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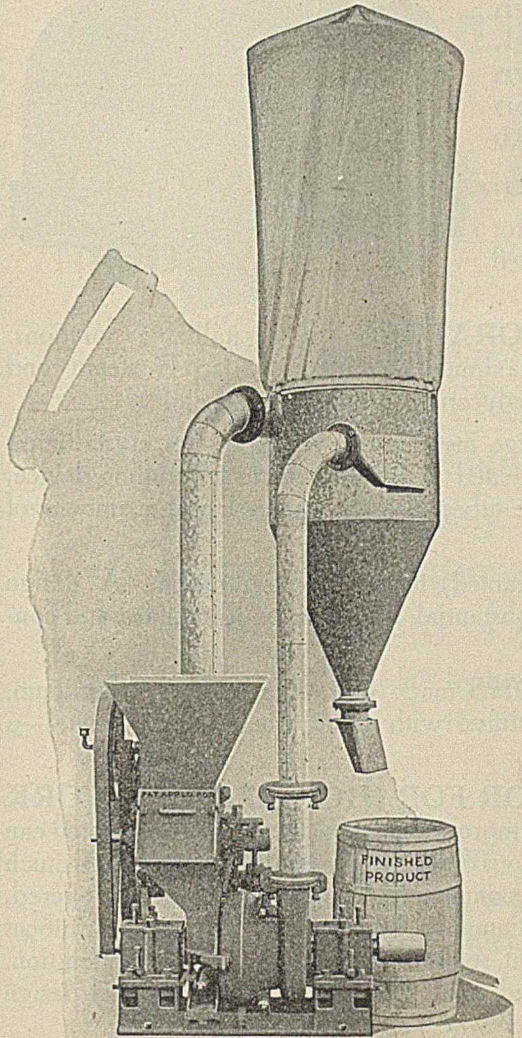

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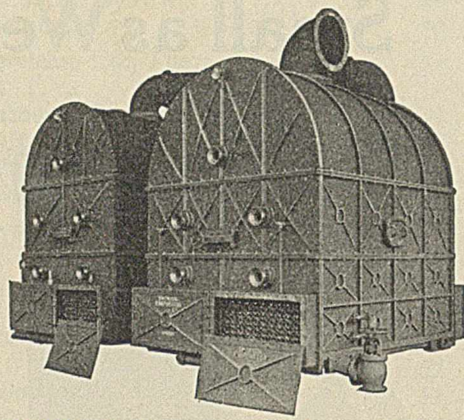

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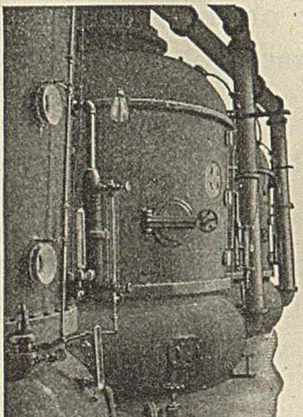

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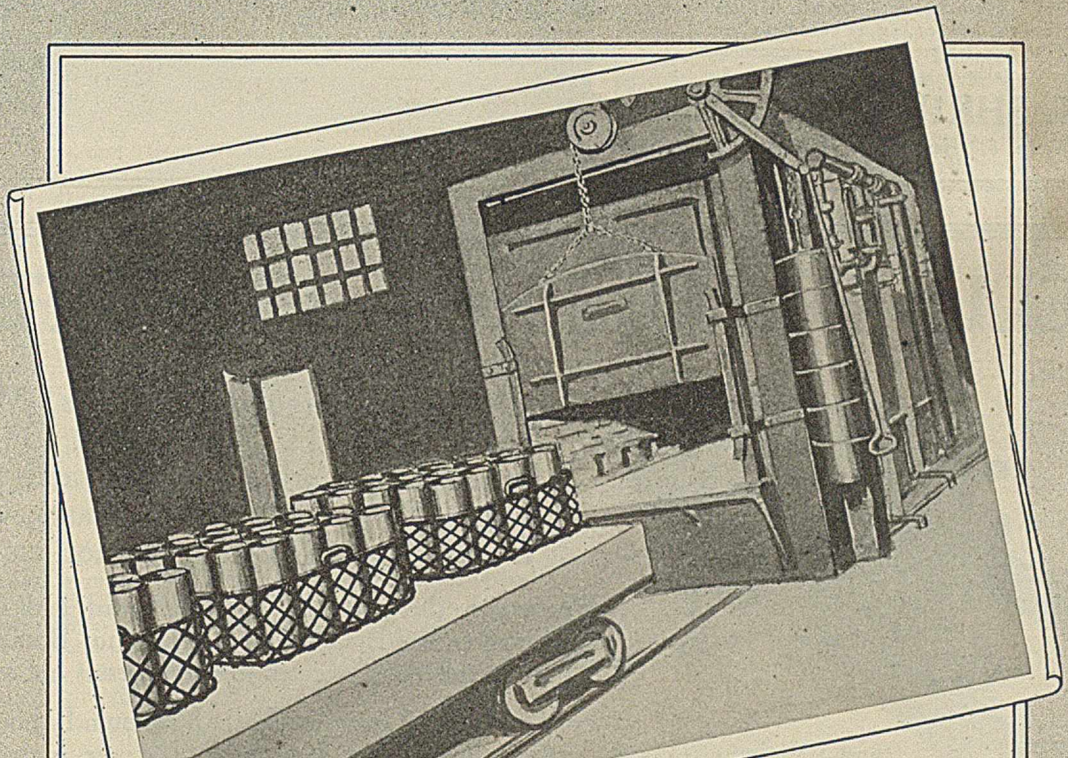

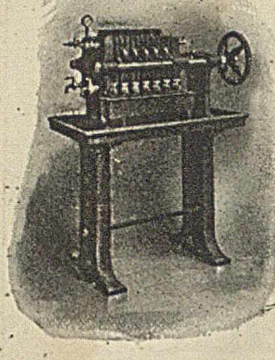

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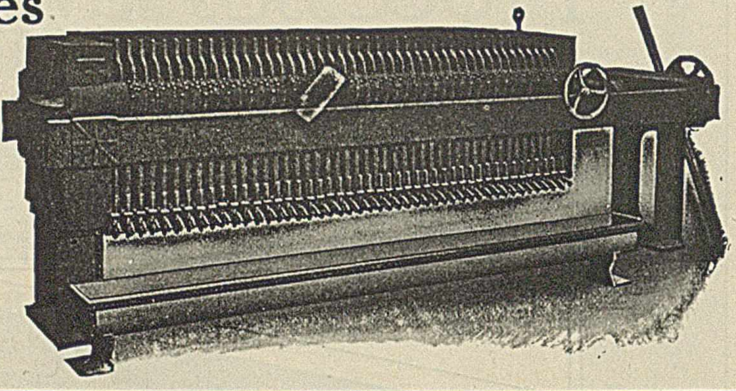

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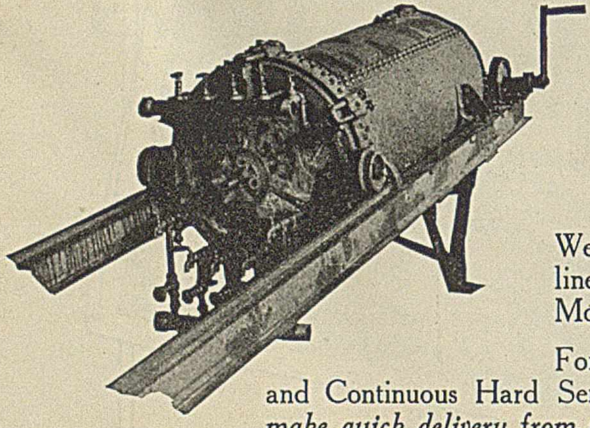

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FOREST PRODUCTS SYMPOSIUM

Papers presented at the 51st Meeting of the American Chemical Society, Seattle, August 31 to September 3, 1915

THE CHEMICAL ENGINEERING OF THE HARDWOOD DISTILLATION INDUSTRY

By James R. Withrow

It would require more time than we have at our disposal to go into the details of the chemical engineering of the hardwood distillation industry. They will be apparent to a certain extent in the accompanying lantern slides, though many interesting points must not even be mentioned. General features will be pointed out sufficiently to give a broad survey of the problems involved in plant operation.

The industrial demand which the hardwood distillation industry meets is sufficiently indicated by the mere mention of acetic acid, methyl alcohol, acetone, chloroform, and formaldehyde, without burdening you with statistics of their production, consumption, exportation and importation. The number of industries and objectives in the arts which benefit by the availability of these substances is surprisingly great, though not all of them are exclusively the products of this industry.

DEVELOPMENT

The development of the industry appears to have been very simple. From the earliest times charcoal appears to have been used in the arts and especially in metallurgy. That liquid products could be formed, during the charcoal production, was very early observed. Uses were gradually found for these products and in course of time a demand was created for them with the result that wood carbonization was largely changed in method, so as to improve the yield of the desired by-products. The historical development of this industry is one of the most interesting in the annals of Industrial Chemistry, because it illustrates so well the battledore and shuttlecock fortune of so many of these industries. So often the by-product of to-day becomes the main product of to-morrow; and the new industry, which appears the worst competitive enemy of one industry, becomes the greatest blessing of the old industry in the next decade. The liquid products accompanying charcoal production are said to have been recovered by the ancient Egyptians and utilized by them in embalming the dead. Pliny mentions their recovery in Syria and Theophrastus in Macedonia. As chemical knowledge in those days appears to have been very limited and engineering skill as much so, the recovery of liquid products must have been trifling and it was many centuries before chemical knowledge made possible and the arts demanded intelligent development. Notwithstanding the later development of the engineering as well as chemical side of the industry, the essential features of primitive wood carbonization have come down practically unaltered to the present day. This primitive charcoal burning is still practiced wherever wood is plentiful in Russia, Scandinavia, Austria-Hungary, Germany (Westphalia and the Harz), New Jersey, Maryland, etc.

In spite of various uses to which they were put in early times no real appreciation of the value of the liquid substances known to be formed during carbonization of wood came until the dawn of modern chemical investigation. Lack of sufficient fundamental chemical knowledge was largely responsible for this. For instance as early as 1658 Glauber identified the so-called "burnt-wood acid" with acetic acid, yet not until 1802 was it identified with the acetic acid of fermentation, by Thenard. In 1661 Robert Boyle discovered wood-spirit in wood distillate, yet not until 1812 did Taylor notice its analogy to grain alcohol. The true character of this "spirit" was established only after a series of investigations by Colin, Dobereiner, Gmelin, Liebig, Sweitzer and especially Dumas and Pèligot, ending about 1835. As a result of these investigations together with those on the composition of the wood-tar and wood gas, there came an increased effort to recover such of these by-products as were suitable for use in the arts in general, as well as in the organic chemical industries that were at this time struggling for a footing upon the investigations of the great masters of our science. The first step in this revolution was the use of by-product kilns and retorts, in place of the old sod covered pits or heaps, for carbonization. It would seem that from this point, at least, the development of the industry should have been consistent and perhaps rapid but this was not the case.

Apparently the first product to have been systematically recovered was the wood-gas. This was first used in England for illuminating purposes somewhat prior to the chemical developments just mentioned. The honor, however, for the exhaustive investigation of the illuminating, heating and power value of this gas, belongs to the Frenchman, Phillip Lebon.

The weakly illuminating wood gas was soon outclassed, however, by the development of coal gas production and the whole hardwood distillation industry had a precarious existence during a large part of the last century even in spite of the fact that a satisfactory method of purifying the crude acetic acid had been devised as early as 1824 by Mollerat and Jasmeyer. The products of the destructive distillation of coal, such as coke, illuminating gas, tar and tar-distillates were sharp competitors of the wood products. Yet when the wood industry seemed about to be relegated to the primitive woods, the sudden development of the coal-tar color industry started a demand for acetic acid, methyl alcohol and acetone which has since been augmented by the demand for the same products in the production of celluloid and smokeless powder as well as many synthetic organic substances used in pharmacy and the arts. The demand for methyl alcohol within a few years after Perkins' discovery of aniline dyes became so great that the price was enormous. It is not surprising, therefore, that at that time in England wood was distilled with the sole object of getting wood alcohol. A like situation is said to have arisen at one time in benzol manufacture, coal having been for a while distilled with the sole object of obtaining benzol for aniline production.

These general statements regarding the development of the industry bring us to the present situation where we recover mainly the primary products wood gas, raw liquor (pyroligneous acid), tars and oils, and charcoal. It should be noticed in passing that while most engineering skill is a matter of quite recent development and chemical engineering is perhaps the most recently developed phase of engineering, yet it has been able reasonably to keep pace with chemical development in this industry at least, and this may be due largely to the fact that the development of engineering methods and the understanding and fabrication of engineering materials of construction developed along with the science of chemistry and are somewhat dependent upon it.

THE PROBLEM OF THE INDUSTRY

Broadly speaking, the problem of the hardwood distillation industry is the complete utilization of the weight of the original wood as far as this is possible by economical chemical engineering manipulation, for the production of the products which it will yield of main industrial use or value, *i. e.*, charcoal, acetic acid and methyl alcohol.

Economical chemical engineering involves the use and sequence of such operations as will give the maximum financial return, per unit of raw material consumed, consistent with a proper balance between low cost of operation, low initial cost of plant and highest speed of turn over (processing raw material).

When wood is heated sufficiently in the atmosphere it tends to burn of course to residual ash, steam and carbon dioxide, for the most part. If the air is partially excluded we obtain residual charcoal and volatile products varying widely in composition, depending upon the extent of air exclusion, from the condition of almost complete combustion of volatile products, to practically entire elimination of combustion or atmospheric oxidation when air is excluded as completely as possible. The problem of the industry may be met therefore by heating the wood sufficiently to carbonize it and in such a way as to eliminate as far as possible any atmospheric oxidation of the volatile products, and at the same time handle the distillates in such a way as to minimize refining or purification difficulties.

Modern chemical engineering skill has met the conditions of reasonably efficient solution of the problem with fair success as is shown by the extent of the industry[1] and without doubt it will advance still further.

Every problem, however large, is merely a series of smaller sub-problems and each of these in turn is often resolvable into still other problems. In studying the problem of any chemical industry it is natural to divide it as far as possible into:

1—The nature of the demand or market for the products, or objectives of the industry;

2—The chemistry involved;

3—The operations or engineering involved.

The chemistry of the hardwood distillation industry finds its basis in the phenomena attending destructive distillation. Much more should be known about destructive distillation than *is* known. Our concern at this time, however, is with the engineering problems involved in the utilization of this chemistry. Suffice it to say in the matter of the chemistry involved, that the products of heating the wood, as is usually the case in destructive distillation, are gaseous, liquid (condensate) and solid (residual charcoal). The gas as a whole is combustible. The liquid is acid and in two (or three) nearly non-miscible phases—tar (and oil) and dilute alcoholic acetic acid, with dissolved tar and other substances.

CHEMICAL ENGINEERING OPERATIONS

The engineering operations of the industry group themselves mainly into four divisions as follows:

I—Preliminary handling of raw material.
II—Destructive distillation proper or so-called primary distillation.
III—Treatment of the liquid distillate or secondary distillation.
IV—Refining operations and derived product production.

The latter is not generally connected with what is known as the Crude Hardwood Industry and will, therefore, be indicated with great brevity.

The engineering operations themselves are as follows:

I—Preliminary Handling of Wood
  1—Harvesting the timber $\left\{\begin{array}{l}\text{Cutting}\\ \text{Splitting}\end{array}\right.$
  2—Seasoning, transportation and storage
II—Primary Distillation (*Destructive distillation, proper*)
  1—Loading oven cars
  2—Charging and sealing ovens
  3—Firing ovens (*Distillation*)
  4—Condensation and fuel gas recovery
  5—Pulling ovens (*Discharging hot charcoal cars*)
  6—Cooling charcoal
  7—Pulling charcoal coolers
  8—Ageing charcoal
  9—Screening charcoal
  Final Product: Charcoal for the Market
III—Secondary Distillation (*of condensate obtained in II, 4*)
  1—Settling "Raw Liquor" (*condensate II, 4*)
  2—Separation of tar (and oil) from "Raw Liquor" (*continuous decantation*)
  3—Redistillation of settled tar (*for further recovery of "Raw Liquor"*) from copper or wooden stills.
  4—Condensation of acid distillate [*Oil added to "Boiled Tar," water added to original "Raw Liquor" (condensate II, 4)*]
  5—Redistillation of settled "Raw Liquor" (*for further tarry bodies elimination*) from continuous copper stills
  6—Condensation of "boiled liquor"
  7—"Mixing" "boiled liquor" with lime (*Neutralization*)
  8—Distillation of neutralized "boiled liquor" in lime-lee still (iron)
  9—Condensation of weak alcohol-acetone
  10—Settling or filtering lime-lee still residue (*aqueous calcium acetate*)
  11—Evaporation and crystallization of lime-lee residues
  12—Drying (sacking, weighing, analysis) crystallized acetate of lime
  Product: Gray Acetate of Lime (80 Per Cent) for the Market
  13—Redistillation of weak alcohol-acetone for oil separation and concentration (*Burcey pans, fractionation*)
  14—Condensation of alcohol-acetone in fractions
  Product: Crude 82 Per Cent Alcohol-Acetone for Market or Refinery
IV—Refining and Derived Product Manufacture
  (*a*) *Alcohol:*
    1—Chemical treatment of crude alcohol (NaOH or H_2SO_4)
    2—Distillation and condensation for elimination of chemicals, etc.
    3—Fractional distillation in column still
    4—Condensation with "heads and tails" elimination
    Product: Refined Methyl Alcohol and Acetone-Alcohol for Market
  (*b*) *Gray Acetate:*
    1—Distillation with sulfuric acid (or HCl)
    2—Dust elimination from vapors
    3—Condensation of acetic acid (*Commercial*)
    4—Refining by redistillation and fractionation after chemical or electrical treatment
    Product: Refined and Concentrated Acetic Acid for Market
  (*c*) *Gray Acetate:*
    1—Destructive distillation
    2—Dust elimination from vapors
    3—Condensation of crude acetone and oils
    4—Separation of oils from acetone
    5—Chemical treatment for acid elimination
    6—Redistillation for "white oil" elimination
    7—Fractional distillation of acetone
    8—Condensation
    Product: Acetone for the Market

Each and every one of these operations calls for its own special form of apparatus or construction and modifies to a varying extent the design and operation of the plant. They are the essential operations or chemical engineering proper which must be carried out to secure the results obtained in first-class practice. There has been indicated also, in a limited way, how some of these operations must be carried out. Sufficient preliminary discussion of the development of the industry has been given to illustrate the connection which appears to have existed between the development of both chemical knowledge and engineering skill, and the growth and development of this industry.

In concluding I must express my indebtedness to Mr. Edward H. French, to whom I owe my first opportunity (a number of years ago) of working upon the problems of this industry, upon which he was himself engaged, and who has been of much assistance in securing plant photographs from which slides have been prepared.

Ohio State University, Columbus

WHAT CHEMISTRY HAS DONE TO AID THE UTILIZATION OF WOOD

By S. F. Acree

An attempt to discuss fully the influence of chemistry on the utilization of waste wood leads so deeply into practically all fields of chemistry that a paper of this kind must necessarily be limited to a small number of topics. Attention is given, therefore, to those phases of this subject which are of most importance commercially and which seem to show greatest promise for the future. The number of eminent chemists who have contributed to the few subjects to be discussed is so large that it is impossible to treat their individual researches as they deserve. Such being the case, a brief mention here and there of a few investigations by the Forest Service, with which the writer

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[1] This Journal, 7 (1915), 47.

is personally familiar, will not be understood as a failure to appreciate the work of others.

The technical processes involved in the apparently simple distillation and extraction of woods lead both into the abstruse problems of thermodynamics and into the most refined technique of organic chemistry, and advances in these investigations have been described by Klason, Wislicenus, Klar, Withrow, Palmer, and others. The distillation of waste hardwoods is now one of the most important industries in this country and has reached a high state of efficiency in the hands of Messrs. Stevens, Quinn Brothers, Troy, Gaffney, Clawson and the Cleveland Cliffs Chemical Company. There are over 100 plants in operation, in which about 1,200,000 cords of wood are distilled annually. The chief products obtained are charcoal, acetic acid, methyl alcohol, wood oils, and wood tars, and these are so valuable that extended researches have been made by the Forest Products Laboratory to increase the yields. The quantitative studies on the regulation of the reactions have resulted in improved processes and increased earnings. Charcoal is the least valuable by-product of the wood distillation industry, but is used in large quantities as a household fuel, in decolorizing grain alcohol and other liquids, in the manufacture of gunpowder and lubricants, and in the manufacture of fine charcoal steel. The acetic acid is utilized for the production of such substances as mordants for dyes, acetanilide, and sugar of lead. Acetone is produced by the distillation of calcium acetate and is used to the extent of 3,000,000 gallons annually in the manufacture of denatured alcohol, chloroform, photographic films, celluloid, and, especially, explosives.

Wood alcohol is one of the most striking illustrations which we have today of the value of chemistry and chemical methods in the purification and utilization of a waste product. When Mr. E. B. Stevens, President of the Wood Products Company of Buffalo, N. Y., constructed his first still out of a tin can and attempted to purify the evil-smelling distillate from a near-by wood distillation plant, he little realized what a tremendous future awaited his efforts as an enthusiastic boy of seventeen. Today, crude 82 per cent wood alcohol is changed by simple chemical and physical methods into a product which has a purity of 99.95 per cent. This substance is used all over the world and new fields are being discovered for its utilization. The 10,000,000 gallons produced in this country are used for making celluloid and similar products, dyestuffs, denatured alcohol, photographic films, formaldehyde, artificial leather, varnishes, shellacs, artificial rubber, and other substances too numerous to mention, in whose manufacture and use hundreds of millions of dollars and large numbers of men are employed. European countries use nearly as much wood alcohol as the United States but chiefly for denaturing ethyl alcohol, for the manufacture of formaldehyde, for the methyl group of aniline colors, and for other purely chemical purposes. There is at present no substitute for wood alcohol in the preparation of the methyl groups of various dyes, especially the violets and blues, and hundreds of millions of dollars are invested in the dyestuff business and in the wool, cotton, linen, and silk cloth industries and others using these colors. These dyes are used today in many cases of disease in staining tissues and bacteria, thus allowing medical men to make a proper diagnosis.

Wood alcohol acts as a poison when taken internally, or when its concentrated vapors are inhaled in enclosed spaces, and sometimes produces blindness and death under these conditions. The wood alcohol manufacturers themselves condemn its use as such in any article of food, drink, or medicinal or toilet preparation. When, on the other hand, these objectionable properties are destroyed by chemical changes, it becomes one of the substances necessary for the production of other products for the protection of the health of the community. Pure methyl (wood) alcohol is the only substance which can be converted on a commercial scale into formaldehyde, which is used universally for disinfection against such contagious diseases as smallpox, scarlet fever, diphtheria, tuberculosis, infantile paralysis, and spinal meningitis in its epidemic form. It should, furthermore, be pointed out that many native and alien crop diseases are becoming epidemics in this country and destroying yearly from ten to twenty million dollars worth of foodstuffs, besides infecting the farming lands and rendering them far less valuable for the production of certain crops. The United States and State Governments prescribe formaldehyde as the disinfectant for the seeds, which are sterilized before planting. By this disinfection crop losses, due to certain fungi, can be greatly reduced. Formaldehyde is practically the only efficient disinfectant which can be legally employed in embalming the dead and preventing the spread of contagious diseases causing their deaths. Formaldehyde and phenol are the basis of the manufacture of bakelite which is now used in tremendous quantities in large chemical apparatus in certain industries, and is finding wide use as insulating material in the electrical world. Formaldehyde is, furthermore, used in large quantities in the preparation of various dyes discussed above.

Another by-product from wood distillation, which has received little consideration up to this time, is the hardwood tar. This substance has a large percentage of derivatives which should find wide use in chemical industries. Already we find small quantities of these tars in use as wood tar creosotes for the preservation of wood, and the insufficient production of coal-tar creosote in this country will, without doubt, enable wood tar creosote to come into its own, especially as it has been found to have a high toxicity toward certain fungi.

In the field of distillation of softwoods, we find that some of our most important commercial products are obtained by this combination of physical and chemical methods. The chief products are turpentine, pine oils, tar oils, rosin and charcoal. The turpentine is used chiefly in the production of paints, varnishes, and shellac. The pine oils are used to a large extent as solvents and as medicine. The tar oils are used for treating such things as rope and fish nets in order to preserve them in salt water. The rosin is used in making soaps, paper sizing, varnishes, and shellacs. Charcoal is used chiefly for fuel.

The value of the paper and pulp industry in this country is apparent immediately when we think of the benefit derived from the tremendous number of books, periodicals and of newspapers. Wood pulp is used for making men's summer clothes, papers of all kinds, binder twine to replace sisal, sacks, filter papers, and rugs. It is used for making fiber vessels, vulcanized fiber for electrical insulation, and is now the basis of viscose which is finding a wide field of application. Viscose is colloidal cellulose which can be made into "fiber silk" thread and sausage coverings. The thread can be woven alone, or in combination with cotton or silk, into all kinds of fabrics, such as artificial silk socks, neckties, and cloth, all of which can be dyed beautifully in any color. Surgeons' thread, and erasers of this material are cheap and efficient.

Cellulose can be made in a very pure form from certain woods and has been used extensively in explosives. Not only is it converted directly into nitrocellulose but we find that wood flour is used very extensively as an absorbent in the manufacture of dynamite. The very finely divided wood flour absorbs 60 per cent of nitroglycerine and is more efficient than kieselguhr. This wood flour is also used in making certain grades of linoleum and is much cheaper than cork.

Turning now to the dyestuffs, we find that the aniline industry of Germany has not entirely driven vegetable colors from the market. A number of West Indian and South American woods, such as the fustic, logwood, Brazil wood, and peach wood, are imported into this country and the Forest Products Laboratory has recently been instrumental in calling the attention of manufacturers to the fact that Oklahoma and Texas contain large quantities of osage orange[1] which can be used very cheaply as a source of a valuable yellow dye for textiles and leather. The enormous leather industry is dependent not only upon the use of vegetable dyestuffs but especially upon the tannins obtained from hemlock, oak, chestnut and other materials. Certainly the preparation of tanning material involves the most delicate physical and chemical, as well as biological, operations, as has been shown by the work of Proctor, Stiasny, Levi, Balderston and others.

That the production and consumption of ethyl alcohol in the United States is an important activity is emphasized by the fact that the taxes on distilled spirits and fermented liquors amounted in 1913 to $223,000,000, which was over 65 per cent of the entire internal revenue of the country. It will be recalled that over 100 years ago Braconnot carried out an investigation in which wood was hydrolyzed by acids into glucose, which was then fermented into ethyl alcohol. In recent years, this problem has been studied energetically by Simondson, Classen, Cohoe, Ewen and Tomlinson and others, and four plants were erected in the United States. Three of these failed but the experience gained thereby and the research work on this problem by the Du Pont de Nemours Powder Company and the Forest Products Laboratory have now made the outlook for this industry very bright. It is believed that a plant could now be constructed under proper conditions of location, transportation, and other such factors, which would make possible the manufacture of ethyl alcohol from sawdust at a price which would allow it to compete with the production of ethyl alcohol from molasses. Especially important in this connection is the discovery by the Forest Products Laboratory that western larch contains about 10 per cent of a galactan yielding only galactose. If fermentation methods can be devised for the commercial conversion of galactose into ethyl alcohol, the western larch butts, which are at present waste material, will become a very cheap source of alcohol. The increased efficiency of alcohol at high pressures in internal combustion engines may thus place this material in a position where it can compete with gasoline when this becomes scarcer.

The preservation of woods in railroad ties, telegraph poles, heavy bridge timbers, structural timbers, and in houses, is one of the most important phases of forest conservation before the public today. In this preservation over 100,000,000 gallons of coal-tar creosote, water-gas-tar creosote, and mixtures of these chemicals with petroleum residues are used. Aqueous solutions of zinc chloride, sodium fluoride, copper sulfate, and many other inorganic chemicals are also used very widely. Millions of dollars are thus involved in an industry whose business it is primarily to preserve this investment. It follows then that chemical methods must be developed for the analysis of the creosotes, and that chemical studies should lead to the manufacture of better materials at lower cost. The Forest Products Laboratory has made investigations on the analysis of creosotes and the isolation of their many constituents, together with tests of the toxicity of these compounds toward wood-destroying fungi, and these researches have shown that great improvements could easily be made in the preservation of wood by the use of better specifications for the materials and methods of treatment.

In the above paragraphs, only a few of the industries involving the utilization of chemical methods in the use of waste wood have been discussed. But daily the problems along these lines are being extended and the future will probably show that the application of chemistry to the utilization of forest products extends into nearly every field of human endeavor.

FOREST PRODUCTS LABORATORY AND UNIVERSITY OF WISCONSIN
MADISON

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[1] THIS JOURNAL, 6 (1914), 462.

THE TANNIN CONTENT OF PACIFIC COAST CONIFERS

By H. K. BENSON AND THOS. G. THOMPSON

The tannin extract industry of the United States utilizes for its raw material chestnut wood and the bark of the Eastern hemlock and the chestnut oak. From these species a standard extract containing 25 per cent tannin is manufactured, annually amounting to over 200,000,000 lbs. An equal or greater quantity of extract is either imported, or made from materials imported, of which quebracho is of chief importance. In view of the large sources of available supply of tannin-containing materials on the Pacific Coast, a study was made of the tannin content of Western hemlock, Douglas fir and Western spruce.

WESTERN HEMLOCK

The Western hemlock extends from Alaska to Marin County, California, and eastward, in some places, as far as Montana, thriving in the cool, moist climate characteristic of western Washington and Oregon. It grows along with spruce, cedar and fir, often being left as useless by the timber cutters. An excellent example of the distribution of these trees is found in a recent survey of the merchantable timber of the Olympic National Forest. It was found that out of this forest, containing 25,500,000,000 ft. of timber, 35 per cent was Western hemlock, 37 per cent Douglas fir, and the remainder spruce and cedar.

The bark of the Western hemlock is thinner than that of the Eastern hemlock, but, as is commonly known, it contains a higher tannin content. This fact was first brought to the attention of the Washington public in 1893 when several samples of the bark were submitted to Dr. H. W. Wiley, who reported the following analyses:

SAMPLE:	Ashford	Enumclaw
Moisture	10.55	10.01
Total solids	24.03	22.16
Soluble solids	22.59	20.58
Reds	1.44	1.58
Non-tannins	6.59	5.76
Available tannins	16.00	14.86
Woody fiber	65.42	67.83

The first results that were published[1] calling attention to the differences in percentage of the tannin content of the Western and Eastern hemlock were the following analyses made by H. G. Tabor of hemlock bark from trees in Washington, Pennsylvania and Quebec:

SOURCE:	Washington	Pennsylvania	Quebec
Tannin	17.04	13.28	10.16
Non-tannin	6.40	7.52	4.56
Reds	1.56	3.48	1.92
Woody fiber	75.00	75.72	83.36

Western hemlock bark has been used as the exclusive tanning agent in at least one tannery in Washington for a number of years. This concern manufactures chiefly skirting leather for saddles. Its larger use has been prevented by the lack of tanneries, due to economic reasons, rather than unsuitableness. The prevailing cost of hemlock bark f. o. b. tannery has been $11.50 per cord.

EXPERIMENTAL

Inasmuch as few references to the tannin content of Douglas fir and Western spruce could be found, and as far as is known no effort has ever been made to demonstrate the tannin content of sawmill waste from these species, an experimental study was undertaken.

PREPARATION OF SAMPLE—The sawmill waste taken for analysis consisted of bark, slabs, and sawdust from the crosscut saws. Samples of 100 lbs. were taken for the bark, from 100 to 150 lbs. for slabs, and about 25 lbs. for the sawdust. The bark and the slabs were each reduced in a Mitts and Merrill chipper and the chips quartered to obtain an average sample. Before subjecting the sample to analysis it was further reduced to a fine powder in a Grumbaugh mill.

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[1] *Hide and Leather*, June 24, 1893.

EXTRACTION OF SAMPLE—The apparatus used for extraction is shown in Fig. I. From 50 to 75 grams of the sample were placed in the percolator A. The flask B contains the water for extraction. On boiling, the steam flowed through the tube C, and entered the percolator where partial condensation occurred in D. Boiling water continually drops upon the material in A until it reaches the height indicated by E, after which it is returned to the boiling vessel B. To prevent rifts or channels in the sample, the latter was mixed with broken glass before extraction, and to prevent particles of bark or wood from siphoning over with the liquid, a small perforated porcelain disk and glass wool were placed over the outlet tube. It was found advisable to use as large an outlet tube for the condenser as possible and to spread glass wool or wire gauze over the top of the sample in the percolator. Both the extraction and the analysis were made in accordance with the Official Method of the American Leather Chemists' Association.

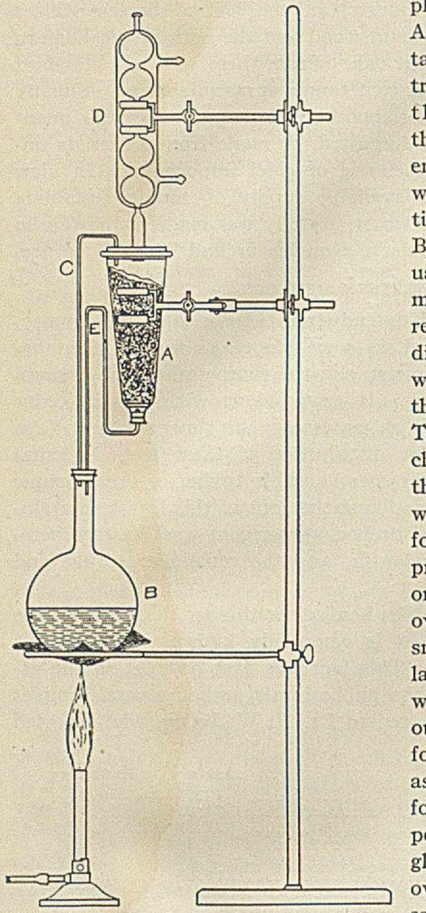

Fig. I

TANNIN CONTENT OF DOUGLAS FIR

The extract obtained from the Douglas fir was reddish brown in all cases except from the freshly cut bark which was yellow-brown and contained a yellow precipitate. The logs from which the samples were obtained had been cut for a period of eight months and during a portion of this time they were in water. However, the bark of the Douglas fir is so thick that there is probably very little loss of tannin by leaching in floating the logs to the sawmills. From the yields given in Table I, it is shown that the cambium layer, which is dark brown in seasoned wood and yellow-brown in freshly cut wood, contains most of the tannin. When a sample of bark is taken from a seasoned log not all of the cambium layer is taken. The slab, however, contains the cambium layer in its entirety with a relatively small quantity of wood in addition to the bark proper. The analyses of various samples appear in Table I, the results being reported on the dry material.

TABLE I—TANNIN CONTENT OF DOUGLAS FIR

SAMPLE:	Sawmill bark	Sawmill slab	Fresh bark	Cambium layer	Sawdust
Total solids	14.75	14.92	11.31	21.96	5.78
Soluble solids	13.36	13.02	9.36	19.28	4.40
Reds	1.39	1.90	1.95	2.68	1.38
Non-tannins	7.02	7.10	6.74	9.36	3.34
Tannin	6.34	5.92	2.62	9.92	1.06
Moisture	9.06	6.91	14.27	20.59	15.51

TANNIN CONTENT OF WESTERN SPRUCE

The extract from spruce was a clear brown in color. The bark is much thinner than in fir. On account of remoteness of the mill from which the samples were obtained no history of the logs is available but it is probable that they were floated in water a longer period than in the case of the fir logs. While check analyses were made, time did not permit as complete an examination as for fir. The results of the analysis, reported on a dry basis, are given in Table II.

TABLE II—TANNIN CONTENT OF WESTERN SPRUCE

SAMPLE:	Sawmill bark	Sawmill slab
Total solids	12.83	11.25
Soluble solids	12.30	10.38
Reds	0.53	0.87
Non-tannins	6.42	6.79
Tannin	5.88	3.69
Moisture	15.23	9.15

TANNING VALUE OF FIR EXTRACT

The fir chips from the bark and slabs not required for samples were extracted by the West Coast Tanning Company, Seattle, and several skins tanned in the resulting extract. A very desirable leather was produced, having a color similar to that obtained from oak tannin.

COMMERCIAL POSSIBILITIES FOR FIR EXTRACT

When it is remembered that the tannin content of chestnut wood is 6.62 per cent, it appears that the low cost of fir slabs containing an average of 5 per cent tannin should make the use of fir slabs a possible raw material for the tannin extract industry. It is known to the writers that fir slabs can be purchased from some of the mills at as low a cost as 75 cents per cord. In mills located in large cities, where they are sold for fuel, the cost is usually \$1.50 to \$2.00 per cord. Compared with Western hemlock bark at \$11.50 per cord, it is found that three cords of fir slabs will yield as much as one cord of hemlock bark but at less than one-half the cost of the bark.

CONCLUSIONS

I—The tannin content of fir sawmill waste is above 5 per cent while that of spruce is above 4 per cent.

II—It is believed that fir waste is a suitable material for use in the tannin extract industry.

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YIELD OF BY-PRODUCTS FROM DESTRUCTIVE DISTILLATION OF SOME WESTERN CONIFERS

By H. K. BENSON AND MARC DARRIN

The utilization of forest wood and mill waste by destructive distillation has been attempted in the Pacific Northwest on a commercial scale, but without success. It has accordingly been thought worth while to undertake a study of the yields obtained from various species and from various classes of waste wood from a given species. For this purpose an experimental plant for operation on a semi-commercial scale was erected, coöperatively, by the United States Forest Service and the University of Washington. The design of this plant has already been described.[1]

This plant was operated by chemists detailed by the United States Forest Service, in coöperation with the University of Washington. This paper presents a résumé of results obtained in a series of runs, part of which were made by G. M. Hunt, and part by Marc Darrin, chemists in Forest Products, United States Forest Service.

SPECIES AND CLASSES OF WOOD USED

For Douglas fir three classes of wood were distilled: (1) common run mill waste; (2) selected mill waste; (3) common run stump wood. In the case of the Western yellow pine both common run stumps and common run mill waste were distillled while for Western hemlock only the common run mill waste was used for distillation. Before final and conclusive results covering the Western conifers can be obtained, similar studies of selected Western yellow pine and of spruce should be made.

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[1] THIS JOURNAL, 5 (1913), 935.

METHOD OF OPERATION

The capacity of the retort is about one-half cord. For distillation, the wood is cut into 2 ft. lengths, measured, weighed, and loaded vertically into three retort buggies. These are run into the retort, which is fired with heat control for 12 hours. The temperature at various points of the retort is measured by four thermo-couples. Water manometers are used to ascertain pressure conditions. The rate of distillation is determined by measuring in a graduate the quantity of distillate per minute. The gross distillate from the retort is redistilled in a copper still heated with steam in coils at 125 lbs. pressure. The liquids and gases from distillation flow from the bottom of the retort into the tar separator, from which the retort tar is drawn. After passing through the cold water condenser the fixed gases pass through a rotary pump which maintains the proper pressure in the retort and also acts as a mechanical separator of the tar globules in the gas. This tar is removed by means of a trap attached to the pump. From the rotary pump the gas passes through a 60-light meter for measurement, and thence into a holder.

PRODUCTS OF DESTRUCTIVE DISTILLATION

"TURPENTINE AND LIGHT OIL" are made to include all oils of a turpentine character. While much of this fraction is not pure turpentine, the general uses of this fraction in paints and for other technical purposes justify the grouping of these oils under one class.

"TAR" includes both the yields of the so-called retort tar and still tar, *i. e.*, residues from still. These tars in distillation yield about a third of their weight of tar oils, which consist principally of wood creosote, fir oil, some turpentine, and other wood oils. The residue, after removal of tar oil, is a heavy black pitch. The retort tar is heavier and darker than the still tar and contains a lower percentage of the tar oils. The gas tar from the rotary pump is included in the yields of still tar. It is very light and fluid and contains a high percentage of tar oils.

"WOOD ALCOHOL" was measured by means of a Westphal float attached to an analytical balance. This method computes the acetone as wood alcohol. In the case of the Douglas fir, the proportion of acetone in the wood alcohol is about 20 per cent.

"ACETATE OF LIME" was computed from a titration of the aqueous distillate with potassium hydroxide, with phenolphthalein as the indicator.

"CHARCOAL" contains from 80 to 90 per cent fixed carbon, and 1 to 2 per cent ash.

The "GAS" was measured from the time that a small pilot burner in the main caused continuous ignition. A uniform sample from this point in the run was obtained from the holder and its heating value determined in a Sargent calorimeter. The results of these determinations show its heating value to be a little below that of commercial illuminating gas.

DOUGLAS FIR COMMON RUN MILL WASTE

Douglas fir common run mill waste consists chiefly of slabs, edgings, and some inside wood. This wood was taken just as it left the saws and was passing up the conveyor. It was in no way selected. In fact pitchy pieces were discarded as not of an average character, in order that the results obtained might be of a conservative nature. Altogether on this class of material, 21 runs were made. These consisted of four or five runs from four different sections of the state. There was found, however, so little difference in the yields from the various localities, that the tests on the remaining conifers were made only on species from one locality.

The average amount of common run Douglas fir mill waste for the four principal mills in Seattle is 32.2 cords per day per 100,000 feet cut.

DOUGLAS FIR SELECTED MILL WASTE

Douglas fir selected mill waste consists chiefly of inside wood, more or less of a split and shaky nature around the pitch seams. The fact that the pitchy mill waste, in the case of Douglas fir, is of this split character, is the probable cause that this lumber is not suitable for any commercial use as lumber, whereas, in the case of Western yellow pine, it was found that most of the pitchy material occurred in hard inside material which is suitable for some commercial uses. The raw material was taken from the mill just as it came from the saws, but included only that portion which was especially suitable for distillation because of its resinous nature. In determining whether the material was of a resinous nature or not every stick passing up the conveyor which contained a single seam of pitch on the sides or ends, was taken, care being exercised to insure that an average sample of the selected mill waste was being obtained. The average amount of common run Douglas fir mill waste for the four principal mills, as previously stated, is 32.2 cords per day per 100,000 feet cut. Of this amount 13.3 per cent consisted of the selected material. This amounts to 4.3 cords per day per 100,000 cu. ft.

DOUGLAS FIR COMMON RUN STUMP WOOD

Douglas fir common run stumps consist of large, solid pieces. There was little or no rotten material in connection with the stumps, although many of them had been in the ground for a long number of years after the trees were cut. The stumps were taken just as they occurred in the clearing of logged-off land, and were strictly unselected, being an average sample of the common run from the locality. Before the material was selected, the ground was gone over in order to determine if the stumps occurring in that logged-off district could be considered a fair average for the western part of the State. In the selection of the individual stumps none showed signs of pitch on the outside.

WESTERN YELLOW PINE COMMON RUN MILL WASTE

Western yellow pine common run mill waste consists chiefly of small slabs and edgings and practically no inside wood. This material was taken from the mills just as it came from the saws and was strictly unselected, being an average sample of the common run of yellow pine mill waste. It should be especially noted in connection with this material that there is much less pitchy and inside wood than in the case of the mill waste from Douglas fir. This observation is apparently at variance with the well-known fact that yellow pine is especially pitchy, but may be explained as follows:

An examination of the logs as they are being first cut by the band saw shows, in many cases, considerable dark, pitchy wood, especially in the inside of the logs. Instead of cutting out this pitchy material, and making small lumber out of the remaining portion, as in the case of Douglas fir, the entire pitchy portion is cut into heavy planks which are suitable for use on roads, etc.

WESTERN YELLOW PINE COMMON RUN STUMP WOOD

Western yellow pine common run stumps, before being cut up, varied in diameter from 11 to 32 in., and in height of the crown above the roots from 26 to 36 in. The stumps were from a few months' to six years' standing, and in several instances showed signs of decay. After the material was cut into two-foot lengths they could be roughly classified as follows: (1) top crown of stump; (2) lower body of stump; (3) roots. The top crowns of the stumps constitute about 40 per cent of the total, and are similar to ordinary forest cord wood, but are a little more resinous. The lower bodies of the stumps constitute nearly a similar portion, and are for the most part large, knotty pieces which are difficult to handle or split. Little difficulty was found later, however, in charring even these

TABLE I—AVERAGE RESULTS OF TESTS ON VARIOUS WASTE WOODS BY DESTRUCTIVE DISTILLATION

WOOD MATERAL TESTED	Av. wt. of measured cord Lbs.	Turpentine and light oil Gal.	Tar Gal.	Wood alcohol Gal.	Acetate of lime Lbs.	Char-coal Lbs.	Gas Cu. ft.
Douglas fir common run mill waste	3,330(*a*)	3.40	27.80	3.90	75.0	977	...
Douglas fir selected mill waste	4,250(*b*)	10.86	46.37	3.71	74.3	900	...
Douglas fir common run stump wood	3,260(*b*)	5.59	19.88	2.60	55.8	675	6,270
Western yellow pine common run mill waste	2,840(*b*)	4.91	24.80	3.25	73.4	478	6,120
Western yellow pine common run stump wood	2,620(*b*)	6.06	23.05	1.83	60.8	520	3,950
Western hemlock common run mill waste	3,270(*c*)	2.76	21.60	5.00	94.0	938	...

(*a*) Based on 15 half-cords. (*b*) Based on 4 half-cords. (*c*) Based on 5 half-cords.

larger pieces. The roots constitute 10 to 20 per cent of the total, and are for the most part rotten, except in the interior, which, in many cases, is hard and resinous. Only two of the stumps showed signs of pitch on the outside. The stumps were taken just as they occurred in the clearing of logged-off lands, and were strictly unselected, being an average sample of the common run of pine stumps for the Eastern part of the State of Washington.

WESTERN HEMLOCK COMMON RUN MILL WASTE

This material consisted chiefly of slabs, and some edgings and inside wood. It was obtained in the same manner as the Douglas fir common run mill waste.

DISCUSSION OF RESULTS

The results of the runs on the various woods are given in Table I; they show that the heaviest material is Douglas fir selected mill waste, and the lightest, Western yellow pine common run stumps. This difference is due chiefly to the fact that the former consists mostly of inside wood piled up closely with a small percentage of voids, whereas the stump wood can be piled, even after cutting into 2-ft. lengths, in a very irregular manner, with a large percentage of voids. The common run mill waste from the Douglas fir and the Western hemlock is several hundred pounds heavier than the similar material from Western yellow pine. Likewise the fir stumps are heavier than the pine stumps. In this connection it should be remembered that the yields given in Table I are based on the measured cord, as this is the method employed in the ordinary commercial handling of wood, and moreover, as it is this volume factor which limits the capacity of any distillation apparatus.

The yields of gas are available only in three cases, the greatest being for Douglas fir stumps. Although Douglas fir and hemlock mill waste were not tested for the yield of gas, it is possible that the yield may be higher in these cases than those recorded, as it is seen that the yield from the mill waste in the case of the pine is greater than that from the stumps.

CONCLUSIONS

None of the classes of the materials tested gave a higher yield of *all* the by-products than any other class of raw material. The question of *which is the most suitable class of material for distillation*, therefore, limits itself to a function of the market values of the respective by-products, the cost of obtaining the raw material, and the operating expenses.

Although one set of conditions might favor the use of one class of material to the exclusion of all others, still, conditions, such as the cost of raw material, market for charcoal, etc., might be so changed as to reverse this.

Under the present conditions, unless other factors enter into the consideration, such as the increased value of the land, due to the removal of the stumps, the order of commercial suitability for distillation is somewhat as follows:

1—Douglas fir selected mill waste
2—Douglas fir common run mill waste
3—Western hemlock common run mill waste
4—Western yellow pine common run mill waste
5—Western yellow pine common run stump wood
6—Douglas fir common run stump wood

A change in the market conditions, especially for charcoal, or a possible sale of the gas, or a possible utilization of the pyroligneous acid for the production of acetone,[1] or a change in the cost of raw material, may greatly change the order of the commercial value. The value of the Douglas fir selected mill waste, however, is so far advanced over that for the other raw materials tested that there is little doubt it is the most suitable for distillation.

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THE USE OF AMMONIUM HYDROXIDE FOR THE EXTRACTION OF ROSIN FROM WOOD

By H. K. BENSON AND HERBERT N. CRITES

The extraction of rosin from "light wood" in the Southern States is accomplished by first steaming the chips, next covering them with a petroleum distillate, reheating until the distillate begins to vaporize, and then withdrawing the solution of rosin. By subjecting this solution to re-distillation, the solvent is recovered first, and finally the turpentine and pine oil, leaving the rosin as a residue. The main difficulty with this method is the loss of solvent in the cycle, which, according to operators, is as high as 50 gallons per cord of wood extracted.

Various plans have been suggested to overcome this loss. Clopé proposes to use[2] the volatile distillate boiling between 170 and 185° C. obtained from the steaming of wood, as a solvent for rosin. The loss of this solvent is reported at less than 6 gallons per cord of wood extracted, and as it is being constantly produced in the process, no additions from external sources need be supplied. This solvent is, however, a fraction of turpentine generally permissible in commercial grades. It consequently diminishes the yield of turpentine per cord of wood, incurring a financial loss equal to or greater than that prevailing with the petroleum solvent.

To a limited extent a hot bath of some non-volatile or high-boiling material, such as molten rosin, pine tar, pine oil, etc., has been employed but has been abandoned by the commercial plants in which it had been attempted. Recently the use of alkalies such as caustic soda or soda ash has been suggested[3] for the extraction of rosin. The alkali combines with the rosin acids and forms a soluble rosin soap while the turpentine and pine oil may be removed from the solution by distillation with steam. The difficulty in this method lies in the form in which rosin is finally recovered, *i. e.*, as a soap, for which apparently no great demand exists as obtained from wood by this process.

The present investigation was based upon two facts: (1) the instability of ammonium salts when heated, and (2) the completeness of recovery of ammonia as commercially carried on in the coal gas plants of this country.

EXPERIMENTAL

Powdered rosin was added to a 25 per cent solution of warm ammonium hydroxide and formed a thick, light yellow, translucent solution which became quite firm on cooling. It slowly decomposed while standing in the air and on adding heat, the ammonia was driven off, leaving rosin as a residue.

GENERAL METHOD OF PROCEDURE

The next step consisted in the use of resinous fir wood containing from 20 to 30 per cent rosin. The wood was used in

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[1] THIS JOURNAL, 7 (1915), 927.

[2] U. S. P. application, Serial No. 875,463.

[3] U. S. Department of Agriculture, Bureau of Chemistry, *Bull.* 159.

the form of chips (1 in. × 1/8 in. × 1/8 in.), shavings, and sawdust. The general method of procedure was to cover a weighed quantity of wood with a known quantity of ammonium hydroxide. This was heated in pressure bottles to various temperatures for several hours, and while still warm the brown syrupy liquor was poured off and the wood washed with very dilute ammonium hydroxide. The combined liquors were heated, the ammonia passing off and being regained in wash bottles containing cold water. After complete evaporation, a black, tarry mass remained, which was next treated with gasoline and filtered. The filtrate is a solution of rosin while the solid matter upon the filter paper consists of portions of the wood which are also soluble in ammonium hydroxide and which for convenience will be designated as "humus." Upon evaporation of the gasoline, a residue of rosin of fine golden color remains. To ascertain the efficiency of extraction, the extracted chips were dried, finely ground in a Grumbaugh mill and extracted with ether in a Soxhlet apparatus.

TABLE I—PRELIMINARY EXTRACTION EXPERIMENTS

	Conditions of Extraction						Wash Liquor		Per cent Rosin		Efficiency
Exp. No.	NH_4OH used Per cent	Cc.	Time Hrs.	Temp. ° C.	Washing Time	Hrs.	Vol. Cc.	Per cent NH_4OH in	Extracted	Not Extracted	of rosin extraction Per cent
1	25	150	500	18	2	6	150	3	16	12	57
2	25	200	500	18	2	6	200	3	22.8	2	92
3	25	150	500	18	2	5	100	3	12	10	54.5
4	25	90	6	100	1	2	100	5	18	5.6	76
5	3	70	7	100	1	1	80	1	18	9.05	64.5
6	5	70	7	100	1	1	80	1	21.6	5.6	79.5
7 (NaOH)	2.6	90	14	100	1	1	80	0	16	1	94

TABLE II—EXTRACTION OF RESINOUS CHIPS

	Conditions of Extraction				Wash Liquor		Per cent rosin extracted in liquor		Per cent humus extracted in liquor		Per cent rosin	Efficiency
Exp. No.	Liquor used Cc.	Time Hrs.	Washing Time	Hrs.	Vol. Cc.	Per cent NH_4OH in	1st	Wash	1st	Wash	not extracted	of rosin extraction Per cent
1	100	3	1	3	80	5	16.9	1.5	7.7	6.9	7.4	71
2	175	8	1	3	90	1	19.7		21.0		2.2	90
3	200	6	1	3	90	1	15.8		14.9		2.0	89
4	200	5	1	3	60	1	16.4	1.0	10.4	2.2	0.8	95.6

PRELIMINARY EXPERIMENTS

In Table I are given the results obtained from the preliminary experiments. The efficiency of extraction is found by dividing the amount of rosin extracted by the sum of the quantity extracted plus that not extracted. Owing to the manner in which rosin is distributed in Douglas fir, it is difficult to get samples with uniform rosin content. This fact accounts in part for variations in total rosin content. Experiments 1, 4, 5, 6 and 7 were with chips, while in 2, sawdust, and in 3, shavings were used: 25-gram samples were used in each case.

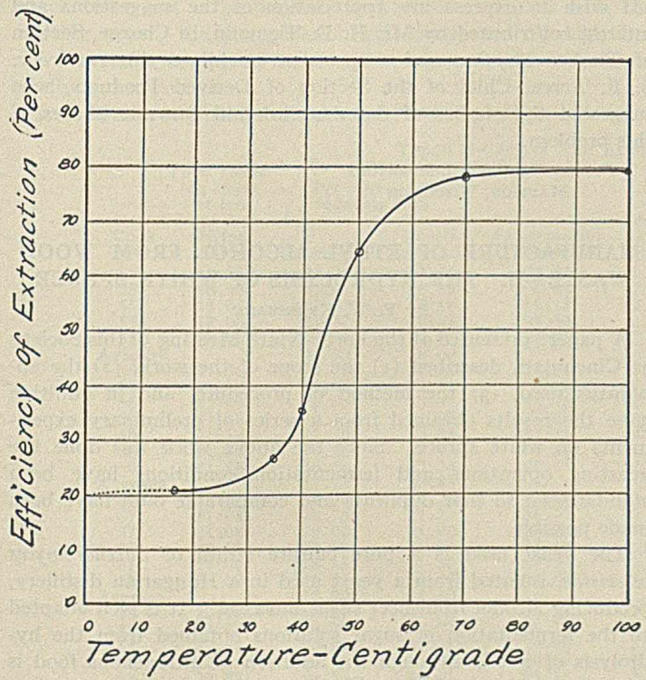

FIG. I—EFFECT OF TEMPERATURE ON EXTRACTION

Experiments 1 and 3 show that extraction of chips and shavings is impracticable at room temperatures while sawdust can be thus extracted. While extraction was not complete in Experiments 4, 5 and 6, the latter shows that a five per cent solution of ammonium hydroxide is equally efficient as concentrated ammonium hydroxide under the same conditions. Experiment 7 was made with sodium hydroxide for comparison. The quantity of rosin extracted in this experiment was obtained by acidifying the sodium resinate solution, drying and weighing the rosin thus obtained. Fineness of wood, higher temperatures (involving closed vessels under pressure), and sufficient volume of liquor are consequently necessary factors for efficient extraction.

EFFECT OF TEMPERATURE ON EXTRACTION

A series of seven extractions was undertaken and carried out under the same set of conditions with the exception of temperature. A concentration of 5 per cent ammonium hydroxide was used. The efficiency of extraction is shown in Fig. I., from which it is observed that rapid extraction begins at 40° to 50° C. and reaches its maximum at 70° C.

EFFECT OF VOLUME OF LIQUOR AND TIME OF EXTRACTION

To ascertain the effect of the volume of ammonium hydroxide solution and the period of time of extraction, a number of extractions were made, the results of which are given in Table II. Twenty-five grams of chips, a 5 per cent solution of ammonium hydroxide and a temperature of 70° C. for extraction and washing were used in each case. In Experiments 2 and 3 the percentage of rosin and of humus in the wash liquor were not determined, but only the total quantity of each in the combined extraction and wash liquors.

In Experiment 1, with a short period of extraction and a small volume of extracting liquor, poor extraction was obtained. While only a small amount of rosin is obtained by washing, the quantity of humus is nearly as great as in extraction. In Experiments 2 and 3 the effect of 2 hours' heating is found to be very slight on the yield of rosin, but high on humus. Maximum rosin and minimum humus extraction are obtained in Experiment 4, in which the minimum period of heating and volume of wash liquor are employed.

USE OF HUMUS

While this investigation did not include a study of the humus, the dry powdery form in which it is separated from the rosin makes it adaptable to such uses as have been suggested by Cram[1] for the humus obtained in the soda process for wood pulp.

CONCLUSIONS

I—When resinous wood is treated with a five per cent solution of ammonium hydroxide equal to eight times the weight of the

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[1] THIS JOURNAL, 6 (1914), 896.

wood at 70° C. for five hours, nearly complete extraction of rosin occurs.

II—From the general properties of ammonia and its complete recovery in gas works, it is believed that nearly complete extraction of rosin from wood may be made and complete recovery of ammonia obtained.

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DISCOLORATION OF MAPLE IN THE KILN

By Roy C. Judd

In kiln-drying green frozen maple lumber, a brown stain or discoloration occurs unless the most favorable conditions are maintained. An investigation of the causes and of the nature of the discoloration was made. Irving W. Bailey[1] states that some sap staining is due to an enzyme. To substantiate this view, Mr. Bailey states that the action can be arrested by simply dipping the lumber in hot water.

To study the possibility that this action is due to some enzyme, such as an oxydase, several small blocks of maple were given various preliminary treatments, and then placed in an oven where the humidity exceeded 90 per cent. The temperature was maintained at 90° C. for from one to three hours. The preliminary treatments were made as follows:

1—Dipping in water at 90° C.

2—Soaking in boiling water for 1, 2, 5, 60, and 200-minute periods.

3—Steaming at 100° C. for 5, 60, and 200-minute periods.

4—Heating in a drying oven at 105° C. for 10 min., 30 min., 1 hr., 2 hrs., 10 hrs., and 20 hours.

5—Previous treatment with solutions of germicides:

a—Zinc chloride
b—Mercuric chloride
c—Sodium fluoride
d—Phenol
e—Arsenious oxide
f—Hydrogen peroxide
g—Sodium carbonate

Discoloration appeared in every case when these samples were finally subjected to the unfavorable conditions of a warm humid atmosphere. The stain was formed on the surface first, but, on remaining for a longer period in the moist air, the stain appeared throughout the specimen. The possibility of the discoloration being caused by an enzyme was disproven because at least some of the above treatments would have destroyed the enzyme. If dipping maple in hot water prevents subsequent staining in the kiln, it is not because the enzymes are destroyed but because the heated wood dries more readily.

A series of experiments were carried on in an autoclave. Pieces of fresh green maple were given the following treatments:

1—Enough of a 3 per cent solution of hydrogen peroxide was added to cover the sample. The air was pumped out and the temperature of 75° C. maintained.

2—An atmosphere of sulfur dioxide was supplied and the temperature kept at 80° C.

3—An atmosphere of carbon dioxide was added and the temperature maintained at 80° C.

4—The air was removed and live steam passed through continuously.

5—An NH_4OH solution was placed in the autoclave and the air exhausted. The temperature was kept at 75° C.

6—A little water was added and air was left in. A temperature of 80° C. was maintained.

The duration of the above experiments was from two to four hours. The results of these experiments showed that discoloration did not appear in the presence of hydrogen peroxide, carbon dioxide, sulfur dioxide, nor with steam in the absence of air. In an atmosphere of air saturated with moisture and also in the experiment with ammonium hydroxide, discoloration was very appreciable.

Three sets of experiments were carried on simultaneously. Pieces of maple were placed in flasks containing distilled water. Air was continuously bubbled through one and carbon dioxide and sulfur dioxide, respectively, through the other two. The initial temperature of 40° C. was maintained for one hour. Then the temperature was raised 10° at one-hour intervals till the temperature of boiling water was obtained. At the end of the process the pieces of maple in the flasks through which carbon dioxide or sulfur dioxide had been passed were even lighter colored than the original, while the maple in the flasks through which air had been passed was very badly discolored. The first evidence of discoloration appeared at about 60° C. and became more pronounced as the temperature was raised. Several experiments indicated that weak solutions of organic or mineral acids prevent discoloration. Some experiments were carried out on a small scale in laboratory drying ovens. It was found that no staining took place when the humidity of the air in immediate contact with the drying wood was kept low. The investigation seems to indicate that the staining is probably of a physical chemical nature and is independent of enzymes.

This paper is not intended to cover the subject of kiln-drying but is written for the purpose of showing that it is possible with conditions well under control to dry green maple lumber artificially and still retain its natural color. The conditions that prevail in a commercial kiln for drying lumber are not as easily controlled as those in a small laboratory apparatus. The section of timber physics in the Forest Products Laboratory has worked out its application in a dry kiln. The only available means of overcoming this difficulty in commercial practice is by the use of a low temperature (120° to 130° F.) and a low humidity (60 per cent or less) in the dry kilns. Fortunately, maple is a wood which does not check easily and hence these low humidities are quite practicable on a commercial scale.

I wish to express my appreciation of the suggestions and interest contributed by Mr. H. D. Tiemann, in Charge, Section of Timber Physics, and a specialist in kiln-drying, and Dr. S. F. Acree, Chief of the Section of Derived Products, who suggested the studies of the chemical and enzymic phases of this problem.

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MANUFACTURE OF ETHYL ALCOHOL FROM WOOD WASTE—II. THE HYDROLYSIS OF WHITE SPRUCE

By F. W. Kressmann

A paper[1] presented at the Forty-Ninth Meeting of this Society at Cincinnati, described (1) the scope of the work, (2) the apparatus used, (3) the method of procedure, and in addition gave the results obtained from a series of preliminary experiments on white spruce. Since the above work was done the yeasting operations and fermentation conditions have been standardized so that duplicate and comparable data have been made possible.

The yeast used is a pure culture strain of *Saccharomyces cerevisiae*, isolated from a yeast used in a Hungarian distillery, producing alcohol from beet sugar molasses. It is well adapted to the fermentation of sugar solutions obtained from the hydrolysis of wood, although the addition of nitrogenous food is necessary. This yeast nutrient is a mixture of ammonium sulfate and malt sprouts. The details of yeast propagation and the preparation of the mashes for fermentation along with other details of the present work will be given later in the form of a professional paper of the United States Department of Agriculture. In all cases, 7,500 cc. of a solution having a gravity

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[1] *Botanical Gazette*, **50**, 142–147, No. 2, August, 1910.

[1] This Journal **6** (1914), 625.

around 12.5° Brix were used for fermentation. The fermentations were carried out in a constant temperature room, maintained at 30° C. ± 0.5°, which could be kept in a sterile condition. The beers were distilled after 96 hours (distilleries operating on this type of materials are classed as sour mash distilleries by the Bureau of Internal Revenue) and from the sugar content before and after fermentation and the specific gravities of the mash and beer along with the alcohol content of the latter it was possible to calculate the fermentation efficiencies and yields. Since about 2.5 per cent of the total alcohol in the beers is obtained from the molasses solution, which is the medium in which the yeast is propagated, and which is added with the starting yeast, the fermentation efficiencies are all high but serve well for comparative purposes, since the error is practically a constant one.

TABLE I—FERMENTATION RECORD FOR WHITE SPRUCE

Fermenter set at 9.15 A.M. Distillations begun at 9.15 A.M. five days later. Fermentation continued during 128.5 hours, including the time of filling the fermenter, which took 32.5 hours

		Yeast					Mash before Fermentation					Brix Record						Beer					
		Seed yeast		96 Hour yeast																			
Fermentation No.	Cook No.	Brix.	Acidity.	Sp. gr. at 15° C.	Alcohol. % by weight	Acidity	Sp. gr. at 15° C.	Reducing sugar Grams per liter	Acidity	Total solids Per cent	Brix	1st day	2d day	3d day	4th day	5th day	Attenuation Degrees	Brix 4th day	Reducing sugar G. per liter	Sp. gr. at 15° C.	Acidity	Total solids % by weight	Alcohol % by weight
17	55	11.4		1.0216	5.922	18.7	1.0470	64.48	3.2	9.426	11.8	3.6	1.5	0.3	0.0		5.4	6.4	14.74	1.0245	8.4	5.603	2.382
17	55	11.4		1.0216	5.922	18.7	1.0454	62.44	3.0	9.115	11.4	3.5	1.5	0.2	0.2		5.4	6.0	13.76	1.0237	8.8	5.347	2.315
16	55	13.9	18.5	1.0223	6.167	26.2	1.0498	58.12	2.6	9.266	11.2	3.5	1.5	0.2	0.1		5.3	6.9	10.04	1.0274	8.0	5.971	2.311
16	55	13.9	18.5	1.0223	6.167	26.2	1.0478	56.40	2.5	9.564	11.9	3.3	1.6	0.2	0.1		5.2	6.7	10.28	1.0269	7.6	5.783	2.259
18	55	11.4		1.0210	6.009	22.3	1.0498	59.74	3.2	10.422	12.2	3.7	1.3	0.3	+0.2	—0.1	5.1	7.1	10.64	1.0275	7.6	6.289	2.224
16	56	13.9	18.5	1.0223	6.167	26.2	1.0514	65.20	4.0	9.567	12.4	3.4	1.6	0.5	0.1		5.6	6.8	14.54	1.0270	8.6	5.842	2.390
16	56	13.9	18.5	1.0223	6.167	26.2	1.0509	64.60	4.0	8.858	12.4	3.1	1.7	0.6	0.2		5.6	6.8	14.42	1.0268	8.6	5.848	2.395
17	56	11.4		1.0216	5.922	18.7	1.0475	57.34	2.4	9.428	12.0	3.1	1.8	0.2	0.1		5.2	6.8	10.22	1.0268	7.6	5.873	2.259
17	56	11.4		1.0216	5.922	18.7	1.0498	60.10	2.6	9.979	12.4	3.4	1.7	0.1	0.0		5.2	7.2	11.00	1.0278	7.4	6.160	2.345
18	56	11.4		1.0210	6.009	22.3	1.0487	66.52	4.4	11.110	12.3	3.7	1.5	0.3	0.0	0.1	5.7	6.6	14.60	1.0262	11.2	6.070	2.543
20	56	13.1		1.0230	5.650	18.0	1.0429	56.904	4.5	8.606	10.7	3.6	1.1	0.2	0.0		4.9	5.8	12.92	1.0227	9.6	5.278	1.968
20	56	13.1		1.0230	5.650	18.0	1.0542	71.288	4.8	10.750	13.1	3.4	1.8	0.7	0.1		6.0	7.1	16.24	1.0287	12.0	6.493	2.590
17	57	11.4		1.0216	5.922	18.7	1.0529	70.24	2.7	10.614	13.1	4.0	1.5	0.3	0.1		5.9	7.2	13.54	1.0279	7.4	6.264	2.558
17	57	11.4		1.0216	5.922	18.7	1.0509	66.32	2.4	10.076	12.5	3.5	1.7	0.3	0.2		5.7	6.8	13.78	1.0263	7.4	6.023	2.543
18	57	11.4		1.0210	6.009	22.3	1.0463	62.02	2.8	9.962	11.5	3.9	1.2	0.2	+0.1	—0.1	5.2	6.3	12.76	1.0247	7.6	5.781	2.318
18	57	11.4		1.0210	6.009	22.3	1.0400	52.26	2.4	8.705	10.0	3.7	0.9	0.2	+0.1	0.0	4.7	5.3	11.24	1.0213	6.0	4.948	1.909
19	57	12.9		1.0232	5.541	16.5	1.0474	60.984	3.0	8.539	11.7	3.6	1.3	0.4	0.1		5.4	6.3	12.86	1.0240	8.8	5.805	2.266
19	57	12.9		1.0232	5.541	16.5	1.0472	60.476	3.0	8.465	11.5	3.3	1.5	0.4	0.0		5.2	6.3	12.48	1.0246	8.8	5.951	2.274
18	58	11.4		1.0210	6.009	22.3	1.0468	75.81	3.2	10.996	11.7	3.8	1.3	0.1	0.0	0.0	5.2	6.5	29.26	1.0258	7.2	6.204	2.218
18	58	11.4		1.0210	6.009	22.3	1.0497	81.48	2.6	11.006	12.4	3.9	1.4	0.1	0.0	0.0	5.4	7.0	31.12	1.0275	6.3	6.484	2.117
19	58	12.9		1.0232	5.541	16.5	1.0523	83.152	3.0	9.679	12.7	3.5	1.4	0.4	0.1		5.4	7.3	32.28	1.0285	7.8	7.196	2.421
19	58	12.9		1.0232	5.541	16.5	1.0540	87.192	3.0	10.598	13.3	3.5	1.5	0.6	0.1		5.7	7.6	34.12	1.0300	9.2	7.453	2.500
20	58	13.1		1.0230	5.650	18.0	1.0553	87.792	2.5	11.180	13.2	3.2	1.8	0.6	0.1		5.7	7.5	33.80	1.0300	10.0	7.063	2.456

In addition, it permits of the expression of yields in the same way that they would be obtained commercially, since in either case about 1 per cent by volume of the total mash consists of the starting yeast solution. Tables I and II, showing the fermentation record and fermentable sugar and alcohol yields, from four different cooks, give an indication of the method of fermentation, the average results and degree of accuracy that are being obtained at present.

TABLE II—SUGAR AND ALCOHOL YIELDS FROM WHITE SPRUCE

		Per cent of total reducing sugar			Alcohol in Beer			Alcohol Yields		
Cook No.	Total reducing sugar Per cent of dry original wood	Fermentable	Non-fermentable	Fermentation efficiency	Actual alcohol yield	Theoretical alcohol yield	Per cent of total sugar obtained as alcohol	Per cent of original dry wood	Gallons absolute per dry ton	Gallons 190 Proof per dry ton allowing 5 per cent distillation loss
55	18.06	82.35	17.65	98.95	2.311	2.330	81.48	7.520	22.746	22.703
55	18.06	81.41	18.59	100.88	2.259	2.239	82.11	7.580	22.927	22.884
55	18.06	81.82	18.18	99.11	2.259	2.289	81.09	7.485	22.640	22.597
55	18.06	81.37	18.63	98.34	2.345	2.381	79.02	7.294	22.062	22.021
55	18.06	80.04	19.96	95.53	2.224	2.328	76.45	7.058	21.348	21.308
Average		81.40	18.60					7.387	22.340	22.300
56	21.56	77.16	22.84	97.73	2.390	2.446	75.41	8.307	25.126	25.079
56	21.56	77.68	22.32	98.09	2.395	2.440	76.20	8.395	25.392	25.345
56	21.56	76.63	23.37	98.75	2.382	2.412	75.67	8.338	25.220	25.172
56	21.56	77.53	22.47	97.80	2.315	2.367	75.82	8.355	25.271	25.224
56	21.56	77.56	22.44	101.11	2.543	2.515	78.42	8.641	26.136	26.087
56	21.56	76.85	23.15	91.83	1.968	2.143	70.57	7.778	23.520	23.475
56	21.56	76.66	23.34	97.73	2.590	2.650	74.92	8.256	24.972	24.925
Average		77.15	22.85					8.295	25.09	25.04
57	21.08	80.26	19.74	93.46	2.558	2.541	75.01	8.082	24.446	24.400
57	21.08	78.73	21.27	100.07	2.543	2.468	78.78	8.488	25.674	25.625
57	21.08	79.00	21.00	96.86	2.318	2.393	76.52	8.249	24.951	24.904
57	21.08	78.09	21.91	95.16	1.909	2.006	74.31	8.006	24.216	24.170
57	21.08	78.43	21.57	97.09	2.266	2.334	76.15	8.204	24.815	24.768
57	21.08	78.91	21.09	97.64	2.274	2.329	77.05	8.301	25.108	25.060
Average		78.90	21.10					8.222	24.87	24.82
58	22.82	60.62	39.38	98.84	2.218	2.244	59.92	6.989	21.140	21.100
58	22.82	60.98	39.02	87.52	2.117	2.419	53.37	6.225	18.829	18.793
58	22.82	60.29	39.71	99.43	2.421	2.435	59.95	6.992	21.149	21.109
58	22.82	59.95	40.05	98.62	2.500	2.535	59.12	6.895	20.855	20.816
58	22.82	60.56	39.44	95.38	2.456	2.575	57.76	6.737	20.377	20.339
Average		60.48	39.52					6.768	20.47	20.43

In the former paper the influence of temperature and pressure and time of cooking were discussed. The ratio of water to dry wood was 400 to 100 and the ratio of acid to dry wood was 1.8 to 100. In view of better fermentation conditions, some of the above work was repeated and in addition the following variables were studied: (1) ratio of water to dry sawdust, (2) ratio of catalyzing agent to dry sawdust, (3) concentration of catalyzing agent in the water added. The first variable was carried from 4 water : 1 dry wood (called 400 per cent water) down to 1 water : 1 wood. The best operating condition finally chosen was 125 per cent of water. This was done: (1) To insure a better mixture of acid and wood, which is easier to accomplish with increasing amounts of water, (2) to use the maximum amount of water possible, and still obtain a digested sawdust that had no drip; *i. e.*, no free water or liquid should be present after digestion. This is highly desirable from an operating standpoint and, although 100 per cent added water is to be preferred to 125 per cent added water when steam consumption is taken into account, it was felt that the better mixture of acid and wood obtained with the 125 per cent water outweighed the small increased steam consumption and justified its adoption as a standard in our work.

Table III shows no great differences in yields resulting in the reduction of the added water from 400 to 125 per cent, which is of exceeding importance commercially.

Table III

1.80 to 1.85 Per Cent H_2SO_4 0 Minute Time 7.5 Atmos. Pressure

Water Per cent of dry wood	Total Sugars	Per Cent of Total Sugars Fermentable	Per Cent Alcohol
100	21.09	60.68	6.440
125	21.96	59.29	6.805
250	23.75	55.31	6.648
300	23.16	54.87	6.096(*a*) 6.645(*b*)
400	22.24	56.19	6.154

(*a*) Actual average, containing one poor fermentation.
(*b*) Result recalculating poor fermentation to a normal efficiency.

Sulfuric acid was used as a catalytic agent in amounts varying from 0.5 per cent to 4 per cent of the dry weight of the wood. The results obtained with a zero minute cooking period and 125 per cent water cooked at 7.5 atmospheres pressure are given in Table IV, which shows that not only total sugars increase with increasing acidity, but especially that the portion which is fermentable increases so that the alcohol yields increase proportionally.

Table IV

Concentration H_2SO_4	Total Sugar Per Cent	Per Cent of Total Sugars Fermentable	Per Cent Alcohol
0.5	17.42	43.13	4.172
0.75	21.83	56.03	6.085
1.00	21.68	56.43	6.506
1.40	23.09	53.93	6.502
1.83	21.53	59.98	6.623
2.50	22.11	63.16	6.927
4.00	21.10	66.63	7.000

A consideration of the above two variables will show that the concentration of the catalytic agent expressed in terms of dry wood is the single and great determining factor and this is especially true when the following work on the effect of time of cooking is studied. The concentration of acid expressed in terms of the added solution is without effect (contrary to a number of patents that have been granted) except where such variation also varies the ratio of acid to dry wood. In the paper referred to in the beginning of this communication, the conclusion was reached that "a cooking period of 0 minute, that is, blowing off from 7$^1/_2$ atmospheres as soon as that pressure was obtained, was advantageous." This was the result of a study of total sugars only, as shown in Figure 3 of that paper where the total sugar obtained from a series of cooks varying from 0 to 30 minutes varied less than a few tenths of 1 per cent. A repetition of that work has shown practically the same results so far as the total sugar is concerned, but careful fermentations have shown a decided difference in composition of that sugar under different conditions. This difference is shown particularly when the acid concentration is fairly high, as in the case of the 2.5 per cent acid, although the rise in fermentable sugars is noticeable even in 1.4 per cent acidity.

Table V

7.5 Atmos. Pressure and 125 Per Cent Water

Time of Cook Minutes	Total Sugar Per cent	Per cent of Total Sugar Fermentable	Alcohol Per Cent of Dry Wood
	With 1.40 Per cent H_2SO_4		
0	23.09	53.93	6.502
10	23.45	55.08	6.456
30	22.34	63.22	6.862
	With 2.50 Per cent H_2SO_4		
0	22.11	63..6	6.927
10	22.36	68.53	7.662
20	23.61	71.44	8.537
45	21.56	77.15	8.295
90	18.06	81.40	7.387

Table V shows these variations and fermentable sugar and alcohol yields. In addition it will be noticed that with 2.5 per cent acid there is a gradual decrease in total sugar as the length of the cook is largely increased (beyond 20-minute cooking periods), even though the portion that is fermentable increases quite decidedly. The causes and explanations of these differences have not been fully worked out, although a number of lines of attack suggest themselves, and we hope to take up this phase of the problem in detail in a subsequent publication. This brief summary of results is given purely for its technical value for the assistance that it may give in the proper choice of operating conditions as determining those prime variables which affect the total and fermentable sugar yields.

CONCLUSION

From an operating standpoint, and taking into consideration, (1) economic operation, which includes acid cost, lime or other neutralizing cost, and steam consumption; and (2) plant depreciations, we feel that the following cooking conditions give the greatest yields of alcohol:

I—Pressure of cook, 7$^1/_2$ atmospheres.
II—Time of cook, about 20 minutes.
III—Water to dry wood ratio, 125 to 100.
IV—Acid to dry wood ratio, 2$^1/_2$ to 100.

The writer wishes to thank the E. I. du Pont de Nemours Powder Company, and Mr. John Boyt and Mr. J. S. Groves, Superintendent and Chemist, respectively, of the alcohol plant of the du Pont Company at Georgetown, S. C., for the many courtesies and suggestions rendered in the yeasting and fermentation work. The writer further wishes to thank Mr. Homer Cloukey, of the Forest Products Laboratory, for the patience and skill exercised in the many hundred analyses required in the study of this problem.

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THE MANUFACTURE OF ETHYL ALCOHOL FROM WOOD WASTE—III. WESTERN LARCH AS A RAW MATERIAL

By F. W. Kressmann

The logging of western larch (*Larix occidentalis*) shows a woods loss of 8 per cent due to butting off the lower portion of the tree.[1] This practice is due chiefly to the presence of shakes in the butt. In addition, the base of the tree is usually swelled. The swelled portion is denser than the rest of the trunk and usually sinks, thus preventing rafting. The lengths of the "butts" left in the woods vary from 4 to 8 feet, although occasionally a 16-foot piece is rejected.

The utilization of this waste material up to the present time has not met with success and it was hoped that it might be profitably employed as a raw material in the production of ethyl alcohol.

A sample of sawdust from a butt log was cooked with 1.8 per cent sulfuric acid, 125 per cent of water, at 7.5 atmospheres pressure for 10 minutes. A yield of sugar equal to 29.72 per cent, and of total solids equivalent to 35.18 per cent, of the dry weight of the wood was obtained. Under the same conditions, white spruce would yield about 22 per cent of sugar, of which from 60 to 65 per cent would be fermentable, making an alcohol yield of 6.8 to 7 per cent of the dry weight of the wood, or from 20 to 21 gallons of absolute alcohol per dry ton. The extracts obtained from the hydrolysis of the larch were fermented under standard conditions, the fermentation records and alcohol yields being given in Tables I and II.

The larch yielded about 35 per cent total sugar more than spruce and yet only 37.9 per cent of that sugar fermented compared with 60 or 65 per cent from spruce. Mr. A. W.

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[1] U. S. Department of Agriculture, *Forest Service Bulletin* 122, "The Mechanical Properties of Western Larch," O. P. M. Goss.

TABLE I—FERMENTATION RECORD—WESTERN LARCH

		Yeast								Mash before fermentation											Beer						
		Seed yeast		96 Hours yeast											Brix record Decrease in Brix												
Fermentation No.	Cook No.	Brix.	Acidity.	Sp. grav. at 15° C.	Alcohol. % by weight	Acidity	Fermenter set. Date	Hours filling fermenter	Hours fermenting, including time of filling	Sp. grav. at 15° C.	Reducing sugar Grams per liter	Acidity	Total solids Per cent	Brix	1st day	2nd day	3rd day	4th day	5th day	Attenuation Degrees	Brix. 4th day	Distillation Date	Reducing sugar Grams per liter	Sp. gr. at 15° C.	Acidity	Total solids % by weight	Alcohol. % by weight
10	50	..	..	1.0243	6.170	18.6	1/21/15	32.5	128.5	1.0495	94.92	3.2	10.301	12.1	3.1	0.3	+0.1	—0.2	0.0	3.5	8.6	1/26/15	56.28	1.0341	7.0	7.207	1.4707
11	50	..	22.0	1.0262	5.398	26.4	2/11/15	32.5	128.5	1.0543	97.936	2.4	12.325	13.3	3.0	0.5	0.2	0.2	0.0	3.9	9.4	2/16/15	61.744	1.0392	7.2	9.490	1.598
14	50	..	22.5	1.0235	5.784	24.5	3/18/15	32.5	128.5	1.0440	83.38	2.8	9.190	11.0	2.9	0.4	0.3	+0.2	0.0	3.4	7.6	3/23/15	51.264	1.0302	5.6	6.438	1.320

TABLE II—SUGAR AND ALCOHOL YIELDS FROM WESTERN LARCH

						ALCOHOL IN BEER			ALCOHOL YIELDS		
Species of wood	Cook number	Total reducing sugar in per cent of dry original wood	Per cent of total reducing sugars fermentable	Per cent of total reducing sugars non-fermentable	Fermentation efficiency	Actual alcohol yield	Theoretical alcohol yield	Per cent of total sugar obtained as alcohol	Per cent of original dry wood	Gallons of absolute per dry ton	Gallons of 190 proof per dry ton allowing 5% distillation loss
Western larch....	50	29.72	37.70	62.30	85.77	1.320	1.539	32.34	4.970	15.033	15.004
Western larch....	50	29.72	39.83	60.17	79.85	1.470	1.841	31.80	5.048	15.269	15.240
Western larch....	50	29.72	36.04	63.96	92.23	1.578	1.711	33.24	4.912	14.857	14.829
AVERAGE........			37.89	62.11					4.977	15.05	15.03

Schorger, of this laboratory, has analyzed both of these woods with the following results:

	WESTERN LARCH (Base) Per cent	WHITE SPRUCE Per cent	WHITE SPRUCE (4 Samples) Average
Soluble in ether...............	9.75	0.90 to 1.95	1.36
Soluble in cold water..........	14.47	0.82 to 1.45	1.12
Soluble in hot water...........	16.52	1.88 to 2.52	2.14
Soluble in 1 per cent NaOH, 10 min. heating................	32.72	6.72 to 8.84	7.70
Soluble in 1 per cent NaOH, 60 min. heating................	38.58	11.18 to 13.87	12.21
Pentosan........................	6.99	10.04 to 10.78	10.39
Methyl pentosan...............	3.42	3.08 to 3.95	3.55
Cellulose......................	42.57	51.95 to 58.47	56.17
Volatile oil....................	0.84	... ...	...
Ash............................	0.36	0.285 to 0.326	0.307

It will be noticed that the larch contains a large amount of water-soluble material and a proportionally small amount of cellulose. In all cases, the above analyses were made on a fresh sample taken from the same lot. In a subsequent paper, Mr. Schorger will take up the composition of this water-soluble material, the chief constituent of which is a galactan which yielded approximately 10 to 12 per cent of the dry weight of the wood of galactose, which in turn accounts for the high sugar yields obtained from the larch.

It has been suggested by Körner,[1] although disputed by Gallagher and Pearl,[2] that in the acid hydrolysis of cellulose-containing material, the source of the fermentable sugars and, therefore, the alcohol, is the cellulose itself.

If the sugar yield for the larch is recalculated so that it is proportional to the cellulose content, assuming 22 per cent of sugar from the spruce, we would have then 16.7 per cent of sugar instead of 29.7 per cent, as actually obtained. However, as noted above, about 10 or 12 per cent of galactose was obtained, which subtracted from the total sugar yield of 29.7 per cent, would leave from 17.7 to 19.7 per cent of sugar comparable to that obtained from spruce. Under normal conditions, with a 94 per cent efficiency and a yield of that per cent of the theoretic amount of alcohol formed, which is 51.1 per cent, we get for 60 or 65 per cent of total sugar fermentable the following alcohol yields:

$$16.7 \times 0.511 \times 0.94 \times 0.60 = 4.83 \text{ per cent alcohol}$$
$$16.7 \times 0.511 \times 0.94 \times 0.65 = 5.23 \text{ per cent alcohol}$$

The actual alcohol yield is 4.977 per cent, which corresponds to about 62 per cent of total sugar fermentable, which is the average of the above figures chosen for spruce. It appears, therefore, that the yield of fermentable sugar and alcohol is proportional to the cellulose content of the wood, irrespective of other materials that may be contained therein. In addition, western larch butts will be a good raw material for the production of ethyl alcohol if a yeast is found which will ferment the galactose as well as the dextrose within the time limit and other limitations as prescribed by the Bureau of Internal Revenue. This phase of the subject has been reserved and will be taken up in the near future.

FOREST PRODUCTS LABORATORY
MADISON, WISCONSIN

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[1] *Zeitschrift für angewandte Chemie*, **1908**, 2353.

[2] *Proc. Eighth Intern. Cong. Appl. Chem.*, **13**, p. 147.

THE APPLICATION OF THE DAVIS SPOT TEST IN THE PRELIMINARY EXAMINATION OF CREOSOTES

By HOMER CLOUKEY

The tests undertaken and included in this report were run for the purpose of verifying the "absorption spot test" given in an article by T. H. Davis on "The Examination of Creosote,"[1] and described in this article as follows:

"*Absorption Spot*—Allow six drops of the sample to fall from a burette upon the surface of clean, white blotting paper. If tar, carbon, or dirt is present, it is very easily observed, as it quickly segregates at the center. The paper should be laid away, in a flat position, for several hours, in a place free from dust. If then examined, foreign matter will be observed in a distinct zone at the center of the spot; the outer zone very readily indicates the character of the oil."

In order to establish some idea of the sensitiveness of this test for carbon and dirt in creosote and make it fairly quantitative a series of spots was made from a carbon-free creosote with definite graduated amounts of carbon in the form of lampblack added.

This series comprises six mixtures of creosote and lampblack as follows:

Per cent lampblack added.........	0.0	0.005	0.01	0.05	0.10	0.50

This series is shown in Fig. I. The results obtained show an increasing gradation in the density of the free carbon ring at the center and indicate that 0.005 per cent is easily shown by this test. In heavier percentages than 0.5 per cent, the amount in an unknown sample would be difficult to estimate by comparison.

The appearance of several authentic samples of various creosotes and oils used for wood-preserving purposes is shown in Fig. II. No. 1 shows coal tar creosote with the characteristic brown gradually shading on the edges to a lighter zone: if carbon and dirt are present, these are segregated in an inner circle which is the size of the original spot before diffusion. No. 2 is a characteristic water gas tar creosote with the large spot and outer yellow zone characteristic of this oil. No. 3 illustrates the character of the spot afforded by a paraffin base crude oil used for wood-preserving purposes: this is almost colorless

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[1] *Oil, Paint, and Drug Reporter*, February 14, 1910.

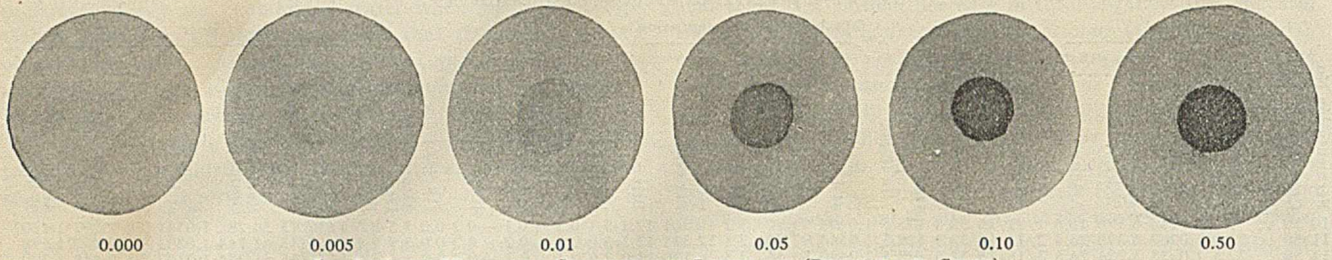

FIG. I—SPOTS MADE WITH CREOSOTE AND LAMPBLACK (PERCENTAGES GIVEN)
Reduced a little over one-half. Color—Light brown with black centers

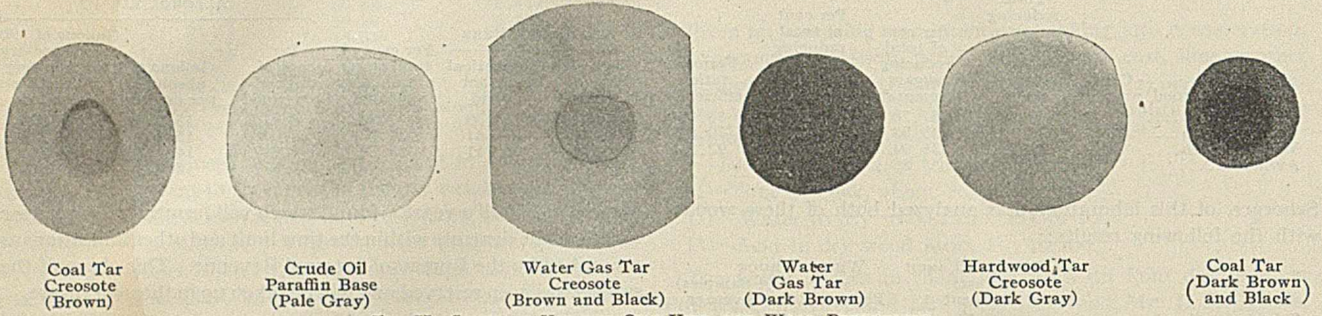

FIG. II—SPOTS OF VARIOUS OILS USED FOR WOOD PRESERVING
Reduced a little over one-half. Colors as noted

while an asphaltic base crude oil shows a browner center. No. 4 shows the characteristic dead gray-black of creosote made from hardwood tar. No. 5 is the even dark brown spot of a typical water gas tar: this tar is often used in creosote tar mixtures at the present time. No. 6 is the spot of a coal tar with the heavy carbon in the center and the small diffusion of the pitchy mass.

The spots given in Fig. II represent only the typical spots of the various oils used for wood-preserving purposes. In actual practice, these oils are used in more or less admixture with each other and a large number of spots can be obtained grading from one of these types to another.

The admixture of tar can be determined roughly from the size (smaller) and the general character of the spot. The heavy tar does not diffuse with the speed of the lighter creosote oils.

CONCLUSIONS

I—The presence of dirt and free carbon in creosote oil is indicated in very minute quantities by this test.

II—If the creosote spot shows a dense black center it will probably be necessary to run a free carbon analysis to determine if the creosote passes the free carbon specification.

III—The various types of wood-preserving oils can be easily distinguished from each other by this test when they are true type samples.

IV—In the large number of intermediate or mixed commercial oils, the value of this preliminary test will depend on the experience of the one applying it together with the possession of a large number of authentic samples for comparison. On applying this test to a number of oils compounded from known authentic samples it was possible to tell the constituents with a reasonable degree of accuracy.

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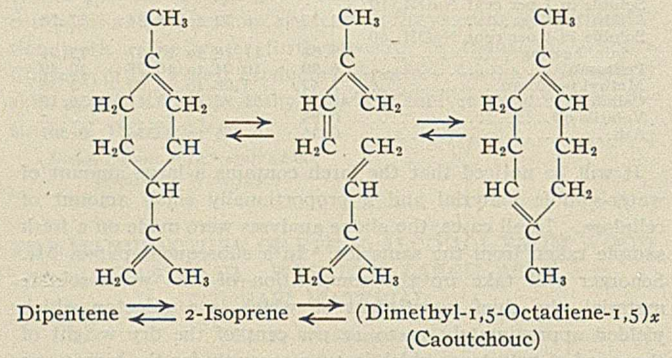
ISOPRENE FROM β-PINENE

By A. W. SCHORGER AND R. SAYRE

The discovery during the latter half of the past century of a close relation between isoprene, the terpenes, and caoutchouc has naturally directed considerable attention toward the utilization of the terpenes as a source of isoprene. This relation may be represented by the following reversible reactions:



It was to be expected that attempts would first be made to utilize α-pinene as a raw material, since, in the form of turpentine, it can be more easily and cheaply obtained than any of the other terpenes. The results of former experiments clearly indicate, however, that only comparatively low yields are possible from turpentine. Tilden,[1] by passing turpentine through a red-hot tube, obtained about 20 cc. of isoprene from a liter of turpentine. By means of his isoprene lamp, Harries[2] obtained about 1 per cent of isoprene from commercial pinene and attributed even this small amount to the presence of dipentene in the turpentine employed. Herty and Graham[3] obtained 5.5 per cent (by volume) of isoprene from turpentine and 8.0 per cent from a fraction boiling between 155 and 156°. These authors are of the opinion that the isoprene obtained from turpentine is not due to dipentene present in the turpentine as asserted by Harries, an opinion that we believe is fully justified.

Apparently the only terpene yielding considerable amounts of isoprene is limonene (dipentene). Harries[2] obtained 30 to 50 per cent of isoprene from commercial limonene while Herty and Graham obtained 12 per cent from a limonene fraction. Staudinger and Klever[4] found that by working at a pressure of about 4 mm., a yield of 60 per cent of exceptionally pure isoprene could be obtained from limonene.

According to patents held by Schering and Company,[5] con-

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[1] *Chem. News*, **46** (1882), 220.

[2] *Ann.*, **383** (1911), 228–9.

[3] THIS JOURNAL, **6** (1914), 803–4.

[4] *Ber.*, **44** (1911), 2212.

[5] German Patent 260,934; K. Stephan, U. S. Patent 1,057,680 (1913) (Assignor to Schering & Co.).

siderably larger yields of isoprene can be obtained from β-pinene than from α-pinene. As has been previously shown by one of us,[1] the oil from the oleoresin of western yellow pine (*Pinus ponderosa*, Laws.) consists mainly of β-pinene with small amounts of α-pinene and limonene. Large stands of this species are available for tapping purposes on the Pacific Coast and to our knowledge offers the only cheap source of β-pinene. Since no data was available on the yields of isoprene to be obtained from β-pinene, it was determined to investigate this point. The above oil would be additionally promising, owing to the presence of limonene.

It was found, however, that α-pinene and β-pinene gave approximately the same yield of isoprene when both terpenes were passed through the same apparatus for the purpose of direct comparison.

EXPERIMENTAL

The isoprene lamp of Harries[2] offers a convenient means of "cracking" terpenes in the laboratory. After considerable experimentation to avoid certain mechanical disadvantages a modified form of apparatus of the type shown in the accompanying Fig. I, *A*, was employed. The heating coil consisted of a platinum wire 150 cm. long wound in the form of a spiral through two parallel rows of holes in the edges of a strip of "transite," a heat-resisting, insulating material, composed of asbestos and cement; the temperature of the wire was regulated by means of a sliding rheostat, the best results being obtained at a low red heat.

The vapors after passing the coil entered the Hopkins condenser where the less volatile portions were condensed and returned to the boiling flask through the trap as indicated. The vapors passing through the Hopkins condenser, passed in turn through a second condenser fed with cold water, then through a spiral glass tube surrounded by a freezing mixture of salt and ice, and finally into a receiving flask likewise surrounded by a freezing mixture.

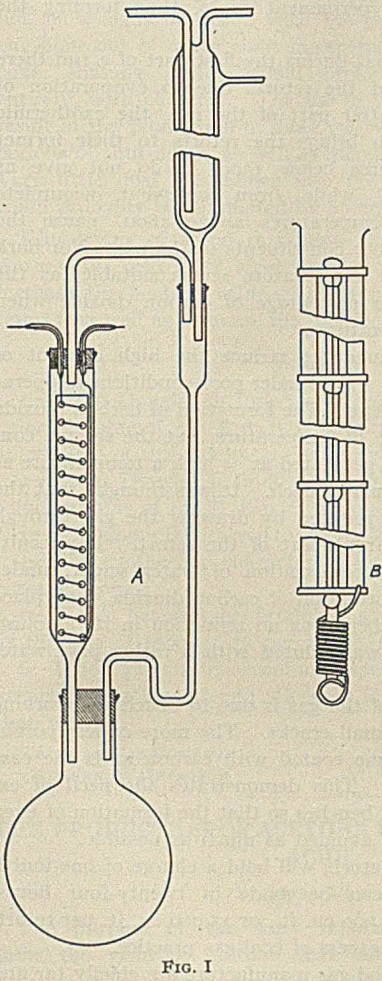

Fig. I

The turpentine employed was obtained by rectifying ordinary gum turpentine. The β-pinene employed was obtained by repeated fractionation of the oil of western yellow pine.

At first the boiling was continued until the contents of the flask had completely polymerized, but it was found that practically no isoprene was formed after the first three or four hours. Although great pains were taken to secure complete condensation, it was evident that some of the isoprene escaped with the non-condensable gaseous decomposition products. Immediately after the completion of a run the distillate was fractioned by means of a 12-inch Hempel column, filled with glass beads, and the fraction boiling between 35–37° considered as isoprene. To what extent the crude isoprene was possibly contaminated with trimethylethylene was not determined. The results of some typical runs are given in Table I.

TABLE I

No.	ORIGINAL MATERIAL	Weight Grams	Time hrs.	PER CENT OF ORIGINAL Residue	Total distillate	Loss	Yield of isoprene % by weight
1	Turpentine.............	400	6	31.7	35.1	33.2	8.1
2	Turpentine.............	283	5.5	46.7	35.3	18.0	10.3
3	β-Pinene (b. p. 162–165°)	300	6	40.0	37.4	22.6	9.4

The production of isoprene in the experiments described above would appear to be due largely to the catalytic effect of the platinum, since a nichrome wire was found to act in an entirely different manner. In order to use a long wire in a compact space a nichrome wire was stretched back and forth between perforated disks of transite fastened on a glass rod Fig. I, *B*.

Owing to the great elongation on heating, the wires sagged, thereby causing short circuits. This difficulty was successfully overcome by attaching a section of strong steel door spring to the lower disk to take up the slack in the manner illustrated. When the terpene vapors came in contact with the nichrome wire, heated to the same intensity as the platinum wire, thick deposits of carbon were formed immediately. The wires were short-circuited so quickly by the masses of carbon deposited that it was impossible to continue the experiment beyond this point.

Several experiments were performed by passing the terpene vapors through an iron tube filled with pumice stone and heated in a combustion furnace. With an apparatus of this type it is difficult to return the unchanged terpenes to the boiling flask. Owing also to the difficulty of temperature control, the terpenes in some cases passed through largely unchanged while in others considerable amounts of tar and members of the naphthalene series were formed from too high temperatures. The results are accordingly not comparable with those obtained with the isoprene lamp.

The small yields of isoprene obtained by Tilden were probably due to the small positive catalytic effect of the iron tube. To obtain the desired catalytic effect platinum black was deposited in the pores of the pumice by reduction of potassium chlorplatinate. It was found advisable to boil the liquid very gently and maintain the tube as nearly as possible at a barely visible red heat. Some of the results obtained with the tube method are given in Table II (no platinum black was deposited in the pumice used in Experiment No. 1).

TABLE II

No.		PER CENT OF ORIGINAL Residue	Distillate	Loss	Yield of isoprene % by weight
1	Turpentine.................	6.7	85.6	7.7	3.5
2	Turpentine.................	6.0	74.2	19.8	8.0
3	β-Pinene (b. p. 163–166°).....	3.6	64.2	32.2	9.6

It will be noted that the yields of isoprene obtained from turpentine are considerably higher than those obtained by Herty and Graham. This may be due to the employment of a larger catalytic surface in our experiments.

CONCLUSIONS

The results obtained show that turpentine and β-pinene under the same conditions yield about the same amount of isoprene, approximately 10 per cent.

The isoprene obtained from turpentine is certainly not due to the cracking of dipentene or limonene originally present in the turpentine; but the opinion is advanced that the isoprene results indirectly from dipentene. It is a well-known fact that α-pinene can be converted into dipentene by heat; the condition

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[1] Schorger, *Bull.* **119**, U. S. Forest Service (1913).

[2] *Ann.*, **383** (1911), 228–9.

obtaining in the apparatus would be favorable to such a transformation. The changes may be represented thus:

PINENE $\longrightarrow$ DIPENTENE $\longrightarrow$ ISOPRENE

Since the first reaction would probably not proceed to a great extent under the conditions of the experiment the yield of isoprene would necessarily be low.

It is not probable that either α-pinene or β-pinene can be made to yield directly sufficient isoprene for the commercial production of rubber. However, since good yields of isoprene are possible from dipentene, an attempt to obtain an approximately quantitative conversion of pinene into dipentene is worthy of further consideration.

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THE DISTILLATION OF DOUGLAS FIR AT HIGH TEMPERATURES

By BAILEY TREMPER

Inasmuch as the cost of manufacturing illuminating gas from coal and oil is gradually increasing in the small plants, the possible substitution of wood as the raw material is of interest. This is especially the case in sections of this country such as the Pacific Northwest, where thousands of cords of mill waste are destroyed in mill incinerators. For several years an experimental gas plant using sawmill waste has been in operation in this state, and recently several plants have been, or are being, constructed for commercial operation. It is the purpose of this paper to present some of the results obtained in one of these plants, located at Auburn, Washington.

The plant is patterned closely after standard coal-gas installations. Benches of four 9-ft. clay retorts are used. These are connected in turn to a hydraulic main, condenser, exhauster, and dry scrubbers; thence through the station meter to the holder.

The big stumbling block in wood-gas manufacture has been the accumulation of tar in the pipes, especially near the offtake. This has been overcome by waterjacketing the standpipes from mouthpiece to bridge pipe. Special design of the mouthpieces provides for drainage of the condensed liquor. Particles of tar and pitch are readily removed by the action of the thin liquor resulting from the condensation of portions of the gas. Trouble from stoppage further on is also minimized.

The wood for gas making is bought in 4-ft. lengths. It is tied into bundles of such size that two may be placed side by side in the retort, each retort requiring four bundles. The bench is maintained at 1400 to 1600° F. The exhauster is regulated so that there is neither vacuum nor pressure on the retorts. The time of carbonization is one hour and forty to fifty minutes.

Both the quantity and the quality of the gas vary with the grade and variety of the wood used. In commercial practice Douglas fir only is used but trial runs were made with red cedar and Western hemlock.

Forest wood cut from live trees, seasoned three months, fairly resinous, and weighing 3700 lbs. per cord, an average of the butt of a tree, produced 25,000 cu. ft. of gas per cord of 128 cu. ft.; 19,000 cu. ft. of this gas had an average heating value of 538 B. t. u. per cu. ft. as determined with a Junkers calorimeter. The average of the entire run was 482 B. t. u. per cu. ft. The maximum heating value during the run was 560 B. t. u. Bark alone from this wood gave 17,000 cu. ft. per cord, averaging 494 B. t. u., with a maximum of 567 B. t. u.

From unselected mill waste, thoroughly air-dried, weighing 3300 lbs. per cord, 18,000 cu. ft. of gas were obtained as the average yield per cord during two months' operation. The average heating value of the gas is 470 to 480 B. t. u. In each run a maximum heating value of about 515 B. t. u. is attained when the run is one-third over.

From hemlock mill waste, very wet, and weighing 4000 lbs. per cord a yield of from 15,000 to 17,000 cu. ft. of gas with an average calorific value of 414 B. t. u. was obtained. Cedar gave the same yield with a heating value of 460 B. t. u.

A typical analysis of the gas as determined with the Morehead apparatus is as follows:

	Per cent
Carbon dioxide (CO_2)	17.4
Illuminants	6.0
Oxygen (O_2)	0.0
Carbon monoxide (CO)	31.5
Methane (CH_4)	21.7
Hydrogen (H_2)	18.3
Nitrogen (N_2)	5.1

The calculated heating value of the gas is 500 B. t. u. and the calorimeter test of the same sample was 509 B. t. u.

It will be seen from this analysis that the gas resembles more or less closely carbureted water gas in composition, and it is of value to consider the reactions in the retort in this light. Wood charged into the hot retort soon acquires a coating of charcoal. Water vapor driven out from the wood comes in contact with the hot carbon and is decomposed. Inasmuch as the initial temperature is comparatively low, the layer of charcoal thin, and steam greatly in excess, the formation of carbon dioxide is favored. The resinous and oil-forming portions of the wood, on vaporization, are apparently cracked and partially converted into permanent gases, thus forming the enriching constituents.

It has been observed that during the first part of a run there is considerable cooling of the retorts due to evaporation of moisture. During the latter part of the run, the exothermic character of the reaction brings the retorts to their former temperature. Temperatures below 1300° F. do not give as good results as outlined, while, from somewhat incomplete data, it appears that temperatures above 1800° cause the breaking down of the richer constituents of the gas. For bark alone, however, a higher temperature seems suitable, as the gas contains a very high percentage of carbon dioxide when formed at medium temperatures.

Attempts have been made to reduce the high content of carbon dioxide in the gas, which under poor conditions of operation has reached 22 per cent. The formation of carbon dioxide is decreased with increase of temperature, but the sample containing 17.4 per cent was generated at as high a temperature as is practicable in the ordinary bench. It was thought that the carbon dioxide might be reduced by drawing the gas through beds of charcoal in the front part of the retort. The results showed, however, that decomposition of water vapor, under conditions favoring the formation of carbon dioxide, took place to such an extent that there was no reduction in its amount. In other words, the gas was diluted with a rather poor water gas.

The nitrogen content of the gas is due to bench gas working into the retort through small cracks. The more or less porous new retorts do not become coated with carbon as is the case in coal-gas manufacture. This demonstrates the need of extreme care in heating the benches so that the formation of even the finest cracks may be avoided as much as possible.

A bench of four 9-ft. retorts will hold a charge of one-fourth cord. Twelve charges can be made in twenty-four hours, giving a gas output of 60,000 cu. ft., or 15,000 cu. ft. per retort, an amount somewhat in excess of coal-gas practice.

The by-products of wood-gas manufacture are chiefly tar and charcoal. Wood alcohol, acetic acid, acetone, and part of the tar are largely decomposed under the temperature of distillation. Analysis of the aqueous distillate shows 1.0 to 2.5 per cent acetic acid, 0.2 per cent wood alcohol and acetone, and less than 1 per cent soluble tar.

The yield of tar varies from 14 to 22 gallons per cord according to the quality of the wood, the temperature of distillation, and the pressure on the retorts. The tar is much more easily recovered than when produced by low temperature distillation, and settles out in eight hours, carrying less than 5 per cent water. On redistillation it yields 1.5 per cent wood spirits, boiling below 100° C. and 2.5 per cent light oils boiling below 150° C. It is completely dehydrated at 150° C. This tar sells readily in local markets for 12.5 cents per gallon.

By employing a lower temperature the yield of by-products is increased. While a lower heating value of the gas results, it would still be useful for industrial purposes.

Charcoal is obtained at the rate of 700 to 800 lbs. per cord, for which a good market does not at present exist in the Northwest. Its quality is impaired by the presence of charcoal from bark, but as the latter is a good producer of gas, it is not feasible to remove it from the sawmill waste. The amount of charcoal obtained is ample to furnish fuel for open-fired benches and with regenerative furnaces an excess would be produced. This excess quantity or the entire amount can be used in water-gas manufacture, as demonstrated by trial runs made in a 3-shell Lowe water-gas set at the plant of the Tacoma Gas Company. The charcoal held its heat so that 3-minute periods of run and blast could be maintained. The same amount of oil was used as with coke. A yield of 1000 cu. ft. of carbureted gas was obtained from each 25 lbs. of charcoal as opposed to 40 lbs. of coke. The clinker formed was practically negligible and did not show signs of fusion with the generator lining. Whereas one hour out of every eight is generally required for clinkering coke, shaking grates would probably eliminate clinkering entirely, with charcoal. The use of the latter would therefore result in the production of more gas, with a saving in labor and periods of shut-down, as well as increase the life of the furnace. Very little dust was blown into the carburetor and all of it could probably be eliminated by baffles.

Kiln-drying of wood to remove the considerable amount of water still held after air-drying would doubtless still further increase the heating value of the gas and the capacity of the retorts, as well as decrease the fuel required.

SUMMARY

1—From Douglas fir forest wood, 25,000 cu. ft. of gas with a calorific value of 482 B. t. u. is obtained per cord of 37000 lbs.; 19,000 feet of this gas has a heating value of 538 B. t. u.

2—From Douglas fir mill waste 18,000 cu. ft. of gas with a calorific value of 475 B. t. u. is obtained per cord of 33000 lbs.

3—Enough tar is obtained to pay for the cost of wood at the plant.

4—The exothermic character of the reaction favors low fuel consumption.

5—Water gas can be produced satisfactorily from charcoal at the rate of 1000 cu. ft. per 25 lbs. of charcoal.

6—Wood gas and charcoal are especially available for metallurgical operations at mines located far from fuel supplies other than wood.

Auburn Gas Company
Auburn, Washington

THE PRODUCTION OF ACETONE FROM PYROLIGNEOUS ACID[1]

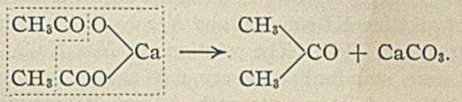
By Marc Darrin

The purpose of the work reported in this paper is to show the yield of acetone that can be obtained from pyroligneous acid by means of a continuously operated electric furnace, containing a catalyzer, which converts the acid directly into acetone without necessitating the intermediate production of the acetate.

The raw material consisted of pyroligneous acid obtained by the destructive distillation of Douglas fir mill waste at the half-cord wood distillation plant designed by the author,[1] and erected coöperatively by the University of Washington and the U. S. Forest Service. Before treatment, the pyroligneous acid was distilled in order to separate and recover the turpentine, oils, tar, alcohol, acetone, etc., since it was thought that these substances might have an injurious effect on the catalyzer and since these by-products are necessarily separated and recovered in commercial practice, up to the point where the re-distilled acid is neutralized with milk of lime for the manufacture of the gray acetate. At this stage, the present practice is to mix the pyroligneous acid with the lime in large tanks until the neutralization is complete, as shown by the color change. The principal acid constituent of pyroligneous acid is acetic acid.

$$2CH_3COOH + Ca(OH)_2 \longrightarrow (CH_3COO)_2Ca + 2H_2O.$$

It is then evaporated to a pasty consistency, dried, and shipped away as "gray acetate of lime" to the acetone plant, where it is destructively distilled in steel retorts, breaking down into acetone and calcium carbonate.



$$\begin{matrix} CH_3CO\,O \\ CH_3\,COO \end{matrix}\!\!>\!Ca \longrightarrow \begin{matrix} CH_3 \\ CH_3 \end{matrix}\!\!>\!CO + CaCO_3.$$

The acetone is condensed, washed and refined in column distillation apparatus.

APPARATUS

The apparatus used for these experiments is shown in the accompanying illustration. Its operation is as follows: The pyroligneous acid is introduced into the reservoir *A* and allowed to drop slowly into the round bottom flask *B* regulated by means of the cock *C*. The acid in the flask is heated by a carefully regulated burner *B*, which is so controlled as to cause the liquid to evaporate at the same speed with which it is dropping into the flask. The acid vapors pass through the tube *E* into the electric furnace *V*, which consists of a quartz tube *G*, surrounded by insulating material *J*, containing the electric heating element. The interior of the quartz tube is filled with the catalyzer *H*, and it is in this region that the conversion takes place. The entrance and exit connections to the heated quartz tube are made by asbestos stoppers, *F F*. The vapors leaving

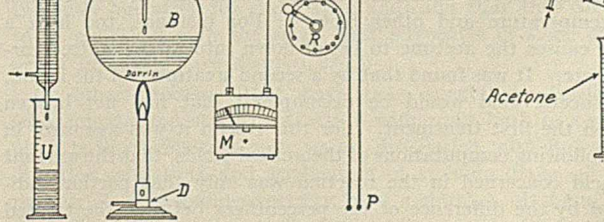


Fig. 1—Acetone Apparatus

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[1] references—U. S. Pat. (1910), 933,107: Formation of acetone from zinc, barium and magnesium carbonates at 575° C.

Pharm. Ztg., **54** (1910), 880: Formation of acetone by passing over base heated at a high temperature at 300–350 mm. pressure.

German Pat. (1910), 214,151. Addition to patent 198,852: Manufacture under reduced pressure.

Ber., **43** (1911), 2821: Acetone and ketone formed to extent of 10% when passed through pumice heated to 500–600°.

Brit. Pat. (1907), 13,263: Generation of acetone.

Ger. Pat. (1908), 198,853: Generation of acetone under reduced pressure.

Fr. Pat. (1908). Addition to 6,531 to 361,379: Generation of acetone under reduced pressure.

[1] For design of plant, see This Journal, **5**, 935.

the furnace are condensed in the condenser *K*, and the liquid caught in the graduate *L*. The temperature within the interior of the electric furnace is measured by means of the thermocouple *I*, which records the temperature on the millivoltmeter *M*. The electric furnace is connected at *N N* to 110 volt switch, *P*. The temperature within the electric furnace is regulated by means of the rheostat *R*, connected in series with the furnace. A sample of the pyroligneous acid vapors before treatment is taken from the flask *B*, by means of the tube *Q*, the amount being regulated by means of the cock *S*, so as to be the same as that coming out of the condenser *K*. These sample vapors are condensed in the condenser *T*, and caught in the graduate *U*.

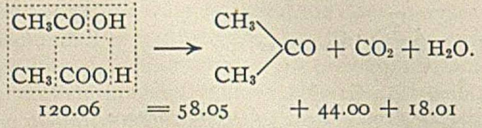
ANALYSIS

The two distillates in graduates *L* and *U* were analyzed separately for their acidity, by titration with $N/2$ KOH, using phenolphthalein as an indicator. The acidity of the liquid in each graduate was computed to the per cent by weight of pure acetic acid, CH_3COOH. The difference in the per cent of acetic acid, that the liquid in graduate *L* was less than that in graduate *U*, measured the amount of acetic acid that had been decomposed in the electric furnace.

The two distillates in the graduates *L* and *U* were also analyzed separately for the acetone, by titration[1] with $N/2$ $Na_2S_2O_7$, using starch solution as an indicator, after first adding an excess of alkaline KI solution and $N/2$ NaOCl, and neutralizing with 6 per cent HCl. The solutions of $N/2$ $Na_2S_2O_7$ and $N/2$ NaOCl were standardized every day against a standard acetone solution of known strength. A small amount of higher ketones, aldehydes, and similar bodies, which react with the NaOCl, are estimated as acetone. To eliminate the error that this would cause in the acetone determination while working with the pyroligneous acid, the blank tests for acetone were made on the untreated acid in the graduate *U*. Subtracting the amount of ketones which were determined in the untreated acid, from the amount of acetone determined in the treated liquid, gave the actual per cent by weight of acetone which had actually been formed in the furnace.

CALCULATIONS

The maximum amount of acetone that can be obtained theoretically from the decomposition of acetic acid is shown by the following equation:



$$\begin{array}{l} CH_3CO{:}OH \\ CH_3{:}COO{:}H \end{array} \longrightarrow \begin{array}{l} CH_3 \\ CH_3 \end{array}\!\!>CO + CO_2 + H_2O.$$

$$120.06 \quad = 58.05 \quad + 44.00 + 18.01$$

The figures below the chemical equation show the relative weights of the products formed; that is to say, 120.06 g. of acetic acid have a maximum theoretical yield of 58.05 g. of acetone.

The decomposition of the acetone does not always proceed as in the above equations, except under the proper control of temperature and other factors. For instance, too high a heat causes the acetone to break down into methane, free carbon, etc. It was found that by a second treatment of the liquid, any acetic acid would be decomposed that had not broken up on the first treatment. For this reason it was assumed, in the following computations of theoretical yields, that the amount of acid concerned in the reaction was only that portion indicated by the difference of the percentages between the treated and untreated liquids. The method has the further advantage of eliminating the error which is caused by the presence of other acid bodies than the acetic acid, but which the analysis computes as acetic. The theoretical yield is, therefore, computed from the analysis by means of the following formula:

$$Y = 100\frac{(B_2 - B_1)}{(A_1 - A_2)}\,\frac{120.06}{58.05} = \frac{(B_2 - B_1)}{(A_1 - A_2)}\,207$$

where Y = per cent of theoretical yield of acetone,
A_1 = per cent of acetic acid in the untreated acid,
A_2 = per cent of acetic acid in the treated acid,
B_1 = per cent of acetone in the untreated acid,
B_2 = per cent of acetone in the treated acid.

ACETIC ACID

Before the work was taken up with pyroligneous acid, the exact conditions under which the reactions best took place were carefully studied, using pure acetic acid. Altogether, 57 different samples of acetic acid were tested under various conditions of control. These were tested continuously, at the rate of about 2 a day, the entire operation on pure acetic acid extending over a period of a little over a month.

During this time the effects of various types of catalyzers were studied, the influence of the initial acid concentration was observed, and the effect of speed and temperature carefully noted. The effect of steady and intermittent heating of the electric furnace caused by the automatic breaking of the circuit at intervals of two seconds was also studied.

Table I is an extract of the analytical data, after the control conditions had been adjusted to run in a satisfactory manner.

TABLE I

Run No.	Per cent acetic acid in Untreated acid	Treated acid	Per cent acetone in Untreated acid	Treated acid	Per cent of theoretical yield of acetone
33	3.09	0.33	0.00	1.25	94.0
34	3.78	0.48	0.00	1.34	84.2
35	3.69	0.48	0.00	1.52	98.0
36	3.00	0.21	0.00	1.07	79.2
52	2.49	0.33	0.00	0.87	83.5
53	2.34	0.24	0.00	0.87	86.0
54	2.43	0.12	0.00	1.02	91.5
55	2.80	0.09	0.00	1.07	81.8
56	3.18	0.15	0.00	1.22	83.0
57	5.58	0.60	0.00	1.98	82.4
AVERAGE	3.24	0.30	0.00	1.22	86.4

When working with 2 to 4 per cent solutions of pure acetic acid, the electric furnace is able to convert 86.4 per cent of the maximum theoretical into acetone. The original strength of the acetic acid has little effect on the process as equally good results were obtained with 50, 25, 10, 5 and 2 per cent solutions. The majority of the tests were made on 2 to 4 per cent solutions.

PYROLIGNEOUS ACID

Having established the conditions under which the maximum yields were obtained working with pure acetic acid, a study was made of the yields from pyroligneous acid. Operating under the same conditions of temperature, speed, etc., that were found to give the best results for the pure acid, the data given in Table II were obtained.

TABLE II

Run No.	Per cent acetic acid in Untreated acid	Treated acid	Per cent acetone in Untreated acid	Treated acid	Per cent of theoretical yield of acetone
58	2.52	1.14	1.07	1.45	57.8
60	2.43	1.23	0.71	0.95	40.9
61	2.52	1.08	0.53	0.81	48.5
62	2.49	0.93	0.71	1.05	45.4
63	2.58	0.69	0.71	0.99	30.2
64	3.18	0.72	0.66	1.42	63.6
66	3.99	0.48	0.66	1.38	42.2
79	2.04	0.99	0.53	0.98	87.5
81	3.18	0.24	0.55	1.35	52.0
86	4.68	0.27	1.08	2.32	58.3
92	2.88	0.36	0.47	1.24	63.0
AVERAGE	2.95	0.74	0.70	1.27	53.6

Because of the small size of the furnace (capable of treating about 25 cc. of the acid per hour), it was impossible to control the speed of the distillation as accurately in proportion to the size as could be done with a commercial apparatus treating several hundred gallons per hour. Consequently small variations which were followed by similar variations in temperature within the furnace, caused local overheating, resulting in a loss of acetone. It is this variation which is the principal cause for the different yields found in various runs shown in Table I. Probably in a larger apparatus with the speed and temperature

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[1] Kebler's method, see THIS JOURNAL, **19**, 316.

properly controlled the yield would be higher than 86.4 per cent, approaching 100 per cent as a maximum.

Applying the same line of reasoning to the yields from pyroligneous acid as shown in Table II, a larger apparatus would produce a yield about 15 per cent higher than that shown in the data, or would approach a maximum of about 65 per cent.

There is still another factor which the author believes, if looked into more thoroughly than the scope of this investigation permitted, would show that the actual yield was even greater than 65 per cent; *viz.*, that the higher ketones, aldehydes, and similar bodies present in the untreated pyroligneous acid are decomposed to a considerable extent by the temperature of the furnace. If this is the case, the true amount of acetone formed by the furnace is not the *difference* between the determinations before and after treatment, but the *whole* amount after treatment. With the assumption that half of the higher ketonic bodies are destroyed in passing through the furnace, the average yield of acetone computed from the data in Table II would be 86 per cent. This is approximately the same figure as that obtained from the pure acid. The assumption that the higher ketonic bodies are destroyed in the furnace is further supported by the observation that the cloudy, yellow iodine compounds, caused by certain higher, ketonic bodies in the untreated acid, do not appear in the acetone determination of the treated acid, the iodoform settling in a flocculent state and leaving a clear, supernatant liquid.

CONCLUSIONS

I—The percentage of the theoretical yield that can be obtained by treating acetic acid in a continuous electric furnace is a little over 85 per cent, approaching 100 per cent as a maximum, under favorable conditions.

II—The yield from pyroligneous acid is similar to that from pure acetic acid, being around 85 per cent.

III—The concentration of the solution being treated has little effect on the operation.

IV—The temperature and speed are the principal factors influencing the yield, the most suitable for the apparatus employed being approximately 435° C. at a speed of 25 cc. per hour.

V—The catalyzer can be used continuously with little deterioration, a sample of barium acetate having been used continuously a little over thirty days.

VI—The process of the direct and continuous conversion of pyroligneous acid into acetone in an electric furnace containing a catalyzer is adaptable to commercial usage in plants treating several thousand gallons of pyroligneous acid per day.

VII—The advantages over the ordinary method of making acetone from the acetate, are considered to consist chiefly in:

1—Higher yields in acetone.

2—Lower operating expenses.

3—Elimination of the large consumption of lime.

4—No drying of the acetate with attendant losses.

5—Continuous operation, converting the pyroligneous acid directly into acetone without intermediate handling.

6—Low cost of apparatus.

7—Easy control of apparatus, adapting itself to an automatic thermostate arrangement, assuring uniform results.

SEATTLE, WASHINGTON

WASTE PINE WOOD UTILIZATION

By John E. Teeple

This is an old, old industry. As far back as we have definite information, people were using tar on their cordage and pitch on their ships. Even Noah's ark you recall was "pitched within and pitched without," and the ark of bullrushes that supported the baby Moses was "daubed with pitch." We lack positive assurance that a pure pine pitch was used in these cases, but it may easily have been cypress pitch, which is not so widely different. Since the industry is such an old one, the chemist is not to be either censured or credited with its results until within the last thirty years, and more especially within the last ten or fifteen years. The method commonly used formerly was to pile the wood in large heaps, cover it with earth, and by burning a portion of the wood accomplish a destructive distillation of the remainder. The products were tar and charcoal, although some few attempts were made to recover the volatile spirit for use as an illuminant.

About thirty years ago the use of iron retorts was introduced, and this made possible the recovery of an unsatisfactory turpentine, a little of the natural pine oils, and considerable tar oil, creosote oil, pitch and pyroligneous acid, in addition to tar and charcoal. The turpentine had a vicious odor, but being in small amount, it could usually be worked off by persuasion. Tar was a standard commodity, but the trade soon began to differentiate between this retort tar and ordinary kiln tar, and the purchaser demanded a reduction in the case of the former, or refused it. Charcoal could be sold for household fuel provided the plant was located in the neighborhood of a fair-sized city. Otherwise it was used to fire the retorts. Gas was also at a time recovered and burned under retorts. When pitch was not readily salable it could be dissolved in the tar oils or creosote oils and sold as tar, but these various light and heavy distillation oils accumulated, and, depending on his ingenuity, the manufacturer worked them up into paint oils, mixed paints, insecticides, disinfectants, fungicides, embalming fluids, medicinal products, and so on until the catalogue becomes alarmingly long. This destructive distillation industry still exists. It is one of the two methods of treating wood to obtain Naval Stores that has been able to operate continuously under even the present very adverse conditions. Many improvements have been made in the type of the retort, in the regulation of heat, and in the separation and preparation of products for the market. In many cases the turpentine now obtained is of excellent odor and of surprisingly good quality, but its success has usually depended on the ability of the manufacturer to work up his oils into lines of specialties under carefully established trade names, and thus protect himself from too vigorous competition. He has improved his retort tar so that now it commonly commands the same price as kiln tar, and is sometimes quoted at a higher figure. With the present high prices ruling for acetate of lime, due to the strong demand for acetone, he is even proceeding in some instances to the manufacture of this product from his pyroligneous acid. A considerable part of these improvements has, of course, been due to chemists, but it must be admitted that a further very considerable part of the preparation of products for the market has been due rather to business men who are good observers but were not primarily trained as chemists. It seems to me that the destructive distillation of wood is wrong in principle, with our present knowledge of its composition, but at the same time the fact that some of these plants have been continuously operating for many years indicates that they are not to be abolished with the speed that advocates of some of the newer processes have assured us.

Of other processes that have been suggested—distillation with superheated steam, extraction with rosin bath or tar and pitch bath, distillation with water, distillation with hot gases, digestion with soda and application of vacuum have all had their trial in a commercial way, and a number of them have seemed to operate successfully for a while, due usually to special market conditions, but for the present these have all passed into history. It is very doubtful whether any of them will appear again, at least alone, even under improved conditions. While they lasted, each one found vigorous supporters and provoked much discussion. The question, for example, whether the steam should preferably pass upward or downward through a retort, was considered of considerable moment, but now it hardly arouses our interest. Some chemist usually took an active part in developing all of these processes, but the fact that none of them are operating to-day should not be laid entirely against him. In many cases he was responsible only for the laboratory results, and plants were constructed without the aid of chemical engineers, or, in fact, of chemists or engineers of any kind. In many other cases he had no first-hand knowledge of the industry himself, and relied on faulty information as to cost of wood and market conditions, supplied by men who were promoting the plants. In any case, in developing these processes the chemist and chemical engineer accumulated, and frequently put on record, a fund of very valuable information which is not at all lost, but which will have an important bearing on the future development of the industry.

In one important respect the chemist performed valuable service during this period of development. For the first time there was produced under his supervision a quantity of wood turpentine and pine oil sufficient to have an appreciable effect on the market. He succeeded in making a wood turpentine of a much higher and more uniform grade than had ever been attained before, and by his knowledge of its uses he overcame the prejudice against it, due to its different odor and different source, in enough places so that a firm and steady outlet on good prices was obtained for all that could be supplied. For the first time, too, there was a very considerable amount of the heavy natural oils of the wood, known as pine oil, to be disposed of. This had no regular market, and he succeeded in finding a use for it in a very wide range of industries, where it has so firmly established its value that at the present time the supply cannot begin to equal the demand.

The one other process which has apparently proved of value, and which is still in operation today under adverse market conditions in at least two or three plants, is that of extraction by means of a volatile solvent. This method was at one time very extensively employed, producing more Naval Stores from waste wood than all other systems combined. The amounts of turpentine and pine oil were large and of good quality, and one new product was added from the wood—rosin. This was ultimately refined to a grade approximately corresponding to F and G grades of gum rosin. At first, progress in marketing this was rather difficult, but the oils and tarry matters affecting its use were in part eliminated, and in part the customer was taught what changes were necessary in order to use it as satisfactorily as gum rosins, so that to-day again the demand exceeds the supply.

The worst feature, of course, in the development of this industry during the last few years has been the wide fluctuation in prices. In 1911 F rosin reached a high price of $8.45 per barrel of 280 pounds, at Savannah: to-day it is $3.00—scarcely more than one-third of the high figure, and it has been lower. In 1911 turpentine reached a high price of $1.07 per gallon at Savannah: to-day it is 38c. and has been lower. Most of these plants were built on the assumption that turpentine would not go below 50c. per gallon, nor rosin below $5.00. These conditions were freely predicted at the time by people who had spent their lives in the Naval Stores industry, and so probably the chemist should not be blamed too much for the failure of processes to operate successfully at much lower figures, when he had based all his calculations on the higher figures and on the best judgment of people well informed as to conditions.

Now as to the future of the industry: Let us look first at the composition of the material with which we have to deal. Assuming that this is what we know as fat lightwood or as fat lightwood stumps, it contains rosin, turpentine, pine oil and the wood fiber. The problem is: in what way can all these products be extracted and placed on the market to bring the largest net amount to the manufacturer? In some places where the timber has been very largely the long-leaf pine, there is the additional factor of so locating his plant that he can entirely clear the land and largely enhance its value at the same time that he is furnishing his plant with raw material. In the future this will no doubt have an important bearing on the establishment of plants, but it will not be considered here. Suppose we have 5000 lbs. of very rich, fat lightwood stumps. Suppose this contains 20 per cent, or 1000 lbs., of rosin; 40 gallons or 300 lbs. of turpentine and pine oil, and 15 per cent, or 750 lbs., of water. This leaves nearly 3000 lbs. of wood fiber. Now if we subject this 5000 lbs. of fat lightwood to destructive distillation, no matter how carefully, we shall not get all the turpentine and we get only a small portion of the pine oil, before the rosin and the wood itself begin to decompose. The rosin would be valuable if we could get it, or its decomposition product, rosin oil. The pine oil would be valuable if we could get it, but before either the rosin oil or the pine oil leave the retort in any quantity they become thoroughly mixed with the tar and tar oils from the decomposition of the wood fiber, and there is no convenient way of ever separating them again in marketable conditions from the resulting mess. If we investigate any of the other methods that have been discussed above, we find an important loss in some one or more of the products originally present in the wood. If we digest with soda there is a very fair recovery of the turpentine and a moderately good recovery of the pine oil, but the rosin and resinate are not in good marketable condition, and it would be very difficult to get them in any condition for anything but very special markets. On the other hand, if we use a volatile solvent, properly selected and properly applied, we can recover nearly the whole of the rosin, turpentine and pine oil, and have left the wood fiber unchanged. The rosin, the turpentine and the pine oil will be in such condition that they can be made into the best grades it is possible to produce from wood. The wood fiber is free of rosin, and is in just the condition required for digestion to manufacture wood pulp. According to information and experiments it gives a good yield of a very strong-fibered pulp. Probably it will work up best into what is known as Kraft pulp, or undercooked pulp, and this 3000 lbs. of wood fiber remaining should furnish about 1500 lbs. of good marketable pulp.

Our wood fiber has two disadvantages as a source of pulp. In the first place it cannot be entirely freed from the charcoal and charred and burned wood, and in the second place it will not be entirely free from bark, but there is no question that it will produce a good grade of brown wrapping paper, and with this addition we have made a very complete utilization of our 5000 lbs. of wood. The difficulty heretofore has been that the Naval Stores manufacturer has approached the problem with the view of making the wood pulp plant an annex to his existing plant. The wood pulp plant is the more expensive, requires the more skilled labor, and is by far the more important, so that the rosin and turpentine production should be considered rather as an annex to the pulp plant, or, better still, the whole should be considered as one complete proposition. Many people have this combination of rosin, turpentine, pine oil and wood pulp in their minds at the present time. There is no doubt that it will be tried before long. It is only to be hoped that when it does receive its trial it will be under the supervision of men thoroughly acquainted with both the wood pulp industry and the wood extraction industry, and with the aid of the most competent chemical engineers there are available. In this way it can build on the mistakes of its predecessors, and it seems to me to stand an excellent prospect of success. There has too much good work gone into this industry, and there is too much accumulated information available to allow it to languish. I feel sure it has an important future, and when its success ultimately comes, very large credit will be due to the chemists and chemical engineers who have made it possible.

Chemists' Building, 50 East 41st St.
New York

SYMPOSIUM ON THE CONTRIBUTIONS OF THE CHEMIST TO AMERICAN INDUSTRIES

Papers presented at the 51st Meeting of the AMERICAN CHEMICAL SOCIETY, Seattle, August 31 to September 3, 1915

CONTRIBUTIONS OF THE CHEMIST TO THE NAVAL STORES INDUSTRY

By John E. Teeple
Consulting Chemist and Chemical Engineer

The Naval Stores Industry includes primarily the production of rosin and turpentine, and secondarily that of tar, pitch and various oils. The raw material in this country is the longleaf pine and some similar pines in the South Atlantic and Gulf States. The United States produces more turpentine and rosin than all the rest of the world, our total annual production being probably worth over $40,000,000; our exports in one year exceeded $27,000,000, and are usually above $20,000,000.

The industry is not a new one. When America was discovered, Russia was furnishing tar and pitch and turpentine for the cordage and ship building industries, and in our own country we were exporting turpentine and rosin in considerable quantities before the year 1800. Nearly forty years ago our exports were nearly half in quantity what they have been in recent years, though having only about one-fifth the present value.

Notwithstanding the magnitude and age of the Naval Stores Industry, however, it developed largely without the aid of the chemist. Only within the last ten or fifteen years can he claim to have played more than a minor part, but recently his contributions have been of extreme importance. We may discuss the chemical contributions under four heads:

1—Improving the quality and quantity of rosin and turpentine produced from the living tree.

2—Providing standards, and detecting and preventing adulteration.

3—Finding new uses and enlarging the markets, particularly for rosin.

4—Utilizing the rich dead pine (lightwood) and various pine wastes for the manufacture of Naval Stores.

This last field is usually classed as wood distillation, or wood extraction, and as there is a special symposium on that subject at this meeting it will not be further discussed here. When this utilization reached its maximum in 1911 to 1913, it is probable that it furnished over 10 per cent of the total Naval Stores production of the United States, and it seems likely to increase in importance in the future when conditions and prices have readjusted themselves.

The methods of producing rosin and turpentine from the living tree made very little progress during the whole nineteenth century. Timber was plenty, land, leases and labor were cheap, there was a profit to be had by existing methods, so why worry. The method consisted in hollowing out a cavity near the base of a pine tree, and cutting away bark and wood, to leave a smooth face for a few inches above it. Then every week from Spring till Fall a V-shaped chip about an inch wide was cut through bark and wood above this face, each chip being just above the preceding one. Thus inch by inch the scarred face crept up the tree, to the extent of say thirty inches a year, and at each chipping the oleoresin from the wound flowed down the scarred face till it reached the cavity at the bottom, whence it was dipped out for distillation with water over direct fire, to produce volatile turpentine and non-volatile rosin. Every year the thick, gummy exudate had to flow over a longer stretch of discolored scarred face, and be exposed to longer evaporation of turpentine before it reached the collecting cavity at the bottom. And so every year the producer's rosins were darker colored, and his yield of turpentine smaller.

But conditions began to change. Many lumber men refused to have their trees bled for turpentine, as it decreased the value of the lower end of the tree for lumber, and very largely increased their loss of timber from forest fires and from windstorms. Timber was not so plentiful as had been supposed. Prices of lands, leases, labor and materials were all rising. The cost of producing turpentine and rosin to-day is probably three times what it was twenty-five years ago. So the producer called for help. He looked first to France. There they were able to bleed a tree for many years—one is said to have survived two hundred years—while here four years was about the limit of profitable operation. There they planted large orchards of pine trees for the express purpose of turpentining them. There they made much narrower gashes in the trees, used a cup and gutters to collect the oleoresins, instead of allowing them to flow long distances over the scarred face into a hole in the tree, and there they accordingly made lighter colored, higher priced rosins, and bleached them still further in shallow pans in the sun, changing for example a K rosin into the much more valuable water-white rosin.

A study of the French methods seemed, however, to show that they were not, as such, applicable to existing conditions here. But there was an idea there, and to C. H. Herty is due the credit first of working out that idea in a scientific and practical way to fit conditions here, and second, what was probably much more difficult, of persuading the operators to use his methods for their own good. Herty conducted careful comparative scientific experiments, and showed clearly:

I—That there was no physiological reason for the marked coloration of rosins during the later years of operation as compared with the first or virgin year, but that this coloration was due to oxidation of the resin acids during the longer flow from the point of exudation to the receptacle, "the box," a large hole cut with an axe in the base of the tree, and especially to the absorption by the fresh oleoresin of the old oxidized resin on the face of the tree scarified during the previous years. By using an outside receptacle attached to the tree and moving it annually up the tree near the point of scarification, only the best grades of rosin were obtained.

II—The flow of the oleoresin over long surfaces resulted in heavy loss of the volatile spirits of turpentine.

III—The cutting of "the box" resulted in destruction of much of the timber by fire, wind and insects.

IV—Most important of all, it was demonstrated that the unboxed pine tree would produce 25 per cent more crude turpentine than the boxed tree, due to greater vitality of the tree.

The total value of the annual Naval Stores output is approximately $40,000,000. It is easily seen, therefore, that the above losses in material, both as to quantity and quality, represent an annual waste of from $10,000,000 to $15,000,000.

Early commercial confirmation of the experimental results led to the rapid substitution of the long used "box system" by the practical, cheap and efficient "cup system." The use of the cup system was not thoroughly efficient for several years, as was evidenced by the low yields of early Spring as compared with the later yields. The explanation of this shortcoming was apparent when Tschirch, of the University of Berne, published his views on resin flow and then by a slight modification of woods practice the difficulty was overcome and a further annual increase of crude turpentine valued at a half million dollars was effected.

But the end is not yet in sight, for, according to Herty, laboratory experiments on perfectly fresh specimens of crude turpentine show that its normal content of spirits of turpentine is approximately 30 per cent, while under most favorable methods of collection and distillation at present yields of only 18 to 20 per cent are obtained. This loss of at least one-third of the volatile oil by evaporation remains still to be overcome.

Some chemists have established new industries, and many have coöperated to improve old ones, but this work of Herty's in making, practically alone, such a change in an industry that had been stationary in methods for a hundred years, is almost unique.

As to other improvements in manufacture, not much progress has been made. The French method of bleaching rosin seems too slow and laborious, and many chemical methods have been suggested instead, but up to the present have not made much headway. Methods of distillation without the use of direct heat, such as using superheated steam, or distillation in vacuum have been suggested but have had little influence as yet on methods of manufacture. The United States Bureau of Forestry has recently been studying the question of chipping, and has published results indicating that a $^1/_2$ inch chip gives about as good a flow of oleoresins as an inch chip, besides being less injurious to the tree. They further recommend chipping every four days instead of once a week, thereby increasing the yield in a season. It has been objected that the tree may not be able to stand this excessive bleeding. The method is still to be tried in practice.

When turpentine was comparatively high in price, many substitutes, chiefly from petroleum distillates, and many methods of adulteration were found. Presumably we must give the chemist credit for these too. The question of adulteration particularly became a serious problem and provoked a flood of literature devising tests for various impurities and additions. Most of the large consumers finally evolved specifications to fit their requirements, and many states have passed laws for preventing and punishing adulteration, so that conditions now are much improved, for which we may also thank the chemist. There is still much work to be done, however, in standardizing turpentine specifications to fit the consumers specific requirements. As they now stand, with rather arbitrary figures on specific gravity and distillation, we, no doubt, often include material he really does not want, and exclude material he could perfectly well use.

Rosin has usually been too cheap to suffer from adulteration except with dirt or brickbats. The chemist has little to do with its purity or grading. Recently, however, the United States Bureau of Chemistry has proposed a series of permanent standards, for the different grades of rosin, based on definite transmissions of red and yellow light for each grade.

In extending the markets for Naval Stores the chemist has done little for turpentine. It has had three uses for many years: (1) As a solvent for gums in the manufacture of varnishes; (2) as a vehicle in the manufacture of paints; (3) as a thinner in the application of paints. The chemist has more often striven to avoid its use in these fields than he has to extend it. Two new fields have been opened within recent years, by chemists—one the manufacture of synthetic camphor. This has been carried on to a considerable extent in a commercial way, and will be again, whenever satisfactory relations exist between the market prices of turpentine and camphor. If the yields of camphor could be made more satisfactory it would no doubt become a permanent industry. But at present, with the price of the raw material, turpentine, and of the finished product, camphor, independent variables, and no large margin in any case existing above the fixed manufacturing cost, the outlook is not enticing to the investor. The second new field opened by the chemist is the manufacture of synthetic rubber. Synthetic rubber can be made from isoprene, and isoprene can be made from turpentine, but whether this is the best way to make synthetic rubber, and whether turpentine is the best source of isoprene, and whether synthetic rubber can compete with natural rubber in the long run anyway, are still open to discussion.

With rosin the chemist has had more opportunity. He puts it into shrapnel shells and sometimes into varnish. In the presence of an alkali, rosin acts as an acid, forming resinates. Combined with an amount of soda insufficient to saponify the whole, it forms rosin size, extensively used in the manufacture of papers, where the rosin deposited in the paper prevents the spreading of ink and moisture. This accounts for a very considerable amount of the medium grades. A somewhat higher grade of rosin, when saponified, forms a valuable constituent of, or addition to, many soaps. Combined with zinc, manganese or the alkaline earths, rosin gives resinates used extensively in varnishes.

A very large amount of rosin, particularly of the lower grades, is distilled for the production of rosin oil. The lower grades of rosin oil when mixed with lime, set to form a semi-solid mass (axle grease) extensively used as a lubricant. Better grades are used extensively in the manufacture of printing ink, so that the marvelous increase in printing and paper during the last fifty years has furnished an outlet for rosin in two directions. All of these varnish, soap, paper and printing industries have furnished abundant fields where the chemist has exercised and continues to exercise his ingenuity on rosin and its products.

In the manufacture of rosin oil the yields and quality are not always what they should be. Recent suggestions of different methods of distilling promise much larger yields of better quality oil with smaller amounts of by-products.

To conclude, we find the chemist has been particularly valuable in three departments of the Naval Stores Industry. He has improved largely the quality and quantity of product, has standardized grades and prevented adulteration, and has been very successful in finding products and derivatives to fit the markets, or in finding markets to fit his products. This last field especially is far from worked out. There is still a wide scope for the energies of the chemist.

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CONTRIBUTIONS OF THE CHEMIST TO THE STEEL INDUSTRY

By Geo. W. Sargent
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To demonstrate the service that chemistry has rendered to an industry such as that of steel, whose whole foundation is chemistry, and where even the messenger boys about the works speak of carbon, phosphorus, sulfur, silicon, etc., almost as freely as they use the terms of the baseball field, would require volumes. In fact, volumes have been published on the part taken by one element alone. To present, therefore, in a brief outline the success that chemistry has achieved in this tremendous industry is a most difficult task, and it is with considerable hesitation that this paper is presented. Perhaps by picturing the past at a time when the chemist was an almost unheard of factor in the iron and steel industry, in contrast with the present conditions, the object of this paper might be attained.

Harking back somewhat more than half a century with an old furnace man who possessed a mind as active and clear as it was at the time of which he spoke, despite his ninety years, he made the following statement: "A water wheel at the flour mill ran the blower, supplying air to us at the forge several hundred feet distant. Here the pig iron, brought up in canal boats from a charcoal furnace forty miles down the river, was melted in the run out, subsequently tapped into two forge fires, there worked to nature, and forged into blooms under a trip hammer run by the old-fashioned overshot water wheel... The blooms were cut in two pieces about 80 to 100 pounds each and then shipped by canal to Pittsburgh to be rolled into sheets, bars or rods, and then into wire; or to be melted in crucibles and made into steel. Charcoal was the fuel we used. It made mighty fine iron and steel, and that without your chemists. Two tons was a day's output, but we made it right and never had any rejected."

Picture the forge with its two "fires," but little larger than fire-places, its "run out" an enlarged blacksmith's forge on top of a hearth or crucible, and its ten men coming to work at 3 A.M. and quitting at 3 P.M. with two tons of good iron, then worth almost $250, to the credit of the sweat of their brows and twenty cords of wood sent up in smoke. Then remember that before this iron became steel it must be heated, forged or rolled into bars, cut up, charged into crucibles, melted, cast and again hammered into bars at the expense of much time and labor. This was the iron and steel business previous to 1860, with wood for fuel and no chemist. The product was expensive and its use limited; only 40,000 tons of cast steel were produced in 1872, sixteen years after the introduction of Henry Bessemer's process.

In the year 1915, with this picture in our minds, we walk through the latest steel plant, wherein are embodied the experience of the many previous years of the chemists and the engineer's efforts.

The locomotives are placing drafts of cars of 50 tons capacity, so that their contents may be added to the almost mountainous piles over which moves what appears to be a bridge without any approaches, but which is a huge crane capable of handling 2500 tons every 12 hours. It scoops up, at each mouthful, tons from the piles and quickly places the ore, limestone and coke in the respective bins from which definite measured quantities are being continually taken and by means of an inclined elevator carried to the top of a blast furnace stack and dumped into its capacious mouth, fully 85 feet above the ground. Every twenty-four hours this stack has produced 500 tons of iron of definite predetermined composition and at a fuel efficiency of 60 per cent, the highest so far obtained from any heating furnace using fuel. The molten slag, instead of being thrown over the dump, is granulated and used for making cement or as a fertilizer. The waste gases from the stack, after all possible heat has been abstracted, are burned to generate steam for the turbines producing the electricity used about the plant. To keep this blast furnace in operation requires engines for blowing 40,000 cu. ft. of air per minute at 15 to 20 lbs. per sq. in. pressure, a condensing pump, ten 350 H. P. boilers, and a water purifying plant with a capacity of 60,000 gallons per hour.

The molten iron as it comes from the blast furnace is caught in ladles holding 30 tons each and hauled by a locomotive to the open hearth department 1000 yards away. This department is contained in a building almost 1000 ft. long × 175 ft. wide × 90 ft. high. It includes, in addition to the 500-ton electrically tilted mixer, ten 75-ton open hearth furnaces. Into the mixer the molten iron is poured as one would pour cream from a pitcher, and from this mixer or reservoir a weighed quantity of molten iron is poured into a smaller ladle and charged into the open hearth furnace together with definite predetermined quantities of scrap steel, iron ore, and limestone or sand.

The steel making is begun, and after about eight hours of boiling, as it were, the requisite amounts of ferro-manganese, ferro-silicon or aluminum are added; then the 75 tons of steel are teemed into ingots weighing from 3 to 8 tons. As soon as the steel has solidified the ingots are stripped from the moulds by a huge machine, almost human in its action, then carried off to the soaking pits where they are kept hot until ready to be rolled and cut into billets. The capacity of this department is over 2000 tons of ingots per day. In the meantime drillings from a test ingot have been analyzed by the chemist, thus checking the efforts of the melter to produce steel of a definite desired composition, and the ingots are bloomed to the correct size for the next step. This blooming is done in a building over 300 ft. long × 75 ft. wide, containing, in addition to the soaking pits at one end, a blooming mill equipped with the necessary mechanical handling devices, operated by a 20,000 H. P. reversing engine and also hydraulic shears capable of cutting a 20 × 20 in. bloom.

Following our product from the blooming mill to the bar mill, we see it charged, perhaps in the form of billet 4 in. × 4 in. × 11 ft. into a continuous furnace of a capacity of 150 tons per day; thence it emerges, almost automatically, at the opposite end heated to the correct rolling temperature, and twisting and writhing like a snake it passes from one set of rolls to another and then loops back and forth through another set of rolls, finally emerging a bar over 300 ft. long, 2 in. wide and 0.260 in. thick. This mill will turn out 300 tons of this per day in straight lengths, and varying in thickness not more than 0.010 in. from the size mentioned.

The steel mill just pictured is the home of small things as well as large, and just as the efforts of the engineer have produced the 500 tons per day furnace, the 20,000 H. P. engine and the trains of rolls weighing tons in themselves, seemingly so rough or crude, and just as these in turn have enabled the manufacturer to turn out bars of steel with a variation in size of 0.010 in. and less from the desired thickness, so have the efforts of the chemist been bent towards not only estimating and controlling those elements which are found in steel in large quantities, but also those to be found there in the most minute amounts, all with the greatest accuracy and speed.

Steel would not exist were it not for the element carbon; therefore, the determination of this elment is most frequent. Although the most obvious method was to burn the metal and collect the CO_2, it was not until the last five years that this was done with assurance—a result attained through the high temperature obtainable with the electric furnace, the development of refractories and the ease with which oxygen can be obtained. Fifteen to twenty minutes after the sample is taken the melter is advised of the carbon content of his 75 tons of molten steel, the necessary additions are made and steel to the required analysis is cast; and this whether or no that analysis includes simply the usual elements, carbon, manganese, phosphorus, sulfur and silicon or the elments chromium, nickel, vanadium, tungsten, titanium or any of the other elements which are used in the production of the alloy steel. For so has the analytical chemist improved his methods that to-day the estimation of almost all the afore-mentioned elements is but a matter of minutes, and the accuracy a few thousandths of one per cent.

The extent of the analyst's work may be judged from a consideration of the data in Table I.

TABLE I—INGOTS AND CASTINGS—GROSS TONS

Years	Bessemer	Open hearth	Crucible	Electric and all other	Total
1898......	6,609,017	2,230,292	89,747	3,801	8,932,857
1899......	7,586,354	2,947,316	101,213	4,974	10,639,857
1900......	6,684,770	3,398,135	100,562	4,862	10,188,329
1901......	8,713,302	4,656,309	98,513	5,471	13,473,595
1902......	9,138,363	5,687,729	112,772	8,386	14,947,250
1903......	8,592,829	5,829,911	102,434	9,804	14,534,978
1904......	7,859,140	5,908,166	83,391	9,190	13,859,887
1905......	10,941,375	8,971,376	102,233	8,963	20,023,947
1906......	12,275,830	10,980,413	127,513	14,380	23,398,136
1907......	11,667,549	11,549,736	131,234	14,075	23,362,594
1908......	6,116,755	7,836,729	63,631	6,132	14,023,247
1909......	9,330,783	14,493,936	107,355	22,947	23,955,021
1910......	9,412,772	16,504,509	122,303	55,335	26,094,919
1911......	7,947,854	15,598,650	97,653	31,949	23,676,106
1912......	10,327,901	20,780,723	121,517	21,162	31,251,303
1913......	9,545,706	21,599,931	121,226	34,011	31,300,874

Included in the total for 1913 are about 714,357 tons of ingots and castings which were treated with ferro-vanadium, ferro-titanium, ferro-chrome, nickel, or other alloys, of which about 625,430 tons were ingots and about 88,927 tons were castings, as compared with about 892,501 tons in 1912, of which about 689,392 tons were ingots and about 103,109 tons were castings. Of the total in 1913 about 74,924 tons were Bessemer steel ingots or castings, about 599,890 tons were open hearth, about 28,279 tons were crucible, about 11,254 tons were electric, and about 10 tons were miscellaneous. Of the total production of steel ingots in 1913 about 625,430 tons were treated with ferro-vanadium, ferro-titanium, ferro-chrome, nickel or other alloys, of which about 53,751 tons were Bessemer ingots, about 535,577 tons were open hearth, about 25,281 tons were crucible, and about 10,821 tons were electric.

Therefore, assuming the sizes of the heats of steel made by the respective processes to average 10, 50, 1.5 and 6 tons, and each heat to have required one determination of each of the five usually estimated elements, we would have for the year 1913 the interesting data in Table II, in which round figures only are used.

TABLE II

DETERMINATIONS	Bessemer	Open hearth	Crucible	Electric
Carbon	954,000	432,000	81,000	5,700
Manganese	954,000	432,000	81,000	5,700
Silicon	954,000	432,000	81,000	5,700
Sulfur	954,000	432,000	81,000	5,700
Phosphorus	954,000	432,000	81,000	5,700
Nickel	.....	7,000	3,400	1,800
Chromium	.....	5,400	7,700	900
Vanadium	.....	2,700	1,700	500
Titanium	5,300	1,100	...	..
Total	4,775,300	2,176,200	417,800	31,700
GRAND TOTAL		7,401,000 determinations		

These figures are startling, and yet they do not, by any means, wholly represent the analytical work connected with the production of the steel itself. It is not to be understood that these figures represent the actual conditions, for it is very questionable if one-half the Bessemer steel heats are analyzed; on the other hand, however, many companies make two and three check determinations on a single heat of open hearth steel. The figures as a whole are very probably far short of showing the work of the analytical chemist associated with the production end of the steel industry. The analyses connected with the blast furnace, such as those of pig iron, ores, slag, coal, coke, gases, refractories and water, should be added to these and also those determinations made upon the ordinary or regularly used ferro-alloys and metals, not to mention those necessary for the production of alloy steels containing chromium, nickel, vanadium, titanium, cobalt, tungsten, copper, molybdenum, and perhaps uranium, either singly or in combination.

Frequently much of this steel when it reaches the consumer is again analyzed so that the part analytical chemistry plays in the steel industry is seen to be extensive and absolutely indispensable. Without it we could not ride from New York to Chicago in twenty hours nor drive with safety in an automobile at a fair rate of speed, nor would we have our high structures, huge bridges or viaducts, nor any of the modern mechanical equipment for operating our factories.

To obtain the high fuel efficiency of the blast furnace, the control of the Bessemer converter and of the open hearth furnace has necessitated an extensive and careful study of the weights and volumes of gases, high and low temperature measurements, heats of combustion of gases and fuel. Thus physical chemistry is seen to be an essential aid, since the metallurgy of steel involves principles which are not only chemical but also physical and mechanical. The mositure of the air was of sufficient economic importance to the efficiency of the blast furnace that its removal by refrigeration was undertaken, following a study of the physical chemistry of the blast. In the Talbot open hearth furnace, a bath of molten steel is used to excite the reaction between molten pig iron and iron ore, with a saving of fuel, since the materials themselves burn each other as it were, an achievement following the study of the thermochemical reaction between carbon and oxide of iron.

Not only has inorganic chemistry in its analytical, physical and thermo phases played a highly important part in the steel industry, but also organic chemistry is entering the field. The production of coke is accompanied by the making of coal tar which is the source of benzol and many other valuable bases used throughout the chemical and allied industries. It is now proposed by one of the largest steel producers, to take up this feature and develop it.

In concluding this presentation, which, on account of the broadness of the subject is necessarily very incomplete, mention should be made of the part electricity has more or less recently played. While the process of making steel with electricity is primarily electrothermic, an extensive chemical study of refractories and electrodes was necessary before the process attained its present status. The high temperature which could thus be gotten enabled the use of hitherto impossible combinations for slags with the result that steels of a greater purity became possible and these from scrap or iron containing high percentages of objectionable elements such as phosphorus and sulfur; the chemist was thus also enabled to produce ferroalloys otherwise impossible.

It is hoped that this article may give to those unfamiliar with the metallurgy of iron and steel some idea of the all-important part chemistry has had in the success of one of the greatest of the world's industries.

PITTSBURGH, PA.

CONTRIBUTIONS OF THE CHEMIST TO THE IRON AND STEEL INDUSTRY

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The contribution of chemistry to the iron and steel industries may be said to have begun with the introduction of the great pneumatic processes of steel making in the latter part of the 19th century. The Bessemer process, brought out in 1863, and the Siemens-Martin open hearth process which followed soon after, produced a revolution in the iron industry that gave the chemist his first opportunity. Before the days of steel, iron making was largely an empirical art, and no one considered the estimation or control of the impurities which usually accompany the metallurgy of iron to be a matter of vital importance. It was, of course, known through the operation of the puddling and crucible processes that certain elements could be depended upon to confer hardness and toughness, but the very important quantitative rôles in the metallurgy of iron played for good or ill by the ever-present five elements—carbon, manganese, sulfur, phosphorus and silicon—were not understood. It was by means of his original researches into the methods for determining and controlling these so-called impurities that the chemist began to force his coöperation upon the more or less unwilling body of contemporary metallurgists. Even within the metallurgical memory of the present writer, which does not comprise more than a third of a century, iron masters were loathe to recognize the necessity for well-equipped laboratories under the charge of college-bred chemists.

After the ore is mined and shipped, the iron industry begins in the blast-furnace in which chemical reactions involving enormous energy exchanges take place. Just as soon as the professional chemist was allowed to win his way from the laboratory into the works, revolutionary changes in output and economy were very soon accomplished. This move forward constitutes, in the opinion of the writer, the first great contribution to the iron industry made by the chemist.

In the meantime, the steel maker, in charge of the rapidly developing pneumatic processes, required to know and follow the content of carbon, manganese and other impurities during the progress of a heat. Analytical chemistry came to the rescue by devising quick methods of analysis. So rapid has been the development along this line of chemical contribution that the modern metallurgist can literally obtain his information while he waits and thus modify and control the degree and duration of his heats. The writer has information that by a recent method even carbon by combustion can be accurately determined in eight minutes from the time the sample drillings are delivered to the laboratory. The development of modern methods of rapid and accurate analysis may be held to constitute another great contribution of chemistry to the metallurgy of iron and steel.

Modern metallurgy has not been satisfied with the development and improvement of ordinary carbon steels, for modern industrial requirements have demanded the production of alloy steels possessing marvelous and, until very recently, unheard of and undreamt of physical properties. The ores of the rarest elements have been unearthed to produce these wonderful alloy steels, and the chemist has been called upon to devise ways and means for their development and study. This evolution has indeed presented many a knotty problem to the analyst as well as to the chemist in the works. It is no easy chemical task to separate, estimate and control such elements as tungsten, chromium, vanadium, titanium, nickel, cobalt, molybdenum and tantalum as every-day constituents of steel.

Space will permit of only the briefest reference to the very important place that the curious alloys have made for themselves in the metallurgy of iron and steel. The tungsten, chromium, cobalt and molybdenum alloys of varied composition constitute the modern "high speed" steels which have revolutionized machine shop practice. Vanadium and chromium are used in the manufacture of the so-called anti-fatigue steels for springs and other rapidly moving machine parts. At the New York meeting of the Eighth International Congress of Applied Chemistry, in 1912, Dr. Carl Duisberg referred to the use of some of these new alloys as follows:

"Of great importance are those alloys of iron with chromium, tungsten and vanadium which possess a high degree of hardness even at 400–500° C. They are needed by engineers for the construction of steam turbines, for the embossing and spraying of metal objects when heated to redness, a process which has lately found extensive application. Chemists use these kinds of steel wherever chemical reactions are carried out at high temperatures and pressure, as in the synthesis of ammonia according to Haber's process."

According to Dr. Duisberg, the very latest alloy manufactured by Krupp for the manufacture of safety vaults and safes can neither be drilled nor exploded nor can it be cut by the oxyhydrogen flame. In the light of subsequent events we are led to presume that the manufacture of safes was not the only purpose in the minds of our chemist-colleagues across the water. It is interesting to note in passing that unless the chemists of Germany had worked out the synthesis of nitrogen compounds from atmospheric nitrogen as well as the special steels suitable for the construction of the necessary apparatus, the present great world war would have to be brought to an early close.

Another important contribution of chemistry to the iron industry which may be called purely American is along the line of the manufacture of pure iron on the same scale which is usual in steel production. This development, which the writer is in large measure responsible for, has had to win its way against many difficulties and even much prejudice and hostility. Many hundred thousand tons of commercially pure iron are now produced annually in this country and are in demand for many purposes for which iron is more suitable than steel. Pure iron is soft and ductile with a high electrical conductivity and useful magnetic properties; for many deep drawing and enamelling purposes it is unequalled, while its slow rusting qualities have given it a wide-spread reputation and use. Some years ago the demand for pure iron could be filled only by the importation of Norway and Swedish charcoal irons; at the present time the industry is fully established in this country on the large scale which our industrial and economic conditions demand. That the chemist has contributed to this line of development there can not be the slightest doubt. It has been found necessary to eliminate the gaseous as well as the solid impurities from these pure irons, and this difficult problem has engaged the closest coöperation between the men in the laboratory and the mill.

In a brief review of this nature it is possible to touch only lightly on the many contributions of chemistry to the iron and steel industry, but the writer's experience is that unless the divisional superintendents in a modern iron and steel plant are themselves chemists as well as metallurgists, they can not be depended upon to carry on progressive work.

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CONTRIBUTIONS OF THE CHEMIST TO THE ART OF SOAPMAKING

By MARTIN HILL ITTNER
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Although the art of soapmaking is of long standing it has undergone considerable improvement during recent years. The advance in the science of chemistry has contributed in no small way to the number and magnitude of the successes in this field.

Soapmaking is connected almost inseparably with a number of other related industries among which the chief are the manufacture of alkali, glycerine, edible fats and oils, and perfumery, while the manufactures of candles and lubricants are industries closely associated with soapmaking.

Although many of the larger soap manufacturers make their own caustic soda from soda ash, the manufacture of alkali is a great industry in itself. I would be neglectful, however, if I failed to state here that the manufacture of alkali has advanced so that most of the alkali now on the market is of very high quality and is sold at a very reasonable price.

It was only a comparatively few years ago that the greater portion of the glycerine formed during the process of soapmaking was wasted. It was thrown away with the spent lyes in the process of making "curd" soap, or was left in the soap in the so-called "cold-made" soap. An increased demand for pure glycerine for the manufacture of explosives, confections, and pharmaceuticals has stimulated the soapmaker to recover his glycerine and an article that was once a neglected by-product has now assumed so much importance that no soapmaker could operate with any degree of success without recovering his glycerine, and the profit from the recovery of glycerine is frequently greater than that from the manufacture of soap itself. A knowledge of the physical and chemical properties of glycerine has been utilized in the various processes which have been devised to recover and purify this valuable body. The most important part of these processes is the distillation of the glycerine by means of steam *in vacuo*. It is not necessary to operate at a temperature sufficiently high to cause any decomposition of glycerine, as was the case in older methods, and improvements in processes employed have worked economics in yield and cost of production. Glycerine of the highest degree of purity is now regularly made on an enormous scale from soap lyes.

During the operation of soapmaking the glycerine is gradually removed from the soap by successive washes. Chemical analysis enables the soapmaker to estimate the total amount of glycerine possibly obtainable in a given operation. Chemical control and a consideration of the cost of operation taken together with the value of glycerine enable him to ascertain the most economical conditions of working.

The process of making "settled" soap, carried on under chemical control, enables the soapmaker to readily produce soap of uniformly high quality.

With an aim to the simplification of the process of soapmaking and the realization of greater economy, new methods of hydrolyzing fats and oils have been devised. In some of these methods, notably those in which sulfo-fatty acids are used, the hydrolysis is effected at atmospheric pressure. Hydrolysis by means of enzymes is also used to some extent. By these processes the larger part of the glycerine is readily recovered and fatty acids suitable for soapmaking are produced. When materials of poor quality are used, as, for instance, cottonseed soapstock, the fat may be hydrolyzed into fatty acids and glycerine, the glycerine recovered, and the dark acids subjected to distillation with steam *in vacuo*, whereby white fatty acids, suitable for making good quality soap, may be obtained.

A new method of lime saponification gives a lime soap that is brittle and readily pulverizable and which is insoluble even in boiling water. By this method, which requires somewhat more mechanical handling than other methods, all of the glycerine may be recovered and the lime soap may be converted into soda soap by means of soda ash, giving a soap of good quality.

The soapmaker is not blessed with a market in which he can obtain at will a steady supply of fats and oils of uniform quality at uniformly reasonable prices. His supplies must be drawn from all over the world and consist of animal and vegetable fats and oils. They include tallow, lard, cotton oil, peanut oil, soya-bean oil, olive oil, cocoanut oil, palm oil, marine animal oils, etc. His principal competitor in the markets of the world is the manufacturer of fat and oil food products. In some cases the soapmaker is also a manufacturer of edible products and at times converts a large part of the fats and oils which he handles into first-class food products, and sells them as such. It has thus become necessary for the soapmaker to develop methods of refining and purification whether the product is to be used as food or to be made into soap. A knowledge of the physical and chemical properties of various fats and oils used in soapmaking makes it possible to get a uniform and satisfactory result by the proper employment of different combinations, thus enabling the soapmaker to utilize the cheaper materials available at any given time. If he could not accomplish this result he would be a more serious competitor to the manufacturer of edible fats, with the result that both our edible fats and oils, and soaps would cost more to the consuming public.

The process of hydrogenating soft oils whereby hard fats may be obtained, has been worked out by a number of soapmakers. Although hydrogenated oils have been and are used on a comparatively large scale in soapmaking, their principal employment up to the present time has been in the manufacture of edible fats.

The soapmakers are the largest consumers of perfume materials in the world. Most of the soaps on the market, even the cheaper grades, are perfumed. There is a popular misconception that perfume material is used to cover up inferior materials. If this was ever the case it can scarcely be so considered now. Perfumes are added to soaps for two reasons: to make them more attractive to the consumer, and because of the fact, well known to all soapmakers, that practically all of the perfume materials exercise a preservative action on soaps, keeping them sweeter than they would be if no perfume material was used.

The manufacturers of toilet soaps include the largest perfumers in the world and the chemical study of perfume materials has come within the province of the soap manufacturer, while some of these manufacturers have entered the field of the distillation of essential oils and the manufacture of synthetic perfume materials.

The successes which have been realized in the soapmaking industry through the aid of chemistry and engineering have been of inestimable value but they are not final. The practice in this industry, however much it may have been improved, has not been perfected, and the wide-awake manufacturer is more than ever convinced by each new improvement that chemistry and engineering must be employed and followed if continued success is to be realized.

Colgate & Company, Jersey City, N. J.

CONTRIBUTIONS OF THE CHEMIST TO THE PERFUMERY AND ESSENTIAL OIL INDUSTRY

By Edward T. Beiser

Perfume Chemist, F. W. Fitch Company

As in many other fields, the contributions of the chemist to the domain of perfumery constitute a glorious romance which would fill many volumes of no small size.

Once secluded to the adornment of the royalty, perfumery has been augmented in volume and lowered in cost until today the most meager toiler may revel in "amber scents of odorous perfume." This is due to the many improvements manufacturing processes have undergone and our advanced knowledge of chemistry.

The synthesis of flower oils has, without a doubt, been the greatest achievement made in elevating the art of perfumery. With all due credit, however, to the perfumer, without whose highly trained sense of smell perfumery perfection could not be attained, yet when it comes to making plain extract of civet cat masquerade as the delicate perfume of a spring Nosegay, we have just simply got to hand it to the chemist. Through his untiring efforts we need no longer depend upon cow dung, or the vomit of the whale as the source of ambergris, but ambrein, the odor constituent of ambergris, in a fine state of perfection, is now produced in the laboratory, and from substances which have a very different effect upon our gustatory functions.

One of the first synthetic materials to find successful application in perfumery was vanillin—the flavoring constituent of vanilla beans. In 1876 Tiemann took out his first patent on a process to produce this methyl ether of protocatechuic aldehyde by means of the acetylation of eugenol and its subsequent oxidation.

The first vanillin sold for nearly $800 per pound. Through advances in chemical discovery, improved manufacturing processes, increased consumption and the opposition of rival patentees this price has been gradually reduced until today vanillin can be bought for less than one one-hundredth the introductory price.

A later discovery which gave renewed impetus to the industry of synthetic perfumery was that of Tiemann and Kruger. These chemists, after many years of patient research, finally succeeded in preparing an artificial violet perfume which they called ionone.

Ionone was formed by condensing the olefinic aldehyde citral with acetone. The olefinic ketone in the presence of alkalies formed pseudoionone of the formula $C_{13}H_{20}O$. By inversion with dilute acid pseudoionone was converted into ionone. This methyl ketone, a straw-colored oil, had such a powerful violet odor that one pound of it was equal to the perfume contained in all of the violets that you could pack into a fair sized room. Perfumers who, up to the time of this discovery, refused to admit the value of synthetic perfumes, were soon won over by the exquisite rendering of the violet made possible with this valuable chemical invention.

In rapid succession, one discovery followed another, until to-day the modern perfumer can choose from several hundred important aromatic chemicals and produce thousands of fine combinations satisfying to the most fastidious. Synthetics have made possible the perfection of many old perfumes and enable the perfumer to produce new scents undreamed of heretofore.

As mere traces of impurities will often seriously alter the basic odor of a synthetic perfume, it must, besides being chemically pure, respond to the olefactory test. And while the sense of smell in man is inferior to that developed in animals, it is nevertheless marvelously delicate. It has been found that air containing one twenty-three billionth of a milligram of mercaptan to the cubic centimeter could be detected, and it was estimated that only one four-hundred-sixty millionth of a milligram of this substance was necessary to excite the sensation of smell.

So, to pass the cultivated nose, great care is exercised by the chemist to free his laboratory creations from detrimental impurities.

Few of the natural flower oils are made up of a single aromatic substance, but are very complex, some oils having upwards of half a hundred individual constituents. Some of these constituents, while having a decided effect upon the character of the complete odor, are present in minute traces only, making recognition exceedingly difficult. But in spite of these many handicaps the chemist has produced a number of artificial flower oils which compare favorably in quality, are always uniform, and in every instance very much cheaper than the oils extracted from the flowers.

In the essential oil field also the chemist has rendered great service. It is no longer necessary to depend wholly upon the nose test in judging the value of an essential oil. Such progress has been made in the way of analysis that the perfumer is enabled to obtain not only pure oils, but such that are rich in active constituents. The composition and properties of the essential oils are so well understood to-day that the Pharmacopoeias have adopted requirements concerning them which are specific and exacting.

While not an extremely large industry, perfumery does figure in the daily life of the average American, and without a doubt brings occasional aesthetic delights to every living being who is blessed with the sense of smell. In the United States, the value of finished products, in which perfumery plays an important part, amounted to several hundred million dollars last year.

Just as the chemist has found the delightful fragrance of new mown hay to be due to the crystalline lactone coumarin, so in time he will discover and synthesize the refreshing odor of the sea-borne breeze, the exhilarating fragrance abounding within the forest after a warm rain, and the many charming odors which prevail at the various seasons in the fields.

712 Third Street, Boone, Iowa

CONTRIBUTIONS OF THE CHEMIST TO THE LEAD INDUSTRY

By G. W. Thompson
Chief Chemist, National Lead Company

To start with, we must admit that the possibilities for accomplishment in the lead industry by the chemist are, for obvious reasons, much more limited than in some of the newer industries. Lead smelting and the manufacture of most lead compounds was fairly well developed before modern chemistry began. Lead was known to the ancients, as well as were most of the lead compounds, which are now produced in large quantities. The great help modern chemistry has afforded is in the direction of preventing losses and systematizing smelting and manufacturing operations.

In the smelting of lead, there have been no radical departures due to the influence of chemistry. The old methods of oxidation and reduction are still in vogue. Many attempts have been made by chemists, particularly electrochemists, to develop simpler methods for the production of lead from its ores. None of these, however, has been commercially successful.

The principal influence of the chemist, as indicated above, has been in the study of the losses incident to smelting and the reducing of these losses to a minimum. The chemist has therefore contributed here principally to a better understanding of the operations of smelting. His influence has also been felt in the development of methods for the concentration of lead from its ores. Most of these methods are, however, distinctly mechanical, and while the modern methods of oil flotation may be considered chemical, their influence upon lead concentration has not been so great as it has been with other metals. It is true also that the chemist has done his part in those operations connected with the recovery, utilization and production under controlled conditions of fumed products, particularly in the production of basic sulfate of lead and the leaded zinc oxides.

In the refining of lead the chemist has helped through the introduction of electrolytic methods, as in the Betts process. This process, however, could hardly compete in cost with the older methods devised by Pattinson and Parkes, which methods, while involving principles of physical chemistry, can hardly be considered as distinctly chemical processes. The use of the Betts process has been particularly serviceable in the recovery of bismuth from metallic lead, which bismuth remains as a slime or mud.

It can hardly be said that the uses of metallic lead have been increased through the help of the chemist. Sheet lead and lead pipe have, of course, been used to a large extent in most chemical operations, particularly in the manufacture of sulfuric acid, and the chemist has assisted considerably in determining the best leads for such purposes. The development of lead alloys since modern chemistry has made its influence felt has been comparatively slight. Metallographical work has thrown a great deal of light upon these alloys and indicated many things with regard to their constitution and possible usefulness. It has helped in the determination of relative values. It cannot be considered, however, that chemistry has developed new lead alloys of great commercial value. The uses of metallic lead have increased considerably, more, however, through the development of other branches of science.

When we come to the compounds of lead and their uses, we find that here too most of the processes now in general operation were developed before the advent of modern chemistry. White lead is still made by the old Dutch process, and while in Germany and France the chamber and precipitation processes have been developed, no new qualities of value have been imparted to white lead by these processes, and Dutch process white lead is still considered the standard. In this country the manufacture of white lead by the Carter process has shown considerable growth, and it was the understanding of the chemical reactions taking place in the manufacture of white lead by the Dutch process that brought about the final development of the Carter or cylinder process, as well as the more recent so-called "mild" process invented by Rowley.

Chemistry has indeed been a great help in the understanding of all of the reactions that take place in the manufacture of white lead and it has helped here to reduce manufacturing costs and save losses. All of these things have been accomplished simply because of the better understanding of the reactions which take place. No specifically great improvement, however, has resulted from this better understanding of reactions. The oxides of lead, litharge and red lead, are manufactured practically as they were before the advent of modern chemistry. It is true that in the manufacture of nitrite of soda by the lead method, a considerable amount of litharge was made until the advent of cheaper methods of manufacture of nitrite of soda as a by-product of the nitrate industry in Norway. The development of the storage battery industry has created a great demand for red lead and litharge, and as the storage battery industry is electrochemical in its character, it may be considered with propriety that in this branch of industry chemistry has been of great assistance. The use of lead oxides in the curing and vulcanization of rubber goes back to the time of Goodyear, and it is very doubtful if at that time or even at the present day, the function which lead oxides perform is properly understood. The best information obtainable indicates that the lead oxides act as catalyzing agents, producing greater uniformity in vulcanization, carrying on reactions which are started either with or without heat and bringing the curing and vulcanization about with more uniform results. The oxides of lead which are used in the glass industry are made by the old methods. The glass industry itself follows the old empirical methods, in which chemistry has had little part in improving results. The study of glass, however, has resulted in the development of a better understanding of what glass is and how it can be made more suitable and permanent.

The function of lead salts in driers in the paint industry is practically chemical, and much information has been obtained as to the action of driers, giving quantitative knowledge, which has helped in the preparation of better driers and product containing them. The influence of the chemist upon the paint industry involving the use of lead compounds has been entirely subordinate to the practical results obtained. Undoubtedly the influence of the chemist has been felt in the better understanding of paint problems, but progress in this direction has been unfortunately handicapped by the development of many fallacious hypotheses, which the chemist is only now beginning to overcome. Very few new salts of lead have been developed having commercial value or importance. Acetate of lead and nitrate of lead are still manufactured by the old processes and chemistry has helped in their manufacture only to a slight extent. The use of arsenate of lead as an insecticide having qualities superior to the ordinary arsenates and arsenites of copper, etc., has grown within the last few years, with promise of still greater progress. Arsenate of lead has the advantage over other arsenical compounds of not injuring foliage to as great an extent, and remaining where it is put for a longer time.

It will be seen, therefore, that what I stated in the beginning is true. The chemist's principal influence in the lead industry has been in the better understanding of chemical reactions involved in the production, manufacture and use of lead and its compounds. Chemistry has helped in the modification of lead products to make them better and more suitable for use. It cannot be said, however, that outside of lead refining by the Betts process, the use of lead compounds in storage battery manufacture and in the production of arsenate of lead, that chemistry has had a conspicuous part. Chemistry has undoubtedly been responsible for many lead industries being able to hold their own in competition with other products, but beyond this and the part which this has in the general application of chemical knowledge, chemistry has done little.

129 York Street, Brooklyn, N. Y.

CONTRIBUTIONS OF THE CHEMIST TO THE PAINT AND VARNISH INDUSTRY

By Maximilian Toch

Director of Laboratory of Toch Brothers

The contribution of the chemist to the paint and varnish industry is not a very long story, for prior to twenty-two or twenty-three years ago the paint and varnish industry was based on rule of thumb entirely, and it is only within the last ten years that the raw materials pertaining to the manufacture of paint have been systematically classified and illustrated by photomicrographs.

If you take the example of white lead, which has been mentioned by Pliny as having been used four hundred years before Christ, re-invented by the Dutch over three hundred years ago, it will be found that this material was empirically made and in a "hit or miss" fashion. It is only within the last few years that the large lead concerns have employed able chemists who have systematized the manufacture of white lead so that all batches are uniformly produced.

It was the custom in the paint industry that dry color manufacturers, whose business really was the manufacture of high type chemicals, had practical men who owned formulas and guarded them as zealously as the alleged secrets of the masonic order are hidden. I knew a well-known manufacturer of chrome green, which in the paint industry is a mixture of prussian blue and chrome yellow, who was entirely at the mercy of his foreman because his foreman never would divulge the actual working formula of the green that he made.

For many years I have enjoyed the confidence of many of our competitors, and it would not be amiss to cite a single example of the method by which the paint or dry color industry was conducted in former years. A very large manufacturer of chrome yellow came to me one day and told me that he made upwards of one million pounds per year and did not make any money. He showed me the cost of his raw materials and the cost of his labor and could not understand where his loss came in. I examined his factory, calculated for him the amount of acetate of lead necessary to react upon a given amount of potassium dichromate and then made an analysis of the waste water from his factory, and showed him that 40,000 pounds of sugar of lead solution were going into the sewer every year, simply because he had no man in charge who knew what he was doing scientifically. I could give many instances of this kind, and it is a gratification for me to know that many of the chemists now employed in the paint and varnish industry have been originally selected by me, and that my firm, though really insignificant as compared with some of the large paint concerns, has, at this writing, five chemists including myself, and that we have had some slight measure of success is due to the scientific control of the products that we make.

Fifteen years ago I doubt whether there was any large varnish concern in the United States who had a chemist; in fact, chemists were then as they are now—frowned down upon by the practical superintendent, who always desired to surround his doings with a witchcraft and chicanery that made him imposing in the eyes of his principal; but fortunately those days have passed and the varnish foreman is more or less dominated now by the chemist. I have been so personally identified with the chemistry of paints and varnishes that those who read or hear this will know that there is absolutely no egotism in what I am saying. In the old days of the Chemists' Club, the late Professor Morris Loeb and I were the targets of every unemployed chemist in the City of New York, at least that is the way it appeared: the present existing Bureau of Employment was started by me with the help of Professor Loeb, and many of the paint and varnish chemists today owe their positions to this Bureau of Employment. This has been further reaching than the paint and varnish industry, because the oilcloth, linoleum, leather and printing ink industries, all of which use paint and varnish in different forms, are now more or less conducted on scientific principles, and many such large works are supervised by competent chemists who were originally recommended by the Bureau of Employment of the Chemists' Club.

I think this small contribution will indicate what a tremendous influence the chemist has had upon the paint, varnish and allied industries, particularly the paint chemist, who was unknown only a few short years ago.

320 Fifth Ave., New York

CONTRIBUTIONS OF THE CHEMIST TO THE PHOTOGRAPHIC INDUSTRY

By Francis C. Frary

Professor of Chemistry, University of Minnesota

If we ask what the chemist has done for photography within the last quarter-century, we may well receive the answer, "everything." For indeed there is little in modern photography which is more than a quarter-century old, and it is the developments of the last two decades that have popularized photography and at the same time wonderfully increased its usefulness to science.

Among the pioneers in the development of the modern "gaslight" papers, with their wonderful adaptability and variety, we are proud to number one of the foremost chemical engineers in this country; and none of the host of competing papers which has since arisen, could have been produced without the chemist. Careful chemical control of the quality of the paper-stock, the gelatine, and the silver salts used, is one of the first requisites in the manufacture of any photographic paper. The older printing-out paper, with all of its beauty of detail and gradation, required negatives of a quality which the average amateur seldom obtains; and it requires a modern paper to make a satisfactory print from the under-timed "snap-shots" which are the inevitable result of the popularizing of photography. Moreover, the possibility of making prints by any common artificial light has placed photography within the reach of thousands who could never have used the older papers.

The successful manufacture of the photographic film has been a great achievement for the chemist. Especially is this the case when the motion-picture film is considered. The problem of making thousands of miles of the support, of uniform high quality, and coating this with an emulsion of uniform and maximum sensibility and satisfactory stability, is one with which the chemist has had many a struggle, and for the solution of which he is entitled to much credit.

The advance in the manufacture of dry-plates in the quarter-century has been remarkable. In uniformity, keeping power, speed, and most of all in color-sensitiveness, their quality has been steadily improving. Much of this improvement has been due to the careful chemical studies of the emulsion, both in the factories and in the K. K. Graphische Lehr- und Versuchs-Anstalt, in Vienna, where a notable group of chemists have devoted themselves to a thorough study of all phases of the photographic industry. It is due to chemical research work of a high order, that we now have panchromatic plates which are sensitive over practically the whole range of the visible spectrum, and can even photograph the infra-red rays which our eyes do not perceive. The beautiful three-color work in our modern magazines would no more be possible without the panchromatic plate, than would modern press photography without the high-speed plate.

In color photography we have made considerable progress. The solution, by the Lumière Brothers, of the problem of the manufacture of a plate combining a permanent screen in three colors with a stable panchromatic emulsion, first brought natural-color photography within the reach of any well-informed photographer. Many similar plates have been introduced, and the results with this type have had a great value in many lines, particularly in medical science. Other processes for transparencies, such as those of Ives and the Eastman Kodak Co., and several for the production of natural-color prints on paper, owe their development and perfection to chemical research of a high order. Suitable aniline dyes for sensitizing the panchromatic emulsion are of course the fundamental need of all such processes, but the proper selection and adjustment of filters, color-screens, and other factors to the characteristics of the panchromatic emulsion chosen, involve a mass of careful work.

The chemist has made for us a large number of new developing agents, which have many advantages over the old-fashioned pyrogallic acid and ferrous oxalate. Improvements in the quality and uniformity of other chemicals have been greatly appreciated.

As yet we are somewhat in the dark as to the exact nature of the latent image, the theory of the action of some of the constituents in the developer, and many similar things, for enlightenment on which we must turn to the chemist. It is a very encouraging sign of good things to come, that one of the largest photographic manufacturers in the world has installed and is maintaining a large research laboratory, where chemical and physical research work on all phases of photography is being carried on by a corps of thoroughly trained men who are in touch with actual manufacturing conditions. We may certainly expect that the next twenty-five years will bring us many more great developments in photography, through the work of these and other chemists and physicists.

University of Minnesota, Minneapolis

CONTRIBUTIONS OF THE CHEMIST TO THE MANUFACTURE OF PHARMACEUTICAL PRODUCTS

By Frank R. Eldred
Chief Chemist, Eli Lilly & Company

The manufacture of medicines is not confined to pharmaceutical houses, since they do not produce many of the medicinal chemicals, volatile oils and other products which may be said to constitute a large portion of their raw materials. Many of these products, however, must pass through the hands of the pharmaceutical manufacturer in order that they may be put in a form suitable for use. It is not easy to draw the line between pharmaceutical manufacturing and the closely allied industries as their fields of activity will frequently be found to overlap; for instance the study and manufacture of certain alkaloids have been left almost entirely to pharmaceutical chemists and manufacturers although most of the alkaloids have been produced by distinctively chemical manufacturers.

Few industries have been as dependent upon the work of the chemist as that of pharmaceutical manufacturing. Many industries have been developed up to a certain point without the direct assistance of the chemist but the very beginnings of pharmacy and chemistry were closely linked together and pharmaceutical manufacturing was made possible by the work of the early chemists. It is true that pharmaceutical manufacturing has not always kept pace with the progress in chemistry, yet the chemist, although at times very imperfectly trained, has always been an indispensable factor in the development of the industry and to-day the successful manufacturers are those who are making use of the most recent discoveries in chemistry and medicine and to that end have built up organizations of well trained and highly specialized scientific workers.

The work of the chemist in the pharmaceutical industry has been characterized by the development of new products and improvement in the quality of products already established rather than by any reduction in manufacturing costs, although at the present time much attention is also being given to the latter problem. For many years it has been the custom of the more progressive pharmaceutical manufacturers to devote considerable sums of money to research along the various lines connected with their business and for this reason much of the credit for the development of our present materia medica should be given to them. Chance and uncertainty in pharmaceutical manufacturing have been largely eliminated, and while ten or fifteen years ago much of the routine manufacturing could be carried on without the direct supervision of the chemist, it is now necessary to have every crude material thoroughly examined, every process controlled and every finished product assayed or inspected by competent chemists.

Among the earlier improvements, due entirely to the pharmaceutical chemist, the standardization of preparations made from vegetable drugs deserves especial mention. The crude drugs were found to vary enormously in strength and as their active principles became known and assay processes were developed the manufacturers adjusted their preparations so that they were always of uniform strength. At that time the only pharmacopoeial standard for such preparations was the fixed amount of drug used in their manufacture and the preparations when finished of course varied according to the strength of the drug from which they were made. The standards established by the manufacturers were based upon the strength of an average prime drug and the quantity of drug required varied inversely with its strength. Most of these standards were adopted by the Pharmacopoeia many years later. This like many other improvements made by the chemist in this industry resulted in increased costs not only on account of the analytical work required but also because of the greater care necessary to maintain these standards.

Thousands of different products are manufactured by every pharmaceutical house, and it has been the duty of the chemist during the past twenty-five years to study these products in order to discover their faults and improve them by modernizing the methods of manufacture. On account of the great number of products to be studied and the diverse problems involved, progress may seem slow but if we look back even ten years we cannot fail to recognize the great improvement which has been made in the general line of pharmaceutical products. The keeping qualities of many products have been thoroughly studied although only a few years ago very little was known in regard to the stability of medicines. Much remains to be done and progressive manufacturers are sparing no effort to improve the quality and raise the standards of their products.

Many of the older classes of pharmaceutical products, however, are rapidly giving way to new medicinal agents which are established on a scientific foundation. This necessitates research with the object of developing new products to take the place of those which are falling into disuse, for the manufacturer who neglects this will see his business gradually going to his competitor who is awake to this necessity. Extracts and tinctures made from vegetable drugs can hardly be considered scientific products even though standardized to a definite content of the active principles since they always contain many other substances which are either inert or possess undesirable physiological activity. Alkaloids and other definite principles isolated from vegetable drugs have already, to a large extent, taken the place of the various extracts and we may expect even greater progress in this direction during the next few years. The preparation of active extracts and definite principles from the ductless glands is one of the remarkable developments of the last fifteen years. Most of us have witnessed with interest the increase in synthetically prepared medicines from a very small beginning to a point where they are made to meet almost every therapeutic indication. The continued advance along the line of rational and exact medication has led the manufacturer to develop many of these new and improved therapeutic agents.

In one respect the position of the pharmaceutical manufacturer differs from that of manufacturers in other lines. The U. S. Pharmacopoeia, a book of standards published under the authority of a convention whose members are appointed by various medical and pharmaceutical bodies, and the National Formulary, published by the American Pharmaceutical Association, have been made the legal standards for all preparations and substances described by them. It is evident that such legal standards are necessary and that the manufacture of medicinal products should be very carefully controlled. This being granted it is very important that the standards be correct and the control judicious. The U. S. Pharmacopoeia is revised decennially by a revision committee chosen by the convention already referred to. In making this revision the committee draws upon the published or privately communicated work of chemists and pharmacists and to some extent upon work carried on under its own direction. Much of the work of chemists for the manufacturing houses has been utilized in the compilation of the Pharmacopoeia. Since the revision committee is a representative body in which all classes interested in the preparation and use of medicinal products have a voice, probably no better standards could be established, but it must be recognized that the progress which necessitates the revision of the book every ten years also renders many of the old standards obsolete long before the new edition appears. The National Formulary is a valuable book which was designed to serve as an unofficial guide in preparing commonly used preparations which were not described by the Pharmacopoeia and its elevation to the rank of a legal standard seems to have been due to a misconception of its function.

While these two standards have been legalized by the Federal Food and Drugs Act a very wise clause was inserted in the act which provides that preparations may differ from the standards so established if the standard be *plainly* stated on the label. This clause allows uninterrupted progress during the interval between revisions of the Pharmacopoeia and insures new material and improved standards for the use of each revision committee. It is this clause which now enables the chemist to contribute actively to the manufacture of medicinal products.

In some quarters the repeal of this provision of the law has been advocated and since it has such an important bearing upon the relation of the chemist to pharmaceutical manufacturing, it should receive our careful consideration. The repeal of this clause would make it unlawful to market any product which differed in any way from the product described by the Pharmacopoeia or National Formulary; manufacturers therefore could not profit by any improvement which they might make and all research tending toward the improvement of such products would be discontinued. When it is remembered that the most important medicines are described in the Pharmacopoeia, that many are included in the National Formulary which afterward find their way to the Pharmacopoeia, and that many of the articles dropped from the Pharmacopoeia are subsequently inserted in the National Formulary, it will be seen that no more effective bar to progress in the production of medicinal products, than the elimination of this clause from the law, could be devised. It may be pointed out that the standardization of extracts and tinctures as well as many other improvements could never have been introduced by manufacturers if no variation from pharmacopoeial standards had been allowed. Under such a law while manufacturers in every other line would be stimulated by competition to improve their products, the manufacturer of medicines would be legally prohibited from doing so. The contributions of the chemist to this industry would then be confined to routine analytical work and to the development of products which could be protected by patents; all improvements in medicinal substances now included in the National Formulary and Pharmacopoeia would have to be made by the committees of revision with limited time and facilities and without manufacturing experience. It seems doubtful if such a law could be enforced but if the matter is placed before our law makers in the proper light there can be no doubt that the federal law will remain unchanged and that the state laws will conform to it in this respect. If this course is followed the progress in this industry, which has never been as rapid as at the present time, will be uninterrupted.

INDIANAPOLIS, INDIANA

CONTRIBUTIONS OF THE CHEMIST TO THE HARDWOOD DISTILLATION INDUSTRY

By S. W. KATZENSTEIN

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The hardwood distillation industry has been brought to popular attention of late, on account of the rapid increase in value of one of its products, acetate of lime, which is being converted, in large amounts, into acetone for the manufacture of smokeless powder. This recent impetus has been a great boon to the industry for, with low prices for wood alcohol since the passage of the denatured alcohol act, overproduction and low prices for acetate of lime and a constantly decreasing premium for charcoal pig iron, the outlook for the industry has not been brilliant for the past few years.

It was the last named condition which made the advent of the chemist into the wood distillation industry imperative. Necessity called him in to show how efficiency of operation, with low prices, might replace obsolete processes, stimulated by high prices. He came into the industry as efficiency engineer.

It has been in the treatment of the crude pyroligneous acid resulting from the wood distillation and in the recovery and refining of the valuable products that the contribution of the chemist has been of greatest value. Although he has shown the importance of temperature control of carbonization; of the physical condition of the wood; of the proper condensation and scrubbing of the non-condensable gases; although he proved beyond question the necessity of the abandonment of the old kiln process and the adoption of the more efficient oven or retort system; yet it has been in the treatment of the products of wood distillation that he has served the industry most usefully.

To outline this treatment of the pyroligneous acid, we have the following steps, after the settling of the crude liquor for the purpose of removing the insoluble wood tar:

1—Distillation of the crude acid liquor for the purpose of removing the tar in solution.

2—After neutralization of the crude acid liquor by means of milk of lime, separation of the volatile products, *viz.*, methyl alcohol and acetone, from the acetate of lime of the neutralized liquor.

3—Concentration and drying of the acetate of lime.

4—Concentration and refining of the wood alcohol.

The primary distillation of the crude liquor was usually a simple distillation under atmospheric pressure, carried out in copper stills, fitted with cylindrical steam coil, copper goose and vertical tubular condenser. The operation consisted of feeding the crude liquor into this still continuously, while the process of distillation was carried out by means of live steam in the coil. This was continued ordinarily for three days when the addition of crude liquor was stopped and the distillation continued until no further product was obtained. Then live steam was entered in the still and a further amount of distillate, containing a large percentage of acetic acid was obtained. The matter of keeping the coils of these stills free from tar and carbon deposit was a difficult one, especially where the liquor was obtained from the kiln process. For the retort plant, it was necessary only once a week to enter a charge of settled tar which, in this process, contains a large amount of oils.

The crude liquor generally entered these primary stills at the temperature at which it was obtained from the retort or kiln condenser. Seldom were preheaters, operated either by the use of the steam condensate from the coil or the heat in the distilling vapor used. When it is remembered that substantially 90 per cent of the crude liquor is distilled, it will be readily seen that the process was a very costly one, not only on account of the high steam consumptoin, but also on account of the large investment in copper stills, coils and necks, all of which required frequent renewal.

The separation of the volatile constituents, *viz.*, methyl alcohol, acetone and methyl acetate, from the acetate of lime of the neutralized liquor was carried out by a simple distillation in the so-called "Lime-Lees" stills—vertical iron tanks, fitted with copper coil on the bottom, with a vertical goose, consisting of an iron pipe of varying heights, leading to a surface condenser, similar in construction to that used in the primary stills. The operation of these stills consisted of alternately filling the still with the neutralized liquor and, by means of live steam in the coil, distilling the volatile constituents until the watery distillate showed, by hydrometer test, no further alcohol content. According to the nature of the liquor, that is, if from retort or kiln and also depending on the nature of the goose, the distillate obtained in this process would vary from 5 to as high as 50 per cent alcohol.

The weak acetate of lime solution remaining in the Lime-Lees stills was then pumped or drawn into storage tanks, settled and fed to open, double bottom graining pans. The solution was evaporated down and grained, with the necessary stirring or "spudding" to secure proper grain of the final product. The final drying of the acetate of lime was then carried out, either on the brick tops of the retort setting by means of the waste heat from the retort, or on an iron dry floor, set on brick flues and heated at one end by coal fire, or by means of waste boiler gases. This process required a large amount of labor in the graining and raking of the drying acetate on the floor.

The watery alcohol distillate from the Lime-Lees stills was generally concentrated in intermittent stills, consisting of an iron still, fitted with scroll coil, the vapors passing through copper columns, of various design, frequently built with perforated plates and return pipes, with tubular separator and condenser at the top for increasing the proof of the distillate by means of fractional condensation. Frequently, the refining distillations are carried out in columns built of plates, with a large number of boiling pipes and caps for the purpose of better separating the more volatile acetone from the methyl alcohol.

The process indicated above involved, in some plants, as many as seven separate distillations from the pyroligneous acid to the refined alcohol; and, where carried out with live steam under atmospheric pressure, it resulted, of course, in great fuel consumption and excessive amounts of water required for condensing and cooling purposes. To these may be added the constant loss of products, due to the rehandling of the volatile material, especially when it is remembered that, even in the northerly plants, the available water frequently rises in temperature in the summer months to a point at which losses of volatile material may be considerable. The improvements undertaken, therefore, in the last few years have had these objects in view:

1—Economy of steam in evaporation and distillation.

2—Economy of water for condensing and cooling.

3—Greater simplicity of operation involving the elimination of repeated condensations and consequent redistillations in the step-up process.

The greatest steam consumer was the first step in the process, the distillation of the acid for the removal of the tar in solution. The first departure, therefore, from the atmospheric evaporation, was the introduction of vacuum evaporators. With the recognized advantage of single- or multiple-effect evaporation, the introduction into this industry was long delayed for various reasons. In many plants which operated in connection with sawmills, refuse has been sold to the chemical plant at a round figure, generally quite favorable to the chemical plant. With the large excess of waste available, the necessity of steam economy was, therefore, not present at these plants and it was at the plants without this favorable connection or at which the utilization of lumber was closer, that the introduction was first made.

The nature of the problem involving the evaporation of a liquid containing volatile products (methyl alcohol and acetone) together with an acid (acetic) with a tarry residue, in solution and held colloidally, presented many new conditions for the builder of evaporators. Generally, their work had involved the concentration of a dilute aqueous solution, in which case the removal of the distillate at a high temperature was permissible. With pyroligneous acid, however, the presence of methyl alcohol and acetone necessitates the use of very efficient condensing and cooling apparatus to prevent losses of these valuable products. Again, the tar in solution presents a substance which rapidly forms a coating on the evaporator tubes, quickly reducing the heat transference and decreasing the boiling and circulation in the evaporator. The following conditions were essential, therefore:

1—Complete accessibility of all evaporator tubes for cleaning, inside and out. The cleaning problem varies with the nature of the crude liquor: that is, if derived from retort or kiln process, and the matter of keeping the evaporator clean is the most serious problem met with in the use of the vacuum evaporator. It is at this point that the attention of the chemist is most necessary, for it is his duty to indicate, judging from temperature and pressure conditions, in the different vapor chambers of the several effects, just where the accumulation of tar has brought about the decrease in heat conductivity.

2—Efficient condensing and cooling apparatus is of vital importance on account of the difficulty of complete condensation, while working under the high vacuum. The design of the condensing apparatus, therefore, in this case, should involve a considerable length of path for vapors and gases before the noncondensable gas is allowed to escape from the apparatus.

3—The prevention of entrainment is important on account of the possible contamination of the distillate by a small amount of tar which might be thrown over and which causes difficulty in the later drying of the acetate of lime solution. Hoods, built within the evaporator body, itself, where the evaporator tubes are vertical, have lately been found to be of great advantage in this regard and permit rapid circulation in the boiling chamber, without fear of entrainment.

Again, in the concentration of the dilute acetate of lime liquor, the introduction of the vacuum evaporator has brought about great savings in steam and consequent lowering of costs. Here, also, the possibility of losses by entrainment was present, but has been completely eliminated by the use of horizontal tubes in the construction of the evaporator. The particular type of evaporator introduced has depended upon the saving to be brought about, in other words, upon the availability of exhaust steam or sawmill refuse. Hence, the single- or double-effect evaporator would be introduced where considerable exhaust steam was available or where fuel was cheap, while the triple-effect would find its place where coal at a high price was used for fuel. Combination evaporators have been introduced with success in several plants. Thus, a common type is a double-effect evaporator, the first effect of which is an iron pan, equipped with horizontal tubes, serving to concentrate the acetate of lime solution, while the second effect, operated by the vapors from the acetate pan, is a copper pan with steam chest equipped with vertical tubes for the distillation of the pyroligneous acid. Further economy has been secured by the introduction of preheaters for the crude liquors, which use the various waste condensates as the heating medium.

The final drying of the concentrated acetate of lime solution from the evaporators is now carried out very efficiently by means of mechanical driers which have eliminated the high labor costs of the old dry floor process. Rotary cylindrical driers, operated with steam within the cylinder, with the concentrated solution without, and fitted with scrapers to remove the acetate mud, have been eminently successful, the final drying taking place on horizontally moving canvas belts traveling over a series of pipes heated by exhaust steam or, better, the wet material is fed into a continuous wire belt moving vertically counter to a current of waste gases from blast furnace stoves, or boilers, reduced to the proper temperatures for drying. The latter arrangement produces a uniform product, especially free from dust, with one-quarter the labor previously used on dry floors and with a considerable saving of steam.

Returning to the second step of our process, the separation of the volatile components from the acetate of lime solution, we find that the introduction of continuous columns has halved the amount the steam formerly used in the old Lime-Lees and the distillate is brought to high proof in one operation, thereby eliminating the step-up process of intermittent distillation. These continuous columns are of various types, frequently of plates equipped with boiling pipes and caps, the preheated neutralized liquor entering at the middle part of the column, the lower half then serving to deprive the neutralized liquor of the volatile products, the upper half concentrating the volatile distillate continuously. An improved arrangement which has been put in successful operation during the last few weeks consists of two separate columns, the first one serving only to remove the alcohol from the neutralized liquor and discharging a low-proof vapor into the middle of the second column, which, operating on this vapor, produces a high-proof distillate, miscible with water, while discharging waste oils and watery condensate from its base. This latter arrangement is a considerable improvement over previous columns in that this latter discharge will not dilute and contaminate the acetate solution, as has been the case heretofore.

In refining the crude wood alcohol, continuous columns have, in some plants, replaced the intermittent stills to a considerable extent. Columns are now in successful operation which separate the acetone from the methyl alcohol, so that a 50 per cent solution of acetone and, simultaneously, methyl alcohol containing under 0.1 per cent of acetone, are obtained continuously. The chemist's ingenuity in the production of various special solvents and mixtures has been of great value in the creation of new uses for methyl alcohol, which to-day are of great importance in disposing of the product, when the market for the regular grades is poor.

It is not within the scope of this paper to mention the work that has been done in the further development of wood distillation products, namely the manufacture of acetone and acetic acid from acetate of lime; of formaldehyde from methyl alcohol and of wood creosote from wood tar. We need only mention that these are purely chemical developments and their increasing importance to-day is a tribute to the American chemist.

Thus, the work of the chemist is apparent at every important step of the hardwood distillation industry and it may safely be said that the introduction of the improved apparatus described and its successful operation would never have been accomplished without the guidance of the chemist.

Mancelona, Michigan

CONTRIBUTIONS OF THE CHEMIST TO THE PACKING HOUSE PRODUCTS INDUSTRY

By Arthur Lowenstein
Technical Director, Morris and Company

The modern large packing house is composed of a group of industries. The packing phase itself consists usually of the slaughter of animals, resolving the carcasses into various commercial cuts, curing, smoking, canning, assorting the various by-products and their conversion into finished products, the manufacture of sausage and other meat food products, etc., etc. In addition to this phase, however, many of the large packers are engaged in a number of specialized industries on a good-sized commercial scale, which are distinctive in themselves. Some of these are: the cottonseed oil industry; the poultry industry; manufacture of "compound" (a substitute for lard), oleomargarine, fertilizers—both animal and mineral (acid phosphate), sulfuric acid, glue, hair felt, curled hair, anhydrous ammonia (from ammoniacal liquor from coal), creamery butter, canned specialties, soda-fountain products, soap and toilet specialties, pharmaceuticals—from animal products, glycerine refining, etc., etc.

The contributions of chemists to some of these specialized industries referred to have already been ably discussed in papers presented earlier in this symposium and others are to be discussed at this meeting. That which follows in this paper will therefore deal largely with the contributions of the chemist to essentially the packing house phase of the subject rather than its allied industries in which the packing house chemist and chemical engineer today actively participate.

It may be interesting to quote from a book entitled the "Modern Packing House" by F. W. Wilder under the caption "Chemical Department:" "This is a department which in all of the large well-regulated packing houses is considered very essential and the information obtained therefrom in the saving and the turning into commercial value of the by-products, as well as in determining the value of the products already produced is turned to good account. Laboratories in the larger plants are fitted up for making exhaustive tests as to the value of the products as well as for ascertaining the most practical methods for manufacturing, and many of the departments of the modern packing house had their origin in the chemical laboratory."

Another book entitled "Packing House Industries" of the International Library of Technology, after discussing the importance of the application of artificial refrigeration to these industries says: "In addition to artificial refrigeration, another most potent factor in developing the modern scientific packing industry is applied chemistry. Were it not for the ingenuity of the chemist in finding a way to utilize the offal of slaughtered animals, the price of meat would necessarily be very high. The chemist has turned into practical channels of income what was in former times not only waste, but a source of expense for its removal."

This group of industries, certainly from the time when it can be termed "modern," does not date back more than thirty-five to forty years. Chemists and chemistry have played an important part in its development almost from its inception. It is interesting to note that the leading pioneer chemists in this industry are living to-day. They have generously responded to the request of the writer for authentic information relative to the early contributions of the chemist to this industry but unfortunately, owing to the limited time and space provided for this paper, only a small portion of this evidence can be included.

In 1886, H. B. Schmidt was the first chemist to be regularly employed by a packer in the Union Stock Yards, Chicago. In the same year Dr. Rose was employed as a consulting chemist by another packer and prior to 1886 one of the packers had employed Prof. Haynes in a consulting capacity in a suit brought against them by the owners of the Mege Mouries patents for the making of oleo oil. This date, then, marks the beginning of the application of chemistry to the packing house industry in this country by the packers themselves through their own chemists. Prior to this date, however, they disposed of some of their by-products in a crude or unfinished state to concerns who employed chemists. Pressed, undried tankage and blood, glue in jelly form, evaporated tank water commonly termed "stick" are examples. Also steam-rendered lard which in those days was frequently discolored, was first improved by a chemist, W. B. Allbright, in 1879–1880, although not at that time identified with a packing house. Mr. Schmidt's description, with dates of installation of improved machinery for evaporating, drying, pressing, refining, etc., of various by-products is exceedingly interesting. These improvements generally were the result of the combined efforts of the chemical, engineering and operating staff.

The installation of vacuum evaporators, first single-effect, and later multiple-effect, stimulated the recovery of boiled beef liquors. These evaporated liquors were at first further concentrated into paste of "solid" beef extract by drying on cast-iron steam-heated revolving roll evaporators, and it was not until some years later that vacuum kettles with agitators supplanted them. It is rather interesting to note that practically since the days of Liebig the development of the extract of meat manufacture has been under the direction of chemists.

The recovery of tank water, containing dissolved nitrogenous constituents resulting from the cooking of various kinds of packing house offal, marked an important step forward both from a sanitary as well as economic viewpoint. A concern in Hammond, Indiana, employing a chemist, Dr. Van Ruymbeke, are said to have been the first to develop the evaporation of tank water, which was evaporated in a Yar Yan double-effect evaporator to a jelly form. This work was done under the supervision of Dr. Van Ruymbeke, who was the first to do this work in the Stock Yards. The material was then shipped to Hammond and mixed with copperas and dried in pans. This latter treatment with sulfate of iron was patented by Jobbins and Van Ruymbeke. Later O. T. Joslin, a chemist for one of the packers, substituted a process of his own, wherein the evaporated tank water was coagulated with a ferric sulfate solution and dried on the roll invented by him. This roll system was devised in 1892–93 and is in general use to-day.

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MANCELONA, MICHIGAN

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By ARTHUR LOWENSTEIN
Technical Director, Morris and Company

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E. K. Nelson says: "The manufacture of neat's foot oil in its various grades, lard oils, tallow and tallow oils, etc., was improved if not by the chemist, at least with his coöperation. All the daily runs were checked up constantly in the laboratory until the quality of the products was worked up to a high standard. This same thing applies to all by-products as you know."

The work of the present-day chemist, chemical engineer or technologist in these industries in addition to analytical work for control of product and supplies, may be summed up as follows:

I—Waste prevention.

II—Improvement of existing methods and processes of operation.

III—Utilization or recovery of products in a more valuable form.

IV—Initiation of new processes.

V—Direction of industrial research and application of the results of the researches until placed on a commercial basis.

VI—Investigation of and putting on a commercial basis new industries and lines of activity allied and associated with the packing industry.

Union Stock Yards, Chicago

CONTRIBUTIONS OF THE CHEMIST TO THE ELECTROCHEMICAL INDUSTRY

By W. S. Landis
Chief Technologist, American Cyanamid Company

When I was first requested to say something about the relation of the chemist to the electrochemical industry I thought I was being handed one of Goldberg's Foolish Questions. The spontaneous answer that occurred to me—he has been the foundation and is at present the mainstay of the industry, however, on mature thought requires reconsideration, for while his present status is well provided for, we should pay some attention to the growing tendency of the industry to replace his services by other classes of engineers.

An analysis of the industry, directing our attention only to that part concerned with the production of compounds, in distinction to the branch concerned with the extraction of metals, shows it to be of comparatively recent origin, beginning largely with the installation of the Niagara power developments in 1896. Most of the men who founded the industry are still alive and occupying prominent positions in the operating departments of the present plants. No one seems to have cared about compiling their biographies, though an attempt was once made in the first volume of "Electrochemical Industry." For some reason only five Americans were included in the series before the work was abandoned. Of these five, three possessed special chemical training, and while the other two would be more properly classed as electrical engineers, they were men well versed in chemical principles and were closely associated with other well-known chemists. It is unfortunate that this series of biographies was so abruptly terminated, for it would have made this note of mine superfluous.

As before mentioned, the chemist played by far the most important part in the development of the industry, and to-day I estimate that he occupies over 90 per cent of the more prominent positions in the plants in the Niagara district, which I take as typical of the industry as a whole. He grew with the industry, and with a few exceptions where others have been brought in for office, sales, and executive positions as the plants enlarged, all the staffs are still predominated by men of essentially chemical training.

But among the younger men now taking their places in the Niagara plants one notes an increasing number of men with mechanical training. This seems to surprise many not directly informed as to the present status of the industry, for next to the chemist it is not unnatural to suppose that the electrical engineer would closely press the chemist for place. It must, however, be remembered that the power companies maintain most highly organized electrical engineering staffs, which at all times are available for consultation, and many times force, I may add, almost unwelcome attention upon us when in our endeavor to push things we unbalance their circuits, or cause some other equally unintelligible trouble. It is thus seen that there is really no need for the chemical plants to carry highly specialized electrical departments. They have standardized their electrical equipment to forms of installation which demand little highly skilled attention. Most of them use low tension currents with simple distribution systems, and when the electrical equipment has once been properly installed there are very few electrical problems arising. They are much more concerned with the handling of solids, liquids and gases, the transportation and preliminary treatment of raw materials, the finishing of the product, than with their power departments, all of which emphasizes the fact that in the training of the newer electrochemists too much stress can be laid on complicated and involved alternating current theory at the expense of the elementals of chemical engineering.

My experience in this field is not large, but I cannot help but remark my discouragement with many recent electrochemical graduates who seemingly are well informed about high tension electrical engineering, but cannot instal a simple pipe line for delivering a definite amount of gas or liquid under given conditions, and a conveyor system for moving a given tonnage of raw materials in some new and unheard of device. When they come into the industry they will probably find that the 10,000 volt circuits are already installed and need no further attention from the plant management, but they will meet every day with problems of moving materials faster and more cheaply, particularly as plants enlarge and efficiencies increase.

In my opinion, it is therefore necessary to the chemist, if he is to hold his own in the present development of the industry and not give way to the mechanical engineer, to familiarize himself with the advances of the industry along mechanical lines, preferably by leaving the narrow confines of the school laboratory and spending some of his time in the other departments of technical education. Analysts will always be in demand, for chemical control of the electrochemical industries is very rigid, and particularly in the field of electric furnace products is there an unlimited opportunity for highly trained analytical chemists.

The chemical engineer possessing a representative training need have no fear on first entering the industry of finding himself in foreign parts. At Niagara the sky is just as clouded with smoke as any similar chemical center in the vicinity of New York City, all of which goes to show that combustion, steam generation, heating, evaporating, crystallizing and other well-known chemical operations have not been completely replaced by electricity. The standardization of the electrical end of the plant can be assumed before his arrival, and the new agent electricity will be found a much more tractable reagent than many of the purely chemical ones he is familiar with. But this reagent rarely starts with a crude material as such, or turns out a ready finished product from the furnace or cell, and the chemical equipment used in the preparation and the finishing is the same old apparatus found in most other branches of the pure chemical industry.

My greatest regret is that the secrecy that is maintained about the electrochemical plants cannot be withdrawn just long enough to bring in the chemical faculties of our schools, so that in the formation of the courses designed to train men for this industry a proper balance of chemical, mechanical, and electrical work may be incorporated. The future of the chemist in the industry would then be assured, and not at the expense, either, I desire to emphasize, of sacrificing that very essential of all mechanical training—fundamental general principles to specialized details.

NIAGARA FALLS, ONTARIO, CANADA

CONTRIBUTIONS OF THE CHEMIST TO THE EXPLOSIVES INDUSTRY

By CHARLES E. MUNROE
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The explosive industry is essentially a chemical industry and is most efficiently conducted under close chemical supervision. It is true that a large part of the explosives used or proposed for use in commerce are mixtures of various components, that variations in the proportions of the components or modifications of the physical conditions of the components, or of the product, may fit it better for certain of the multifarious uses to which explosives are put, and that this has given large opportunities for the exercise of empiric ingenuity which practically controlled the industry for centuries; but modern developments have proven that these mixtures can be most economically, uniformly and satisfactorily produced when their manufacture is supervised by skilled chemists who prove the degree of purity of each ingredient and, by a systematic scientific series of tests, determine quantitatively the characteristics of the product.

As a fact, while the empiricist has dominated the industry up to recent times he has nevertheless benefited from the altruistic services of the chemist, for gunpowder, the mixture which almost alone served man's purpose as an explosive through five centuries, was brought to the attention of western nations by Roger Bacon, whom E. von Meyer includes as an alchemist in his history of chemistry, and it was brought to a higher state of usefulness and reliability through the researches of Lavoisier, Berthollet, Gay Lussac, Violette, Chevreul, Bunsen, Linck, Karoyli, Debus, and other chemists, and a variation in the oxidizer was wrought when Berthollet proposed in 1785 the substitution of a chlorate for the nitrate.

With the discovery of picric acid by Woulfe in London in 1771, its confirmation by Hausmann in 1788, its identification as trinitrophenol by Laurent in 1843 and the demonstration of its explosiveness *per se* by Sprengel in 1873 began the era of strictly chemical explosives. This was followed in 1800 by the discovery of mercuric fulminate by Howard, and the elaborate investigations of the fulminates by Liebig and his students; by the discovery of guncotton and the cellulose nitrates in 1845 by Schönbein and in 1846 by Böttger; by the discovery of nitroglycerin by Sobrero in 1846; by the discovery of diazo compounds by Griess in 1858; and by the discovery of hydronitric or triazoic acid and its salts by Curtius in 1890.

With the demonstration by Berthelot of the explosiveness of other nitrosubstitution compounds than picric acid there came a widely extended and constantly increasing use for them; and the number of different compounds made use of is constantly growing for while the cellulose nitrates, with nitroglycerine, are playing the rôle of propellants, the nitrosubstitution compounds are the explosives which are being made use of in the high explosives shell now working such devastation in the present war.

I believe I have said enough to make it obvious that only by the liberal employment of trained chemists can this industry be continued or extended, and in recent years this view has been accepted and followed by manufacturers.

It may be worth while to recall here that when the Census of the Chemical Industries was taken in 1900 an inquiry was made as to the number of chemists employed, because it was then recognized that this afforded a criterion by which to determine the intelligence and foresight with which the businesses were conducted. As shown in Bulletin No. 210 of that Census there was reported but 276 chemists employed in 1740 establishments then reporting, and that but 32 chemists were employed in the 97 explosives works from which returns were secured. It is not known what effect, if any, this inquiry had but it is understood that at present a single explosives company in the United States employs alone many times this number of chemists. This improvement is gratifying to record but it should serve only as an example to be emulated.

WASHINGTON, D. C.

ORIGINAL PAPERS

THERMAL REACTIONS OF PETROLEUM HYDROCARBONS IN THE VAPOR PHASE[1]

By WALTER F. RITTMAN

Received Sept. 8, 1915

One of the most widely studied and important chemical problems of the present day is the thermal decomposition or "cracking" of petroleum hydrocarbons. A considerable number of investigations have been undertaken along this line, most of which were conducted with the primary intention of developing commercial processes. The data thus obtained have been necessarily of a rather special character and, in addition, are not particularly available to the scientific world. In view of the widespread need for clear-cut and comprehensive information concerning the "cracking" reaction a series of experiments has been outlined for the purpose of studying the problem consistently and impartially in all its phases.

The first experiments[2] were conducted in connection with the problem of oil-gas production and were limited to such conditions as might apply in that field. Attention was given to the influences of temperature, pressure and concentration on the end products of the reaction, a maximum of care being bestowed upon the properties of the evolved gases. Liquid products were examined only with regard to general physical and chemical properties.

In the present experiments it has been found unnecessary to give more than a minimum of attention to quantitative relations among gaseous products. The results of the earlier experiments were so definite and so exactly in accord with theoretical considerations that little has been added to them in the present connection. It has been possible, however, to learn facts of the greatest importance concerning liquid products of the cracking reaction and, in addition, valuable information has been obtained regarding the course and mechanism of the process.

The results at hand have led to the following conclusions with regard to the cracking reaction:

1—The nature, both physical and chemical, of an oil is of secondary importance, compared with the influence of temperature, time, and pressure, in controlling the products of the cracking reaction. Under

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[1] Published with the permission of the Director of the Bureau of Mines.

[2] Whitaker and Rittman, THIS JOURNAL, 6 (1914), 383 and 472.

like conditions approximately similar results have been obtained from five different oils and it has seemed that the minor existing differences may as probably be due to variation in rate of reaction as to the actual production of unlike equilibrium products.

2—From any oil it is possible to make any desired type of hydrocarbon by adjusting properly the conditions of treatment.

(*a*)—Most favorable conditions for gasoline production are temperatures of about 500° and pressures higher than 6 atmospheres.

(*b*)—Low-boiling aromatic hydrocarbons are produced best at temperatures between 600° and 700° with pressures above 4 atmospheres.

(*c*)—Higher temperatures favor the production of carbon and gas at the expense of the liquid reaction products.

3—The course of the cracking process is one of dehydrogenation.

4—The formation of aromatic compounds seems to occur in either of three ways:[1]

(*a*)—The original oil may be decomposed to small molecule compounds of the acetylene series which subsequently polymerize to form the larger aromatic nuclei.

(*b*)—There may be a simple splitting of polycyclic (asphaltic) hydrocarbons.

(*c*)—There may be a dehydrogenation of naphthene hydrocarbons.

THEORETICAL

The characteristic feature of the present set of experiments is that cracking has been conducted in the vapor phase. The advantages of this method of procedure are considerable and, as will be shown in a later connection, have as much importance from a commercial as from a purely scientific point of view.

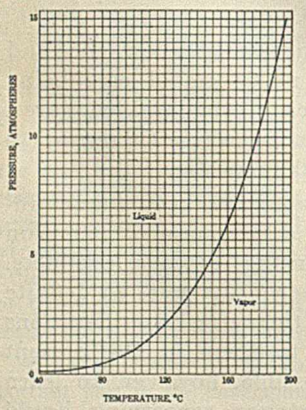

FIG. 1—VAPOR PRESSURE OF WATER

The primary advantage of the one-phase system is that both temperature and pressure may be controlled separately and at will. In a two-phase system each is a function of the other. The simple vapor pressure curve diagram for water (shown in Fig. I) illustrates the characteristic difference between a one-phase and a two-phase system. If liquid and vapor are in equilibrium with each other we are confined to the line representing the variation of vapor pressure with temperature. If an attempt is made to change one of these variables and keep the other constant a phase disappears. Thus any attempt to heat water above 100° C. at atmospheric pressure tends to produce entire conversion to the state of vapor. If, however, we deal with the vapor phase only we may obtain any desired combination of temperature and pressure as long as conditions are avoided which cause condensation. The vapor pressure of petroleum, which is a mixture and not a simple chemical compound, is not exactly analogous to that of water as the liquid and vapor phases differ in composition and thus permit the establishment of various conditions of equilibrium; if temperatures are raised sufficiently high, however, the liquid will disappear, as in the simpler case.

As applied to present needs the limitations of the two-phase system become evident. If it is desired to heat to 850° C. an oil boiling below 300° enormous pressure must be developed to retain the liquid phase, more pressure, probably, than apparatus of ordinary construction will withstand. Yet it is perfectly easy and simple to pass oil vapor through a tube heated to 850° and at the same time maintain in the system any desired pressure from a fraction of an atmosphere to over thirty atmospheres.

Other advantages of the vapor-phase system for heat decomposition of petroleum hydrocarbons are more mechanical than chemical. The influence due to differences among the physical properties of various original oils is eliminated. In systems which involve cracking by distillation under pressure the amount of pressure developed in getting an oil up to decomposition temperature depends upon the volatility of the oil. The total conditions and range of applicability of a process may be controlled by this factor. When, however, as in the vapor-phase system, the interdependence of the two variables, temperature and pressure, has been eliminated, the influence of volatility of the oil is a minor consideration. It is controlled by adjustment of the vaporizing device, through which the oil passes before entering the body of the cracking furnace.

The vapor-phase cracking process, as here conducted, possesses another characteristic which is advantageous, both from a scientific and from a commercial point of view. In the present work and in a majority of commercial cracking processes the production of gas is undesirable. It will be noted that the method here employed minimizes its formation. The mechanism by which this end is accomplished is as follows. In the cracking furnace we have established an equilibrium which may be represented by the following equation:

$$\left.\begin{array}{l}\text{Condensable}\\ \text{Original Hydrocarbons}\end{array}\right\} \rightleftarrows \left\{\begin{array}{l}\text{Permanent}\\ \text{Gases}\end{array}\right\} + \left\{\begin{array}{l}\text{Condensable}\\ \text{Cracked Hydrocarbons}\end{array}\right.$$

At the lower end of the cracking tube is a Liebig condenser which removes by cooling the unreacting residuum of original hydrocarbon and the condensable products, but which does not affect the permanent gases (hydrogen, methane, etc.). As a result when more of the original hydrocarbons enter the furnace a new equilibrium is established without any further production of gas. In actual practice this condition is not attained in absolute degree but the above description indicates a strong general tendency.

The factor of safety is another important consideration. In the course of the cracking reaction only a

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[1] Lewes, *Jour. Soc. Chem. Ind.*, **11** (1892), 584; R. Meyer, *Ber.*, **45** (1912), 1609; R. Meyer, and Tanzen, *Ber.*, **46** (1913), 3183; Ipatiew, *Ber.*, **44** (1911), 2978; Brooks, Bacon, Padgett and Humphrey, THIS JOURNAL, **7** (1915), 180.

small portion of oil is in the zone of action at any one time and in case of failure of apparatus to remain tight there is no large volume of hot oil to be discharged.

Valuable indications of what may be expected as products of the cracking reaction may be obtained by considering equilibrium relations between a typical hydrocarbon and its possible decomposition products. The initial substance chosen for present purposes is a hydrocarbon of the paraffin series, representative of the kerosene cut which modern commercial conditions have rendered desirable of transformation. The compound $C_{12}H_{26}$, boiling at about 215°, has been employed as a basis for a number of typical reactions (Table I), it being understood, however, that with each of the major reactions is coincident a number of minor reactions.

It is also to be understood that present considerations do not neglect the possibility of cracking polycyclic hydrocarbons directly into aromatic compounds. Here the case may be one of simple splitting, such as occurs in the transformation of a paraffin molecule to a paraffin and an olefin. By starting with a paraffin the case is made inclusive and is therefore best suited to purposes of illustration.

The possibilities of decomposition may be classified under the following four heads:

1—Paraffin formation
2—Olefin formation
3—Acetylene formation
4—Aromatic formation

The list of typical equations includes all these cases, and the approximate equilibrium relations point out general tendencies of the various reactions. It is to be noted that for all reactions save paraffin rearrangement (Equations 1 and 2) and aromatic formation (Equations 15 and 16) the effect of pressure is inimical, as the transformations are accompanied by increases in total volume. This consideration is, however, modified by the necessity of taking account of the factor of reaction velocity. According to the law of mass action, reaction velocity is proportional to concentration, and concentration is a derived function which varies in the same direction as pressure. If we allow reactions sufficient time to proceed to completion low pressure is favorable for the transformations represented by Equations 3 to 14. When, however, time is limited to a fixed amount the effect of low pressure may be unfavorable unless the reaction occurs with considerable rapidity.

All the reactions indicated, except the last two, take place with absorption of heat and therefore are favored by high temperature. This is indicated by the magnitudes of the approximate equilibrium constants which are figured according to the Nernst equation.[1] Here again it must be remembered that the case is not a simple one and that certain of the reactions take place at the expense of others. Especially is it true that the formation of hydrogen and carbon, undesirable end products, is favored by high temperature and it cannot be said, therefore, that to obtain a maximum yield of such a substance as acetylene (Equation 7) it is desirable to heat as strongly as possible.

A few specific cases deserve attention. It seems entirely reasonable to assume that olefin formation is intermediate to acetylene formation. Likewise there are clear indications to the effect that aromatic compounds are polymerization products of acetylenes. Hence the following relations seem to hold for the effects of temperature and pressure. Acetylenes are formed at moderately high temperatures and being intermediate to aromatics the latter are likewise high

TABLE I—QUANTITATIVE STUDY OF EQUILIBRIA

No.	REACTION	Heat of reaction (a)	Volume change	(Formulas refer to partial pressures)	APPROXIMATE		
					K_{500}	K_{600}	K_{750}
1	$2C_{12}H_{26} \rightleftarrows CH_4 + C_{23}H_{48}$	— 500	2 to 2	$K = \frac{CH_4 \times C_{23}H_{48}}{(C_{12}H_{26})^2}$	0.306	0.308	0.309
2	$2C_{12}H_{26} \rightleftarrows C_{10}H_{22} + C_{14}H_{30}$	± 000	2 to 2	$K = \frac{C_{10}H_{22} \times C_{14}H_{30}}{(C_{12}H_{26})^2}$	1.0	1.0	1.0
3	$C_{12}H_{26} \rightleftarrows CH_4 + C_{11}H_{22}$	— 16750	1 to 2	$K = \frac{CH_4 \times C_{11}H_{22}}{C_{12}H_{26}}$	645.	2820.	15500.
4	$C_{12}H_{26} \rightleftarrows C_{10}H_{22} + C_2H_4$	— 16100	1 to 2	$K = \frac{C_{10}H_{22} \times C_2H_4}{C_{12}H_{26}}$	1950.	8300.	43500.
5	$C_{12}H_{26} \rightleftarrows C_{12}H_{24} + H_2$	— 31800	1 to 2	$K = \frac{C_{12}H_{24} \times H_2}{C_{12}H_{26}}$	0.0044	0.0616	1.02
6	$C_{12}H_{26} \rightleftarrows 5C_2H_4 + C_2H_6$	— 80400	1 to 6	$K = \frac{(C_2H_4)^5 \times C_2H_6}{C_{12}H_{26}}$	1.25×10^{16}	1.58×10^{19}	6.3×10^{22}
7	$C_{12}H_{26} \rightleftarrows 6C_2H_4 + H_2$	—111600	1 to 7	$K = \frac{(C_2H_4)^6 \times H_2}{C_{12}H_{26}}$	1.2×10^{14}	2.0×10^{18}	1.6×10^{23}
8	$C_{12}H_{26} \rightleftarrows C_2H_2 + 2C_5H_{12}$	— 47600	1 to 3	$K = \frac{C_2H_2 \times (C_5H_{12})^2}{C_{12}H_{26}}$	7.1×10^2	4.0×10^4	3.9×10^6
9	$C_{12}H_{26} \rightleftarrows C_2H_2 + 2C_4H_{10} + C_2H_4$	— 61100	1 to 4	$K = \frac{C_2H_2 \times (C_4H_{10})^2 \times C_2H_4}{C_{12}H_{26}}$	7.4×10^6	1.4×10^9	6.2×10^{11}
10	$C_{12}H_{26} \rightleftarrows 6C_2H_2 + 7H_2$	—381000	1 to 13	$K = \frac{(C_2H_2)^6 \times (H_2)^7}{C_{12}H_{26}}$	1.0×10^{-20}	5.0×10^{-7}	1.2×10^9
11	$C_{12}H_{26} \rightleftarrows 5C_2H_2 + 6H_2 + C_2H_4$	—336800	1 to 12	$K = \frac{(C_2H_2)^5 \times (H_2)^6 \times C_2H_4}{C_{12}H_{26}}$	3.5×10^{-15}	7.1×10^{-3}	2.9×10^{11}
12	$C_{12}H_{26} \rightleftarrows 3C_2H_2 + 4CH_4 + C_2H_4$	—154400	1 to 8	$K = \frac{(C_2H_2)^3 \times (CH_4)^4 \times C_2H_4}{C_{12}H_{26}}$	1.1×10^{11}	5.6×10^{16}	2.4×10^{23}
13	$C_{12}H_{24} \rightleftarrows C_2H_2 + C_{10}H_{22}$	— 29300	1 to 2	$K = \frac{C_2H_2 \times C_{10}H_{22}}{C_{12}H_{24}}$	0.89	10.2	162.
14	$C_{12}H_{24} \rightleftarrows 4C_2H_2 + 4CH_4$	—167600	1 to 8	$K = \frac{(C_2H_2)^4 \times (CH_4)^4}{C_{12}H_{24}}$	5.6×10^7	7.1×10^{13}	7.8×10^{20}
15.	$3C_2H_2 \rightleftarrows C_6H_6$	+130700	3 to 1	$K = \frac{C_6H_6}{(C_2H_2)^3}$	2.0×10^{20}	6.3×10^{15}	5.7×10^{10}
16	$2C_2H_2 + C_3H_4 \rightleftarrows C_7H_8$	+131900	3 to 1	$K = \frac{C_7H_8}{(C_2H_2)^2 \times C_3H_4}$	6.3×10^{20}	2.2×10^{16}	1.6×10^{11}

(a) Whenever possible heats of reaction (for constant pressure) are based on values obtained by experimental methods. In other cases calculated values are used, based on the empirical formulae given by Thomsen. ("Thermochemischer Untersuchungen," Part IV, Chapter XIV.)

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[1] Nernst, "Theoretical Chemistry;" also text-books of physical chemistry —Whitaker and Rittman, *Loc. cit.*

temperature products. Acetylene formation appears to be favored by low pressure but the polymerization is a reaction helped by high pressure and this, in addition to the factor of reaction velocity, leads to the expectation that aromatic formation must be favored by pressure.

The ultimate products of the cracking reaction are carbon and hydrogen, their formation being favored by high temperature. Being inactive and undesirable end products they should be minimized by moderation in securing the latter condition.

EXPERIMENTAL

GENERAL SCHEME OF PROCEDURE—A scheme of procedure similar to that described in an earlier paper[1] has been adopted for the present series of experiments, though the study of products has been conducted on a broader scale. The course of a single experimental unit was as follows: a quantity of oil, generally between 400 and 600 grams, was run through an electrically heated furnace in which were maintained carefully regulated and measured conditions of temperature and pressure. After the completion of the "run" the quantity of resulting oil was determined and the deposited carbon scraped out of the furnace and weighed. The evolved gas was collected in a suitable receiver and its volume read and recorded, although no use has been made of this information in the present connection.

In connection with each major experiment or run a number of supplementary determinations were necessary. Both uncracked and cracked oils were submitted to analytical distillations and specific gravities were measured for total oils and for distillation cuts. Viscosities were determined for original and recovered oils. A number of refractive indices were measured, though these, like gas volumes, are not recorded. Refractive indices have been found to agree so exactly with specific gravities[2] that these constants furnished no additional information of value.

In some cases when the presence of aromatic hydrocarbons was indicated by the gravity figures, chemical examinations were made and the reliability of the former determinations verified. The matter of chemical methods of separation has received consideration in other communications.[3] Samples of evolved gases were removed from the holder and later analyzed. This work will be described later.

The total scheme of procedure may be summarized as follows:

"RUN" OR MAJOR EXPERIMENT

1—Cracking of 400 g. to 600 g. of oil in furnace at regulated temperature and pressure.

2—Determination of quantities of oil, gas and carbon resulting.

SUPPLEMENTARY DETERMINATIONS

1—Distillations of: (*a*) uncracked oil, (*b*) cracked oil.

2—Determinations of specific gravities of uncracked oil, cracked oil and individual distillation cuts.

3—Determinations of viscosity and refractive index.

4—Chemical tests on distillates.

5—Analyses of evolved gases.

CRACKING EXPERIMENT OR "RUN"—The apparatus and general method used for making a run was the same as that described in an earlier paper.[1] The apparatus consists essentially of an electrically heated furnace body eighteen inches long and one and a half inches in diameter. At the upper end is attached an oil feed cup and a vaporizing device. Below the furnace is a Liebig condenser discharging into a receiver which is connected with either a rotary type vacuum pump or a pressure release valve. A final connection is made with a gas holder of 12 cu. ft. capacity. The whole order of the apparatus was therefore: (1) oil feed cup, (2) vaporizer, (3) furnace proper, (4) condenser, (5) first receiver, (6) vacuum pump or pressure valve, (7) second receiver in case of vacuum, and (8) gas holder. The system is a closed one and any desired pressure from a thirtieth of an atmosphere up to 30 atmospheres has been maintained. The electrical heating equipment of the furnace body permits the regulation of temperature at any desired height up to 1000° C. The temperature measurements were made by means of a thermocouple inserted in the interior of the furnace body.

DISTILLATIONS were made in flasks of 250 cc. bulb capacity, with necks $^5/_8$ in. in diameter and 6 in. long to the outlet tube. In each case the neck of the flask was equipped with a 5 in. column of the Hempel type, filled with aluminum beads. Original charges of 200 grams were distilled in most cases and cuts were made at 100° C. and therefore at intervals of 50°.

SPECIFIC GRAVITIES were determined either by means of a Westphal balance with plummet of one cubic centimeter displacement or else with Drushel type pycnometers.[2]

VISCOSITIES were measured in the Engler viscosimeter and results are expressed in Engler degrees, which represent ratios between rates of flow of oils and water.

REFRACTIVE INDICES were measured with the Pulfrich refractometer.

GENERAL RESULTS OF EXPERIMENTS

The results of a large number of the cracking experiments appear in Table II. The necessary arrangement of figures does not render them particularly easy to interpret, and more readable tables are given in connection with the various points set forth by these experiments (see various sections of Table III). It will be noted, however, that experiments were performed with five different oils, most attention having been given to three, which were a Pennsylvania refined burning oil, an Oklahoma fuel oil and a California Kern River crude. These oils were particularly suited to the present experiments as they are devoid of low-boiling constituents; they are, therefore, of the class least valuable commercially, which renders them most promising as starting material for cracking re-

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[1] Whitaker and Rittman, *Loc. cit.*

[2] Rittman and Egloff, THIS JOURNAL, 7 (1915), 578.

[3] Rittman, Twomey and Egloff, *Met. Chem. Eng.*, Oct. 1, 1915; Rittman and Moore, *Ibid.*, Oct. 15, 1915.

[1] Whitaker and Rittman, *Loc. cit.*

[2] Rittman and Dean, THIS JOURNAL, 7 (1915), 185.

actions. They are also typical of the three great fields of the country, the eastern, the mid-continent and the western. In addition to the above three Mexican and Russian crude oils were examined and sufficient data obtained to show that, in spite of their different chemical nature and physical properties, these do not vary radically in their behavior from the others. Figures for the Russian oil were so few that they have not been used in the later tables prepared for purposes of comparison.

It will be noted that the ranges of temperature and pressure are considerable, and that the entire regions of importance in the cracking reaction have been covered thoroughly. Temperatures lower than the ones used do not produce decomposition with sufficient rapidity while those higher in the scale generate as maximum end products undesirable carbon and gas. The range of pressure was as great as could be managed with apparatus of ordinary construction.

STUDY OF RESULTS (See Tables III and IV)

CARBON FORMATION—The first factor to be considered is one which, though of the greatest importance from a commercial point of view, is of but minor theoretical interest. Carbon formation was determined by the rather crude method of scraping the interior of the cooled cracking tube after each run and weighing the solid material thus removed. In spite of the roughness of the method valuable indications were obtained as to general tendencies.

A study of the data in Table III shows first that there are characteristic differences among the series run with the various original oils. These differences may be attributed to a combination of two causes, one chemical, the other physical. The chemical factor may in turn be subdivided. The four oils varied in their carbon content, partly because there were differences in the average sizes of the hydrocarbon molecules and partly because of chemical differences among the basic natures of the oils. Those of an asphaltic base have a higher carbon percentage than those of paraffin base. Hence the following order: The Pennsylvania oil, which deposited least carbon, was a "water-white" kerosene of purely paraffin base and boiling so that 90 per cent came over below 250° C. The Oklahoma oil was intermediate and was of mixed paraffin and asphaltic base; a so-called "fuel oil" of which only 22 per cent distilled below 300°. Still more carbon was deposited by the California oil, a heavy Kern River crude of asphaltic base and giving a distillation cut of only 17 per cent below 300°. The Mexican oil was lighter than the California and gave a distillation cut of 37 per cent below 300° but it had a peculiar physical property of causing much heavy tarry material to be included in the carbon deposit. As a result the figures for carbon formation are high and the yields of recovered oil and the gravities of recovered oils low.

A comparison of averages of irregular sets of figures showing such marked variations is necessarily inaccurate and is almost unscientific, yet it happens to show the result which would be expected from theoretical considerations. Carbon deposition is favored by both temperature and pressure.

VISCOSITY OF RECOVERED OIL—In a considerable number of experiments it was inconvenient to make measurements of viscosity on account of insufficiency in the quantity of recovered oil.

It will be noted that, although there are considerable differences among the viscosities of the original oils, the analogous figures for recovered oils show but moderate variations. This fact is one of a number going to show that the conditions of treatment of an oil are of much more influence than any properties, physical or chemical, which it originally possesses.

A specific comparison brings this out clearly. The ratio of viscosities between the original Pennsylvania and Oklahoma oils was about 1 : 35. The ratio between averages of the two series of recovered oils is about 1 : 3, and, by omitting two vacuum runs of Oklahoma oil, the ratio can be brought down to 1 : 1.5.

No attempt has been made to average the viscosity values, as sufficiently instructive indications can be obtained by a study of any one of the series. That for the Oklahoma oil is most complete and it has been selected for use in Table III. Here it appears that for runs up to 750° C. viscosity decreases with temperature and with pressure. It does not, however, seem probable that this rule will hold for higher temperatures and the single figure obtained for 800° goes to prove the truth of this reasonable expectation. A similar figure for California oil indicates likewise an increase in viscosity as the 800° mark is approached.

SPECIFIC GRAVITY OF RECOVERED OIL—The variation among the specific gravities of the different sets of recovered oils is likewise a factor of interest. Here, as in the case of carbon deposition, it will be noted that there are characteristic differences among recovered oils from the various sources and that these are of the same order as the gravity differences among the original oils. Pennsylvania cracked oils are lightest, Oklahoma next and California heaviest. The Mexican, on account of the tendency of heavy constituents to stick to the carbon, are a little lighter than the California, although the original oil was heavier.

Here again average values are an uncertain proposition and must be regarded with due caution. It appears definitely, however, that gravities increase with temperature. It is to be expected that they would likewise increase with pressure and there are indications that such is the case. Most of the deviations can be explained on the basis of probable experimental error.

PERCENTAGE OF OIL RECOVERED—The percentage of oil recovered is a factor of the greatest importance but, unfortunately, the necessary conditions of the present experiments prevented its determination with any degree of accuracy, especially in the cases of reactions under pressure. The pressure in each case was "built up" by gas generation due to cracking of original oil and there was therefore in each experiment a considerable period during which pressure was not up to the prescribed mark. In addition it was

TABLE IIA—RESULTS OF EXPERIMENTAL RUNS WITH PENNSYLVANIA, OKLAHOMA AND CALIFORNIA OILS

Temperature:	850	850	800	750	750	750	750	650	600	600	600	600	600	600	550	550	500	500	500	500	500	500	450	450	ORIG.
Pressure—Atmospheres	1	Vac.	Vac.	12	6	1	Vac.	Vac.	24	18	12	6	1	Vac.	24	18	24	18	12	6	1	Vac.	18	12	OIL
PENNSYLVANIA OIL:																									
Oil used (grams)	731	.....	.....	372	545	893	800	.....	568	572	547	861	591	750	.....	.....	576	539	561	951	750	750	.....	575(a)	.....
Oil recovered (per cent)	9.7	.....	.....	15.9	16.9	16.5	33.9	.....	37.3	40.5	43.6	31.4	59.9	97.4	.....	.....	72.5	79.7	84.2	57.8	94.5	(753 g.)	.....	93.1	.....
Carbon (per cent)	7.4	.....	.....	24.3	15.3	3.7	16.1	.....	7.5	5.3	3.6	2.4	0.6	0.0	.....	.....	1.2	0.0	0.0	0.0	0.0	(6 g.)	.....	5.1	.....
Sp. gr. of oil recovered	1.026	.....	.....	0.910	1.020	1.012	0.905	.....	0.962	0.926	0.918	0.885	0.840	0.811	.....	.....	0.828	0.819	0.808	0.810	0.799	0.802	.....	0.809	0.802
Viscosity	.....	.....	.....	.....	.....	.....	1.04°	.....	.....	.....	1.11°	1.00°	.....	1.04°	.....	.....	.97°	.96°	.98°	1.00°	1.00°	1.04°	.....	1.02°	1.05°
First drop	70°	.....	.....	75°	70°	60°	.....	.....	36°	30°	33°	.....	31°	.....	.....	.....	32°	30°	30°	40°	36°	.....	.....	30°	120°
Temperature	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%
100	18.0	.....	.....	30.2	31.6	16.8	3.7	.....	19.0	19.0	18.0	14.5	8.7	0.5	.....	.....	13.5	9.5	9.5	5.0	1.0	0.0	.....	3.5	.....
150	7.0	.....	.....	9.4	9.7	14.3	14.3	.....	21.5	25.0	24.2	25.0	18.5	3.5	.....	.....	16.2	14.5	14.3	10.0	6.7	3.0	.....	8.3	4.8
200	10.0	.....	.....	17.9	8.3	8.9	39.2	.....	9.3	12.7	15.0	21.0	36.5	44.5	.....	.....	27.3	32.7	34.0	38.8	42.5	46.0	.....	34.0	46.7
250	30.0	.....	.....	25.5	22.2	20.7	23.7	.....	13.5	15.3	14.0	16.5	23.5	41.0	.....	.....	25.0	28.5	27.3	33.0	38.0	37.5	.....	33.0	39.0
300	.....	.....	.....	.....	.....	8.2	6.3	.....	.....	.....	.....	7.5	5.7	.....	.....	.....	.....	.....	.....	8.0	.....	.....	.....	.....	.....
150	25.0	.....	.....	39.6	41.3	31.1	18.0	.....	40.5	44.0	42.2	39.5	27.2	4.0	.....	.....	29.7	24.0	23.8	15.0	7.7	3.0	.....	11.8	4.8
250	40.0	.....	.....	43.4	30.5	29.6	62.9	.....	22.8	28.0	29.0	37.5	60.0	85.5	.....	.....	52:3	61.2	61.3	71.8	80.5	83.5	.....	67.0	85.7
Temperature	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G	S. G.	S. G.	S. G.
100	.875	.....	.....	.865	.876	.876	.800	.....	.837	.816	.807	.772	.759	.....	.....	.....	.726	.714	.725	.735	.718	.....	.....	.715	.....
150	.859	.....	.....	.834	.860	.878	.832	.....	.862	.855	.851	.829	.803	.770	.....	.....	.802	.790	.786	.780	.761	.760	.....	.778	.763
200	.885	.....	.....	.824	.873	.922	.848	.....	.876	.860	.857	.838	.815	.791	.....	.....	.813	.806	.801	.793	.787	.786	.....	.793	.784
250	Solid	.....	.....	Solid	Solid	Solid	.904	.....	Solid	.931	.930	.889	.850	.818	.....	.....	.850	.839	.831	.820	.814	.813	.....	.820	.811
300	.....	.....	.....	.....	.....	(b)	.956	.....	.....	.....	.....	.956	.901	.....	.....	.....	.....	.....	.....	.851	.....	.....	.....	.....	.....
150	.873	.....	.....	.858	.872	.877	.826	.....	.847	.837	.833	.809	.790	.769	.....	.....	.771	.765	.763	.767	.758	.760	.....	.764	.763
250	.....	.....	.....	.....	.....	.....	.858	.....	....	.900	.893	.861	.827	.804	.....	.....	.832	.819	.814	.804	.800	.798	.....	.805	.797
OKLAHOMA OIL:																									
Oil used (grams)	.....	862	937	596	703	1325	912	1159	581	574	554	813	982	1692	577	570	590	.....	518	601	705	536	550	547	.....
Oil recovered (per cent)	.....	23.1	29.3	20.2	22.2	17.5	51.7	88.7	33.9	35.6	62.2	37.3	50.8	100.0	38.1	55.0	63.8	.....	88.2	91.8	99.0	99.8	76.9	87.7	.....
Carbon (per cent)	.....	5.8	4.1	24.6	20.0	11.3	4.1	2.2	17.2	13.2	6.7	9.6	3.1	0.0	9.2	7.0	4.6	.....	1.4	1.2	0.0	0.0	0.0	0.0	.....
Sp. gr. of oil recovered	.....	0.997	0.963	0.965	1.040	1.039	0.925	.....	1.008	0.973	0.907	0.931	0.906	0.899	.....	0.908	0.877	.....	0.870	0.867	0.872	0.901	0.882	0.873	0.894
Viscosity	.....	.....	1.64°	.....	.....	.....	1.44°	.....	.....	.....	1.48°	1.08°	1.27°	7.29°	1.05°	.....	1.25°	.....	1.32°	1.54°	2.51°	13.42°	1.50°	1.73°	35.40
First drop		70°	46°	50°	.....	65°	38°	62°	37°	35°	30°	32°	38°	66°	30°	40°	30°	.....	30°	33°	47°	100°	30°	35°	.....
Temperature	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%
100	.....	8.9	6.5	34.4	19.6	13.7	5.3	1.2	21.0	24.0	10.0	16.5	10.3	1.0	17.5	16.3	13.0	.....	9.3	6.2	2.3	0.0	6.2	5.0	0.0
150	.....	9.7	13.0	9.6	7.1	13.4	9.3	2.5	17.4	18.0	9.8	21.7	15.0	1.8	15.5	15.5	9.3	.....	6.7	6.0	2.7	0.8	6.3	5.3	0.3
200	.....	6.8	7.5	8.3	5.8	5.1	9.0	4.0	4.5	5.7	5.0	12.3	10.5	3.7	9.3	7.5	9.0	.....	7.5	5.8	4.2	2.2	4.5	5.5	2.0
250	.....	18.9	11.2	18.7	19.2	19.1	10.7	11.0	11.8	10.8	11.2	16.0	11.2	9.5	11.0	11.0	12.5	.....	10.7	9.7	11.8	7.0	8.8	9.7	5.5
300	.....	.....	10.8	.....	.....	6.9	12.5	15.5	.....	.....	14.0	8.0	14.0	13.0	.....	.....	14.5	.....	14.3	16.0	11.2	13.5	.....	14.3	14.0
150	.....	18.6	19.5	44.0	26.7	27.1	14.8	3.7	38.4	42.0	19.8	38.2	25.3	2.8	33.0	31.8	22.3	.....	16.0	12.2	5.0	0.8	12.5	10.3	0.3
250	.....	25.7	18.7	27.0	25.0	24.2	19.7	15.0	16.3	16.5	16.2	28.3	21.7	13.2	20.3	18.5	21.5	.....	18.2	15.5	16.0	9.2	13.3	15.2	7.5
Temperature	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.
100	.....	.873	.827	.858	.879	.873	.775	.....	.845	.833	.785	.805	.769	.....	.758	.752	.718	.....	.712	.704	.724	.....	.748	.713	.....
150	.....	.868	.856	.853	.857	.876	.827	.798	.871	.867	.839	.850	.817	.791	.842	.833	.803	.....	.790	.777	.766	.....	.813	.783	.....
200	.....	.921	.872	.855	.850	.917	.849	.826	.900	.891	.848	.853	.840	.817	.865	.859	.827	.....	.821	.810	.802	.806	.827	.811	.801
250	.....	.....	.919	Solid	Solid	Solid	.879	.848	.987	.977	.873	.902	.872	.839	.917	.905	.863	.....	.853	.840	.835	.829	.856	.839	.826
300	.....	.....	.928	.....	.....	.994	.893	.855	.....	.....	.885	.956	.894	.850	.....	.....	.886	.....	.868	.858	.848	.844	.....	.855	.841
150	.....	.870	.847	.857	.874	.875	.811	.793	.855	.860	.813	.832	.798	.791	.786	.792	.755	.....	.752	.741	.752	.....	.782	.755	.....
250	.....	.....	.899	.....	.....	.....	.852	.842	.963	.948	.866	.....	.855	.827	.893	.887	.847	.....	.739	.828	.827	.824	.847	.829	.819
CALIFORNIA OIL:																									
Oil used (grams)	.....	646	582	315	539	603	578	.....	569	517	597	987	611	551	563	.....	571(c)	.....	621	571	514	510	.....	613	.....
Oil recovered (per cent)	.....	34.2	43.7	21.1	20.9	20.9	47.4	.....	31.6	33.7	52.5	43.9	46.6	81.8	40.6	.....	53.6	.....	64.8	71.5	91.3	73.6	.....	75.4	.....
Carbon (per cent)	.....	7.1	6.9	38.8	36.2	9.9	8.7	.....	26.7	27.5	19.3	16.2	12.3	13.6	21.1	.....	11.7	.....	14.5	10.1	3.7	11.9	.....	2.3	.....
Sp. gr. of oil recovered	.....	1.025	1.017	1.022	1.025	1.092	0.963	.....	1.030	0.997	0.996	0.984	0.966	0.960	0.991	.....	0.942	.....	0.925	0.928	0.939	0.950	.....	0.924	0.970
Viscosity	.....	.....	3.95°	.....	.....	.....	.....	.....	.....	.....	1.79°	1.15°	1.98°	.....	.....	.....	1.35°	.....	1.35°	1.52°	6.16°	.....	.....	2.71°	35.7°
First drop	.....	.....	.....	70°	67°	70°	.....	.....	40°	33°	67°	35°	33°	.....	30°	.....	33°	.....	35°	38°	50°	.....	.....	40°	.....
Temperature	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%	%
100	.....	8.0	12.5	32.8	31.1	13.4	1.7	.....	11.3	21.3	11.0	16.5	8.3	3.3	12.5	.....	12.8	.....	11.0	8.5	3.2	0.3	.....	4.3	.....
150	.....	6.0	7.5	14.8	8.4	10.8	6.8	.....	22.7	17.5	11.5	19.2	14.5	3.2	17.3	.....	12.3	.....	11.0	10.7	4.5	1.2	.....	5.2	.....
200	.....	8.0	8.3	6.5	2.6	3.6	7.1	.....	4.7	5.3	7.0	7.5	8.0	4.0	8.2	.....	7.0	.....	8.3	7.8	5.3	1.0	.....	5.5	1.8
250	.....	14.7	14.0	22.2	11.1	17.5	10.8	.....	10.6	13.1	10.5	10.8	12.0	11.3	11.0	.....	12.3	.....	10.7	9.5	8.8	5.3	.....	9.3	5.5
300	.....	9.0	10.0	.....	.....	.....	13.3	.....	.....	.....	14.0	10.0	15.5	9.7	.....	.....	.....	.....	10.5	11.0	13.2	12.0	.....	11.5	10.0
150	.....	14.0	20.0	47.6	39.5	24.2	8.5	.....	34.0	38.8	22.5	35.7	22.8	6.5	29.8	.....	25.1	.....	22.0	19.2	7.7	1.5	.....	9.5	.....
250	.....	22.7	22.3	28.7	13.7	21.1	17.9	.....	15.3	18.4	17.5	18.3	20.0	15.3	19.2	.....	19.3	.....	19.0	17.3	14.1	6.3	.....	14.8	7.3
Temperature	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.	S. G.
100	.....	.864	.849	.852	.879	.870	.....	.....	.837	.847	.828	.826	.767	.800	.787	.....	.736	.....	.729	.759	.727	.....	.....	.720	.....
150	.....	.861	.859	.839	.864	.875	.814	.....	.871	.870	.853	.863	.831	.832	.862	.....	.828	.....	.812	.815	.785	.807	.....	.793	.....
200	.....	.879	.874	.862	.886	.917	.841	.....	.908	.893	.870	.887	.861	.847	.894	.....	.863	.....	.848	.858	.824	.....	.....	.825	.806
250	.....	.953	.929	Solid	Solid	Solid	.874	.....	.990	.980	.922	.952	.902	.883	.966	.....	.910	.....	.891	.893	.865	.848	.....	.867	.854
300	.....	.962	.963	.....	.....	.....	.906	.....	.....	.....	.946	.988	.937	.905	.....	.....	.....	.....	.920	.919	.891	.883	.....	.894	.886
150	.....	.863	.852	.848	.875	.873	.814	.....	.862	.867	.842	.847	.808	.817	.829	.....	.783	.....	.776	.794	.765	.....	.....	.764	.....
250	.....	.926	.908	.....	.....	.....	.859	.....	.964	.953	.903	.926	.884	.873	.936	.....	.893	.....	.872	.876	.848	.845	.....	.851	.841

(*a*) 7 to 9 atmospheres.
(*b*) Viscous at 21°.
(*c*) 28 atmospheres.

necessary for the experiments at lower temperatures to have the furnace greatly superheated at the start in order to get the system up to the desired pressure before too large a proportion of the original charge of oil had been run through.

If a run at 500° and 24 atmospheres is taken as an example the following conditions obtained: the furnace was started at 650° and atmospheric pressure and oil run in till the pressure reached the desired height while, at the same time, the temperature was allowed to fall. In such an experiment it was necessary to run through from 150 to 200 grams of the total 600-gram charge of oil before desired conditions were established. It must be understood therefore that the percentage recoveries are invariably low for pressure reactions and that the error is considerable in the case of the lower temperatures.

The first point to be noted in the data is that the differences among original oils seem to have no marked influence over the magnitude of the percentage recoveries. There is an exception in the case of the Mexican crude where a large amount of the liquid product of reaction was held by the carbon deposit. Among the other oils the variations, though of some magnitude, are distributed with perfect impartiality. Thus the Pennsylvania recovered oils are liable to show either a maximum or a minimum percentage recovery.

It is indicated with entire definiteness in the tabulation of averages that percentage recovery decreases with increase of temperature. It appears also that vacuum recoveries are greater than those at atmospheric pressure. Indications for pressures above one atmosphere are not entirely correct on the basis of figures given here. It was noted, for instance, that in the case of a pressure reaction at 500° gas formation was practically nil after the initial period during which conditions were being established. In this case there should have been a recovery of practically 100 per cent as carbon is not formed in the absence of gas and the only possible products must have been liquid. The low yields here indicated were due to the preliminary cracking period during which pressure was low and temperature high. The indications for the effect of pressure at higher temperatures are undoubtedly correct, being in accord with theory and not explainable on the basis of any known experimental error.

TABLE IIB—RESULTS OF EXPERIMENTAL RUNS WITH RUSSIAN AND MEXICAN OILS

	RUSSIAN OIL:			MEXICAN OIL:							
Temperature:	675°	500°	Orig.	750	600	600°	600°	500	500	400	Orig.
Pressure—Atmospheres	12 atmos.	12 atmos.	oil	Vac.	12	1 at.	Vac.	12	1	10–11	oil
Oil used (grams)	443	387	...	812	479	628	500	573	591	527	...
Oil recovered (per cent)	28.3	77.3	...	37.3	47.2	40.2	66.0	54.5	72.8	73.5	...
Carbon (per cent)	21.7	0.0	...	10.5	13.1	16.4	9.0	16.2	9.8	13.8	...
Sp. Gr. of oil recovered	.965	.880	.897	.992	.941	.992	.942	.918	.930	.934	.986
Viscosity	...	1.44°	11.92°	2.70°	...	1.96°	6.73°	1.15°	1.88°	4.43°	Over 300°
First drop	40°	30°	...	...	30°	40	70°	30°	55°	35°	...
Temperature	% S. G.	% S. G.	% S. G.	% S. G.	% S. G.	% S. G.	% S. G.	% S. G.	% S. G.	% S. G.	% S. G.
100	30.5 .863	7.3 .718	0.0	8.3 .822	13.3 .765	13.0 .802	2.3 .751	13.5 .721	7.0 .739	4.8 .729	0.0 ....
150	16.1 .867	7.8 .796	0.0	5.0 .836	12.5 .829	8.3 .837	3.5 .782	13.3 .807	8.7 .792	6.0 .789	1.1 .759
200	4.7 .891	5.7 .829	1.5	7.7 .868	8.0 .846	9.2 .860	8.3 .811	11.0 .837	8.5 .825	7.0 .816	2.5 .798
250	13.9 Solid	13.8 .856	10.7 .836	10.5 .915	11.0 .899	13.0 .923	10.5 .846	13.0 .884	12.5 .875	11.0 .856	8.3 .843
300	5.9 .964	16.5 .881	16.3 .859	14.5 .941	11.7 .943	12.5 .968	15.5 .880	12.2 .945	11.7 .902	13.7 .898	25.2 .871
150	46.6 ..865	15.1 .761	....	13.3 .827	25.8 .877	21.3 .816	5.8 .771	26.8 .766	15.7 .770	10.8 .762	1.1 .759
250	24.5	19.5 .846	12.2 .836	18.2 .897	19.0 .797	22.2 .909	18.8 .829	24.0 .862	21.0 .854	18.0 .843	10.8 .833

TABLE III—CARBON FORMATION, PHYSICAL PROPERTIES AND PERCENTAGES OF OILS RECOVERED

		PERCENTAGE OF CARBON FORMATION					PROPERTIES OF OILS RECOVERED										PERCENTAGES OF OIL RECOVERED				
							Viscosities (Engler degrees)					Specific gravities									
OIL:		Pa.	Okla.	Cal.	Mex.	Av.	Pa.	Okla.	Cal.	Mex.	Russ.	Pa.	Okla.	Cal.	Mex.	Av.	Pa.	Okla.	Cal.	Mex.	Av.
Temp. ° C.	Pres. atm.																				
850	1	7.4	..	..	..	7.4	..	...	...	..	...	1.026	...	...	...	1.026	9.7	...	..	..	9.7
	Vac.	..	5.8	7.1	..	6.4	..	...	...	..	...	...	0.997	1.025	...	1.011	...	23.1	34.2	..	28.6
800	Vac.	..	4.1	6.9	..	5.5	..	1.64	3.95	..	...	...	0.963	1.017	...	0.990	...	29.3	43.7	..	36.5
750	12	24.3	24.6	38.8	..	29.2	..	...	...	..	...	0.910	0.965	1.022	...	0.965	15.9	20.2	21.1	..	19.1
	6	15.3	20.0	36.2	..	23.8	..	...	...	..	...	1.020	1.040	1.025	...	1.028	16.9	22.2	20.9	..	20.0
	1	3.7	11.3	9.9	..	8.3	..	...	...	..	...	1.012	1.039	1.092	...	1.048	16.5	17.5	20.9	..	18.3
	Vac.	16.1	4.1	8.7	10.5	13.1	1.04	1.44	...	2.70	...	0.905	0.925	0.963	0.992	0.946	33.9	51.7	47.4	37.3	42.6
650	Vac.	..	2.2	..	..	2.2	..	...	...	..	...	...	...	...	...	...	...	88.7	..	..	88.7
600	24	7.5	17.2	26.7	..	17.1	...	...	...	..	...	0.962	1.008	1.030	...	1.000	37.3	33.9	31.6	..	34.2
	18	5.3	13.2	27.5	..	15.3	1.11	1.48	1.79	..	...	0.926	0.973	0.997	...	0.965	40.5	35.6	33.7	..	36.6
	12	3.6	6.7	19.3	13.1	10.7	1.00	1.08	1.15	..	...	0.918	0.907	0.996	0.941	0.940	43.6	62.2	52.5	47.2	51.4
	6	2.4	9.6	16.2	..	9.4	..	1.27	1.98	1.96	...	0.885	0.931	0.984	...	0.933	31.4	37.3	43.9	..	37.5
	1	0.6	3.1	12.3	16.4	8.1	1.04	7.29	...	6.73	...	0.840	0.906	0.966	0.992	0.926	59.9	50.8	46.6	40.2	49.4
	Vac.	0.0	..	13.6	9.0	7.5	..	...	...	..	...	0.811	0.899	0.960	0.942	0.903	97.4	100.0	81.8	66.0	85.0
550	24	..	9.2	21.1	..	15.1	..	1.05	...	..	...	...	...	0.991	...	0.991	...	38.1	40.6	..	39.3
	18	..	7.0	..	..	7.0	..	...	...	..	...	...	0.908	...	...	0.908	...	55.0	..	..	55.0
500	24	1.2	4.6	11.7	..	5.8	0.97	1.25	1.35	..	...	0.828	0.877	0.942	...	0.882	72.5	63.8	53.6	..	63.3
	18	0.0	..	..	..	0.0	0.96	...	...	..	...	0.819	...	...	...	0.819	79.4	...	..	..	79.4
	12	0.0	1.4	14.5	16.2	8.0	0.98	1.32	1.35	1.15	1.44	0.808	0.870	0.925	0.918	0.880	84.2	88.2	64.8	54.5	72.9
	6	0.0	1.2	10.1	..	3.8	1.00	1.54	1.52	..	...	0.810	0.867	0.928	...	0.868	57.8	91.8	71.5	..	73.7
	1	0.0	0.0	3.7	9.8	3.6	1.00	2.51	6.16	1.88	...	0.799	0.872	0.939	0.930	0.885	94.5	99.0	91.3	72.8	89.4
	Vac.	6.0	0.0	11.9	..	5.9	1.04	13.42	...	..	...	0.802	0.901	0.950	...	0.884	100.0	99.8	73.6	..	91.1
450	18	..	0.0	..	..	0.0	..	1.50	2.71	..	...	...	0.882	...	...	0.882	...	76.9	..	73.5	75.2
	12	..	..	..	..	..	1.02	1.73	...	4.43	...	...	...	...	...	...	...	...	..	..	..
ORIGINAL		..	..	..	..	..	1.05	35.40	35.7	Over 350	11.92	...	...	...	...	...	...	...	..	..	..

	Average Percentages Carbon Formation						Viscosities for Oklahoma Series						Average Specific Gravities of Recovered Oils						Average Percentages of Oil Recovered					
Pres. atm. / Temp.	Vac.	1	6	12	18	24	Vac.	1	6	12	18	24	Vac.	1	6	12	18	24	Vac.	1	6	12	18	24
450°	..	...	..	2.5	0.0	..	...	..	..	1.73	1.50	..	...	...	...	0.868	0.882	...	..	...	..	85.4	..	..
500°	5.9	3.6	3.8	8.0	0.0	4.8	13.42	2.51	1.54	1.32	..	1.25	0.884	0.885	0.868	0.880	0.819	0.882	91.1	89.4	73.7	72.9	79.4	63.3
550°	..	...	..	..	7.0	15.1	...	..	..	...	..	1.05	...	...	...	...	0.908	0.991	..	..	...	..	..	39.5
600°	7.5	8.1	9.4	10.7	15.3	17.1	7.29	1.27	1.08	14.8	..	..	0.903	0.926	0.933	0.940	0.965	1.000	85.0	49.4	37.5	51.4	36.6	34.2
650°	2.2	...	..	..	..	..	...	..	..	..	..	..	...	...	...	...	...	...	88.7	..	..	..	..	..
750°	13.1	8.3	23.8	29.2	..	..	1.44	..	..	...	..	..	0.946	1.048	1.028	0.965	...	...	42.6	18.3	20.0	19.1	..	..
800°	5.5	...	..	..	..	..	1.64	..	..	...	..	..	0.990	...	...	...	...	...	36.5	..	..	..	..	..
850°	6.4	7.4	..	..	..	..	...	..	..	...	..	..	1.011	1.026	...	...	...	...	28.6	9.7	..	..	..	..

FORMATION OF GASOLINE AND AROMATIC HYDROCARBONS

GASOLINE FORMATION—From both a scientific and a commercial point of view the most important factors to be considered in the cracking reaction are the physical and chemical relations between original and recovered oils. The chief commercial interest in the cracking reaction has of late centered on the problem of gasoline production. It is rather a difficult matter to establish a satisfactory definition of gasoline and for present purposes it is unnecessary, as the differences obtained among reaction products have been so great that no fine distinctions are necessary. In the present connection the magnitude of the distillation cut up to 150° C. has been considered a measure of the "naphtha" or "gasoline." On this basis the amount of gasoline production has been figured for the different runs and results listed in Table IV. The calculations from experimental figures were conducted as follows. The amount of oil in the original sample distilling below the 150° mark was deducted from the distillation figure for the recovered oil. This last corrected percentage was reduced to terms of the original oil, giving a percentage which represents its degree of transformation into hydrocarbons boiling below 150° C.

TABLE IV—FORMATION OF GASOLINE AND AROMATICS

		PERCENTAGE GASOLINE FORMATION					FORMATION OF LOW-BOILING AROMATIC HYDROCARBONS				
Temp. °C.	Pressure	Pa.	Okla.	Cal.	Mex.	Av.	Pa.	Okla.	Cal.	Mex.	Av.
850	1	2.3	..	..	..	2.3	2.4	..	..	...	2.4
	Vac.	..	4.2	4.8	..	4.5	..	4.1	4.4	...	4.3
800	Vac.	..	5.6	8.8	..	7.2	..	4.6	7.1	...	5.9
750	12	5.5	8.8	10.0	..	8.1	5.6	7.7	8.0	...	7.1
	6	6.2	5.8	8.2	..	6.7	6.8	5.9	7.9	..	6.9
	1	4.3	4.7	5.1	..	4.7	5.1	4.7	5.0	..	4.9
	Vac.	4.5	7.5	4.0	4.6	5.1	4.1	4.2	2.2	2.9	3.4
650	Vac.	..	3.0	..	..	3.0	..	1.4	..	...	1.4
600	24	13.3	12.9	10.7	..	12.3	12.4	11.2	9.7	...	11.1
	18	15.8	16.6	13.0	..	15.1	13.4	14.0	12.3	...	13.1
	12	16.3	12.1	11.8	11.6	12.9	13.4	6.9	8.8	4.1	8.3
	6	10.0	14.1	15.7	..	13.3	7.1	9.8	12.2	...	9.7
	1	13.4	12.7	10.6	8.1	11.2	7.3	5.8	5.1	4.3	5.6
	Vac	0.0	2.5	5.3	3.1	3.6	1.0	1.1	3.0	0.4	1.4
550	24	..	12.4	12.1	..	12.3	..	4.7	7.7	...	6.2
	18	..	17.3	..	..	17.3	..	7.3	..	...	7.3
500	24	18.1	14.0	13.5	..	15.2	7.0	2.1	4.2	...	4.4
	18	15.3	13.8	..	..	14.6	5.6	..	..	...	5.6
	12	16.0	10.9	14.3	14.6	13.7	5.6	1.8	3.7	0.9	3.0
	6	5.9	4.7	13.7	10.6	8.7	2.6	..	5.4	...	4.0
	1	2.7	0.0	7.0	..	4.9	1.8	0.6	1.2	1.1	1.2
	Vac.	0.0	..	1.1	..	1.1	0.7	..	..	...	0.7
450	18	..	9.4	..	..	9.4	..	3.3	..	...	3.3
	12	6.5	8.8	7.2	..	7.5	2.9	1.3	1.1	...	1.8
400	10	..	..	..	7.1	7.1	..	..	..	0.2	0.2

	AVERAGE PERCENTAGES GASOLINE FORMATION						AVERAGE LOW-BOILING AROMATIC HYDROCARBONS					
Pres. atm.	Vac.	1	6	12	18	24	Vac.	1	6	12	18	24
Temp.												
450°	...	..	..	7.5	9.4	..	...	...	...	1.8	3.3	..
500°	1.1	4.9	8.7	13.7	14.6	15.2	0.7	1.2	4.0	3.0	5.6	4.4
550°	...	..	..	..	17.3	12.3	...	...	...	...	7.3	6.2
600°	3.6	11.2	13.3	12.9	15.1	12.3	1.4	5.6	9.7	8.3	13.1	11.1
650°	3.0	..	..	..	..	..	1.4	...	...	...	..	..
750°	5.1	4.7	6.7	8.1	..	..	3.4	4.9	6.9	7.1	..	..
800°	7.2	..	..	..	..	..	5.9	...	...	...	..	..
850°	4.5	2.3	..	..	..	..	4.3	2.4	...	...	..	..

Thus for example, in the case of an experiment at 500° and 12 atmospheres with Pennsylvania oil the distillation cut given in Table I is 23.8 per cent up to 150°. The original oil distilled 4.8 per cent up to the same temperature. The increase is therefore 19.0 per cent. But of the original oil only 84.2 per cent was recovered and the gasoline formation was therefore 84.2 per cent of 19.0, or 16.0 per cent.

An examination of the results shows first of all a remarkable agreement among the effects of the same conditions of temperature and pressure on the different original oils. The comparison of the average percentages indicates that the formation of a large percentage of hydrocarbons boiling below 150° C. is favored by pressure and that the optimum temperatures are in the neighborhoods of 500° and 600°. It is not, however, sufficient to limit our considerations to the data appearing in Table IV. There are vast physical and chemical differences between cuts of approximately the same magnitude formed respectively at 500 and 600° as will be noted when a comparison is made of the figures for specific gravities.

DIFFERENCES IN SPECIFIC GRAVITY OF CUTS TO 150° FROM RUNS AT LIKE PRESSURES AND TEMPERATURES 600 AND 500°

Pressure	Pennsylvania	Oklahoma	California	Mexican
Vac	0.009	...	...	...
1	0.032	0.046	0.043	0.046
6	0.042	0.081	0.051	...
12	0.070	0.061	0.056	0.032
18	0.086	...	...	...
24	0.088	0.111	0.053	...

The above is a list of these density differences between distillates obtained from runs at the same pressures and at temperatures respectively of 600 and 500°. The gravities of the 600° products are invariably the higher. It will be noted also that the 600° products represent a large percentage of a small recovery while the reverse is the case for 500° products. *In addition it must be remembered that the inherent error of operation made low the amounts of gasoline recovered for pressure runs.*

The obvious conclusion is that the favorable conditions for producing gasoline, as determined by the comparison of results of the present series of experiments, involve a moderate temperature and a high pressure.

FORMATION OF AROMATIC HYDROCARBONS—It has long been known that aromatic hydrocarbons may result from the cracking of petroleum. A recent study[1] of the properties of gas oils and derived tars has demonstrated this fact again with entire definiteness, and the present experiments have served to determine only what are the most favorable conditions for this type of transformation.

As was indicated in the preceding section the characteristic property of aromatic hydrocarbons is their great density. Benzene, which boils at 80.5°, has a specific gravity of 0.883 and toluene, with a boiling point of 111°, has a gravity of 0.872. Paraffin or olefin hydrocarbons of equivalent boiling points possess much lower gravities. This is indicated by the following figures for straight chain hydrocarbons boiling at temperatures slightly higher than those required for benzene and toluene.

HYDROCARBON	Boiling point	Specific gravity
Heptane	98.0	0.712
Octane	125.5	0.708
Heptylene	98.0	0.703
Octylene	123.0	0.721

The quantitative separation of the different groups of hydrocarbons is not a process which can be conducted with ease and accuracy. There are chemical methods which have been recommended and these have been tried with results which have appeared in another communication. For present purposes the indications furnished by specific gravity measurements have been found sufficiently clear-cut to differentiate results with a satisfactory degree of accuracy.

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[1] Rittman and Egloff, THIS JOURNAL, 7 (1915), 481.

A scheme has been devised for the approximate calculation of the percentage formation of hydrocarbons of the aromatic series, boiling below 150°. The assumption has been made that the average gravity of the mixed aromatics benzene, toluene and the three xylenes is 0.875. This figure is undoubtedly very near the true value and is justified by the results obtained through its use. By studying some results of a previous paper[1] it was learned that with the methods of distillation here employed the average specific gravities of 150° distillation cuts of uncracked oils are as follows:

Pennsylvania	Oklahoma	California	Mexican
0.721	0.734	0.742	0.759

Using these figures in connection with a value of 0.875 for the mixed aromatics a series of calculations was conducted and values obtained for the percentage formation of low-boiling aromatic hydrocarbons.

The following example is typical of one of the less favorable runs. For a California (Kern River) oil, cracked at 600° and 12 atmospheres the percentage recovery was 52.5 per cent and this oil gave below 150° a 22.5 per cent distillation cut of gravity 0.842. The last figure, according to the scheme indicated above represents a mixture containing 75 per cent of the aromatics. The total aromatic formation was therefore 75 × 22.5 × 52.5 per cent or 8.3 per cent on the basis of the original oil.

Figures obtained in this way are shown in Table IV and again there appears an excellent agreement among the different original oils. The variations are moderate and are not characteristic. In consideration of the several approximations and the inherent experimental errors the agreement may be considered as striking. Again there is proof that the conditions of treatment are of such importance that any original tendencies on the parts of the oils used are rendered of negligible importance.

It will be noted from the average figures for low-boiling aromatics in Table IV, that most favorable conditions for the formation of benzene, toluene and xylenes seem to be 600° and above and at pressures greater than 4 atmospheres.

SUMMARY

Results of experiments here described have indicated the following conclusions:

I—Equilibrium products of the cracking reaction seem to be independent of the influence of chemical and physical properties of the original oils or at most are affected only in minor degree by such tendencies. The matter of carbon formation is the one clear-cut exception. Carbon is a residual product and its formation is proportional to the amount originally contained in the oil. Viscosities and specific gravities seem to show in slight degree the influence of properties of original oils but the differences are so slight that they may well be explained on the basis of failure to reach complete equilibrium.

II—The formation of aromatic hydrocarbons occurs best at moderate to high temperatures and under high pressure.

III—The formation of gasoline (low-boiling, low gravity hydrocarbons) is favored by moderate to low temperatures and by high pressure.

IV—The results obtained by the series of laboratory experiments indicate striking commercial possibilities.

The experimental work connected with this paper was carried out in the laboratories of the Department of Industrial Chemistry of Columbia University, New York.

Chemical Section of Petroleum Division
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[1] Rittman and Egloff, This Journal, 7 (1915), 578.

A QUANTITATIVE METHOD FOR THE DETERMINATION OF THE ADULTERATION IN CHINESE WOOD OIL[1]

By J. C. Brier

Received October 5, 1915

Examination of a large number of shipments of Chinese wood oil extending over a period of several years, each shipment being checked against its behavior in the varnish kettle, has convinced the writer that the advocated methods of testing Chinese wood oil, while affording valuable indications in certain cases, can be used with any degree of accuracy only on grossly adulterated oils.

Hoffman[2] reports that the adulteration of wood oil with foreign oils was very common in China in former years and that the oils, pressed in China, hardly ever reached the market in a pure state. According to him, oil from the kernel of the vegetable tallow seed, especially, was employed for the fraudulent blending of wood oil. Boughton[3] states that Chinese wood oil is frequently adulterated with soya bean oil. From numerous inquiries the writer believes that the larger amount of intentional adulteration is confined to the use of these two oils, but that frequent accidental adulteration happens with ground nut oil, sesame oil, and occasionally, with tea seed and perilla oils. Importers, who are familiar with the production of Chinese wood oil and the various stages and methods of its transportation until barreled ready for export, have informed the writer that adulteration, although not nearly as prevalent as at the time at which Hoffman wrote, is still exceedingly common.

It is well known that the oil is obtained from a large number of small producers by the middlemen or merchants, who transport it in baskets made of bamboo lined with oiled paper (the average capacity of these baskets being approximately 17 gallons). The native boats, which transport the oil to the export markets, frequently carry mixed cargoes of the oils occurring in that section of China where Chinese wood oil is produced. The frail baskets being piled on top of each other often become leaky, thus allowing the oils from the upper baskets to contaminate the contents in the others. Then, too, the oils that drip on the floor are collected and distributed among the various baskets throughout the cargo. Such cases as those just cited make it difficult for the Chinese wood oil importers to be sure as to the purity of the oil they are buying,

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[1] This method was developed in collaboration with Dr. C. D. Holley in the Research Laboratory of the Acme White Lead and Color Works.

[2] *Seifenseider-Zeit.*, **35** (1908), 169.

[3] *Drugs, Oils and Paints*, **29**, 252–256.

since the methods of detecting adulteration are so unsatisfactory.

The demand for Chinese wood oil for the manufacture of varnishes has increased very rapidly during recent years owing to the fact that with certain gums, as well as rosin, this oil, when properly manipulated, gives a very high-grade product for certain uses. Since slight variations in the purity of the oil affect the finished product it is necessary that the varnish maker have a uniform grade of oil, otherwise every shipment will mean an experimental batch.

METHODS OF EXAMINATION

The oldest method for testing Chinese wood oil is the practical kettle test. This, however, is hardly feasible when one considers that large plants use from 100 to 300 or more barrels per month.

The methods of Bacon, Worstall, Potsdammer, Browne and others,[1] are based on the well-known characteristic that Chinese wood oil has of polymerizing when heated to 280° C.

Other methods of testing Chinese wood oil are the McIlhiney,[2] the iodine jelly[3] and the Ware Schumann.[4]

The urgent need for standardizing the methods of testing Chinese wood oil, led Sub-committee D1 of the American Society for Testing Materials to take up the problem. Their work has covered a period of several years and the various methods have been tried out. However, specifications for raw Chinese wood oil were not proposed until the report of the committee was given at the meeting of the society held June 22, 1915. These specifications were based on the analytical constants which had been determined in previous years along with the modified Browne's method and the modified iodine jelly test, which were reported on at this time. The work for this year was done on the following five samples:

OILS	PER CENT PRESENT IN SAMPLE NO.				
	1	2	3	4	5
Pure China wood oil....	100	95	90	95	90
Soya bean oil..........	...	5	10	..	..
Linseed oil............	...	..	..	5	10

From the results obtained by the various operators on these samples the time limit at or under which pure oil should polymerize was specified as 12 minutes when using the Browne test, while a time limit of four minutes was specified for the setting period in the iodine jelly test.

The wide variations in the results obtained by the different members of the committee indicate the necessity of working both the tests against standards, and not to a time limit as proposed in the specifications. A brief résumé of the results obtained show the large personal equation involved:

(*a*)—Accepting the 12-minute time limit in the case of the Browne test, at or under which the oil is to be considered acceptable, and above which it is to be rejected, the pure sample should be rejected by one operator, the sample adulterated with 5 per cent soya bean oil should be accepted by 5 operators and the sample adulterated with 10 per cent soya bean oil should be accepted by one operator. The sample adulterated with 5 per cent linseed oil is acceptable by the reports of 3 operators.

(*b*)—Accepting the time limit of 4 minutes, as is specified in the case of the iodine jelly test, 6 out of the 9 operators reporting gave figures below the time limit on sample No. 2. With sample No. 4, the same 6 operators gave in their reports figures which were below the accepted time limit for pure Chinese wood oil.

The contention that all tests on shipments of wood oil should be made against standards is further supported by the supplementary report of Boughton (meeting held June 22, 1915), which shows that variations in temperature of the bath, the weight of oil used, the amount of chloroform taken, and the strength of the iodine solution, cause large differences in the results obtained.

The index of refraction has been reported as giving valuable information as to the purity of Chinese wood oil by Wise,[1] Chapman,[2] Hoepfner and Burmeister,[3] and Boughton.[4] Dr. Holton[5] noticed, while working with the Abbey instrument on the index of refraction, that it was necessary to set the compensator at an abnormal point when taking readings on Chinese wood oil.

Shook,[6] while working on the dispersion of various oils with the Pulfrich refractometer, noticed that Chinese wood oil showed anomalous dispersion; *i. e.*, while the spectrum of all the other oils appeared with the red on the upper side and the green and blue on the lower side (Fig. 1), Chinese wood oil completely reversed this condition, showing the blue and green on top with the red on the lower side of the spectrum (Fig. 2). This phenomena probably accounts for the observations made by Holton.

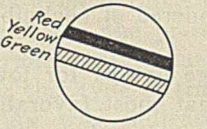

FIG. 1
SPECTRUM SHOWING NORMAL DISPERSION

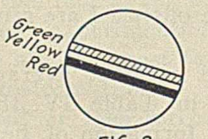

FIG. 2
SPECTRUM SHOWING ANOMALOUS DISPERSION (CHINESE WOOD OIL)

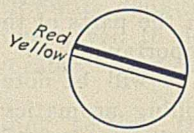

FIG. 3
APPEARANCE OF SPECTRUM AT TURNING POINT.

APPARATUS

The principle used is that of the Pulfrich refractometer. The essential features of the apparatus are as follows: Referring to Fig. 4, *X* is the source of light and in this work was a tungsten light. *L* is a condensing lens. The prism *P*, the most essential part of the apparatus, is a 90° prism having an index of refraction of 1.62. The cell *C*, in which the oils were placed, is so mounted on the prism that the upper surface of the prism is above the joining of the cell and prism. The telescope *T*, which picks up

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[1] Boughton, "Testing of Chinese Wood Oil," *Proc. Am. Soc. Test. Materials*, **13** (1913), 923.

[2] THIS JOURNAL, **4** (1913), 496–497.

[3] *Proc. Am. Soc. Testing Materials*, **1914**, 242.

[4] THIS JOURNAL, **6** (1914), 806.

[1] *J. Soc. Chem. Ind.*, **4** (1912), 497.

[2] *Analyst*, **37** (1912), 543.

[3] *Chem. Zeit.* (**1913**), No. 2, 18.

[4] *Drugs, Oils and Paints*, **29**, 252–256.

[5] Private communication to Dr. C. D. Holley, Chief of this Laboratory.

[6] *Met. and Chem. Eng.*, Oct., 1915, and also private communication.

the spectrum, is bent at a right angle of 90° between the objective *O* and the eyepiece *e* for convenience in observing the spectrum.

EFFECT OF DIFFERENT OILS ON THE SPECTRUM OF CHINESE WOOD OIL

Additions of linseed oil to Chinese wood oil up to about 8 per cent show very little change in the spectrum. Beginning with 9 per cent adulteration, the yellow becomes more pronounced while the red begins to disappear. However, when the adulteration reaches 10 per cent, the green takes the form of a more distinct band with the red slowly decreasing in intensity. With 11 per cent adulteration, the green band becomes more marked and the line of demarcation from the yellow is much sharper than with 10 per cent adulteration. The red shows now only to a very limited degree. The addition of 12 per cent linseed oil to the Chinese wood oil shows a spectrum consisting of only two colors, green and yellow, which appear as very distinct bands of about the same width, the green still remaining upon the upper side. The appearance of the bands with 13 per cent adulteration is very little different from that with 12 per cent, the green, however, being a trifle less distinct. With 13.5 per cent the conditions remain practically the same, while with 14 per cent and 14.5 per cent the green shows very indistinctly, and when 15 per cent linseed oil has been added, only a distinct yellow band can be seen. The next change noted, occurring with 15.5 per cent adulteration, is the red beginning to appear in a distinct narrow band on the upper side of the spectrum. This is the most noticeable change and the one most readily observed, and in the work which follows this point is called the "turning point" (Fig. 3). Further additions of linseed oil increase first the distinctness of the red, and when about 16 to 16.5 per cent has been added, the green begins to appear. From this point on the colors gradually develop until finally the spectrum has been completely and distinctly reversed.

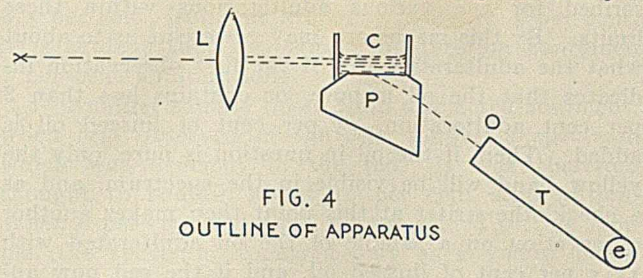

FIG. 4
OUTLINE OF APPARATUS

All other oils used as adulterants of Chinese wood oil affected its spectrum in the same manner, the changing point varying over a range of 2.5 per cent depending on the particular oil used. To illustrate: light tallow seed oil and perilla oil begin to affect the spectrum of Chinese wood oil at about 9 per cent, while the "turning point" is at 17 per cent adulteration. On the other hand, with ground nut, cotton seed, sesame and rape oils this effect is first noticed at a little below 7 per cent and the "turning point" takes place with 14.5 per cent adulteration. Candle nut, linseed, poppy seed, dark tallow seed, and soya bean oils affect the spectrum of Chinese wood oil at a point about midway between the cases just cited, the "turning point" taking place with 15.5 per cent adulteration in the case of poppy seed, linseed, and candle nut oils, with 16 per cent of dark tallow seed, and with 15 per cent adulteration of soya bean oil.

TABLE I—APPEARANCE OF THE SPECTRA OF ADULTERATED CHINESE WOOD OIL AT 25° C.

Adulterant Present Per cent by weight	Tallow Seed Oil		Perilla Oil	Candle Nut Oil	Linseed Oil		Poppy Seed Oil	Soya Bean Oil	Sesame Oil	Ground Nut Oil	Cotton-Seed Oil	Rape Oil
	(Light)	(Dark)			(Raw)	(Refined)						
13.5	....	....	....	....	....	....	....	Green Yellow	Green Yellow	Green Yellow	Green Yellow	Green Yellow
14.0	....	....	....	Green Yellow	Green Yellow	Green Yellow	Green Yellow	Green Yellow	...... Yellow	...... Yellow	...... Yellow	...... Yellow
14.5	....	....	....	Green Yellow	Green Yellow	Green Yellow	Green Yellow	...... Yellow	Red Yellow	Red Yellow	Red Yellow	Red Yellow
15.0	....	Green Yellow	....	...... Yellow	...... Yellow	...... Yellow	...... Yellow	Red Yellow	....	....	....	....
15.5	Green Yellow	Green Yellow	Green Yellow	Red Yellow	Red Yellow	Red Yellow	Red Yellow	....	....	....	....	....
16.0	Green Yellow	...... Yellow	Green Yellow	....	....	....	....	....	....	....	....	....
16.5	...... Yellow	Red Yellow	...... Yellow	......	......	......	....	....	....	....	....	....
17.0	Red Yellow	....	Red Yellow	....	....	....	....	....	....	....	....	....

Table I shows the effect of the individual adulterants on the spectrum of Chinese wood oil at varying percentages just below and at the "turning point."

Reading from top to bottom, it will be noticed that at 0.5 per cent below the "turning point" the spectrum showed, in each case, only a yellow band; while, with an adulteration of one per cent less than this, the spectrum showed two bands, namely, green and yellow. In all cases these bands were very definitely divided and not blended one with the other.

TABLE II—APPEARANCE OF SPECTRA OF ADULTERATED CHINESE WOOD OIL

Adulterant Per cent 20° C.	by weight 30° C.	Tallow Seed Oil (Dark)	Linseed Oil (Raw)	Soya Bean Oil	Cotton-Seed Oil	Ground Nut Oil	Sesame Oil
13.75	12.25	....	....	....	Green Yellow	Green Yellow	Green Yellow
14.25	12.75	....	....	Green Yellow	Green Yellow	Green Yellow	Green Yellow
14.75	13.25	....	Green Yellow	Green Yellow	...... Yellow	...... Yellow	...... Yellow
15.25	13.75	....	Green Yellow	...... Yellow	Red Yellow	Red Yellow	Red Yellow
15.75	14.25	Green Yellow	...... Yellow	Red Yellow	....	....	....
16.25	14.75	Green Yellow	Red Yellow	....	....	....	....
16.75	15.25	...... Yellow	....	....	....	....	....
17.25	15.75	Red Yellow	....	....	....	....	....

Temperature influences the effect of the adulteration on the spectrum of Chinese wood oil to a very marked degree. However, this factor seems to be the same for each of the oils used. For example:

the "turning point" with soya bean oil at 25° C. takes place with 15 per cent adulteration, at 20° C. with 15.75 per cent adulteration, and at 30° with 14.25 per cent adulteration. With linseed oil the "turning point" at 25° C. takes place with 15.5 per cent, at 20° C. with 16.25 per cent, and at 30° C. with 14.75 per cent adulteration. A comparison of Tables I and II clearly shows the influence of temperature on the spectrum at the different adulterations.

It is obvious from these results that either a correction for temperature should be made when making a determination, or, better, all readings should be made at one temperature. In the remainder of the work this latter plan has been adopted.

Up to this point in the investigation the work has been carried on using only one sample of Chinese wood oil. This oil is No. 1 in Table III, which shows the effect on the spectra of different Chinese wood oils when the same soya bean oil is used as an adulterant. It will be noticed that the "turning point" in the cases of oils 1, 2 and 3 takes place at the same point, that is, with an adulteration of 15.0 per cent, in the cases of oils 4, 6 and 7 with 14.5 per cent, and with oil 5, the poorest one given in the table, with 13.5 per cent.

TABLE III

Soya Bean Oil Per cent by weight	Appearance of the Spectra of different Chinese Wood Oils Adulterated with Soya Bean Oils 1	2	3	4	5	6	7
13.0	....	....	....	Green Yellow	...... Yellow	Green Yellow	Green Yellow
13.5	Green Yellow	Green Yellow	Green Yellow	Green Yellow	Red Yellow	Green Yellow	Green Yellow
14.0	Green Yellow	Green Yellow	Green Yellow	...... Yellow	....	...... Yellow	...... Yellow
14.5	...... Yellow	...... Yellow	...... Yellow	Red Yellow	....	Red Yellow	Red Yellow
15.0	Red Yellow	Red Yellow	Red Yellow	....	....	....	

Samples 1, 2, 3 5, 6 and 7 were taken from shipments covering a period of two years and from different importers. Sample No. 4 was furnished to Dr. C. D. Holley by Mr. E. W. Boughton and is the *Aleurites fordii* used by him in his investigations on the various oils from the Orient. In actual use, oil No. 5 proved to be inferior to oils 1, 2, 3, 6 and 7.

Of the oils mentioned above, the ones generally supposed to be used as adulterants are soya bean, tallow seed, sesame, and occasionally ground nut and candle nut oils. It has been pointed out that the effect on the spectrum of Chinese wood oil when adulterated with these oils is to cause the "turning point" to take place at adulterations varying between the limits of 14.5 to 17.0 per cent. As a working basis for an analytical method, the writer assumes an arbitrary adulteration of 15.5 per cent, at which the "turning point" will take place, *i. e.*, when the red first appears on the upper side of the spectrum.

ANALYTICAL METHOD

Since 15.5 per cent of linseed oil in Chinese wood oil causes the "turning point" to take place, it is used as the oil to be added when necessary to bring about the "turning point."

The author's procedure is as follows: First a sample of the oil in question is placed in the cell and its spectrum examined. If the oil is pure, or contains less than 8 per cent of adulteration, the spectrum will be inverted and appear practically the same in all cases. If the percentage of adulteration is over 8 per cent and under the "turning point" (15.5 per cent), the spectrum will appear as previously described for the various adulterations within these limits. By this means one may get a clue as to about what the adulteration is. If the first observation indicates that the oil is pure, or contains less than 8 per cent adulteration, 15 per cent of linseed oil is added. Then, if the oil in question is pure, only the yellow band will be visible in the spectrum, and as a check, the writer at this point then makes another observation on a sample of the oil adulterated with 15.5 per cent of linseed oil, and if the red now appears on the upper side of the spectrum, the sample of Chinese wood oil may be considered pure beyond question. If, on the other hand, the spectrum has reversed to the normal it is evident that the oil is adulterated, since more than 15 per cent of foreign oil is required to bring this reversal about. The amount of adulteration will, however, not exceed 8 per cent, as shown by the preliminary examination. The depth of the colors of the spectrum will give some idea as to whether or not the adulteration is very small or quite large. If the green is very indistinct the adulteration will probably not be over two per cent. However, if more than two per cent of adulteration is present, the colors will be more intense, since the intensity of both the green and the red vary directly with the adulteration when over 15.5 per cent foreign oil is present. In order to determine the percentage it is necessary to find out how much adulterant must be added before the "turning point" is reached, then, by subtracting the amount added from 15.5, the amount present in the oil may be obtained.

If the preliminary examination shows the adulteration to be between the limits of 8 per cent and 15.5 per cent, successive additions of the adulterating oil are added to the sample in question until the "turning point" has been reached. Then by subtracting the amount added from the arbitrary figure, 15.5 per cent, the amount of adulteration in the Chinese wood oil is obtained, as in the case when the foreign oil present is less than 8 per cent. This procedure is best illustrated by concrete examples.

1—A sample of oil which was rejected on the kettle test during the past year, was examined as follows: the oil when placed in the cell for preliminary examination showed the characteristic spectrum of pure Chinese wood oil, or of an oil not adulterated over 8 per cent. Upon adding 15 per cent of linseed oil the spectrum was observed to have been completely reversed, showing the colors red, yellow and green comparatively strong, thus indicating that the oil contained about 5 to 8 per cent adulteration. Seven per cent of linseed oil was then added to the original sample. The spectrum of the oil thus adulterated showed a distinct green band on top with a yellow band on the bottom, indicating that sufficient adulterant had not been added. When a sample of the original oil to which 8 per cent of linseed oil had been added was examined, its spectrum showed only a yellow band; 8.5 per cent of linseed oil was then added to the original sample and the observation made; the "turning point" was now reached, shown by the characteristic red band appearing on the upper side of the spectrum. Subtracting the amount added, 8.5 per cent, from the arbitrary figure 15.5 per cent, it is evident that the sample in question contained 7 per cent adulteration.

2—Another sample taken from a rejected shipment, when first observed in the cell, showed a spectrum which was deficient in the red. It was therefore thought to contain about 10 per cent adulteration. When 6 per cent of linseed oil was added to this sample, the spectrum appeared only as a yellow band, and when 6.5 per cent of adulterant was added to the sample in question the red appeared on the upper side of the spectrum. Subtracting 6.5 from 15.5 the adulteration was shown to be 9 per cent.

3—Another sample, taken from a rejected shipment, when examined by this method, showed an adulteration of 7.5 per cent, and a sample marked questionable by the chemist of a large varnish works, showed an adulteration of 4.5 per cent.

The sample of pure Chinese wood oil used by the American Society for Testing Materials, and reported on as an exceptional oil, required an addition of 17.0 per cent linseed oil before the "turning point" occurred; hence in examining the four adulterated samples of this oil, which were used by the society in the work for 1915, it was deemed advisable, for sake of comparison, to use the figure 17.0 as well as the arbitrary figure of 15.5. Table IV gives the results in both cases:

Table IV

Sample No.	Per cent adulteration found by using as "turning point"	
	17.0 per cent	15.5 per cent
2	5.25	3.75
3	10.50	9.00
4	5.0	3.5
5	10.0	8.5

If these samples had been examined without having the pure oil the last column would have been considered as the percentage to which the Chinese wood oil had been adulterated.

SUMMARY OF RESULTS

If the adulterating oil is known, very accurate results can be obtained by this method, otherwise the accuracy is limited to 1.25 per cent either way from the true value, owing to the fact that the turning point varies between the limits of 14.5 to 17.0 per cent and the arbitrary value of 15.5 is not correct.

If, however, the oil with which the Chinese wood oil is adulterated can be determined, the accuracy is thereby increased. Lewkowitsch,[1] in commenting on tallow seed oil, says: "The most characteristic property of this oil is that it rotates the plane of polarized light to the left, *viz.*, 6° 45′ in a 200 mm. tube in a Laurent polarimeter, corresponding to —29.9 saccharimeter degrees. The optical activity is confirmed by Nash." This is being investigated as a means of detection in Chinese wood oil at the present time in this laboratory. If the attempt is successful the accuracy for adulterations with other oils will be increased, since the only other oil which causes the "turning point" to take place as high as 17.0 per cent adulteration is perilla oil, which is used only occasionally as an adulterant of wood oil.

The detection of sesame by the Baudouin test and the detection of cottonseed oil by the various methods as described in Lewkowitsch, would eliminate most of the oils which affect the turning point as low as 14.5 per cent. The remaining oils affect the turning point within such a close range of each other that the arbitrary figure assumed as the turning point would be very close to the true value.

The writer believes that concentration of the adulterating oils by the light break method described by Ware and Schumann[1] may facilitate the detection of those oils readily identified.

Another factor which must be taken into consideration is the fact that the prism itself has a dispersive power and if its dispersion is too large the anomalous dispersion of Chinese wood oil will be counteracted. Also, prisms, though having a dispersion low enough to be used in this method, may vary from each other, so that in using an instrument of this type one must first calibrate his prism before making determinations. This is readily done by finding the "turning points" brought about by the addition of different oils to Chinese wood oil.

The fatty acids of Chinese wood oil also show anomalous dispersion. This fact gives promise of interesting information when studied in connection with other fatty acids.

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COMPOSITION OF WOOD TURPENTINE[2]

By Maxwell Adams

Received June 14, 1915

Because of the shortage in the supply of "gum" turpentine, and the enormous wood waste of our lumber mills during the past ten years, many methods for the extraction of turpentine from wood have been proposed. The economics of steam, solvent, alkali digestion, oil bath, destructive distillation, under both increased and diminished pressure processes have been given thorough consideration, but very little attention has been accorded the composition of the turpentine obtained by these different procedures. Although the products obtained by these various methods differ widely as to color, odor and physical constants, without any attempt at standardization, they have all been classed together. The result has been that a strong prejudice has arisen against any product sold under the name of wood turpentine.

The physical properties of wood turpentine obtained, presumably, from long leaf or Cuban pine, have been

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[1] "Chemical Technology, and Analysis of Oils, Fats and Waxes," Vol. II, p. 70.

[1] This Journal, 6 (1914), 806.

[2] This work was in part done in Prof. Wallach's laboratory at Göttingen, Germany, and supported by the Adams Fund of the Nevada Agricultural Experiment Station.

worked out by Hawley,[1] F. W. Richardson and J. E. F. Whitaker,[2] Marcille,[3] Parry.[4] Others have published methods for the analysis of different kinds of turpentine, but for the most part these workers have been concerned with the identification of adulterants, or with physical constants; little attempt has been made to identify or separate the different terpenes present, and in no case observed has wood turpentine obtained from any variety of Western pine been investigated.

The present investigation was undertaken for the purpose of determining the physical properties and identifying the various terpenes present in the wood turpentine prepared from pine growing in the western part of the United States. The "gum" turpentines of the varieties chosen have previously been examined by Schorger.[5]

Samples of wood from Western yellow pine (*Pinus ponderosa*), obtained from trees growing in the neighborhood of Independence Lake, Cal., Jeffrey pine (*Pinus jefreyi*) from trees growing on the Eastern slope of Mount Rose and near Galena Creek, Nev., and from single leaf nut pine (*Pinus monophylla*) growing on the western slope of the mountains between Steamboat Springs and Virginia City, Nevada, were collected. Each sample was distilled under diminished pressure in a retort, around which circulated hot oil, according to the method described by Adams and Hilton.[6] The distillate from the wood was separated roughly into three fractions: (1) that which comes over below 160°, (2) the distillate between 160 and 220, and (3) that driven over above 220°. In most of the following experiments only the first fraction was used. The second fraction contained considerable quantities of decomposition products, which could be removed only by alternate treatments with sulfuric acid and caustic soda, together with steam distillation. Since treatment with these chemicals might affect the chemical composition of the oil, the second and third fractions were discarded. This procedure has doubtless slightly increased the percentage of the lower boiling terpenes over that which would be found in ordinary crude wood turpentine.

SINGLE LEAF NUT PINE

A sample of wood turpentine obtained from the distillation of the wood of single leaf nut pine, according to the above method, when redistilled with steam, had the following physical constants: d_{15° 0.9702, n_{D15° 1.4771, α_{D20° +21.15. The oil was colorless and had a very characteristic aromatic odor. A sample of the oil was dried over fused potassium carbonate, distilled at a pressure of 15 mm. and separated into three portions:

I—B. p. 50–60°, consisted of 77.9 per cent of the original oil and had the following properties: d_{15} 0.8691, n_{D15° 1.4693, α_{D20° +29.76

II—B. p. 60–80°, consisted of 7.6 per cent of the original oil and had the following constants: d_{15} 0.8721, n_{D15° 1.4777, α_{D20° +11.78

III—B. p. 80–135°, consisted of 9.2 per cent of the original oil and had the following constants: d_{15} 0.8982, n_{D15° 1.4856

There was a residue left in the distilling flask consisting of 5 per cent of the original oil. Each fraction decolorized an alkaline solution of permanganate and absorbed bromine readily, but none gave a test with sodium bisulfite or semicarbizide for aldehydes or ketones.

α-PINENE—From Fraction I, when distilled over sodium, there was obtained a material with a boiling point of 155–156°, which when dissolved in acetic acid and treated with amyl nitrite and hydrochloric acid gave crystals of pinene nitrosochloride. After dissolving in chloroform and reprecipitating with methyl alcohol the crystals melted at 103°. The nitrol piperidene, prepared in the usual manner[1] from the nitrosochloride melted at 118°. The above results prove the presence of *d*-α-pinene.

β-PINENE—Fraction II, after several redistillations over sodium, yielded 15 per cent, or about one per cent of the original oil, which boiled at 167°; 3.7 grams of this oil were added to 4.9 grams of potassium permanganate, and 1.8 grams of sodium hydroxide dissolved in 11 grams of water and crushed ice. The mixture was agitated on a shaking machine for 6 hours, according to the method of Wallach.[1] As the ice melted the temperature was allowed to rise to that of the room. The hydrocarbons not acted upon were driven off with steam and the solution remaining in the retort was filtered from the oxide of manganese and the filtrate evaporated, while a constant stream of carbon dioxide was conducted through the solution. Crystals of sodium nopinate separated when the solution cooled. The yield, however, was so small that an attempt to prepare the nopinic acid in sufficient quantity for complete identification failed.

CADINENE—The third fraction, which was light yellow in color, when heated at atmospheric pressure began to boil at 170° but turned brown, indicating some decomposition. A second portion was therefore distilled at 15 mm. pressure and separated into two portions. The lower boiling fraction was tested for limonene and phellandrene without positive results. The higher boiling fraction, after distilling over sodium, thereby removing considerable tar, boiled without any apparent decomposition at 205–215°. This fraction, thus purified, was dissolved in four parts of glacial acetic acid, cooled in brine and ice, and saturated with dry hydrogen chloride. A heavy brown liquid separated, which when placed in an open dish evolved considerable gas and quickly solidified. This solid was dissolved in acetic acid and allowed to stand for 12 hours. A good yield of white crystals separated, which melted at 117°, thus establishing the presence of cadinene.

These results, when compared with those of Schorger[2] on the volatile oil obtained from the oleoresin, indicate that wood turpentine obtained from this species is almost identical in both physical properties and chemical composition with the pure "gum" spirits.

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[1] *Eighth Int. Congr. Applied Chem.*, **1912**, and Forest Service, *Bull.* **105**.

[2] *J. Soc. Chem. Ind.*, **30**, 115.

[3] *Bull. soc. chim.*, **2**, 762.

[4] *Chem. and Drugg.*, **81**, 52.

[5] THIS JOURNAL, **5** (1913), 971, and U. S. Forest Service, *Bull.* **119**.

[6] *Ibid.*, **6** (1914), 378.

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[1] *Ann.*, **356**, 288.

[2] *Loc. cit.*

JEFFREY PINE

In the following experiments on the volatile oil from Jeffrey pine especial care was taken to identify the wood used, because the differentiation between *Pinus jefreyi* and *Pinus ponderosa* is very close, and some confusion has arisen on this account. Sadtler[1] reports having obtained heptane from *Pinus ponderosa*. This error probably arose from a confusion of Yellow pine with Jeffrey pine.

Sudwirth[2] says in writing of Jeffrey pine: "Some specialists consider it a variety of *Pinus ponderosa*, which it resembles so closely in its habits and soil and climate requirements that from the foresters' point of view there appears to be no practical reason for distinguishing the two."

Jepson[3] says, "The Jeffrey pine which merges insensibly into the Yellow pine is in its typical form found at high elevations ranging from 6000 to 9000 feet. By some writers this variety is regarded as a species. While the typical form is clearly to be recognized it is to be said, on the contrary, that Jeffrey pine not only passes over into Yellow pine, but that the transition forms in the intermediate region are quite as numerous and occupy as extensive an area as the true Jeffrey pine itself."

In the publications of the United States Forest Service[4] similar ideas are expressed as follows: "The Jeffrey pine bears very close resemblance to western yellow pine and some authorities regard it simply as a variety."

Because of the distinct chemical difference in the volatile oils obtained from the woods of these trees, as shown by the following analyses, there is good evidence for the contention that Yellow and Jeffrey pine are distinct species; at least there is a definite chemical method for distinguishing between them. It would be interesting in this connection to examine chemically the volatile oil from a specimen which the botanist considers a transition form.

A sample of wood turpentine obtained from distilling carefully selected wood from a well identified Jeffrey pine tree, was distilled under diminished pressure. The distillate thus obtained was steam-distilled, the resulting volatile oil dried over potassium carbonate and separated into four fractions by distilling at a pressure of 15 mm.:

Fraction	I	II	III	IV	Residue
Boiling point	25–30°	30–35°	35–65°	65–125°	
Per cent obtained	74	13	6	4	3

The first three fractions distil without decomposition at ordinary pressure. They were combined and distilled over sodium and a portion, consisting of about 90 per cent of the entire volatile oil, was obtained which distilled between 99 and 100.5°. This distillate gave no test for the double bond with von Beyer's reagent, neither would it absorb bromine. When added to amyl nitrite in acetic hydrochloric acid solution it gave no test for pinene. The physical properties were d_{15° 0.6877, n_{D20°, 1.3890, optically inactive. These tests prove that the volatile oil from Jeffrey pine wood contains from 90 to 95 per cent of normal heptane.

The fourth fraction was redistilled at diminished pressure and separated into two parts. The lower boiling fraction was distilled over sodium and tested for limonene and dipentene. It absorbed the calculated amount of bromine to form the tetrabromide, butr cystals were not obtained from the oil. The higher boiling fraction gave a precipitate with sodium bisulfite, which would indicate the presence of citronellal, but the small amount of the material available prevented making confirmatory tests.

YELLOW PINE

A sample of volatile oil obtained from Yellow pine, according to the method already described, was clear, water-white, and had the odor which is characteristic of Yellow pine wood. Its specific gravity at 15° was 0.8626, index of refraction 1.4727, specific rotation —13.15.

On distilling 500 grams of the oil, using a Norton and Otto distilling tube, and at a barometric pressure of 642 mm., the following results were obtained:

FRACTIONAL DISTILLATION OF THE VOLATILE OIL OBTAINED FROM THE WOOD OF *Pinus ponderosa*

Temp. ° C.	Distillate Observed	Distillate Cumulative	Specific gravity, 15°	Index of refraction, 15°	Specific rotation, 20°
156–158	4.72	4.72	0.8467	1.4621	—15.14
159.9	9.39	14.11	0.8525	1.4710	—14.48
161.2	9.20	25.32	0.8546	1.4731	—14.57
162.1	9.99	33.32	0.8552	1.4732	—13.68
163.0	10.08	43.40	0.8559	1.4738	—12.15
163.9	9.51	52.91	0.8562	1.4740	—11.89
164.9	9.65	62.56	0.8590	1.4744	—11.04
176.7	9.95	72.51	0.8603	1.4745	—10.56
169.8	6.958	79.47	0.8633	1.4752	—10.97
173.4	7.86	87.35	0.8733	1.4913	—14.12
180.3	9.36	97.71	0.9246	1.5038	—17.22
Residue	2.3				

When the above table is compared with a similar one prepared by Schorger[1] on "gum" spirits, it will be noted that the wood turpentine contains more of the low boiling and less of the high boiling fractions than that obtained from the oleoresin of western pine. This difference is perhaps due to the fact that wood turpentine was obtained from the first fraction of the wood distillate and that the barometric pressure was abnormally low.

A second analysis, which was made upon a smaller but a more representative sample of Yellow pine wood distillate, and which contained all the volatile oil given off up to the temperature at which the wood began to decompose, gave the following results:

Fraction	I	II	III	Residue
Distilling temperature	156–164°	164–172°	172–180°	
Per cent distillate	8.7	72.5	16.4	2.4

Fraction I of the oil when tested for α-pinene readily yielded nitropiperidine which melted at 118°. Fraction II, when oxidized with potassium permanganate according to the method previously described, gave an abundant yield of crystals of nopinic acid, which melted at 125°. Fraction III gave crystals of limonene tetrabromide when treated with bromine.

CONCLUSIONS

The presence of practically every constituent of the

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[1] *Am. Jour. Pharm.*, **51**, 176.

[2] "Trees of the Pacific Slope."

[3] "The Silva of California," p. 80.

[4] Silvica Leaflet, p. 21.

[1] U. S. Forest Service, *Bull.* **119**, 12.

volatile oil of the oleoresin has been identified in the wood turpentine of the three varieties of wood under examination. The physical properties and the percentage composition do not always agree, yet they coincide as closely as could be reasonably expected, when we consider the inaccuracies of some of the methods available. The results thus far obtained justify the conclusion that the volatile oil obtained from distilling wood under diminished pressure is similar to that obtained from the oleoresin of the same species of tree.

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LABORATORY AND PLANT

SOME IMPROVED ELECTRICAL LABORATORY APPARATUS

By Chas. Morris Johnson

Received July 23, 1915

I—QUICK HEATING ELECTRIC FURNACE FOR THE DETERMINATION OF CARBON IN STEEL, ETC., WITH TRAIN

The train (Fig. I) consists of: (1) a mercury tube to detect leaks and stoppages; (2) a safety jar containing a 1 : 1 solution of KOH; (3) a tower filled loosely with a plug of glass wool followed with anhydrous calcium chloride, a layer of soda lime and another plug of the wool; (4) a jar of short pieces of stick caustic potash with a plug of glass wool at the top and at the bottom; (5) a clay and rubber connector doing away with rubber stoppers at the charging end; (6) the vitrified clay combustion tube with the tapered outlet doing away with the rubber stopper at this point also; (8) is the jar of granulated zinc to stop acid and sulfur; (9) is a jar of phosphoric acid powder and (10) is the bulb for absorbing and weighing the CO_2. This train was designed by the writer, being a simplification of his train described in *J. Am. Chem. Soc.*, May, 1908.

The writer has now used the tapered clay combustion tubes for over two years for the determination of carbon in steel, etc., and has been using them for some months for oxygen determinations also.

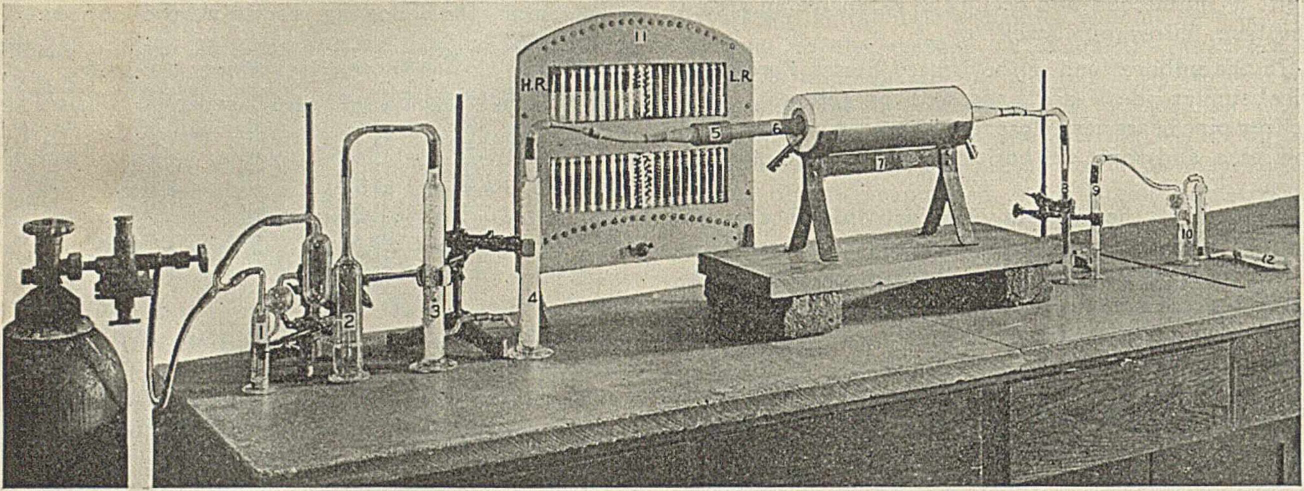

Fig. I

The new split muffle type of furnace was designed about a year ago. It is of most simple construction, consisting of a split muffle of kieselguhr, a coil of nichrome wire embedded in alundum cement as a replaceable heating element, and a supporting stand. The furnace will heat from the cold to 1000° C. in 20 to 25 minutes with a current consumption of 3 to 4 amperes. It can be cooled rapidly, if desired, by lifting off the top half of the split muffle. The writer has supplanted for some time in this laboratory, and in another laboratory under his direction, all of the type introduced by him in 1908[1] with this new type shown in Fig. I.

This type of electric furnace can be built in any length of heating surface with coils connected in parallel. The type shown in Fig. I is 13 inches long by $3^{5}/_{32}$ inches O. D. and $1^{15}/_{16}$ I. D. The writer built the same type for organic work 18 inches long. By using three rheostats a long furnace can be made to heat to three different temperatures at once or it can be heated in one part if desired.

The furnace presents a bright neat appearance with its white muffle; new heating elements can be kept in stock and quickly put in place in case of a burn-out as there are no screws or powdered heat insulation to bother with. In case the muffle becomes soiled it can be put in a muffle furnace and heated to a red heat for about 20 minutes when it becomes as white as new. The simple construction makes the furnace a very inexpensive one.

By using a split insulation muffle of $5^{5}/_{8}$ inches O. D. and $2^{5}/_{32}$ I. D., the writer obtained a furnace of this type which is developing 1020° C. with a current consumption of only 2.7 amperes. This furnace has been in daily service in this laboratory for several months.

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[1] *J. Am. Chem. Soc.*, **30** (1908), 773.

II—A NEW TYPE OF PORTABLE LABORATORY RHEOSTAT

Fig. II shows the rheostat designed by the writer with a view to making repairs more easy and to have a piece of apparatus with sufficient capacity to fill the needs of most laboratories. It consists of 21 coils of nichrome wire giving a total resistance of 30 ohms and a maximum capacity of 14 amperes. It can be used either on the furnace shown in Fig. I or with the large one given in Fig. III. It holds the large furnace in Fig. III for any length of time at any temperature from 200° C. up to 1000° C.

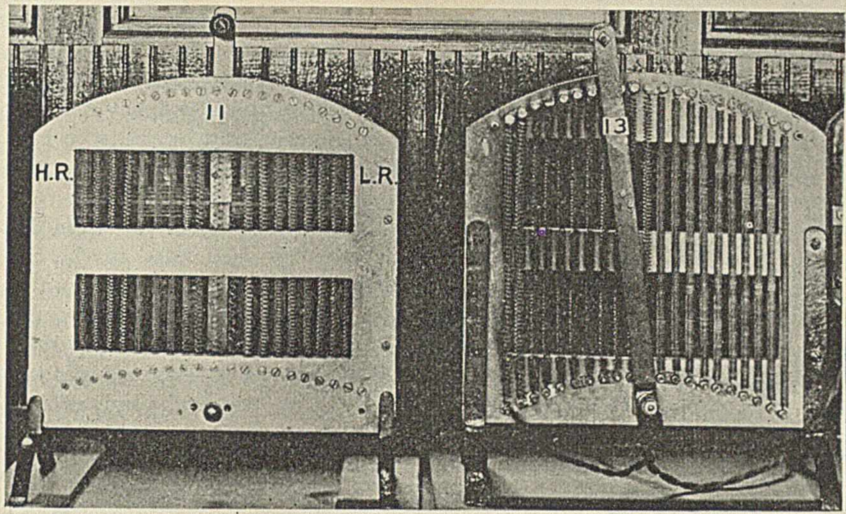

Fig. II

The rheostat occupies but a few inches of table width and can be set behind the other apparatus as illustrated in Fig. I. Repairs are easily made and the white board of hard asbestos board gives a cheerful effect. If desired, two such rheostats can be bolted together making a double instrument. The rheostat is practically indestructible so long as its capacity is not exceeded as there is nothing about its design to get out of repair. In Fig. II, 11 is the front and 13 the back view showing details of construction.

III—A NEW RECTANGULAR LABORATORY ELECTRIC MUFFLE FURNACE WITH SOLID HEAT INSULATION CASE

The furnace shown in Fig. III is in principle just the same as the cylindrical furnace described in Fig. I. A white solid case of kieselguhr surrounds the heating element and working muffle which has an available working space of $11^1/_2 \times 5 \times 7$ in. This is ample for most laboratories. To control the temperature of the furnace the rheostat shown in Fig. II is used and by means of it any desired temperature can be maintained for hours at a time up to 1000° C. The author keeps three furnaces of this size in constant service.

On account of the simplicity of construction, the repairs can be easily made and, as no insulating powder is used, the furnace is free from powder falling out into the working chamber. With the absence of the heat insulating powder the repairs are much simplified. The whole furnace is supported and enclosed in a stout, soft steel frame which makes it very rigid.

The white casing makes an attractive looking piece of apparatus. The interior muffle on which the wire is wound is of a special composition which is now being tested out. This same composition used in combustion boats made the remarkable run of 525 steel combustions per boat with the test boats still in commission but considerably worse for wear. It is highly probable that the muffle will be equally superior to the old mixture for heating core muffles.

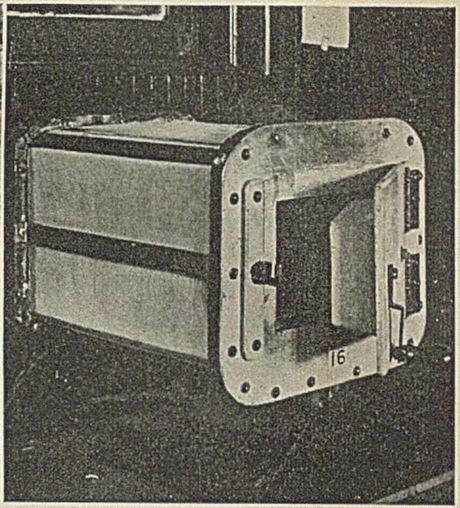

Fig. III

The scheme is to keep these working muffles already prepared with the heating wire cemented on them ready for use so that repairs can be quickly made. As the furnace consists of three parts its construction it is considerably cheaper than that of the old types, besides having the advantages already mentioned.

The door opens horizontally: the author has found this arrangement to be much more desirable than the vertically opening door used in some of the old types.

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A NEW DIRECT-READING VISCOSIMETER

By R. F. MacMichael

Received September 8, 1915

Viscosimeters are instruments for measuring the viscosities of fluids. They operate on two general principles:

I—The *time* required to produce a definite relative movement of the fluid particles under a given force.

II—The *force* required to produce a definite relative movement of the fluid particles in a given time.

Familiar examples of the application of the *first* principle are:

a—Long capillary tube, used by scientists.

b—Short-tube commercial instruments, *e. g.*, Saybolt, Engler and Redwood.

c—Falling bodies within the fluid, *e. g.*, Arnold, Ladenburg and Flowers.

d—Rising bubble.

e—Retarded surface, *e. g.*, Doolittle and Stormer.

In each of these instruments the time required for a definite movement of the fluid particles to take place is noted. The acting force in each case, except that of Doolittle, is gravity. This may be the weight of the fluid itself, or the weight of a foreign body acting within or upon the fluid, or bouyancy of an air bubble. In the case of Doolittle, energy is stored up in a steel spring, and allowed to expend itself in rotating a disk.

In well designed instruments of this class the control of the acting force is almost absolute. However, the time required to make a test is frequently a drawback, and the difficulties of maintaining an exact temperature during the test are often very serious. The fluid must usually be strained before testing; with very viscous fluids, this necessitates heating; Small particles of foreign matter within the instrument may interfere with proper action, and at times will introduce very large errors. It is only rarely that colloidal or glue-like substances, or suspensions of finely divided solids, such as clay slips, can be handled successfully in these instruments.

Results are usually expressed in seconds of time. Even in the simplest forms of instruments, elaborate and involved calculations are required to reduce the readings to scientific units, while most of the instruments noted above are not even susceptible to such calculations. In the latter case the results are expressed in an arbitrary manner in empirical units. The personal equation is large, and considerable care and skill are required if accurate results are to be obtained. The difficulties and annoyances attending the constant use of a stop-watch are too well known to require comment.

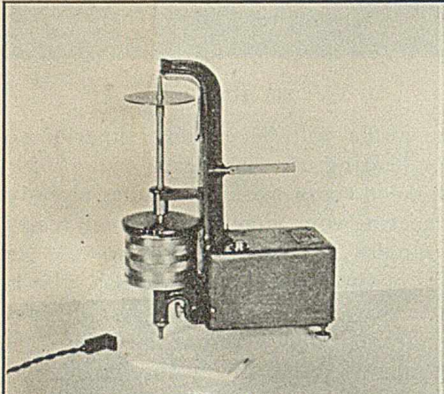
Fig. 1

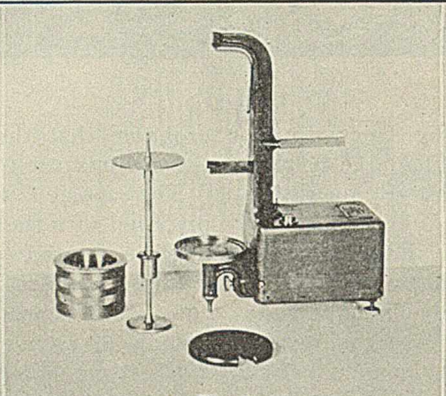
Fig. 2

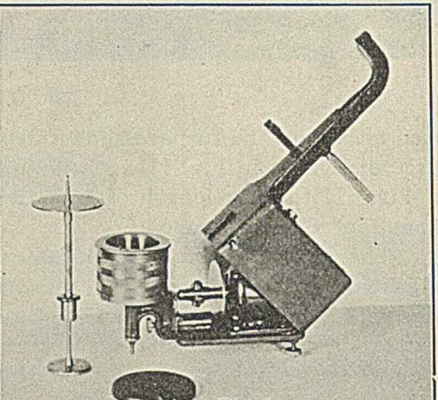
Fig. 3

Viscosimeters of each of the above types have been before the public for many years. Up to the present it has not been found possible to combine in any one instrument based on the first principle all of the essential features required in a viscosimeter for general scientific and commercial use. It is not to be asserted that this cannot be done, but merely that after many years of effort, it has not been done.

The most prominent examples of instruments based on the *second* principle are those of Couette, Clark and MacMichael. In each of these instruments a cylinder or disk is suspended in a cup of fluid; then either the disk or the cup is revolved, and the torsional force noted. Couette's experiments were conducted at an early date, and though the principles involved were sound, mechanical difficulties were encountered which proved insurmountable at that time. In the more recent Clark machine, the tangential force applied to the rotating member is measured: this instrument under proper conditions gives excellent results, but the construction introduces mechanical complications which render it unsuitable for general use.

In the author's viscosimeter, the force exerted by the rotation of the fluid itself on the stationary member is measured. This force is equal and opposite to that applied to the rotating member. A description of this instrument follows.

APPARATUS—Fig. 1 shows the machine assembled and ready for a test. A disk is suspended in the cup of fluid by a torsion wire about 10 in. long, running down through the stem of the plunger, and fastened near the bottom. The head of the torsion wire is triangular, and is held between two grooved pins at the top of the standard. The cup and plunger may be removed and replaced without manipulating any catches or fastenings.

Fig. 2 shows the cup and plunger removed for cleaning. All surfaces are smooth and rounded and may be cleaned with the greatest ease. The cup is oil-jacketed, being formed of two pieces of heavy spun brass. Within the oil jacket is immersed an electric heating coil. This coil draws current from the same line as the motor, only one connection being necessary.

The fluid to be tested is heated in place, no other heating device being required. The operation is very rapid. Stirring is effected by a slight vertical movement of the plunger. A bent thermometer inserted through an opening in the cover indicates the temperature, the bulb being immersed in the fluid. The temperature during test may be controlled to within a small fraction of 1° F. For low temperature work, the fluid and the adjacent parts are chilled in an ice bath or brine solution.

The graduated dial at the top of the plunger is secured to the stem by a friction disk, permitting the adjustment of the zero mark to its proper location.

The fine adjustment is effected by means of the steel wire pointer at the head of the standard.

The same figure shows the dash pot on the stem of the plunger. This device is frictionless and automatic in action, and requires no attention from the operator. Its function is to check incipient vibrations and to permit quicker readings by damping the action.

In Fig. 3 the standard and hood are thrown back, showing the driving mechanism. The speed control is of the familiar phonograph type and gives excellent results. The motor is specially wound for this service and is furnished either in a. c. or d. c. for use on ordinary lighting circuits. Variations in voltage do not affect the accuracy of the determination.

OPERATION—In operation, the cup is filled to the mark on the side with the fluid to be tested. This requires about 100 cc. The cover is put on and the thermometer inserted. By means of the heating coils the temperature is raised to nearly the desired point. As expansion of the fluid will probably have taken place, the excess fluid is removed by means of a pipette furnished with the machine. This automatically fixes the proper level, without further attention from the operator. The temperature is brought exactly to the desired point, the thermometer removed and the motor started. Within a few seconds the dial will have come to rest, and will remain steadily at one point. The deflection noted is the viscosity of the fluid. This entire operation is very rapid, so that the drop in temperature on ordinary work is entirely negligible.

For extreme accuracy, the temperature may be raised slightly above the desired point, and an allowance made for the drop up to the moment of reading. This will seldom be found necessary in actual practice.

READINGS—The readings are in degrees of angular deflection, 300° to the circle, designated as °M. The practical working unit is 1/1000 of the absolute unit. As water at 20° C., or 68° F., has exactly 1/100 of the absolute unit of viscosity, water at this temperature reads 10° M. Thus, by shifting the decimal point, practical units, absolute units, and specific viscosity may be obtained at one reading. Readings are taken directly from the dial, no intermediate calculations being required.

Torsion wires ordinarily give water as 10° M., and may be used up to 500° M. This covers the range of most liquids ordinarily to be tested. For extremely thin and extremely thick liquids, lighter and heavier wires giving readings for water of 100° and 1°, respectively, are supplied. This covers every range of viscosity up to about 5000° M., or 500 times that of water.

CALIBRATION of the instrument is effected by testing with a standard fluid of known viscosity. Sugar syrup is used for this purpose. Water may be used for calibrating, but this is not advisable as it is too thin for the best results. Should the reading of the dial, on test, be found to vary slightly from the correct value, the speed of rotation of the cup is altered by means of the speed control screw at the lower front of the machine. The deflection of the dial will vary directly as the speed, other factors being constant, and the correct indication may be obtained. When calibrated accurately for one value, the readings will be correct for any other value.

The accuracy of the instrument under ordinary working conditions should be well within an error of $^1/_2$ per cent above or below the true value. For extremely accurate work, extra precautions should insure results within a small fraction of this error.

As the time required for a reading is extremely short, determinations may be made on suspensions, such as clay slips, which settle rapidly.

Colloidal solutions such as glues, gums, starch solutions, gelatine, and other similar materials can be handled successfully. Likewise, the viscosity of complicated mixtures such as catsup, or other food materials or chemical substances may be readily determined. Straining is unnecessary as small particles of foreign material do not in any way affect the accuracy of the determination.

It is believed that this instrument offers an accurate, rapid, convenient and thoroughly satisfactory means for determining the viscosity of fluids.

SEATTLE, WASHINGTON

ADDRESSES

COAL-TAR DYES AND THE PAIGE BILL[1]

By BERNHARD C. HESSE

The Paige Bill is the latest of a number of attempts to saddle compulsory working of patents upon American inventors in the United States. Its text is as follows:

A BILL

To amend Sections Forty-Eight Hundred and Eighty-Six and Forty-Eight Hundred and Eighty-Seven of the Revised Statutes, relating to patents.

Be it enacted by the Senate and House of Representatives of the United States of America in Congress assembled, That Section Forty-Eight Hundred and Eighty-Six of the Revised Statutes, as amended by Act of Congress approved March Third, Eighteen Hundred and Ninety-Seven, be, and the same is hereby, amended so as to read as follows:

"SEC. 4886. Any person who has invented or discovered any new and useful art, machine, manufacture, or composition of matter, or any new and useful improvement thereof, not known or used by others in this country before his invention or discovery thereof, and not patented or described in any printed publication in this or in any foreign country before his invention or discovery thereof or more than two years prior to his application, and not in public use or on sale in this country for more than two years priors to his application, unless the same is proved to have been abandoned, may, upon payment of the fees required by law and other due proceedings had obtain a patent therefor: *Provided, That no patent shall be granted on any application filed subsequent to the passage of this Act upon any drug, medicine, medicinal chemical, coal-tar dyes or colors,*

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[1] Address before the Rochester Section of the American Chemical Society, Rochester, New York, October 4, 1915.

or dyes obtained from alizarin, anthracene, carbazol, and indigo, except in so far as the same relates to a definite process for the preparation of said drug, medicine, medicinal chemical, coal-tar dyes or colors, or dyes obtained from alizarin, anthracene, carbazol, and indigo."

SEC. 2. That Section Forty-Eight Hundred and Eighty-Seven of the Revised Statutes, as amended by Act of Congress approved March Third, Eighteen Hundred and Ninety-Seven, and as further amended by Act of Congress approved March Third, Nineteen Hundred and Three, be, and the same is hereby, amended so as to read as follows:

"SEC. 4887. No person otherwise entitled thereto shall be debarred from receiving a patent for his invention or discovery, nor shall any patent be declared invalid, by reason of its having been first patented or caused to be patented by the inventor or his legal representatives or assigns in a foreign country, unless the application for said foreign patent was filed more than twelve months in cases within the provisions of Section Forty-Eight Hundred and Eighty-Six of the Revised Statutes, and four months in cases of designs, prior to the filing of the application in this country, in which case no patent shall be granted in this country.

"An application for patent for an invention or discovery or for a design, filed in this country by any person who has previously regularly filed an application for a patent for the same invention, discovery, or design in a foreign country, which, by treaty, convention, or law, affords similar privileges to citizens of the United States, shall have the same force and effect as the same application would have if filed in this country on the date on which the application for patent for the same invention, discovery, or design was first filed in such foreign country: *Provided,* That the application in this country is filed within twelve months in cases within the provisions of Section Forty-Eight Hundred and Eighty-Six of the Revised Statutes, and within four months in cases of designs, from the earliest date on which any such foreign application was filed. But no patent shall be granted on an application for patent for an invention or discovery or a design which had been patented or described in a printed publication in this or any foreign country more than two years before the date of the actual filing of the application in this country, or which had been in public use or on sale in this country, for more than two years prior to such filing: *Provided, however, That in case any drug, medicine, medicinal chemical, coal-tar dyes or colors, or dyes obtained from alizarin, anthracene, carbazol, and indigo, on which a patent for a definite process for the preparation thereof had been granted on any application filed subsequent to the passage of this Act, is not manufactured in the United States by or under authority of the patentee, within two years of the granting of said patent, and after the commencement of said manufacture the same is not continuously carried on in the United States in such a manner that any persons desiring to use the article may obtain it from a manufacturing establishment in the United States, then said patentee shall have no rights under the patent laws of the United States as against any citizen of the United States who may import such drug, medicine, medicinal chemical, coal-tar dyes or colors, or dyes obtained from alizarin, anthracene, carbazol, and indigo, into the United States or who may produce or manufacture the same in the United States or who may handle for sale or use such article so imported or manufactured.*"

(NOTE—Italicized matter is the new matter introduced by this Bill.)

The Paige Bill was introduced for the purpose of forcing European dye and medicinal makers to the United States: it is utterly incompetent and unable to do anything of the sort.

The compulsory working of patents is a legislative device which appeals very strongly and persistently to the very great majority of lay minds as a most efficient, equitable, just and effective mode of introducing the industry relating to the subject matter of the patent into the country in which the patent runs. There seems to be nothing on its surface nor immediately beneath its surface to suggest difficulties in enforcement and inequalities, injustices and economic errors in such an enactment.

However, the legislative experience of 57 countries, extending over a period certainly for the past 83 years, has taught that the compulsory working of patents, if it is to be just, economically profitable and enforceable, is a thing almost impossible of achievement and presents obstacles which, to the most astute minds and the most experienced persons, seem unsolvable.

Contrary to all expectations, manufacture within the country is not so stimulated but dumping from without is thereby increased.

GENERAL NATURE OF RESULTS

To go no further back than 1832, when the United States enacted its compulsory working clause,[1] a study of that enactment shows compulsory working as absolute and as bare as any enactment could be, yet it was repealed within four years and no similar statute has ever been placed upon our statute books.

Coming next to the attempts of the French Government in 1844 and the modifications that it has from time to time added thereto, it is correct to say that the legislative measures of that country have been a series of compromises and modifications, all of them aimed at reducing the harm that was being done to France through this legislation, and at the same time retaining, in some measure, the benefits that were supposed to have accrued to France thereby.

The experience of other countries is very much the same and the latest efforts in that direction have been the conclusion of treaties between various countries, which amount in effect to an abrogation of these laws, so far as the citizens or subjects of the two countries involved are concerned. That is, two countries get together and say to each other: "If you will not enforce your working clause against my citizens, I will not enforce my working clause against your citizens."[2]

To put it in a few words: Experience has shown that this seemingly innocent legislative device is possessed not only of a "punch" but also of a "kick" (*i. e.,* it hurt and oppressed domestic inventors); that the "kick" in most cases is more powerful than the "punch" and no legislative efforts have been directed towards removing the "kick" without, at the same time, interfering with the "punch." At the end of about 80 years of legislative experimentation these 57 countries have drifted into the position of removing the "kick" by removing the "punch" at the same time. In other words, having a compulsory working law which is a dead letter towards some peoples and, at any rate, a sleeping letter towards other peoples and when enforced doing quite as much, if not more, harm than good.

THE BRITISH WORKING CLAUSE

When the British compulsory working clause was adopted in 1907, the world had a right to expect that that Act would contain the very best legislative devices that could be constructed in the light of the past experience of the world. The present European war, however, has shown how futile this British working clause was; that it failed utterly in introducing within the British realm those industries which it was the particular aim and object of that enactment to bring into Great Britain from without. Instead of the $500,000,000 of fresh capital that were lavishly promised to be introduced into Great Britain because of this enactment, and in place of the thousands, if not hundreds of thousands, of fresh labor to be employed there-

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[1] THIS JOURNAL, **7** (1915), 304.

[2] Transactions International Association for the Protection of Industrial Property, **16** (1912), 183.

through, the best obtainable information[1] is that not to exceed fifty different firms came to England from without, that not to exceed $5,000,000 of fresh capital has found its way into Great Britain, and not to exceed 8,000 additional people have been employed in Great Britain. No new self-contained industries of any kind were thereby brought to Great Britain; on the contrary, dumping from without grew apace by virtue of revoked patents.[2]

WHY BRITAIN FAILED

Without attempting to go into the refinements of the various reasons that have been given for the failure of the British working clause, it can be fairly stated that the main reason why it failed of efficient enforcement is that it called for an encroachment upon or an abrogation of personal and private rights which in all civilized countries are regarded as inalienable, namely: that the burden of proof of a charge lies upon him who makes it. In order to make the British working clause efficient, it was necessary to reverse that principle and hold every charged person guilty until he proved himself innocent. In other words, in order to make the British working clause effective it is necessary to regard every patentee as dishonest and to regard everybody else as thoroughly honest and to put every patentee in a position where he must establish his innocence[3] by full disclosure to the public of all of his business dealings on the subject matter of the patent.

GERMAN WORKING CLAUSE AND DYE PATENTS

It has been asserted[4] that the presence of a compulsory working clause in the German patent law is *the* cause of the existence of a coal-tar chemical industry in Germany, because unless patents were worked efficiently in Germany they could be revoked and would revert to the German public and that is why non-Germans do not take out German coal-tar process patents. Nothing could be further from the truth than that assertion. So far as I have been able conveniently to ascertain, not one single German coal-tar patent, whether held by a foreigner or by a German subject, has ever been revoked for non-working, and yet I personally know of any number of German patents for processes for making coal-tar products, which are not worked, never have been and probably never will be, worked in Germany. Americans, British, Swiss and French have taken out patents in Germany for coal-tar products and dyes.

The rate of revoking patents in Germany for non-working is probably not in excess of four patents per year;[5] non-Germans and Germans as well owned the patents so revoked. The British rate of revoking patents for non-working is about 5 per year.[6]

Germany herself is very willing to repeal her working clause if the other countries will do the same.[7]

COMPULSORY WORKING DOES NOT CREATE A DYE INDUSTRY

If a compulsory working clause necessarily gives birth to a coal-tar chemical industry, then it necessarily follows that France should dominate the world, because the French working clause dates back to 1844, or 12 years *before* the first coal-tar dye (apart from picric acid) was invented, and the German compulsory clause dates from 1877 only, or 21 years *after* the first coal-tar dye was invented and 17 years after the German coal-tar dye industry started; Germany had *no* patent laws prior to 1877. Switzerland had no compulsory working until 1907, or 48 years *after* its coal-tar dye industry started; on this reasoning of the supporters of compulsory working, Switzerland, which has for 40 years at least been second to Germany in the coal-tar dye business, should not have had any coal-tar dye industry at all. Furthermore, a French patent is invalidated if the subject of that patent or materials similar to the subject of that patent are imported into France or French territory from without. Theoretically, this certainly is a very effective way of insuring that the consumption within the country would be produced within the country. However, France, either in 1844 or shortly thereafter, proceeded to remove the "kick" by inserting a modification that partly manufactured materials for use in a process patented in France could be imported into France without invalidating the patent. That is the milk in the cocoanut: France has had for a great number of years, certainly since 1873, within her borders, branches of practically all of the German coal-tar dye factories, but they were merely, or, almost merely, assembling plants and Germany shipped into France its partly manufactured materials, carried out the patented steps in France, complied with the French working law, but absolutely nullified the hope for favorable economic effect. In short, when France could no longer trade with Germany, France was left holding the empty bag; she had "assembling" plants but she had nothing to "assemble." Russia and England were in precisely the same fix, while Switzerland was not much better off.

The following letter (in translation), dated Sept. 13, 1915, and addressed to me by the French branch factory, established in 1873, of a very large German coal-tar dye plant, reinforces this statement:

"We are purchasers of beta-naphthol, beta-naphthylamine, diphenylamine, dimethylaniline.

"Will you please tell us whether you sell these products, and in case you do, please inform us of your conditions and the quantities which you can offer us.

"At the same time, we beg you to inform us of other raw materials of interest for the manufacture of dyestuffs which you may have to offer us, aside from those mentioned above.

"In case your answer should be negative, we should be obliged to you if you would kindly indicate to us the addresses of firms to whom we could address ourselves with prospect of success."

GENERAL SURVEY OF COMPULSORY WORKING

Without going into all of these enactments in detail, it will be enough to say that some 15 years ago it became my specific duty to examine the working clauses of all countries and to report to my principals, who were then in Germany, as to the possibility or feasibility of the enactment of a compulsory working law in the United States that could force them to come to the United States. After giving more than six months to a study of this subject, my report was in substance that I could not draft up any legislative enactment which would force an industry into the United States without doing the domestic industry of the United States in that and in other directions an amount of harm far in excess of any possible good that could flow from it and therefore that, in my judgment, a compulsory working act which would force the transplantation of the complete coal-tar dye industry into the United States would work more damage to the country at large than it could possibly do it good,[1] and that therefore in my opinion such legislation would not be enacted.

When the British working clause of 1907 was passed, I frankly confess I felt for a while that I had skipped a cog in my judgment; it seemed on the surface as though they had worked out something that I had overlooked. Be that as it may, my conclusion as to the ultimate effect of this sort of legislation was and is correct.

BELGIUM'S EXPERIENCE

How compulsory working operates in smaller countries, such as Belgium, is described as follows by Jacques Gevers, Patent Agent of Antwerp, in Vol. XVI of *Transactions of the International Association for the Protection of Industrial Property*, pp. 168–169:

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[1] THIS JOURNAL, 7 (1915), 306.

[2] *Ibid.*, 7 (1915), 306, 311, 315.

[3] *Ibid.*, 7 (1915), 313, 315, 316.

[4] *Textile World Record*, **1906** (Nov.), p. 65 and **1914** (Sept.), p. 56.

[5] *Trans. Int. Soc. Protection Indust. Property*, **16**, 177; see also *Textile World Record*, May, 1915, p. 92.

[6] THIS JOURNAL, 7 (1915), 311.

[7] *Ibid.*, 7 (1915), 305; *7th Int. Congr. Appl. Chem.* (London, 1909), Sec. XI, p. 9.

[1] See also THIS JOURNAL, 7 (1915), 311.

"The general interest that there is in abolishing reciprocally the requirement of working has been abundantly proved by the numerous papers written on this question. It is, however, of interest to consider exactly why small countries such as Belgium have still greater interest than other countries in the abolition of this requirement.

"At present the Belgian inventor or manufacturer who is the proprietor of patents is obliged to manufacture the patented article separately in Great Britain, Germany, France, Austria, Hungary, Italy, Spain, Portugal, Denmark, etc.

"Instead of being able to extend the manufacture in Belgium and export to these other countries, he is obliged, under risk of having his patent annulled, to manufacture in each country the patented articles which he sells there. This splitting up of manufacture increases the cost in a manner which is prejudicial to the success of his enterprise. Existing laws, therefore, militate against manufacture for exportation. It will be seen that exporting countries are those which suffer most from the present law. Belgium, which exports about three-quarters of its total industrial production, is obliged, in the case of patented articles, to manufacture these for the most part elsewhere, in order to maintain the validity of the patents. On the other hand, the principal argument of those who are in favor of maintaining the law as at present, is that, Belgium being a small country, foreign inventors must be prevented from importing into Belgium, notwithstanding that in return Belgium manufacturers are still to be obliged to manufacture in all foreign countries.

"But do the existing laws prevent importation into Belgium and does the foreigner really manufacture in our country the patented articles which he sells there? Facts show that he does not.

"There are used in Belgium large quantities of sewing machines, typewriting machines, cash registers, and photographic apparatus for which thousands of patents have been granted by the Belgium Government (the number, according to the Dictionnaire des Brevets, is 4,687). So that, although each Letters Patent granted says 'you are obliged by law to manufacture the patented articles in Belgium,' not 1 per cent of these articles are in fact manufactured there.

"Let us take the case of Van Berckel's patent which was annulled in 1911. These are the facts: A meat-cutting machine which is to be found in every meat salesman's establishment, was patented by Van Berckel. The inventor and his legal representatives brought successfully a number of infringement actions. The reports of these actions prove the extensive use of the machine. In Belgium alone more than 2,000,000 francs worth were sold, and as many could be furnished as required. Van Breckel's patent was subsequently attacked for non-manufacture. Notwithstanding the steps usually taken by interested parties, the Government annulled the patent, once more interpreting the law in the sense that 'exploiter' meant 'fabriquer.'

"A more attentive examination of the operation of economic laws shows also how it happens that the requirement to manufacture is nothing more or less than an obstacle to the establishment of the prosperous manufacture in Belgium of patented articles. An invention of value is never patented in Belgium alone. If, therefore, an inventor is to maintain his patents he must either manufacture in each country, or he must compromise by manufacturing only in certain countries and importing into others. If he takes the first course he must establish a factory in each country, which is impracticable. The multiplication of factories entails too large costs, raises the price of the article and prevents manufacture on a large scale and upon uniform lines.

"If he chooses between the countries, where shall he manufacture? In a small country where the consumption is small, or in a large country where the consumption is considerable? Which is the patent to be safeguarded, the patent with a limited value in a small country, or the patent granted in a large country where it is of ten times the value? The answer is obvious. Manufacture of patented articles is perhaps established in the larger countries to ensure the validity of the patent there. The establishment of manufacture in smaller countries is omitted in order to avoid multiplication of factories. That is why small countries, which have a limited domestic market and export by far the largest part of their manufactured products, have the greatest possible interest in abolishing upon reciprocal terms this requirement of manufacture under patents which is so prejudicial to export trade."

No one should attempt to pass upon this subject without first having given careful thought to the Symposium of the Imperial Industries Club of Great Britain printed in full in THIS JOURNAL, 7 (1915), 307–317 and to the facts collected in the paper published in THIS JOURNAL, 7 (1915), 304–307.

NO BENEFITS FROM COMPULSORY WORKING

The supporters of the compulsory working of patents have not yet come forward with any specific or concrete instance of advantage arising therefrom and to the enacting country; as much as they have done is to indulge in glittering generalizations, vague promises and hypothetical assurances; both sides were ably represented at the Symposium of the British Industries Club just above referred to, yet the supporters failed utterly to make out any reasonable case in their favor.

COMPULSORY WORKING A DELUSION

Personally, I would most heartily welcome a compulsory working law that would force industries from without into the United States and at the same time would do no harm to the United States in other directions, or at any rate, not enough to counterbalance any benefits that might flow from such introduction of extra-territorial industries. My own view is that any attempt to solve a legislative problem of that kind is of the same order, not merely as to attractiveness and desirability, but as to absolute unsolvability as is the problem of perpetual motion. The reason seems to be that you cannot treat domestic and foreign inventors each and all alike *and at the same time* "play favorites" toward the domestic inventors; it is most frequently *not* to the best interests of domestic inventors to be compelled to work their patents; therefore the makeshifts, compromises and the like that have everywhere been introduced in the hope of helping domestic inventors but in fact and at the same time benefitting foreign inventors to the same extent; hence, the inoperativeness of all such enactments.

Therefore, believing as I do, and for the reasons given, I am convinced that if we embark upon a policy of compulsory working of patents, we shall simply be living in a fool's paradise; we shall be leaning on a broken reed and we shall be fooling ourselves thoroughly and from the ground up. In the language of the day: "It can't be done."[1]

In the past 20 years, numerous attempts have been made in the Congress at Washington to pass some such legislation. However, and fortunately as I firmly believe, such legislation has not been passed. The judgment of the American Bar Association and of the American Patent Law Association is almost unanimously against any such legislation. As a result of a study of the various hearings held in Congress, I have come to the conclusion that those who favor compulsory working do not take anything but a very personal, that is, a very narrow view of such legislation. The most any legislation can hope to accomplish is the greatest good to the greatest number; to condemn legislation because in individual or isolated cases it does not work quite right, is visiting condemnation upon practically all of our legislation and an indictment of all law.

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[1] THIS JOURNAL, 7 (1915), 311.

I believe that those who take the entire interests of the entire United States into consideration and balance benefit against disadvantage, are of the opinion that such an enactment will do us more harm than it can possibly do us good (even though it might rectify certain abuses to-day existent) because, as a result thereof, other abuses and inequalities of far greater extent and of greater economic effect would necessarily arise at once.

PAIGE BILL SHOULD BE KILLED

What I have particularly in mind is that if we enact the pending so-called Paige Bill we would meet retaliatory legislation from other countries. That Bill picks out a certain branch of chemical industry in which compulsory working of patents is to be introduced, and exempts the rest of the chemical industry, and in fact all the other industrial activities of the United States. It is unreasonable to expect that other countries would not at once pass similar laws and, instead of limiting them to that particular branch of chemistry that this Bill has selected, would select some industrial branch in which we are supreme and would shut our products out of their markets or would force our industries to go to those countries and establish plants there for the manufacture of their patented products.

When Great Britain passed its compulsory working act, Germany made her compulsory working act sharper against Great Britain, and so did France; then Great Britain raised the barrier. Germany and France did the same. Each was hurting the other as well as itself, and in view of the very best information that I can get, not only by consultation of public documents but by correspondence with most competent men abroad, it is certain that these laws have not been effective in the direction in which they were expected to be. Judging from the reams upon reams of discussion appearing in the various *Proceedings of the International Association for the Protection of Industrial Property*, every one of the countries that has a working clause would be willing to drop it if everybody else would drop it; one is afraid to drop it and the other dare not. Some countries have made mutual treaties abrogating those laws with respect to each other. There you are! Should we commit ourselves to a policy of legislation which we adopted 83 years ago, rejected 79 years ago, and have since not put on our statute books and with which 56 other nations have been tinkering for over 70 years with an amount of success far less than the effort put forth and the damage incurred?

TEXTILE INTERESTS AND THE PAIGE BILL

The chemists of this country are, or ought to be, particularly interested in the so-called Paige Bill, officially known as H. R. 19187, 63rd Congress, 2nd Session, which was introduced into Congress by Hon. Calvin D. Paige, of the Third District of Massachusetts, October 8, 1914, to force European dye and medicinal makers to come to the U. S. This Bill came up for endorsement on the part of the National Association of Cotton Manufacturers at its meeting in Boston on April 28, 1915, but was not so endorsed. What cotton manufacturers know about the chemical business it is difficult to understand. They have constantly opposed the enactment of suitable tariff provisions so that the dye industry could thrive in this country, on the ground, among others, that it would raise the price of a dozen red flannel undershirts by 25 cents[1] and that dyes were their raw materials, that increase in the cost of dyes would increase the cost of goods to the United States consumer and would interfere with their ability to do export business; that a protective tariff on dyes would simply mean an increased cost in goods with no added public benefit.

The facts seem to be that prior to the tariff of 1882 the cost to the textile people of at least two colors *did* drop,[1] *i. e.*, magenta dropped from $6.50 in 1860 to $2.50 in 1882, and rosaniline blues, which commanded $4.00 in 1881, were sold in this country at $2.50 in 1882. The cause of this drop in prices of these two products is ascribed, by trustworthy sources, to the internal competition within this country between the nine coal-tar dye factories then operating. The textile men have conclusively proven that they guessed things absolutely wrong on the tariff for dyes and that certainly is no qualification for them to offer themselves as infallible or desirable guides or as even probably good guides in so complicated a question as patents. The answer to them is that they have in the past been swayed altogether too much by self-interest; they have disregarded the rights of others and their duties to others; they have run the steam-roller of tariff legislation over our chemical indus-

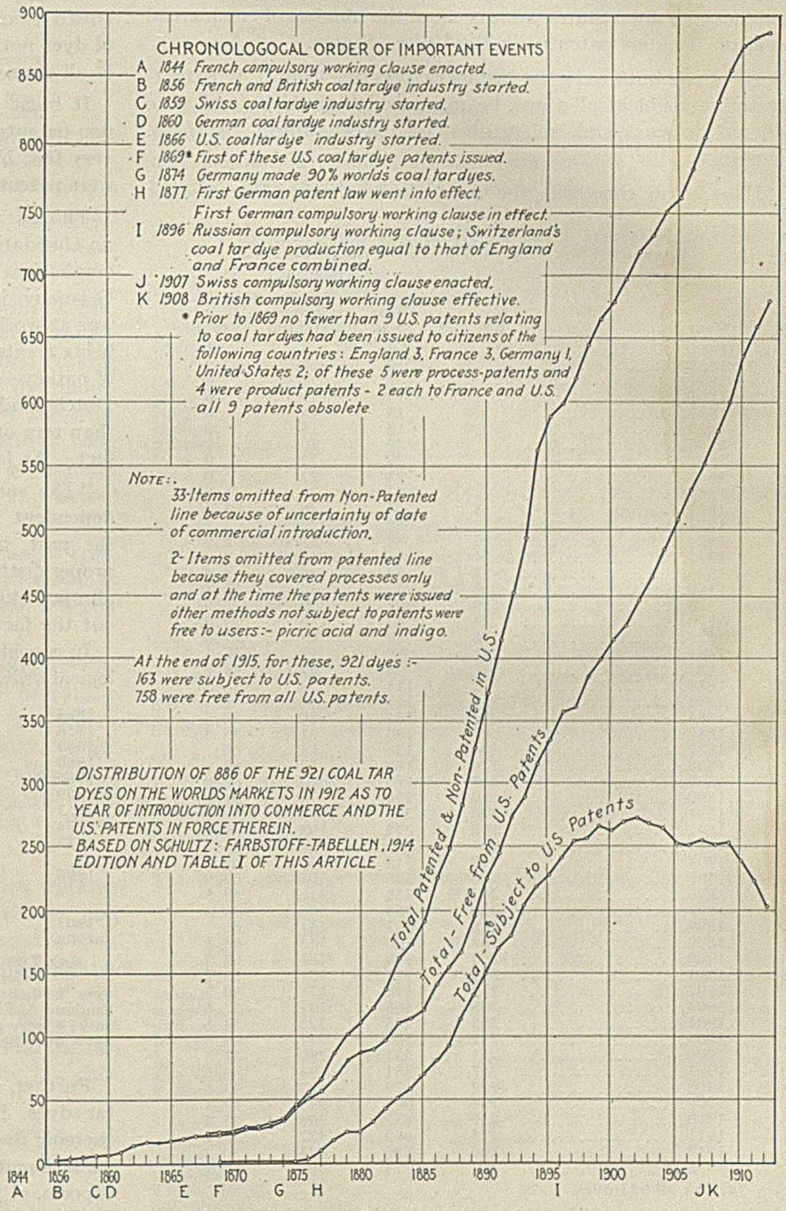

Fig. I

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[1] This Journal, 7 (1915), 694.

[1] This Journal, 7 (1915), 695, 696, 697.

tries and now propose to run the patent-law steam-roller over them as well, and in those circumstances, the chemists of this country must wake up and see to it that their interests in Congress are not further jeopardized by the selfish and short-sighted activities of the textile people.

PAIGE BILL NO HELP TO AMERICAN COAL-TAR DYE INDUSTRY

Can the Paige Bill help the coal-tar chemists? The answer is: it cannot. The reasons are:

I—At no time in the history of the development of the coal-tar dye industry did the number of commercial coal-tar dyes subject to United States patents equal the number of commercial coal-tar dyes *not* patented in the United States.

II—In all but a very few instances dyes free from U. S. patent-restraint could at all times be made which could be and were offered as successful substitutes for dyes subject to U. S. patents.

III—At no time was the American industry throttled or even handicapped by U. S. patents held by foreigners to such an extent that it could not offer successful substitutes for the great majority of patented articles; the American industry always had at its disposal means of getting into the market and which were free from U. S. patent-restraint, *provided* that industry was strong enough to get into the market.

TABLE I—COMMERCIAL DYES PATENTED AND NOT-PATENTED IN U. S. YEAR BY YEAR (EXPIRED PATENTS ALLOWED FOR)

Year	Pat.	Free to all	Excess of free to all over patented	No. of patented per 100 free to all
1856	...	4	4	0
1858	...	5	5	0
1859	...	7	7	0
1861	...	9	9	0
1862	...	15	15	0
1863	...	18	18	0
1866	...	20	20	0
1867	...	22	22	0
1868	...	23	23	0
1869	1	23	22	4.8
1871	1	27	26	3.7
1873	1	30	29	3.3
1874	1	34	33	2.9
1875	1	44	43	2.3
1876	3	52	49	5.8
1877	9	58	49	15.5
1878	19	68	49	27.9
1879	24	82	58	29.3
1880	24	89	65	27.0
1881	33	90	57	36.1
1882	41	98	57	41.8
1883	52	111	59	46.8
1884	59	115	56	51.3
1885	71	121	50	58.7
1886	82	143	61	57.3
1887	94	155	61	60.7
1888	116	168	52	69.1
1889	133	196	63	67.9
1890	150	224	74	67.0
1891	170	246	76	69.1
1892	180	273	93	65.9
1893	205	289	84	70.9
1894	218	316	98	69.0
1895	228	336	108	67.9
1896	242	358	116	68.9
1897	256	364	108	70.3
1898	258	387	131	66.7
1899	267	400	133	66.8
1900	263	416	153	63.2
1901	270	427	157	63.2
1902	272	448	176	60.7
1903	268	465	197	57.6
1904	265	485	223	54.6
1905	252	509	257	49.5
1906	251	531	280	47.3
1907	257	551	294	46.6
1908	252	579	327	43.5
1909	254	603	349	42.1
1910	239	637	398	37.5
1911	223	660	437	33.8
1912	206	680	474	30.3
1912(*a*)	207	714	507	29.1

(*a*) For all 921 dyes.

These statements are conclusively proven by the data in Table I based upon the 1914 edition of Schultz's "Farbstofftabellen" for 886 of the 921 commercial dyes there listed and for which dates of appearance on the world's markets could be fixed with any approach to reasonable accuracy; of the 35 entries not considered, 33 never were subject to U. S. patents and 2 were subject to U. S. patents, namely, picric acid (expired) and indigo (some still alive).

At the end of 1912, 206 dyes were subject to U. S patents (or 207 adding indigo) and 680 (or 714 adding picric acid and the 33 others above referred to) were then free from all U. S. patent-restraint to U. S. makers.

Limiting further discussion now to these 886 dyes out of those 921, the Table I speaks for itself; under the column "Pat." are given the numbers of dyes subject to U. S. patents for the year given (all patents 17 years old being counted as lapsed); under the column "Free to all" are given the number of dyes not subject to U. S. patents for the year given *inclusive* of all dyes whose U. S. patents had then lapsed.

It must be remembered that the intermediates used in the non-patented dyes were not patented in the U. S. and that very few, if any, of the intermediates used in the patented dyes were patented in the U. S.

This 1912 situation is visualized in part in Fig. I, which needs no elucidation. It shows the rapid growth of the business and emphasizes that whatever brought the coal-tar dye industry to Germany, it was not the U. S. patent system. Something else was at work—the market was being developed and the industry being created. Both these things were being done by a number of nations—Swiss, British, French, Germans, Austrians, Italians, Dutch and Americans, but the Germans succeeded better than any or all of their competitors. There is no blinking that fact. As far back as 1887, Otto N. Witt[1] wrote as follows:

> "The success of a coal-tar dye factory is to-day no longer dependent upon the careful guarding of factory secrets as in the past, but upon a systematically arranged plant and the proper distribution therein of the work to be performed and above all upon skillful commercial management, both within and without the factory."

In considering this diagram, however, the following chronological order of events must not be lost sight of:

1844	French compulsory working clause enacted.
1856	French and British coal-tar dye industry started.
1859	Swiss coal-tar dye industry started.
1860	German coal-tar dye industry started.
1866(*a*)	U. S. coal-tar dye industry started.
1869(*b*)	First of these U. S. coal-tar dye patents issued.
1873	French branch factories of German dye-works erected.
1874	Germany made 90 per cent of world's coal-tar dyes.
1877	German compulsory working clause enacted; first German patent law.
1896	Russian compulsory working clause enacted; Swiss coal-tar dye production equal to that of France and England combined.
1907	Swiss compulsory working clause enacted.
1908	British compulsory working clause effective.

(*a*) THIS JOURNAL, **7** (1915), 695.

(*b*) Prior to 1869 no fewer than 9 U. S. patents relating to coal-tar dyes had been issued. Classified according to citizenship of owners: England 3, France 3, Germany 1, U. S. 2; five were specific process patents only; 4 were product patents, 2 each to France and to the U. S.; all of these have since become obsolete.

Further, in 1912, Germany exported $51,545,326 of coal-tar dyes, intermediates and crudes in excess of her imports thereof; Switzerland had a corresponding excess of $3,794,898, while Great Britain had the reverse, an excess of imports over exports, of $6,275,775.[2]

A most liberal estimate of the production in 1912[3] of all coal-tar dyes for those five countries is as follows:

Germany	$68,222,846
Switzerland	6,452,651
England	5,982,676
France	5,000,000
United States	3,750,000

In 1896 Switzerland (eleven years *before* it had a compulsory working clause and 37 years *after* it started its coal-tar dye industry) produced $3,200,000 worth of coal-tar dyes,[4] *i. e.*, as much as France and England together in that year. French

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[1] *Chemische Industrie*, **1887**, p. 215.

[2] THIS JOURNAL, **6** (1914), 1015.

[3] *Ibid.*, **6** (1914), 1017.

[4] *Ibid.*, **6** (1914), 1026.

figures for 1897 and later are not accessible, but France's production is smaller than that of Switzerland.

Finally, Germany produced:[1]

Year	Alizarin	All other coal-tar dyes
1874	\$3,000,000	\$ 3,000,000
1878	\$6,250,000	\$ 3,750,000
1882	\$8,750,000	\$ 3,750,000
1890	\$6,250,000	\$10,000,000

The cause of this slow increase of values is the great price-reduction per unit of dyestuff; the 1890 unit-values are barely 40 per cent of the 1879 unit values. Germany's excess of exports over imports[1] is as follows:

Year	Anilin oil and salt	Anilin, etc., dyes	Alizarin 10 per cent waste
1876	175 tons	175 tons	Not known
1880	345 tons	1640 tons	5871 tons
1885	1241 tons	4213 tons	4145 tons
1890	3411 tons	6695 tons	7892 tons
	\$1,706,000	\$8,656,000	\$3,157,000

The 1890 German export excess totals \$13,519,000; the corresponding figure for 1912 is \$51,545,326, or very nearly fourfold.

The above productions are 90 per cent of the world's production[2] for the years shown. Therefore, Germany in 1874—three years before she had a patent law or a compulsory working clause, fourteen years after she started the coal-tar dye industry, and 18 years after this industry started in France and England—had even then outstripped all of her competitors combined, by 9 to 1, and that in spite of France having a compulsory working clause from 1844 (12 years *before* this industry started) down to the present.

This state of affairs certainly cannot be charged up to compulsory working laws, and, therefore, those who attempt to force compulsory working in the U. S. will have to find a better excuse than have the advocates of the Paige Bill.

It is perfectly obvious and self-evident that the value of the markets in the 207 dyes still patented in the U. S. in 1912 is very much smaller than the value of the markets in the 714 dyes then free from any and all U. S. patent-restraint. Since 1912 U. S. patents on 44 further dyes out of the above 207 have expired, leaving 163 dyes of these 921 subject to U. S. patents, or about 4.5 free dyes for each dye subject to U. S. patents.

Under this state of facts it is clear that the French working clause did not keep the coal-tar dye industry in France and that the German working clause did not create or retain the coal-tar dye industry for Germany, nor did it create the coal-tar dye industry for Switzerland; neither can the U. S. patent system be charged with Germany's present-day success because in 1868 (12 years after this business started) the U. S. had the advantage over Germany in this respect (see foot-note (*b*), page 968 and Table V).

For the purpose of this discussion, the time from 1856 to 1912 (both inclusive) has been divided into three periods:

I	1856–1882
II	1883–1896
III	1897–1912

The first period corresponds to the time when the U. S. industry had a protective tariff high enough to enable it to compete with Europe.

The third period was chosen to show approximately the course of events during the past 17 years and represents the conditions during the life of those patents now (1915) about to expire and issued from 1897 onward; overlapping earlier patents are not considered, and no account of dyes introduced in 1913, 1914 and 1915 could be taken since that information is not accessible except at prohibitive labor.

The commercial products whose patents issued in each of these three periods and those non-patented commercial products of each of these three periods is given in Table II (no allowance for expired patents).

Table II

	Patented	Not-patented	Totals
1856–1882	41	98	139
1883–1896	225	236	461
1897–1912	187	99	286
Totals	453	433	886

In Table III is shown the number of patents taken out in these three periods by the different countries and also the number of non-patented dyes invented and exploited by these countries together with their respective totals as shown.

Table III—Showing Patented and Non-Patented Dyes for Each of the Three Periods as Stated

Total 886 dyes—no allowance for expired patents

Country	1856–1882 Pat.	1856–1882 Not-pat.	1883–1896 Pat.	1883–1896 Not-pat.	1897–1912 Pat.	1897–1912 Not-pat.	Totals Pat.	Totals Not-pat.	Grand Totals
Germany	38	53(*a*)	159	202	122	74	319	329(*a*)	648(*a*)
Switzerland	0	9(*b*)	48	11	56	19	104	39(*b*)	143(*b*)
England	1	18(*c*)	11	12	2	2	14	32(*c*)	46(*c*)
France	2	27(*d*)	4	3	3	1	9	31(*d*)	40(*d*)
Italy	0	0	0	1	0	2	0	3	3
Holland	0	0	0	1	0	1	0	2	2
Austria	0	0	1	3	0	0	1	3	4
U. S.	0	0	2	3	4	0	6	3	9
	41	107(*e*)	225	236	187	99	453	442(*e*)	895(*e*)

(*a*) 7 simultaneously with 2 or 3 other countries.
(*b*) 2 simultaneously with 2 or 3 other countries.
(*c*) 2 simultaneously with 2 or 3 other countries.
(*d*) 6 simultaneously with 2 or 3 other countries.
(*e*) Less 9 for simultaneous invention by 2 or more countries.
Note—Differences in totals are accounted for by the above foot-notes.

In Table IV are given the distribution of the patented and non-patented 886 dyes over the 17 chemical classes, no allowance being made for expired patents. This table shows at a glance that except for two, namely, auramines and indophenols, each chemical classification had at all times members free from U. S. patent-restraint; in the majority of the other classifications the non-patented dyes were very well provided for as against the patented dyes.

Table IV—Chemical Distribution of Patented and Non-Patented Dyes

No Allowance for Expired Patents. (Total 886 Dyes)

Dye class	1856–82 Pat.	1856–82 Not-pat.	1883–96 Pat.	1883–96 Not-pat.	1897–1912 Pat.	1897–1912 Not-pat.	Totals Pat.	Totals Not-pat.	1912 Totals
Nitroso	..	2	1	1	...	..	1	3	4
Nitro	1	1	...	...	1	..	2(*a*)	1	4
Stilbene	..	..	2	5	3	..	5	5	10
Pyrazolone	..	..	1	1	6	4	7	5	12
Azo	24	43	137	165	43	37	204	245(*b*)	462
Auramines	..	..	2	...	...	..	2	...	2
Triphenyl- and diphenylnaphthylmethane	3	17	20	16	9	4	32	37(*c*)	72
Xanthone	..	18	8	5	3	1	11	24	35
Acridine	..	1	3	2	...	2	3	5	8
Quinoline	3	1	...	...	...	..	3	1	4
Thiobenzenyl	..	..	3	2	...	..	3	2	5
Indophenols	1	..	...	...	...	..	1	...	1
Oxazine and thiazin	2	1	16	12	13	3	31	16(*d*)	48
Azine	2	8	11	15	...	..	13	23(*e*)	38
Sulfur	..	1	8	2	21	19	29	22(*d*)	52
Anthraquinone and related dyes	5	5	13	9	52	23	70	37(*f*)	116
Indigos	..	..	...	1	36	6	36(*a*)	7(*g*)	48
Totals	41	98	225	236	187	99	453(*e*)	433(*h*)	921

(*a*) Add 1 for patented but uncertain date of introduction.

(*b* to *h*) Add for non-patented but uncertain date of introduction: (*b*) 13; (*c*) 3; (*d*) 1; (*e*) 2; (*f*) 9; (*g*) 5; (*h*) add 33.

U. S. COAL-TAR DYE PATENTS—PRIOR TO 1883

Before the end of 1882, 138 U. S. patents for coal-tar dyes had issued. Table V gives details, as shown, for them.

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[1] Wichelhaus, "Wirthschaftliche Bedeutung Chemischer Arbeit," pp. 33 and 34.

[2] "Guide Book for the Exhibition of the Chemical Industry of Germany at the World's Fair, Chicago," **1893**, p. 68.

Table V—Details of 138 Patents Issued Prior to 1883

P = Product patent M = Method or process patent

	France		England		Germany		U. S.		
Year	P	M	P	M	P	M	P	M	Totals
1861	..	1	...	...	..	..	..	...	1
1863	..	...	...	1	..	..	..	...	1
1864	..	...	...	1	..	..	..	...	1
1865	..	...	...	1	..	1	..	...	2
1866	1	...	...	...	..	..	..	...	1
1868	1	...	...	...	..	..	2	...	3
1869	..	1	...	...	..	2	..	...	3
1870	..	...	...	...	..	..	..	...	0
1871	..	...	...	...	..	..	..	...	0
1872	..	...	...	1	..	..	..	...	1
1873	..	...	...	...	..	..	..	...	0
1874	..	...	...	...	..	2	..	...	2
1875	..	...	...	...	..	..	..	...	0
1876	..	...	1	...	..	..	..	...	1
1877	..	1	...	2	..	1	..	...	4
1878	1	...	...	...	5	..	..	...	6
1879	3	...	...	...	3	..	7	...	13
1880	1	...	...	...	11(*a*)	..	..	...	12
1881	1	...	2	...	23(*b*)	2	1	...	29
1882	..	2	...	3	31(*c*)	9	13	...	58
Totals	8	5	3	9	73	17	23	...	138
Per cent of total	5.8	3.6	2.2	6.6	52.9	12.3	16.7	...	...
Total per cent by countries	9.4		8.8		65.2		16.7		
Stayed until 1912	0	0	1	0	28	2	0		31(*d*)
Efficiency—per cent	0		8.3		33.3		0		

(*a*) 7 for synthetic indigo all obsolete.

(*b*) 11 for synthetic indigo never commercially useful.

(*c*) 7 for synthetic indigo.

(*d*) Covering 33 different dyes still commercial in 1912; for 8 of the 41 dyes on the markets in 1882 the U. S. patents did not issue until *after* 1882.

UNITED STATES A POOR SCORER

In the language of the baseball score, at the close of 1882:

	At bat	Runs
France	13	0
England	12	1
Germany	90	30
U. S.	23	0
Total	138	31

Umpires—World's Markets and Dye Users

Therefore, it appears that each of these four countries made many wrong guesses as to the dyes suitable for exploitation and which stayed on the markets until 1912. On that basis France and the U. S. each made no score, England made 1 score, Germany made 30 scores. In percentages, the efficiency of "guessing the market" is, France and U. S. each zero; England 8.3 per cent; Germany 33.3 per cent.

Everybody knows the multitude of different reasons that are generally given for the failure of the home-team to win the pennant; and after it is all over the real sportsman says: "The best team won." Why should this case be an exception?

Whatever may have been the reason why "our team" did not win, the Paige Bill could not have helped it win. Many a ball-team has played poor ball for lack of proper home-support.

From Table IV it follows that the U. S. received outright and as a free gift and free from any and all U. S. patent-restraint 466 (433 and 33) dyes out of 921 products on the world's market in 1912; from Table I it follows that of the 455 dyes once patented in the U. S., 247 had become free from all patent-restraint by the end of 1912. By the end of 1915, 44 more patented dyes will have become free, making 291, or 64.2 per cent, of the 455 commercial and once patented dyes of 1912 free from all U. S. patent-restraint. Therefore, at the end of 1915 the condition for these 921 is:

Free to all	758 or	82.3 per cent
Patented	163 or	17.7 per cent
Total	921	100.0 per cent

The condition at any year is shown in the diagram. From Table III it follows:

For 886 of the 921 dyes on the markets in 1912:

I—The U. S. originated but 9, patenting 6 and not-patenting 3.

II—Germany patented 319 and did not patent 329.

III—Switzerland patented 104 and did not patent 39.

IV—England patented 14 and did not patent 32.

V—France patented 9 and did not patent 31.

The 35 dyes not contained in Table III, distributed as to originating countries, are as follows:

	Germany	Switzerland	England
Patented	2	..	..
Not-patented	26	6	1

Therefore, Germany, France and England each treated Switzerland and the U. S. with greater liberality than the latter did the former.

For the 921 commercial dyes of the Schultz "Farbstofftabellen" the total number of patents taken out in the four principal countries is as follows:

Germany	810	United States	487
England	524	France	399

From Table V it follows that while the U. S. did take out some patents it did not succeed in "picking any winners" that lasted till 1912.

From all this it irresistibly follows that there is nothing in the history of the development of the coal-tar dyes industry that could have been cured at all by compulsory working, which failed so utterly in France, Russia and in England.

This is reinforced by the history of this industry as follows:

GERMANY'S FIGHT FOR SUPREMACY

The following (in translation) from the pen of Otto N. Witt, of Berlin, in the Introductory Chapter to the "Sammel-Ausstellung der Deutschen Chemischen Industrie" for the Paris Exposition of 1900 (pp. 5 and 6) bears on this point:

"The almost simultaneous discovery of the first two aniline dyes, Mauvein by Perkin and Anilin-red by Natanson, occurred in 1856; the first practicable manufacturing process for Anilin-red was worked out by Verguin in 1859. Immediately and almost simultaneously the new chemical industry of artificial dyestuffs arose in both France and England. Seldom has any new industry come into being with such brilliancy and such immediate success as has this industry. To none as to this industry has been spared the reaction, after the first access of enthusiasm has died out. Its development is a series of triumphs which continue even to-day after an existence of more than 40 years.

"Germany contributed to the rise of this industry since one of its greatest chemists, August Wilhelm Hofmann, helpfully participated at the time of its introduction and development. But only with hesitation did Germany decide to take up this new industry. And yet Germany was particularly well prepared for such an industry; for only one step was needed to pass from the manufacture of fine chemicals, which had steadily progressed since its early introduction into Germany, to the dyestuff industry. Experience gained in the one could very profitably be translated to the other field; Germany's surplus of well-trained help could not find better employment than in an industry which was to flourish upon a foundation so complicated and theoretical.

"If Germany, nevertheless, allowed years to pass before she turned to the production of artificial dyestuffs the reason was probably that here likewise the fundamental economic conditions for the industry were more favorable abroad, and particularly in England, than at home. Not only were the necessary auxiliary substances, such as fuel and the products of the industries of acids and alkalis much cheaper in England, but Germany lacked also particularly the raw material—tar and its distillation products—because the gas industry was here not nearly so well developed as in England and in France."

So does the following from Heinrich Caro's lecture on the development of the coal-tar dye industry[1] (in translation):

"This industry, which arose in England and France, has finally and after a mighty struggle found its principal home in Germany, and that in spite of our being in an unfavorable geographical location and far removed from the source of raw materials and the consumers of the products of this industry and hampered

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[1] *Berichte*, **1892** (3), p. 956; see also *Jour. Soc. Chem. Ind.*, **1885**, p. 436; *Jour. Soc. Arts*, **1886**, p. 759; *Jour. Soc. Chem. Ind.*, **1886**, p. 351 for more reasons.

as we are on many sides through unfavorable foreign tariff and patent laws."

Finally, the following from Sir William Henry Perkin,[1] the founder of the coal-tar dye industry, is suggestive:

"And, moreover, who could have formed any idea of the wonderful development of the coal-tar color industry that exists to-day, started from such small beginnings, but which through the researches of an army of scientific men, is now of such colossal magnitude."

In view of all this it is clear that the Paige Bill is no relief whatever for the condition it seeks to cure. On the contrary, it is full of harm to us as a nation and can do us no good.

PAIGE BILL BROADLY EXAMINED

Before proceeding to any detailed discussion of the Paige Bill, I want to say that I consider it to represent an attack upon the best interests of this country, as ill-advised as it is full of pernicious potentialities. We in this country believe that class legislation is intolerable; that being so, what must we think of *sub*-class legislation? The textile people believe that compulsory working is good—for a few of us chemists; in other words, the Paige Bill selects a few of us upon which to experiment with this compulsory working of patents. If compulsory working of patents is a good thing for the chemists, or a few of us, we are certainly willing to share our good fortune with the rest of the country. If it is a doubtful thing or a bad thing, then there is no reason why some of us should be selected for the purpose of "trying it on the dog;" under these circumstances, the whole country ought to step in and make every man take his just share of the risk, and not as this Bill contemplates, enable the textile and other people to stand from under and simply shove us chemists, or some of us, out where we are exposed to injury.

Congressman Paige himself is reported[2] as saying of this Bill at the banquet given in Boston by the National Association of Cotton Manufacturers on April 28, 1915:

"There were those who feared that the Bill I introduced would be applied to all patents, and they were naturally lukewarm, if not actually opposed to it, but the Bill contemplated nothing of the kind. It was introduced solely for the purpose of relieving American manufacturers from the deplorable conditions which existed in September last.

"I think the textile manufacturers of the country should have investigated the merits of the Bill, even if they did not feel warranted in urging its passage. But the manufacturers who ought to have been so deeply interested in this problem in New England got cold feet because it was represented to them that the Bill would be of no avail."

Since compulsory working of textile patents is regarded as bad by our textile interests why should compulsory working of chemical patents be considered good for our chemical interests? If compulsory working is as good as represented by our textile interests why should our textile makers "fear" that the Paige Bill would apply to *their* patents? If *they* "fear" under those circumstances why should chemical manufacturers not do the same? Since some of our textile interests are "naturally lukewarm" or "actually opposed" to any effort to enforce compulsory working of textile patents why should our chemical interests do otherwise with respect to the compulsory working of chemical patents? Why should our chemical manufacturers be so recklessly sacrificed for the benefit of our textile interests? The above quotation throws a convincing light upon the mental attitude of the supporters of this Bill and proves beyond question that compulsory working is in itself a bad thing and as such is instinctively dreaded by those likely to be affected thereby.

Not only is the Paige Bill badly constructed from the point of view of the needs of the chemists, but it is the very poorest kind of law-building; it attempts to inject into a Patent Act the language of a Tariff Act; in other words, it has selected, from a law-building point of view, the entirely wrong tool with which to do its work.

The Paige Bill is bare and unequivocal compulsory working, absolutely devoid of any of the legislative and administrative compromises which the experience of the world has unqualifiedly proven to be absolutely essential; it absolutely ignores the legislative experience of 57 countries for the past 80 years.

PAIGE BILL REVERSES TWO LONG-ESTABLISHED U. S. POLICIES

The U. S. Patent Act of April 10, 1790, did not specify "composition of matter" which is the basis of our product claims, among the patentable subject-matter; this Act was repealed February 21, 1793, and "composition of matter" was then made patentable and has remained so ever since, or for 122 years.

The U. S. Patent Act of July 12, 1832 (repealed July 4, 1836, 79 years ago) provided for compulsory working of all U. S. patents to be held by foreigners;[1] no such legislation has since been passed.

Those who wish to bring about such fundamental reversals in our policies of such long standing surely must sustain the burden of showing positive advantages and the sponsors of compulsory working have utterly failed to do so.[2]

The past has taught nothing to the advocates of this Bill.

SIXTEEN CRITICISMS OF PAIGE BILL

Without attempting a minute and exhaustive analysis of this Paige Bill, it is sufficient to point out in sketchy fashion the following 16 directions in which it is open to serious criticism:

1—The things to be affected by this Paige Bill are "any drug, medicine, medicinal chemical, coal-tar dyes or colors, or dyes obtained from alizarine, anthracene, carbazol and indigo." These things are to be deprived of the right to a product patent, to a generic process patent and are to be limited to *the* process of manufacture specifically described and no other; all other products to continue to enjoy the rights thus taken away from these enumerated products.

2—Process patents relating to these designated things must be worked within this country by the patentee or by a licensee inside two years from the issue date of the patent, and the product must be continuously manufactured within this country thereafter throughout the life of the patent.

3—The products of these patents must be for sale at the plant where they are made; they must be sold to all comers without exception.

4—A *citizen* of the United States escapes infringement if he uses the process of the product of a patent covering any of these things, if the patentee fails to observe any of the aforementioned requirements; a *resident* of the United States and *not* a citizen thereof does not so escape infringement.

5—A person who deals in the products of such processes of patents so invalidated is free from suit for infringement; a person who uses that material is not at all exempt from such pursuit and by implication is open to such pursuit.

6—A person using benzol which has been produced from coal tar is put on an entirely different footing from a person who uses benzol recovered from the gas of a by-product coke oven or benzol that may be made by the Rittman process.

To show how these things may work out, it is enough to call your attention to the fact that if Inventor A makes a so-called coal-tar dye from coke-oven benzol he can get a product patent, a generic process patent, and he can do as he pleases with his product and his process; if, however, Inventor B uses coal-tar benzol he *cannot* get a product patent for his new dye therefrom; he cannot get a generic process claim; he can merely have a specific process claim and he *must* operate that process *con-*

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[1] *Jour. Soc. Chem. Ind.*, **1906**, p. 785.

[2] *Textile Manufacturer's Journal*, May 1, 1915, p. 50; *Textile World Record*, May, 1915, p. 126.

[1] This Journal, **7** (1915), 304.

[2] *Ibid.*, **7** (1915), 307–317.

tinuously for the last fifteen years of the statutory life of the patent and he *must* sell at his plant to all comers. It is not only novel but pernicious to attempt to define patentable invention by the *source of the material* employed in carrying out that invention.

7—A man may take coal-tar benzol and therewith invent a new photographic developer or a non-medicinal material or a non-dyeing material which is useful in the arts, obtain a product patent on it, a generic process patent on it, and do absolutely as he pleases with both.

8—If it be contended that the expression "coal tar" as used in the Paige Bill means things that are contained in coal tar, then where do we draw the line? Ammonia is a constituent of coal tar and is obtained from it. Would a dye-product, made with the aid of ammonia, be a coal-tar product *because* of that ammonia? Water is a component of coal tar. Is every dye in whose manufacture water is used a coal-tar dye? These illustrations are enough to show the pernicious effect of the loose, vague, ambiguous and indefinite language of this Bill.

9—Is there any reason why a man who invents a dye from coal-tar benzol should be compelled to have a selling outfit at his manufacturing plant, and a man who makes a photographic developer from coal-tar benzol should *not* have a selling outfit at his plant?

10—Suppose a man invents a new member of the products affected by this Paige Bill, and, in order to protect himself, he proceeds to invent, let us say, five different processes of making that particular new thing; suppose he is operating one of those processes in such a manner that with ten days' production he can supply the needs of the entire United States for the whole year; suppose that only one of these processes is for him a commercially profitable process. Must that man operate his plant 355 days in the year when he cannot sell the output? Must he operate each and every one of those processes throughout the entire year and every day or moment of that year? Must he sell to anybody any amount of a product produced by any one of these processes that a whimsical buyer may specify? If so, it simply opens the door to sandbagging—an unfair attack that certainly cannot appeal to the American people. It takes no great imagination to figure out the pernicious combinations that are possible under this state of affairs. For example, suppose a man decided that A must deliver to him 10,000 lbs. of his product made by his least profitable process; A decides upon a price, which the prospective buyer declines to pay. This Bill says, in effect, that A must sell to B; they cannot agree on the price. Who decides? Or shall B be in a position to browbeat A into selling him the product of a process at a loss?

11—Suppose A and B are competitors. B needs the product in his business that A has invented, just as does A. Suppose B goes to A and insists on buying that product. At what price shall A deliver these goods to B? There is no provision whatever in this Bill for the determination of any such disputes and they are bound to arise. If A's product were not patented he would *not* have to sell to B.

12—Further, suppose A invents a new process of producing a so-called coal-tar dye, by itself, as a pigment and upon fiber. Must he embark not only in the venture of making the coal-tar dye itself, but also of the pigment and of the colored fiber? If A fails to embark in either of these ventures or is unable to obtain a licensee, does he lose his entire patent, and if not, how much and what part does he lose?

13—Suppose A, during the first two years, cannot embark upon the enterprise or is not able to find a licensee. *Must* he lose his patent? If so, this Bill merely puts a prize on prospective licensees to band together, string the inventor along, and when the two years are up, jump in and take for nothing that which they did not create.

14—Suppose an American invents a new and very useful drug derived from animal sources entirely, and a foreigner discovers a way of making that same drug from so-called coal-tar materials. Is this foreigner to be placed in a position to drive the American out of his own market? That is precisely what the Paige Bill makes possible and enthusiastically invites and encourages.

15—Suppose that the inventor does not succeed before the expiration of the twenty-fourth month, in getting his plant in shape, but does during the twenty-fifth month. Is he to be deprived of his patent? If that be so, why should we trouble at all to invent and would it not be easier to let the other fellow do all the work while his time is running against him, interfere with delivery of machinery and the like to him, and when the twenty-fourth month is up, simply rob him of what he has? If this is not putting a premium on piracy, underhanded dealing and double-crossing, there never could be any such inducement. Suppose his plant and stock burn up during the third year and it takes him a week to rebuild, does he lose his patent? If not, why not?

16—This Bill attempts to do away with product patents and substitute specific process patents therefor and abolishes generic process patents. Everybody knows that under the American system, chemical process patents are almost impossible of defense; infringement is difficult to detect and to prove; orders of inspection are very rarely given. If we are to substitute process patents for product patents on the ground that that is the German practice, then we should also incorporate into our system of jurisprudence the German judicial point of view of process patents. While the German claims are in form process claims, yet judicial interpretations have given them the effect of product claims; furthermore, the patentee, upon a reasonable *prima facie* showing, forces the defendant to disclose to three men skilled in the art and acceptable to the Court that which he actually does and the Court then decides whether the thus disclosed matter is or is not an infringement of the patent. For how long does any one suppose the American public would stand for a judicial procedure of this kind? I have yet to see the American chemist who, after fully appreciating this German procedure, was for one instant in favor of abandoning our product claims and product patents for process claims and patents.

PROCESS AND PRODUCT CLAIMS

The difference between our present practice, now 122 years old, and the practice proposed by this Paige Bill may be fairly illustrated as follows: A prospector locates a valuable gold mine after much wandering and traveling; he stakes and registers his claim; that gold mine is his and all are trespassers who enter upon his claim by whatever route. The Paige Bill proposal would have it that a person reaching that staked and registered mine by a route different from the trail taken by the first locater has equal rights upon the property with the first locater; this Bill wants to try this departure, new to the U. S., upon a few of us chemists, while the textile people continue to live and work under the old plan. In other words, the Paige Bill ranks a finder of a second trail to a gold mine, that he did not discover, as of equal importance with the man who had the grit to go out and find that unknown gold mine; "claim-jumping" is not regarded as particularly honorable and clean dealing and why should the Paige Bill encourage it? To bring it nearer home: suppose you buy a lot and build a house on it; everybody is a trespasser who enters that lot by any route whatever. The Paige Bill says that any one is *entitled* to full and free use of that lot and house and all its contents equally with you, *provided* he enters the lot by any means other than the front gate which happens to be your customary way of entering your grounds. Can you imagine the disturbance that this sort of a proposition would make among householders? Then why should the chemists of this country tolerate any such treatment?

PAIGE BILL INCURABLY BAD

In short, this Bill opens the way to innumerable avoidable disputes without providing any judicial means of settling them, but leaves it to the individual to work out a set of conclusive judicial interpretations through costly, time-consuming court proceedings. We now have a large body of such decisions based upon our present policy of patent-protection now over 122 years old. To my mind, it is worse than useless to throw away such positive results and send some of us out on another expedition for the same purpose which might require fifty years to accomplish and with every reasonable prospect of not hitting the mark at all, and all at the cost of a selected few of us.

There is no question that many of the above specified objections to this Bill can readily be cured by suitable alterations in its language. But what an indubitable index such imperfections are as to the seemingly haphazard and thoughtless manner in which this legislation was conceived and proposed! With so many on-the-surface faults and shortcomings why is there not just ground for well-founded distrust of the more hidden effects of such proposed legislation, so-called "jokers?" No matter what changes may be made in this Bill as to details like those enumerated, the three insurmountable and irremovable objections to it still remain:

1—Class and subclass legislation.

2—Abandonment of a settled judicial policy and substitution therefor of an effort to create a new judicial policy which the world's experience to date shows conclusively will lead to failure.

3—Introduction of a new system of judicial procedure which cannot be otherwise than obnoxious to American principles and thought.

Experience is said to have shown that manufacture within the country is not stimulated but that dumping is. Shall the Paige Bill stimulate dumping? If so, for whose benefit? Any one else than the dye-*users?*

COMPULSORY WORKING IN OTHER COUNTRIES

With the aid of the book entitled "Foreign and Colonial Patent Laws," by W. C. Fairweather (published London, 1910) and other publications, I have compiled the following list of countries having compulsory working clauses and have given the date of enactment thereof, so far as it could be ascertained with any approach to accuracy; as to dates this compilation needs checking up.

Date	COUNTRY	Date	COUNTRY	Date	COUNTRY
1832[1]	United States	1887	Sweden	1902	Nigeria (Northern)
1844	France	1888	India	1902	Spain
(1856	Coal-tar dye industry founded)	1894	Portugal	1902	Transvaal
		1896	Costa Rica	1903	Barbados
1864	Liberia	1896	Negri Sembilan	1904	Rhodesia (Southern)
1869	Peru	1896	Russia	1905	Trinidad and Tobago
1877	Germany	1897	Guatemala	1906	Ceylon
1880	Ecuador	1898	Finland	1906	Leeward Islands
1880	Luxemburg	1898	Granada	1906	Roumania
1880	Turkey	1899	Nicaragua	1907	Switzerland
1882	Brazil	1901	Seychelles	1908	Great Britain
1885	Norway	1902	Bermudas	1908	New Zealand
1885	Uruguay	1902	British Guiana	1908	Panama
				1909	Japan

[1] Repealed 1836.

Those countries whose enactment-dates have not been determined:

Argentine
Australia
Austria
Bahamas
Belgium
Bolivia
Canada
Chile
Colombia
Cuba
Denmark
Hungary
Italy
Iceland
Malta
Newfoundland
Nigeria (Southern)
Orange River Colony
Venezuela

Why do not *all* these countries have coal-tar dye industries? They all have compulsory working laws!

A half-dozen hours' casual reading of the working clauses as set out in this Fairweather publication will convince any one of the futility of the Paige Bill.

COMPULSORY WORKING AN ABSOLUTE FAILURE

In conclusion, the following from Lord Moulton, the foremost patent-counsel of Great Britain and one of Great Britain's highest law officers of the present day with regard to the British Compulsory Working Act of 1907 and uttered by Lord Moulton in 1914[1] after more than 6 years of enforcement of that Act, is pertinent and suggestive:

"Well, gentlemen, so far as the legislation, as it stands, about compulsory working is concerned, I am afraid I am going to use adjectives. I think it is not only mischievous, it is also idiotic. It is one of those cases which are growing to be common, where the layman rushes into legislation with little or no acquaintance with the subject on which he is legislating. Knowledge has gone so far that the layman cannot keep up with the technical knowledge of a subject, even in politics. In my opinion, it would have been impossible for any man who understood this matter to devise the present legislation.

"Now let us take one example, and remember when you are testing whether a thing is right or wrong by an example, you must never take an extreme example; you must take one which fairly represents it. Let me suppose that a man makes an invention and contents himself by taking out an English patent. The invention turns out to be of world-wide utility; it is adopted, we will say, by every civilized nation in the world; but the inventor has no rights abroad: he has rights in England only. Then comes some person and says, 'Your patent is worked mainly abroad!' Of course it is. Big as England is, the world outside is bigger. Then he appeals to this legislation, and claims that the patent must be revoked because it is worked mainly abroad. In this he is justified and the consequence is that an invention that is universally valuable and universally appreciated stands in the eye of the legislation of England, as it at present exists, as condemned to death, and it will be revoked unless the poor inventor can explain how it is that a useful thing is used more abroad than it is in England.

"It is no use arguing about legislation of that kind. It is self-condemned.

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"The mischief that has stirred up people to pass this legislation is that manufacturers, not inventors, stop the use of inventions in this country. Just think of the cases. Do you think that a man who invents a new dye would not like it to be made by Mr. Levinstein as well as everybody else? But it is a different question when a great foreign firm gets it. They do not want to protect the inventor's rights. They want to protect the manufacturer's rights. The consequence is that the whole cause of the outcry is the manufacturer who wants to get a monopoly by means of the rights which we have given to the inventor as a reward for his original thought, and you never find any mischief of the kind that it is supposed we must guard against as long as the patent remains in the inventor's hands. I smile when I hear those who take the opposite side in this controversy talk about the advantage of compulsory working because it brings trade into England and gives work to our working class. Mr. Levinstein is an old friend and an old client of mine, but do you think he conducts his campaign in order to bring the Badische Anilin and Soda Fabrik into England? No, no, no. If you strip the cases of the formal language in which they are put, you will always find that the man who wants to revoke a patent is a manufacturer who wants to prevent the inventor proper from getting the return of his invention. I have said that I never knew any inventor who kept the interest in his invention in his own hands, who was ever unwilling that it should be worked as largely as possible in every country in the world. Occasionally you may find a crank who puts too high a value on his invention, and will let the few years of the patent pass and his invention remain unused, simply because he thinks that the royalties offered are too low, but those cases are so rare that they may be neglected. No, the applications to revoke patents are not for the purpose of increasing the manufacture in England; they are for the purpose of increasing the importation into England. It is this which makes it so important to give to people clear ideas on the

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[1] THIS JOURNAL, 7 (1915), 314, 315.

subject. As you know, it is quite possible that the specious appearance of a movement in favor of English manufacturers (and it can be clad in those words) may bring some attempt to increase the severity of the law. This can be resisted only by showing to the world, first of all, that the movement is always from the manufacturer—the inventor has no interest in limiting the use of his patent—and, secondly, that the aim of all these attempts is to increase importation into England, and not manufacture in England.

"So much for the real substance of the case. What then is the true remedy? The true remedy is that you ought to protect the monopoly of every inventor who behaves reasonably and punish those only who act unreasonably. If an inventor sells his rights to a foreign manufacturer, and that foreign manufacturer uses the English patent to prevent manufacture in England, a manufacturer who is willing to make the article in England ought to be able to get a license to do it. No patent ought to be used for the purpose of checking work in the country itself. But let me point out the dangers that there are in taking a principle like that and using it without knowledge and without caution. There are many things that are best manufactured by one or two firms. The demand is small for them, and one or two firms can well satisfy it. If you threw it open to hundreds of firms you would get no greater benefit to the country. (A voice: 'Less.') Therefore, we have adopted the rule, and I think it a wise one, that the inventor should be left to manage his own patent, fix his own royalties, and get as much from it as he can with his own personal knowledge of the matter. But if he acts so unreasonably that the patent is having the effect of seriously checking working in England, then give the power to the Courts to grant a compulsory license. That is remedy enough, and I should counsel you not to do anything else whatever. If you do you will find that all you are doing is to help people to take the ideas of the inventor without giving any fair return, and to help people who manufacture abroad to import their goods into this country without remunerating the inventor. We do not want to see that, because we desire to see English manufactures increase, for we feel that we have a growing working population. We are glad to welcome new industries to develop in our midst; but, if we try to bring that about by unjust laws, you may be perfectly certain you will do more harm than good, and ultimately you will regret it."

CONCLUSIONS

I—Compulsory working has nowhere been successful, is a gross economic error, increases dumping and does not increase home-production, and all other countries are anxious to find some way of dropping it.

II—The Paige Bill takes no notice whatever of the experience of the world for the past 80 years, proposes to start where the rest of the world, including the U. S., began 80 years ago, and wants to re-prove at the expense of a few of us chemists what six hours' reading of past legislation in other countries would teach, to any one, was and is a colossal mistake.

III—The U. S. rejected one of the Paige Bill proposals in 1793 and the other in 1836; no reasonable proof of advantage for such a reversal as now proposed has been promised, much less offered by the supporters of the Paige Bill.

IV—The Paige Bill was devised for the purpose of forcing European dye-makers to the U. S.; compulsory working had nothing whatever to do with the dye industry development.

V—The Paige Bill as it stands puts a premium on dishonest practices and unfair methods of dealing and invites and encourages such conduct.

VI—Compulsory working should not be adopted by the U. S. and the Paige Bill and all Bills like it should be killed for good and all.

90 William Street, New York City

DISCUSSION

By H. E. Stonebraker

Gentlemen of the Chemical Society:

The request of your committee to have placed before you the other side of the question which Doctor Hesse has so ably presented, places me in an anomalous position, but with your assurance that the following remarks will not be construed as my personal views, or the views of the patent bar generally, I will endeavor to explain the causes for the proposed Bill, and the advantages to be derived, as drawn from my impressions after a study of the Bill in connection with similar acts in foreign countries and the conditions surrounding the much recently proposed patent legislation in the United States.

Before entering into a detailed discussion I want to call attention to several points brought out by Dr. Hesse: *First*, I do not understand that the Bill excludes "generic process patents." It states that patents may be obtained on applications relating to "a definite process," as distinguished from a product, and there is nothing in this to indicate that the Bill contemplates the refusal of a patent on a generic process, since a definite process may be either generic or specific. *Secondly*, the patented product does not necessarily have to be sold at the place where manufactured, as the Bill merely provides that the product shall be "manufactured in the United States by or under authority of the patentee within two years of the granting of said patent, and after the commencement of said manufacture" it is to be continuously carried on in the United States "in such a manner that any persons desiring to use the article may obtain it from a manufacturing establishment in the United States." Under this provision, I do not understand that the patentee shall be compelled to sell at *any* price, but merely that he shall offer it for sale so that it may be obtained by anyone who is willing to pay the required price.

The Paige Bill is divided into two sections, one of which deals with the exclusion of certain patents, except in so far as they relate to a definite process, or, in other words, product patents, whereas the latter part of the Bill deals with the compulsory working feature, which is the more important consideration of the two.

The elimination of product patents will not work any serious injury to an inventor and, in fact, will be of considerable assistance in connection with litigation involving chemical patents, where it is very difficult, and sometimes impossible, to identify a particular product with a patent. It is hard to conceive of a case where an inventor would not be protected as to his product, if a patent is granted to him covering the process employed in making that product, so that the inventor would not suffer except in the unusual case where the product could be manufactured by more than one process. Under such a condition, a generic process patent would afford complete protection, and if the inventor is not entitled to cover a generic process there is no reason why he should be granted a patent covering the specific process which he has developed for producing a certain formula, and every other process that might be developed by others after him, but of which he had no conception. It has been held, as instanced in the case of Downes *vs.* Teter-Heany Development Company, 150 Fed. 122, that where a patent includes claims both for a process and a product of the process, the claims for the product are to be construed in connection with and are limited in scope by the claims for the process and are not infringed unless the process claims are also infringed. In the case of Kuehmsted *vs.* Farbenfabriken, 179 Fed. 701, Hoffman patent No. 644,077, for Acetyl Salicylic Acid, known medically as Aspirin, was involved, and covered the product of a new process. The Court experienced great difficulty in finding any difference between the patented product, old products, and the infringing product and held that "a prior known compound having the same formula is not conclusive evidence of the lack of patentable novelty in a chemical compound," and the patent was sustained on the ground that the product claimed was produced by a new process. So that in reality the invention was for a process and not for a product, and if the patent had been based on the process instead of the product, much labor and expense would have been eliminated in litigating it. In commenting on the invention and the patent the Court said:

"The fact that the formulae are identical cuts little figure. A chemical formula is simply the symbolical expression of the composition or constitution of a substance; as the formula for water is H_2O. Customarily, chemists who intend to produce a combination of two substances write the formula of the product in advance of making it. And, assuming that the formula actually expresses the constitution of the substance chemically, the substance physically, and in consequence, therapeutically, may be widely different, as, for instance, the water of the seas differs in its physical body from the water of certain springs though the chemical formula for water, whether of sea or spring, is H_2O. That is to say, two substances having the same chemical formula may differ widely as to impurities upon qualitative analysis."

The net result seems to be that the real invention in every instance of this class of patents resides in the process and not in the product, and to eliminate product patents would be of no serious consequence to the inventor, and, on the other hand, the requirement that he define his invention in terms of the process might be of great help in cutting down litigation and in enabling him to prove infringement with greater certainty.

Turning now to the compulsory working phase of the bill, a great deal has been said in the numerous discussions of compulsory license and working-laws about what the respective laws fail to accomplish, but nowhere has there been pointed out a specific instance of harm having been done as a result of such laws. The disadvantages claimed seem to be in the nature of generalizations, and the hypothetical cases which are cited to show the bad effects of the laws in operation are usually most exceptional instances. In the talk to which you have listened this evening, three reasons have been assigned as a basis for the statement that the Paige Bill cannot help the chemists. These I will take up in the order given.

(1) Commercial coal tar dyes subject to United States patents have never equaled the number of commercial dyes not-patented in the United States. In answer to this, it can be said that the Paige Bill will not affect in any way the unpatented dyes, and it will insure the manufacture of patented dyes or, as an alternative, the lapsing of the patents.

(2) The second point advanced was that in all but a very few instances, dyes free from United States patent-restraint could be made and were offered as successful substitutes for dyes subject to United States patents. This admits the existence of cases where United States patents stood in the way of open competition, and if any of these patents were pigeonholed, it would certainly be a disadvantage to the public in general, and the Paige Bill would cure just such an evil.

(3) At no time was the American industry throttled or even handicapped by U. S. patents held by foreigners to such an extent that it could not offer successful substitutes for the great majority of patented articles. If this condition continues to prevail there may arise no cases that will require operation of the Paige Bill, but if the Bill did nothing more than to insure the continuance of such a happy industrial condition, it would be worth putting into effect for this purpose, in the absence of proof of any actual harm that might be done by it.

The Bill before us is one of several that have been presented to Congress as a result of a combination of conditions that have arisen in recent years, due in part, as claimed by many, to our patent system, and partly to some of the unfortunate business policies that have grown up with our trusts and larger combinations, and it may be of interest to digress for a moment and look at some of the causes for the general state of unrest surrounding our patent institutions.

A patent monopoly in an invention for a limited period is beneficial so long as the patent is not employed for illegitimate purposes, and the patent evils complained of arise only from the perversion of patent monopolies to illegitimate uses. Such illegitimate use has consisted in securing a monopoly more extensive than that covered by the patent, and by accumulating a number of patents and suppressing part of them. Our present-day trusts have come into existence *after* the enactment of our patent laws, at a time when competition was universally accepted in business and combinations for restraining trade were not dreamed of. Under such conditions a patent monopoly was a healthful stimulation for competition, but as soon as competing concerns attempted to combine for dominating any branch of industry, the patent monopoly was put to a use for which it was not originally intended and resulted in extortionate prices and an arrest of progress.

The inventors' guild, which includes in its membership such inventors as Thomas A. Edison, Peter Cooper Hewitt, Michael I. Pupin and H. Ward Leonard, define in the following language the danger that the country faces:

"It is a well-known fact that modern trade combinations tend strongly toward constancy of processes and products and by their very nature are opposed to new processes and products originated by independent inventors, and hence tend to restrain competition in the development and sale of patents and patent rights; and consequently tend to discourage independent inventive thought to the great detriment of the nation, and with injustice to inventors whom the constitution especially intended to encourage and protect in their rights."

It has been clearly demonstrated from experience that certain patent practices, when pursued in a competitive business way are harmless, and sometimes beneficent, whereas the same practices are attended with serious evils when pursued for the purpose of restraining trade, and it has been a world-wide practice to buy up patents for precluding competition, the harm of which has been recognized and dealt with in almost every nation except the United States. Great Britain, Canada, Germany, France and many other nations provide the government with power to liberate the manufacture, sale or use of all patented articles after a fixed period, which affords the patentee sufficient time for supplying the patented product to the public. In case of his failure to do so, in some countries the patent right is forfeited and the privilege of making and selling becomes common to everyone, while in other countries the owner of the patent by his inactivity brings himself within the terms of a compulsory license law.

There is no question but that patents have been bought up in large numbers in the United States for suppressing competition, and instances can be found in the reports of decisions of Federal Courts. Thus in Columbia Wire Co. *vs.* Freeman Wire Co., 71 Fed. 302, the Court, in referring to one of the parties stated that:

"It has become possessed of many if not all of the valuable patents for the manufacture of barbed wire and machines for so doing."

In the case of Indiana Manufacturing Co. *vs.* J. I. Case Threshing Machine Co., 148 Fed. 21, the Court found that one of the parties had acquired over a hundred patents pertaining to straw stackers.

The National Harrow Company was charged with having acquired eighty-five patents on spring-tooth harrows in National Harrow Co. *vs.* Bement, 21 Appellate Division, New York, 290.

And in the so-called Locks case, 166 Fed. 560, the Court said: "It is a fact familiar in commercial history that patent rights have a commercial value for purposes of extinction; that many patents are purchased in order to prevent the competition of new inventions and of new machines already installed."

Another and more famous case is that involving the Paper Bag patent, 150 Fed. 741, in which the complainant owned two patents covering machines for making paper bags, one of which patents was deliberately suppressed, and on this unused patent suit for infringement was brought. The suit was successful, although in both the Court of Appeals and the Supreme Court of the United States there were dissenting opinions, contending that the complainant, having purchased and suppressed the patent to prevent competition, was not entitled to the aid of a court of equity to enjoin and infringe it.

Other cases may be cited to show the harmful practice of buying up and suppressing patents, the effect of which is to shut out competition and to build up a monopoly of monopolies, equivalent to a patent on the industry, and such a combination is not a monopoly in the beneficial use of a specific invention (148 Fed. 25) but is a monopoly created by commercial cunning in the use of great wealth, and it is to cure this evil and prevent the continuation of such a practice that compulsory working is provided.

Another practice that has grown up is for a vendor of a patented device to place a notice on the product, requiring the purchaser to obtain his unpatented supplies, to be used with the patented product, *exclusively* from the owner of the patent under penalty of his being dealt with as an infringer if he fails to do so, and also, patented articles are sold with notices stipulating a fixed retail price below which the purchaser is forbidden to sell the article. This practice was first recognized in the case of Heaton Peninsular Button Fastener Co. *vs.* Eureka Specialty Company, 77 Fed. 288. The complainant owned a patent for a machine for fastening buttons to shoes by means of metallic staples, and placed a notice on the machines which it sold, to the effect that the purchaser was licensed to use the device only with staples purchased from the owner of the patent on the machine. The staples were not patented. The defendant sold staples to the purchaser who used the same on the patented machine, and the Court held that the act of selling the staples was one of contributory infringement, and enjoined a further sale of the *unpatented* staples.

The most recent upholding of this doctrine was in the case of Henry *vs.* Dick Co., which the Supreme Court decided March 11, 1912. In this later case, the Dick Company made and sold a patented mimeograph with a notice that it was to be used only with ink, paper and other supplies made by the Dick Company. The decision of the Court was that the sale of ink by Henry, who knew of the restriction, to the owner of the mimeograph for use therewith was contributory infringement.

The restriction as to resale price was however invalidated by the Supreme Court in the more recent case of Bauer & Co. *vs.* O'Donnell, decided May 26, 1913. According to the facts in this case, the complainant prepared and sold a compound made according to a patented formula with a notice stating that the contents of the bottle was patented, and that it was sold under license whereby the purchaser must retail it at not less than one dollar, and that anyone selling it for a less price or using it when so sold would be prosecuted for infringement. A druggist in Washington sold a quantity of the patented goods at a cut price and was sued for infringement, and the Supreme Court, by a five to four decision, declared that there had been no infringement of the patent, and while the court endeavored to distinguish this case in principle from the earlier case of Henry *vs.* Dick, involving the mimeograph they have failed to convince many persons that any distinction really exists that should cause the application of different principles to the two cases. I will not endeavor to reconcile these cases, and do not care to enter into any discussion as to whether the Supreme Court was right or wrong in the decision in either case, but am citing them to draw attention to the changed order of things that business conditions have brought about, whereby the highest court in our land has been influenced to decide by a most narrow margin in favor of one doctrine that has prevailed practically since the establishment of our patent system, and to throw down utterly another doctrine closely analogous. Something must be wrong with our laws that needs remedying, so that the public will at least be in better position to judge of the probable result in any case, and so that the people may not be forced to suffer individually or collectively under an illegal use of patent monopoly at the hands of any combination, built up directly or indirectly, for the purpose of restraining trade and competition.

I have here a statement of the working laws in the important civilized countries which may be of interest, but I will not burden you with reading this entirely. The laws in Germany and Great Britain are practically the same and require working only in the event that the patented article or process is manufactured or carried on exclusively or mainly outside the country. Only three applications for revocation of a patent on the ground of non-working were filed in Great Britain in 1912, and only one patent was revoked during the same year, and this was by consent of the patentee. A second application was withdrawn, while in the third case the patent was revoked in 1913.

During 1913, according to the official notice, eight applications for revocation were filed, of which seven were withdrawn, and only one patent was revoked, and this by consent of the patentee. Considering that more than a hundred thousand patents were in force in England on 1913, the figures above indicate that manufacturers are not in any way prejudiced by the working act.

One of the chief objections to a compulsory working law is that inventions are not suppressed, and that the supposed evil is fanciful and has no reality, but if there are no instances of suppressed inventions, and the proposed law is directed to an imaginary evil, it is strange that opposition should be made to it when it should affect no one according to this argument.

Let me read you some extracts from what Mr. Walter Reid, Chairman of the Institute of Inventors of Great Britain, had to say before the Imperial Industries Club on April 1, 1914.[1]

"We for a very long time had no compulsory working; we had allowed foreign inventors here to acquire monopolies in certain branches of industry and they had made use of those monopolies in a way which those who were acquainted with the details of the subject could no longer permit Great Britain to labor under the disadvantage of. On the occasion when we had the last deputation to Mr. Lloyd George a number of cases and details were given of industries that had suffered in Great Britain because foreigners had been granted monopolies here and had not worked the patents in this country, but, having the monopoly, they could demand from our own people prices which they at home could not obtain. A very familiar case was that of the alizarine industry, where our manufacturers here who had to use such dyes were paying about half a crown, and the practical real value of the material was about seven pence. Of course, our manufacturers were suffering there under a great disability as regards foreign competition because they required that dye, it was a dye which was necessary and the wool dyers in Bradford and elsewhere had those excessive prices to pay whereas their German competitors had not.

Now, I think, if we consider that, it is a clear case where the British industry is handicapped and unfairly handicapped; and I would ask you to bear in mind that the first and original idea of a patent was a monopoly for the introduction of an industry into this country, and it was only at a later stage that the inventor was given a monopoly for the produce of his brains.

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[1] See THIS JOURNAL, 7 (1915), pp. 307–308.

Nobody will deny that an inventor ought to have something for inventing a thing; otherwise we should have no inventors in this country; they would go elsewhere. But I do think, myself, and, so far as I have been able to learn, the opinions of inventors generally are, that it is an unfair thing to British inventors that a foreign inventor should come here and acquire a monopoly and not do what the British inventor in most cases has to do from the very nature of the subject, namely, work the invention. I think that we, as an Imperial Industries Club, ought to see to it that the British inventor is not, at any rate, worse off than the foreign inventors. He cannot in many cases acquire foreign patents abroad in the same way that a foreigner can acquire a patent here because there is compulsory working.

"Now, as a consequence of that Act during the time when, if I may use the word, it was active, several firms abroad came and established factories in this country and a number of factories have undoubtedly been established under the pressure of the Act as we interpreted it. It is not enough to say that one firm or another has put up a factory in England, and they are not doing much, that they have done it just simply as a blind, and so on; that is not so much the question; those who are behind the scenes know very well that a number of patents have been introduced into this country under royalty to British firms that would not otherwise have been introduced. When an industry is introduced into this country, whatever it may be, there is employment for British capital, there is employment for British workmen and the industry becomes established in this country and after the lapse of the patent the industry becomes an English one."

The same reasons apply in the United States that apply in England in favor of compulsory working. The evil of illegal monopolizing of patents exists beyond a doubt. Only several days ago a decision was handed down in the United States District Court for Eastern Pennsylvania by Judge Dickinson against the Motion Picture Patents Company, the General Film Company and many other concerns and individuals manufacturing moving picture films and accessories or controlling the rights of their manufacture, and by this decision the so-called moving picture trust has been dissolved. The Court discusses the rights of the holders of patents in the following language:

"It is the right of a patentee through having the exclusive sale of the patented article to control and in that sense to monopolize the trade in it. It is wrong by an illegal restraint of trade to monopolize it or any part of it."

"We feel constrained on the authority of this case alone, to find that the agreements and acts of the defendants in the present case went far beyond what was necessary to protect the use of the patents on the monopoly which went with them and that the end and result which would be expected to be and was accomplished, was the restraint of trade condemned by law."

Let me call your attention to the fact that the entire world-wide industry of moving pictures and film manufacture has been dominated by probably less than ten men, and were it not for the obstruction which the United States has placed in their way under the Sherman Law, it is not improbable that in a little while the public would be compelled to pay one and two dollars for moving picture exhibitions that now cost five and ten cents. There should be some other way to prevent this practice than the slow and difficult procedure, responsibility for carrying on which rests with the United States Department of Justice, and a compulsory working law would in a large measure prevent the evil, as it would compel a controlling company either to operate under all of its patents or else lay the patents open to public competitive operation and under the present existing conditions it is submitted that the United States people as an entirety would derive benefits from a compulsory working system that would more than counterbalance any bad effects that might be found to accrue therefrom.

Wilder Building, Rochester, New York

DISCUSSION

By B. C. Hesse

Whether the language of the Bill in respect of "definite process" excludes generic processes and in respect to "obtain it from a manufacturing plant" is or is not mandatory, is open to question. Under the stress of litigation, on the Bill as it now stands, it might very well be that the quoted language would be stretched to the limit to curb generic process claims on the one hand and to enforce sale of goods made by patented processes in the U. S., on the other hand. Practice under the Rules of the U. S. Patent Office would favor limiting "definite process" to "specific process," thus excluding "generic processes." Under these conditions the only safe course for the chemists of this country to pursue is to assume the, to them reasonable, adverse extreme of the purport of the Bill and its language and the conditions that wou'd likely thereunder arise.

In view of the doubtful and limited protection afforded by U. S. chemical process-patents and in view of the way in which Germany, England and France have each stretched chemical process-patents into product-patents there is no doubt that the chemists of this country would be greatly hampered and harrassed by elimination of product-patents, as such. If a product-patent does not contain tests for the certain identification of its product and for its differentiation from all other like substances, then that is due to improper and defective patent-drafting and is not a defect in the patent-law, as such.

As to specific instances of harm done by compulsory working: while the summing up by Lord Moulton:

"No, the applications to revoke patents are not for the purpose of increasing manufacture in England; they are for the purpose of increasing importation into England,"[1] is a general statement, yet it is so clear and authoritative in its statement of final result and outcome as to satisfy any reasonable demand for specific effect. In an endeavor to get specific and precise instances I addressed letters in May of this year to two of the supporters of the Compulsory Working Act under discussion at the Symposium of the Imperial Industries Club[2] requesting them to send me specific instances of benefits and directed their specific attention to their own generalized statements of benefits; to these no reply has been received; I also sent at the same time a like letter to one of these opponents of Compulsory Working and asked him for specific instances of harm—no additional information was received. In addition to Lord Moulton's statement, just referred to, are the statements of Gevers in my paper of this evening and those of A. J. Walter,[3] H. A. Colefax[4] and of J. Hunter Gray;[5] as against these set the statements of Walter F. Reid,[6] of Alexander Siemens[7] and of Ivan Levinstein[8] for specific benefits and compare them with the statement of actual results achieved.[9] The balance is not in favor of the supporters of Compulsory Working. In reading the quotation from Mr. Reid as given by Mr. Stonebraker, above, it must be remembered that commercial forms of process of making alizarine were patented separately in England by Perkin, of England, B. P. 1948 of June 26, 1869 and by Caro, of Germany, B. P. 1936 of June 25, 1869; that the British Alizarin Company was originally based upon the above Perkin Patent.

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[1] This Journal, 7 (1915), 314, 2d. col. last half and top of p. 315.

[2] *Ibid.*, 7 (1915), 307–317.

[3] *Ibid.*, 7 (1915), 311–312.

[4] *Ibid.*, 7 (1915), 313.

[5] *Ibid.*, 7 (1915), 316.

[6] *Ibid.*, 7 (1915), 307–309.

[7] *Ibid.*, 7 (1915), 310.

[8] *Ibid.*, 7 (1915), 305.

[9] *Ibid.*, 7 (1915), 306–307.

Compulsory working will *not* force manufacture of coal-tar dyes in the U. S.; France, Russia and England have all and long ago found that out.

A law like the Paige Bill will in no wise influence the U. S. coal-tar dye market. On the other hand it would discourage any American inventors from attempting to break into the business because the American is not yet industrially equipped to compete with foreigners and when he does become so equipped he will recognize in legislation like that of the Paige Bill, an enemy and not a friend.

Whether compulsory working would overcome the improper acquisition of patents and concentration of their ownership is something into which I have not looked with any detail; however, the habitual concentration of chemical patents (other than coal-tar dyes) in a few hands in Great Britain and in France and its comparatively rare occurrence in Germany does not, at first sight, seem to augur well for the success of compulsory working in that direction; having failed in so many different directions and in all directions for which glowing success was confidently predicted,[1] there does not seem to be any real ground to hope that compulsory working would effectively help the Sherman Law or the Clayton Law.

APPLIED CHEMISTRY[2]

By L. H. Baekeland

It is only three years ago that a Brooklyn alderman, who, in the absence of the Mayor of New York, had to welcome the visitors to the International Congress of Chemistry, addressed them as if they were druggists or pharmacists.

After all, he made not much greater a mistake than many so-called educated men who obtained a B.A. and yet are ignorant enough of elementary scientific knowledge to imagine that the main occupation of a chemist is to analyze substances and detect falsifications.

Even in England, a pharmacist is currently designated as "chemist," while a real chemist is called an "analytical chemist."

But the European war has done much to correct some of these mistaken notions of the public at large. Our daily press has to a certain extent acquainted this country with the fact that in our national make-up, there are such things as chemical problems. I doubt, however, whether the unthinking masses have begun to realize that aside of the so-called chemical industry, practically every other industry, in fact, every enterprise, has chemical questions to contend with, and that chemical industry itself is intimately interwoven with the great network of every modern industrial or agricultural condition; that the economic welfare of our country, and the health of its citizens is largely dependent on the way we utilize our chemical knowledge.

CHEMICAL WARS

The present war has been aptly called a "chemical war," because efficient work of every department of the fighting armies, from the Red Cross service to the manufacture of guns and explosives, involves incessantly chemical knowledge and—still more chemical knowledge.

But do not imagine that this is the first chemical war. The art of killing and robbing each other became "chemical" the day gunpowder was invented; at that time, however, the existing knowledge of chemistry was just of pinhead size. Napoleon knew very well how to use adroitly exact knowledge and chemistry for furthering his insatiable ambition to dominate the world; so he surrounded himself with the most able chemical advisers and scientists, and, for awhile at least, he placed himself at a decided advantage over his many enemies; incidentally, he thus helped to lay the foundation for some very important branches of chemical industry.

"Les chiens ont appris quelquechose," exclaimed the Corsican conqueror when he realized that his enemies began to adopt the same means which had given him temporary mastership over them; but those whom he called so contemptuously "the dogs" finally beat him at his own game.

Ever since then, science, technology and chemistry in particular, have played a rôle of increasing importance in the armament of nations. This accounts perhaps for the strange fact that the really great military inventions have practically all emanated from civilians and from non-military nations like our own. If the men of the military class, essentially conservative in all countries, had been left to their own devices, they would probably still be fighting with bows and arrows—or perhaps, with the traditional sling. Nor should the pacifist blame the chemist if the latter's most beautiful conquests in science, if his proudest discoveries, have been turned into means of relentless destruction and human slaughter. Do not reproach chemistry with the fact that nitrocellulose, of which the first application was to heal wounds and to advance the art of photography, was stolen away from these ultra-pacific purposes for making smokeless powder and for loading torpedoes. Do not curse the chemist when phenol, which revolutionized surgery, turned from a blessing to humanity into a fearful explosive, after it had been discovered that nitration changes it into picric acid.

As well might you curse written speech or language or the art of printing—by which the most noble thoughts of the human race have been expressed, disseminated and preserved—if it has been used also to distribute the vilest lies and the most damnable errors.

Knowledge is like a knife: in the hands of a well-balanced adult, it is an instrument for good of inestimable value; but in the hands of a child, an idiot, a criminal, a drunkard, or an insane man, it may cause havoc, misery, suffering and crime.

Science and religion have this in common, that their noble aims, their power for good, have often, with wrong men, deteriorated into a boomerang to the human race. Our very successes will threaten to devour us as long as all of us have not yet become imbued with the truth that greater knowledge, like greater possession of wealth or power, demands a greater feeling of responsibility, greater virtues, higher aims, better men.

Let us hope, in the meantime, that war carried to its modern logical gruesomeness, shorn of all its false glamor, deceptive picturesqueness, and rhetorical bombast, exposed in all the nakedness of its nasty horrors, may hurry along the day when we shall be compelled to accept means for avoiding its repetition.

Will you take it amiss if I made a digression from my subject as an answer to some repeated attacks which have been made of late by men who blame our increasing scientific knowledge in general and our chemical science in particular, for the excesses of the present European war?

THE PEACEFUL WORK OF AMERICAN CHEMISTS

But let us turn our attention to more peaceful chemical pursuits and more particularly to the chemists of this country.

Their work is difficult to understand and still more difficult to be appreciated by the uneducated or uninitiated; nor do chemists court the plaudits of an ignorant public that cannot understand them; they feel fully compensated by the results of their work if it only meets with the approval of a few of their fellow-chemists, irrespective whether it brings them financial results or not; in fact, most chemists are so much in love with their work, that very often they neglect the financial side to their own immediate detriment.

Unlike the physician, lawyer, clergyman, actor, writer, artist, or business man, the chemist does not depend on the public at large; he is either engaged in some private enterprise or he acts in a consulting capacity for a few people, if he is not engaged

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[1] This Journal, 7 (1915), 301, top of 2nd col.

[2] Presented at the 51st Meeting of the American Chemical Society, Seattle, August 31 to September 3, 1915.

as professor or teacher in some educational institution. Popularity in the usual sense has little or no value for the chemist.

No wonder then that the chemists of this country, numerous and active as they are, have hardly been noticed among the daily noise of newspaper sensation and shrieking publicity—no more than a skillful watchmaker would be noticed among the hammering of a busy steam-boiler manufacturing plant.

And yet, right here in the United States, the chemical profession has taken such a root, such a development during the later years, that we have here assembled at this very meeting, the representatives of our national American Chemical Society, which counts over 7000 members, by far the largest membership of any chemical society in the world, with all due respect to England, France and Germany—a society which finds it possible to spend yearly over $100,000 on its three chemical publications, copies of which are to be found all over the world in every well-equipped scientific library. Nor is the study of chemistry in this country a matter of recent occurrence. Our European friends are astonished when we tell them that as far back as 1792, there existed already the Chemical Society of Philadelphia, which was probably the first chemical society ever organized in the world; even to-day, some of the papers read at the meetings of that early scientific body furnish very interesting reading. Some of our American universities equipped chemical research laboratories for students, at a time when exceedingly few of the best-known European universities possessed any such facilities.

Nor should we overlook the fact that notwithstanding the essentially pragmatic tendencies of our country, the United States has given to the world a Willard Gibbs, who outtheorized existing chemical theories and whose mathematical deductions are still, after his death, furnishing food for profound thought to the most renowned physical chemists of Europe, to whom they have opened entirely new fields in the study of chemical dynamics.

THE ANILINE-DYE SITUATION

I mention this more particularly because some of our aniline dye consumers have taken the chemists of the United States bitterly to task and have made decidedly unfavorable comments on their abilities, because since the European war, dyes could no longer be imported from Europe. But Dr. B. C. Hesse, an American-born chemist, a graduate of the University of Michigan, has already ably answered this indictment of the American chemists. In a paper full of information on this subject, which he presented at our last General Meeting,[1] but which, unfortunately, has received little or no attention from our daily press, he has clearly demonstrated that the aniline dye consumers of the United States can have all the chemists and all the dyes they want; provided they are willing to make the necessary investments of capital and to submit to the risk of uncertain profits by starting their own dye-manufacturing establishments here in the United States instead of, as in the past, favoring imported dyes, either through personal choice, or by fostering legislation which forbids the home manufacturers to utilize such methods as selling agreements, "Kartels," or other consolidation of interests, "dumping,"—so as to kill new competitors in the field, while making up the temporary loss by increasing the price of other products,—and in general, any of the many other trade arrangements and trading tricks freely and openly utilized by European manufacturers so as to stifle possible competition of our home aniline dye producers.

The outcry which has been raised as to our shortage of artificial dyes is out of all proportion if we take into consideration that the annual importation of dyes and synthetic products from Germany amounts only to about $9,000,000. As our former President, Mr. A. D. Little, pointed out[2] this represents about the same sum of money as the amount of candy sold annually by the Woolworth Ten-Cent Stores.

LOCAL OPPORTUNITIES OF AMERICAN CHEMICAL INDUSTRIES

The development of any chemical industry is a matter of local opportunities; for instance, the manufacture of cellulose, as well as the industry of wood distilling, have taken a greater development in the wood-covered sections of the United States than in Germany or any other country in the world.

The magnitude and earning capacity of the largest German chemical enterprises, however imposing they may be, look less astonishing if you take into consideration that some of these companies have been in existence for more than half a century. Much younger American chemical enterprises, which make American specialties—for instance, the Eastman Kodak Company, which sends its films and photographic papers throughout the whole world—have annual earnings decidedly greater than the most successful German chemical works of much older existence. Nor is the value of the output of our largest purely chemical companies much less important than that of the German concerns.

SULFURIC ACID

This country is now the greatest producer of sulfuric acid with an annual production of about 3,000,000 tons. Yet it is not so long ago that the first maker of sulfuric acid could not even find a purchaser for a trifling small production of a few tons per week. It needed the opportunity of a home market; by and by this was created by the refining of petroleum after the discovery of our oil fields, .the discovery of natural phosphates and the resultant industry of superphosphates, the use of dynamite for blasting, the development of the glucose industry, and electrolytic copper refining. These and many other new industries all required large amounts of sulfuric acid, and gave this country an opportunity of developing sulfuric acid manufacturing to its present importance. In fact, the same reasoning holds good for all of our industries. I doubt very much whether the talented foreigners, who have now beome proficient in chemical manufacturing, would have tied their initiative and enterprise by specializing in coal-tar dyes manufacturing, if they had had the limitless opportunities of an immense undeveloped country like ours, to which to give outlet to their spirit of pioneership in mining, transportation, agriculture, and similar subjects, all beckoning for more urgent attention, and offering at the same time, more immediate rewards.

AMERICAN ELECTROCHEMICAL INDUSTRIES

In the meantime some of our other chemical industries, better suited to our local conditions, have taken such an enormous development here as to make the United States an undisputed leader in at least some of them. Such products as the various acids and salts, aluminum, artificial abrasives, soda and caustic alkalies, bleaching powder, chlorine products, electrolytic copper, are decidedly more important in value and in economic importance than the few million imported coal-tar dyes.

Mr. F. A. Lidbury[1] pointed out rightly that if there had been a shortage in some of the products of our electrochemical industries in which the United States has been a pioneer, the consequences to our national economics would have been so serious that the present complaint of our aniline dye users would have sounded like a timid whisper, compared to the bellowing lamentations of so many more important industries which would have become absolutely paralyzed. The fact is that few men realize how many industries are directly dependent on the work of American chemists. If the aniline dye industry has been somewhat neglected in this country, there are many other good reasons for it; not only was the possibility of reasonable profits too scant to offer special inducement to clever-headed business

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[1] This Journal, **7** (1915), 293.

[2] *Ibid.*, **7** (1915), 237.

[1] F. A. Lidbury, *Metallurgical and Chemical Engineering*, **13** (1915), 277.

men to risk their capital in this branch of manufacturing when they had so much better choice in other channels of enterprise, but the first raw material, suitable coal tar, was not abundantly available here as it is in Europe, for the simple reason that this country long ago discarded the older and more expensive methods of gas manufacturing generally used in Europe, and which give coal tar as a by-product. The less expensive and simpler water-gas process, adopted in the United States, gives no suitable gas tar; it is only of late, by the introduction of the by-product coke ovens that we can look forward to an almost unlimited supply of coal tar.

In the meantime, the German manufacturers had every opportunity in specializing in these coal-tar industries and could afford to concentrate their efforts so as to supply not only their home consumption and that of the United States, but that of the whole world; in about the same way as the United States sends to the remotest corners of the globe its sewing machines, its typewriters and its Ford cars.

Judging from the past history of the chemical industry in America, I have little doubt that the day it will be found profitable to manufacture all kinds of synthetic dyes here in the United States, instead of a few as is the case now, there will be little further delay in supplying the demand by a hustling and bustling home production.

In fact, it is quite possible that under present abnormal conditions, this branch of manufacturing may be stimulated to the point as to result in overproduction after the war is over.

CHEMICAL RESEARCH IN UNITED STATES

If hitherto our chemists have been deficient in this special line, we can, with some satisfaction, point to better efforts in other chemical industries. For instance, it is not sufficiently known how many research chemists in our different American manufacturing establishments are busily occupied in studying and improving manufacturing processes, nor what large sums of money are devoted every year to industrial chemical research. If we hear it constantly repeated that some of the largest German chemical companies have hundreds of chemists and engineers, it is less known that right here in the United States, the number of chemists employed in some of our better organized chemical enterprises is scarcely less; but nobody finds it necessary to boast about it. In fact, the most striking symptom is that so many engineering enterprises, for instance, some of our large electrical companies—although their field of action seems rather remote of chemical subjects—have now elaborate chemical research organizations, of which the record is well known by their excellent results.

Conditions were quite different some fifteen or twenty years ago; but this country has grown, and as the requirements and opportunities grew up at the same time new chemical problems arose thereby.

THE ANALYTIC AND CONSTRUCTIVE TURN OF MIND

The urgent nature as well as the magnitude of some of these new chemical problems, is shaking our chemists awake,—is making new men of them.

Professor Whitaker is probably right when he says that the chemists are thirty years behind the engineers as far as method and attitude of mind is concerned, but this same criticism holds good for chemists all over the world. The fact is that the engineer was called first, and he was born centuries before the chemist, but the latter is now making up for lost time.

New conditions, new problems, are compelling the chemist to learn to tackle a proposition in a true engineering spirit and—to hitch some business sense to it. He is learning to forget thinking or acting on the test-tube plan; he is thrown more and more in contact with business men; he begins to realize that too one-sided theoretical considerations are sometimes more dangerous than complete ignorance and that a sense of proportion and relative values is the first requirement for good practical effort.

Here, indeed, is one of the weakest spots of the chemist. Aside from the fact that the chemical profession seems one of those vocations which have fascinated a large number of intellectual freaks, it has generally attracted men of an analytical, rather than a constructive turn of mind. Successful engineering is essentially constructive. The most urgent work for the chemist of to-day must be constructive—he must learn how to cement together the vast amount of data which already lie at his disposal, even if he, himself, has to provide this very cement by further research.

THE CHEMIST IN OTHER INDUSTRIES

The chemist of to-day is no longer confined to purely chemical enterprises; even the most stubbornly conservative manufacturers have learned, through competition, that every industry, however mechanical be its nature, has its chemical problems. Things have changed rapidly since the day Andrew Carnegie listened with a sly twinkle in his eyes to the fun his competitors were poking at him when he first engaged a spectacled professor to investigate the chemical problems in his iron works. Conditions have now become reversed; to-day, a steel or iron works without a competent chemist justly provokes contempt and distrust.

Nor is the time so far distant when even our biggest railroads had not begun to realize how they missed the constant services of a staff of chemists, so as to advise them in the endless chemical problems which present themselves in the operation of a well-organized railroad.

Some time ago, I visited the plant of the National Cash Register Company, in Dayton, Ohio; one of its most interesting departments was its well-equipped chemical laboratory, where no end of chemical questions relating to the manufacture of purely mechanical devices have to be studied and solved.

No up-to-date motor-car works is complete without its chemical department and the same remark holds good for all well-organized engineering concerns.

In this country, the importance of chemistry has been first appreciated in its relations to agriculture. So obvious was this, that we set an example to all other nations of the world by the number of our federal and our state chemical agricultural laboratories. This, more than anything else, was the entering wedge of applied chemistry in this country, which extended later on in the Government Service, Geological Survey, Bureau of Standards and the Bureau of Mines. Nor did the useful effect stop there. Many of our federal chemists, our state chemists, have left public service to accept better-paying positions in private industries; but the men trained in public service implanted their high aims and scientific ways in some of our commercial enterprises which needed it badly. I know of some cases where this beneficial influence changed radically the whole spirit of the commercial organization, from its manufacturing to its selling department, and introduced instead of reckless, sordid commercialism, a spirit of fairness and efficiency which soon proved the more profitable policy.

ETHICS OF THE CHEMIST

In this and similar directions the chemist can exercise a valuable moral influence on the community. If you think it over, you will find that the quest of efficiency lies quite close to the path of honesty, justice, and equity.

Here also the chemist has much to learn. In some instances, I have been astounded at the almost childlike attitude of mind of some of our chemists who are too ready to sell their services to anybody who has a temporary use for them, irrespective of the underlying motives or purposes.

Some lawyers tell me that they never have the slightest difficulty in hiring chemical experts to defend contradictory opinions. It is quite amazing how some chemists in their eagerness of pleasing their employers will overlook their own ignorance of the most elementary principles of patent law, as well as their superficial acquaintance with the many details of intricate technical questions while not hesitating to furnish cocksure opinions which encourage infringers or industrial pirates to trespass on the rights of intellectual property of others. Much ruinous patent litigation would be avoided in this country, and invention would be better encouraged if we had more men of the type of a well-known British electrical expert, who never hesitates in Court to tell the simple and direct truth, regardless whether it kills or saves the case of his client; his statements are so highly valued and respected that the judges accept them without suspicion, and the same expert is frequently retained by the two opposing parties, whom he serves impartially, and who gladly pay him higher fees than to a mere litigation-acrobat-expert, or a chemical "ambulance-chaser."

EMPLOYERS AND EMPLOYEES

The ethics of our profession have been dealt with by the American Institute of Chemical Engineers, and have been embodied in its recently adopted Code of Ethics, which may furnish a good guide for younger or less-experienced chemists. And this leads me to state that many more manufacturers or business men would be induced to utilize the services of chemists, if they could feel confident that in so doing they are not putting themselves at the immediate mercy of a stranger, by confiding to him facts or processes which it has cost them many sacrifices of time and money to accumulate, and the undivided knowledge of which constitutes sometimes one of their most valuable assets. On the other hand, a chemist can hardly be of any service unless his client or employer is just as frank with him as he would be with his lawyer or physician. However, this mooted point is easily overcome by referring to the Code of Ethics to which I have just alluded, or, better, by making a preliminary agreement between the chemist and his client or employer, safeguarding the interests of both parties. But in such a case, the compensation to the chemist should be made commensurate to the occasion.

This same principle holds good in the employment of chemists in manufacturing plants, where the chemist is either engaged in research or in a manufacturing capacity. An employer should not expect an intelligent chemist to render him important services without proper compensation, and in as far as the practical value of the work of a chemist can seldom be determined in advance, it will pay the employer to offer special inducements or rewards for initiative; he can well afford to give his chemist some share of the increased profits he has received through his work; to do otherwise would be narrow-minded, short-sighted, and detrimental to the direct interests of the employer. The work of a research chemist cannot be performed nor measured like that of a bookkeeper or a laborer; the results of his work are uncertain; delays and obstacles beset him at every turn; sometimes luck plays an important rôle; but good-will, enthusiasm, and persistent endeavor are indispensable factors and these may be encouraged or killed by the attitude of the employer. An employer who is unfair, or who cannot arouse the respect or the enthusiasm of his chemists, cannot get the best there is in them. He must make them feel that if their work turns out well for him, they will get some decent share of compensation. Therefore, a reasonable salary ought to be supplemented by the possibility of a bonus or some share in the profits based on the earnings brought about directly by the work of the chemist.

On the other hand, the chemist must not overlook the financial sacrifices and business risks assumed by his employer. He should specially bear in mind that knowledge or experience gathered at great cost by his employer, or through expensive factory equipment, or other facilities, have in most cases enabled him to take up his own work at an advanced stage. It would be rather unfair, unless otherwise stated, that a chemist should be allowed during or after his period of employment, to divulge or take advantage of all knowledge or information gathered around the plant in which he is engaged; or patent for his exclusive benefit, any inventions he may make on those particular subjects for which he is engaged, as long as the stimulating ideas themselves have been gathered by the very means put at his disposal by his employer. All these questions should be provided for and embodied in an equitable contract which will necessarily vary with special circumstances. But here again, niggardliness, or too great cunningness of the employer, will hardly pay. Unless his chemist be a fool—and a fool of a chemist is not worth anything—he will lose the good will and confidence of the very man whose work is primarily dependent on these factors.

Faithful and generous observance of these conditions have brought about the most excellent results in many instances. I know that the contract system, with a salary supplemented by a bonus, or participation in profits in special departments, has been used with great advantage to all concerned, by some of the most successful chemical companies in Continental Europe, and in some of the more progressive American enterprises.

It has been objected that a contract of the kind merely binds the employer who has tangible assets, while in most cases it would be difficult to enforce it against faithless employees. But even then, a clear and well-defined contract will prevent many misunderstandings which will crop up in the course of time. It has been my experience that direct dishonesty and faithlessness are merely exceptions among chemists, whatever their other shortcomings may be.

THE CHEMIST AS A RESTLESS PROSPECTOR

We know where the work of the chemist begins. We can never tell where it ends, and through what unexpected ramifications it may lead. It is just this fact which adds some zest to the life of the struggling, hard-working chemist, and brings to his work frequently as much excitement as the best of sports; his hopes and disappointments can be compared to those of the restless prospector.

Pasteur, while he was professor at the University of Lille, was consulted by a local alcohol distiller about some irregularities in the fermentation processes. Little did the great French chemist dream, when he tried to solve this purely industrial problem, that by doing so he was going to establish such an amount of new and unsuspected scientific facts, destined to upset all formerly accepted notions, not merely on fermentation, but on life, disease, contagion and epidemics; that he was about to revolutionize surgery, sanitation and medicine, and create several new departments of medical science; that he was going to save millions of lives, reduce sorrow and misery. So little were the men of that period prepared for all these stupendous revelations that this great benefactor of the human race had to suffer most from the gibes and violent attacks of some of the best-known men of that very medical profession into which he was going to infuse new life by placing it on a true scientific basis. The history of the stubborn polemics and angry discussions at the French Academy show that, at that time at least, the imagination even of men of science could not expand to the point of perceiving that medicine and surgery were to be remodeled by the hands of a mere chemist.

YONKERS, NEW YORK

THE INDUSTRIAL RESOURCES AND OPPORTUNITIES OF THE NORTHWEST[1]

By H. K. BENSON

The industrial aspect of the Northwest has been towards the East. From thence flowed the great streams of capital which

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[1] Presented at the 51st Meeting of the American Chemical Society, Seattle, August 31 to September 3, 1915.

have furrowed the continental barriers and spread a network of transportation to every storehouse of natural treasures; or locked in mountain fastnesses have generated the power with which the wheels of industry may be driven. The time is coming and is now here when we may well turn from the broader phases of external development to a more minute and critical inspection of the resources about us on every hand and utilize them in the greater intensification of industry and commerce.

The Pacific Northwest constitutes an industrial unit which comprises the states of Oregon, Idaho, and Washington, the Province of British Columbia in Canada, and the territory of Alaska. Although composed of different political parts, whereby the natural course of industry may be hindered or benefited, yet the ultimate outlet for commodities is largely confined to the cities touching the waters of the Pacific Ocean. Material development of any portion of this area is felt throughout all of it. While it would be of great interest to comprehensively discuss the potentialities of the Pacific Northwest as a whole, the lack of published data would make it necessary to collect and compile at first hand much of this information. Time does not permit of such an extended investigation at present, and it is proposed therefore to present in some detail the resources of the State of Washington as a typical example of what may be found in varying degrees throughout the entire Northwest.

The total area of the Pacific Northwest includes nearly a million and a quarter square miles, distributed as follows:

OREGON	WASHINGTON	IDAHO	BRITISH COLUMBIA	ALASKA
96,607	69,180	83,888	395,500	586,400 sq. mi.

Harney County, alone, in Oregon, contains as much tillable land as the State of Maryland, while the area of Alaska is greater than that of Norway, Sweden, Finland, England, Scotland and Ireland taken together. While a large portion of Alaska is mountainous, less than 1 per cent of its area consists of permanent snow fields and its agricultural valleys contain as much tillable land as is cultivated in the States of New York, Pennsylvania, and Ohio.

LUMBER AND WOOD PRODUCTS

We call Washington the "Evergreen" State, because its hills and mountains are enveloped with forests of fir, cedar, spruce and hemlock. With the pines of Oregon and Idaho, these forest species represent the greatest resource of the Pacific Northwest. In the states of Oregon, Washington and Idaho there are still uncut more than 50,000,000 acres of merchantable timber, from which it is estimated that over 850,000,000,000 feet, board measure, of lumber may be cut. This is more than one-third of the standing timber of the United States. The annual cut of lumber in the two states of Oregon and Washington is slightly in excess of 5,000,000,000 feet in lumber and 8,500,000,000 shingles. Over 2,000 wood manufacturing establishments, employing about 65,000 men, are required in the production of the lumber and timber products of Oregon and Washington annually. The value of the timber products annually manufactured exceeds $80,000,000, of which 80 per cent is paid out for wages, taxes, rent and other production costs. When there is added to these forest areas, the 180,000,000 acres of forest lands in British Columbia with its annual cut of $1^1/_3$ billion feet and an estimated stand of merchantable timber amounting to 130,000,000,000 feet and the more or less indefinite content of 20,000,000 acres of merchantable timber in Alaska, it becomes apparent that the timber products of the Northwest will continue for many years as an asset of great importance.

FERTILIZERS

FISHERIES PRODUCTS—The fertilizer resources of the Pacific Northwest are almost of equal importance with its forest products. From the waters of Puget Sound and the North Pacific the annual harvest of fisheries products exceeds $53,000,000. In 1913 the fisheries products of British Columbia were almost one-half of the total production of the Dominion, amounting to $14,500,000 and twice the value of those of Nova Scotia. The salmon industry of Alaska now extends from Ketichan in Southeastern Alaska for a distance of 2000 miles to beyond the Bay of Bristol in Bering Sea. In 1913 the 79 salmon canneries of Alaska represented an invested capital of $31,341,670.00 and the annual catch of halibut, codfish, herring and whales required an additional investment of over $6,000,000.

From the annual packing of 8,000,000 cases of salmon on the Pacific Coast results an annual production of 140,210 tons of cannery waste, which might be augmented considerably by the non-edible fish which are rejected and never reach the cannery. This waste contains in the fresh condition 10 per cent oil, 3.5 per cent bone phosphate, 1.5 per cent phosphoric acid, and 3 per cent nitrogen. Of the total waste in 1913, only 16,300 tons were utilized on the Pacific Coast of the United States. From this waste were obtained 1630 tons of dry scrap and 293,000 gallons of oil. More complete utilization would increase the possible output of fertilizer and oil by tenfold.

POTASH—Floating upon these same waters are the great beds of the giant kelps or sea weeds. These have been mapped by the United States Bureau of Soils, and according to this survey, they yield annually over 20,000,000 tons of kelp equivalent to 750,000 tons of potassium chloride. The total imports of potash salts of all kinds in normal times is equivalent to about 400,000 tons annually. From the kelps of the North Pacific waters may be extracted not only the potash salts for domestic consumption, but a surplus as well, which eventually may enable this country to export potash salts to other countries.

ROCK PHOSPHATE—Until the discovery of the rock phosphate deposits of the Northwest the only commercial quantities that were known to exist in the United States were found in Florida and Tennessee. The phosphate deposits of Idaho are thirty times greater than the other known deposits of the United States. In the areas thus far surveyed by the United States Geological Survey it is estimated that over 2,500,000,000 tons of phosphate rock with 35 to 37 per cent phosphoric acid (P_2O_5) may be found.

In the ultimate utilization of cannery waste and of non-edible fish, of the potash in kelps, and of phosphate rock, the Pacific Northwest offers a most promising field for the upbuilding of a great fertilizer industry.

POWER

COAL AND OIL—In the past, the industrial welfare of a community was dependent upon its nearness to a coal deposit but in the Northwest coal has competitors in the great quantities of oil from California and the electrical power of the mountain streams of the Northwest. The annual production of coal in the State of Washington is nearly 4,000,000 tons, while that of British Columbia is 2,500,000 tons. The total supply of coal in the Pacific Coast states is estimated by Campbell and Parker at 22,000,000,000 tons of which 20,000,000,000 tons are located in the State of Washington. The coal fields of Alaska cover 12,667 square miles. The estimate of the Geological Survey places the minimum coal resources of Alaska at 150,000,000,000 tons, which are far in excess of the original coal supply of Pennsylvania. Unlike the latter, however, much of this coal consists of lignites, the utilization of which calls for new methods and processes. Even in the State of Washington and adjacent to the coal fields themselves fuel oil is transported from California displacing annually a quantity of coal greater than that consumed.

WATER POWER—The water power resources of the Pacific Northwest constitute an important factor in power productions. The estimated developed water power of the Pacific Northwest, exclusive of Alaska, exceeds 650,000 H. P. The estimated undeveloped power of the Columbia River Basin is approximately 20,000,000 H. P., or one-third of all the undeveloped water power in the United States, and equivalent to all the water power found in the twenty-six states east of the Mississippi River. Within economical transmission of our larger cities on tide water may be generated at least one-fifth of the available water power of the United States, exclusive of Alaska. The water power resources of the latter are said to exceed those of the Pacific Coast states.

MINERALS

METALS—Much has been written of the mineral industry of Alaska and the Northwest, and it would become tedious to particularize concerning its extent and importance. The annual production of gold and silver in normal times is approximately $35,000,000. For the year 1913, the production of copper was valued at $11,300,000, of lead at $15,600,000, and of zinc at $2,500,000. The development of deposits of minor minerals has not been extensive, due to their remoteness from suitable smelting facilities. On the Seward Peninsula is a tin-bearing district of 450 square miles. Alaska has produced since 1902 over 560 tons of tin. Antimony ores are found in Alaska and Washington, molybdenum in British Columbia and Washington, tungsten in Alaska and Washington, while platinum is found in the sluice boxes of many of the placer camps of Alaska. Arsenic ores abound in the Monte Cristo district of Washington, and are smelted with the recovery of white arsenic at the Tacoma smelter.

GRAPHITE, GYPSUM AND BARYTES exist in commercial quantities in Alaska. In Washington and adjoining portions of British Columbia occur the only large deposits of LIMESTONE located on deep water along the entire Pacific Coast of the United States and Canada. Extensive deposits occur in the northern tier of countries from Puget Sound to the Idaho boundary. These deposits constitute the sources for six Portland cement, one hydrated lime, and numerous lime-kiln plants now located within the State of Washington.

CLAY—Clay deposits suitable for use in common brick manufacture are widely distributed throughout the Pacific Northwest, as are also those adapted to the manufacture of drain tile. In numerous places are also found deposits of clays which can be used for the manufacture of paving brick, sewer tile, terra cotta, and firebrick. Both Washington and British Columbia are producers of refractory brick of a high grade. Washington now ranks fifteenth in the production of clay products in the United States, forty-five firms being engaged in the industry, and producing wares valued at over $2,000,000 annually.

MISCELLANEOUS—Deposits of diatomaceous earth of superior quality have been investigated in five locations within the State of Washington. Apparently enormous quantities of this material exist in this state. Deposits of magnesium carbonate, and other magnesium salts have been reported, but are not utilized. Among other raw materials available in the Pacific Northwest are iron ores suitable for mineral paints and perhaps for pig iron; talc for use as a filler in paper manufacture; silica for glass and sand-lime brick; and large deposits of stone for structural purposes. Among the latter especial mention should be made of the Alaskan marble and of the granites and sandstones of Washington. At least three extensive deposits of tufa rocks occur in Oregon and Washington.

MANUFACTURING INDUSTRIES

The opportunities of the Pacific Northwest are to be found in the utilization and distribution of the resources heretofore cited. Less than 100,000 persons are now engaged in manufacturing industries, which produce products valued at $400,000,000 in the States of Oregon and Washington. An inspection of the foodstuffs of the city of Seattle reveals what is typical of the entire Northwest. In this city of 300,000 population, during the past year 7,207 cars of vegetables and fruit were shipped into the city. This is equivalent to a carload for every seven families. Of three million packages making up the total receipt, a million and a quarter came from outside the State of Washington.

AGRICULTURE NEGLECTED

Agriculture is a dormant, undeveloped industry. Less than 10 per cent of the agricultural land of Washington has been improved and is utilized for agriculture. More than 2,500,000 acres of logged-off land of agricultural character remain uncleared and idle. This is due chiefly to the high cost of clearing the land by the methods which are commonly in use. The farms which have been developed from the wilderness of débris left by the logger represent a monument of industry to those who have reclaimed them and made them factors of production. In Eastern Washington the problem has been different. Instead of the jungle the desert must be overcome. Irrigation has transformed large areas into orchards and meadows of great beauty and wonderful productiveness. But even there, out of 2,000,000 acres of irrigable land, not more than 25 per cent has thus far been reclaimed. In British Columbia the estimated acreage of tillable land is 253,000,000 acres, of which not more than 12 per cent is occupied, and only about one-fifth of that is under cultivation. The great need of the Pacific Northwest is settlement by colonists. Transportation is available, water and power are abundant, natural resources of unknown extent and character await development: in brief—the harvest is great, but the laborers are few.

CONSERVATION NEEDED

In the industries already established greater intensification and closer utilization are urgent. Fully as much timber as is manufactured into lumber is either wasted by burning or used in an uneconomic manner. As will be shown in papers presented before the Convention, the fir slabs of the sawmills contain nearly as much tannin as the chestnut wood which is the basis of the tannin extract industry of the United States. But no extract plant exists in the Northwest, and even tanneries have had hard struggles for existence in spite of the fact that great quantities of hides are readily available. Men have looked at our great forests and have seen nothing but lumber in them, and, following the line of least resistance, they have cut down these forests and manufactured a single product.

In the fisheries industry the results have been largely similar. Mr. John N. Cobb, editor of the "Pacific Fisherman" calls attention to the neglected fishery resources of the Pacific Coast in an admirable paper presented before the first annual meeting of the Pacific Fisheries Society. In the 120,000 tons of salmon offal annually wasted are millions of pounds of salmon eggs. Siberia in 1913 prepared 500,000 pounds of salmon eggs as caviar, while 24,000 pounds were prepared upon the Pacific coast of America in the same time. In Japan whale meat is of considerable economic importance as a food product. The whaling stations of the Pacific Coast ship certain portions to Japan, but by far the largest portion is either made into fertilizer or else wasted. Methods of processing whale meat so that it could be marketed as a food product should be worked out by the technical men of the fisheries industry, whereby millions of pounds of meat might be added to our national supply annually.

These are random instances of the wasteful methods whereby our natural resources are being developed. A more complete study of existing conditions would simply tend to multiply their number. The Pacific Northwest needs to apply the methods of self-examination which have been so successfully applied to the industries and resources of the more congested sections of this and other countries. It needs a closer alignment with the technical thought and progress of the day. In a word, it needs not capital, but captains of industry. With more care for the utility of natural resources and with the coöperation of the technical skill of the day, opportunities will spring from hidden places, and industries will multiply, and the Pacific Northwest will take its place in the moving streams of commerce and trade.

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CHEMICAL PATENTS—III AND IV

III—CONTRACTUAL RIGHTS RELATING TO LETTERS PATENT

By Seabury C. Mastick[1]

1—ASSIGNMENTS

An assignment of a patent or application for patent is an instrument in writing which, in the eyes of the law, purports to convey the entire title to that patent or application, or to convey an undivided share in that entire title.

The patent or application assigned ought to be described in the assignment by its number and date, or serial number and date of filing, as the case may be, and by the name of the patentee or applicant and the title of the invention. In the case of assignment before or cotemporaneous with the application, it should specify the name of the inventor and the title of the invention in manner sufficient to identify it, giving also the date of execution of the application papers, if possible. Where the assignment is made before the payment of the final fee, if so requested in the assignment, the patent will issue to the assignee.

The assignment, whether of a patent or of an application, should be acknowledged before a Notary Public, although this is not requisite for validity, and unless the assignment of an issued patent is recorded in the Patent Office within three months of its date it is void as against any subsequent purchaser or mortgagee for a valuable consideration without notice.

An assignment of an application is not required to be recorded, although as a matter of practice it should be. Such assignment, even though unrecorded, is valid against all subsequent assignments, even though the latter assignees have no notice of the prior unrecorded assignment. Even though the patent issue to the inventor or to the assignee of an assignment subsequent to the prior unrecorded assignment, the assignee of the latter has both equitable and legal title to the patent and may lawfully bring suit for infringement thereof.

Rights of action for past infringement of a patent are not conveyed by the mere assignment of that patent but should be specifically mentioned in the assignment. No action can be brought for acts of infringement occurring before the issue of the patent, so such need not be mentioned in an assignment of an application.

Interests in patents may be vested not only in assignees but in grantees of exclusive sectional rights, in mortgagees, and in licensees.

(1) An assignee is a transferee of the whole interest of the original patent or of an undivided part of such whole interest, extending to every portion of the United States. The assignee of an undivided interest, no matter how small, has the same right to make, use or sell the patented invention, or to license others to do so, as have the owners of the remainder of the interest, even though the latter interest is much greater than the former, subject only to an accounting of profits to the other owners of the patent in the proportion of the respective interests.

(2) A grantee acquires by the grant, which must be in writing, the exclusive right, under the patent, to make, use and sell and to grant to others the right to make, use and sell, the thing patented within and throughout some specified part of the United States.

(3) A licensee takes an interest less than or different from either of the others. It may be exclusive and for the whole country, or exclusive for a part of the country, or limited to the making, or using, or selling or in any other way. A license may be oral or written and need not be recorded in order to be valid against subsequent purchasers of the patent or subsequent licensees. In the case of a license the title to the patent remains in the patentee or assignee and all suits for infringement must be brought in the latter's name.

A license not expressly limited in duration continues until the patent expires or the license is surrendered or forfeited in accordance with its own terms.

No license is assignable by its licensee to another unless it contains words which show that it was intended to be assignable.

2—LICENSES, EXPRESS AND IMPLIED

Express licenses are those given directly either orally or in writing. Implied licenses may arise from the conduct of patentees or assignees, or from the recovery by them of profits or damages for certain classes of infringements.

Implied licenses arising from the conduct of owners of patent rights are generally found to arise by virtue of acquiescence in acts which constitute a license or by action of estoppel. For example:

Where the patentee knows of the use and does not object, especially if he receives a portion of the profits for such use.

Where the owner of a patent explains the invention to another who is ignorant of the patent and advises the latter to make, use or sell the patented invention with intent to cause him to infringe unwittingly, an implied license would result by estoppel to the person thus misled.

Where a patentee sees an infringing article sold without authority from him, but makes no objection, an implied license by estoppel results to the purchaser.

A recovery of damages by way of royalty as against an infringing manufacturer operates an implied license to the purchaser from that manufacturer. But a recovery of profits only from an infringer for use of a patented device gives no license to the subsequent purchaser as each infringer by way of use stands alone to account to the owner of the patent for his profits in its use.

3—EMPLOYER AND EMPLOYEE

Where a man is employed for the purpose of making inventions, all such inventions are the property of the employer, who can compel assignment of them to him, the inventor having no property right in them whatsoever.

Where a man is employed generally by another to work in the business of the latter, and not specifically as an inventor, and, during the period of such employment, conceives an invention, applicable to the employer's business, and uses the property and time of the employer to reduce the invention to practice, an implied license or "shop right" results to the employer. This is especially the case if the employee sees the employer make, use or sell the thing invented, without making objection.

If the invention was made and the drawings prepared outside the hours and shops of the employer, but the invention was reduced to practice and made in the employer's shop at the employer's time and expense, an implied license results to the employer.

Where a person has discovered a new and useful principle in a machine, manufacture or composition of matter, he may employ other persons to assist in carrying out that principle, and if they, in the course of experiments arising from that employment, make discoveries ancillary to the plain and preconceived design of the employer, such suggested improvements are in general to be regarded as the property of the party who discovered the original principle, and they may even be embodied in his (the employer's) patent as part of his invention.

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[1] Special lecturer on Chemical Patents, Department of Chemical Engineering, Columbia University, 1915.

But persons employed, as much as employers, are entitled to their own independent inventions, and if the suggestions communicated constitute the whole substance of the improvement then the patent should be applied for and would be the property of the employee subject to the qualifications as to actual employment as an inventor and implied license as above pointed out.

If you are an employer the safe way is to have a definite contract in writing with the employee, specifying that all inventions made while in said employ are to be assigned to the employer, together with any future improvements on the several specific inventions.

If you are an employee the safe way is either to have a definite agreement in writing with your employer or not to do any inventing or reducing to practice of such invention on the premises or on the time or at the expense of or with the tools of the employer.

IV—ACTIONS FOR INFRINGEMENT

I—THE CASE IN THE LOWER COURT

The District Courts of the United States in the several States and the Supreme Court of the District of Columbia have original and exclusive jurisdiction of all suits, either at law or in equity, arising under the patent laws of the United States. The action must be brought in the district in which the defendant is an inhabitant, or in any district in which the defendant, whether a person, partnership or corporation, shall have committed acts of infringement and have a regular and established place of business.

Actions brought to enforce or set aside contracts relating to patents, such as license contracts, contracts to assign patents or applications, or for specific performance of such, are not actions arising under the patent laws of the United States and are not within the jurisdiction of the United States Courts unless diversity of citizenship and the amount involved give such jurisdiction. Such actions are properly within the jurisdiction of the several State Courts.

In any suit or action brought for the infringement of any patent there shall be no recovery of profits or damages for any infringement committed more than six years before the filing of the bill of complaint or the issuing of the writ in such suit or action.

In any suit for infringement brought by a party failing to mark the patented article with the word "patented," together with the day and year the patent is granted; or when, from the character of the article, this cannot be done, by fixing to it, or to the package wherein one or more of them is enclosed, a label containing the like notice, no damages shall be recovered by the plaintiff, except on proof that the defendant was duly notified of the infringement, and continued, after such notice, to make, use, or vend the article so patented.

The decisions of the District Courts of the United States may be appealed to the proper one of the United States Circuit Courts of Appeal for the several Circuits (nine in number) and the judgments or decrees of the Circuit Courts of Appeals are final in all cases arising under the patent laws, excepting that said Court may certify to the Supreme Court of the United States any questions or propositions of law concerning which it desires the instruction of that Court for its proper decision; and excepting also that it shall be competent for the Supreme Court to require, by *certiorari* or otherwise, any such case to be certified to the Supreme Court for its review and determination.

As a matter of practice the decision of the Circuit Court of Appeals is generally final and very few cases go up to the Supreme Court.

Actions at law for infringement of patents may be before a jury or, if both sides waive a jury, before a judge without a jury, or before a referee appointed by the Court with consent of both parties. The only relief in actions at law is to give damages for past infringements of patents. Actions at law are infrequent excepting where the action is brought for damages for the infringement of an expired patent.

Actions in equity for infringements of patents are before a judge without a jury and must be so brought where an injunction to restrain future infringements is desired. The Court sitting in equity has jurisdiction to direct an accounting for profits and damages which the complainant has sustained on account of the defendant's infringement. Almost all actions for infringement are brought on the equity side of the Court as an injunction is almost universally the chief remedy desired.

It is unnecessary to go into the details of a trial in these lectures further than to state that the pleadings comprise a complaint and an answer; that the complaint must set up the patent, that the complainant has title thereto, that it is valid and that it has been infringed by the defendant; that the answer must set up the defense, controverting the allegations of the complaint, and specifically setting forth the prior uses or prior patents relied upon for invalidating the patent or restricting it so that infringement cannot be held.

The testimony, where possible, should be given in open Court but when the witness lives at a greater distance than one hundred miles from the place of trial or the Court, upon application of either party (in cases permitted by the statute), for good and exceptional cause for departing from the general rule, gives permission, the testimony may be taken by deposition before an Examiner or other qualified officer.

The question of the validity of the patent sued upon is determined in accordance with the rules heretofore given in the two preceding lectures. At the outset of the trial the patent is presumed to be *prima facie* valid and the complainant is not required (and generally not even permitted) to introduce any evidence as to validity. He must, however, prove infringement. This is done by offering in evidence a sample of the infringing device or by introducing testimony clearly describing the alleged infringement if, from the nature of the case, such as in certain process or certain complicated or cumbersome devices, it is not possible to produce bodily the matter constituting the infringement. Expert evidence may be introduced to explain the operation of relevant arts, processes, machines, manufactures or compositions of matter, and of the meaning of terms of art or science and of diagrams or formulae, but the witnesses are not permitted to give their opinion as to the meaning of any patent claim or specification. This latter is solely for the Court to consider.

A patent for a process is infringed by one who, without authority, uses substantially the process of the claim, whether or not he uses substantially the apparatus or materials which the patent describes, or uses equivalents therefor. The definition of an equivalent has been given in one of the preceding lectures as "a thing which works in substantially the same way to accomplish the same result or perform the same function as the thing for which it is substituted." No process patent claim is infringed if one of the steps of the process is omitted by the alleged infringer unless some equivalent step is substituted for the one omitted.

A patent for a composition of matter or a machine is infringed by one who, without authority, makes, uses or sells the thing covered by any claim of the patent. It is not an infringement of a patent, whether in a process patent or in one for a composition of matter or a machine, to use, or to make, use or sell, a thing described in the specification but not claimed in the claims, as the measure of the patent protection is defined only by what it claims. Of course the rule of equivalents also applies in composition of matter and machine cases.

Whoever infringes any one claim of a patent infringes the patent, whether or not it contains other claims which are not infringed.

Addition to a patented machine or article of manufacture does not void infringement. Addition to a process or to the ingredient of a composition of matter does not avoid infringement, unless a substantially different process or composition results.

Omission of a claimed part of a machine or of a claimed ingredient of a composition of matter, no equivalent being substituted, negatives infringement of said claim, as a combination is an entirety and if one of its elements is omitted, the claimed thing disappears. Every part of a combination claimed is material, and evidence to the contrary is inadmissible.

Changes of proportions of the ingredients of a composition of matter, unless the claim gives specific proportions, will not avoid infringement.

It is not necessary that the infringer should have known of the patent at the time he infringed it or should have known that what he did was an infringement even if he did know of the patent. In either case an action for injunction will lie although profits and damages could not be recovered unless the patented article was marked "patented" or the infringer had received actual notice. If the patented article had never been manufactured and the patent was a mere "paper" patent, or in the case of a process where marking "patented" is impossible, profits and damages could probably be recovered in the absence of marking "patented" or of actual notice, although, in this case, actual notice is usually required.

As is stated above, it is unnecessary to go into details in these lectures. The subject of infringement is a large one and it has been the intention to point out only certain of the more prominent features. For greater detail it will be necessary to consult the text-books on Patent Law.

THE HALL PATENT LITIGATION

In order to illustrate the practice in connection with patents, both with relation to the trial of a case and the way in which the Courts handle the matter, I have chosen the litigation concerning the Hall Patent (No. 400,766, April 2, 1889) for the electrolytic production of aluminum. We have already traced the history of this patent through the Patent Office. Now let us trace its history through the Courts.

We shall see the practical application of many of the rules heretofore stated and shall learn something of the trials and tribulations of the patent litigant.

About the first of July, 1886, Hall was introduced to the Cowles brothers who had already organized the Cowles Electric Smelting & Aluminum Co. and later entered into an optional agreement with them to sell them his patent. The option ran for a little less than a year. During the period of the option, which expired sometime in July, 1888, he was given facilities to experiment and develop his process at the Cowles plant at Lockport, New York. His proposed process was one wherein the crucibles were heated externally and copper anodes were used. Hall was unable to satisfy the Cowles Company that his process was a success as they believed that internally heated crucibles with carbon rods for anodes were necessary. Internal heating, however, was alleged to be within the scope of certain prior Cowles patents and applications and Hall would not, at that time, deviate from his original idea.

Early in July, 1888, one of the employees of the Cowles Company resigned and went to Pittsburgh, where he began the organization of what became The Pittsburgh Reduction Company. At the close of July, after the option had expired, Hall went to Pittsburgh also, joined forces with the organizers of the new company and in November, 1888, began work at the factory at New Kensington, near Pittsburgh, producing some 50 pounds of metal a day which sold at $2 per pound. For the first six months they operated according to Hall's original ideas. At the close of this period they lined their crucible with a heavy coat of carbon and adopted internal heating with carbon anodes. This proved successful and their progress was rapid.

It is to be noted that the final amendment to the Hall application, wherein a new specification was filed (corresponding to that of the issued patent) and the claims for copper anodes were dropped and those involving carbonaceous anodes substituted, was filed on September 4, 1888, after he had left the Cowles Company and while he was beginning work at Pittsburgh.

Immediately following Hall's departure from Lockport, the Cowles Company began working on cryolite and alumina, keeping the bath fused by the current. For a period there was an attempt made to merge the two companies and when this failed a suit was brought in 1891 by the Cowles Company against the Pittsburgh Reduction Company for infringement of certain Cowles patents and a counter-suit was brought against the Cowles Company by the Pittsburgh Reduction Company for infringement of the Hall patent. Before the taking of testimony had started in the suit brought by the Cowles Company, the Bradley patent (No. 464,933 dated Dec. 8, 1891, but applied for February 23, 1883) had issued. We shall discuss this patent later. We might note here, however, that it covered one of the chief points at issue between the two companies but did not enter into the controversy until later and after the Cowles Company had acquired it.

The case brought by the Pittsburgh Reduction Co. against the Cowles Electric Smelting & Aluminum Co. was brought to final hearing and on January 20, 1893 (55 Federal Reporter 301) an interlocutory decree in favor of Hall was rendered upon an opinion written by Judge Taft, then U. S. Circuit Judge. The case then went before a Master who eventually assessed an amount of $292,000 against the Cowles Company, damages and profits, in favor of the Hall Company. In view of future developments, to be hereinafter considered, this sum was never paid. The litigation involving the Bradley patent was pending so that no effort was made to push the case before the Master and it dragged along until 1903. A rehearing in the Hall case was then ordered but was never had. After the Cowles Company obtained title to the Bradley patent, as we shall see, it brought suit against the Hall Company on the Bradley Patent, which suit was lost in the lower Court but reversed and decided in favor of the Cowles Company on appeal. We shall take up these various phases of the litigation in order, referring first to the decision in The Pittsburgh Reduction Co. *vs.* Cowles Electric Smelting & Aluminum Co. In this case the Court said in part as follows:

"TAFT, CIRCUIT JUDGE—This is a suit *in equity* by the Pittsburgh Reduction Company against the Cowles Electric Smelting & Aluminum Company, to restrain the infringement of a patent process for reducing aluminum by electrolysis (letters patent No. 400,766) owned by the complainant under an assignment from the original patentee, Charles M. Hall. The patent was applied for July 9, 1886, and was granted April 2, 1889. The defenses to the suit are: *First*, that the patent is invalid for want of novelty; and, *second*, that the defendant does not infringe. * * * *

"The two claims of the patent in suit which are here involved are as follows:

"'1—As an improvement in the art of manufacturing aluminum, the herein described process, which consists in dissolving alumina in a fused bath composed of the fluorides of aluminum, and a metal more electro-positive than aluminum, and then passing an electric current through the fused mass, substantially as set forth.

"'2—As an improvement in the art of manufacturing aluminum, the herein described process, which consists in dissolving alumina in a fused bath composed of the fluorides of aluminum and sodium, and then passing an electric current, by means of a carbonaceous anode, through the fused mass, substantially as set forth.'

"The defendant is said to infringe both these claims. The validity of the first, so far as it covers defendant's process, and of the second, is attacked by the defendant.

"We shall first consider the validity of the claims, and in that connection must refer to the history of the art. It is said on behalf of the defendant that as far back as 1859, De Ville, a famous French chemist, published to the world the process which Hall has included in his patent. De Ville gave a great deal of time to aluminum and its production from its compounds. He was the discoverer of the purely chemical process by which, without the aid of electricity, pure aluminum has been manufactured since his day down to the present time. He also gave some attention to the manufacture of aluminum by the process of electrolysis. In the publication by De Ville on aluminum, its properties, its manufacture, and its uses, in the year 1859, we find this statement:

"'*Aluminum by the Current.*—The same bath of the double chloride of aluminum and sodium can be used for coating with aluminum, especially copper, on which Captain Caron and myself have worked. In order to succeed well, one must employ a bath of the double chloride, which has been carefully purified from all metallic substances by the action of the current itself. When the aluminum which deposits at the negative pole appears pure, one attaches to this pole the piece of copper to be aluminized, and to the negative pole a bar of aluminum.

"'(NOTE—A compact mixture of carbon and alumina, which is transferred into chloride of aluminum gradually and in measure as the deposit of aluminum takes place, keeps the composition of the bath constant for an indefinite period of time.)

"'(The temperature must be kept somewhat below the melting point of aluminum. The deposit takes place with great facility. It is very adhering; but it is difficult to prevent the metal from getting impregnated with double chloride, which attacks it the moment one washes the piece, while, with some precaution, one succeeds. The washing of the piece must be done with much water, and for a long time. Cryolite, in the same way, can be used in this operation, but it must be made more fusible by mixing it with a little double chloride of sodium and aluminum, and with potassium chloride.'

"The process here described is not for producing pure aluminum, but for plating aluminum on copper. It will be seen that the bath which is first and chiefly recommended by De Ville is not a fluoride of aluminum and sodium, but a double chloride of aluminum and sodium, *i. e.*, a double compound of chlorine with each of these metals. Alumina will not dissolve in a double chloride of aluminum and sodium, heated to a fused state. * * *

"Finally, it will be observed that De Ville suggests that instead of the double chlorides of aluminum and sodium, cryolite may be used, but that it must be made more fusible by mixing it with a little double chloride of sodium and aluminum and with potassium chloride; and it is the proposition of De Ville to use cryolite in this way as the main part of the bath, and an anode of carbon and alumina in accordance with the foot-note, which the defendant's counsel and experts contend is substantially the same process as that on which Hall claims a patent.

"We are clear in our opinion that this is not an anticipation of Hall's patent. The primary and most important step of Hall's patent is the dissolving of the alumina in the fused bath of the double fluoride of aluminum and sodium. This happens before any electrolysis occurs at all. When the electric current is applied, it disrupts the alumina, sending the aluminum to the cathode and the oxygen to the anode, where it unites with the carbon anode, and becomes carbonic oxide gas. The bath material in which the alumina was dissolved—that is, the double fluoride of aluminum and sodium—is not affected by the electric current, and the process is made continuous by the simple addition of alumina to the bath. In the De Ville process, however, alumina is not dissolved in the bath. The fused mass subjected to the current is composed of a double fluoride of aluminum and sodium with no alumina in it. No effect comes from the presence of the alumina in the anode until after the electric current is passed through, and the fluoride of aluminum has been disrupted, and fluorine gas has been produced at the anode; then the gas unites with the aluminum of the alumina, and restores the bath, in a manner, to its former state, to be again disrupted by the application of the current. In the one case there is a simple solution of the alumina in a bath and its disruption by the current, the bath remaining constant; while in the De Ville process the bath itself is disrupted, and restored again by the union of one of the disrupted elements with an element of the alumina in the compact anode. In the Hall process there is simply solution of the alumina and electrolysis; in the De Ville process there was electrolysis, followed by what is called an 'electrochemical solution of the alumina.'

"Defendant's counsel and experts, in their effort to make the two processes the same, rely upon the supposition that when the compact anode of carbon and alumina was inserted by De Ville into the bath of cryolite some of the alumina was necessarily dissolved in the cryolite, and so the current electrolyzed that alumina as in the Hall process. In the first place, the experiments made on behalf of complainant demonstrate that a compact mixture of carbon and alumina (which can be made only by the use of some binding material) may be kept in a bath of cryolite for many hours without dissolving the alumina at all. Some counter-experiments by defendant are by no means so satisfactory, because the results may be attributed to the presence of other causes than the solution of the compact anode.

"But suppose it to be a fact that in De Ville's process alumina was dissolved in the bath from the anode, and that thereupon it was electrolyzed as in the Hall process, it was a mere accident, of which De Ville made no note, and which, therefore, we may reasonably infer, he did not observe. Accidents of this character cannot be relied on as anticipations of a patented process (Tilghman *v.* Proctor, 102 U. S. 707,711) when the operator does not recognize the means by which the accidental result is accomplished, and does not thereafter consciously and purposely adopt such means as a process for reaching the result. De Ville did not intend that the alumina should be dissolved in the bath. If he did, why did he unite it with carbon by means of pressure and a binding material? If he had intended to dissolve the alumina, he could not have done anything which would have so interfered with his purpose. Just as Prof. Langley says, such a course would have been as reasonable as it would be for a man, wishing to dissolve a lump of sugar in his coffee, to varnish it or grease it. De Ville did not intend to dissolve the alumina in the bath: *First*, because as we shall see hereafter, he had no knowledge that it would dissolve therein; and, *second*, because, until the fluoride of aluminum had been decomposed in the bath by the current, he did not need anything with which to restore that fluoride to the bath. He felt that it was necessary to have the alumina in a solid form in close connection with the carbon, because, without the strong attraction that the carbon would have for the oxygen of the alumina, the fluorine gas could not get at the aluminum of the oxide.

"De Ville was seeking to electrolyze the fluoride of aluminum, while Hall was seeking to electrolyze alumina. It was a condition precedent to Hall's success that his alumina should be in a liquid state. As we have seen, it was essential to De Ville's process that his alumina should be where it was, *i. e.*, mixed in solid form with the carbon. * * * * * * *

"We have read with care every part of this voluminous record of over 1,500 pages, and, while we are not chemists, we have obtained a sufficient understanding of the principles applied in the Hall and De Ville processes to be entirely confident that they are wholly different, and that no skilled chemist and electrician could have developed the Hall process from the De Ville process without a real discovery.

"The Bell patent of 1861, the Johnson provisional specifications of 1879, the Johnson specification of 1883 and the Graetzel patent of 1884, which are all recorded in the English patent office, and are produced here by the defendant and relied on as anticipations of

Hall's patent, are mere reproductions of the De Ville bath-regenerating process, with various attempts to avoid the difficulties which the process presents in its practical operation. The Graetzel patent—the latest of them—was a failure. This is admitted by the patentee himself, who was a witness in the case. The Johnson specifications were never even proceeded with to the procuring of a patent, and the Bell patent has never been used at all to make aluminum commercially. It is said that one reason why these electrolytic processes, including De Ville's, were not brought into actual use, was that, until within a very few years, there were no dynamos capable of furnishing sufficient electric volume and force to make them practicable for commercial purposes. That the improvements in the machines for producing electricity have greatly facilitated the use of electrolysis as an agent in commercial processes is not to be denied, but the failure of the De Ville process for making aluminum by electrolysis finds a far better reason in the inherent difficulties of the process itself than in the mere expense of electricity. If the De Ville process is operative with the present modes of producing electricity, why does not the defendant adopt it? It is free to any one. The various futile attempts to relieve the De Ville process of its defects are strong proof that it was and is a failure without regard to the development in the making of electricity.

"There are other patents introduced to show anticipation than those which involve the De Ville process, but of these there is only one which, it is seriously contended, discloses the Hall process. This is a French patent of Fuerst, dated August 8, 1884. In this patent the patentee says:

> " 'The process of which I claim the industrial property consists in making the electrolysis of alkaline aluminates, or alkaline earthy aluminates, in the condition of solution or of fusion. For this I take an alkaline aluminate, or earthy alkaline aluminate; that is to say, one of the aluminates of potassium, of sodium, or of barium, etc. Into this body, in solution or fusion, according to the circumstances, I introduce the two poles of an electric current of suitable tension. There is nothing further to do than to receive the aluminum upon the cathodes employed (negative poles), where I collect it in order to melt or utilize it alone or in different alloys for all the industrial uses to which it can lend itself. As to the anodes (positive poles), these anodes can be, according to my desire, soluble or insoluble, and I reserve to myself the industrial property of each of these two cases. While I employ anodes which are insoluble, or are considered such industrially, I reserve to myself to maintain a constant composition in these electrolytic baths by adding to them, as is needed, oxide of aluminum.
>
> " 'The electrolytic decomposition produced by the electric current in an alkaline aluminate, or in an alkaline earthy aluminate, can also be produced equally well whether this salt be in a state of purity or whether several aluminates be mixed together, or whether they be mixed with other salts or foreign materials giving rise to double salts or any secondary combinations whatever, provided, let it be understood, that these salts of foreign materials, whatever they may be, will not produce decomposition of the aluminates employed. It is thus that I reserve to myself to introduce into these baths salts of ammonia, alkaline sulfites, alkaline phosphates, alkaline cyanides, alkaline chlorides, etc., the presence of which seems to have the power of improving the practice of this electrolysis.'

"Later on, Fuerst deposited an addition to his patent, in which he says:

> " 'In this certificate of addition I claim as my industrial property my process for the industrial production of aluminum by electrolysis of alkaline aluminates or alkaline earthy aluminates in solution or igneous fusion. And by 'alkaline aluminates,' I mean every compound into which alumina and alkaline base enter, even if there should enter into that compound one or more of the following bodies, which I shall call 'useful' or 'indifferent' auxiliary bodies: Hydrocyanic acid, cyanides, cyanates, phosphoric acid (pyro and meta), boric acid, silicic acid, hydrofluoric acid—in the state of acids or salts. For me there is aluminate of alkali as soon as there is in a body alumina and an alkaline base, even in the presence of the auxiliary bodies above mentioned, whatever otherwise be the hypothesis that might be established respecting the numerous molecular groupings which might exist or be supposed to exist.'

"An alkaline aluminate is either an aluminate of potassa or an aluminate of soda. Aluminate of soda is a compound of alumina and soda, *i. e.*, of the oxides of aluminum and sodium. It is a triple compound of oxygen, aluminum and sodium. An alkaline earthy aluminate is an aluminate of baryta, strontia, lime, or magnesia; *i. e.*, a compound of alumina with baryta or strontia or the other substances. The three elements of such a compound are oxygen, aluminum, and barytum, or strontium, or the other substances. The theory of the patent—and it is only a theory, as we shall see—is that if these triple compounds of oxygen, aluminum, and some other metal can be reduced only to a liquid form either by aqueous solution or igneous fusion, the current will decompose the compound by depositing the aluminum at the cathode, and will leave the compound of oxygen and the other metal in the bath. By adding alumina to the bath he proposed to restore the aluminate in the bath. Now, the electrolysis of an aqueous solution of the aluminates to produce aluminum is impossible. This is not denied. One-half of Fuerst's patent is demonstrably inoperative. Secondly, so far as the patent calls for the igneous solution of the aluminate of soda, it is also inoperative, because that compound is infusible; and even if it were fusible, the current would precipitate the sodium, and not the aluminum. This is conceded by defendant's experts. Such facts are strong evidence that Fuerst wrote out a theory for the production of aluminum by the current without knowing by experiment whether it would work or not. All the experts in the case agree that he was a very poor chemist. In order, however, to catch future inventors in the toils of his dragnet, he mentions a great many chemical compounds which could be added to his bath of aluminates, and not interfere with its working, and might even aid the electrolysis. It is perfectly obvious that he never tried any of them, and it has not been shown at the bar that one of the innumerable baths proposed by him would, when electrolyzed, produce aluminum. It does appear, however, that if hydrofluoric acid, which is one of the indifferent auxiliary bodies mentioned by Fuerst, be mixed with aluminate of soda so that the mixture shall contain eight times as much acid as aluminate, the acid will entirely decompose and destroy the aluminate, and, after evaporation and fusion, we shall have a bath, not of the aluminate at all, but of the fluorides of aluminum and sodium. If, then, alumina be added, and the current applied, we shall have the Hall process exactly. An experiment of this kind was tried on behalf of defendant, and an account of it is produced in evidence as demonstrating the identity of the Fuerst and Hall processes. It is perfectly evident that no one without a knowledge of the Hall process, and without the deliberate intention of producing Hall's bath, would ever have derived from the Fuerst patent any information leading to such a treatment of the aluminate of soda with the hydrofluoric acid. In the first place, according to Fuerst's specifications, the acid is to be only indifferent or auxiliary. Who would think, therefore, of using eight times as much acid as aluminate in preparing an aluminate bath? Again, one imperative injunction which Fuerst imposes in his specifications was flatly disobeyed in defendant's experiment. He says he has no objection to the introduction of foreign materials as indifferent or auxiliary bodies, 'provided, let it be understood that these salts or foreign materials, whatever they may be, will not produce decomposition of the aluminates employed.' The addition of hydrofluoric acid in excess to aluminate of soda completely decomposes and destroys the aluminate, so that the bath which results after evaporation and fusion has nothing like an aluminate in it, but is a bath of fluorides. In view of the necessity for this flagrant violation of Fuerst's injunction in order to produce the Hall bath, it is absurd to say that the bath can be found described within the four corners of the Fuerst patent. Fuerst does suggest that his bath can be kept constant by adding alumina, but, as his bath is not Hall's bath, he does not thereby come any nearer the Hall patent.

"The other patents pleaded as anticipations of Hall's are even less like it than those already considered, and they were not pressed in argument.

"It is objected to the validity of the Hall process that it is not operative. The argument is that it has no utility, as described in the patent, for three reasons: '*First,* because the fusion of the bath is maintained by external heat, and except with internal heat, produced by the current, the process is worthless and inoperative; *second,* because it contemplates continual interruptions in the process to remove the aluminum already deposited from the cathode, which would make the process too cumbersome and expensive for commercial use; and, *third,* because it does not provide for sprinkling powdered alumina over the top of the bath, to be gradually stirred down into it, without which the depositing of aluminum cannot go on without interruption.'

"There is nothing in any of these claims. The patent disclosed a process and suggested an apparatus by which the process could be operated. * * *

"Now, it may be that the changes in the mode of using the Hall process, indicated in the objections of defendant stated above, from that recommended by Hall in his patent, are great improvements, but that does not in the slightest degree affect the validity of the patent if it appear that at the time when the application was made a new and useful result could be accomplished with the process by the mode which Hall suggested. * * * *

"Hall's process is a new discovery. It is a decided step forward in the art of making aluminum. Since it has been put into practical use the price of aluminum has been reduced from $6 or $8 a pound to 65 cents. This is a revolution in the art, and has had the effect of extending the uses of aluminum in many directions, not possible when its price was high. An effort has been made to show that this reduction in the price is due to the improvements in the application of electricity to the manufacture of aluminum. That the new inventions in the line of producing electric currents of great volume and intensity have contributed to render the Hall process an economical one is true, but without the Hall process the manufacture of pure aluminum must have continued to be a purely chemical one. The Cowles brothers made aluminum alloys by the use of electrical furnaces which they have brought to a high state of perfection, doubtless, and that had an effect to reduce the price of aluminum alloys, and perhaps indirectly affected the price of pure aluminum. The fact was that the price of pure aluminum was so high that its uses were few, and the market for it was small. When Hall's process, however, came into the field of commercial manufacture, pure aluminum was largely substituted for aluminum alloys, and, if alloys are now desired for particular purposes, they are generally made from pure aluminum. Hall was a pioneer, and is entitled to the advantages which that fact gives him in the patent law.

"One other thing must be alluded to before we close the long discussion of the validity of Hall's patent and that is the French patent of Héroult. Héroult has a patent granted to him by the French Government, dated April 23, 1886. The process he described is substantially one of those described by Hall. He uses a fused bath of cryolite in which alumina is dissolved, and from which, by the electric current, aluminum is deposited at the cathode. His anode is of carbon. Héroult made application for a patent to the United States patent office May 22, 1886. Hall did not make his application until July 9, 1886. An interference was declared in the patent office between Hall and Héroult. Hall adduced evidence to show the Commissioner of Patents that he made his invention and put it into operation February 23, 1886. The same evidence has been introduced here. It establishes beyond all reasonable doubt by written evidence that Hall did put his process into successful operation on February 23, 1886. The patent office decided the interference proceeding in Hall's favor, and it was clearly right. There was no evidence, there or here, to show that Héroult discovered the process before Hall, and the fact that Héroult's French patent antedates Hall's application does not affect Hall's right to a patent, because by Section 4887, Rev. St., an inventor's right to a patent in this country is not debarred by reason of the fact that the invention has previously been patented in some other country, provided that it has not been more than two years in use in the United States. The counsel for defendant do not dispute the correctness of this conclusion, but they rely on the Héroult interference proceedings to attack the validity of that part of the second claim in the Hall patent where he specifies as part of his process the use of a carbonaceous anode. It is conceded by them that Hall had used a carbon anode in his process before filing his application, but the contention is that because in his application he alluded to it as having some disadvantages, and did not include it in his first series of claims, which he afterwards amended, and did not finally include it in his claims until Héroult's patent suggested its value to him, and more than two years after his application, he thereby abandoned the use of a carbon anode to the public. No authority is cited to sustain this argument. * * * *

"And now we come to the question of infringement. The evidence leaves no doubt that the defendant company began their manufacture of pure aluminum in January, 1891, with the aid of one Hobbs, who had been the foreman of the complainant company, and engaged for it in superintending the manufacture of aluminum by the Hall process. By that time the complainant company had adopted several improvements in the apparatus described in the patent for working the process. The defendant copied these improvements, as well as the process, and has sought to escape responsibility by maintaining that without these improvements the process was inoperative, and the patent which did not contain them was invalid. Much the same course is taken as to the defense of infringement. The improvements are said to make the present process a different one from that described in the patent, and, therefore, the defendant does not infringe. It is needless to say that a court is not inclined to favor such defenses. There is evidence tending to show that the defendant used Hall's preferred bath, but with the admissions in this case it is unnecessary to consider the issue made on that point. It is admitted that the defendant is using a process in which alumina is dissolved in a fused bath of the double fluoride of aluminum and sodium and in which an electric current is passed through the bath containing the alumina thus dissolved, whereby aluminum is deposited at a carbon cathode and oxygen is released at a carbon anode. It is said, however, that they are not infringing the Hall patent, because the Hall patent does not cover the particular double fluoride of aluminum and sodium which they are using, which is cryolite; and because the Hall process contemplated and called for a fusion of the double fluoride by external means of heating, whereas the defendants are using, to fuse the bath, the heat generated by the resistance of the bath to the electric current. The whole defense of infringement is based on a narrow and wholly impossible construction of the Hall patent. * * * *

"Coming now to consider the claim that the patent does not cover fused cryolite as a bath, it should first be noted that cryolite is a double fluoride of aluminum and sodium, a metal more electropositive than aluminum. It is, therefore, exactly within the words of both claims here in suit. But the contention is that the patentee has disclaimed any right to the exclusive use of cryolite. It is not denied that the patentee had used cryolite as a bath before he made his application, but it is said that he described its use in his first application, and stated, in substance, that it was not so good as a different proportion of the fluorides, and then subsequently omitted reference to it as a bath altogether in his final specifications. * * * *

"The patentee states first the proportion of the aluminum fluoride and sodium fluoride which he prefers for his bath. They are 84 parts of sodium fluoride and 169 parts of aluminum fluoride. Then he proceeds to tell a convenient way for reaching those proportions. Cryolite, as we have said, is an article of commerce. He suggests that the best bath can be made by taking cryolite and adding to it 338/421 of its weight in aluminum fluoride. After this, for the very object of including cryolite, which, as we know, he had used for the purpose, and all other double fluorides of sodium and aluminum which would work, he says that the proportions may be varied. What proportions does he mean? Why, the proportions of the two fluorides, of course. The labored construction that the proportions to be varied are those of the cryolite and the aluminum fluoride is wholly untenable. How may they be varied? Within certain limits. What does that mean? The patentee goes on to state when he says 'that any proportions which may be found suitable may be employed.' Cryolite is now found suitable, and the patentee had found it suitable when he made his application. Could anything be more unlikely than that Hall or his patent solicitor, after they had been successful in an interference controversy with Héroult, whose only bath was one of cryolite, would have framed his specifications with the intention of not including a cryolite bath in the monopoly he was seeking? We think the patent very aptly drawn to cover the use of every double fluoride of aluminum and sodium which can be made to produce aluminum when used as a bath in the Hall process. The proportions are immaterial so long as it is a double fluoride of these metals.

"Next is the defense based on the use of external heat in Hall's apparatus as described in his patent, and defendant's use of internal heat. Hall's claims do not say what heat shall be used to fuse the bath. The process is described as beginning with a fused bath. The argument is that because Hall in his specification describes an apparatus for the fusion of the bath by external heat, therefore he limits himself to a process in which external heat is used, and confers upon the world at large the right to use his process if only some other mode of applying the heat is employed; and this in the face of the words of the patent: 'Nor does this apparatus described herein with more or less particularity form any part of the invention herein.' We do not see how the patentee could have used stronger words to avoid the difficulty in which defendant wishes to involve him. Then, too, we have the refined point that the language of the claim itself excludes the possibility of electric heating because it speaks of dissolving alumina in a fused bath of the double fluorides, 'and then passing an electric current by means of a carbonaceous anode, through the fused mass.' This language is as applicable to electric heating as to any other. The word 'then' is used to indicate that the electrolysis is to follow the solution of the alumina in the fused bath. Now, whether the current or a furnace fire fuses the bath, the current which is passed through the fused mass to perform electrolysis is passed through after the fusion. * * *

"Finally, it is said the defendant does not infringe, because the claim calls for a carbonaceous anode and the defendant uses a carbon anode. 'Carbonaceous' means 'pertaining to carbon,' or 'made of carbon.' It includes anodes made partly of carbon and partly of some other substance, but it certainly covers an anode made all of carbon.

"A decree will be entered for the complainant, finding that complainant's patent is valid, and that the defendant infringes both the first and second claims thereof, and perpetually enjoining the defendant from further infringement, with the usual reference to a master to determine the damages."

Following this decision the defendant took an appeal to the United States Circuit Court of Appeals, but when the appeal was called by that Court in June, 1893, it was dismissed on motion of the appellant. The master then proceeded with the hearing of evidence on the question of damages. Pending this hearing and on June 21, 1894, a motion was made by the defendant to reopen the case, for leave to introduce new evidence, and for a rehearing.

The principal ground for asking the rehearing was that the Court made an error in holding that the De Ville process for making aluminum by electrolysis was not an anticipation of the complainant's process, which error it was purposed to show more fully by subsequent experiments made by the defendant and now offered in evidence.

In the opinion denying the rehearing (64 Federal Reporter 125), the Court said in part:

" 'The evidence which the defendant now seeks to bring to the court's attention consists of certain patents issued to the Cowles Brothers and to Bradley for the reduction of refractory metallic ores by electric currents of great intensity, of quotations from books on chemistry to show that cryolite was known to the art for many years before Hall's alleged discovery as a suitable flux for the reduction of alumina, and of subsequent experiments with the De Ville electrolytic process. Cowles' patents describe an electric furnace into which he introduces the aluminum compound mixed with small granular particles of carbon. An intense electric current is passed through the furnace, and is carried by the granulated carbon. A degree of heat is thereby produced which is intended to smelt the aluminum compound, and to permit an alloy with some other metal, as copper. The Bradley invention contemplates the introduction of the refractory ore into a furnace in which are a movable carbon anode and a cathode. These are placed so near together at the beginning of the operation that, when the current is applied, an electric arc is formed between them. The arc generates enough heat to melt the ore near the anode and cathode, and this melted portion of the ore thereafter carries the current, and gradually effects the melting of the entire mass of ore in the furnace. As soon as any part of the ore is melted, electrolysis is intended to take place. The gist of the invention is the use of the electric current, first to fuse the refractory ore and subsequently to electrolyze it, and disrupt it into its constituent parts. * * * *'

"The Cowles patents, here relied on, were in evidence at the original hearing. The Bradley patents were not introduced in evidence, though well known to defendant and its counsel. The present claim of the defendants is that, as cryolite was a well known flux for alumina, it was mere mechanical skill, and involved no discovery or invention, to use alumina in the Cowles and Bradley furnace (which were patented some years before the patent in suit), with cryolite as a flux; that the use of cryolite as a flux for alumina in the Cowles or Bradley furnaces must result in the electrolysis of the alumina, and the deposit of the aluminum at the cathode and would be the patent process claimed by complainant. * * * *

"The evidence which has been introduced by the defendant consists of the deposition of another expert and further quotations from De Ville's works and from subsequent chemists. Everything which has been shown is carried back to the process for making aluminum by chemical reactions discovered by De Ville, and which was the only practical process until Hall's process by electrolysis was discovered to the world. The evidence is simply cumulative, and does not at all change the conclusion of the Court with reference to its effect. Neither the Bradley nor the Cowles patents contain the slightest suggestion that cryolite should be used as a flux for the refractory alumina, and no evidence is sought to be introduced that any one ever actually used cryolite in furnaces for that purpose. The question presented by the defendant, therefore, is exactly the same as that which the Court originally passed upon, and which was fully argued to the Court. * * * * * It should be remarked, moreover, that the Hall process would still be a patentable process, and the only one known for practically making aluminum by electrolysis, even if it had been well known that alumina would freely dissolve in fused cryolite; for it was conclusively shown by the evidence that never before Hall did it, had alumina been disrupted by electrolysis into its constituent parts. It therefore follows, even if the conclusion already reached is not well founded as to the effect of the evidence touching the solubility of alumina in fused cryolite, that Hall's patent would still be a valid one. * * * *"

After this rehearing was denied the inquisition of damages before the master was continued and about 10 years later, at the beginning of 1903, a second petition for rehearing was made, based on the question whether the commercial success, which had so greatly influenced the Court before, resulted from the practice described in the Hall patent or from some important modification of, or addition to, such process, and whether or not internal heating sufficient to fuse is necessarily incident to the use of the electric current in electrolysis.

The Court granted the rehearing on condition that defendant pay all the costs of the accounting before the master. This rehearing was never actually had. Other matters intervened and subordinated the interest in this case to one in which the former defendant was the complainant and the former complainant was the defendant.

This latter case and one of the conditions precedent to it will be considered next.

(*Part IV to be concluded in December issue*)

2 Rector Street, New York

THE STATUS OF THE CHEMICAL INDUSTRIES IN THE UNITED STATES AT THE END OF 1915[1]

By I. F. Stone

Mr. Chairman and Gentlemen:

When your Chairman asked me to address you on the status of the chemical industries in the United States at the end of 1915, I considered the subject with much trepidation, and felt that if I tried to speak on this topic I would be falling into a teeming cauldron of trouble, as with conditions so abnormal as they are to-day, any one trying to describe these conditions as they are and as they may be for the next year or more, is very likely to prove a false prophet. But notwithstanding these conditions, I will do the best I can, and hope anything I may have to say will be of interest to you.

Right after the beginning of the war there was an immediate demand from all sides for products which had formerly been supplied from Europe, which were apt to be scarce as a result of the war. Buyers attempted to secure as large supplies as possible in order to carry them through during this war. As a result the prices of European products immediately commenced to advance and finally reached the most fabulous figures. Those of us connected with the lines of business dealing in these products then thought that no more abnormal and exciting condition could be possible. But strangely enough, now, a year after the war, conditions are more exciting than ever, in that most of the European products which were then procurable in some quantity, have practically disappeared, while the demand for them still continues to a great extent, but is impossible to meet.

The products manufactured in America, not dependent entirely on Europe, did not follow immediately the advance of the European products, but gradually, as the manufacturers commenced to see how things were developing and what their cost of raw material would likely be in the near future, they advanced their prices until they are now many times the normal in a good many instances.

American manufacturers also began to consider the question of manufacturing larger quantities of what they were already making, and also taking up new products which formerly could be obtained only from Europe, and I am glad to say that a great deal of progress has been made along these lines. I can say definitely that the chemical industries in the United States at this time are developing rapidly, and are in a stronger and better condition at this time, the end of 1915, than ever in the past. Many of the products which are now being manufactured will, in my opinion, continue to be manufactured after normal conditions are again in vogue, and we need no longer be dependent on Europe for such products. I will be more specific as I go along, but simply want to mention the fact at this time that the condition and progress of the chemical industries have developed and are now in a stronger position than ever before.

At the time of the war, or just before the war, the two most important chemical products furnished by Europe to the United States, in this case by Germany, were potash and coal-tar products. We were entirely dependent on Germany for muriate and sulfate of potash, which as you know are used very largely for fertilizers, and in a smaller way for the manufacture of potash products such as bichromate of potash, yellow prussiate of potash, caustic potash, etc., the manufacture of which was dependent on Germany for their supply of raw material. Unfortunately, since the war and the embargo on the shipment of potash to the United States, none now being received, no substitute has been found, and in spite of the reports from the Department of Commerce that large quantities could be secured from a seaweed called kelp, which is in beds or meadows along the Pacific Coast, belonging to this Government, nothing so far has been practically done to recover it. There are reputed deposits of potash in Utah and California, but again nothing practical has ever been done with them, so that at the present time consumers of potash or potash products are entirely dependent on what is left of the German shipments before and right after the war; when these stocks are exhausted, no one knows what can be done.

PRODUCTS FORMERLY OBTAINED FROM EUROPE

BENZOL—On the other hand, as to coal-tar products, it was immediately discovered by investigation in this country, that we had large quantities available if they only could be recovered and developed. In other words, the production of benzol, which is the principal base for the manufacture of most coal-tar products and chemicals, could be immediately increased, and this was and is being done. Some of the large steel works, who formerly did not recover their benzol, are now producing it, and the production, which was about 3,000,000 gallons before the war, is now increased to about 15,000,000 gallons. While, unfortunately, its demand for war purposes in the manufacture of explosives, etc., is so great that even the present supply is not sufficient, and prices are almost too high for manufacturers to use it for the manufacture of ordinary products not connected with war products, yet the fact remains that this product has increased in production over five times, and that this production is here to stay; at normal prices it will be largely used for the manufacture of other products, which will develop with it. There is one use for benzol, for instance, which is practical and sure, and that is its use for motive purposes in automobiles in place of gasoline. It is being largely used in Europe for this purpose, and the only reason it has not been used here has been the limited production and the higher price, it normally being about double the cost of gasoline. With the present immense production, however, the cost has been reduced until now I believe that it can be manufactured and sold at a profit at the proportionate price of gasoline, and that this will be done as soon as the present war is over. When I say proportionate price I mean that careful experiments for automobile purposes show that benzol has a motive power about 25 per cent better than gasoline; it consequently would have 25 per cent advantage at the same price, so that even with benzol 25 per cent more in cost it would still be money value, but I also really believe that it could be actually produced now and sold at the same price as gasoline if it were necessary to do so. Therefore, the importance of this matter is obvious. Not only is the above true, but it is a fact that the use of gasoline

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[1] Address before the New York Section of the American Chemical Society, Chemists' Club, October 8, 1915.

for automobile purposes is so large (100,000,000 gallons) that it is very difficult for the oil companies to produce enough to meet the demand; consequently the entrance of the new product for the same purpose will be very important, and a great relief.

ANILINE PRODUCTS—Another large increase in production through the present conditions, is the manufacture of aniline colors and other coal-tar products in this country, which will be much to the relief of consumers who at the present time are unable to get anything like the supply of colors which they need in the conduct of their business. The five factories already established in this country before the war, are all extending their production to their utmost capacity in keeping with safety for their investment, and I believe that in 1916 the production of aniline colors in this country will be at least three to four times the production before the war. This production could again be largely increased if the manufacturers were sure of some protection from the Government in the way of higher tariff, or Government manufacture of intermediate materials, which would enable them to get the same at the same price as paid by European manufacturers; then again putting into effect some anti-dumping clause, which is now promised by the Government, to prevent the dumping into this country of colors at lower prices than they are sold elsewhere, for the purpose of preventing their development and manufacture here. Whatever the Government may finally decide to do towards the protection of this industry, there remains the fact that a great impetus in the development is already under way, with the hope of Government protection, so that the Europeans will find them strongly entrenched in any event after the close of the war. I am speaking now of the factories already in operation, but in addition to these there are many new factories projected, and some in operation. I have a list of 23 new factories, the last of them with a proposed capital of $15,000,000, and while a good many of these may not materialize, something will surely come of some of them. Up to now none have actually manufactured any aniline colors, but some are operating with a production of some intermediate products like aniline oil, beta naphthol, paranitranilin, etc. Speaking of aniline oil, with the one factory established before the war and the increase in its works since the war, and the number of new factories making or about to make aniline oil, it looks as if the production of this product would be 4,000 to 10,000 tons as against the normal consumption of about 4000 tons; in other words, the proposed manufacture seems larger than the consumption, but as the consumption is also increased, possibly the whole amount projected can be used. At any rate, there will be enough manufactured in this country to take care of the whole consumption, so that Europe need not be depended upon.

CARBOLIC ACID—Another article which has increased largely in production is carbolic acid, the consumption of which in the United States is about eight to ten million pounds yearly. It is true, however, that this has not been available for ordinary use, as most of the new factories have used their product for the manufacture of picric acid, which is sold for war purposes, with the exception perhaps of the works of Thos. A. Edison, who, in an interview, said that his production was about 12,000 lbs. daily, which is used for his records. The point about this article is that practically none of it was manufactured here prior to the war, but it is now produced in large quantities, and after the demand for picric acid is over, for war purposes, then the production can be used for other commercial purposes to the advantage of the country, and so relieve them of depending on Europe for their supply.

NAPHTHALINE—Another article which has increased largely in production here is naphthaline, which is also a coal-tar product. Before the war the production in this country was about 2,500,000 pounds, while now it has increased to something like 7,000,000, perhaps more, the normal consumption being about 9,000,000; the difference between what was produced in America and the total was obtained from England and Germany, which countries hereafter should be practically unable to ship over here, by reason of the new large production here.

BARIUM PRODUCTS—This is about all I will say in connection with aniline or coal-tar products, but as it is obvious that there is a great development taking place in this industry, it is certainly a very satisfactory condition at this time. Leaving the aniline industry, and going to other products not connected with aniline, but the development of which has gone on very quickly since the war, I shall refer first to barytes and barium products. Before the war there had been a yearly average importation to this country of crude barytes of about 40,000 tons coming from Germany, practically all of which was used in the manufacture of lithophone, which was about the only product of barytes made largely in this country prior to the war. There are now six manufacturers who are turning out large quantities, and the business on this product will remain with the American factories. I wish to say, however, that since the beginning of the war no barytes has been coming in from Germany but it has been supplied from mines and deposits in the States of Tennessee, Kentucky, Virginia and Missouri, and possibly some others, so that now the thing to do is to continue to use this American product and keep away from the European barytes. Formerly there was a duty of $1.50 per ton on foreign barytes, but this was reduced in the last tariff to 15 per cent which was only about half the former duty. The German barytes was formerly delivered at a cost of about $5.00 per ton at such ports as Philadelphia and New York, the duty of 15 per cent per ton included, while the American barytes under normal conditions could not compete and can sell now only because no German goods can be secured. The German barytes tests higher in barium sulfate content, averaging about 96 per cent and is almost free from objectionable impurities, so that the consumer obtains a better yield at a lower cost than by the use of the available American barytes, which analyzes only from about 83 to 95 per cent barium sulfate, averaging, say, 92 per cent and much of it is contaminated with iron. The point is then that a duty should be placed on foreign barytes high enough to offset the difference in quality and price, and insure the continued use of American barytes by American manufacturers. In other words, the duty should be advanced, instead of standing at the present rate of 15 per cent per ton. In addition to using the American barytes for the manufacture of lithophone, since the war four or five responsible factories have started up to manufacture other barium products such as chloride of barium, carbonate of barium, hydrate of barium, nitrate of barium, and binoxide of barium, which means an increased use of the crude barytes, giving still further production to American producers, possibly double the quantity formerly used. It is important that American producers should continue to furnish the crude barytes to these factories in spite of the German competition, which is bound to come again after the war, and, as far as I know, an additional duty is the only way it can be done. With the manufacture in this country of the products just mentioned, *viz.*, chloride, carbonate, hydrate, nitrate and binoxide of barium, most of which were never made in this country before successfully, we have a practically new industry created, which will make us independent of Europe in the future, some of these factories being already in operation successfully, and the full production of all of them will undoubtedly be on the market before very long. This makes the barytes and barytes products in this country practically a new industry and one which could be held in the future, and is of great importance.

CARBON TETRACHLORIDE—Another product, the manufacture of which has increased largely in this country since the war, is carbon tetrachloride, which was formerly made exclusively in Germany, but later taken up by American manufacturers, who at the time the war began were probably producing half the consumption here. Since the war they have increased their plants very largely so that they are now supplying all of the American trade, and while still somewhat short of the requirements, new factories are being constructed so that in the end the whole consumption of the country will be manufactured here. This article is perhaps not so well known, but is one of considerable importance, and the consumption is continually increasing in view of the many purposes for which it can be used.

INCREASED PRODUCTIONS IN ESTABLISHED INDUSTRIES

I have given, up to now, the situation on those articles for which we formerly depended largely on Europe, but the manufacture of which has increased largely in this country, which gives a distinct advantage, and increase in our chemical industry. I will, therefore, now refer to a number of products which have always been produced largely in this country, and are not so susceptible to European competition, simply to advise you the condition of the manufacture of these articles and the present and future conditions regarding them.

ACIDS—First in importance, I presume, is the manufacture of such acids as sulfuric, nitric and muriatic, which are the basic materials for practically all of the great chemical industries of the country, and on which there has never been any foreign competition because largely of the heavy expense of transportation, the fact being that American manufacturers were able to make at such prices as rendered importation unprofitable. Up to within a few months ago they were able to supply the demand of the country as usual, but as the war ran on, and the demand for ammunition and explosive purposes became larger and larger, the demand for acids increased to such an extent that at this time the manufacturers are absolutely unable to supply it; as a consequence American consumers find themselves unable to get enough to conduct their business, or, when they do get enough for their present business, they are unable to obtain any additional quantities for an increased business, so that the general situation on acids is very serious at this time. Many increases are planned and under construction, but it will be a number of months before they can be completed, and not much relief is expected for some time to come.

ALKALIS—Perhaps the next important manufactures in volume and value, are caustic soda, soda ash, and bleaching powder, all of which are now manufactured largely in this country, the quantity I am told being from 1,250,000 to 1,400,000 tons of the three products together. For a short time after the war, the manufacturers were able to continue their supply in their ordinary way, but the stopping of shipments from European countries to other countries by reason of the war, led to a demand for American manufactures. to supply this shortage, *i. e.*, countries who had formerly bought from England and Germany and who could not get any from Germany at all and not enough from England, turned to America for their supply; this created a large export business which brought the manufacturers to their full production and has kept them very busy up to this time. By reason of this unusual export demand, and the consequent shortening of stock, the condition of these products means great prosperity to the American manufacturers.

CHLORATES, PRUSSIATES, BICHROMATES—Other products manufactured largely in this country are such products as yellow prussiate of soda and potash, chlorate of soda and potash, bichromate of soda and potash, the manufacturers of which hold the whole American trade, as there are practically no importations from Europe. The manufacture of yellow prussiate of potash, however, is somewhat limited, owing to the inability of manufacturers to get muriate of potash from Germany, so most of them are working almost exclusively on soda, and the paint manufacturers, who are large users, are now trying to make their Prussian blues from soda instead of potash, and if successful, as some of them seem to be, the soda will probably be used exclusively for a long time to come, and there will be no need to depend on European sources for potash.

The manufacture of chlorate and bichromate of soda continue without hindrance, and in increasing quantities, but chlorate and bichromate of potash are apt to be somewhat limited, owing again to their inability to get proper quantities of the muriate potash, these manufactures depending, as far as I can find out, on muriate of potash which was brought in from Germany before or just after the war; while they are fortunate in having enough to go on with their manufacturing, it must be evident that sooner or later their potash products must stop, and soda products substituted wherever possible.

CAUSTIC POTASH—The condition of caustic potash, another potash product, is also interesting; its manufacture was commenced in this country some years ago and up to the time of the war was constantly gaining in importance and production. Unfortunately, however, the manufacturers were dependent entirely on Germany for their muriate of potash; when shipments of this were stopped the factory was compelled to slow up and is now in a position where they can only make that quantity of caustic potash for which they can secure the raw material. Should the muriate of potash be found or produced in America, then this caustic potash could be made largely, but while depending on Germany, muriate of potash must necessarily continue to be limited.

OXALIC ACID—Another important article, the manufacture of which has developed in this country, is oxalic acid. The factory was established some years ago, but up to the time of the war had trouble competing with the European product on account of the reduction of duty in the last tariff, and was not able to develop the production as they wished; but since the war practically stopped the importation of foreign acid, they have taken care of practically the whole American consumption, and will continue to do so to the limit of their ability. They are not yet able to produce the full quantity, so there is a considerable shortage in supplies. Consequently the price is very high, but eventually there is no reason why this product should not be manufactured to the full extent of the American consumption, and the business held here.

There are many other chemical products which are manufactured in this country, but not of enough importance to enumerate separately, so all I can say in finishing is that the status of the chemical industries in the United States at this time is very satisfactory, practically every manufacturer being engaged to the limit of his capacity, and from every indication this prosperity will continue for the following year, for the reason that most of them have made contracts covering their production for this period.

You will notice that in speaking of these various products manufactured in America, I have made frequent reference to tariff, and the necessity of advancing the duty if articles manufactured are to be developed successfully. While I have no intention of making this an address on tariff, at this time, it is so obvious that the tariff should be reformed upward to retain the present manufactures successfully, that I cannot help mentioning it.

100 WILLIAM STREET, NEW YORK

THE BARIUM INDUSTRY IN THE UNITED STATES SINCE THE EUROPEAN WAR[1]

By MAXIMILIAN TOCH

Within the last eighteen years there have been three attempts to establish a barium industry in the United States prior to August 1, 1914, and all three of them were unfortunate failures.

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[1] Address before the New York Section of the American Chemical Society, Chemists' Club, October 8, 1915.

I have investigated the cause of these failures and I find that two of them failed through financial and chemical mismanagement, and one of them was utterly destroyed on account of its inability to compete against foreign competition.

I have been identified with the manufacture of barium products for nineteen years, and while the venture of my firm has been successful it was never very large and was confined to one particular barium salt, which sold entirely on account of its quality rather than its price.

A well known and successful chemical corporation manufactured barium chloride and barium sulfate in New York City a little over fifteen years and made very, very good products. As soon as they turned out reasonable quantities some foreign chemical manufacturers undersold them to such an extent that they found they were unable to continue, and as soon as they were promptly and properly put out of business the foreigners raised their price and have kept the trade ever since, for they taught the American manufacturer the unfortunate lesson that if he were to establish an industry he could not continue it successfully, for the well-known methods of "dumping" large quantities on this market, in addition to the meagre tariff, would soon teach them that they had better not try to make American goods again.

I believe I was the first one who pointed out to the Department of Commerce the shortcoming of our own laws. If a manufacturer in this country attempts to put a competitor out of business by what is known as unfair methods, all one has to do is to file a complaint with the Federal Trade Commission, and the matter is considered at once by its examiners and attorneys; but it is quite obvious that any law made in this country has no sanction against a foreign corporation outside of the jurisdiction of the United States any more than a firm in the United States can be held responsible for its actions according to the law of England, France or Germany.

Two weeks after the great war in Europe was under full swing, my firm recognized that a very interesting portion of its business would be obliterated if we could not obtain barium compounds, and, to be exact, on the 14th of August I started negotiations for a plant at Sweetwater, Tennessee, for the purchase of a large tract of land containing the mineral barite, and in October, just a year ago, the first carload of barium salts was shipped from Sweetwater, Tennessee, to our Long Island City works. From that day to this the plant has been working night and day, additional mines have been purchased, additional installation has been erected, and the corporation is destined to continue to grow, provided our own parsimonious and shortsighted Government will not aid in its final destruction.

There has been a great deal of talk about the dye industry in the United States, and any number of capitalists would invest the necessary funds to-morrow to establish a permanent aniline industry in the United States if they had some kind of assurance that the laws which prohibit unfair competition among ourselves could, in some manner, be made active as against those outside of the United States. There has been a tremendous amount of talk with reference to enacting an "anti-dumping" law. For months this has gone on and up to now it is simply nothing but talk. A great deal of publicity has been given by public officials about the new and wonderful methods and short cuts that have been discovered for the manufacture of aniline dyes out of soft coal to such an extent that it has made the finest and best method for stock jobbing that has ever existed, but there is not a color maker in the United States who can buy for immediate delivery the necessary aniline colors for the manufacture of the lake colors in any reasonable quantity in spite of all this caloric atmospheric discussion.

The barium industry in the United States is not an industry that can be started over night, and among the failures that I referred to one of them was due to the fact that theory took the place of practice. The starting point of the barium industry in the United States is the admixture of barium sulfate and coal, both of which are finely divided, and heated, when the coal converts the entire oxygen into carbon dioxide and leaves barium sulfide behind. The best that any man can do in practice is to get 70 to 75 per cent reaction. In our works we have had runs where we have gone as high as 85, but I think this is purely accidental; yet theoretically the reaction ought to be complete, and the difference between theoretical chemistry and chemical engineering shows that the chemical engineer must devise apparatus which will fit his raw material. Not all barite is suitable for reduction. I have seen disastrous results where calcium fluoride was contained in the barite, and where certain forms of iron would actually produce a paralysis in the reaction, and if up to now I have had a slight measure of success in the manufacture of these compounds it is because I had a knowledge of the manufacture of these compounds prior to the European war. Chemical engineering is 49 per cent chemistry and 51 per cent business acumen. I place the business end ahead of the chemical end for the obvious reason that ability to finance, ability to sell at a profit, and ability to organize are ahead of the chemical reactions involved. You can buy chemical talent, but you cannot buy business efficiency. You can buy barytes of any kind, but you cannot buy in the open market the skill necessary to design this apparatus so that the results will be uniform. I have seen a chemical industry fail because no commercial method of water purification necessary for the reaction was at hand, and if the subject had been carefully studied in advance a fortune could have been saved.

There are many arguments in favor of protection of the chemical industries in the United States, and what I say about the barium industry might refer practically to any other chemical industry, for we have economic differences between Europe and America which cannot be neutralized excepting by a system of protection in addition to an "anti-dumping" law. The freight rate from Tennessee to the coast on our raw material is about $4.50 per ton; that is to say, barite cannot be shipped for less than that except by rail and water, which is a purely theoretical rate, for under existing circumstances a fifty ton car of barium ore cannot be transferred to a steamer unless the material is packed in sacks or barrels. The same material pays $1.80 freight from Bremen to New York, and I have it on very good authority that the rate from the mines of Germany to New York is $2.00 per ton, because the German Government has always fostered its commerce by making special freight rates to export shippers. The railroads in this country, which are, of course, all privately owned, show that they cannot carry trade for less than a fixed sum, owing to their overhead charges; but their overhead charges, which start with the interest on their watered stock, are so great that the shipper must suffer. An 800 mile haul from Tennessee to New York pays $5.00 per ton, and a 1200 mile haul from Missouri to New York pays $4.00 per ton, and the one railroad can afford to do it and the other railroad cannot afford to do it. If the unsuspecting public is forced to support an overburdened railway the Government should, at least, equalize this matter by giving us a protection in the form of tariff.

It is very amusing to read in the papers almost daily about wonderful new methods and new corporations who are about to manufacture all the dyestuffs in the United States and that the shortage of dyes and chemicals necessary for dyeing is about to disappear. It seems to be a pity that any Government employee will lend himself to the exploitation of what looks like stock jobbing schemes without due investigation, for I have not been able to buy any American dyes and no one else that I know of has been able to buy any American dyes such as are generally used for the manufacture of lake colors. On the same sheet of the daily press in which these forthcoming industries are heralded you could invariably find a news item stating that the dye works are practically shut down for want of even simple dyes.

It is at once amusing and pitiable to note the superficiality with which these investigations have been conducted. Three weeks ago in the *New York Times* there appeared a statement that a corporation was formed and very shortly there would be no dearth of the barium chemicals necessary for the manufacture of mordants and paper glazing material, and that a great deal of credit should be given to this concern as being the first concern to manufacture the barium chemicals in the United States, and this just exactly one year after the industry with which I am connected has been steadily turning out hundreds of carloads. It may be true that I have not trumpeted from the housetops that the concern which we started was supplying material with great regularity and of chemical uniformity, and our own Government did not know it. On the other hand, last Spring a friend of mine sent me a clipping from the German commercial reports giving a full description of the materials that we are making, and an item concerning myself, for the investigator had stated that many German chemists would recognize my name as having been president of a section of the Eighth International Congress of Applied Chemistry. This was last Spring, and now our own wide-awake Government is noting the fact that soon there will be a barium industry started in the United States.

One of the very serious drawbacks concerning the new barium industry in the United States is the inability to obtain raw materials and equipment. There isn't a steel factory in the United States that is not working overtime at high prices on appliances which are designed to kill people, and as long as this condition obtains we peaceful manufacturers who want new rotaries and new apparatus will simply have to abide our time and pay exorbitant prices until we can obtain parts of new equipment. To give you a striking example, we needed in one of our works a set of bevel gears made of a special metal, and after waiting twelve weeks, because there was no shop in the country which would pay any attention to us, we finally had to devise gears ourselves as a makeshift. Nor does this apply to machinery alone, for no one could take any contract on any of the barium salts like the chloride, nitrate or sulfate and hope to deliver any part, because it is absolutely out of the question to obtain acids. The people with whom we have dealt for our acids for years are simply either refusing to give us any materials or supplying us with ridiculously small lots at enormous prices, for there are other channels which buy larger quantities and pay more than the peaceful manufacturer can afford.

I mentioned before that industrial chemistry was 51 per cent business and 49 per cent chemistry, and these figures will serve for an example. I was often asked why we located at Sweetwater, Tennessee, and while I did not have very much time to look around I found that that was the logical and strategic location for coal, labor, water and ore, for all of these materials combined are perhaps more reasonable at that locality than anywhere else I know of. In the lantern slides, which I will show you later, you will see a graphic description of the method by which the ore is mined, and in order to make a success of this enterprise the works should be located within such a radius of the mine that the ore can be delivered at a reasonable rate.

The great and only question with reference to the barium industry is, shall this industry live after the European war is over, and I could not answer that categorically without some explanation. It depends entirely upon our own Government. If, on account of the economic conditions which I have described, foreigners are prevented from underselling us, we shall be able to live. Our own manufacturers should get together and understand that the underlying principle of successful business is to have a profitable business, for no argument is necessary on this point other than to prove that when you have a growing and going manufacturing concern, in order to have peace and success you must be able to pay your men a living wage, and you must be able to pay them regularly, for the shut-down of any works in any community, particularly in a small one, produces disastrous results. Innocent women and children are made to suffer through it, so that in the greed of obtaining business manufacturers must understand that cut-throat methods lead to bad results for all parties concerned. I hope that when this war is over the barium industry will be firmly established and protected, as the economic conditions in this country would demand.

What a remarkable difference there is between the enterprise in Japan as compared to the United States. Japan has suffered from a lack of chemicals and dyestuffs just as much as we have, and both Houses of the Japanese Government have voted to subsidize all corporations and firms who are about to engage in the manufacture of chemicals and dyestuffs that were not formerly made in Japan to the extent of $3,500,000, the other half to be subscribed by the manufacturers themselves. It is understood that the subsidy granted is for ten years in order that the new manufacturing concerns may pay a dividend of at least 8 per cent. In addition to this it is understood that the Japanese Government has protected these manufacturers in the form of a high tariff. And that is the country to whom we have been sending missionaries in order to convert them from their archaic beliefs and notions. The old order of things could be reversed and Japan could now send missionaries to this country. It seems to me that it is a great pity that nothing has been done up to now to protect the American manufacturer, for a subsidy from the Government is out of all reason, even though this is at present the richest Government in the world and Japan rated as one of the poorest. As I said before, we can hope to succeed only if our Government will help us from being destroyed by outside influences.

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NOTES ON THE EUCALYPTUS OIL INDUSTRY OF CALIFORNIA

By P. W. Tompkins

Received April 26, 1915

Eucalyptus globulus oil produced in California has been characterized, since the Pure Food and Drug Act, by its nonconformity to the U. S. P. solubility standard in three volumes of 70 per cent alcohol by volume, a guarantee now required by wholesale druggists. While this difficulty can be readily overcome by appropriate fractionating, it is at the expense of a certain portion of the oil and a cost not favorably comparable with Australian conditions of production, with which we must compete.

A feature developed during an investigation of the industry begun in 1912 satisfactorily explains the wide variation occasionally encountered in California oils. A considerable proportion of the trees cut are topped or stumped and the subsequent growth develops a very prolific and dense foilage. Since this new wood is sometimes recut, its influence on the oil derived exclusively from mature trees was determined.

Two sections were found where the new foilage (all globulus) was fairly accurately established as being five years old and was from topped trees while the other was a lot of one year sucker shoots from tree stumps. Each batch of 400 pounds (with a minimum of terminal twigs) was separately distilled and is compared with an oil from trees about 30-years old, with salient characteristics briefly determined as follows:

Oils from *Eucalyptus globulus*, All from San Francisco Co.

	1 year	5 years	30 years
Yield, per cent	0.09	0.21	0.84
Sp. gr.—25°	0.890	0.906	0.908
Sol. 70 per cent alcohol by wt.	15 vols.	3.25 vols.	1.5 vols.
Sol. 70 per cent alcohol by vol.	Insoluble	45 vols.	15 vols.

Obviously the young growth furnishes much less oil in proportion to its age, and its inferior quality would have an undesirable influence on any run. Aside from a very marked difference due to locality, observed in oil from mature trees, this young growth would apparently account for the abnormal character of California oils noted from time to time. What age the new growth must attain before its oil fully matures, would be an interesting subject for those having the opportunity to investigate.

However, the greatest difficulty the California industry has to contend with, aside from the decreased oil recovered, in order to bring its product up to present U. S. P. solubility, lies in the fact (practically speaking) that *Eucalyptus globulus* is the only species available in quantities for oil production at the present time, whereas in Australia more productive species requiring no treatment are almost exclusively worked for export. This subject is best illuminated by quoting some correspondence with Richard T. Baker[1] in part as follows:

"There is, however, an erroneous opinion commercially as to the percentage content of cineol in the oil of *E. globulus* and we have species growing naturally in Australia, the oils of which are much richer in cineol than that species, and also yield a much greater quantity of oil. These oils are now largely exported from Australia, particularly that of *E. polybractea* and are without doubt often sold as the oil of *E. globulus* as indicated by the records of their sale. In fact, there is at the present time very little oil of *E. globulus* distilled in Australia, as it cannot compete against the more prolific oil-yielding species which are richer in cineol.

"It is not usual to fractionate the oil of *Eucalyptus globulus* and the other rich eucalyptol oils so as to remove terpenes and thus endeavor to increase the eucalyptol content, as this is unnecessary with these species. Rectification by steam distillation is all that is necessary. There are species, however, the oil of which can be much improved by fractional distillation and the oil of the New South Wales form of *Eucalyptus amygdalina* can be, and is so increased in eucalyptol, by separating the large fraction boiling at about the temperature of eucalyptol; the other portions of the oil are then used for industrial purposes other than pharmaceutical. As a rule fractional distillation, on a large scale, is not practiced to any great extent in Australia in preparing eucalyptus oils for market."

In addition to this unequal basis of competition they now employ, in Australia, digesters of much greater capacity than are used here, some of these taking over 3 to 4 tons of green leaves to the charge. A most interesting feature was developed in the fact that the raw globulus oil from Australia does not conform to U. S. P. solubility, unless fractionally distilled like our own, though with less loss. Notwithstanding the popular idea that the raw Australian globulus oil is soluble in 3 volumes of 70 per cent alcohol by volume, it is in reality not so.

Through the kind assistance of Mr. Baker this valuable comparison was made possible and in submitting the first sample (broken in transit) Mr. Baker stated in part:

"After considerable difficulty I have at last been able to obtain a sample of the oil of *Eucalyptus globulus* which has been forwarded, etc." "The oil was without doubt distilled from *Eucalyptus globulus* and is typical of the oil of this species as grown in Tasmania."

The second sample also from the Island of Tasmania was received with the comment:

"I have after considerable trouble secured another sample of *Eucalyptus globulus* oil," etc.

Compared with the average raw California globulus oils from San Francisco County, and normal extremes observed from properly distilled leaves (mature) of several localities, the Australian sample gave the following, compared with Baker and Smith's observations of the New South Wales growth:

VARIOUS SAMPLES OF OILS	CALIFORNIA (mature growth) Extremes observed	CALIFORNIA (mature growth) Average S. F. Co.	AUSTRALIA (Tasmania) Our sample	AUSTRALIA (N. S. W.) "Baker & Smith"
Sp. gr. —25°.....	0.901 to 0.909	0.906	0.9123	0.913
Rotation $[\alpha]_D^{25}$(a)	+11.4 to +7.0	+8.0	+3.9	+9.2
Sol. 70% alc. by wt.	1.7 to 1.4 vols.	1.5	1.2	1.5
Sol. 70% alc. by vol.	35 to 13 vols.	15	8	?
Per cent cineol (U. S. P. Method)..	47 to 54	52	54	About 50
Phellandrene test	Negative	Neg.	Neg.	Neg.

(a) Burke and Scalione, THIS JOURNAL, **7** (1915), 206, found $[\alpha]_D^{20}$ +14.4 from trees at Berkeley, Cal.

Inasmuch as most oils from Australia are soluble in 1.8 to 2.2 volumes of 70 per cent alcohol by volume, it would lead to the conclusion, particularly in view of Mr. Baker's statement and our examination of the Tasmania globulus oil, that this species is not represented in the products received here. Merely in support of its scant production, is the fact that of some ten commercial varieties of eucalyptus oil representing the Australian exhibit at the P. P. I. E., *Eucalyptus globulus* is the species conspicuously absent.

It could hardly be considered guess work to assume that the raw globulus oil of Australia is in reality as variable in 70 per cent alcohol by volume as our own, when we compare the constants of the Tasmania sample and those found by Baker and Smith, with over twice the specific rotation and 0.3 cc. less soluble in 70 per cent alcohol by weight, a very great factor of influence on the solubility in 70 per cent alcohol by volume.

Probably the greatest relief home industry could receive, would be by appropriate modifications of U. S. P. requirements in the coming edition, which would permit within reasonably practical limits the use of our globulus oil. The limitations should be sufficiently broad to include normal variations, particularly the solubility in 70 per cent alcohol by "volume." Among the proposed requirements are: specific gravity at 25° —0.905 to 0.925 (same as at present); soluble in 4 volumes of 70 per cent alcohol (undoubtedly meaning 70 per cent by volume); and containing not less than 70 per cent cineol by the resorcin method.

However, if 70 per cent cineol is to constitute the minimum limit, then a gravity as low as 0.905 is unnecessary. The proposed solubility in 4 volumes of 70 per cent alcohol by volume with oils containing a minimum of 70 per cent cineol when the present U. S. P. calls for solubility in 3 volumes with not less than 50 per cent cineol, reverses the needs, since the solubility increases as the cineol increases. That is, if by increasing the solubility to 4 volumes, a more liberal scope is intended, the 70 per cent cineol makes this increase unnecessary and would be a solubility more in keeping with present U. S. P. requirements for 50 per cent cineol. If, on the other hand, a gravity of 0.905 (about that of California's) is meant to include the conditions of a broader range of oils, then the solubility in either 3 or 4 volumes of 70 per cent alcohol by volume is entirely inadequate; nor could a natural oil of this gravity contain as much as 70 per cent cineol.

SUMMARY

I—Appropriate fractional distillation will render California eucalyptus oil up to present U. S. P. requirements, but at the expense of about 15 per cent of the original volume as determined by the present official method. The quality is further improved by the use of caustic soda.

II—Neither Australian nor California globulus oils in the raw state or redistilled in a current of steam are soluble in 3 or 4 volumes 70 per cent alcohol by volume, without fractionating, though this variety is not the competitive species we have to deal with in the Australian trade.

III—If the phosphoric acid method is retained as official in the coming U. S. P. the minimum per cent of cineol should remain as at present, 50 per cent. If the resorcin method is

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[1] Curator of the technical museum of Sydney. Baker and Smith—"A Research on the Eucalyptus with Particular Reference to Their Essential Oils" published by the Government of Australia in 1902.

adopted the minimum per cent of cineol could be placed at 60 per cent without materially interfering with the California industry, but the proposed 70 per cent would entail a loss of at least 30 per cent of the original volume to bring it up to this figure if determined by the phosphoric acid method, and about 15 per cent loss if the cineol is determined by the resorcin method.

IV—The solubility in 70 per cent alcohol by volume should be increased to at least 18 instead of 4 volumes, if the average raw California oils redistilled in a current of steam without fractionating (Australian practice) are to be included on an equal basis with the foreign importations.

V—The by-product oil of refining as well as the crude (if sufficiently reasonable and in quantities) might be utilized for the separation of metallic sulfides by the flotation process, as extensively applied in Australia and to some degree here with the foreign oil.

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CURRENT INDUSTRIAL NEWS

MARKET CONDITIONS

Active inquiry, alarming advances and continued scarcity have been noted for many chemicals and raw materials during the past month. A combination of peculiar circumstances that is unprecedented has resulted in a very sensitive market. Conditions prevailing are such that quotations seldom hold for more than a day. Manufacturers having a surplus of some articles are selling at a handsome profit and small fortunes have been realized by speculators. Manufacturers in many lines have been compelled to alter their formulae to allow for the use of substitutes where the necessary materials are abnormally high or impossible to obtain.

These unusual conditions are justly attributed to the European war but there are some features only remotely connected with the hostilities of nine nations and some causes entirely independent of the European situation. The cessation of all shipments from Germany is probably the largest single factor although home requirements and embargoes have prevented England, France, Russia and Italy from permitting or making normal exportations of many commodities. The war has resulted in a scarcity of freight room on ocean vessels, higher rates for freight and insurance, and a general uncertainty.

Contracts for the manufacture of explosives for the entente Allies are responsible for the scarcity and high prices noted for carbolic, picric and nitric acid, nitrate of soda, glycerine, ether, benzol, toluol, aniline oil and many other coal-tar derivatives.

Nitrate of soda in normal times enters commerce principally for fertilizer. During the early months of the war, large stocks were held at most receiving and shipping points; with a large portion of foreign markets closed, operations in Chile were brought almost to a standstill and prices were very low. As the war continued the demand for nitrate increased and operations at producing points were increased to normal with quotations slowly advancing. About a month ago unusually large purchases in Chile were credited to explosive interests and great strength developed. Then came the last big slides in the Panama Canal and the subsequent announcement that Canal traffic would be suspended indefinitely. This resulted in the market reaching $3.00, the highest point in several years. Cargoes will now come "round the Horn" and increased freights and the growing scarcity will probably result in further advances.

Glycerine was not attracting any particular attention in the markets a month ago and C. P. was available at 26 cents and "dynamite" at 25 cents. To-day it is difficult for any buyer to secure supplies, except subject to delays, although the current quotation is 60 cents per pound for both C. P. and dynamite grades. One consumer was in the market a few days ago for 1,500,000 pounds. The high prices and active demand have resulted in an increased production and this is proving the most profitable business to most of the soapmakers. Vegetable oils are being purchased in large quantities for this purpose and linseed oil appears to be the only one not profitable.

Inability to secure supplies of potash salts has resulted in abnormal prices for many chemical compounds and the elimination of some from the market. Dealers and brokers have verily scoured every corner of the globe in search of old stocks of German potash, so that now little muriate or sulfate is to be had except from third hands who naturally look for a substantial profit. Muriate of potash, however, is available in a limited way at $245 to $250 per ton.

Scarcity of the various forms of potash has found the soda compounds being substituted to such a degree that sharp advances are common. Yellow prussiate of potash is to-day selling at 87 cents per pound as compared with $11\frac{1}{2}$ cents before the beginning of the war. Red prussiate of potash commands $2.60 to $2.75 per pound as compared with 21 cents in July, 1914. These advances result in such an increased use of soda compounds that prussiate of soda is to-day selling at 44 to 45 cents per pound, as compared with $8\frac{1}{2}$ to $8\frac{3}{4}$ cents during July, 1914. Bichromate of potash is held at 21 cents per pound as compared with a normal price of 7 cents or less. Bichromate of soda is selling at 15 to 16 cents and quoted at 14 to $14\frac{1}{2}$ for winter deliveries as compared with $4\frac{3}{4}$ cents before the war. Chlorate of potash is to-day bringing 37 to 38 cents per pound as against the old price of $7\frac{1}{4}$ to $7\frac{1}{2}$ cents. Chlorate of soda sells to-day at 22 to 23 cents per pound on spot with contracts for future deliveries commanding 14 to 16 cents f. o. b. works, as compared with $7\frac{1}{4}$ to 8 cents a little more than a year ago.

In July, 1914, aniline oil was selling at $10\frac{3}{4}$ to 11 cents per pound while to-day one can sell at 95 cents to $1.50 per pound, according to urgency of the consumer's requirements. Aniline salts at present command $1.35 to $1.50 per pound whereas 9 cents was the average price fifteen months ago. Benzol is now selling at 80 to 90 cents; not long ago many orders were filled at $1.25 to $1.40; just a little over a year ago, plentiful supplies could be had at 22 cents.

The foregoing are only a few instances of the upward trend; further reference to a price current of July, 1914, shows the price of oxalic acid at $7\frac{1}{4}$ to $7\frac{1}{2}$ cents; this week sales were recorded at 45 and 46 cents and quotations are as high as 50 cents. We have depended on Germany and other foreign sources to supply us with oxalic.

Lead products were very high for a time but present levels are close to normal. Zinc products are high and scarce but not as high as a few months ago. Quicksilver sells to-day at $93 to $95 per flask as against a normal figure of $75.

One of the principal reasons for the extreme high prices is that manufacturers have contracted for their entire output and consumers not fortunate in holding a contract for regular deliveries are at the mercy of second hands who are reaping profits while they may. How long present conditions will prevail is a question that interests many. The duration of European hostilities is the most important factor although this may be overcome, to a degree, by more generous supplies of needed raw materials. Further advances are predicted and are to be expected for many articles. A sudden cessation of the war would result in declines but well-posted factors claim that it would be many months before the old levels were restored.

But few commodities have remained practically unchanged during the fifteen months of the war or have found lower levels. These include most of the vegetable oils, naval stores and a small list of articles that are of domestic origin.

Unusual opportunities have been opened for American chemists and manufacturers and it is pleasing to note that most of them are being grasped and developed.

THE VALIDITY OF EMPLOYMENT CONTRACTS

In a Court of Appeals of England, an interesting decision has been rendered, affecting the relation of employer to employee. An account of this appears in the *India Rubber Journal*, for April 24, 1915, as follows:

> A draughtsman in a certain plant reached the position of chief draughtsman, and eventually became head of the sales department in the company. On attaining this position he signed an agreement, that if he left the company's employ he would not for seven years thereafter enter the employ of any other concern in the same business. This agreement he did not observe, but shortly after leaving the first firm he was engaged by a second,—a competitor. Action was taken by the first company to compel its former employee to observe his agreement. The trial Justice rendered a decision in favor of the employee, on the ground that such an agreement was against public policy and could not be enforced. The case was appealed, and the Court of Appeals upheld the original decision on the same ground stating: "On the one hand it was in the public interest that a covenant should be observed and enforced but it was also in the public interest that a man should be free to use his skill and experience to the best advantage."

This decision is of considerable interest to Industrial Chemists in the United States as numerous attempts have been made by employers limiting or seeking to limit the usefulness of former employees. It would seem that the German industrials had found a better way out of the difficulty by keeping a former employee on half salary for five years in case he promises not to enter the employ of a competitor within that time.

DEVELOPMENT OF SOUTH AUSTRALIAN COPPER AND OIL RESOURCES

A recent bulletin of the South Australian Official Intelligence Bureau at Adelaide states that with the object of making a systematic attempt to provide a means whereby the relatively low-grade copper deposits of South Australia may be turned to profitable commercial account, the Government, with the assistance of the University of Adelaide, has secured the services of a metallurgist for a period of two years, to experiment in hydrometallurgical methods.

The direct aim of this experimental work will be the drawing up of working schemes, wherever practicable, for the treatment of ores that have been tested.

A small experimental plant will be erected in South Australia for the carrying out of a series of tests on the copper ores of the State. Should this experimental work be successful, it is hoped that some of the copper mines of the State which are now lying idle or being worked on a restricted scale, will become important contributors to the annual output of copper.

With reference to the offer made by the Government of South Australia of a bonus of £5000 to the person or body corporate first obtaining from a well or bore, situated in the State, 100,000 gallons of crude petroleum containing not less than 90 per cent of products obtainable by distillation, the South Australian Department of Mines reports the results of investigations into the supposed oil-bearing areas of South Australia. It is stated that the prospects of obtaining a supply of petroleum from the districts investigated are very slight indeed. The far Southeast carries a faint possibility which is based on surmise and no evidence. In the interior of the continent the Coonanna Bore gave some signs which have resulted in nothing tangible when tested by further boring. Shales which may produce oils on distillation are also known to occur in these far removed areas, but difficulties connected with climate, water, and communication will probably cause a long period to elapse before the actual value of these interior deposits can be adequately tested and made known, and will largely discount their economic value for many years.

With regard to lignite beds which underlie areas nearer the coast, the analysis of average samples shows them to be of such poor quality that all attempts to utilize them at present would result in financial loss.

The report concludes with the suggestion that the offer of the bonus should be extended so as to include oil obtained by distillation from shales or lignites.—D. G. ANDERSON.

GLAUBER'S SALT FROM THE CASPIAN SEA

The Russian *Agricultural and Land Gazette* calls attention to the immense riches in Glauber's salt, soda, etc., to be found in the Karabagasky Gulf, of the Caspian Sea. This gulf had been particularly investigated already in 1897, when it was shown that there are beds of Glauber's salt, unequaled in purity by that ordinarily prepared in the chemical factories. It is estimated that the deposits there contain 181,000,000 pounds of the salt. The production of this article, as well as others which do not lie deep in the gulf, might be undertaken with simple excavating machinery. Naturally, stress is laid on the value of exploiting these deposits at the present moment, as their exploitation would free Russia of dependence on foreigners in respect to Glauber's salt, and might bring about a complete revolution in the soda business.—A.

BRITISH BOARD OF TRADE

During the month of August the British Board of Trade have received inquiries from firms in the United Kingdom and abroad regarding sources of supply for the following articles:

Acetanilide
Borax
Caliper gauges
Combs, vulcanite
Cobalt oxide
Cobalt chloride
Cocaine
Codeine
Glass chimneys
Glass ampoules
Graphite
Iceland spar
Irish moss
Leather, imitation
Lithophone
Magnesium ribbon
Milk, powdered
Mica
Manure, artificial
Manganese dioxide
Naphthaline
Papier mâché
Phenazone
Phenyl salicylate
Rubber sponges
Silica
Stearine pitch
Steel wool
Tungsten powder
Wood wool
Acetyl-salicylic acid
Asbestos and asbestos goods
Boxes with aluminum lids
Compressed air brushes for applying color
Copper tubes, elliptical section
Felt tape for insulated wire covering
Glass capsules for holding soup
Glass tubes for pills
Hexamethylenetetramine
Horn and tortoise shell waste
Incandescent mantles
MACHINERY FOR MAKING:
    Metal clips
    Press buttons
    Safety pins
    Tin cans
    Stamping ivory nut buttons
    Grinding barytes
    Wire-stitching
Paper for wrapping crystallized fruit
Phosphor bronze, hard spring quality
Pumice, rough lumps
Quebracho extract
Resin, powdered
Spirit level bubbles as used in clinometers
Steel cylinders for compressed gas
Steel rings, polished, 1 in. diameter
Steel wire for bookbinding
Tinfoil for packing
Ultramarine-blue powder

Firms who may be in a position to supply any of the above articles are asked to communicate with the Director of the Commercial Intelligence Branch, Board of Trade, 73 Basinghall Street, London, E. C.—A.

POTASH DEPOSITS IN SPAIN

Extensive deposits of sulfate and carbonate of potassium are reported to have been discovered at Cordova. A Belgian company paid £20,000 for an option on the bulk of the deposits and has now taken over all the land on which it had an option. Sinking operations to determine the extent and commercial value of the deposits are still being carried on, however, and the deposits are, so far, not being worked as a commercial success, being hardly out of the experimental stage.—A.

CHINA CLAY

The export of China clay from Cornwall has been prohibited except to British possessions or protectorates, but supplies will be allowed under licenses granted by the War Office Department. It is feared, however, that licenses will not be granted very easily and difficulty may arise in Cornwall from which about 60 per cent of the clay exported goes to the United States. Representations are being made to the British Government that the export trade in China clay with the United States should be continued as what is sent there is so used that there is little chance of it finding its way into the hands of the enemy. —A. McMillan.

PRODUCTION OF ZINC

According to the *Chemical News*, the Swansea Vale Spelter and Zinc Smelting Works, Wales, which were built and until recently controlled by Germans, have now been taken over and will henceforth be under the control of an entirely English group, with the consent and approval of the British Government. The works, which have been erected upon the most modern principles, are likely to be enlarged on an extensive scale and the development of the zinc ore deposits of Great Britain is likely to be encouraged. The head of the group is Mr. R. Tilden Smith, who is associated with many important enterprises of the kind, including the zinc mines of Burma from which he intends to obtain supplies of metal. The plant is exactly similar to those which have been erected in Germany, Belgium and France, and was part of the organization which enabled the owners to dominate the zinc market of Europe.—M.

CONCRETE PLUGS IN MINES

Concrete plugs have been used by the Warwickshire Coal Company at their colliery at Keresley, near Coventry, England, in order to stop the inrush of water and as a result of the treatment in one shaft practically no water is being met with instead of 2,600 gal. per minute before applying this treatment. A concrete plug 20 feet thick was placed at the bottom of each shaft which cut off completely the water and allowed boring (twelve holes in each shaft) for the injection of cement from the bottom of the shafts.—M.

TUNGSTEN MARKET IN GREAT BRITAIN

According to the *Engineer* the British-made tungsten powder market is being well supplied from various sources. The element tungsten is recovered from Wolfram ore, well nigh inexhaustible supplies of which are available within the British Empire. In order that these ore reserves may be better handled, the Government has assumed control of them and now it is understood that it has decided to control the tungsten supplies. One of the first evidences of this is seen in the regulation of values. After the outbreak of war the price quoted was about four times the price of pre-war days. It appears that under the Government scheme the market is fixed at $1.24 per lb. for tungsten powder and 4 cents less for ferro-tungsten. In some quarters, exception is taken to this arrangement, but if the scheme gets into working order, high-speed steel makers should benefit considerably.—M.

THE NEED FOR PETROL SUBSTITUTES

Professor Vivian B. Lewes, lecturing recently at the Royal Society of Arts, London, stated that the need of petrol substitutes lay principally in the fact that in the next, say 50 years, the natural supplies would be diminished to such an extent that the depth from which the oil would have to be obtained would render them practically commercially unavailable. The world's total output of crude oil, which in 1914 amounted to 97 million tons, had, he said, increased at an annual rate of only 6 per cent for the past three years and the amount of petrol obtainable from the crude oil could be put at only 10 per cent. America itself used no less than 1,200,000,000 out of the world's 1,700,000,000 gallons and England in 1914 imported 120,000,000 gallons, making a total of 1,320,000,000 gallons, leaving only 380,000,000 gallons for the remainder of the world.—M.

HARDENED FILTER PAPER

Writing in the *Pharmaceutical Journal* on a process for toughening ordinary filter paper, Mr. W. R. Rankin states that a substitute for the smooth, hardened filter paper of German make may be prepared by dipping the best English filter paper very quickly in nitric acid (sp. gr. 1.4), draining and washing in running water until most of the acid is removed. The remainder is neutralized by immersion in 0.5 per cent ammonia solution. The paper is afterwards washed thoroughly, pressed and dried at 100° C. When dry, the paper is subjected to the same treatment. Excessive temperatures should be avoided in drying, as the cellulose is nitrated to some extent. A shrinkage of about 10 per cent should be allowed for. An inferior substitute may be got in a single operation by dipping filter paper in a mixture of 65 parts sulfuric acid (sp. gr. 1.84) and 35 parts nitric acid (sp. gr. 1.42).—M.

BORNEOL FROM WASTE SULFITE LIQUOR

In a note on the distillation of waste sulfite liquor given in *Papierfabrikant*, the isolation of inactive borneol is described. When methyl alcohol is distilled from waste sulfite liquor, a heavy oil is recovered towards the end of the distillation. This oil contains furfural and, when cooled, deposits small crystals. These are washed and crystallized from petroleum ether. They form large, flaky, translucent crystals which have a camphor-like odor. The results of elementary analysis, the melting point, boiling point and the fact that it forms bornyl chloride on treatment with phosphorus pentachloride, confirm the substance as being inactive borneol.—M.

THE WORLD'S COAL SUPPLY

A matter of great interest, though not an acute problem at present, is the probable nature and extent of the coal fields yet in reserve and of known locality. According to the 12th International Congress of Geology held at Toronto, close on to 72 billion tons are still available.

	Anthracite (in million tons)	Common coal (in million tons)	Lignite (in million tons)	Totals
America	22,542	2,271,080	2,811,906	5,105,528
Asia	407,637	760,098	111,851	1,279,586
Europe	54,346	693,162	36,682	784,190
Australia	659	133,481	36,270	170,410
Africa	11,662	45,123	1,054	57,839
Total	496,846	3,902,944	2,997,763	7,397,553

Naturally it is impossible to claim great accuracy for the above estimates and for years to come we may reasonably expect the discovery of further deposits that will compensate for current consumption.—M.

NOTES AND CORRESPONDENCE

NOTE ON "CHEMISTS' PROTECTIVE ASSOCIATION"

Editor of the Journal of Industrial and Engineering Chemistry:

The recent proposal [THIS JOURNAL, 7 (1915), 798] to form a "Chemists' Protective Association" seems to call for discussion.

In the first place, as a loyal and interested member of the American Chemical Society, the present writer feels that this Society *is* intensely practical in that membership in it tends to increase the efficiency of its members as chemists.

Any organization which has this effect is bound to react favorably upon those responsive to such influences, and this reaction will ultimately find expression in a fatter pay envelope, if we wish to look at that aspect of increased efficiency, apart from all other considerations.

In the second place, any attempt to define a "chemist" as a person having "spent four or more years at an accredited technical school or University in the didactic and laboratory study of all the regular branches of chemistry, and who has received formal statements represented by a degree or degrees that the prescribed courses of study have been satisfactorily completed," will justly be doomed to sure and certain failure. There are many men who have had all the advantages of technical training but who are lacking in the essentials of interest, clear-headedness and broad-mindedness. Such men, it is needless to say, will never be successful chemists. On the other hand, many so-called "quasi chemists" and "laboratory boys," in the proper surroundings, may develop into very competent and efficient workers. Men of this type, with a little encouragement, succeed in spite of a lack of technical training.

In the third place, the proposed salary scale based on a classification of chemists as "Assistant Chemists" and "Chief Chemists" is artificial, academic, and impractical. Most chemical work in a technical laboratory naturally falls into one of the following classes: (*a*) analytical and control; (*b*) factory development; (*c*) fundamental research. A given piece of work may overlap the boundaries of two or even all three of the above divisions. It also happens that the aptitude of different men for different classes of work is different so that some men make good research men, but would be dismal failures in developing processes on a commercial scale, or in analytical work. The training a man has had, of course, plays a large part in making him fit or unfit to do a given class of work, but the native ability of the man himself, his enthusiasm, his "chemical instinct" and "optimistic activity," or lack of these qualities, must surely be considered in placing men in laboratories and in their subsequent advancement.

Looking at this same subject from another point of view, it so happens that, in different laboratories, the relative importance of the above classes of chemical work may differ. Thus, one company may require but little research work and a great deal of analytical and control work; or a great deal of factory development work may be necessary to put into practical operation the results of some fundamental research. Now, this question of the class of work required in any one instance, the native ability of a man and his attitude toward his work are all inseparably connected with his advancement. Of these, his ability to take on added responsibility; to grow and become more efficient; the proper receptive attitude which enables him to apperceive new ideas and meet new conditions, are of the greatest importance, in determining his salary. If he is capable of doing low-grade routine work, and nothing else, it is but right that he should receive a low salary; for, the lower the grade of work required, the easier it is to train an ordinary boy to do the work. In all laboratories that the writer knows of, there is considerable routine work which is easily mastered by any boy of average intelligence and no trained man should be foolish enough to waste his time doing such work; if such a chemist should be found he could rest assured that the business man would pay him no more than he would pay the laboratory boy, because he would be worth no more.

On the other hand, some of the so-called "laboratory boys" or "quasi chemists" happen to be above the ordinary in intelligence and men of this sort will rise and justly so.

It seems to the present writer that these questions of the definition of "chemist," the classification of chemists, and the salaries they are to receive, should be, and are generally in actual experience, answered on the basis of the efficiency of the individual worker: $1200.00 per year is too much to pay to an inefficient chemist who lacks interest and enthusiasm in his work and whose growth is at a standstill; $5000.00 per year is too low a limit to place for an active, energetic and growing head of a laboratory. In fact, why place any limits in either direction?

In conclusion, the writer feels that any proposal to form a "Chemists' Protective Association" should be most emphatically opposed by all chemical workers and chemically trained men who have the real interests of the chemical profession at heart.

CLEVELAND, OHIO WILLIAM C. MOORE

ON GERMICIDAL EFFICIENCY OF DENTAL CEMENTS

Editor of the Journal of Industrial and Engineering Chemistry:

A letter written by W. V-B. Ames, representing the W. V-B. Ames Company, referring to my paper on "The Germicidal Efficiency of Dental Cements" (THIS JOURNAL, 7, 195), appeared in THIS JOURNAL, 7, 723.

In the first paragraph Ames misquotes from my article as shown by comparing the following excerpts:

From Ames' letter—THIS JOURNAL, August, 1915:	From "The Germicidal Efficiency of Dental Cements" by Paul Poetschke, THIS JOURNAL, March, 1915.
In THIS JOURNAL, March, 1915, appeared an article by Paul Poetschke, entitled "The Germicidal Efficiency of Dental Cements." The concluding paragraph promised reports on the comparative strength and other properties. These reports would be, as Mr. Poetschke states, "of more immediate interest to the dental profession," and it will be by clinical experience in the hands of the dentist that the subject will receive its final analysis and probably be reported mainly in the dental literature.	FROM INTRODUCTION: "It is possible to measure the germicidal efficiency of material of this character in precisely the same way as it is possible to measure the germicidal efficiency of disinfectants. It is therefore obvious, especially in view of the varied composition of these cements, that a thorough investigation of this subject is of immediate interest to the dental profession, as well as to chemists and bacteriologists who may be called upon to report on the germicidal power of dental cements. The object of this paper is to show the comparative germicidal power of these cements, and also some of the compounds used in their production." FROM CONCLUSIONS: "The germicidal efficiency of a dental cement is merely one of the properties which are of importance. Many other physical properties such as resistance to saliva, hardness, crushing strength, constancy of volume, etc., are also of importance. The relation of the germicidal efficiency to these other properties is being investigated in this laboratory, and the results will be published in future papers."

The exact quotations from Ames' letter and my article are given because of the evident intention of Ames to minimize the importance of this investigation to the dental profession. As far as the clinical significance of these bacteriological tests is concerned, one can hardly fail to appreciate that a copper cement is used primarily on account of its germicidal properties. Therefore, such tests must be relied upon to secure first-hand evidence in this direction. In surgical practice the carbolic coefficient of a disinfectant is accepted as a reliable criterion of its efficiency, and likewise the germicidal efficiency of a copper cement can be accepted as a reliable indication of its clinical efficiency.

In connection with this letter Ames reproduces an illustration which he says is "the one illustration criticized by Mr. Poetschke, the criticism being mostly that only comparative inhibition has been shown and that there had been no bacterial count." Here again, Ames attempts to minimize the criticism which I made of his paper. This illustration was the only one which appeared in Ames' paper, and therefore the only one which was open for consideration. However, reference to my article will show that every statement made by Ames, bearing directly on the subject of germicidal properties, was fully quoted and criticized in my paper.

A copper cement exerts its germicidal effect in two distinct stages, that is, both in "plastic" and "set" condition. It thus exerts an initial sterilizing influence when inserted and a permanent germicidal influence after it is set. The initial sterilizing influence is not of much importance because the local application of a liquid disinfectant would accomplish the same purpose. The permanent germicidal action of the cement in the set condition is the main desideratum. Ames apparently fails to appreciate these facts in referring to "a cavity from which all infected material had not been removed, there being, for this reason, a need of a sterilizing filling."

Ames further states in his letter: "It is well known by the dental profession that a real copper oxid cement will furnish the means of sterilizing of such an infected area, and yet furnish a mass, after proper setting, which is so highly insoluble that it would show potency only in the hardened state equal to the best of the so-called copper cements tested by Mr. Poetschke, including that made by the L. D. Caulk Co., the employers of Mr. Poetschke." In this statement Ames intends to create the impression that the dental profession accepts and knows these statements to be facts, when as a matter of fact, the dental profession does not know or accept these statements. Nowhere in the dental literature does any authoritative statement or proof appear which substantiates this assertion. Ames insinuates that the black copper cement of his manufacture, which is referred to in the illustration, is not more highly germicidal in the hardened state, because of its "highly insoluble" character. My experimental data, which I shall shortly publish, shows that this black copper cement, made by Ames and referred to as so "highly insoluble," suffers the greatest relative loss in strength in saliva of twelve copper cements which are on the market to-day with but one exception, and that is also a black copper cement. Ames has attempted to claim the authority of the dental profession for a statement which has absolutely no basis in fact.

The ambiguous statements made by Ames regarding the "cement-making property of the cuprous and cupric oxids" have absolutely no relation to the subject under discussion, because a copper cement need not contain cuprous or cupric oxides in order to possess the requisite germicidal power, strength, resistance to saliva, or any of the properties demanded of a copper cement. It is immaterial whether a copper cement contains cuprous or cupric oxide, but it must contain some compound of copper which in itself has sufficient germicidal power to secure the required germicidal efficiency in the finished cement. Of course, it is understood that any copper cement must have, in addition to germicidal power, secured by the addition of a potent compound of copper, all of the other physical and chemical properties demanded of a product of this character. My investigations, which will be published shortly, show that cuprous and cupric oxides need not be depended upon for any cement-making property which they may possess, and their elimination from copper cements, as Ames infers, is greatly desired by the dental profession, because of the limitation imposed on copper cements containing cuprous and cupric oxides on account of their color.

Ames refers to a "difference in germicidal efficiency between a real copper oxid cement and an oxyphosphate of zinc cement, more or less modified by a copper compound, each in the freshly mixed state" as being "so great that results may be shown which appeal to the naked eye instead of needing microscopical count." I have referred at length, in my paper, to the fallacy of testing a dental cement for its germicidal efficiency in the "freshly mixed state," and I wish to emphasize the statement contained in my original article, namely, that the tests employed by Ames failed to give any information concerning the germicidal efficiency of a dental cement. I fail to understand Ames' reference to "microscopical count" except to assume that Ames does not know that the colonies of bacteria are not counted by the aid of the microscope, but by the naked eye.

In the last paragraph of his letter Ames describes his illustration in part by stating, "No. 2, a zinc oxid cement carrying a small percentage of oxid of mercury (yellow)," whereas his original paper read, "No. 2, oxyphosphate of zinc containing a small percentage of mercurous iodid." This certainly cannot be a typographical error and it is needless for me to call attention to the radically different character of these two compounds of mercury. This does not inspire confidence in the illustration. Ames states that this illustration shows "inhibition of bacterial growth" and that the "inoculation was overwhelming," yet reference to the illustration fails to show a single colony of bacteria, notwithstanding the fact that it is supposed to depict an inoculated Petri dish. The illustration is plainly not a photograph and naturally cannot be accepted as showing "inhibition of bacterial growth." Even if it did show what is intended, the test is of no value as regard establishing germicidal efficiency of the material.

In conclusion, I wish to point out that Ames does not present a single criticism of my article but merely repeats and adds to the statements contained in his article which I fully criticized as being of pseudo-scientific character. In this letter additional erroneous statements and conclusions are presented by Ames which have no basis in fact and are unsupported by any evidence whatever. The illustration presented is not worthy of consideration, because it does not show "inhibition of bacterial growth," and further, the description does not agree with Ames' original article.

PAUL POETSCHKE, *Director*

DEPARTMENT OF CHEMISTRY, THE L. D. CAULK CO.
MILFORD, DELAWARE, AUGUST 26, 1915

THE "PURE FOOD LAW"

Editor of the Journal of Industrial and Engineering Chemistry:

For the past nine years it has been of profound interest to follow the prosecutions under the "Pure Food Laws." Being of an analytical mind I carefully went into the fundamental conceptions of the law, its rigid legal and logical interpretation, its effect upon commerce, international and interstate, and finally I examined the details of the administration of this, and attempted to ascertain, as nearly as possible, whether justice or injustice had been inflicted on those who had come into contact with the law as defendants in criminal prosecutions brought by the government.

My conclusions, after the nine years of investigation, are that in many cases the defendants pleaded guilty to the charges brought against them, although they were neither *legally* nor *morally* guilty, merely to escape the annoyance of a trial, always distasteful to busy and enterprising men of affairs.

To confirm my conclusions, I shall give a strict legal interpretation of the law in such particulars as are of essential importance for the views advanced above.

Section 3 of the law *does not* confer legislative power on the three secretaries—

Purely executive powers are conferred, and therefore the rules and regulations of the Bureau of Chemistry are of a similar nature, as rules and regulations enacted by the district attorney of New York County for the purpose of administering the *executive* functions of his office.

The legal reasoning is clear, for Congress cannot delegate *its legislative* powers to any other body. Notwithstanding the efforts of certain editorial writers on various trade journals, no decision of the Supreme Court exists which contradicts this statement: Congress cannot delegate its legislative powers.

The second point I wish to make is the definition of the word, Drug.

Many definitions for various purposes may be found but for the enforcement of the "Pure Food Law" only the definition given by that law may be used. It has surprised me how rarely, if at all, courts have attempted to dwell on this point. Section 6 of the law reads: That the term Drug "as used in this Act shall include all medicines and preparations recognized in the United States Pharmacopeia or National Formulary for internal and external use, and any substance or mixture of substances intended to be used for the cure, mitigation, or prevention of disease of either man or other animal."

Let us examine this definition. The phrase, "all medicines and preparations *recognized* by the United States Pharmacopeia or National Formulary for internal or external use" requires that the prosecution must prove

1—That it is medicine or preparation.

2—That said medicine or preparation is *recognized* by the United States Pharmacopeia.

3—That it is so recognized for internal or external use.

What a medicine or preparation is the law does not say, and that question is, therefore, a question of fact for the jury and not a question of legal definition.

But whatever a medicine or preparation is, it must be such as is *recognized* by the United States Pharmacopeia or National Formulary.

Now examining the history of pharmacopeia for the last two centuries throughout all parts of the world, it must be agreed upon that no pharmacopeia recognizes any commodity that does not comply with its tests or definitions.

Therefore, an article of commerce that does not comply with such tests or definitions cannot be defined as a drug under that part of the laws, phraseology as stated above.

Furthermore, such medicines and preparations must be recognized for internal or external use. There are many substances in the U. S. P. *not* recognized for internal or external use. Clearly, then, if an article of commerce does not come under the phrase, of the laws' definition, as stated above, we may proceed and examine the definition as it continues: "And any substance or mixture of substances intended to be used for the cure, mitigation or prevention of disease of either man or animal."

We note the words "intended to be used," and also the words, "for the cure, mitigation or prevention of disease."

A mere intent to be proven by the circumstances of each particular case is all that is necessary, but it must be a direct intent; of indirect intent the law takes no notice.

Furthermore, it must be for the *cure, mitigation* or *prevention* of *disease*. Therefore, articles of commerce used or intended to be used for manufacturing, even though the products resulting from such manufacture are substances intended to be used as a cure, mitigation or prevention of disease, do not come under the law. I call particular attention to this fact, because the administration and enforcement of the law has not always been conforming to this legal conclusion, especially in cases of importations of articles of commerce into United States.

Finally, Section 11 of the law deserves some notice. The notice has gone abroad as evidenced by numerous communications on the subject, that executive officers have absolute legislative and executive powers to exclude goods offered to be imported into this country, that there is no legal remedy for any wrongful acts on the part of such executive officers.

Let me say that the writ of injunction still exists, and that it is applicable where "there is no adequate remedy at law," a statement that should be considered where an executive officer is about to usurp legislative functions.

1221 Lincoln Place, Brooklyn, N. Y.
September 3, 1915

Louis Hogrefe

PAPER REAGENT

Some time ago the writer noticed the following article in a drug journal, the name of which he does not at present recall: "Detection of Wood Pulp in Paper:

"The *Repertoire de Pharmacie* republishes from the *Bulletin de la Société de Chemie Belge* a reagent for the detection of wood pulp in paper. It consists of paranitroaniline, 20 cgm., dissolved in 80 cm. distilled water and sulfuric acid, sp. gr. 1.767: 20 milligrams. This has the advantage over the alcoholic solution of phloroglucin and hydrochloric acid in that it may be kept for years. It is also preferable to anilin sulfate, which, although it is stable, gives with wood pulp a yellow color, which is not easily distinguished by artificial light. The paranitroaniline colors wood pulp orange to brick-red, and it can be seen under any mode of illumination. The reagent has the further advantage of yielding a color to unbleached cellulose."

The writer made up this reagent and tested different papers and pulps with the following results:

1—Mechanical wood-pulp → dull orange to orange.
2—Unbleached sulfite pulp → very similar to above.
3—Bleached sulfite pulp → yellow.

The writer found that the distinction between the mechanical wood pulp and the unbleached sulfite pulp was not very marked and proved rather troublesome when testing unknown pulps.

The writer has discovered, however, a method whereby the detection is made plainer. He used the above reagent in conjunction with ammonium molybdate solution and obtained the following results:

1—Mechanical wood-pulp → bright reddish orange.
2—Unbleached sulfite pulp → dull orange, faint; or faint brown.
3—Bleached sulfite pulp → colorless.

By experiment it was found that when the ammonium molybdate is applied first and then the paranitroaniline, better results are obtained. Also, on waiting for one or two minutes, the reaction is complete and the distinction is then very sharp.

The above certainly seems to be an excellent test for distinguishing paper pulps, and could no doubt be applied to certain other substances as a test for presence of lignin.

National Cloak & Suit Co.
New York City
September 28, 1915

Wm. J. Schepp
Chief Chemist and Textile Expert

THE SCHOOL OF CHEMISTRY OF THE UNIVERSITY OF PITTSBURGH

The new professional School of Chemistry of the University of Pittsburgh began its work on Spetember 27, 1915, under the deanship of Dr. Raymond Foss Bacon, Director of the Mellon Institute of Industrial Research. A prescribed four-year undergraduate curriculum leads to the degree of Bachelor of Chemistry; the staff of instruction includes the regular faculty of the University and fellows from the Mellon Institute of Industrial Research who are especially qualified in various theoretical and technical branches. This combination gives the new school the opportunity to offer not only the usual undergraduate and graduate courses in chemistry and technology, but also specialized work under men who are experts in specific American industries. In addition, thirty special lectures by prominent chemists and technologists in the Pittsburgh district have been arranged for the academic year 1915–16. Attendance at these lectures is required of the student body but they are also open to the public.

The professorate of the new school is constituted as follows: Alexander Silverman, M.S., Professor of Chemistry and Head of the Department of Inorganic, Analytical and Physical Chemistry; David S. Pratt, Ph.D., Professor of Chemistry and Head of the Department of Organic, Sanitary, and Micro-chemistry; Samuel R. Scholes, Ph.D., E. Ward Tillotson, Jr., Ph.D., and Edmund O. Rhodes, M.S., Professors of Applied Chemistry; Benjamin T. Brooks, Ph.D., Professor of Chemical Engineering; William A. Hamor, M.A., Professor of Chemistry; Henry A. Kohman, Ph.D., and Harold Hibbert, Sc.D., Professors of Applied Organic Chemistry; Leonard M. Liddle, Ph.D., and R. Phillips Rose, M.S., Professors of Organic Chemistry; Lester A. Pratt, Ph.D., Professor of Inorganic Chemistry; and C. C. Vogt, Ph.D., Professor of Physical Chemistry. Thirteen assistant professors and ten instructors complete the teaching staff of the school.

NOTE ON ARTIFICIAL ZEOLITE—PERMUTITE

Editor of the Journal of Industrial and Engineering Chemistry:

Having just returned from an extensive tour through Cuba and the Eastern States, I find, on looking through the past numbers of the Journal, in the March number of this year, on page 325, a communication by Mr. Stephen Kocsor: "Preliminary Note on Artificial Zeolite—Permutite."

This note is interesting to me for the reason that for the last two and one-half years I have been, and am still, working along identical lines while Research Chemist of the Kieselguhr Company of America, Van Nuys Building, Los Angeles, California.

During my travels I have freely discussed the results obtained with zeolites thus produced, with a great number of sugar and other chemists whom I have visited professionally during this time.

968 West 42nd Place
Los Angeles, Cal., July 15, 1915

F. R. Bachler

PERSONAL NOTES

Prof. Julius Stieglitz has been made chairman of the department of chemistry of the University of Chicago to succeed the late Prof. John Ulric Nef.

The One Hundredth Meeting of the Syracuse Section of the A. C. S. was in the form of a banquet held on the evening of October 23rd, at "The Onondaga," Syracuse.

The positions left vacant in the chemical department of Syracuse University by the resignations of Dr. E. H. Archibald and Dr. C. R. Hoover have been filled by Dr. Charles Warren Hill and Prof. Merritt R. Grose.

The October Meeting of the St. Louis Section of the A. C. S. was held on the 11th, with the following program: "The Industrial Utilization of Peat," by Mr. H. E. Wiedemann; "An Apparatus for the Determination of the Degree of Fineness of Paint Pigments," by Mr. E. J. Sheppard, of the National Lead Company.

The American Cyanamid Company announces the opening of its general offices at 200 Fifth Avenue, New York City, on October 1, 1915. The Nashville and Buffalo offices were discontinued on that date.

W. A. Gibb Maitland, Government Geologist and Director of the Geological Survey, Western Australia, has been appointed President of the Royal Society of Western Australia for the Session 1915–1916.

A $500,000 electrochemical plant will be built at Niagara Falls by the E. I. du Pont de Nemours Powder Company, of Wilmington, Del.

The former students of Prof. C. E. A. Winslow have given a dinner in his honor on the occasion of his entrance into his new position of professor of public health at Yale University.

Dr. Walter D. Bonner, formerly assistant professor of chemistry in the School of Mining in Queen's University, has recently been appointed Head of the Department of Chemistry in the University of Utah.

The British Association held its annual series of meetings at Manchester at the beginning of September. The inaugural address entitled "The Common Aims of Science and Humanity" was delivered by the President, Professor Arthur Schuster.

Dr. H. H. McGregor, formerly instructor in chemistry in Adelbert College, and recently appointed instructor in biochemistry at the medical school of Western Reserve University, has died of typhoid fever in Toronto, Canada.

Charles Hallet Wing died on September 13th in his eightieth year. In 1870 he became professor of chemistry in Cornell University, from which place he went in 1874 to Boston to accept a like position at the Massachusetts Institute of Technology, where he remained for ten years.

Advance notice has been received of the Joint Meeting of the New York Sections of the American Electrochemical Society and the Illuminating Engineering Society, to be held on Nov. 11, 1915 at the United Engineering Societies Building. The subject for the evening is, "Electrical Phenomena in Vapors and Gases." The following papers will be read: "Unstable States in Arc and Glow," by Walter G. Cady, Wesleyan University; "Gaseous Conductor Light," by D. McFarlan Moore, Edison Lamp Works; "Electric Arc in Complex Vapors," by W. A. Darrah, Mansfield, Ohio. The papers will be illustrated by experiments and lantern slides.

It has been decided that the next meeting of the American Electrochemical Society will be in the Spring in Washington, D. C., with the subject of *Electrochemical Research* as a special order of discussion.

Mr. Lincoln Burrows spoke on "Glycerine" before the October 18th Meeting of the Rochester Section of the A. C. S.

Prof. Albert Victor Bleininger, who has been in charge of the Ceramic Section of the U. S. Bureau of Standards at Pittsburgh since 1912, has been appointed Professor and Head of the Department of Ceramic Engineering of the College of Engineering of the University of Illinois.

The winter meeting of the American Institute of Chemical Engineers will be held in Baltimore, Md., January 12–15, 1916. A number of papers on recent and important developments in some of the chemical industries of the United States are being arranged for. Excursions will be made to a number of the important chemical industries, of which Baltimore is the center. The experimental laboratories of the Johns Hopkins University and the Naval Academy at Annapolis, Md., will be visited.

The following appointments have been made to the faculty of Case School of Applied Science: Melville F. Coolbaugh, South Dakota School of Mines, assistant professor of chemistry; Lawrence G. Wesson, Harvard University, instructor in organic chemistry; Carl H. Wilson, Harvard University, instructor in chemistry.

Mr. William Roy Mott, of the National Carbon Company, addressed the Cleveland Section of the A. C. S. on "The Use of the Flaming Arc in Paint and Dye Testing," on October 11th.

The October Meeting of the Pittsburgh Section of the A. C. S. was held jointly with the American Electrochemical Society on October 21st. Mr. F. T. Snyder, of the Snyder Electric Furnace Company, Chicago, presented a paper on "Electric Furnaces for High Temperature Industrial Chemical Work."

Dr. Edward Bartow, University of Illinois, spoke on "The Purification of Sewage by Aeration in the Presence of Activated Sludge," before the Chicago Section of the A. C. S., on October 15th. The regular November meeting of the Section will be held on November 12th.

The Bausch & Lomb Optical Company received, at the Panama-Pacific Exposition, four Grand Prix on optical instruments, balopticons, engineering instruments and range finders. The first division, called optical instruments, is comprised of seven classes and covers the company's ophthalmic lenses, microscopes, parabolic and mangin mirrors, field glasses, microtomes and magnifiers. A Medal of Honor was awarded Bausch & Lomb photomicrographic apparatus. Bausch & Lomb-Zeiss photographic lenses received the Gold Medal.

Dr. Frank K. Cameron, of Washington, has severed his connection with the United States Bureau of Soils in order to devote his time exclusively to consulting practice, especially in the field of fertilizers and soil chemistry.

In addition to the regular November Meeting of the Philadelphia Section of the A. C. S., a special joint meeting with the Franklin Institute will be held on November 4th. The program will consist of an illustrated lecture on "The Turpentine Industry in the Southern States," by Dr. Charles H. Herty, President of the American Chemical Society.

Mr. Phillip J. Krell will manage the new plant of the International Oxygen Company now being erected at Verona, Pa. (Pittsburgh district), for the accommodation of its customers in that vicinity.

Dr. Frank K. Cameron gave an illustrated lecture on "The Development of a Dynamic Theory of Soil Fertility," before a joint meeting of the Philadelphia Section of the A. C. S. with the Franklin Institute on October 28th.

GOVERNMENT PUBLICATIONS

By R. S. McBride, Bureau of Standards, Washington

NOTICE—Publications for which price is indicated can be purchased from the Superintendent of Documents, Government Printing Office, Washington, D. C. Other publications can usually be supplied from the Bureau or Department from which they originate. Consular Reports are received by all large libraries and may be consulted there, or single numbers can be secured by application to the Bureau of Foreign and Domestic Commerce, Department of Commerce, Washington. The regular subscription rate for these Consular Reports mailed daily is $2.50 per year, payable in advance, to the Superintendent of Documents.

GEOLOGICAL SURVEY

Contributions to Economic Geology, 1913. Part II. Mineral Fuels. Marius R. Campbell and David White. This volume is one of the regular series published annually, being made up of six distinct papers. "The papers included are of two classes: (1) short papers giving comparatively detailed descriptions of occurrences that have economic interest but are not of sufficient importance to warrant a more extended description; (2) preliminary reports on economic investigations, the results of which are to be published later in more detailed form. These papers are such only as have a direct economic bearing, all topics of purely scientific interest being excluded."

The following papers make up the volume:

Oil Shale of Northwestern Colorado and Northeastern Utah. E. G. Woodruff and D. T. Day. 23 pp.

Oil and Gas in the Western Part of the Olympic Peninsula, Washington. C. T. Lupton. 60 pp.

The Moorcroft Oil Field, Crook County, Wyoming. V. H. Barnett. 22 pp.

Possibilities of Oil in the Big Muddy Dome, Converse and Natrona Counties, Wyoming. V. H. Barnett. 14 pp.

Geology and Oil Prospects in Waltham, Priest, Bitterwater, and Peachtree Valleys, California. R. W. Pack and W. A. English. 42 pp.

The Coalville Coal Field, Utah. C. H. Wegermann. 24 pp.

Analyses of Rocks and Minerals, from the Laboratory of the United States Geological Survey, 1880–1914. F. W. Clarke. 370 pp. This bulletin summarizes and tabulates approximately 2800 analyses of rocks, clays, soils, meteorites, minerals, etc. The data are arranged principally under the heading of the geographical source of the rock or mineral, thus giving for the various localities the characteristic analyses which have been found by determinations in the Survey's laboratories.

Nitrate Deposits in Southern Idaho and Eastern Oregon. G. R. Mansfield. Bulletin 620–B. This is an advance publication of one of the contributions to Economic Geology, 1915. Part I.

The article describes the deposits of nitrate which have recently been discovered and discusses the origin, extent, volume, and utilization of these nitrates. Although the author urges further investigation in the hope that a commercially valuable deposit may be found, he does not consider the outlook very promising at the present time.

Field Apparatus for Determining Ash in Coal. C. E. Lesher. Bulletin 621–A. 12 pp. An illustrated description with operating directions are given for a form of portable apparatus which has been designed to meet the needs of field parties of the Geological Survey and which has been in successful use for three years.

Resins in Paleozoic Plants and in Coals of High Rank. David White. Professional Paper 85–E. 32 pp. "This paper presents evidence that anthracite and bituminous coals, as well as those of lower rank, originated as peats, and that the plants from which the Paleozoic coals were formed were in part resin-bearing—in fact, the resin-bearing elements in the coal-forming floras of the Carboniferous period, to which the bituminous and high-rank coals of most parts of the world belong, are perhaps as numerous as in the floras from which the coals of later epochs were formed. The author points out the general prominence of resins in the Mesozoic and Tertiary coals of low bituminous and inferior ranks and contributes observations as to the disappearance of these coals in the course of carbonization under regional metamorphism, the resins disappearing at about the stage of carbonization at which the coking quality of the coals is well developed. After reviewing the evidence as to resin and gum secretion in the petrified fragments of Paleozoic plants described by several paleobotanists, he describes, with illustrations of both common and unique materials, the presence of abundant resinous matter in Paleozoic coals of medium bituminous or lower ranks."

The Fauna of the Batesville Sandstone of Northern Arkansas. G. H. Girty. Bulletin 593. 170 pp., 11 plates.

Geology and Mineral Deposits of the National Mining Dis-

trict, Nevada. WALDEMAR LINDGREN. Bulletin 601. 58 pp., 8 plates, 8 text figures.

SMITHSONIAN INSTITUTION

The following reprints from the 1914 Report of the Smithsonian Institution are now available:

Modern Views on Constitution of the Atom. A. S. EVE. Publication 2326. 9 pp.

Examination of Chinese Bronzes. JOHN C. FERGUSON. Publication 2347. 6 pp.

Form and Constitution of the Earth. LOUIS B. STEWART. Publication 2324. 6 pp.

Explosives. EDWARD P. O'HERN. Publication 2331. 26 pp.

Some Aspects of Industrial Chemistry. L. H. BAEKELAND. Publication 2330. 25 pp.

Recent Developments in the Art of Illumination. PRESTON S. MILLAR. Publication 2350. 18 pp.

DEPARTMENT OF AGRICULTURE

Prickly-Pears as a Feed for Dairy Cows. T. E. WOODWARD, W. F. TURNER AND DAVID GRIFFITHS. Journal of Agricultural Research, 4, 405–50. This includes a considerable number of analyses of feeding materials tested.

Phosphorus Metabolism and Lambs Fed a Ration of Alfalfa Hay, Corn, and Linseed Meal. E. L. ROSS, M. H. KEITH AND H. S. GRINDLEY. Journal of Agricultural Research, 4, 459–74.

COMMERCE REPORTS—SEPTEMBER, 1915

Owing to shortage of the supply of **wood pulp**, efforts are being made to develop its production in New Zealand. (P. 1090.)

The Australian Government has acquired control of the stock of **sugar.** (P. 1096.)

The British **pottery** industry is suffering from lack of a supply of **bone,** formerly obtained from Argentine. (P. 1115.)

A municipal **gas** works at Akkrum, Holland, is now preparing gas from a mixture of two parts coal and one of **peat.** (P. 1151.)

Australian **zinc** concentrates are now being shipped to Galveston *via* the Panama Canal. (P. 1151.)

A large order for **zinc** for Hongkong has been placed in the United States. (P. 1166.)

Bounties are to be paid upon all **zinc** produced in Canada from Canadian ores. (P. 1169.)

The **sewage disposal** plant of Nottingham, England, is being operated with a profit of several thousand dollars per year. (P. 1204.)

Experiments are being conducted on the Pacific coast upon the use of pulverized **coal** as fuel on steamships. (P. 1209.)

Recent rulings of the Steamboat Inspection Service regarding passenger vessels include:

Loaded **shrapnel shells** may not be carried.

Hydrochloric acid is subject to the same conditions as sulfuric acid.

Only non-inflammable, non-combustible, non-explosive **metal polishes** may be carried, or used as stores.

Tanners' oil of high flash point may be carried.

Quicklime, alone or mixed with magnesia, may not be carried.

Sesquisulfide of phosphorus, and **red phosphorus** may be carried in iron drums on deck, while **yellow phosphorus** may not be carried.

Moving picture **films** may be carried under the same conditions as celluloid. (P. 1230.)

Prospecting for **petroleum** is being carried on in Zulia, Venezuela. (P. 1235.)

Efforts to discover a source of mineral **phosphates** in New Zealand have not thus far been successful. (P. 1257.)

The present annual profits of some British **dye works** are nearly 50 per cent of the paid up capital. (P. 1261.)

The Brazilian **piassava** fiber industry is described. (Pp. 1262–3.)

The West Indian **lime-juice** industry also produces **calcium citrate** and oil of limes. (P. 1291.)

Kapok or "vegetable wool" obtained from a tree of the genus Eriodendron in Ecuador and the Dutch East Indies is used extensively in the United States for mattresses, etc. (P. 1294.)

Among the various substitutes for **absorbent cotton** now being used in Europe, are "lignin" (made of pine cellulose), "cellulose wadding" (made from chemical wood pulp), "bog moss" (*Sphagnum cymbifolium*). (P. 1306.)

The mineral products of Alberta, Canada, include **gold, coal, natural gas, cement, clay products, lime, sand-lime brick** and **stone.** (P. 1312.)

In New South Wales, Australia, a Patents Investigation Committee has been appointed to assist in the development of inventions likely to be of general public utility. (P. 1323.)

The output of **pig-iron** from Penhsihu, China, has increased, the demand being large since it is low in phosphorus and sulfur. (P. 1325.)

With the increased production of lemons in California, it is soon expected to produce all the **lemon oil** and **citrate of lime** required in this country. (P. 1345.)

The **nitrate** industry of Chili shows marked improvement. (P. 1250.)

The Austrian government has taken over entire control of the **petroleum** industry of Galicia. (P. 1353.)

An old **porcelain** plant in Kiangsi, China, is to be conducted as a national model porcelain factory. (P. 1360.)

The mineral resources of Manchuria include **gold, silver, lead,** and tungsten. (P. 1362.)

The **flax fiber** industry of Canada is being revived. (P. 1366.)

A committee has been appointed to improve the quality of the **copra** produced in the Philippines. (P. 1389.)

A marked increase is noted in the imports of American **drugs** into Hongkong. (P. 1393.)

The price of **Epsom salt** in America has increased from $1.10 to over $5.00 per 100 lbs. (P. 1395.)

A plant is being established in Russia for the manufacture of **tanning extracts** from oak and chestnut bark. (P. 1405.)

The **oil** from the **coyol** palm nut found in Central and tropical South America, has the following constants: Sp. gr. 25° = 0.9136; melting point, 25°; "clouding point," 17°; free acid, 1.69; saponification number, 246.2; iodine number, 25.2; Reichert-Meissl number, 5. The oil would be valuable for food products, but its commercial extraction is not profitable, owing to the difficulty of cracking the nuts. (P. 1407.)

The Netherlands Government has assumed charge of all purchase and import of **fertilizers** into Holland. (P. 1417.)

The Swedish **match** industry is suffering from a lack of suitable asp wood, formerly obtained from Russia. (P. 1423.)

A factory to manufacture **paper** from an aquatic plant is being erected at Maracay, Venezuela. (P. 1435.)

New **gold** deposits have been discovered in Thunder Bay District, Ontario. (P. 1492.)

A marked shortage of photographic chemicals is noted in Nottingham, England. (P. 1497.)

The following special supplements, containing statistics and other information, were issued during September:

Germany, Frankfurt—6*e*.
Roumania—12.
Spain—15*d*.
Bahamas—22*b*.
Martinique—28*a*.
Turkey—18*c*.
China, Hankow—52*i*.
Samoa—62*a*.

BOOK REVIEWS

An Introduction to the Study of Physical Metallurgy. By Walter Rosenbain, B.A., D.Sc., F.R.S. D. Van Nostrand Co. 6 × 9. 390 pp., illustrated. Price, $3.50 net.

This book is divided into two parts, the first section dealing with the structure and constitution of metals and alloys, the second with the properties of metals as related to their structure and constitution.

Taking up first of all the microscopic examination of metals, the author discusses the preparation of specimens, and the microscope used, then the microstructure of pure metals and alloys. This is followed by the thermal study of metals and alloys, the thermal diagram and its relation to the physical properties. Typical alloy systems are exemplified by the lead-antimony, lead-tin, zinc-aluminum, zinc-copper, tin-copper and certain ternary alloys, followed by the iron-carbon system.

The second part reviews the mechanical testing of metals, the effect of strain on the structure, heat treatment, mechanical treatment and casting, and ends with a discussion of defects and failures.

To review the contents of this book thoroughly would take many pages, because the author has covered the broad field of metallography so thoroughly and so well. This is particularly true of the presentation of the comparatively new ideas on the structure of metals, the effects of strain and of annealing, developed from Beilby's Amorphous Metal Theory. The elongation of the crystals when strained, the production of slip-bands and their nature, the formation of amorphous layers and the hardening of metals by cold work, twin structure, fracture under tensile, shock and alternating stress conditions, and the amorphous cement theory are all most clearly set forth. The criticisms therefore must be on minor points and not on the broad lines of the book.

For example, on page 13, after mentioning the names of the earlier workers, Sorby, Martens, Osmond, Werth, Grenet, Charpy, LeChatelier, Heyn, Wust, Tammann, Andrews, Arnold, Roberts-Austin, Stead, Howe and Sauveur, the author says: "The fact that the present author was privileged to count Roberts-Austen and Osmond among his personal friends and that Arnold and Stead are still actively at work in this field, serves to show how very recent the whole development has been." Besides Arnold and Stead, many of those mentioned are "still actively at work" as current literature in the metallographic field amply proves.

On page 21, in describing the preparation of specimens for polishing, "the necessity of gripping the specimen in the vise" to file, is mentioned. Most people grip the file in the vise and rub the surface of the specimen on it.

On page 31, the reference to etching reagents is too short and might with advantage be expanded.

On page 162, as Ruff's work is mentioned, reference ought also be made to that of Wittorf and of Hanemann.

The photomicrographs are all well chosen and excellently executed, but lose somewhat in not having a title beneath each, rather than having it given only in the list of plates.

In conclusion, the only changes that could be suggested is in the section on the thermal diagram which should contain those diagrams showing partial solubility in the liquid state. A short classification according to solubility in both liquid solid states would help.

The author has succeeded in preparing an excellent book, interesting to the student, valuable to the metallurgist and engineer, and full of ideas for anyone engaged in metallographic research. It is a book that can be recommended to the general reader also, because the style is simple and the ideas are clearly and logically developed and followed. With the growing interest in metallography as a method of testing it will undoubtedly prove very popular.

W. Campbell

The Chemistry and Technology of Printing Inks. By Norman Underwood and Thomas V. Sullivan. New York: D. Van Nostrand Co. 139 pages, 9 illustrations. Price, $3.00 net.

Messrs. Underwood and Sullivan have charge of the Ink-making Division, Bureau of Engraving and Printing, United States Treasury Department, Washington, D. C. This book is an attempt "to give in a brief and practical but yet scientifically correct manner the many facts concerning the raw materials and finished products, used in this industry which they (*i. e.*, the authors) have collected during a number of years of laboratory work and manufacturing experience."

The introduction gives an excellent explanation of the meaning of the many technical terms used. The authors venture the assertion: "A general chemical analysis will not, however, convey any idea of the essential composition of the ink." This is wholly in accord with the opinions of those connected with this industry.

Part I, pp. 15–38, discusses laboratory apparatus, methods of analysis of raw materials and the physical testing of pigments.

In Part II, the manufacture of the various classes of ink-making materials, such as dry colors, oils, etc., is described in detail. The properties of each of these substances as ink-making materials are given very conveniently in tabular form. The constants for raw linseed oil which are given do not agree with those adopted by the American Society for Testing Materials. In giving the specific gravity no temperature is mentioned and the well-known constants of index of refraction, acid number and unsaponifiable matter are not included in the table given.

Part III, pp. 104–132, is devoted to the manufacture of printing ink. No formulae are given and the subject is taken up only very generally. Much attention is given to the making of plate or engraving inks and very little to the manufacture of typographical inks.

There are many mistakes of a kind which are not unusual in a first edition. On page 74 "dilutent" occurs four times; the same mistake was noticed in several other places. "Vermillion" is used repeatedly for "vermilion." On page 97, third paragraph, "there use" should be "their use." On page 44, the fineness of ultramarine blue pigment is given as "very fine impalpable powder."

On page 73, under the heading abrasive qualities and referring to magnetic pigment, it is stated that it is "not abrasive." Since this material is used for sharpening razors one would expect the reverse of this statement. On page 81, third paragraph, "aluminum sulfate" should be "aluminum hydrate."

It is very unfortunate that references to the literature were not put in the book. Only a single reference was found by the reviewer.

The authors hope that the book may "prove valuable and serviceable to the many workers in the art," seems justified yet it is to be hoped that in another edition more extensive material will be included and that the actual manufacture of printing inks will be given more detailed attention.

Philip L. Davis

NEW PUBLICATIONS

By Irene DeMatty, Librarian, Mellon Institute of Industrial Research, Pittsburgh

Agriculture: Laboratory Exercises in Principles of Agriculture. By E. Hopt and R. R. Stafford. 8vo. 192 pp. Price, $0.50. W. M. Welch Mfg. Co., Chicago.

Alcoholometric Tables. By Sir Edward Thorpe. 8vo. Price, $1.10. Longmans, Green & Co., New York.

Asphalt: Beitraege zur Chemie der Asphalte m. besond. Berucksicht. ihrer photochemischen Eigenschaften. By Paul Goedrich. 8vo. 14 pp. A. Hoelder, Vienna.

Chemical German. By Francis C. Phillips. 2nd Ed. 252 pp. Price, $2.00. Chemical Publishing Co., Easton.

Chemistry: Cours experimentale de chimie. By H. Grandmontagne. 8vo. 248 pp. Larousse, Paris.

Chemistry: First Principles of Chemistry. By R. H. Brownlee and Others. Rev. Ed. 12mo. 526 pp. Price, $1.25. Allyn and Bacon, Boston.

Chemistry: Laboratory Exercises Arranged to Accompany "First Course in Chemistry." By W. McPherson and W. E. Henderson. 12mo. 128 pp. Price, $0.60. Ginn & Co., Boston.

Colloidchemistry: Die Welt der vernachlaessigten Dimensionen. By Wolfgang Ostwald. 8vo. Price, $1.50. Theodor Steinkopff, Dresden.

Dynamo-Electric Machinery: Specification and Design of Dynamo-Electric Machinery. By Miles Walker. 4to. 648 pp. Price, $10.00. Longmans, Green & Co., New York.

Electric Light: De la portée des projecteurs de lumière électrique. By Jean Rey. 8vo. 150 pp. Berger-Levrault, Paris.

Electric Measurements and Meter Testing. By D. P. Moreton. 16mo. 328 pp. Price, $1.50. F. J. Drake & Co., Chicago.

Engineering Mathematics: A Vest-Pocket Handbook of Mathematics for Engineers. By L. A. Waterbury. 8vo. 213 pp. Price, $1.50. John Wiley & Sons, New York.

Field Engineering. By W. H. Searles. 17th Ed. 16mo. 630 pp. Price, $3.00. John Wiley & Sons, New York.

Food Products: Laboratory Experiments on Food Products. By E. H. S. Bailey. 44 pp. Price, $0.25. P. Blakiston's Son & Co., Philadelphia.

Gas Engineering and Gas Manufacture. Ed. by C. W. Hastings. J. G. Hammond & Co., London.

Gaseous Combustion at High Pressures. By W. A. Bone. 4to. 65 pp. Price, $0.65. Wm. Wesley & Sons, London.

Glycerine: Its Production, Uses and Examination for Chemists. By S. W. Koppe. 8vo. 258 pp. Price, $2.00. Scott, Greenwood & Son, London.

India-Rubber: Die Synthese des Kautschuks. By R. Ditmar. 8vo. 124 pp. Price, $0.75. Theodor Steinkopff, Dresden.

Industrial Chemistry: Muspratt's encyclopaedisches Handbuch der technischen Chemie. Vol. 3, Pt. 1, Chemische Technologie organischer Industriezweige. Ed. by Arthur Binz. 8vo. 515 pp. Price, $6.50. F. Vieweg & Sohn, Braunschweig.

Iron and Steel: The Canadian Iron and Steel Industry. By W. J. A. Donald. 8vo. 376 pp. Price, $2.00. Houghton, Mifflin Co., Boston.

Mechanics: Cours de mécanique professé à l'Ecole polytechnique. By Léon Lecornu. 8vo. 544 pp. Price, $3.75. Gauthier-Villars, Paris.

Medical Chemistry: A Compend of Medical Chemistry. By H. Leffmann. 6th Ed. 241 pp. Price, $1.00. P. Blakiston's Son & Co., Philadelphia.

Mineral Industry. Vol. 23 (1914). Ed. by G. A. Roush. 8vo. 1000 pp. Price, $10.00. McGraw-Hill Book Co., New York.

Molecules: Studien ueber Molekularstatistik von Emulsionen und deren Zusammenhang mit der Brownschen Bewegung. By M. Smouluchowoski. 8vo. 25 pp. A. Hoelder, Vienna.

Oil: All About Oil for Engineers. By A. Cree. 8vo. Price, $0.75. W. Thacker & Co., London.

Oils: Semi-Annual Report on Essential Oils, Synthetic Perfumes, Etc. By Schimmel & Co. 110 pp. Schimmel & Co., Miltitz near Leipzig.

Organic Chemistry for Advanced Students. Vol. 2. By J. B. Cohen. 8vo. Price, $4.50. Longmans, Green & Co., New York.

Organic Chemistry: Laboratory Experiments in Organic Chemistry. By E. P. Cook. 52 pp. Price, $0.35. P. Blakiston's Son & Co., Philadelphia.

Reed's Handbook to the Examination of Engine-Room Artificers. By E. R. Collings. New and Rev. Ed. 8vo. 356 pp. Price, $1.25. T. Reed & Co., London.

Soil Analysis: Eine neue Methode zur mechanischen Bodenanalyse. By Svén Odèn. 8vo. 55 pp. Price, $0.75. Verlag fuer Fachliteratur, Berlin.

Steam Boiler Economy. By W. Kent. 2nd Ed. 8vo. 717 pp. Price, $4.50. John Wiley & Sons, New York.

Steam Engine: Dampfmaschine oder Elektromotor. By F. Barth. 8vo. 64 pp. Price, $0.50. R. Oldenbourg, Munich.

Storage Batteries: A List of References 1900-1915. By G. S. Maynard. 8vo. 37 pp. New York Public Library, New York.

Turbine Regulator:. Beitrag zur Frage der Stabilitaetsbedingungen der Turbinenregler. By H. Baudisch. 8vo. Verlag fuer Fachliteratur, Berlin.

Ultramarine: Ueber natuerliches und kuenstliches Ultramarin. By C. Doelter. 8vo. A. Hoelder, Vienna.

RECENT JOURNAL ARTICLES

Agricultural Chemists: Journal of the Association of Official Agricultural Chemists. New Journal. Published by Williams & Wilkins Co., Baltimore, for the Association.

Air and Steam as Atomizing Agents. By R. A. Bull. *Iron Trade Review,* Vol. 57 (1915), No. 14, pp. 626–629.

Alcohol: Die Bestimmung von Alkohol in Aether. By P. Szeberényi. *Zeitschrift fuer analytische Chemie,* Vol. 45 (1915), No. 8, pp. 409–411.

Aluminium-Bestimmung in Silikaten. By Emmerich Selch. *Zeitschrift fuer analytische Chemie,* Vol. 45 (1915), No. 8, pp. 395–403.

Aluminum Precipitation at the Mill of the Butters Divisadero Company. By E. M. Hamilton and P. H. Crawford. *Mining and Scientific Press,* Vol. 111 (1915), No. 11, pp. 387–391.

Ammonia Compressor Efficiency. By Charles Johnsson. *Refrigerating World,* Vol. 49 (1915), No. 8, pp. 25–26.

Benzol: Unterscheidung von Benzin und Benzol. By H. Mix. *Kolloid-Zeitschrift,* Vol. 17 (1915), No. 1, pp. 7–9.

Bronze: Ueber elektrolytische Bronzefaellung. By W. D. Treadwell and E. Beckh. *Zeitschrift fuer Elektrochemie,* Vol. 21 (1915), No. 15/16, pp. 374–381.

Cellulose: Experimental Work on Soda Cellulose. By Sidney D. Wells. *Paper,* Vol. 17 (1915), No. 4, pp. 14–15.

Coal Tar: Use of Coal Tar in Flotation. By W. A. Mueller. *Engineering and Mining Journal,* Vol. 100 (1915), No. 15, pp. 591–593.

Corrosion: Influence of Different Elements on the Corrosion of Iron. By L. C. Wilson. *Engineering Magazine,* Vol. 50 (1915), No. 1, pp. 78–86.

Flotation: What is Flotation? By T. A. Rickard. *Mining and Scientific Press,* Vo. 111 (1915), No. 11, pp. 383–386.

Gas *vs.* Electrically Heated Ovens. By G. H. Trout. *Iron Trade Review,* Vol. 57 (1915), No. 12, p. 526.

Gas Producers: Factors Governing Gas Producer Practice. By Franz Denk. *Steel and Iron,* Vol. 49 (1915), No. 26, pp. 911–914.

Gases: The Kinetic Theory of Gases. By Saul Dushman. *General Electric Review,* Vol. 18 (1915), No. 10, pp. 952–958.

Gold Milling in California. By L. A. Palmer. *Metallurgical and Chemical Engineering,* Vol. 13 (1915), No. 10, pp. 617–624.

Hydrogen: Bestimmung des Wasserstoffs in Gasgemengen durch katalytische Absorption. By E. Bosshard and E. Fischli. *Zeitschrift fuer angewandte Chemie,* Vol. 28 (1915), No. 70, pp. 365–366.

Hydroquinone: Die Sulfurierung des Hydrochinons. By Johann Pinnow. *Zeitschrift fuer Elektrochemie,* Vol. 21 (1915), No. 15/16, pp. 380–388.

India Rubber: Arbeiten ueber Kautschuk und Guttapercha. By G. H. Hillen. *Zeitschrift fuer angewandte Chemie,* Vol. 28 (1915), No. 66, pp. 349–354.

Insulating Materials: The Volume Resistivity and Surface Resistivity of Insulating Materials. By H. L. Curtis. *General Electric Review,* Vol. 18 (1915), No. 10, pp. 996–1001.

Kilns: Setting Down Draft Kilns. By A. F. Greaves-Walker. *Brick and Clay Record,* Vol. 47 (1915), No. 7, pp. 501–505.

Leather: Ueber ein Neutralfettverbot in der Lederindustrie. By W. Fahrion. *Chemische Revue ueber die Fett- und Harz-Industrie,* Vol. 22 (1915), No. 9, pp. 77–80.

Oil Engine for Ice Plant Service. By L. K. Doelling. *Ice and Refrigeration,* Vol. 49 (1915), No. 4, pp. 154–162.

Paper Fiber Analysis. By Max Cline. *Paper,* Vol. 17 (1915), No. 3, pp. 19–21.

Petroleum: Beitrag zur Untersuchung und Beurteilung des zu Leuchtzwecken dienenden Petroleums. By R. Jungkunz. *Chemiker-Zeitung,* Vol. 39 (1915), No. 102, pp. 641–642.

Portland Cement: Controlling the Raw Mix in the Manufacture of Portland Cement. By Samuel McAnally. *Concrete-Cement Age,* Vol. 7 (1915), No. 3, pp. 30–33.

Rare Earths: Die seltenen Erden in der Therapie. By C. R. Boehm. *Zeitschrift fuer angewandte Chemie,* Vol. 28 (1915), No. 64, pp. 346–348.

RECENT INVENTIONS

UNITED STATES PATENTS

By C. L. Parker
Solicitor of Chemical Patents, McGill Building, Washington, D. C.

Compressing, Drying, and Transferring Chlorin Gas. J. W. Aylsworth, April 6, 1915. U. S. Pat. 1,134,432. Alternate volumes of chlorin gas and sulfuric acid are caused to continuously enter a helical pipe. The pipe is rotated thus transferring the acid and gas compressed thereby into a receiver. The intimate contact between the acid and gas dries the gas and the acid is finally separated from the dried gas.

Converting Meadow or Swamp Land into Fertilizer. B. Reichelt, April 6, 1915. U. S. Pat. 1,134,760. Meadow or swamp land material containing potash is converted into fertilizer by heating the material to a temperature sufficient to dry it and to drive off the potash therefrom. The dried material is then ground and finally the potash is re-mixed with the ground material.

Nitrogen and Potash Mixture. S. W. Sinsheimer, April 6, 1915. U. S. Pat. 1,134,921. The product is a fertilizing agent consisting of the residue of waste liquid after the separation of sugar from molasses which has been treated with carbon dioxid and evaporated at a low temperature to produce a concentrated product.

Preventing Escape of Sulfur Dioxid in Smelting Sulfid Ores. W. A. Hall, April 6, 1915. U. S. Pat. 1,134,846. A reducing flame together with steam is introduced into the upper level of the furnace at a point near the normal charge level therein and under conditions capable of reducing a part at least of the SO_2 in the exit gases and separating the sulfur from the gases.

Producing Hydroxids of Metals by Means of Ammonia. H. A. Frasch, April 13, 1915. U. S. Pat. 1,135,785. Nickel oxid is precipitated from a solution of nickel ammonium chlorid by boiling in the presence of a solution of ammonium chlorid until the nickel oxid is precipitated, while maintaining the solution alkaline with ammonia.

Treatment of Alunite, Etc. C. H. MacDowell, April 20, 1915. U. S. Pat. 1,136,549. Alunite is calcined for the purpose of driving off anhydrid and sulfuric anhydrid. The product is heated to a temperature sufficient to volatilize and drive off potash and the exit of the volatilized potash from the charge is expedited by carrying it off in a gaseous current.

Continuous Distillation and Rectification of Alcoholic Liquids. V. Slavicek, April 20, 1915. U. S. Pat. 1,136,559. The alcoholic raw vapor is divided into portions. The non-cooled phlegm of one portion of the head product is purified and immediately introduced at the beginning of the rectification into the alcoholic raw vapor of the second portion.

Coagulant for Water Purification. A. Jacobson, April 27, 1915. U. S. Pat. 1,137,005. The coagulant is produced by passing an aqueous solution of sulfate of aluminum over metallic iron so that a part of the iron is dissolved to form iron sulfate and sulfate of aluminum is reduced to a soluble basic sulfate of aluminum.

Rendering Phosphoric Acid Available. W. S. Landis, April 27, 1915. U. S. Pat. 1,137,065. A mixture of finely divided phosphate rock, an alkali metal salt and carbon is heated to a sufficient temperature to eliminate the acid radical of the salt. The mixture is then subjected to a higher temperature sufficient to cause an incipient fusing or clinkering of the mixture.

Carbonitrids. S. Peacock, April 27, 1915. U. S. Pat. 1,137,524. The process consists in heating in the presence of nitrogen an oxid of an element having metallic properties with sufficient carbon and to a temperature sufficient to produce carbonitrid while preventing the partial pressures of the gaseous products from rising sufficiently to interfere with the reaction.

Alkali-Soluble Alumina. P. R. Hershman, April 27, 1915. U. S. Pat. 1,137,617. Alumina in admixture with carbon is heated in the presence of a non-nitrifying atmosphere, to a temperature sufficient to make it soluble in alkali.

Recovering Soda Content of Waste Liquor. H. K. Moore, May 4, 1915. U. S. Pat. 1,137,779. The lignin is utilized in making steam which may be employed in the digestion of the raw wood. Multiple-effect evaporators are used in concentrating the lignin to approximately 355 Bé. and the residuum is mixed with sulfate of soda and a carrier of carbonaceous material such as sawdust, pulverized bark, peat, coke, or the like. The mixture is dried, burned in a smelting furnace and the soda recovered.

Acid Mono-Calcium Phosphate. Robert Stewart, May 4, 1915. U. S. Pat. 1,137,806. Phosphate rock is fed by the conveyor 10 into the upper end of the tube furnace 5, heated by the burner 14 and subjected to the action of steam and smelter smoke introduced through the pipes 12 and 13, respectively. The reaction results in the production of acid mono-calcium phosphate and a solution of acid calcium sulfite.

Treating Aluminate Solutions. H. Howard, May 4, 1915. U. S. Pat. 1,137,860. Silica is removed from aluminate solutions by subjecting the hot aluminate solution, at a concentration not exceeding 300 Bé. to the action of the residues from the decomposition of bauxite by alkali.

Reducing Waste Sulfite Liquor to a Powder. F. H. Kennard, May 4, 1915. U. S. Pat. 1,138,118. The waste sulfite liquor is first concentrated to a viscous state in a vacuum. The viscous material is then applied in a vacuum, in a thin sheet, onto a heated surface and retained thereon until the viscous material is evaporated to dryness.

Treating Hydrocarbon Oils. C. H. Washburn, May 4, 1915. U. S. Pat. 1,138,266. Petroleum oils of from 260 to 488 Bé. are transformed into a product not exceeding 520 Bé. by passing the oil and water in liquid form into a retort where they are converted into vapor, and leading the vapor to a condenser, the terminal of which is closed to create a back pressure, thereby condensing the vapor at a pressure of from three to five atmospheres maintained throughout the distillation and condensation.

Drying Sodium-Bisulfite Crystals. H. Howard, May 11, 1915. U. S. Pat. 1,138,658. Wet sulfite crystals are subjected to a current of hot sulfur dioxid gas.

Nitroglycerin. R. Weyel, May 11, 1915. U. S. Pat. 1,138,917. The separation of a mixture of liquids, containing nitroglycerin and an acid is facilitated by generating silicon fluorid in the liquids by the addition of a fluorid and an insoluble siliceous compound.

Explosive. G. Burgschmiet, May 11, 1915. U. S. Pat. 1,139,339. The composition comprises essentially about 80 per cent of ammonium nitrate, about 18 per cent of dinitrotoluene and about 2 per cent charcoal and wood meal.

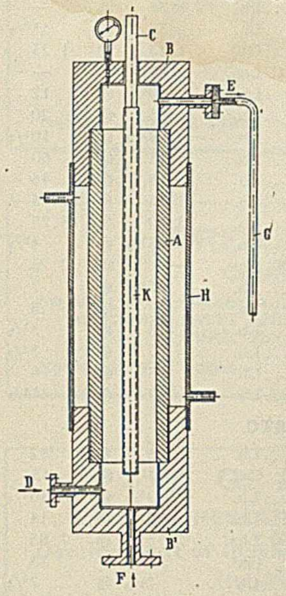
Wood-Preservative. J. A. DeCew, May 18, 1915. U. S. Pat. 1,140,127. The composition comprises a mixture of waste sulfite liquor, creosote oil, and chlorid of zinc.

BRITISH PATENTS

By D. Geddes Anderson
Chemical Engineer and Patent Chemist, Glasgow, Scotland

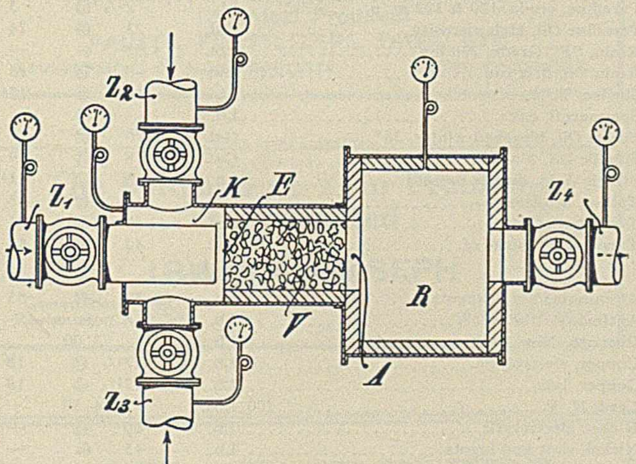
The following abstracts are taken direct from the patent specifications as soon as these are published by the British Patent Office. The date given at the end of the abstract is the date of acceptance.

Manufacture of Hydrogen Peroxide. Henkel and Cie, Nov. 18, 1914. Brit. Pat. 22,714. A is a tube of suitable metal coated inside with a thin layer of cathode material, such as silver amalgam and copper amalgam. The electrolyte is pumped in at D and escapes at E, and oxygen is admitted at F. C is the anode and is covered with the diaphragm tube K.—June 17, 1915.



Production of Esters of the Tertiary Alcohols. Neumann and Co., Chemische Fabrik G. M. B. H and J. Zeltner, Nov. 5, 1913. Brit. Pat. 25,262. Organic acid chlorides, with the exception of the chlorides of carbonic acid and their derivatives are caused to react with non-substituted aliphatic tertiary alcohols in the presence of tertiary bases, *e. g.*, 64 grams of valeric chloride are added to 44 grams tertiary amyl alcohol in 55 grams pyridine. After standing 24 hours, 100 cc. water and 100 cc. hydrochloric acid are added and the tertiary amyl valeriate is distilled *in vacuo*.—July 1, 1915.

Production of Nitrogen and Oxygen Compounds. C. Krauss and P. Staehelin, Nov. 18, 1913. Brit. Pat. 26,499. Liquid or gaseous fuel is burned without flame at high pressure in an atmosphere of nitrogen and oxygen. The combustion chamber is filled with a granular material coated with substances such as oxides of chromium, titanium or vanadium, which facilitate the combination of nitrogen and oxygen.—July 8, 1915.



Plastic Compositions. E. Krause and H. Blucher, Jan. 1, 1914. Brit. Pat. 76. The plastic material is made by treating the albuminous and cellulosic residues from the manufacture of yeast extracts or preparations, with formaldehyde. The residues obtained from yeast in the manufacture of preparations of the nature of meat extract are preferably employed.—July 29, 1915.

Rubber Substitute. J. Baier and A. G. M. Weale, Jan. 15, 1914. Brit. Pat. 1,171. Animal tissue is dissolved in zinc chloride and the thick jelly obtained is mixed with a starchy material such as finely ground tapioca. The thick dough so prepared is mixed with rubber solution, or with a saturated solution of resin in methylated spirit.—July 29, 1915.

Varnish. E. Girzik, Jan. 20, 1914. Brit. Pat. 1,575. Process relates to the manufacture of varnish from semi-drying oils such as train oil. The free fatty acids in the oil are removed by adding a suitable quantity of soda lye, and allowing to stand in the cold for several days. The soap is separated and the oil subjected to a boiling or polymerization process.—July 29, 1915.

Extracting Sulfurous Acid and Oxides from Sulfates. A. Bambach, Feb. 6, 1914. Brit. Pat. 3,174. The sulfates, such as calcium and barium sulfates, are introduced into a shaft furnace in a fragmentary state and heated with a flame from a gas and air mixture. The operation is finished by feeding the flame with excess of air.—July 29, 1915.

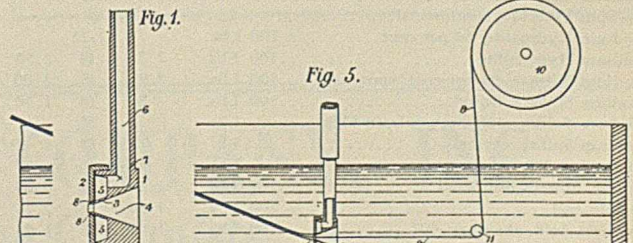
Coating Articles with Aluminum. W. Krumbhaar, Feb. 12, 1914. Brit. Pat. 3,713. The article to be treated is first given a coat of lacquer, and when this coat is perfectly dry, finely divided aluminum powder is rubbed in.—July 1, 1915.

Manufacture of Light Hydrocarbons from Heavy Hydrocarbons. F. Bergius, Feb. 21, 1914. Brit. Pat. 4,574. The hydrocarbons, previous to distillation, are heated in the presence of hydrogen under a pressure of 100 atmospheres. The hydrogen reacts and converts all non-saturated compounds into saturated compounds. The petroleum so treated is then distilled and it is found that the proportion of light constituents is very much increased.—July 8, 1915.

Manufacture of Sulfate of Ammonia, and the Purification of Coal Gas. J. Mackenzie, April 23, 1914. Brit. Pat. 10,059. The gas, freed from tar, is passed into a moist atmosphere of nitrous acid at 200° F. The sulfur compounds contained in the coal gas are oxidized and combine with the ammonia to give sulfate of ammonia. The excess of nitrous acid is absorbed in sulfuric acid.—July 23, 1915.

Carbon Electrodes for Galvanic Cells. V. Scholz, April 24, 1914. Brit. Pat. 10,171. Carbon powder in the form of colloidal graphite is employed in the formation of the electrode. The voltage is rendered more constant by using the graphite in this form.—July 8, 1915.

Manufacture of Tubular Threads from Solutions of Cellulose. Courtnaulds, Ltd., and L. P. Wilson, July 23, 1914. Brit. Pat. 17,495. A jet as shown in Fig. 1 is used, and is immersed in a coagulating bath. The liquid of the bath has access to both the interior and exterior surfaces of the thread being formed. Compound threads can also be formed by projecting a solution of viscose through the orifice of the jet while a free formed thread of suitable textile fiber is introduced into the interior of the tabular thread as in Fig. 5.—July 23, 1915.



MARKET REPORT—OCTOBER, 1915

WHOLESALE PRICES PREVAILING IN THE NEW YORK MARKET ON OCTOBER 20TH. *(See this issue, page 997)*

INORGANIC CHEMICALS

Acetate of Lime, gray	100 Lbs.	4.00	@	4.05
Alum, lump	100 Lbs.	5.25	@	5.50
Aluminum Sulfate, high-grade	100 Lbs.	2.25	@	2.75
Ammonium Carbonate, domestic	Lb.	8½	@	9½
Ammonium Chloride, gray	Lb.	8.25	@	8.50
Ammonium Phosphate, commercial, 98–100%	Lb.	10	@	10½
Aqua Ammonium, 16°, drums	Lb	2¼	-@	2½
Arsenic, white	Lb.	3¾	@	4
Barium Chloride	Ton	90.00	@	100.00
Barium Nitrate	Lb.	15	@	16
Barytes, prime white, foreign	Ton	19.00	@	23.00
Bleaching Powder, 35 per cent	100 Lbs.	3.50	@	4.00
Blue Vitriol	Lb.	6½	@	7
Borax, crystals, in bags	Lb.	5½	@	6
Boric Acid, powdered crystals	Lb.	10	@	10¼
Brimstone, crude, domestic	Long Ton	22.00	@	22.50
Bromine, technical, bulk	Lb.	1.50	@	1.75
Calcium Chloride, lump	Ton	—	@	11.78
Calcium Chloride, granulated	Ton	—	@	14.78
Caustic Soda, 70 @ 76 per cent	100 Lbs.	5.25	@	5.50
Caustic Soda, powdered or granulated, 76 per cent,	100 Lbs.	5.50	@	5.75
Chalk, light precipitated	Lb.	4¾	@	5½
China Clay, imported	Ton	20.00	@	25.00
Feldspar	Ton	8.00	@	12.00
Fuller's Earth, foreign, powdered	100 Lbs.	80	@	1.00
Glauber's Salt, in bbls.	100 Lbs.	60	@	75
Green Vitriol, bulk	100 Lbs.	70	@	80
Hydrochloric Acid, commercial, 18°	100 Lbs.	1.75	@	2.00
Hydrochloric Acid, C. P., conc., 22°	100 Lbs.	2.25	@	2.50
Iodine, resublimed	Lb.	4.30	@	4.35
Lead Acetate, white crystals	Lb.	11⅝	@	12⅛
Lead Nitrate	Lb.	11½	@	12
Litharge, American	Lb.	6¼	@	—
Lithium Carbonate	Lb.	97	@	1.00
Magnesium Carbonate	Lb.	5½	@	—
Magnesite, "Calcined"	Ton	34.00	@	36.00
Mercuric Chloride, commercial	Lb.	1.50	@	--
Nitric Acid, 68 per cent, sp. gr. 1.42	Lb.	nominal		
Nitric Acid, fuming	Lb.	nominal		
Phosphoric Acid, sp. gr. 1.750	Lb.	28	@	31
Phosphorus	Lb.	40	@	1.00
Plaster of Paris	Bbl.	1.50	@	1.70
Potassium Bichromate, casks	Lb.	21	@	22
Potassium Bromide	Lb.	2.50	@	2.51
Potassium Carbonate, calcined, 80 @ 85%	100 Lbs.	27	@	29
Potassium Chlorate, crystals, spot	Lb.	38	@	42
Potassium Cyanide, bulk, 98–99 per cent	Lb.	28	@	32
Potassium Hydroxide	Lb.	37	@	40
Potassium Iodide, bulk	Lb.	3.70	@	3.75
Potassium Nitrate	Lb.	19	@	20
Potassium Permanganate, bulk	Lb.	1.25	@	—
Quicksilver, flask, 75 lbs.		93.00	@	95.00
Red Lead, American, dry	Lb.	6½	@	—
Salt Cake, glass makers'	100 Lbs.	55	@	65
Silver Nitrate	Oz.	31¾	@	33¾
Soapstone in bags	Ton	10.00	@	12.00
Soda Ash, 48 per cent	100 Lbs.	80	@	90
Sodium Acetate	Lb.	5	@	6
Sodium Bicarbonate, domestic	100 Lbs.	1.00	@	1.10
Sodium Bicarbonate, English	Lb.	3½	@	3⅝
Sodium Bichromate	Lb.	15½	@	16
Sodium Carbonate, dry	100 Lbs.	1.10	@	—
Sodium Chlorate	Lb.	38	@	40
Sodium Fluoride, commercial	Lb.	12	@	—
Sodium Hydroxide, 60 per cent	100 Lbs.	2.25		
Sodium Hyposulfite	100 Lbs.	2.25	@	2.50
Sodium Nitrate, 95 per cent, spot	100 Lbs.	2.95	@	3.00
Sodium Silicate, liquid	100 Lbs.	85	@	1.10
Sodium Sulfide, 30%, crystals, in bbls.	Lb.	2	@	2½
Sodium Sulfite, crystals	Lb.	2½	@	2⅝
Strontium Nitrate	Lb.	18	@	19
Sulfur, flowers, sublimed	100 Lbs.	2.20	@	2.60
Sulfur, roll	100 Lbs.	1.80	@	2.15
Sulfuric Acid, chamber, 60°	100 Lbs.	1.00	@	1.25
Sulfuric Acid, conc., sp. gr. 1.842	100 Lbs.	1.75	@	2.50
Sulfuric Acid, oleum (fuming)	100 Lbs.	1.75	@	2.50
Talc, American	Ton	9.00	@	13.00
Terra Alba, American, No. 1	100 Lbs.	75	@	80
Tin Bichloride, 50°	Lb.	10	@	10½
Tin Oxide	Lb.	38	@	40
White Lead, American, dry	Lb.	5½	@	—
Zinc Carbonate	Lb.	12	@	13
Zinc Chloride, commercial	Lb.	8	@	8½
Zinc Oxide, American process	Lb.	8	@	8¼
Zinc Sulfate	Lb.	6½	@	7

ORGANIC CHEMICALS

Acetanilid, C. P., in bbls.	Lb.	1.10	@	1.15
Acetic Acid, 28 per cent, in bbls.	100 Lbs.	3.00	@	3.25
Acetic Acid, glacial, 99½%, in carboys	Lb.	21½	@	25
Acetone, drums	Lb.	28½	@	30
Alcohol, denatured, 180 proof	Gal.	39	@	40
Alcohol, grain, 188 proof	Gal.	2.60	@	—
Alcohol, wood, 95 per cent, refined	Gal.	45	@	47
Amyl Acetate	Gal.	3.00	@	3.25
Aniline Oil	Lb.	1.00	@	1.35
Benzoic Acid, ex-toluol	Lb.	3.25	@	3.30
Benzol, 90 per cent	Gal.	80	@	90
Camphor, refined in bulk, bbls.	Lb.	43	@	—
Carbolic Acid, U. S. P., crystals, drums	Lb.	1.70	@	1.75
Carbon Bisulfide	Lb.	6½	@	7½
Carbon Tetrachloride, drums, 100 gals.	Lb.	17	@	18
Chloroform	Lb.	35	@	—
Citric Acid, domestic, crystals	Lb.	55	@	55½
Cresol, U. S. P.	Gal.	1.15	@	1.25
Dextrine, corn (carloads, bags)	100 Lbs.	2.80	@	—
Dextrine, imported potato	Lb.	10	@	12
Ether, U. S. P., 1900	Lb.	15	@	20
Formaldehyde, 40 per cent	Lb.	9½	@	10½
Glycerine, dynamite, drums included	Lb.	57	@	60
Oxalic Acid, in casks	Lb.	45	@	46
Pyrogallic Acid, resublimed bulk	Lb.	1.45	@	1.55
Salicylic Acid	Lb.	3.50	@	3.75
Starch, cassava	Lb.	3½	@	4⅛
Starch, corn (carloads, bags)	100 Lbs.	—	@	2.25
Starch, potato	Lb.	6¾	@	7
Starch, rice	Lb.	7	@	8
Starch, sago	Lb.	3	@	3½
Starch, wheat	Lb.	5	@	5½
Tannic Acid, commercial	Lb.	75	@	76
Tartaric Acid, crystals	Lb.	47	@	47½

OILS, WAXES, ETC.

Beeswax, pure, white	Lb.	45	@	52
Black Mineral Oil, 29 gravity	Gal.	12½	@	13
Castor Oil, No. 3	Lb.	9¾	@	10¼
Ceresin, yellow	Lb.	10	@	14
Corn Oil	100 Lbs.	7.75	@	7.85
Cottonseed Oil, crude, f. o. b. mill	Gal.	52	@	53
Cottonseed Oil, p. s. y.	Lb.	7⅝	@	7¾
Creosote, beechwood	Lb.	2.50	@	3.50
Cylinder Oil, light, filtered	Gal.	20	@	25
Fusel Oil, crude	Lb.	2.65	@	2.75
Japan Wax	Lb.	12¾	@	13
Lard Oil, prime winter	Gal.	86	@	90
Linseed Oil, raw (car lots)	Gal.	58	@	—
Menhaden Oil, crude	Gal.	nominal		
Naphtha, 68 @ 72°	Gal.	22	@	24
Neat's-foot Oil, 20°	Gal.	92	@	94
Paraffine, crude, 120 & 122 m. p.	Lb.	2½	@	3
Paraffine Oil, high viscosity	Gal.	23	@	24
Rosin, "F" Grade, 280 lbs.	Bbl.	4.15	@	—
Rosin Oil, first run	Gal.	—	@	25
Shellac, T. N.	Lb.	15	@	15½
Spermaceti, cake	Lb.	25		
Sperm Oil, bleached winter, 38°	Gal.	70	@	—
Spindle Oil, No. 200	Gal.	18	@	19
Stearic Acid, double-pressed	Lb.	13	@	13½
Tallow, acidless	Gal.	64	@	65
Tar Oil, distilled	Gal.	30	@	31
Turpentine, spirits of	Gal.	42	@	44

METALS

Aluminum, No. 1, ingots	Lb.	50	@	55
Antimony, Hallets'	Lb.	nominal		
Bismuth, New York	Lb.	3.00		
Copper, electrolytic	Lb.	17¾	@	18
Copper, lake	Lb.	17¾	@	18
Lead, N. Y.	100 Lbs.	4.50		
Nickel, electrolytic	Lb.	50	@	—
Nickel, shot and ingots	Lb.	45	@	—
Platinum, refined	Oz.	45.00	@	50.00
Silver	Oz.	49⅜		
Tin	100 Lbs.	33.50	@	—
Zinc, N. Y.	Lb.	13½		

FERTILIZER MATERIALS

Ammonium Sulfate	100 Lbs.	3.40		
Blood, dried	Unit	2.85		
Bone, 4½ and 50, ground, raw	Ton	33.00		
Calcium Cyanamid	Unit of Ammonia	2.35		
Calcium Nitrate, Norwegian	100 Lbs.	—		
Castor Meal	Unit	—		
Fish Scrap, domestic, dried, f. o. b. works	Unit	3.25	@	10
Phosphate, acid	Unit	75	@	80
Phosphate rock; f. o. b. mine:				
Florida land pebble, 68 per cent	Ton	2.75	@	3.00
Tennessee, 70–80 per cent	Ton	5.00	@	5.50
Potassium "muriate." basis 80 per cent	Ton	245.00	@	250.00
Pyrites, furnace size, mported	Unit	—	@	13
Tankage, high-grade; f. o. b. Chicago	Unit	2.30	&	10

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ANALYSIS OF PULP AND PAPER
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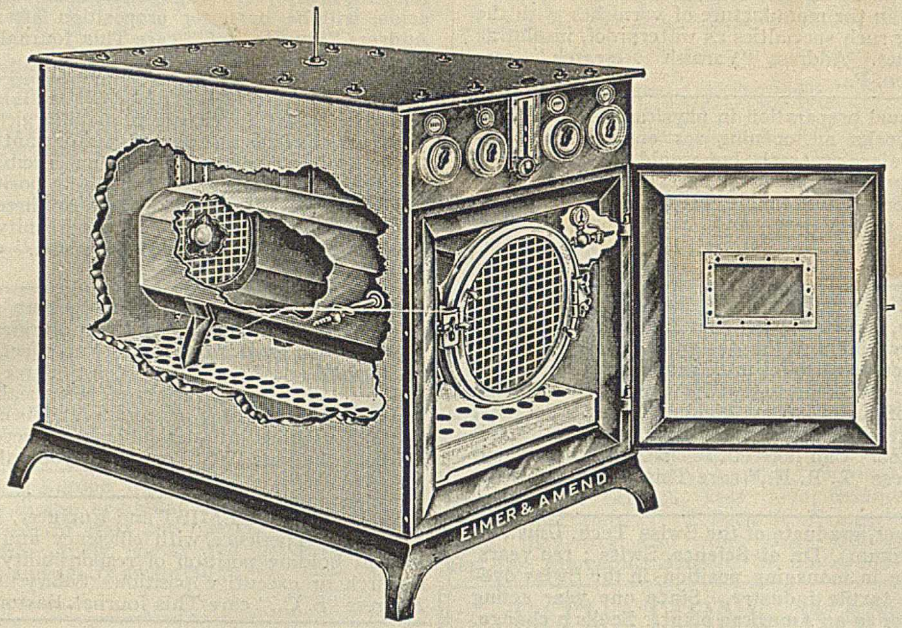

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