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ELECTRIC FURNACE

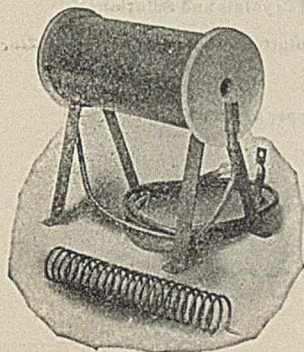
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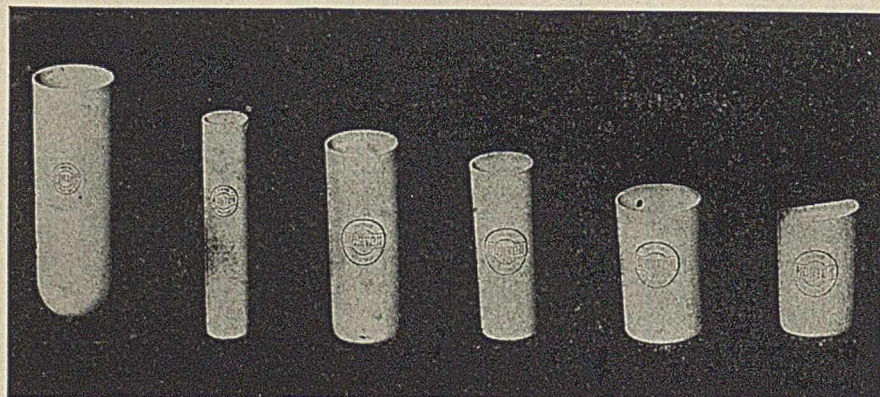
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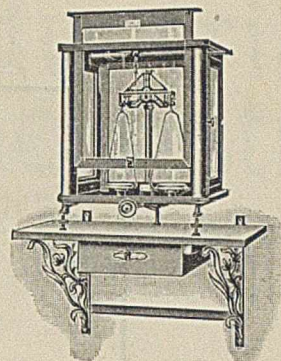
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# The Journal of Industrial and Engineering Chemistry

Published by THE AMERICAN CHEMICAL SOCIETY

Volume IV

JANUARY, 1912

No. 1

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Associate Editors:

Geo. P. Adamson, E. G. Bailey, H. E. Barnard, C. A. Browne, G. E. Barton, Wm. Brady, Wm. Campbell, F. B. Carpenter, Virgil Coblenz, Francis I. Dupont, W. C. Ebaugh, Wm. C. Geer, W. F. Hillebrand, W. D. Horne, Karl Langenbeck, A. D. Little, C. E. Lucke, P. C. McIlhiney, E. B. McCready, Wm. McMurtrie, J. Merritt Matthews, T. J. Parker, J. D. Pennock, W. D. Richardson, Geo. C. Stone, Ernst Twitchell, Robert Wahl, Wm. H. Walker, W. R. Whitney, A. M. Wright.

Published monthly. Subscription price to non-members of the American Chemical Society, \$6.00 yearly. Foreign postage, seventy-five cents, Canada, Cuba and Mexico excepted.

Entered at the Post-Office, Easton, Pa., as Second-class Matter.



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## EDITORIALS

### SOME APPLICATIONS OF WROUGHT TUNGSTEN AND MOLYBDENUM.

Only a few years ago a number of the elements were classed in the literature as brittle and had never been produced in a condition in which they were not brittle at room temperature, but have since been taken out of the brittle list. Among these are tungsten and molybdenum.

Prior to their production in ductile form, tungsten and molybdenum as such (that is, other than in alloys or compounds) had but one application, namely, in an incandescent lamp, the one as filament, and the other as filament support.

Some of the physical properties of these last-named industrially new elements, together with a rough sketch of the investigation work leading up to the production of ductile tungsten, have been published elsewhere (*Trans. Am. Electrochem. Soc.*, 17, 229-234 (1910); *Proc. Am. Inst. E. E.*, 29, Part II, 961-965 (1910)).

It is the purpose of this paper to tell of some of the applications which are being developed for wrought tungsten and wrought molybdenum. The writer presents it not only for the interest of the work itself, but also in the hope that it will stimulate work on the other so-called brittle metals, and it seems to him that some of our industries are almost certain to profit greatly by the discovery of each new ductile element.

#### THE DRAWN-WIRE TUNGSTEN INCANDESCENT LAMP.

A year and a half ago the hope was first publicly expressed that it would soon be possible to manufacture lamps on a large scale from ductile tungsten. This hope has been fulfilled, and the commercial development has been so rapid that already the bulk of the tungsten lamps made in this country are of drawn wire, and the manufacture is already established on a large scale in England and Germany. Some anxiety was at first felt lest it should not be possible to get diamond wire drawing dies of sufficiently small aperture to produce wire fine enough for the lowest candle power lamps which might be desired. But this fear proved groundless and wire is now produced in quantity down to 0.0006 inch in diameter.

Ductile molybdenum is used in some types of lamp as a support material for the tungsten filament.

#### THE TUNGSTEN OR MOLYBDENUM-WOUND FURNACE.

This has already been described by Winne and Dantszen (*THIS JOURNAL*, 1911, p. 770), and has proved to be an exceedingly useful tool, especially so in connection with the production of wrought tungsten and molybdenum. Extensive factory use has shown it to be not only cheaper but, regardless of cost, far superior to a platinum-wound electric furnace. When wound on a body of alundum it permits of the attainment of higher temperature than can be reached with platinum. This attainment of the higher tempera-

ture is often in itself an advantage, and it is always an advantage, in industrial work, to be relieved from the necessity of using the strictest temperature control to avoid melting a furnace winding. This is especially true in cases where the time consumed in heating up the work in the furnace is important. For in these cases much time is saved by not using a constant current supply to the furnace winding, but by first raising the current, upon the introduction of the cold work, and then bringing it down as the temperature of the work rises.

#### ELECTRICAL CONTACTS OF TUNGSTEN AND MOLYBDENUM.

Tungsten and molybdenum are destined to play a very important rôle in the field of contact-making devices. This is due to their high melting point, which prevents them from welding together; to their heat conductivity (about twice that of platinum) which tends to keep down local temperature rise; and to their hardness, which enables them to stand repeated impact without flattening out.

The natural assumption would be that both metals under the heat of even minute arcs, which form when the contacts are separated, would oxidize at the points where arcing has taken place and that non-conducting layers would thus be formed which would produce a high and variable contact resistance. But several things intervene to prevent this. First, there is the relatively good heat conductivity of the dense form of these metals, which distributes the heat. Then the fact that the metals are much less expensive than platinum, for example, makes possible the use of larger contact masses, which again militates against strong local heating. Finally, there is the fact that with both tungsten and molybdenum the oxides which form in the presence of a sufficiently limited amount of oxygen are conducting.

Some promising applications which are being tested out are the following:

As an iridium substitute in the "master" contacts of Tirrell voltage regulators.

As a silver substitute in the relay contacts of Tirrell voltage regulators.

As an iridium substitute in feed-wire voltage regulators.

As a platinum substitute in railway signal relays.

As a substitute for platinum-iridium in the contacts of a synchronously driven vibrating mechanical rectifier.

As a platinum substitute in telephone jacks.

As a platinum substitute in automobile and stationary gas-engine ignition work, for spark coil contacts, magneto circuit-breaker contacts, and spark plugs.

In connection with the above there was at first serious difficulty in getting satisfactory heat contact between tungsten or molybdenum on the one hand, and iron or brass, as the case might be, on the other.

This was due to the fact that neither tungsten nor molybdenum can be satisfactorily soldered by any of the ordinary processes. This difficulty has been finally entirely overcome.

#### TUNGSTEN AS TARGET IN A RÖNTGEN (X-RAY) TUBE.

This has proved to be, both from the scientific and practical points of view, an exceptionally interesting application.

Until recently platinum has been almost universally regarded as the best target material, and it has so long held undisputed sway in this field that the Röntgen ray worker has come to look upon its limitations as inherent in the Röntgen tube.

It has not been possible until recently to produce dense, forged pieces of pure malleable tungsten. But with the advent of this material, the possibilities of the Röntgen tube are greatly extended.

The desiderata in a material to be used as the anti-cathode or target are the following:

1. High specific gravity.
2. High melting point.
3. High heat conductivity.
4. Low vapor pressure at high temperature.

The reasons why the above qualities are desirable follow readily from a brief consideration of the theory of Röntgen-ray production.

From the concave cathode, electrically charged particles, the electrons, are shot out at high velocity in a direction normal to the surface. The paths of these particles converge and the target is placed at or near the point of strongest convergence, the focus point. When the electron meets an obstruction, as the target, its velocity is reduced, and the denser the target the more rapid is the deceleration. The more rapid the deceleration the greater is the amplitude of the electromagnetic pulse, the Röntgen-ray, sent out. Here then is a need for high specific gravity; that of forged tungsten is but little less than that of platinum.

In modern Röntgen-ray practice, powerful apparatus running sometimes to a capacity of six kilowatts is used to excite the tube. The greater part of the energy delivered to the tube is transformed into heat at the point where the cathode rays bombard the target. Where platinum is used it has been found necessary, to prevent melting, to place the target beyond the focus of the cathode so as to spread the bombardment over a larger area. As a radiograph is a shadow picture, and as the source of the Röntgen-ray is the bombarded area of the target, this enlarging of that area is clearly an undesirable thing to do, as the larger area will mean more overlapping and less definition in the resulting picture. In this way, the melting point of platinum has been the limiting feature of the Röntgen tube. The capacity of the tube has been increased by water cooling the platinum or by using as a target a large mass of copper having a very thin platinum face. But the limit, although raised by this artifice, has still been the melting point of the platinum.

Tungsten has a much higher melting point (3000° C.

as against 1755° for platinum), and so, even with sharp focusing of the cathode rays on the target, permits the use of more energy than has hitherto been possible; for the high temperature to which it can run enables it to radiate more heat, and its better heat conductivity permits a more rapid flow of heat from the focus spot to the surrounding metal.

Stability of vacuum in a Röntgen tube is of the utmost importance, as the character of the rays is so largely determined by the vacuum. Metal, which under the influence of the high temperature vaporizes from the target, condenses on the glass in finely divided form and absorbs relatively large amounts of gas, thus changing the vacuum. At high temperatures tungsten vaporizes least of all the metals.

In the above case, practice seems to be in full accord with the theory, and the tungsten target offers great promise to the Röntgen-ray worker. It gives him what he has not had before, an indestructible target, so far as the present electrical apparatus goes, together with sharper definition and much shorter exposure.

#### TUNGSTEN PROJECTILES.

The use of wrought tungsten as a projectile is being carefully investigated. It offers, in this field, possibilities not possessed by any other metal.

The present small arm service projectile is made of lead with a jacket of copper-nickel alloy. The principal advantage of lead over iron, which would of course be cheaper, is that it has a higher specific gravity. Because of this fact a lead bullet will have a smaller cross-section, and will therefore encounter less air resistance to its flight, than will an iron bullet of the same weight, and will therefore give a flatter trajectory and longer range. An iron bullet of the same diameter as the lead bullet could of course be given the same weight by increasing its length. But this would at once necessitate giving it a higher rotational velocity to keep its axis tangential to its flight. To impart this added rotational velocity calls for the expenditure of energy and so leaves less for velocity of translation. The lead bullet then with its higher density makes possible a flatter trajectory and longer range. With the exception of tungsten, lead is the densest metal which can be considered for this purpose, for gold is the cheapest of the other elements having a higher specific gravity than lead. The density of wrought tungsten is 19.3 while that of lead is 11.5.

For military purposes, the softness of lead is not an advantage, a soft nosed bullet being tabooed in civilized warfare. For this reason and because of the fact that it is too weak to hold the rifling, it has to be jacketed with the copper-nickel alloy. To take the rifling and to act as a gas check, the tungsten bullet will require a copper band or its equivalent at the base.

The hardness and high tensile strength of wrought tungsten will give high penetrating power.

The high melting point of tungsten will prevent it from being harmfully upset at the base by the combined action of the high temperature and rapid impact due to the combustion of the powder charge.

(An unsymmetrical upsetting of the base of a projectile is very prejudicial to accuracy.)

It would be a very simple matter to calculate the constants of the trajectory of a tungsten projectile were it not for the fact that the high density removes it too far from the present base line. For such calculations one quantity is lacking, the so-called "form factor." This factor could itself be calculated if it contained, as the name would seem to imply, only the dimensions and the specific gravity; but there are also involved in it all of the errors due to simplifying assumptions which have been made in connection with the mathematical derivation of formulae. It therefore becomes necessary to experimentally determine the constants of the trajectory of tungsten bullets, and preparations for this work are now being made.

W. D. COOLIDGE.

#### "THE FELLOW WHO DOESN'T KNOW ANY BETTER."

There are in the United States according to the census more than eight thousand chemists. Most of these chemists have spent at least three years and many have spent six or seven years in the college or university acquiring a knowledge of chemistry. Above all other men they are acquainted with chemical laws and processes. According to President Maclaurin and President Lowell, to judge from some of their comparatively recent addresses, the earth and the fulness thereof during the present century is to belong to the chemist. Aside from his meekness, however, does the chemist today show any indications of entering into his inheritance? Is the chemist today, in this country, originating as many new products and processes as he should? Is he even originating his fair proportion of new chemical products and processes?

In the older and more strictly chemical industries, such as the acid, heavy chemical, alkali and dyestuff industries for instance, the chemist seems to be in control and progress in these industries may be credited to him. But there are other industries in which chemical principles of the highest order are involved which for the most part are not under chemical control; such as, the paint, varnish, dyeing, leather, alloy, shoe ink and shoe polish industries. There are in some instances chemists in control who are making good, but for the most part the chemist in such industries as are mentioned above is employed in testing raw materials and finished products. He may be even essentially ignorant of the manufacturing processes.

Consider, for example the manufacture of shoe inks and polishes. These are important industries and the manufacture of these products is a chemical process. Moreover, American shoe inks and polishes are sold all over the world, some of our largest companies having branch factories in England, Germany and France. But in this essentially chemical industry there are almost no chemists. Their products and processes have, for the most part at any rate, originated with rule-of-thumb men, the so-called

practical men. Conditions in the other industries mentioned are similar, though in a lesser degree perhaps.

The first really successful one-set edge ink for shoes was stumbled on by a man who added a basic color to an emulsion of carnauba wax and soap, instead of an acid color as called for by the formula. If he had been a chemist he would have known better. He would have known that a basic color would undergo double decomposition with the soap, causing a coagulation of the emulsion. That is just what the basic color did in this case, but after stirring and straining it was just as fluid as before. The blacking man now throws his wax with acid to get the desired result. What chemist in his right mind would have added acid to an emulsion of wax and soap expecting to obtain a desirable result?

It keeps the chemist busy stretching his conception of chemical laws, theories and hypotheses to explain how the despised rule-of-thumb man gets the results he does.

The chemist, at any rate the common, garden-variety of chemist, suffers from a sort of *chemico-sclerosis*. His conceptions of chemical laws become hardened and his theories and hypotheses inflexible. He knows that sodium chloride added to a silver salt solution produces a precipitate, but until colloids became fashionable how many chemists knew that in the presence of a protective colloid-like albumen the silver chloride would not precipitate? He knows that silver iodide, bromide and chloride are insoluble and concludes that silver fluoride is also. He knows that hydrogen sulphide gives black mercuric sulphide with a solution of mercuric chloride and so the United States Pharmacopoeia states that under the conditions of the 'Gutzeit Test' for arsenic, hydrogen sulphide gives a black stain. Under usual conditions, however, the stain is yellow and may be thought to indicate arsenic.

The chemist finds it difficult to work without a theory and neglects to follow the leads that according to his theory do not promise results. But results have been obtained that were not in exact accordance with the preconceived theory. Perkin had a theory for making quinine and found the first aniline dye in spite of this theory.

There is also a certain chemical snobbery that prevents chemists following leads of dubious respectability. In 1873 it was discovered that a dye resulted from the fusion of sawdust and sodium sulphide. The dye was weak and of no ascertainable constitution, but that such a fusion gave a dye at all was a highly significant fact and even a moderate amount of investigation would have improved this dye tenfold. But as the father of the dye was sawdust, of course, no self-respecting chemist would recognize it, and nothing was done on sulphur colors until twenty years later a sulphur dye of perfectly respectable parentage was discovered. Then hundreds of chemists were set to tilling the field.

The practical, rule-of-thumb man with a natural bent for research labors under none of these handicaps. He is free. He staves ahead untrammelled by theory and now and then gets the result. He



may put on the market a wood stain reeking with ammonia as an *acid* stain but it sells and does the work. He gets results often enough to lend a color of truth to the statement that "It's generally the fellow who doesn't know any better who does the thing that can't be done.

The fellow who doesn't know any better," will not make this century the century of chemistry. It will be done by the chemist who realizes the flexibility of our great laws, the instability of hypothesis, the unreliability of analogy and who is not afraid of the dark.

W. H. WATKINS.

## ORIGINAL PAPERS.

### DETERIORATION AND SPONTANEOUS HEATING OF COAL IN STORAGE.<sup>1</sup>

By HORACE C. PORTER AND F. K. OVITZ.

Received December 11, 1911.

Not many years ago, coal was commonly regarded as an extremely unstable material, subject to very serious alteration and losses on exposure to the elements. E. C. Pechin, in 1872, speaking before the American Institute of Mining Engineers, says: "Fuel suffers materially by storage; especially with bituminous and semi-bituminous coals is the loss heavy, an exposure of only two weeks causing a loss of carbon to the extent of 10 to 25%." Similar views have been held in much more recent times. For example, in a paper before the U. S. Naval Institute in 1906 we find the statement: "The pressure of the weight of coal causes gases to be evolved; these gases constitute the chief and only value of the coal in that they furnish the heat units. It is claimed that if a ton of fine bituminous coal be spread out on a concrete pavement in the open air in this climate (Key West, Florida) for one year, it will lose all its calorific properties. The gases are simply free to escape, and when the coal has lost all its gas it will have lost all its heat units and be simply coke." This was only five years ago.

In 1907 a German gas-works engineer claims to have found that moist fine coal sustained an average loss per week of 1.7 per cent., this loss being due to gas. The 1889 edition of Groves and Thorp's "Chemical Technology of Fuels" says: "In some places coal is known to lose 50 per cent. of its heating value in six months." Other statements like these are to be found in recent literature, but probably the great majority of chemists and engineers to-day hold no such exaggerated ideas on the subject. There is, on the other hand, a well-defined suspicion, in the minds of many that sufficient loss of volatile matter and sufficient deterioration by oxidation does occur in coal to be of industrial importance; and for that reason the investigations described in this paper were undertaken by the Bureau of Mines to determine accurately the extent of the deterioration in different types of coal.

First a study was made in the laboratory of the loss of volatile matter from crushed coal during storage. A number of samples (20 lbs. each), representing a variety of types from widely separated fields, were broken to about 1/2-inch size and immediately placed

in glass bottles in the mine. At the laboratory the accumulated gas was withdrawn and a free continuous escape of the volatile products permitted at atmospheric pressure and temperature. The results of these experiments have been published in Technical Paper No. 2, Bureau of Mines, entitled "The Escape of Gas from Coal" and will therefore not be given here in detail. Suffice it to say that while several coals evolve methane in large volumes especially in the early period after mining the coal suffered in one year a loss in calorific value from this cause of but 0.16 per cent., as a maximum.

It seems therefore that the loss due to escape of volatile matter from coal has been greatly overestimated.

At the instance of the Navy Department, however, which is a purchaser of coal to the extent of two or three million dollars annually, and stores large lots in warm climates for long periods of time, more elaborate tests were undertaken to determine the total loss possible in high-grade coal by weathering. The extent of the saving to be accomplished by water submergence as compared to open-air storage was a point to be settled, and there had also arisen the question as to whether salt water possessed any peculiar advantage over fresh water for this purpose. An English railway and dock superintendent in an article in the *London Engineer*, 1903, reports that he has found coal accidentally submerged for 10 years in the salt mud of the English Channel, actually improved in calorific value by 1.8 per cent. which offers a very pretty question to chemists to say why." He claims that salt will preserve the virtues of coal as it does those of many other things, "if coal is given a strong dose of coarse salt and water 12 hours before using, its calorific value is greatly improved."

Coal-storage problems have assumed importance during the last few years on account of the uncertainties of supply due to strikes and transportation difficulties. The naval coaling stations, the Panama R. R. Co., the Great Lakes commercial coal distributing companies, large coke and gas or power plants at a distance from the coal fields and the railroads themselves, particularly those in the west, keep 50,000-500,000 tons in storage a great deal of the time.

In brief outline, the tests by the Bureau were carried out as follows: four kinds of coal were chosen: New River on account of its large use by the Navy; Pocahontas as a widely used steaming and coking

<sup>1</sup> Presented, by permission of the Director, Bureau of Mines, at a joint meeting of the New York Section, American Chemical Society, American Electrochemical Society, and the Society of Chemical Industry, New York, November 10, 1911.

coal in the eastern section, and as being also the principal fuel used in the Panama canal work; Pittsburgh coal as a type of rich coking and gas coal; and Sheridan, Wyoming, subbituminous or "black lignite"—a type much used in the west. With the New River coal, 50-pound portions were made up out of one large lot, which had been crushed to  $\frac{1}{2}$ -inch size and well mixed. These portions confined in perforated wooden boxes were submerged under sea water at three navy yards, differing widely from each other in climatic conditions, and 300-pound portions from the same original lot were exposed to the open air, both out of doors and indoors, at the same places.

With the Pocahontas coal test was made only at one point, the Isthmus of Panama—run-of-mine coal being placed in a 120-ton pile, exposed to the weather. Pittsburgh coal was stored as run-of-mine in open outdoor bins, 5 tons capacity, at Ann Arbor, Michigan, also in 300-pound barrels submerged under fresh water. The Wyoming subbituminous was stored at Sheridan, both as run-of-mine and slack in outdoor bins, holding three to six tons each.

Every test portion was sampled each time in duplicate and in all cases except the outdoor pile at Panama and the 300-pound open-air piles of New River coal the sampling was done by rehandling the entire amount. In the excepted cases mentioned it was not thought fair to disturb the entire lot, and therefore at Panama a vertical section of ten tons only was removed each time (eight samples being taken from the 10-ton section), while in the case of the outdoor lots at the Navy Yards a number of small portions, well-distributed, were taken from each pile, mixed, and quartered down.

Small lots and a fine state of division were conditions purposely adopted with the New River coal so as to make the tests of maximum severity.

Moisture, ash, sulphur, and calorific value determinations were made on each sample, the latter by means of the Mahler bomb calorimeter and a carefully calibrated Beckmann thermometer. The calorimetric work on all except the Sheridan, Wyoming, tests has been done throughout by one man, Mr. Ovitz, and with the same instrument. All the calorific values in the tables have been calculated to a comparable unit basis, viz., that of the actual coal substance free of moisture, sulphur, and corrected ash.

The results show in the case of the New River coal less than 1 per cent. loss of calorific value in one year by weathering in the open. There was practically no loss at all in the submerged samples and fresh water seemed to "preserve the virtues" of the coal as well as salt. There was almost no slacking of lump in the run-of-mine samples and the crushed coal in all cases deteriorated more rapidly than run-of-mine.

The Pocahontas run-of-mine in a 120-ton pile on the Isthmus of Panama lost during one year's outdoor weathering less than 0.4 per cent. in heating value,

and suffered little or no physical deterioration of lumps.

The Pittsburgh gas coal during six months' outdoor exposure suffered no loss whatever of calorific value, measurable by the calorimetric method used, not even in the upper surface layer of the bins.

The Wyoming coal lost as much as 5.3 per cent. in one of the bins during two and three quarter years, and 3.5 per cent. even in the first three months. There was bad slacking and crumbling of the lumps on the surface of the piles but where the surface was fully exposed to the weather this slacking did not penetrate more than 12-18 inches in the  $2\frac{3}{4}$ -year period.

No outdoor weathering tests have been made by the Bureau on coal of the Illinois type. Thorough tests, however, on this type have been reported by Prof. S. W. Parr, of the University of Illinois, and by A. Bement, of Chicago, both of whom find from 1.0-3.0 per cent. in calorific loss in a year by weathering. Mr. Bennet reports a slacking of lumps (in tests on small samples) of over 80 per cent. in one case and about 12 per cent. in another. It is probable that in this type as in the Wyoming, the slacking in a large pile would not penetrate far from the surface.

Storage under water unquestionably preserves the heating value and the physical strength of coal. But it practically necessitates firing wet coal, and therefore means the evaporating in the furnace of an amount of moisture varying from 1-15 per cent., according to the kind of coal. This factor is an important drawback to under-water storage with coals like the Illinois and Wyoming types, which mechanically retain 5-15 per cent. of water after draining, but in case of the high-grade eastern coals, if firemen are permitted, as is ordinarily the case, to wet down their coal before firing, "so as to make," as they say, "a hotter fire," then the addition during storage of the 2 or 3 per cent. moisture which these coals retain would be of little consequence. Submergence storage is an absolute preventative of spontaneous combustion, and on that account alone its use may be justified with some coals, but merely for the sake of the saving to be secured by avoidance of weathering there does not seem to be good ground for its use.

#### SPONTANEOUS COMBUSTION.

Losses in coal due to spontaneous heating are a much more serious matter. Oxidation, *i. e.*, probably in the main an absorption of oxygen by the unsaturated chemical compounds in the coal substance, begins at ordinary temperature in any coal, attacking the surfaces of the particles, thus slowly developing heat. In a small mass of coal this slowly developed heat can readily dissipate itself by radiation and no rise in temperature results. If radiation is restricted, however, as in a large pile densely packed, the temperature slowly rises. Now, the curve of oxidation rate plotted against temperature rises with great rapidity, and when the storage conditions are such as to allow a certain point (near 100° C.) to be passed the rate of oxidation is great enough ordinarily so

that the heat developed overbalances the heat radiated and the temperature will rise to the ignition point if the air supply is adequate. The importance therefore can be seen of guarding against even moderate heating in the coal either from internal spontaneous causes or by radiation from external sources. Increased loss of heating value and of volatile matter occurs at moderately increased temperatures even though the ignition point is not reached.

The amount of surface exposed to oxidation in a given mass depends on the size of the particles and increases very rapidly as the fineness approaches that of dust. Dust is therefore a dangerous thing in a coal pile, particularly if it is mixed with larger sized coal which forms air passages to the interior. Spontaneous combustion is brought about by slow oxidation in an air supply sufficient to support the oxidation but insufficient to carry away all the heat formed. There is a wide variation among coals in friability. In comparative rattler tests under certain standard conditions, Pocahontas, New River and Cambria Co., Pa., coals produced nearly twice as much dust (through  $\frac{1}{8}$ " screen) as a sample from the Pittsburgh seam. This is a large factor in spontaneous combustion. Mixed lump and fine, *i. e.*, run-of-mine, with a large percentage of dust, and piled so as to admit to the interior a limited supply of air, make ideal conditions for spontaneous heating.

High volatile matter does not of itself increase the liability to spontaneous heating. A recent circular letter of inquiry on spontaneous combustion sent by the Bureau to more than 2,000 large coal consumers of the United States has brought 1,200 replies, of which 260 report instances of spontaneous combustion, 220 of them naming the coal. Of these 220, 95 are in semi-bituminous low-volatile coals of the Appalachian region, and 55 in western and middle western coals. This result shows at least no falling behind on the part of the "smokeless" type and no cause for placing special confidence in these coals for safety in storage.

A serious fire in cinder filling under a manufacturing plant in Pittsburgh was recently investigated by the Bureau and all the evidence pointed to spontaneous combustion as the cause, induced by external heat radiated from a furnace. The cinders contained 40 per cent. of carbon. A similar fire occurred two years ago in cinder filling under a smelting plant on Staten Island in which case the cinders contained 33 per cent. carbon. Damage amounting to \$20,000 was done. The cause was not definitely determined, but from the reports of the insurance adjusters spontaneous heating appears to be the most plausible explanation. The volatile matter in the material could not have been a factor in these causes.

Pocahontas coal gives a great deal of trouble with spontaneous fires in the large storage piles at Panama. It is reported also by several large by-product coke concerns to be more troublesome in this respect than their high volatile gas coals. The high volatile coals of the west are, to be sure, usually very liable to

spontaneous heating, but they owe this property to chemical nature of the substances which compose the coal rather than to the amount of volatile matter. Strange as it may seem, a high oxygen content in coal appears to parallel its avidity for oxygen and to promote therefore its tendency to spontaneous combustion.

The influence of moisture and that of sulphur upon spontaneous heating of coal are mooted questions much discussed, not very much actually investigated, and certainly not yet settled. *Richters* has shown that in the laboratory dry coal oxidizes more rapidly than moist, but the weight of opinion among practical users of coal is that moisture promotes spontaneous heating. The observation by the Bureau of many actual cases has not developed any instances where moisture could be proven to have had such an effect. Sulphur on the other hand has been shown by these investigations, to have in most cases only a minor influence. In a number of actual cases, samples of the heated coal from areas where the heat was greatest have been analyzed, both for the total sulphur and that in the sulphate form. The difference between these, or in other words, the unoxidized sulphur was in no case less than 75 per cent. of the average total sulphur in the original. In other words, not more than  $\frac{1}{4}$  of the total sulphur has entered into any heat-producing reaction. The possibility remains, however, that all of the sulphur which was oxidized was concentrated in one pocket of moist flaky pyrites, and thus sufficient heat was developed in one spot to act as an igniter. On the other hand, a Boston company, using Dominion (Nova Scotia) coal of 3-4 per cent. sulphur, has much trouble with spontaneous fires in storage, but a number of samples taken by the Bureau from exposed piles of this coal in which heating had occurred showed that 90 per cent. of the sulphur was still unoxidized. Experiments in the laboratory, passing air over coal at 120° C., have developed enough heat to ignite the coal and no change was found in the form of the sulphur. While not entirely conclusive, these results point to a very minor contribution, if any, on the part of sulphur to spontaneous heating in coal.

Freshly mined coal and even fresh surfaces exposed by crushing lump coal exhibit a remarkable avidity for oxygen, but after a time become coated with oxidized material, "seasoned" as it were, so that the action of the air becomes much less vigorous. It is found in practice that if coal, which has been stored for six weeks or two months and has even become already somewhat heated, be rehandled and thoroughly cooled by the air, spontaneous heating rarely begins again. A large power plant in New York crushes its coal to pass a 4" screen immediately after unloading from barges, the fines and dust, 50 per cent. or more, being left in the coal to be stored. This freshly crushed coal is elevated to iron hopper-shaped bunkers directly over the boilers and the air temperature in these often reaches 100° F. As the coal hangs on the sloping sides sometimes three or four months at a time, it seems hardly surprising that some of the bunkers are on fire practically all of the time.

With full appreciation of the fact that any or all of the following recommendations may under certain conditions be found impracticable, they are offered as being advisable precautions for safety in storing coal whenever their use does not involve an unreasonable expense:

(1) Do not pile over 12 feet deep nor so that any point in the interior will be over 10 feet from an air-cooled surface.

(2) If possible, store only lump.

(3) Keep dust out as much as possible; therefore reduce handling to a minimum.

(4) Pile so that lump and fine are distributed as evenly as possible; not as is often done, allowing lumps to roll down from a peak and form air passages at the bottom.

(5) Rehandle and screen after two months.

(6) Keep away external sources of heat even though moderate in degree.

(7) Allow six weeks' "seasoning" after mining before storing.

(8) Avoid alternate wetting and drying.

(9) Avoid admission of air to interior of pile through interstices around foreign objects such as timbers or irregular brick work; also through porous bottoms such as coarse cinders.

(10) Do not try to ventilate by pipes as more harm is often done than good.

#### THE DISTILLATION OF ALCOHOL.<sup>1</sup>

By A. B. ADAMS, Chief Chemist, Internal Revenue Bureau.

Received July 13, 1911.

The production of alcohol differs essentially from the production of straight whiskey, because it is necessary that the finished product be as near pure ethyl alcohol as is possible, by mechanical means, to distil. To effect this the processes are essentially the same up to a given point; that is, as far as the primary distillation, this being conducted in a continuous beer still which is built exactly similar to the continuous beer still used in a majority of the "Bourbon" houses. The distillate, however, is produced at a much higher proof, generally at about 160 degrees or 80 per cent. alcohol by volume. The high wine distillate is reduced with water to about 101 proof, and passed through charcoal filters under pressure. After filtering, the spirit is emptied into the kettle of the "column" still. This still consists of a series of plates ranging in number from 18 to 30 or more, depending on the type and size of the plant. These plates act as dephlegmators, each one constantly condensing the products of higher boiling points, permitting the products of lower boiling points to proceed upward. When the vapors leave the top of the still they enter a series of condensers, in some types, called a "goose," which consists of a series of pipes arranged very much like a radiator. These pipes are surrounded by cooling water at a temperature practically one degree higher

than the boiling point of ethyl alcohol. The purpose of this apparatus is to condense all products as far as possible, which boil at a higher temperature than ethyl alcohol and which have escaped the action of the plates in the still. From the "goose" the vapors pass into the condenser. In recent years, on account of the excessive weight of the "goose," which in some distilleries is a tank as large as a good-sized room, a simpler apparatus has been devised, called a "tubular separator;" this operates on the same principle as the "goose," namely, is cooled by water at the same temperature. This is considered by many to be as efficient a piece of apparatus.

At the "tail box" or end of the condenser, the distillate is separated into various fractions, depending on the quality, by the operator of the still. These are generally called, in the order distilled, "foreshots," or low wines, "heads," "middle run," "tails," "low wines" and lastly, "fusel oil." The point at which the different fractions are cut out depends upon the class of goods desired by the operator. The "middle run" is the cleanest part of the distillation, and when a very clean spirit is desired is frequently separated in a No. 1 and a No. 2 spirit. The "foreshots" and low wines are the poorest parts of the run, containing the greatest amount of bad smelling impurities, which render them unfit for commercial purposes. In this condition they are generally collected for several days and then redistilled in the "column." (The better part of such a redistillation is classed as "commercial alcohol.") The "heads and tails," if separated fairly clean, are combined and called "commercial alcohol." The "middle run," No. 1 and No. 2 spirit go into the trade as such. The "fusel oil" is not run off at the end of each charge but is permitted to remain in the still and on the plates of the column until two or more charges have been run, when, in the judgment of the distiller, sufficient "fusel oil" has been collected to warrant its distillation; this is conducted by simply turning on more steam when the last of the "low wines" have been run from the charge then in the kettle.

The last portion of the "low wines" will consist of increasing proportions of fusel oil and water. When the per cent. of fusel oil is so great that it does not remain in solution, the flow in the tail box becomes milky, and the subsequent distillate is distilled into the "fusel oil" tank. The fusel oil is gradually forced up the column from plate to plate, being carried upward by "steam distillation." Although water is of a lower boiling point than the amyl alcohols, yet it does remain on the plates of the column after the fusel oil has been driven up. This can be proven by opening the try cocks at each plate in the still during the course of the distillation.

#### METHODS OF ANALYSIS.

The analytical methods used were those as found in *Bulletin 107*, Bureau of Chemistry, Department of Agriculture, under "Distilled Liquors," with changes in treatment for esters and higher alcohols.

*Proof.*—The proof of the samples was determined

<sup>1</sup> Published by authority of the Commissioner of Internal Revenue. See companion article, "The Distillation of Whiskey," THIS JOURNAL, Feb., 1910.

with a regular Internal Revenue hydrometer. One degree of proof is equal to one-half of one per cent of alcohol by volume.

*Acids.*—Fifty cc. titrated with  $N/10$  NaOH; use phenolphthalein as indicator; calculated to acetic acid.

*Esters.*—Saponify with soda, titrate excess with sulphuric acid, calculate results to ethyl acetate. The above method was used for the determination of esters in all of the samples but the following: Nos. 36814 to 36826, inclusive; in these samples, on account of the content of aldehyde bodies, the true ester value could not be determined until they were removed. These samples were treated by the method as given in *Allen*, Third Edition, page 158. The spirit being treated with 1 cc. aniline and 1 cc. phosphoric acid, refluxed, distilled, then proceed in regular manner. I find that this method gives results a trifle lower than are correct, but this was compensated for by adding a correction.

*Higher Alcohols.*—The Allen-Marquardt method was used. Samples Nos. 36814 to 36828 had to be treated to remove the aldehydes and acetals. This was done by successive treatments with alkali to form condensation products, distilling; then treat with HCl to break up the acetal into aldehyde; then treat with alkali and distil, repeating until the distillate was clean. Blanks were run to see that this treatment would give true results.

*Aldehydes.*—Determined according to the prescribed method, being a colorimetric comparison. Test made with a solution of fuchsin dissolved in  $SO_2$ .

*Furfurols.*—Used prescribed method, a colorimetric comparison. Test made by treating the spirit with 2 cc. aniline and  $\frac{1}{2}$  cc. hydrochloric acid.

In proceeding with the plan of the work outlined a distillery was selected which produced alcohol as a by-product from the manufacture of yeast. The mash is made in the usual manner in about the following proportions: Malt 34%, rye 5%, corn 61%.

*Fermentation.*—The fermentation is a very rapid one as the mash is aerated during the entire process, which is complete in sixteen hours. The result of such aeration produces a very pure and a very strong yeast, but at the expense of more or less alcohol which is lost by evaporation, and more particularly by oxidation into aldehydes, etc. On account of the high aldehyde content in such a mash or "beer" it is acknowledged to be one of the most difficult to distil clean; that is, to obtain a good grade of alcohol.

*Primary Distillation.*—The finished fermentation, or as it is called, the "beer," is distilled in a large continuous beer still, the operation of which is described in a previous article.<sup>1</sup>

The still is three feet in diameter, containing fourteen chambers, each eighteen inches in height. It produces continuously a spirit of about 150 proof, called "high wines." Such a still after it has been once set produces a similar distillate during its entire

period of operation, except at the beginning and end, when a full head of beer is not on the still.

*Filtration.*—The product of the beer still is reduced to about 101 proof with pure water and is then passed through charcoal filters under pressure. These filters are cast iron receptacles about  $3\frac{1}{2}$  feet high by 4 feet in diameter, tightly packed with charcoal. The purpose of such filtration is to eliminate from the high "wines" some of the raw odors of the spirit. It was at one time thought that this filtration, or as it is erroneously called, rectification, removed the fusel oil. The results of the analysis of these samples, as well as of the products of six other distilleries using similar filters, prove this to be incorrect, as the analyses in no case show more than a slightly lower per cent. of higher alcohols.

For example:

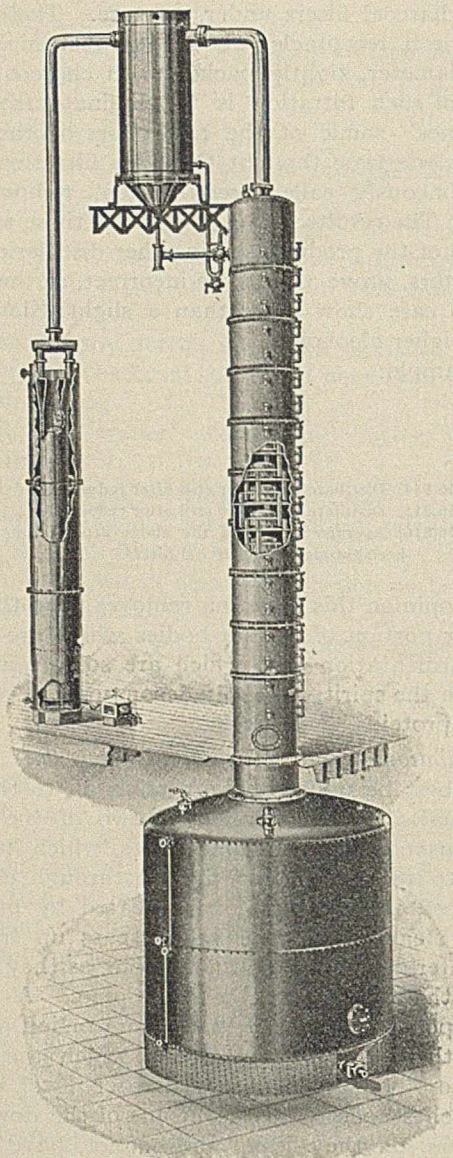
	Higher alcohols. Grams per 100 liters.
Sample No. 36,814: Distillate from beer still after reduction.....	100.2
Sample No. 36,815: Distillate from beer still after reduction.....	84.2
Sample No. 36,816: Average sample of the above after having been through the charcoal filters.....	86.2

In my opinion this filtration removes from the spirit the major portion of certain bodies which are formed during fermentation and which are soluble in water and not in the spirit, probably decomposition products from the proteids of the grain.

*Redistillation.*—The construction of the column still can best be understood by referring to the illustration and the cross section of two of the plates. Four thousand gallons of high wines, which had been reduced to 101° proof and filtered through charcoal, were placed in the kettle; it is heated by means of a closed steam coil. The vapors pass up from the kettle, entering the still at the bottom, as they ascend, striking the bottom of the first plate. A portion of the vapors is condensed; the larger portion proceeds upward through the three pipes which are underneath the "bonnets." As the water level of each plate reaches exactly to the height of the down pipe, the vapors, as they leave the bonnet, have to pass through this level of liquid. More of the vapors are condensed, the remainder pass upward against the next plate and enter the bonnets, and so on. Those products which are condensed flow downward, a constant level being kept on the plates by means of the down pipes. Products which flow to the bottom of the still are returned to the kettle automatically. The column still from which the samples were taken has twenty-three chambers and one boiling dome. The vapors after passing through the highest chamber enter a tubular separator, as previously explained; this is kept very near the boiling temperature of ethyl alcohol. Any products which are condensed are returned to the top of the still by means of the small trapped pipe. The vapors on leaving the separator enter the condenser where they are condensed to a liquid. It takes about thirty hours to run off one charge of the kettle. Three charges of the kettle are run and the fusel oil distilled on the third running

<sup>1</sup> THIS JOURNAL, Feb., 1910.

only, the still being thoroughly cleaned out after this distillation. The samples were taken from the running of such a third charge, and, therefore, include fusel oil samples. By referring to the analytical table

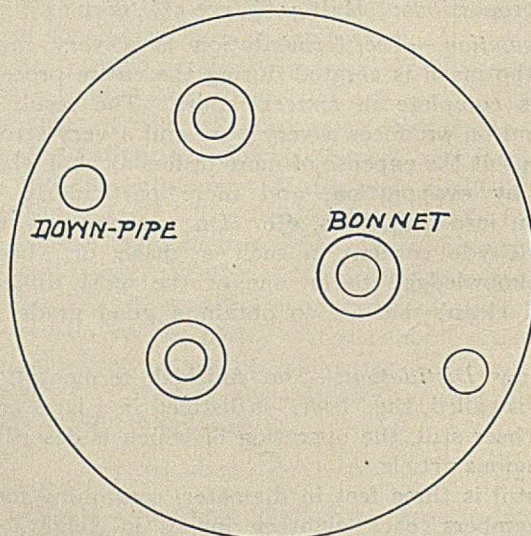
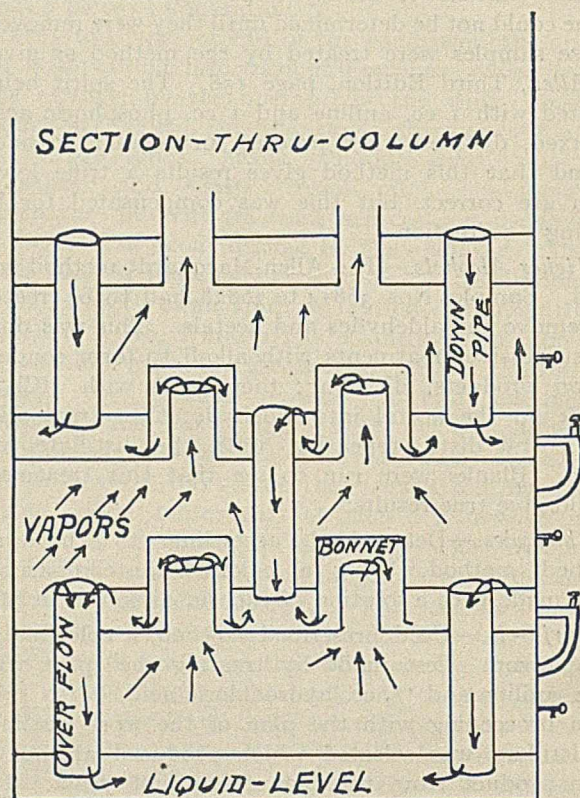


it will be seen, when the samples were taken, what part of the run they represent, and the analysis. Only three samples were taken during the middle run because necessarily this would be a clean spirit. More frequently samples were taken at the beginning of the distillation and at the end on account of the changes occurring in these portions of the runs.

The first two samples, Nos. 36817 and 36818, are not representative of the very first runnings of the charge in the kettle at the time, but contain an admixture of products left on the plates from the two previous distillations. When the third sample, No. 36819, was taken, the still appeared to be running on the charge in the kettle; this is shown by the drop in the proof. The proof gradually rises until  $191^{\circ}$  is reached which is the highest proof wanted. The speed of the distillation is regulated by the operator, it being desired to eliminate, in the first part of the distillation,

as much as possible of the aldehydes and similar products. After the middle run has been distilled the proof gradually drops. It could not be read after 12.21 P.M., when it was  $165^{\circ}$ . It goes lower than this but as the flow of spirit in the tail box is cut out of the hydrometer cup in order not to foul it with fusel oil, the reading could not be made.

*Acids.*—There is a constant appearance of acids in the distillate, although the highest percentage appears in the first portions of the run. By the time the middle run is reached the spirit is nearly free of this constituent.



*Esters.*—The same thing can be said of esters as of the acids.

*The Higher Alcohols.*—The results of the first three samples show a rather definite percentage of

higher alcohols. I am of the opinion that this is due, not so much to the distillation of this charge, but to the fact that fusel oil which had remained upon the plates from the previous distillation, had been forced up the still and into the tail box before the still could be regulated. In the next sample the content has dropped to 19 which is a rather small amount. These results practically remain constant throughout the remainder of the distillation, until about the tail is reached, when the percentage of higher alcohols begins to increase. It is very noticeable in sample No. 36835. In the sample taken forty-five minutes later it has increased to nearly a per cent. In the sample taken sixteen minutes later it has increased to 2.6 per cent., and in the last sample taken, which was just before the "fusel" began to distil, the content was over 16.3 per cent.

*Aldehydes.*—As previously stated, alcohol produced by the aeration of the mash contains an excessive amount of aldehydes. A large portion of the aldehydes go over in the first runnings, the majority having been driven off at the end of two hours' distillation. When the middle run is reached the content of aldehyde is almost *nil* and remains so throughout the distillation, showing that practically all the aldehydes have been distilled.

*Furfurol.*—No furfurol was found in the products of the beer still, and none was found in the distillate of the column. These results bear out the statement, previously made from this laboratory, that furfurol had never been found in the distillate from a continuous beer still.

*Acetal.*—The percentage of acetal and similar bodies, other than aldehydes, was not determined, there being no feasible method. The presence of acetal is shown in samples 36814 to 36828, inclusive; only a very small amount was found in the last few samples. It is to be expected that such bodies will be present in spirit when such a high percentage of aldehydes is contained therein, these products being produced by the action of the aldehydes on ethyl alcohol.

*Quantities in Each Fraction.*—Of the 4,000 gallons of 101 degrees proof of spirit in the charge of the kettle, neglecting the water, about 3 per cent. is separated as "fore-shots" (low wines), 30 per cent. as "heads," 48 per cent. as "middle run," 16 per cent. as "tails" and 3 per cent. as low wines, fusel oil, 0.25 per cent. An examination of the samples both physical and chemical would indicate that the fractions represented by the two samples taken before, and the two samples taken after the middle run could, without detriment, be added to the higher grade spirit increasing the proportion of "middle run" to about 63 per cent. In a distillery producing alcohol from an unaerated mash the proportion of "middle run" is as would be expected larger than this figure, being between 65 and 80 per cent., depending entirely on the quality desired.

#### SAMPLES FROM THE PLATES OR CHAMBERS.

In order to see at just what stage the purification of the alcohol occurs, it was decided to take samples

from the plates of the still from the bottom up. The analyses of these would show at just which point the spirit was cleansed. As previously stated, the charge in the kettle, which was sampled, was a third one; there was, therefore, remaining on the plates and in the kettle just three times as much higher alcohol as was present in one charge. I thought it best to wait until the still has been cleaned out and a new charge in process of distillation before taking samples from the plates. The best time at which to take representative samples is during the middle of the distillation, when the purest spirits come over. I, accordingly, went to the distillery at 10 o'clock P.M., at which time the middle run had been distilling for two hours. By referring to the illustration it will be seen that there is a little cock just above each plate in the still. The cocks are placed about four inches above the water line of the plate. Being so high it made it difficult to obtain samples in some cases, as only vapor came from the cock. The three chambers nearest the bottom could not be sampled as the cocks had been permanently closed. Samples were taken from Nos. 4, 5, 6 and 7 chambers from the bottom, and then from every other chamber to the top, or 23d making twelve samples. For purposes of comparison I have included in this table Sample No. 36829 which is a sample from the previous run, taken at the condenser, at about the same relative time, showing that the highest proof is reached in the top chamber of the still. By reference to the analytical table and curves it is seen that the proof rather regularly increases from the fourth chamber, where it was 164°, to the top chamber where it was 190.7°.

*Acids and Esters.*—The content of the acids and the esters would seem to indicate that both of these products are formed in the column, as, if this were not the case, there would be no reason for a higher content in the upper than in the lower chambers.

*Higher Alcohols.*—Examination of the higher alcohols proves that the major portion of them is condensed out of the spirit on the lower plates, and when the seventeenth chamber is reached it is as free of this product as the still can make it.

*Aldehydes.*—The determination of aldehydes show only a faint trace.

*Furfurol.*—No furfurol was found in any of the samples.

#### CONCLUSIONS.

The results seem to prove that the essential differences between the column still and the pot still, or what should probably be called a "simple" still, are:

First: That a much higher proof can be obtained from the former.

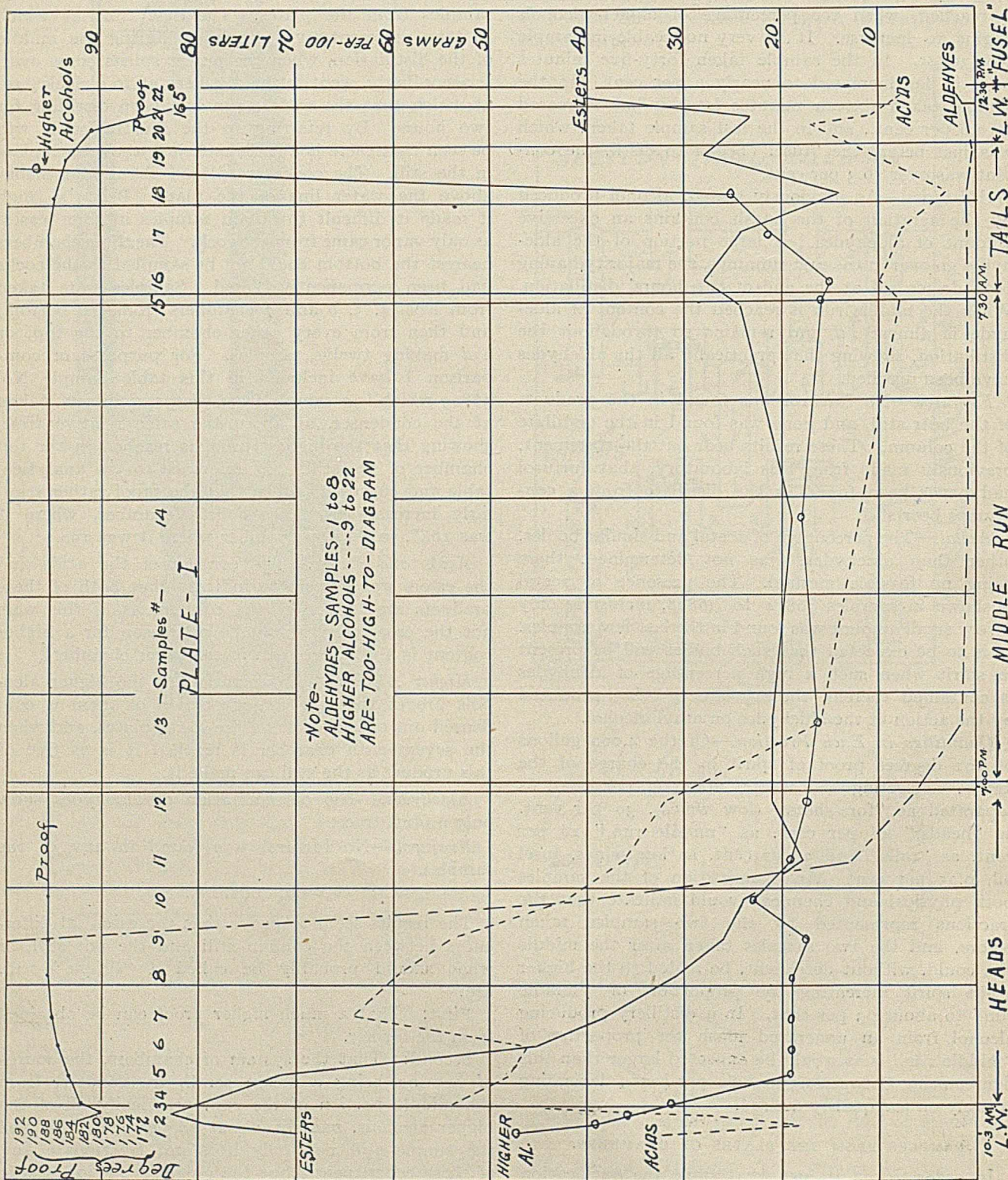
Second: That the system of chambers, the source of heating being a steam scroll, fractionates, from the middle portion of the run, practically all of the higher alcohols, namely, about 80 per cent., whereas the simple still not only does not fractionate any of these constituents, but the greatest portion distils over in the middle part of the run.

Credit should be given Mr. Peter Valaer for the analytical work on the aldehydes and furfurols.

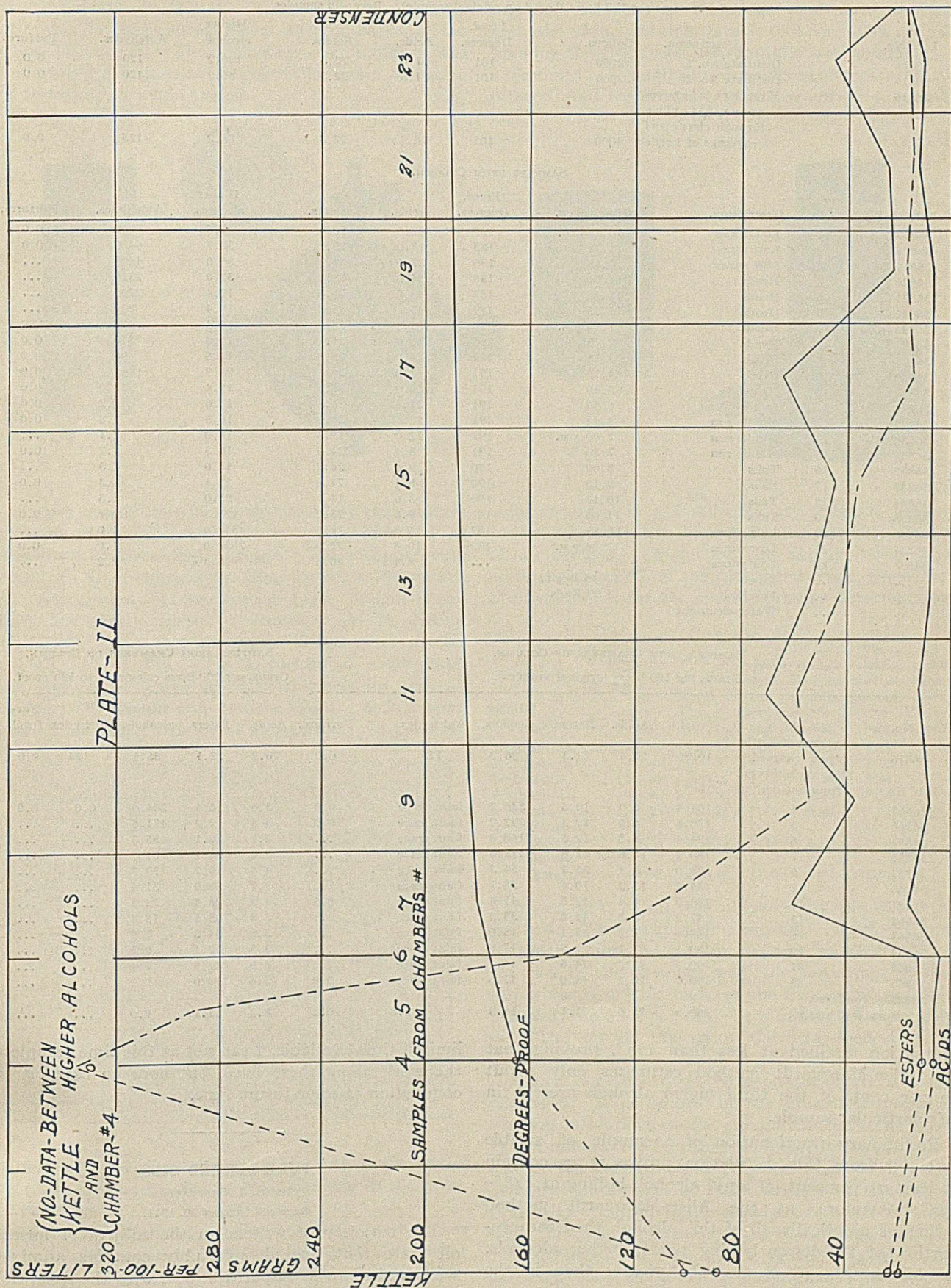
I wish to thank The American Copper & Brass Works, of Cincinnati, Ohio, for the loan of the cut of the column still.

NOTE.—In offering this paper I would state that it is not as complete as it might be, as more work should be done to ascertain the different constituents

of the foreshots and heads, and also the relative proportions of the different higher alcohols in the last portion of the run. For instance, sample No. 36838 gives, with the Allen-Marquardt method, for higher alcohols, 16.3 per cent. by weight; this would be approximately 20 per cent. by volume. After shaking this sample with a large volume of water 40 per cent. of higher alcohols separate, of which a considerable







## SAMPLES FROM ALCOHOL DISTILLATION.

Grams per 100 liters (based on original sample). Beer still samples.

Lab. No.	Description.	Gallons.	Proof. Degrees.	Acids.	Esters.	Higher alcohols.	Aldehydes.	Furfural
36814	Distillate No. 1	2000	101	21.6	22.3	100.2	120	0.0
36815	Distillate No. 2	2000	101	21.6	22.3	86.2	120	0.0
36816	Mixture of above after filtering through charcoal —average of kettle	4000	101	20.4	22.3	86.2	125	0.0

## SAMPLES FROM COLUMN.

Lab. No.	Sample No.	Description.	Time taken.	Proof. Degrees.	Acids.	Esters.	Higher alcohols.	Aldehydes.	Furfural.
36817	1	Low wines	10.13 A.M.	185	32.6	70.4	47.5	6000	0.0
36818	2	Low wines	10.25	185	18.0	79.2	38.7	6400	0.0
36819	3	Low wines	10.45	180	27.6	82.7	36.0	4800	...
36820	4	Heads	11.01	185	54.0	78.6	32.0	2320	...
36821	5	Heads	11.45	187	48.0	71.6	19.4	2200	...
36822	6	Heads	12.35	189	45.6	45.2	19.4	800	...
36823	7	Heads	1.15 P.M.	190	63.6	38.2	19.4	426	...
36824	8	Heads	2.15	191	60.0	31.1	19.0	370	0.0
36825	9	Heads	3.15	191	54.0	25.8	17.6	96	0.0
36826	10	Heads	4.15	191	42.0	24.1	22.9	42	0.0
36827	11	Heads	5.40	191	36.0	17.6	19.4	13	0.0
36828	12	Heads	6.40	191	32.4	21.1	17.6	7.2	0.0
36829	13	Middle run	8.45	191	15.6	21.1	16.3	1.8	0.0
36830	14	Middle run	2.00 A.M.	191	12.0	19.4	18.0	1.1	...
36831	15	Middle run	7.30	191	8.4	24.6	16.3	1.2	0.0
36832	16	Tails	8.00	190	9.6	28.2	15.0	1.3	...
36833	17	Tails	9.13	190	8.4	24.6	21.1	1.3	0.0
36834	18	Tails	10.10	190	3.6	14.1	25.0	1.3	...
36835	19	Tails	11.05	187	9.6	28.2	122.3	trace	0.0
36836	20	Low wines	11.50	182	16.8	17.6	840.0	1.0	...
36837	21	Low wines	12.06 P.M.	172	10.8	22.9	2650.0	1.0	0.0
36838	22	Low wines	12.30	...	8.4	40.5	16.3%	2.2	...
...	..	Fusel oil	12.38 Begins						
...	..	Fusel oil	1.00 Ends						
...	..	Water-clean out							

## SAMPLES FROM CHAMBERS OF COLUMN.

Grams per 100 liters (original samples).

## SAMPLES FROM CHAMBERS OF COLUMN.

Grams per 100 liters calculated to 100 proof.

Lab. No.	Proof.	Acids.	Esters.	Higher alcohols.	Aldehydes.	Furfural.	Acids.	Esters.	Higher alcohols.	Aldehydes.	Fur- fural.	
36816	Average of kettle when charged. No. of chambers from bottom.	101%	20.4	22.3	86.2	125	0.0	20.2	22.1	85.4	124	0.0
36855	4	164.4	6.0	10.6	336.2	faint trace	0.0	3.6	6.4	204.6	0.0	0.0
36856	5	170.8	6.0	12.3	292.9	faint trace	0.0	3.5	7.2	171.5	...	...
36857	6	174.4	4.8	12.3	148.8	faint trace	...	2.7	7.1	85.3	...	...
36858	7	180.4	15.6	61.6	111.0	faint trace	...	8.6	34.1	61.5	...	...
36859	9	183.0	8.4	35.2	54.3	faint trace	...	4.6	19.2	29.7	...	...
36860	11	184.8	13.2	70.4	58.1	faint trace	...	7.1	38.0	31.4	...	...
36861	13	186.0	8.4	52.8	41.4	faint trace	0.0	4.5	28.4	22.3	...	...
36862	15	187.2	7.2	44.0	35.2	faint trace	...	3.8	23.5	18.8	...	...
36863	17	188.4	7.2	65.1	15.0	faint trace	...	3.8	34.5	7.9	...	...
36864	19	189.1	7.2	22.9	17.6	faint trace	...	3.8	12.1	9.3	...	...
36865	21	190.2	13.2	24.6	14.1	faint trace	...	6.9	12.9	7.4	...	...
36866	23	190.7	10.8	44.0	17.6	faint trace	0.0	5.6	23.0	9.2	...	...
36829	at condenser finished alcohol	190.8	15.6	21.1	16.3	1.8	0.0	8.2	11.1	9.0	...	...

proportion distilled at less than 128°, proving that the Allen-Marquardt method estimates only about 50 per cent. of the total higher alcohols present in this particular sample.

Preliminary fractionation of a sample of salable fusel oil from this distillation proves it to contain at least 75 per cent. of amyl alcohol, boiling at 128°–132°; therefore, as the Allen-Marquardt method estimates practically all of this alcohol, then the proportion of the lower boiling point, higher alcohols, in this sample 36838 must be greater than in the marketable fusel oil.

On account of the small samples taken and the

limited time available, I can not at this time complete the work along these lines, but hope to carry it to completion at some future time.

## PURE LINSEED OIL.

By E. J. SHEPPARD.

Received October 20, 1911.

The majority of writers on the subject of linseed oil state that linseed invariably contains admixed seeds and straw as impurities; but, with the exception of the article by Ketel and Antusch,<sup>1</sup> there is no data

<sup>1</sup> Z. angew. Chem., 1896, 581.

available of the precise effect of these impurities on the constants of the oil. Church, however, calls attention to the injurious effect of weed seeds on the color of the oil. The following investigation was undertaken in order to establish the exact influence of these impurities on the oil.

pletely freed of impurities by screening and by hand picking. Portions of the clean seed were analyzed and the data obtained are shown in Table I. On pressing the clean seed, oil sample *C* was obtained.

Samples 1, 2, 3 and 4 are considered as representative of seed used for crushing in this country; 5, 6 and 7

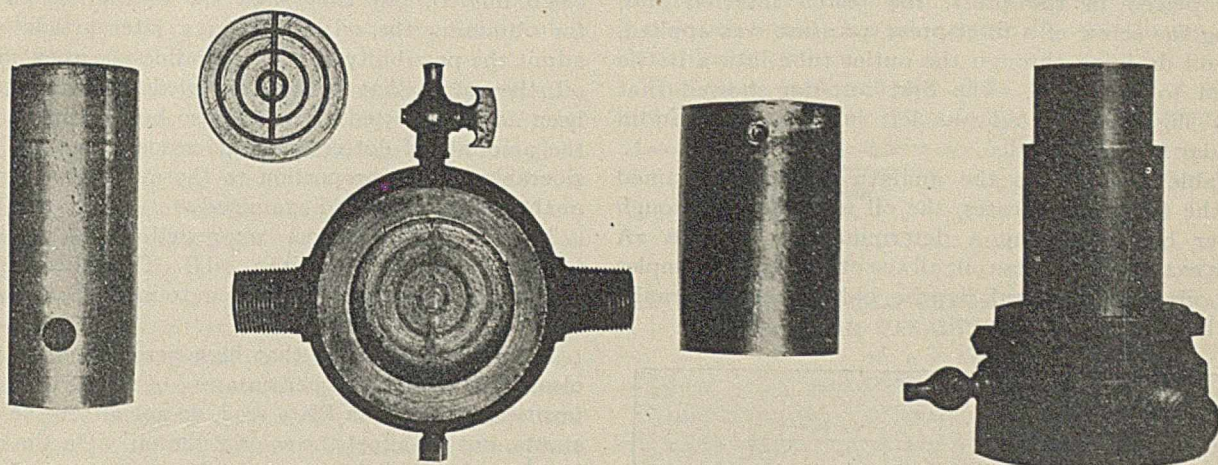


Fig. 1.

It was thought best to obtain the oil by the pressure method rather than by the extraction method as employed by Ketel and Antusch. The accompanying drawings and photographs (Fig. 1) show the details of construction of the special press designed and built for this purpose by Professor A. H. Sabin: *A*, in Fig. 2, is a cast-iron base. *B* is a piece of steel tubing. *C*, in Fig. 3, is a perforated steel plate, the grooves of which serve as channels for the oil; in use it is placed with grooves down. *D* is a solid piece of shafting; this serves as a piston. In practice,

were imported in a small quantity, but are believed to be representative—no authentic factory samples of oil could be obtained. The average weight per seed was obtained on 4000 full-sized seeds; these were afterward used for the specific gravity determination.

TABLE I.

Seed.	Oil. Per cent.	Sp. gr. 15.5° C.	Average weight per seed.		Oleaginous impurities.		Oil in total impurities. Per cent.
			Mg.	Per cent.	Per cent.	Per cent.	
1—American.....	39.67	1.1388	4.61	1.50	1.69	10.0	
2—American.....	39.40	....	4.53	1.01	1.05	..	
3—La Plata.....	36.98	1.1415	5.58	0.58	5.64	14.1	
4—Calcutta.....	40.82	1.1326	5.41	4.85	5.03	14.9	
5—Bombay.....	41.23	1.1182	7.88	0.81	2.80	..	
6—S. Russia (Kertch).....	39.11	1.1375	5.74	5.05	1.71	..	
7—N. Russia (Riga).....	36.95	1.1458	4.19	3.31	1.97	..	

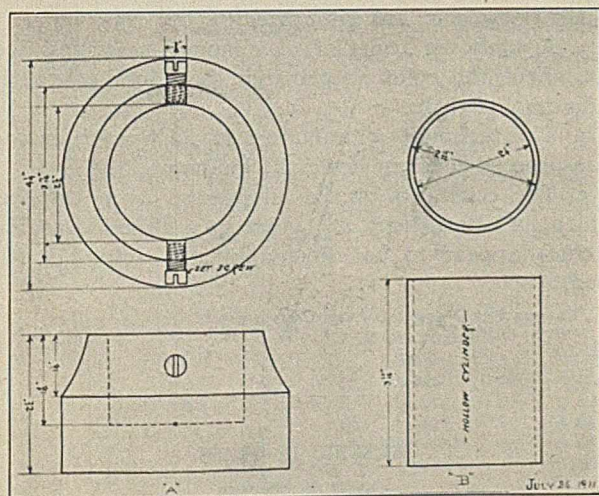


Fig. 2.

a piece of wire gauze fitting inside of *B* is placed on *C*; this prevents the charge from wedging in the holes of the plate *C*.

Samples of seed, as used for pressing, were taken at an oil-mill together with samples of the oil pressed therefrom—Samples A. Portions of the seed as received were pressed in the special press and gave samples B. About 300 grams of the seed were com-

The per cent. of oil was obtained by the extraction method. The percentages of impurities refer, of course, to the amounts present in the seed as received. In the case of the Calcutta seed, the percentage of total impurities is a trifle high, as the average of 11 recent samples was 6.90 per cent.—containing 15.1 per cent. oil. The oil in the clean Calcutta seed averaged 41.01 per cent.

Andes<sup>1</sup> is evidently in error on the average weight of a seed. Wijs' figures<sup>2</sup> on the amount of impurities present in various seeds are correct in some cases. It is a remarkable fact that the percentages of impurities in the Calcutta and Russian seeds do not confirm the statements of most writers on the subject that "Calcutta and Baltic seeds are freer from foreign seeds than the American and La Plata seeds, and that, consequently, the oils are of better quality." Apparently, this prejudice regarding the presence of

<sup>1</sup> "Drying Oils," page 58.

<sup>2</sup> Andes, *Ibid.*, page 318.

foreign seeds in commercial American linseed is unfounded.

In preparing the oil, the seed was ground in a coffee-mill, placed in a Mason jar and steam introduced, the jar covered and placed in a steam oven for 2 hours. On removal, the meal was at once packed in cloth and placed in the press, the piston inserted and, using the screw of a filter-press, pressure was applied, the oil draining through the outlet tube into a bottle wired to the press. The first test-run showed that only slightly more oil was left in the cake than in regular factory practice.

Table II contains the analytical results obtained on the oils. In all cases, the oil was filtered through paper before making a determination. Sample 2A is an extraction process oil; all the other factory samples are oils manufactured by the old process hydraulic method.

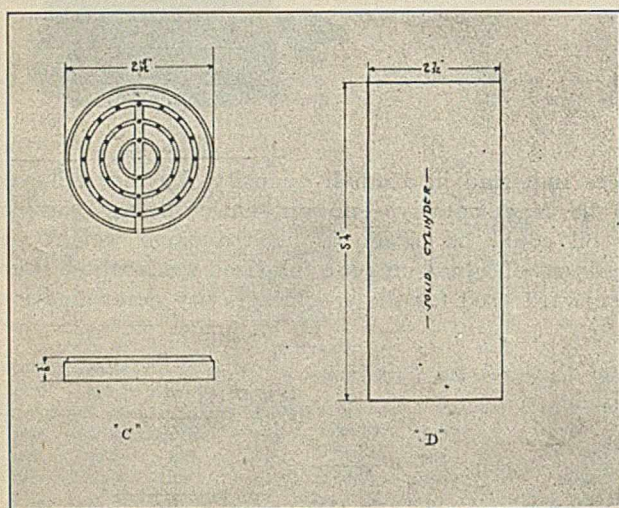


Fig. 3.

Analyses: 1A and 2A give the results obtained recently on the factory samples of oil, while 1A<sub>1</sub> and 2A<sub>1</sub> are the results obtained two years ago on the same samples, indicating that no change in the constants determined took place in the oils. Samples 1B, 1C and 2C were pressed in the laboratory from seed kept 2 1/2 years in closed Mason jars.

TABLE II.

No.	Oil.	Sp. gr.	Ref. index	Iodine	Color.
		15.5° C.		No.	
		15.5° C.	at 25° C.	(Hanus.)	
1	American—A	0.9346	1.4802	187.5	yellow
	American—A <sub>1</sub>	0.9347	1.4800	187.9	yellow
	American—B	0.9347	1.4800	187.6	yellow
	American—C	0.9346	1.4800	187.5	yellow
2	American—A	0.9330	1.4800	185.9	yellow
	American—A <sub>1</sub>	0.9331	1.4797	186.1	yellow
	American—C	0.9330	1.4800	186.5	yellow
3	La Plata—A	0.9316	1.4782	171.4	green
	La Plata—B	0.9316	1.4782	171.8	green
	La Plata—C	0.9317	1.4783	171.1	orange
4	Calcutta—A	0.9329	1.4790	178.9	yellow
	Calcutta—B	0.9327	1.4792	179.3	yellow
	Calcutta—C	0.9327	1.4793	180.5	yellow
5	Bombay—C	0.9316	1.4790	176.9	yellow
6	S. Russian (Kertch)—C	0.9317	1.4795	177.0	orange
7	N. Russian (Riga)—C	0.9354	1.4815	196.4	green

So far as the writer knows, no figures have been

published giving the analysis of oils from picked seed, with the exception of Thomson and Dunlop<sup>1</sup> who obtained 205.4 as the iodine no. on oil from picked Riga seed, but they used the Wijs method. Ketel and Antusch<sup>2</sup> give data of the effect on the oil of varying amounts of dotter and rape seeds added to clean linseed, but they used the extraction method for obtaining the oil; while, in a later article<sup>3</sup> they admit the possibility of a lower iodine no. in extracted oil, they state that to their knowledge this has never been actually tested. It may also be mentioned that the amounts of dotter and rape seeds added are considerably out of proportion to those actually present in the commercial seed examined.

Mastbaum states<sup>4</sup> that "expressed oil has a higher iodine no. than extracted oil." The comparative analysis of 2A and 2C would seem to indicate, however, that this is not necessarily so.

It is worthy of note that high percentages of non-oleaginous and low percentages of oleaginous impurities, as in the La Plata seed, do not affect the constants, but do affect the color of the oil. On the other hand, with the Calcutta seed, the variation in iodine no. can hardly be due to experimental error—more likely to the high per cent. of oleaginous impurities—whereas the color is only slightly affected. The green color of the Riga oil must be due to some coloring matter present in the seed itself.

The typical analyses quoted in Table II fall well within the limits set by Wijs when the usual difference between the Hanus iodine no. and the Wijs no. is considered.

From a consideration of the results it appears that 1. Oil pressed from clean linseed does not differ materially from commercially pure linseed oil.

2. The dark green color of the La Plata oil is due to the non-oleaginous impurities.

3. A high per cent. of oleaginous impurities does not affect the color appreciably, but does affect the iodine no. slightly.

4. The technical manufacture of oil by the extraction process does not lower the iodine no.

5. The constants of the oil pressed from the seed which had been kept 2 1/2 years in a closed container do not appear to be affected by the aging of the seed.

LABORATORY NATIONAL LEAD COMPANY,  
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## ARSENIC IN GLASS.

By S. R. SCHOLES.

Received November 13, 1911.

In Hovestadt's excellent work on "Jena Glass" arsenic oxide (As<sub>2</sub>O<sub>3</sub>) is listed among the constituents of many of the glasses described, in quantities of from 0.2 to 0.4 per cent. The English edition of this book contains a note by Everett, the senior translator.

<sup>1</sup> *Analyst*, 1906, 282.

<sup>2</sup> *Loc. cit.*

<sup>3</sup> *Z. angew. Chem.*, 1897, 210.

<sup>4</sup> *Ibid.*, 1896, 719.

who takes exception to this view, arguing that the pentoxide would be reduced at the glass-making temperature. Rosenhain, author of "Glass Manufacture," supports the latter opinion, and adds that he believes very little arsenic remains in the glass at all. It seemed of interest, therefore, to study this question from the analytical standpoint, by examination of the glass itself. Some unexpected results were obtained.

Two varieties of glass were analyzed for arsenic: No. 1, a soda-baryta glass, of fairly high alkali-content; No. 2, a potash-lead glass, used for cut-glass ware. In No. 1, the arsenious oxide put into the batch, or raw materials, was between 0.3 and 0.5 per cent. of the glass produced, while in No. 2 it was approximately 0.45 per cent. In both these batches, potassium nitrate was used in considerable quantity, and in No. 2 red lead was one of the main constituents, so that there was abundant opportunity for the initial oxidation of the arsenic to the pentavalent condition. The method used for determining the percentage and state of oxidation of the arsenic remaining in the glass was as follows:

Samples of glass, powdered to pass a 40-mesh sieve, were weighed into a platinum dish and treated with 15-20 cc. of hydrofluoric acid, added in three portions. When reaction had ceased, 20 cc. of 30 per cent. sulphuric acid were added, and the mixture evaporated on a water-bath until hydrofluoric acid fumes ceased coming off. It was then rinsed into a beaker, making a total volume of 40 cc., and arsenic acid determined by titration after the manner described in Sutton's "Volumetric Analysis." In the case of the lead glass, the solution was filtered before the addition of potassium iodide, to remove the lead sulphate and avoid formation of lead iodide. After titrating with thiosulphate, the solution was nearly neutralized with sodium carbonate, 20 cc. of a saturated solution of sodium bicarbonate were added, the arsenious acid was titrated with iodine, and the arsenious oxide originally present determined by difference. The standard solutions used were twentieth-normal. Blank determinations showed that the reagents contained no measurable quantity of arsenic. The data obtained are tabulated below.

Glass No.	Grams sample.	Cc. N/20			Per cent.		Per cent.	
		thio- sulphate.	Cc. N/20 iodine.	As <sub>2</sub> O <sub>3</sub> .	As <sub>2</sub> O <sub>5</sub> .	As <sub>2</sub> O <sub>3</sub> .	As <sub>2</sub> O <sub>5</sub> .	
1	6.60	9.0	9.1	0.02587	0.0003	0.39	0.00	
1	4.82	4.2	5.1	0.01207	0.0022	0.25	0.05	
2	4.25	5.9	6.6	0.01696	0.0017	0.40	0.04	
2	7.73	9.6	9.6	0.02760	0.0000	0.36	0.00	
2 <sup>1</sup>	6.00	8.6	8.6	0.02472	0.0000	0.41	0.00	

These results indicate that a great part of the arsenic used in glass-making remains in the glass as the higher oxide, probably as arsenate of soda or potash. This is somewhat surprising, in view of the properties attributed to arsenic in justification of its use. It is expected to volatilize, aiding in the mixing of the batch as the latter melts. Moreover, it is said to have an oxidizing effect, but the fact that the arsenious

Sample fused with sodium carbonate and dissolved in sulphuric acid.

oxide originally employed appears finally in the glass with a gain of oxygen does not bear out this belief. On the contrary, it seems that the use of arsenic must reduce the available oxygen of the batch. At least in cases where nitrates are present and the melting carried on in closed pots, as in the manufacture of the glasses here examined, the value of the arsenic, if any, obviously lies in neither its volatilizing nor its oxidizing powers.

The glass used and the data concerning it were furnished by one of the glass companies of this vicinity.

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PITTSBURGH.

### THE DETERMINATION OF CHROMIUM AND ITS SEPARATION FROM VANADIUM IN STEELS.

By J. R. CAIN.

Received November 10, 1911.

While attempting, recently, to determine chromium in chrome-vanadium steels, difficulties with some of the usual methods were encountered. If a steel containing chromium as chromate and vanadium as vanadate is titrated against ferrous solutions, using ferricyanide to indicate the point at which all the vanadium and chromium are reduced and an excess of titrating solution is present, there is sometimes an indefinite end-point, because as soon as some vanadium is reduced to the vanadyl condition this reacts with the ferricyanide indicator, reducing it to ferrocyanide which then gives the usual color with the ferric salts present.<sup>1</sup> An experienced operator can oftentimes judge the end-point sufficiently closely for practical work, but the difficulty increases with increasing vanadium, and it is almost always necessary to run blanks of various kinds, increasing to that extent the uncertainties of such methods. If the excess of ferrous solution is titrated back with permanganate some correction is also necessary for chromium oxidized by permanganate.<sup>2</sup> If in the preliminary oxidation of the vanadium and chromium, any manganese dioxide separates, as where potassium permanganate or potassium chlorate are used, some chromium may be, and often is, carried down by the manganese.<sup>3</sup> If, in order to separate vanadate from chromate, the nitric acid solution of the steel (with or without preliminary ether extraction of most of the iron) is poured into excess of sodium hydroxide solution and boiled, usually an appreciable amount of chromium goes into the filtrate with the vanadium. Many methods take no account of this chromium. The amount so lost increases with the manganese in the steel and the time of boiling, the manganese being converted in part to peroxide and this, in the strongly alkaline solution, oxidizing the chromium to chromate.<sup>4</sup>

Many careful tests having shown that chromium in

<sup>1</sup> Cain, *THIS JOURNAL*, 3, 476 (1911); other references will be found here.

<sup>2</sup> Cain, *loc. cit.*

<sup>3</sup> Arnold and Ibbotson, "Steel Works Analysis," 3rd Ed., p. 180; also Cain, *loc. cit.*

<sup>4</sup> Cain, *loc. cit.*



much larger amount than the usual commercial steels carry can be precipitated completely in a few minutes by boiling the nearly neutralized (ferrous) solution of the steel with barium carbonate, cadmium carbonate, zinc oxide or magnesium oxide, it was decided to base a method for determining chromium on one of these methods of separation from iron. Probably the reason why such separations have not come into more general use is because practically all writers have directed to make the carbonate or oxide precipitation in the cold, which requires many hours' standing and is even then sometimes incomplete. The work done here has shown that but ten or fifteen minutes' boiling, with proper precautions, is all that is ever necessary, the filtrates being free from even traces of chromium (or vanadium).

Noyes and Bray<sup>1</sup> have given conditions for completely precipitating chromates in the presence of vanadates which make possible a good separation of the two elements. The work here having completely confirmed their results, this separation was incorporated in the method.

#### DESCRIPTION OF THE METHOD.

Dissolve in a covered 300 cc. Erlenmeyer flask an amount of drillings, which will give not to exceed 6 or 7 centigrams of chromium, using about 10 cc. of concentrated hydrochloric acid per gram of steel. The concentrated acid (specific gravity 1.20) seems to dissolve the steel much more readily than a diluted acid. When no more hydrogen is given off, dilute to 100 or 150 cc. with hot water, nearly neutralize with saturated sodium carbonate solution, add barium carbonate emulsion in slight excess, place on the hot plate and boil vigorously ten or fifteen minutes, with small additions of the barium carbonate emulsion every two or three minutes. The flask should be kept covered during all operations so as to exclude air as completely as possible. Too great an excess of barium carbonate should not be used, as this increases the difficulty of extracting the chromium in the subsequent fusion. An excess of a gram or two is the most that should be present. Remove the flask from the plate, let the precipitate settle, and filter at once on a 11 cm. white label No. 589 filter, washing twice with hot water. These operations should be carried out rapidly and without delay. Place the filter and precipitate in a sufficiently large platinum crucible, burn off the filter, add 2 grams of sodium carbonate and about  $\frac{1}{4}$  gram potassium nitrate, and fuse for 20 minutes. Support the inverted and inclined crucible on a glass triangle which has glass legs about  $\frac{3}{4}$  inch long fused to its corners for supports, place on the bottom of a 250 cc. beaker, cover it with boiling water and digest a few minutes until the fusion is disintegrated. Filter into a flask, add 1 or 2 cubic centimeters of hydrogen peroxide to the filtrate and boil for five or ten minutes to destroy the excess of peroxide. Cool, transfer to a 250 or 300 cc. separatory funnel, add a slight excess of nitric acid (1-1), and shake vigorously a few minutes, allowing the liberated

carbon dioxide to escape by inverting the separatory funnel and opening the stopcock. Transfer to a 250 cc. beaker, just neutralize with sodium hydroxide solution, and then add nitric acid (1-1), 2 cc. for each 100 cc. of solution. Add 20 cc. of a 20 per cent. lead nitrate solution to the cold solution, stirring vigorously. The precipitate settles quickly. It is filtered on asbestos, and washed three or four times with cold water. The asbestos mat is transferred to a beaker or flask, and the lead chromate decomposed with hot hydrochloric acid (1-4). The solution is cooled, the volume made up to 150 or 200 cc. and it is titrated against approximately N/10 ferrous sulphate solution with ferricyanide as outside indicator. Or, if desired, an excess of the ferrous sulphate solution is added and the excess titrated back against bichromate. The standard iron solution should be compared with bichromate or permanganate on the day it is used. Table I gives results obtained with synthetic solutions and with the chrome-vanadium standard steel of the Bureau of Standards. The synthetic solutions were made by adding the chromium from a carefully standardized bichromate solution and the vanadium from a sodium vanadate solution to the hydrochloric acid solution of a vanadium and chromium free steel.

TABLE I.

No. of expt.	Iron present. Grams.	Vanadium present.	Chromium present.	Chromium found.	Difference.
1	1	0.0012	0.0140	0.0137	-0.0003
2	1	0.0024	0.0280	0.0277	-0.0003
3	1	0.0024	0.0420	0.0420	0.0000
4	1	0.0036	0.0560	0.0558	-0.0002
5	1	0.0048	0.0700	0.0700	0.0000
6	1	0.0060	0.1120	0.1122	+0.0002
7	1	0.0120	0.1680	0.1685	+0.0005
8	1	0.0180	0.2800	0.2805	+0.0005
9 <sup>1</sup>	4	..	..	0.0536	..

#### NOTES AND PRECAUTIONS.

Numbers 6, 7 and 8 of Table I show that very large amounts of chromium can be completely precipitated by the barium carbonate method carried out as above described. However, if the amount of chromium exceeds that recommended in the method described herein, it cannot be readily extracted by 2 grams of sodium carbonate, particularly if too great an excess of barium is present. The amount of sodium carbonate used was governed by the directions of Noyes and Bray<sup>2</sup> who state that, for a successful separation of chromium, not too much sodium nitrate should be present. The separations described herein were made in a volume of 250 cc., and inasmuch as 2 grams of sodium carbonate were used for fusions, the resulting sodium nitrate concentration was about 1.3 grams per 100 cc. The filtrates were tested for chromium by removing the excess of lead with sulphuric acid, filtering, concentrating to about 10 or 15 cc. and making alkaline with sodium hydroxide. No color indicative of chromate was ever obtained, even after adding a little hydrogen peroxide and boiling to

<sup>1</sup> Chrome-vanadium standard: 4 determinations; each gave 1.34 per cent. Average of results of cooperating analysts: per cent. chromium 1.36, per cent. vanadium 0.204.

<sup>2</sup> *Loc. cit.*, p. 48, Note 4.

oxidize any chromium that may have been reduced. Possibly the larger amounts of chromium could have been completely extracted by one fusion, had more sodium carbonate been used, but the manipulation is then less convenient; moreover, with the usual range of chromium content in commercial steels one need never work with more than 7 or 8 centigrams to secure an accurate determination.

The boiling with hydrogen peroxide in alkaline solutions before precipitation with lead nitrate is done in order to destroy any nitrite formed during the fusion. If this were present when the solution is acidified, the resulting nitrous acid would possibly reduce some chromium or vanadium. Five minutes' boiling will insure destruction of the excess of peroxide if only 2 or 3 cc. of the usual 3 per cent. solution are used. The hydrogen peroxide should be completely removed before acidifying, inasmuch as it reduces chromic acid.<sup>1</sup>

The freeing of the solution from carbon dioxide is an important step, for if this is not done, the precipitate does not settle out rapidly and is not so easy to filter; shaking in a separatory funnel, as described, is a convenient way of accomplishing this.

The solutions containing the chromium, after titration, were treated with enough sulphuric acid to precipitate the lead, which was filtered off and washed with dilute sulphuric acid. The filtrates were evaporated on the hot plate until free from hydrochloric acid and then electrolyzed with a mercury cathode<sup>2</sup> until free from iron and chromium. The solution in the electrolyzing apparatus was then tested for vanadium by hydrogen peroxide, none being found in most cases and in others only traces, showing that the separation under the conditions given is practically perfect. It is well to examine the insoluble from the fusion for chromium by again fusing it with sodium carbonate and potassium nitrate. The solution from the second fusion is almost invariably colorless when working under the conditions herein recommended. Should there be a slight yellow color, however, the chromium causing it can be estimated colorimetrically. A determination of chromium can be made easily in 1½ hours.

#### CONCLUSIONS.

1. Sources of error in some of the usual methods for determining chromium in chrome or chrome-vanadium steels, which limit the accuracy of the results, are described.

2. The precipitation of chromium from solutions of steels and its separation from practically all the iron can be effected quickly and easily by boiling with a number of precipitants, herein described.

3. The chromium may be readily extracted from the precipitates by fusion, and separated from vanadium by precipitating as lead chromate, under the conditions prescribed.

BUREAU OF STANDARDS,  
WASHINGTON.

<sup>1</sup> Perchromic acid is first formed, but is rapidly decomposed.

<sup>2</sup> Cain, *loc. cit.*

#### THE BISMUTHATE METHOD FOR MANGANESE.

By D. J. DEMOREST.

Received October 20, 1911.

There seems to be considerable misapprehension as to the effect which chromium has upon the bismuthate method for manganese. Thus it has been stated that chromium affects the results for manganese by this method. If, however, the method is properly carried on, the chromium has no influence. It is true that the bismuthate oxidizes some of the chromium to chromic acid, and this is titrated along with the manganese if the manganese is titrated by adding an excess of ferrous sulphate and then titrating the excess with permanganate. This is not the proper way. If the permanganic acid is titrated directly with sodium arsenite until the pink color disappears, the chromic or vanadic acids which may be present do not interfere at all and the results are accurate.

To show this, the vanadium steel standard issued by the Bureau of Standards was analyzed for manganese with and without the addition of chromium. The Bureau gives 0.669 per cent. as the manganese in the steel. The following results were obtained.

Without adding chromium.	With 3 per cent. chromium added as K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> .
0.666	0.669
0.666	0.666
0.666	0.671
Another steel was run in the same way obtaining:	
0.468	0.468

The acid open-hearth steel standard issued by the Bureau of Standards, and for which the Bureau gives 0.407 as their average, and the average by the co-operative chemists as 0.412 was analyzed with and without 3 per cent. Cr. The results were 0.403 per cent. Mn without, and 0.405 per cent. Cr with 3 per cent. chromium. Another sample gave 0.489 per cent. without addition of Cr or V and 0.488 per cent. with the addition of 3 per cent. Cr and 1½ per cent. V.

The method as used in this laboratory is as follows: One gram sample is dissolved in 45 cc. of water and 15 cc. HNO<sub>3</sub> (sp. gr. 1.42) and the solution boiled until nitrous fumes are gone. After cooling a little some "bismuthate" is added, a little at a time, until the resulting permanganic acid or manganese dioxide persists after a few minutes' boiling. Now KNO<sub>3</sub> is added to dissolve the MnO<sub>2</sub>, and the solution is boiled a few minutes to expel nitrous fumes. It is then cooled to tap water temperature. When cold, bismuthate is added a little at a time, while the solution is shaken, until about 1/2 gram has been added. After settling a moment the solution is filtered through asbestos on glass wool (for speed) and the asbestos washed well. Then sodium arsenite is run in from a burette until the pink tinge just disappears. There should not be a brownish color at the end. If there is, it indicates insufficient acid.

The arsenite is made by adding to 2¼ g. As<sub>2</sub>O<sub>3</sub> in a beaker a hot solution of Na<sub>2</sub>CO<sub>3</sub> until the As<sub>2</sub>O<sub>3</sub> dissolves. It is then diluted to 2½ liters.

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## SALT-RISING BREAD AND SOME COMPARISONS WITH BREAD MADE WITH YEAST.<sup>1</sup>

By HENRY A. KOHMAN.

Received October 12, 1911.

Bread is made light and porous by two methods of aeration. The oldest of these and the one, which is most extensively used at present, involves a process of fermentation, in which various microorganisms produce the gas necessary to raise the bread; by the other method it is given a porous character, either by forcing carbon dioxide under pressure into the dough while it is being mixed, as is done in making aerated bread, or by mixing with the other ingredients certain chemicals, which, when they come in contact with the water used in making the dough, evolve gas, either immediately upon mixing the bread or later when it becomes hot during the process of baking.

The method of preparing bread with the aid of yeast has been extensively investigated and the function of this microorganism in bread is now thoroughly understood. By means of the saccharifying enzyme, diastase, which is present in flour, a part of the starch is converted into sugar, which as well as that normally present in flours and that added in making the dough either as sugar in some form or other, or malt extract, is partly converted by the yeast into alcohol and carbon dioxide which aerates the bread.

Another type of bread, involving a process of fermentation, the so-called salt-rising bread, is made by many housewives and bakers, particularly in the south and is not so thoroughly understood and offers far greater difficulties in its preparation. In its preparation there is added neither yeast nor a portion of fermented dough from the previous baking, as is done in making certain kinds of rye bread and a type of white bread as well. In fact there is nothing added that can be called leaven, and each day's baking is dependent upon a new and independent spontaneous fermentation. The method that is commonly employed is as follows: Cornmeal, salt and soda are thoroughly mixed and stirred into enough hot milk or water, often boiling, to make a batter of the consistency of corn meal mush. This batter or "emptyings," as it is commonly called, is kept in a warm place 15 to 20 hours or until it becomes light and shows the evolution of gas, and is then mixed with flour and water to make a slag sponge. The sponge is allowed to come up well which may take from one to three hours, and is then mixed with the remainder of the ingredients to make a dough of the usual stiffness. The dough is allowed to stand not longer than an hour but is usually moulded into loaves immediately upon mixing. After it has risen to the degree of lightness desired, it is baked in the usual way.

The literature on the subject indicates that the theory of the leaven in this bread is very incompletely worked out and that there are conflicting views both in regard to the nature of the organism or organisms involved and to its source.

<sup>1</sup> This work was done under the Fellowship established in the University of Kansas, by the National Association of Master Bakers, and the results have in part been made public in papers read in conventions, and through a series of articles in *The Bakers' Review*.

In reference to salt-rising bread Margaret Mitchell<sup>2</sup> says: "When a brew is prepared, but no stack yeast or raw dough is added, it will still be found that in time the mixture will ferment if kept warm. This spontaneous fermentation is due to the fact that yeast spores, when dried, are very light, and are blown about so that they are present almost everywhere. These floating spores may be those of the household yeast, or those of 'wild' yeasts which are common, for instance, in drying fruit, etc., but which are not often cultivated. The spores can enter the brew from the air or from utensils used in mixing. When accidentally planted they grow, as any yeast would, and produce fermentation. Along with the 'wild' yeast obtained in this method of making bread, there are usually also obtained a large number of bacteria, which form bodies of characteristic odor and flavor in the course of their fermentation; and to this is due the peculiar odor and flavor of the salt-rising bread."

She says further: "The uncertainty of this method of making bread is one of its disadvantages, but when it is made, often in the same place the 'wild' yeast most successful in raising this kind of bread is apt to be more abundant in the air, utensils, etc., than other 'wild' yeasts."

Professor F. C. Harrison<sup>3</sup> says the following: "There is another method of making risen bread, that is the method called 'salt-rising.' It is the result of a spontaneous fermentation, and is therefore a matter of chance whether good bread will be produced, although in places where such bread has been made for some time there is less likelihood of failure, as the utensils and air of the rooms in which the bread is made contain large numbers of the desirable germs." Dr. E. H. S. Bailey<sup>3</sup> describes the process as follows: "The salt-rising process depends on preparing a favorable medium in which the yeast germs will grow, and then allowing them to get into the dough from the air, or from the ingredients used in making the sponge. The bread is started by the use of flour, or cornmeal, warm milk, and salt. The meal begins to ferment after a short time, if kept in a warm place, but the fermented material will not have the same taste and odor as the sponge from yeast, as various 'wild' yeasts are sure to be present. It is probable that lactic and butyric fermentation also take place to some extent. Although salt, in any quantities above 1.4 per cent., retards alcoholic fermentation, yet as it even to a greater extent retards the growth of foreign ferments, such as lactic and certain 'wild' ferments, it is probable that its addition is an advantage, on the whole, if this method of fermentation is used. Salt-rising bread is finer grained than yeast bread and has a peculiar and characteristic odor, which is due, no doubt, to the lactic fermentation which has taken place."

Helen W. Atwater<sup>4</sup> describes it thus: "The so-called 'salt-rising' bread is interesting as an illustration of self-raised bread. In it the ferments originally

<sup>2</sup> U. S. Department Agricultural Bulletin, 200, p. 45.

<sup>3</sup> Ont. Agricultural Bulletin, 118, p. 16.

<sup>3</sup> "Sanitary and Applied Chemistry," p. 168.

<sup>4</sup> U. S. Dept. Agr., Farmers' Bulletin, 389, p. 21.



present or acquired from the air produce the fermentation which leavens it. To make it warm milk and cornmeal are mixed together into a stiff batter which is left at blood-heat until the whole mass is sour—that is, until the ferments present have produced fermentation throughout. Next a thick sponge is made of wheat flour and hot water in which a little salt has been dissolved. This sponge and the sour batter are thoroughly kneaded together and set in a warm place for several hours. The leavening action started in the batter spreads through the dough and produces a light, porous loaf, which many persons consider very palatable. Such a bread is comparatively free from acidity, as the presence of the salt hinders undesirable acid fermentation.”

P. G. Heinemann and Mary Hefferan,<sup>1</sup> in an investigation of salt-rising bread, isolated a bacillus which agrees in morphology, staining properties and other cultural characteristics with *B. bulgaricus*. In a stain from the mixture of cornmeal, milk, salt and soda the bacilli present showed a granular appearance, but after growing in milk they stained uniformly. These bacilli, the authors failed utterly to isolate by plating on ordinary laboratory media, but they were readily isolated if cultivated in milk at 37° C. or if plated with milk-agar. In milk the pure culture forms a soft creamy coagulum which does not separate from the whey. After 14 days the milk showed an acidity of 1.65 per cent. and the authors think that the milk is a necessary part as without it the bacillus is unable to produce the acid necessary to liberate the carbon dioxide from the added soda. By inoculating sterile milk with cornmeal it was demonstrated that the origin of the organism was the cornmeal and not the milk.

Erbert J. Clapp,<sup>2</sup> in a paper on salt-rising bread, tells of the extreme difficulties encountered in making it and enumerates the conditions and ingredients that he finds important. It is his experience and that of bakers as well that success depends to a large extent upon the kind of cornmeal used. Kiln-dried meal is found to give poor results. Best of all he finds coarse meal, “hulls, shucks and all,” as he describes it. The hulls and shucks are strained out, however, in making the sponge. He enumerates potatoes which are not generally used as one of the essential ingredients, but says that river bottom potatoes which are high in moisture and low in starch do not give satisfactory results.

Mr. Clapp's formula differs also from the one ordinarily given in that it includes ginger as one of the essential ingredients; milk, on the other hand, is not used at all and he says its use is to be avoided entirely.

He emphasizes the fact that it is important to scald the vessels used in making the bread and to guard against souring. The evolving gas he observed would burn (explode) when a lighted match is applied which he attributes to the generation of alcohol.

It is evident that while there are conflicting views in the above references, there are also some points of agreement. All of the writers agree that the methods

of making this type of bread are exceedingly uncertain; it is a prevalent opinion, too, that success is dependent very largely upon preparing a favorable medium and allowing the desirable organisms to get into it either from the air or utensils. Dr. Bailey and Margaret Mitchell state definitely that the leavening power is due to the presence of “wild” yeasts and that various bacteria contribute more or less to the odor and flavor of the bread; the bacterial fermentation, in the opinion of Dr. Bailey, is due to lactic and butyric ferments. Professor Harrison and Helen Atwater do not specify what the germs are that cause the spontaneous fermentation.

P. G. Heinemann and Mary Hefferan observed a bacillus which they were able to isolate but they do not say whether yeast was present or not. This bacillus, they maintain, is enabled by the use of milk to produce the acid necessary to liberate the carbon dioxide from the soda.

#### “SAUERTEIG” METHOD.

There is another type of bread made by the use of the “Sauerteig,” which, while it differs from salt-rising bread in many respects, has sufficient bearing upon the subject, as will be seen later, to warrant a brief review of some of the literature on the subject.

Long before the existence of microorganisms was discovered, it was known that when meal or flour and water were made into a paste it would, after a time, begin to ferment and evolve gas. This was early made use of in the preparation of bread and it was soon learned that a portion of the dough could be saved to start the fermentation in the next baking. This portion of dough would continue to ferment and become sour, hence its name “Sauerteig,” but when mixed with fresh flour and water it would again become active and raise the bread. This method of making bread was, and in some countries is still, used very extensively, particularly in making whole meal bread (“Schwarzbrot”) and rye bread. It is similar to the salt-rising method in that the fermentation in both is spontaneous; they differ, however, in that the former is started with hot water or milk, usually boiling, while the latter is made with tepid water. They differ also in that salt-rising bread is made from fresh meal each time while by the “Sauerteig” method a portion of fermented dough is saved for the next baking, and when a housewife or baker is out it is usually obtained from a neighbor. This method of making bread, while it is crude and uncertain compared with the methods of to-day which involve the use of compressed yeast, is, as would be expected, more certain than the salt-rising method because each time a portion of dough is saved for the next baking which insures the presence of the essential organisms although they may be badly contaminated with others.

A microscopic examination of a “Sauerteig” reveals both yeast cells and various bacteria in great numbers; at times the former and at times the latter exceed in numbers, and it has been the subject of many investigations to determine the rôle of each in the preparation of bread by this method.

<sup>1</sup> Science, June 25, 1909, 29, No. 756, p. 1011.

<sup>2</sup> Bakers' Helper, Nov., 1908, 22, 1164.

The earliest view was that this bread was leavened by means of an alcoholic fermentation due to the presence of yeast.<sup>1</sup> This view was supported by various analyses that showed the presence of both sugars and saccharifying enzymes in flour, and later the alcohol in the fermented dough. Further, microscopic examination revealed the presence of yeast which was shown to have the cultural characteristics of *Saccharomyces minor* Engel.

In the year 1883 Chicandard<sup>2</sup> advanced a new theory, according to which the fermentation in this bread is due to a bacterium, which he claims produces gaseous products from the albuminous substances in flour.

In support of this theory he says that diastase does not saccharify starch at ordinary temperatures and that there is no decrease in the sugar content of dough during fermentation. As a matter of fact, however, sugar is a normal constituent of flours, and diastase has been shown to be active even at the temperatures at which doughs are ordinarily fermented.

Further, he maintains that there is no alcohol in dough which also has been proved to be contrary to fact. As a further support of his theory he gives an analysis of the evolving gases which consist of about 70 per cent. of carbon dioxide and the remainder of hydrogen and nitrogen. These gases are similar to those evolved from decaying albumin, which he gives as additional evidence in favor of his theory. Peters<sup>3</sup> thinks, however, that the carbon dioxide may have been produced by yeast, that the nitrogen may have come from enclosed air and the hydrogen may have been produced by a butyric ferment.

Laurent<sup>4</sup> found that an organism which he called *Bacillus panificans* is normally present in flours. This organism is killed only after heating to 100° C. for 10 minutes and is probably not killed in the baking of the bread. When growing in dough, it produces acetic, lactic and butyric acid, together with CO<sub>2</sub> but no hydrogen nor nitrogen. According to Laurent it is the cause of ropiness in bread, and in the opinion of Maurizio is identical with *Bacillus mesentericus vulgatus*.

An investigation by Dünneberger<sup>5</sup> leads him to believe that the essential organism in the preparation of bread by this method is yeast and that the presence of bacteria is, at least, unnecessary if not even harmful.

In three papers on "Die Organismen des Sauerteigs und ihre Bedeutung für die Brotgärung," Peters<sup>6</sup> reviews the literature on the subject and gives the results of his own exhaustive investigation. He finds numerous yeast cells and many bacteria as well, in a normal "Sauerteig." He succeeds in isolating *Saccharomyces minor*, which has been obtained by previous investigators, another yeast which resembles this species closely, *Mycoderma vini* (*Sacchar. my-*

*coderma*) and *Saccharomyces cerevisiae*. In addition to these four species of yeast he isolated five different bacteria which he describes minutely. He concludes from the results of his investigation that the fermentation of bread by means of the "Sauerteig" is complex in its nature and that no single organism is responsible for all the changes that take place. The *Saccharomyces* are important in that they produce the alcoholic fermentation which aerates the bread and the bacteria render soluble a portion of the constituents of the flour and produce a certain percentage of acidity which checks various other bacteria that cause diseases in bread.

According to Lehmann's<sup>7</sup> investigation there is present in the "Sauerteig" together with numerous yeast cells a gas-forming bacterium. Upon a gelatin plate made from the dough numerous yeast colonies develop, which upon further examination agree in morphology and cultural characteristics with *Saccharomyces minor* Engel. Among the yeast colonies there usually appear comparatively few colonies of bacteria. If, however, an agar plate is made and incubated at about 37° C. the yeast colonies do not appear, but numerous colonies of bacteria develop, among which one form greatly predominates, others appearing far less plentifully and regularly. This predominating organism which he calls *Bacillus levans* grows on gelatine plates in white water colonies which are spherical in shape with a rather darker zone in the center. It is facultative anaërobe and grows in an atmosphere of CO<sub>2</sub>. It is a gas former and now and then bubbles will appear even in sugar-free bouillon gelatine media. In the presence of sugar it produces gas in gelatine and agar media both on plates and in stab cultures. In bouillon media it produces cloudiness while if sugar is added gas is evolved. The organism is motile; does not form spores.

The gases that evolve from sugar bouillon, outside of a little nitrogen, consist of approximately 1/3 hydrogen and 2/3 carbon dioxide. Hydrocarbons were not detected. In sugar-free bouillon only very small quantities of hydrogen were produced and no carbon dioxide. A more complete analysis of the gases produced by this organism is given by Maurizio.<sup>8</sup> It was grown in sugar-bouillon and the results of the gas analysis follow:

	Beerwort.	Diluted beerwort.	Sugar-free bouillon.
	1.	2.	3.
Carbon dioxide.....	68.9	66.8	63.7
Hydrogen.....	25.4	28.7	31.8
Nitrogen.....	5.7	4.5	5.5

The variation indicated by the figures above he attributes to the fact that carbon dioxide is absorbed to some extent by the medium.

When grown in different media the gases produced by this bacillus show considerable variation.

	Beerwort.	Diluted beerwort.	Sugar-free bouillon.
	4.	5.	6.
Carbon dioxide.....	68.4	63.8	0
Hydrogen.....	22.1	28.7	67.1
Nitrogen.....	9.2	7.5	32.9

*Centralblatt für Bacteriologie*, 1894, p. 350.

<sup>2</sup> "Getreide Mehl und Brot. Maurizio," p. 235.

<sup>1</sup> *Botanische Zeitung*, 47, 404 (1889).

<sup>2</sup> *Ibid.*, 47, 407 (1889).

<sup>3</sup> *Ibid.*, 47, 408 (1889).

<sup>4</sup> "Getreide Mehl und Brot. Maurizio," p. 238.

<sup>5</sup> *Botanische Zeitung*, 47, 410 (1889); (*Botanische Centralblatt*, 1889).

<sup>6</sup> *Ibid.*, 47, 405, 420 and 435 (1898).

There were obtained from 600 cc. 1 per cent. sugar-bouillon 300 cc. of gas, while in sugar-free bouillon only 30 cc. of gas were obtained from the same amount of media.

The acids that this organism normally produces in media containing sugar are designated as acetic, lactic and oxalic.

Lehmann succeeded in setting up a gaseous fermentation in flour, which had been sterilized by submerging in ether for several days and regained from the ether by evaporation, by subsequent moistening with sterile water and inoculating with this *Bacillus levans*. The evolving gases were the same as those from sugar-bouillon fermented by this bacillus, and the odor the same as that of the "Sauerteig." A similar portion of the sterile flour inoculated from the "Sauerteig" fermented in apparently the same way but an analysis of the gases showed, however, that hydrogen was invariably absent, which would indicate that this is due to the presence of yeast and that the gas in the "Sauerteig" is not produced by *Bacillus levans*.

A control in which the sterile flour was inoculated with both *Bacillus levans* and yeast also failed to yield hydrogen in the evolving gases, which would perhaps indicate that the bacillus is not active in the presence of the yeast. Also when both organisms were inoculated into the media the maximum evolution of gas was obtained much sooner and with a smaller degree of acidity than when the fermentation was due to *Bacillus levans* alone.

Outside of the rôle that *Bacillus levans* plays in bread-making, whether it be desirable or not, it is of interest from another point of view. It was observed by Wolffin and Lehmann that it strikingly resembles *Bacillus coli commune* especially in its morphology. Also it was possible to set up the same gaseous fermentation in a paste made from sterile flour and water with *Bacillus coli commune*, as occurs when the same material is inoculated with *Bacillus levans*. Both ferment maltose, grape sugar and lactose. *Bacillus coli* differs, however, in that it coagulates milk and also in the composition of the gases it produces.

When bouillon with 1 per cent. of grape sugar added was fermented with *Bacillus levans* and *Bacillus coli commune* the following gases were obtained:

	<i>Bacillus levans</i> .	<i>Bacillus coli commune</i> .
Carbon dioxide.....	66.5	22.3
Hydrogen.....	28.6	75.6
Nitrogen.....	4.9	2.1

The gases collected from a paste of sterile flour and water exhibit no marked variation from the above table as may be seen from the following:

	<i>Bacillus levans</i> .	<i>Bacillus coli commune</i> .
Carbon dioxide.....	66.5	23.3
Hydrogen.....	27.7	74.0
Nitrogen.....	5.8	2.7

Lehmann questions whether these differences in the composition of the gases are sufficient to distinguish this bacillus from the coli group, particularly

when the variability of these bacteria is taken into account. Members of this group have been known to lose their ability to coagulate milk, or to ferment sugar after having been grown on sugar-free media for some time. Maurizio also is of the opinion that *Bacillus levans* belongs to the coli group and he, too, concludes, as Peters, that it is the function of the yeast to produce the gas necessary to aerate the bread, and further that various lactic bacteria are desirable in that they check the action of objectional forms such as butyric bacteria and members of the coli group.

The disagreement in regard to the theories concerning the leavening agent in salt-rising bread and the necessary ingredients, the uncertainty of manufacture, and the lack of uniformity in the finished product lead the author to investigate the subject, in order to ascertain definitely, if possible, what the leavening agent really is: whether "wild" yeasts, as the majority of writers on the subject claim; or the interaction of lactic acid formed by bacterial fermentation, with soda as others claim; and then perhaps to obtain the desirable organism in pure culture and propagate it for the production of bread of uniform quality, thus substituting more scientific methods for making this type of bread.

#### OBSERVATIONS.

The first step in the investigation was to start the fermentation from cornmeal, as is regularly done, and then also to make the bread in order to be certain that the fermenting mass contained the essential organisms. The first experiences of the author in making this bread were in harmony with the references on the subject in regard to the uncertainty of the method, and even after the bread had been made successfully a number of times, failures were of frequent occurrence, although care was taken to control temperatures, proportions of ingredients, etc.

After having made the bread successfully a number of times, a microscopic examination of the "emptyings" prepared in the customary way was made. This was found to be literally teeming with bacteria while yeast cells were not to be found. Since this was contrary to what was found in the literature, the examination was repeated again and again; each time, however, bacteria were present in great numbers while yeast cells were invariably absent. This indicates that yeasts play no part in this bread, and the question arises as to how these bacteria function in the dough, whether by producing acid which liberates carbon dioxide from the soda or by decomposing some of the constituents of the flour into gaseous products. It was observed that if a portion of these "emptyings" was transferred to sterile milk by means of the platinum loop, gas began to be evolved after 8 or 10 hours, and that a curd which was broken and full of holes, due to gas bubbles, would form. It was possible to transfer these bacteria from tube to tube and each time the same gaseous fermentation would be set up, which would indicate that the evolving gas is produced by the bacteria from the milk itself, and it becomes of interest to determine the relationship of the bacteria present in the fermenting batter.

It is only in exceptional cases, if ever, that a sample of natural media contains but a single species of microorganism when in a state of fermentation. Often the activity of two or more species in the same media produce changes which neither could do of itself. When several species are simultaneously engaged in the consumption of the same medium, their association is termed symbiosis; as an example of this we may refer to the kefir grains which contain at least one species of yeast and two species of lactic bacteria, the simultaneous growth of which produces from milk the beverage known as kefir. Another common association of microorganisms is styled metabiosis, in which one species by its activities renders the medium suitable for the growth and development of another. A good example of this is the formation of vinegar from cider, due to the presence of yeasts which form alcohol from the sugar, and acetic bacteria which, after the alcoholic fermentation has ceased, transform the alcohol into acetic acid. The "Sauerteig" is perhaps an example of both symbiosis and metabiosis, inasmuch as there is the simultaneous consumption of sugar by yeasts and lactic bacteria, which relationship is that of the former, and later the conversion of part of the alcohol formed by the yeast into acetic acid, which, as it occurs in the manufacture of vinegar, has already been referred to as an example of metabiosis.

The microscopic examinations having shown that active microorganisms in salt-rising bread are bacteria, it becomes a question whether or not a pure culture can be obtained from the "emptyings" with which alone the bread can be made, or whether the necessary gaseous products of fermentation owe their origin to the combined activity of several species growing side by side.

After transferring the bacteria from one milk tube to another for several times and incubating at 40° C., it was observed that there was exhibited no great variety of forms, indeed the cells were strikingly alike except for slight differences in the length of rods.

It was therefore assumed that the fermentation is due to a single species and plates were made to obtain it in pure culture. The first set of plates was made with an agar-bouillon media and incubated at 40° C. After about 15 hours a good growth had appeared and a number of milk tubes were inoculated from those colonies which seemed to show differences and incubate at 40° C. The next day all the tubes showed a growth, for a soft curd had formed in the milk, but none of them showed any signs of gas production while the one inoculated from the tube from which the plating was made was giving off gas. Since the first plating resulted in a failure, it was repeated. Again numerous colonies appeared in the usual time and this time a larger number of tubes were inoculated from them and incubated at 40° C. Again a curd formed in each of the tubes but there were no signs of gas. After making numerous plates with this agar bouillon medium and isolating many cultures without obtaining any that would produce gas it was

thought that perhaps the medium was not favorable for the gas forming bacteria and that it either did not appear upon the plates at all or became attenuated during the planting and lost its ability to produce gas.

Since soda is commonly used in making the bread it was thought that an alkaline medium might be more suitable. Therefore a portion of nutrient agar medium with 1.0 per cent. added lactose was titrated with phenolphthalein to an alkalinity of 1 per cent. and another to an acidity of 1 per cent. With each of these plates were made, both directly from the "emptyings," and a tube that was the result of several transfers, and numerous tubes inoculated and incubated at the usual temperature of 40° C., but again all efforts to obtain a gas-forming culture were in vain.

An effort was made to make a medium as similar as possible to the original "emptyings" by adding a portion of cooked flour to the agar medium. This medium, however, was so opaque that it could not be used successfully and was abandoned. As milk is commonly used in making this bread, plates were made with a milk-agar medium and a number of milk tubes inoculated and incubated. Upon observing them the next day one of them showed the characteristic gaseous fermentation. From this tube another set of plates was made and several tubes inoculated. These again were giving off gas after incubating over night. Since milk-agar plates are always rather opaque it was thought advisable to plate this culture upon clear media so as to be more certain that it was pure. This was done but not a tube produced any gas after incubating. This then was either not a pure culture or it lost its ability to produce gas during the plating on clear media. Further attempts to obtain another gas-forming culture by plating with milk-agar failed utterly.

It was observed that when the predominating culture from the fermenting batter was isolated and grown in milk that after a curd had formed the milk began to be peptonized near the surface and after standing a few days a large part of the curd was peptonized. The same effect was observed when milk-agar tubes were inoculated with this culture, only the peptonization proceeded downward in the tube much slower. In milk-agar tubes, inoculated direct from the fermenting batter, the peptonization of the milk was also observed near the surface while farther down gas was formed which at times pushed part of the media together with the cotton plug out of the tube. This latter effect was observed in broth-agar tubes also when the media was boiled to expel air. Plates made from the organisms after having been grown under these anaerobic conditions yielded no culture that would produce gas.

When milk-agar plates are poured in Petri dishes about  $\frac{3}{4}$  of an inch deep and 6 inches in diameter, the milk begins to peptonize from the top downward until after several days the medium loses its opaque character entirely and becomes yellowish in color. In these deep plates gas bubbles at times form near the bottom of the plates which may be due either

to a single organism or to several. To obtain cultures of these, the plate was inverted to allow the medium to drop out, when tubes could be inoculated by touching the gas bubble with a platinum loop. Milk tubes inoculated in this way showed the characteristic gaseous fermentation after incubating over night, but when plates were made from them and tubes inoculated from colonies, no gas appeared.

Single cells isolated from the fermenting batter by Dr. M. A. Barber's pipette method reacted in milk as those obtained by plating. A soft curd was first formed which later was peptonized from the top downward. When allowed to grow under a cover glass in an incubator at 40° C. a good growth was observed both in milk and broth. Spores formed readily. Single spores put into sterile milk and incubated produced the same reaction in milk as the cells themselves. No culture was obtained by this method that produced gas.

After all these failures to obtain a culture that was pure without a doubt and would retain its gas-producing power, it was thought that perhaps this gas formation was due to a mixture of cultures—perhaps a case of symbiosis or metabiosis. Upon this assumption plates were made and a number of tubes inoculated and incubated. After a good growth had set in, the tubes were numbered and fresh tubes inoculated from them by means of a platinum loop, mixing the various cultures. For example one tube was inoculated from Nos. 1 and 2, another from 1, 2 and 3, etc. The results of this mixing of cultures was as fruitless as all previous efforts, not a single tube showed any gas.

When sterile milk is inoculated from the fermenting batter, gas is driven off after incubating 6 to 10 hours and a curd is formed which is carried to the top by the gas bubbles and then assumes a tough leathery character. If after incubating for three or four days, sometimes less, a transfer to sterile milk is made, no gas appears. The same loss of gas production occurs in three or four bays if transfers are made at intervals of either 12 or 24 hours. A falling off of gas production is also observed in the fermenting batter; after gas bubbles begin to form the rate of gas production increases for about three or four hours and then gradually decreases. After fermenting for 8 to 10 hours, it becomes very much weaker and does not regain its strength if used in making the sponge and dough. In the latter the weakness is even more apparent than in the former, and when once the batter has fermented too long, the loaves are apt to be only about one-half the normal size. This gradual weakening of the batter has been observed by bakers, as well, and is popularly termed "working itself out." They also observed that when once it has become weakened and lost its gas-producing power that it will not regain its strength by the addition of fresh food material as is done in making the sponge and dough. While it was not possible to propagate the bacteria in liquid media from time to time to be used in making bread, a dried product

was prepared which could be used at will with good results.

The product was used by the author in making salt-rising bread in numerous experiments and was also given out to a number of housewives who used it successfully. That the artificial cultivation of bacteria can be of value to the manufacturing baker as well was demonstrated by giving this product a thorough trial in a modern baking plant where the bread was not uniform and it was necessary at times to add compressed yeast to insure proper aeration. From 800 to 1000 loaves were made daily for a month and the bread was uniform in quality and was ready for the bench at the desired time every day while formerly it was often ready sooner or later than desired and consequently upset the system of the plant. The batter made from the product begins to ferment not only more regularly but sooner than if made with meal. One pound is sufficient for the production of 400 to 500 loaves of bread.

The fact that the fermenting "emptyings" made from this product reveal cells which are strikingly alike, a large sporebearing motile rod greatly predominating, and that once a gas-forming organism was obtained by plating, and this one lost its ability to produce gas after incubating several days, indicates that the gas formation owes its origin to a single organism, which becomes attenuated during the plating. This is corroborated by the fact that a falling off of gas formation is observed both in the batter and when transfers are made to sterile milk, and is further supported by the fact that from either broth or milk-agar plates, in which the colonies were extremely thick, portions of media so large that all organisms should be included could be transferred to sterile milk and no gas would be evolved, which seems clearly to indicate attenuation.

Another instance in which the loss of gas production was observed may be referred to here. In a modern bakery bread is mixed with huge mixers in lots of about 800 to 1500 pounds of dough. A certain make of these mixers is run at very high speed making about 60 revolutions per minute, which is several times that of the average mixer. This extreme treatment is considered very desirable in making ordinary bread as it develops the elasticity of the gluten and improves the color of the bread. A batch of salt-rising bread was tried in one of these mixers, taking out a portion of dough after mixing 6, 15 and 30 minutes, respectively. The loaves made from the dough that was mixed 6 minutes rose very slowly, and were poor in quality, while the loaves from the portions of dough that were mixed 15 to 30 minutes, respectively, failed utterly to rise, even after standing several hours, while when mixed in a slow-speed mixer they rise in 30 to 45 minutes.

Such variation is not without parallel as may be seen from observations made by Klein,<sup>1</sup> on *Bacillus enteriditis sporogenes*. If deep liquid sugar-gelatine tubes (8 cm.) are inoculated with the bacilli or spores

<sup>1</sup> *Centralblatt für Bakteriologie*, 18, I Abteilung, No. 24, p. 737 (1895).

of this organism and incubated at 20° C., the medium is liquefied after 48 hours, with the formation of very few indeed, if any, gas bubbles and the organism sporulates freely. If, on the other hand, a stab culture is made from the same culture into a similar medium, gas formation begins within 24 hours and the medium is torn and rent by the evolving bubbles. In such a culture in which gas is abundantly given off, complete liquefaction takes 8, 14, or even 20 days, instead of 48 hours, and spore formation is not observed. The gas is principally methane.

A violent evolution of gas, after incubating from 1 to 2 days which breaks the layer of cream, accompanied with the formation of a curd which separates from the clear acid whey, is given by Klein<sup>1</sup> as the typical characteristic of this organism in milk.

If two gelatin tubes are inoculated with spores and the bacilli of the organisms, respectively, and incubated for several days, the results are so different that it is difficult to believe that the two cultures are identical, for the former shows the typical spherical liquefying colonies, while in the latter only small non-liquefying spots appear.

By transferring spores of the organism to fresh sugar-gelatine for a number of times, it undergoes marked changes in its cultural characteristics. It soon acquires greater liquefying powers, a tendency to form threads and the ability to rapidly form spores. In milk these variations are quite pronounced, for it no longer produces the changes which have been described as typical. Instead of the violent evolution of gas which disturbs the layer of cream, the formation of a curd and the separation of a clear whey, the milk undergoes an entirely different transformation, in which there is no gas whatever evolved and the cream is not disturbed. After several days the milk immediately underneath the cream begins to be decomposed into a yellowish solution which remains separate from the white coagulum underneath. The whey in the typical milk culture smells of butyric acid and reacts acid while the yellow solution of the atypical culture is weakly alkaline and has a foul odor. The bacilli in the typical milk culture are short rods without spores, while those of the atypical culture often appear in threads which as well as the short rods sporulate within a few days. When once the organism has changed so that spores when repeatedly transferred to milk continue to give the atypical culture characteristic, the change is permanent and it does not revert to the original form. The virulence in the atypical is almost or entirely lost.

On the suggestion of Professor Stephens some "emptyings" were obtained from a lady who makes salt-rising bread which he at one time found to be teeming with both yeast cells and bacteria. This was contrary to what had been observed, but as it agreed with a number of the references a microscopic examination was made, but again many bacteria were present and no yeast cells. From these "emptyings" a set of plates was made and a number of tubes inoculated. On removing them from the incubator

the next day several of them were evolving gas. One of these cultures was plated repeatedly on both milk and broth-agar media and each time all the colonies appeared alike and all the tubes inoculated were evolving gas after standing in the incubator for about 15 hours, which is sufficient evidence that it is a pure culture, and it becomes a question as to whether it is capable by itself to properly ferment bread. The mere fact that it will produce gas when growing in milk is not sufficient evidence that bread can be made with it, for often the "emptyings" will apparently be fermenting normally, but when made into a sponge by the addition of water and flour the production of gas almost ceases, and when the dough is made it fails utterly to rise. Indeed at times both the "emptyings" and sponge will appear normal and the dough will fail to rise, and it has happened in practice that a batch of about 1000 pounds of dough was run through the mixer, divider and moulder, and after being put into pans failed to rise and had to be taken from the pans and mixed with compressed yeast.

In order to determine whether this bacterium is of itself capable of aerating bread, it is of course desirable to use sterile flour; this unfortunately can be obtained only with difficulty and perhaps not at all without altering the character of the flour and destroying to a certain extent its bread-making qualities.

One of the two methods that have been employed is by heating the flour to a temperature of 115° to 120° C. This temperature is not sufficiently high to completely sterilize the flour for spore-forming organisms certainly survive, and besides it darkens the flour and, according to Peters, destroys its ability to form a dough. The other method of submerging the flour under ether for several days is more satisfactory, as it is less injurious to the flour, but it is a difficult matter to sterilize enough to be used in baking tests.

In these experiments it was thought best not to sterilize the flour but to grow the bacteria in sterile media up to the time the sponge was made, which would give the bacteria in the flour only from two to four hours to become active, for it was never more than four hours from the time the sponge was made until the bread was put into the oven and usually between two and three hours. When a dough was made without the addition of either bacteria or yeast, no marked changes were apparent within three or four hours, and if yeast was then worked into it, it would begin to rise and after fermenting properly, nearly normal bread could be made, which would indicate that the bacteria in flour do not have a marked effect upon a dough within three or four hours.

As already stated, milk is a common ingredient in salt-rising bread, and as it can be sterilized conveniently, it was used in the following baking experiments. With this pure culture bread was made from the following ingredients: 800 grams of flour, 100 cc. sterile milk, 410 cc. water, 12 grams salt and 15 grams sugar. The sterile milk was inoculated with the pure culture obtained and incubated 17 hours, when it was made into a sponge with 175 grams of flour and 200 cc. water. After the sponge

<sup>1</sup> *Centralblatt für Bakteriologie*, 22, I Abteilung, p. 577.

began to drop, which took about two hours, the dough was made by mixing the sponge with the remainder of the ingredients and moulded into loaves and put into pans at once. The bread rose well and was baked in the usual way and had the characteristic odor and taste of salt-rising bread. This experiment was repeated a number of times, which indicates that this bacterium is able by itself to properly aerate bread. Bread was also made by the "straight dough" process in which all the ingredients are mixed at once leaving out the sponge stage. In this experiment the whole of the liquid used was milk fermented by this bacterium, which was made into a dough by mixing with the other ingredients. The dough was at once moulded into loaves and put into pans. It rose rather slower, than when a sponge was made, and produced bread of poorer quality. "Straight dough" bread was also made by using as the liquid  $\frac{1}{2}$  fermented milk and  $\frac{1}{2}$  water; also by allowing the bread to rise, as is always done in making yeast bread, previous to moulding into loaves. This bread rose faster in the pans and the loaves had more of a tendency to crack. The bread was coarser in texture and when cut crumbled easily.

This bacterium could be propagated by transferring to fresh milk occasionally. After standing two months, however, it had become much weaker, and while it would still produce gas when grown in milk, it would no longer raise bread. Even by transferring to fresh milk a number of times it failed to regain its strength. After 200 cc. of milk fermented with this organism had been kept for 9 months in a German flask closed with a cotton plug, it was impossible to obtain any growth whatever by inoculating sterile milk from it.

An effort was made to prepare a dry product containing this organism to be used in making bread. This was done by growing the pure culture in sterile milk and then mixing flour to make a sponge. After the sponge had risen and fallen it was spread on panes of glass in thin layers and allowed to dry. Some of this dried product was mixed with meal and was used in starting the bread in the usual way. When the liquid used in starting the "emptyings" was heated to the boiling point the addition of some of this dried material seemed to make no appreciable difference either in the regularity or the time it would take to show the formation of gas bubbles, as was observed by setting at different times, a number of emptyings in Erlenmeyer flasks plugged with cotton, both with and without any added material. When the meal was stirred into the boiling milk a temperature of  $85^{\circ}$  to  $90^{\circ}$  C. was obtained. When the milk was heated to  $90^{\circ}$  C., it seemed rather doubtful whether the addition of some of this product was noticeable; if heated to  $80^{\circ}$  or  $70^{\circ}$ , however, it was observed that gas began to form both sooner and more regularly if a portion of the dried product was added, which would indicate the possibility of preserving this organism in the dry state for its economic use.

The source of the organisms involved, especially when the bread is made without adding them by arti-

ficial means, is one of the important considerations in making this kind of bread. It is the opinion of Dr. Bailey, Professor Harrison and Margaret Mitchell that they get into the prepared medium either from the air or utensils and that failures are far less frequent in places where the bread has been made for some time. To determine whether or not the desired bacteria get into the medium from the air, a number of Erlenmeyer flasks were partially filled with the usual ingredients in the proportions that they are used in the "emptyings." These flasks were plugged with cotton and sterilized by steaming for about 45 minutes on three or four consecutive days. After incubating for a few days to make certain that all life in the flasks was destroyed, the plugs were pulled and the flasks set in a gas oven which had been repeatedly used as a place to keep the "emptyings" and sponges as well while they were fermenting. If these bacteria are propagated from one baking to another by means of the air then this oven should prove a particularly fertile source, and as the media was properly prepared and the temperature carefully regulated, these flasks should soon show the characteristic fermentation. As a matter of fact, however, it was only occasionally that a gaseous fermentation would develop in these flasks and then with great irregularity; the gas bubbles would first appear near the top of the media and later they would form farther down and never did they occur uniformly throughout the media as is the case when the "emptyings" are set in the usual way. Often an abnormal fermentation would occur in these flasks; at times this would become manifest in a souring of the media without any gas formation, and at other times various moulds would appear on the surface of the media. This would indicate that the air is not the true source of the gas-forming organism, and although it may get into the media from the air at times it cannot be relied upon to produce the desired fermentation with any certainty or regularity.

In how much the utensils serve as a means of propagating the bacteria evidently depends to some extent upon the operator; if they are thoroughly cleaned each time after use, the chances are that the bacteria are nearly all removed with the ingredients; on the other hand if carelessly cleaned, it is possible that enough bacteria will be retained to again start the fermentation. At best, however, this cannot be a reliable source, and as the bacteria would be only on the sides of the vessel, some time would elapse before they would get into the center of a non-liquid medium. It is the experience of the author that it matters little, if any, whether the "emptyings" were made in vessels that had been previously used or not.

Evidently the milk is not the source of the organisms for the bread can be made with water, also it is possible to obtain the gaseous fermentation by inoculating sterile milk with cornmeal. The true source seems to be the cornmeal, and it is the experience of bakers and housewives that the more highly cleaned meals do not give as good results, which would indicate that it is associated in some way with the exterior of the grain of corn. It is the experience

of housewives, and canning factories as well, that it is a difficult matter to can corn so that it will keep; often the ends of the cans bulge out, owing to the formation of gas which may be due to the survival of the salt-rising organisms.

As already stated, it has been a matter of much dispute as to what ingredients are necessary in making salt-rising bread, and something over two hundred baking experiments were made to determine the necessary ingredients, the proportions in which they would be used, the most favorable temperature, etc. These baking experiments were taken up as systematically as possible, observing the effect of changing one thing at a time both upon the rate of the evolution of the gas and the character of the bread produced, and keeping a careful tabulated record of the results, some of which will be given here. One series of these experiments was made to determine the effect of milk, and the following table shows the proportions of the ingredients used, when they were mixed in "emptyings," sponge and dough, and the temperature of each (see Table I). The ingredients in Nos. 101, 102 and

upon the growth of the bacteria and consequently upon the character of the bread produced. Various baking experiments were made to determine to which of the constituents of the milk to attribute this.

It was found that whole milk did not give appreciably better results than skimmed, which would indicate that the butter fat, while certainly it has a "shortening" effect, as all fats do, has no bearing upon the gas production of the organism. The other two important constituents of milk, namely the casein and milk sugar, were used separately in making the bread. Four different samples of casein were obtained: two made from the buttermilk and two from skim milk.

It was found that when the "emptyings" were set as in No. 101 with 10 grams of casein instead of 10 grams of powdered skim-milk, gas began to form several hours sooner, especially when the buttermilk caseins were used, as may be seen from bakings 132 and 134. Even five grams of casein which is about the equivalent of casein in 10 grams of powdered skimmed milk was more effective than the latter

		1909.									1910.
Batter.	Date.....	11/3	11/3	11/3	11/14	11/14	11/14	11/14	11/10	11/10	5/9
	No.....	101	102	103	132	133	134	135	112	115	378
	H <sub>2</sub> O.....	150	150	150	150	150	150	150	150	150	150
	Dried milk.....	10	10	10	10	5A <sup>1</sup>	10A <sup>1</sup>	5B <sup>2</sup>	10M <sup>3</sup>	3C <sup>4</sup>	5
	Cornmeal.....	20	20	20	20	20	20	20	20	20	22
	Sugar.....	4	4	4	0	0	0	0	0	0	...
	Starter.....	2	2	2	2	2	2	2	2	2	2
	Soda.....	2½	2½	2½	2½	2½	2½	2½	2½	2½	2
Time.....	9.30	9.30	9.30	10.30	10.30	10.30	10.30	10.30	10.30	10.30	8.30
Sponge.	H <sub>2</sub> O.....	200	200	200	200	200	200	200	200	200	200
	Dried milk.....	0	10	0	0	0	0	0	0	0	0
	Flour.....	150	150	150	150	150	150	150	150	...	150
	NaHCO <sub>3</sub> .....	0	0	0	0	0	0	0	0	...	...
	Time.....	8.50	9.30	9.30	12.05	9.30	9.30	9.30	8.30	...	2.20
Temp.....	40	41	41	43	42	40	40	40	...	41	
Dough.	H <sub>2</sub> O.....	200	200	200	200	200	200	200	200	...	200
	Flour.....	650	650	650	650	650	650	650	650	...	650
	Sugar.....	0	0	0	0	0	0	0	0	...	0
	Salt.....	12	12	12	12	12	12	12	12	...	12
	Oil.....	0	0	0	0	0	0	0	12	...	0
	Time.....	10.00	11.00	11.40	1.05	10.50	10.45	10.35	9.55	...	3.30
	Temp.....	37	37	37	38	39	38	38	39	...	41
Oven.....	11.00	12.10	...	2.00	10.15	12.00	12.00	11.05	...	4.25	
Out.....	11.45	...	...	...	...	...	...	11.35	...	...	

<sup>1</sup> A in 133 and 134 is casein from skim-milk.

<sup>2</sup> B in 135 is casein from another sample of skim-milk casein.

<sup>3</sup> M in 112 is malt extract.

<sup>4</sup> C in 115 is asparagin.

Note: All batters, except in No. 378, were set in the evening at the time given and the sponges in the morning of the next day. No. 378 was finished in one day.

103 are precisely the same in quantity, differing only in that No. 101 had the milk added in the "emptying," No. 102 in the sponge and 103 in the dough. It is evident from the table that the emptyings in 101 were light and ready to be made into the sponge by 8:50, while 102 and 103 required 40 minutes longer; in the sponge it was observed that 101 was more active than 102 and 103 still more sluggish. In the dough the advantages of adding the milk in the early stages were still more apparent. The bread from 101 was largest in volume and best in every way, No. 102 was rather heavy and 103 failed to rise more than about one-half the usual height. From these experiments it is evident that milk has a marked influence

and almost as effective as 10 grams of the same material. In baking Nos. 133, 134, 135, all of which were made with casein, the gas began to be evolved about 2½ hours sooner than in No. 132 which was made with powdered skimmed milk. No. 134 showed signs of gas about ½ hour sooner than 133 and 135 but it was made into a sponge about the same time.

When lactose was used instead of milk in the above formula the gas began to form little sooner than if it was not used. Both lactose and casein used together were apparently no more effective than the same amount of casein alone. These experiments would indicate that the essential constituent of the milk is the casein rather than the milk sugar or butterfat.



The two samples of buttermilk casein, as stated above, favored the gas production more than those made from skim-milk and also more than the powdered milk itself. This may perhaps be partly explained by the fact that the casein from buttermilk undergoes a partial hydrolysis during the fermentative process of buttermaking, which may render it more assimilable by the bacteria. Also it was observed later that  $2\frac{1}{2}$  grams of soda in the above formula made the media rather alkaline and checked the fermentation somewhat, and as the caseins, especially those made from buttermilk, reacted acid, neutralized a part of this alkali and made the medium more favorable to the growth of the bacteria.

Malt extract which is used extensively in making ordinary bread also proved to be of value in making salt-rising. By the use of 10 grams of malt extract as in baking No. 112 it was possible to make good salt-rising bread. The value of malt extract is perhaps to be attributed both to its content of maltose and various soluble nitrogenous substances, and it is a question whether the enzymes have the same value that they do in making bread with yeast, especially those that have a proteolytic action, for the bacteria themselves have a marked action upon the gluten.

The addition of three grams of asparagin, as in No. 115, was detrimental and there was no gas production even after standing for several days. Ammonium nitrate was added to the formula in quantities varying from  $\frac{1}{2}$  to 5 grams, and it was found to retard the production of gas in all cases and when more than about 2 grams were used, gas failed to be produced at all. Potatoes and ginger, which are at times used, were tried but no beneficial effects were observed.

A series of "emptyings" were set in the same way using different sugars, *i. e.*, maltose, cane sugar, dextrose and lactose, and it was found that the gas began to evolve sooner than if no sugar was added to the meal. These sugars, as near as could be observed from the time bubbles began to form, were all fermented equally well and also as far as the production of bread is concerned.

These results were confirmed by adding 1 per cent. of cane sugar, maltose lactose, dextrose and dextrin respectively to separate portions of a nutrient agar medium and inoculating with a small loop full direct from the fermenting batter. The agar medium to which the carbohydrates, named above, were added contained 10 grams peptone and 5 grams beef extract to a liter of water. Seven hours after inoculating the tubes containing carbohydrates were all rent with gas bubbles and the medium in each tube was raised to about twice the initial volume. The tube containing no sugar showed only a few small bubbles which did not increase appreciably with longer incubation, while in a control no gas bubbles were observed. The dextrin and sugars were all fermented equally well.

The addition of milk accelerates both the production of gases and acids. It was repeatedly observed that those "emptyings" which were set with milk

or casein began to throw off gas sooner and more rapidly and, too, that the evolution of gas ceased sooner and the medium became sour. For example on one occasion the "emptyings" made without milk were allowed to stand at a temperature of  $40^{\circ}$  C. for 31 hours or 25 hours after gas bubbles appeared and it was still alkaline and gas was given off, while when milk is added it becomes sour in a very much shorter time and the evolution of gas ceases. This has been observed by bakers as well, and the author has been told that the addition of milk causes the fermenting material to "work itself out" much sooner. When once the evolution of gas has ceased the "emptyings" no longer work well, if at all, when used in making the sponge. This is quite different in fermenting either a batter or a clear wort with yeast, for after the fermentation has ceased and the yeast settled to the bottom, it can be saved for a week or more and used in making a sponge. This also accounts for the fact that a portion of salt-rising dough cannot be saved for the next baking with the same success as can a portion of dough made with "Sauerteig," for the ferments in the former rapidly lose their gas-producing power and fail to regain it with the addition of fresh flour and water. The "emptyings" as well fall off in gas production after fermenting for some time. After the first bubbles appear, the rate of evolution of gas increases to a maximum and then gradually decreases. When once this maximum has been passed they no longer give good results when used to make bread, for the weakening in gas production becomes even more pronounced in sponge and dough.

It is well known that, in all fermentative processes, the temperature is of great importance, and in a modern baking plant the temperature of the dough is under almost perfect control, varying only a degree or two from day to day during the four seasons of the year. Bakers who make salt-rising bread have found that  $26.6^{\circ}$  ( $80^{\circ}$  F.), the temperature at which ordinary bread is fermented, is far too low for this type of bread. To determine at what temperature the salt-rising organism is most active a number of "emptyings" were made in the usual way and placed in ovens at a temperature of  $35^{\circ}$  C.,  $40^{\circ}$  C.,  $45^{\circ}$  C.,  $50^{\circ}$  C.,  $55^{\circ}$  C. and  $60^{\circ}$  C., respectively. It was found that the optimum temperature lies somewhere between  $40^{\circ}$  and  $50^{\circ}$  and that it is possible to ferment bread of about equal quality anywhere between these two temperatures. At  $35^{\circ}$  the gas is evolved very much slower and when the bread is put into pans it takes considerably longer for it to rise to the usual height. When set at  $55^{\circ}$  C. the gas is formed slower than at lower temperatures while at  $60^{\circ}$  C. only a few bubbles of gas are formed in 24 hours. From these results it is apparent that the bacteria grow and can be used in making bread through as wide a range of temperature as yeast although it is about  $15^{\circ}$  C. higher.

It is the experience of the author that best results are obtained by fermenting the "emptyings" and sponge at a temperature rather lower than the optimum and then taking the water used in making the dough hot

enough to bring it to a temperature of 42° to 46° C. In bread made with yeast as well it is in accordance with the best bakery practice to increase the temperature toward the latter stages of the fermentation.

Sudden changes of temperature seem to have no marked influence upon the bacteria other than accelerating or retarding the rate of fermentation, for good bread was made from "emptyings" that had cooled to 22°, by making the sponge in the usual way at 39° C. and the dough at 42° C. This is of practical importance, for often in practice the "emptyings" are not kept at constant temperature. In the later stages, however, especially in the dough, it is essential that the temperature should be up to 40° C. or over.

[To be continued in February.]

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#### A RAPID METHOD FOR THE DETERMINATION OF SULPHUR IN ROASTED BLENDE.

By C. C. NITCHIE.

Received November 10, 1911.

As usually conducted, the sampling and analysis of the roasted ore drawn from a blende roasting kiln furnishes information useful only for purposes of record. A small portion is taken from each car load drawn and all such portions are mixed at the end of a day to make an average sample, which is analyzed on the following day.

This practice gives only average results, is of no value in determining the quality of ore from the individual draws, and fails entirely to detect the occasional car of poorly roasted ore which is transferred to the storage bins. It relies only on the judgment of the kiln men to determine when the ore is sufficiently roasted to be suitable for use in the spelter furnaces, and when it should be returned to the kiln for additional roasting.

In order to put the disposition of the ore on a definite basis, each draw should be sampled and analyzed for sulphur immediately after leaving the kiln. No ore should be placed in storage until the analysis is reported, showing that the sulphur is below a previously fixed maximum.

The usual gravimetric method of sulphur determination is unsuitable for such control analyses, on account of the time required, which would necessitate holding the ore in the car for a considerable length of time.

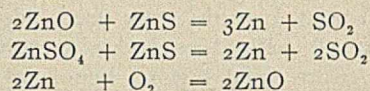
About a year ago Mr. M. F. Chase suggested to the writer that a rapid method, suitable for such determinations, might be devised by heating the ore sample in a current of air, absorbing the resulting oxides of sulphur in an excess of a standard alkali solution and titrating the excess. Based on this suggestion, the method to be described was worked up and has been in regular use since January, 1911.

In the kiln, the constituents of the original ore are mainly converted into oxides. Some remain partly in the original sulphide condition, while a small part

of the zinc, some of the lead, and all of the lime are present as sulphates. On heating this ore in a current of air to a bright red, all of the sulphides are oxidized, giving the corresponding metallic oxides and sulphur dioxide with a little sulphur trioxide. The sulphates of zinc and lead are decomposed, giving zinc and lead oxides and sulphur trioxide. The calcium sulphate, however, is not easily decomposed by heat and remains unchanged, unless the temperature is raised to a point much higher than would be practicable in any roasting furnace. Strontium and barium would probably be present as sulphates. This has not been confirmed as none of the ores on which the method has been used have contained these elements.

At first thought, this property of the calcium sulphate might seem to be a drawback to the method, but, as the result wanted is an index of the completeness of the roast, and as, even under the best conditions at the kiln the calcium in the ore will always retain its equivalent in sulphur, no real error is introduced. The result obtained is the so-called "false sulphur," the sulphur which, under proper conditions, might have been removed from the ore in the kiln.

An unforeseen phenomenon is the evolution of copious fumes of zinc oxide as long as any sulphide remains unoxidized. This undoubtedly comes from the reduction of the zinc oxide and sulphate by the zinc sulphide (reactions analogous to the well known reactions in the smelting of lead sulphide ores) with volatilization of the zinc and subsequent oxidation by the air current.



The cessation of this evolution of zinc oxide furnishes a very accurate indication of the completion of the decomposition of the sulphides and sulphates of the ore, and of the absorption of the sulphur oxides.

The fume is not appreciably dissolved by the alkali solution. Numerous tests after absorption have shown, at most, faint traces of zinc, not enough to affect the determination. To prove that it is zinc oxide, it was collected over distilled water in an inverted bottle which was then covered and allowed to stand until the fume had settled. It was then dissolved in a little dilute hydrochloric acid and potassium ferrocyanide solution added. This gave the white zinc ferrocyanide precipitate.

Phenolphthalein is used as the indicator in the titrations, as it is equally sensitive to sulphurous and sulphuric acid, showing neutrality with the normal salts of both.

For heating the ore sample to drive off the sulphur, nothing can be superior to the electric tube furnace, on account of its simple manipulation, accuracy of temperature control and cleanliness. The furnace which has been in use here is one similar to that described by Mr. G. M. Berry,<sup>1</sup> with a fused silica combustion tube. It has given even better service than was claimed by Mr. Berry, as the original winding was in service over 4,000 hours at 1000° C.,

<sup>1</sup> THIS JOURNAL, 2, 255.

part of that time for about twelve hours a day, and for nearly four months continuously without a shut down.

The ends of the tube are cooled by wrapping with strips of cotton gauze which dip into distilled water.

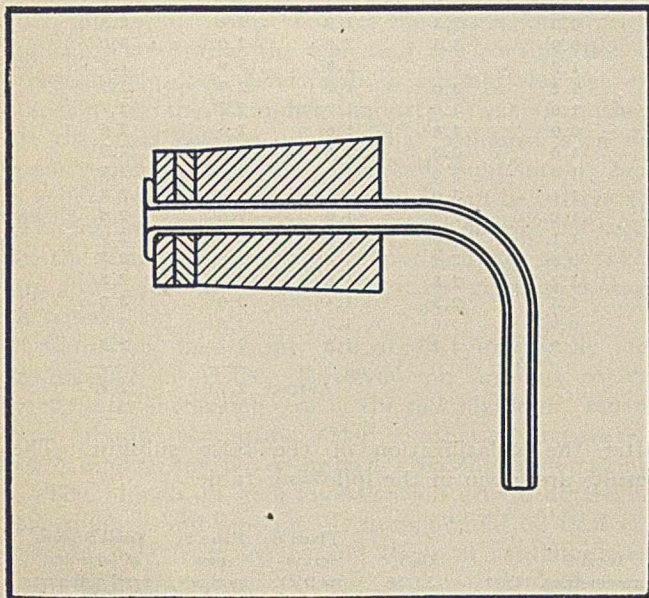


Fig. 1.

Tap water should not be used as it soon forms a crust of salts over the gauze, decreasing the cooling effect. An excellent reservoir for the cooling water consists of a rectangular box of galvanized sheet iron of about the same length as the combustion tube, about six inches high and two inches wide. This is tightly closed except for two small openings, which communicate with laterally projecting troughs at the bottom near each end. This reservoir is filled with water and placed back of the furnace, with the small troughs under the ends of the combustion tube to receive the free ends of the gauze. Atmospheric pressure keeps the water from flowing into the troughs except when evaporation has lowered the water level to the top of the openings. This reservoir contains enough water to last a week or two. Even where the ends of the tube are water-cooled, there is danger that the rubber stoppers may be overheated by the radiant heat. To avoid this the ends of the stoppers are protected by discs of  $\frac{1}{8}$ " asbestos board of the same diameter as

the tube, held in place by bordering the ends of the small glass tubes as shown in Fig. 1.

A convenient device for introducing the boat without the necessity of looking into the tube to make sure that the boat is not overturned is shown in Fig. 2. It is made of a piece of heavy wire, preferably of nickel or some other metal not easily corroded or rusted. One end is flattened out and so bent that the end projects over the end of the boat and in contact with it when both boat and wire are resting on the bottom of the combustion tube. The other end of the wire is bent at a right angle to make a hook for withdrawing the boat and also to indicate when the rod is in the proper position to keep the boat upright. A notch is filed around the wire or a finer wire wrapped around it as a guide at the point which is flush with the end of the tube when the boat has been pushed to the middle of the tube.

The solutions needed are an accurately standardized solution of sulphuric acid and one of equivalent strength of sodium hydroxide. A convenient solution is equivalent to 5 milligrams sulphur per cc. One cc. will then equal 0.5% sulphur on a 1-gram sample. The two solutions should be as nearly equal as possible to avoid the necessity for corrections in reading one in terms of the other. There should be provided a reservoir of distilled water on an elevated shelf, with a siphon tube for use in diluting the contents of the absorption bulb to the proper volume, and for washing it out after the absorption.

The most convenient form of absorption vessel which we have found is the Murray potash bulb.

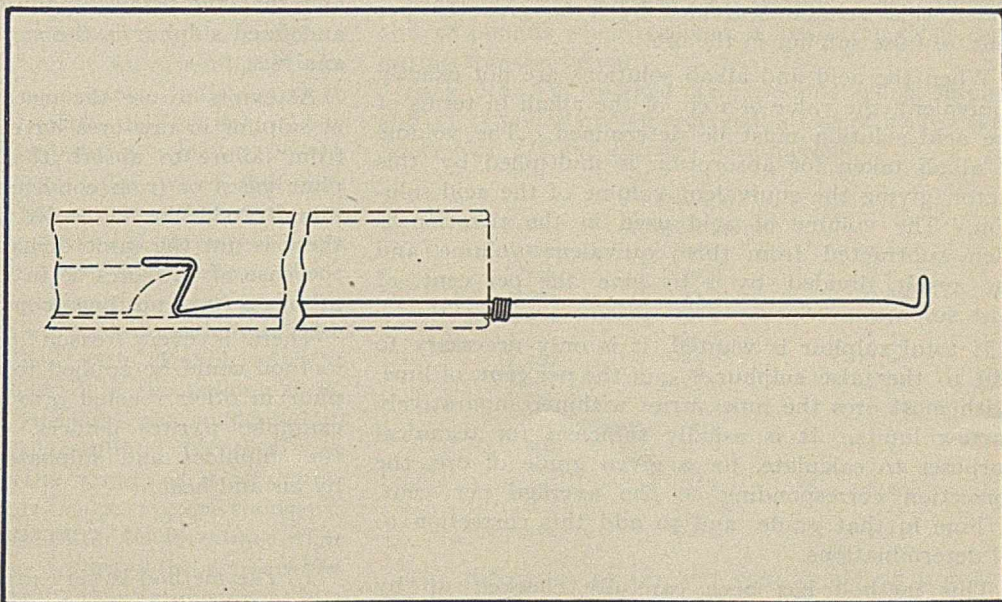


Fig. 2.

The method as in use at present is as follows:

Each car, as it is brought from the kiln, is sampled in two places by a spear sampler. This sample is cut down with a riffle to about 50 grams and ground with a few turns of the muller in a Buck's mortar.

Fine grinding is undesirable as it prolongs the time necessary for the analysis by restricting the free circulation of air through the ore. All that is necessary is to grind fine enough so that a one-gram sample will be fairly representative.

A measured quantity of the standard alkali solution is run into the absorption bulb. Seven to 10 cc. will suffice in practically all cases. This is diluted with distilled water to a volume that will bring the solution into the fifth bulb when the air current is passing. The bulb is then attached by a bent glass tube to the exit end of the furnace.

One gram of the ore is placed in a combustion boat and introduced into the tube of the furnace which has already been heated to about 1000° C. A moderately rapid current of air, freed from carbon dioxide by caustic potash or soda lime, is passed through the apparatus until the sulphur is completely driven over into the alkali solution. This usually takes about six minutes. When the zinc oxide fume has completely disappeared from the large bulb of the absorption vessel, the air current is stopped. The absorption bulb is disconnected and the solution transferred to a beaker, the bulb being thoroughly rinsed out with distilled water. A few drops of phenolphthalein are added and the standard acid is run in until the solution is colorless. If the acid and alkali solutions are exactly equivalent, the volume of acid required is subtracted from the volume of alkali originally taken for the absorption. The difference is the volume of acid equivalent to the sulphur which has been burned out of the ore. If the solutions are of the concentration mentioned above, this volume is divided by 2 to give the per cent. of false sulphur in the ore.

When the acid and alkali solutions are not exactly equivalent, the value of 1 cc. of the alkali in terms of the acid solution must be determined. The volume of alkali taken for absorption is multiplied by this factor, giving the equivalent volume of the acid solution. The volume of acid used in the titration is then subtracted from this equivalent volume and the result divided by 2 to give the per cent. of false sulphur.

If total sulphur is wanted, it is only necessary to add to the false sulphur  $\frac{32}{56}$  of the per cent. of lime. With most ores the lime varies within comparatively narrow limits. It is usually sufficient for technical purposes to calculate, for a given grade of ore, the correction corresponding to the average per cent. of lime in that grade, and to add this correction to all determinations.

This method has been carefully checked up by running daily samples, both by the new method and by the gravimetric method. The accompanying table shows the results for twenty consecutive days on Wisconsin magnetic separator concentrates.

As a further check on the method, a number of ores were analyzed for (1) total sulphur by the gravimetric method, (2) lime, (3) false sulphur by the new method, and (4) fixed sulphur remaining in the ore

False sulphur determined by new method. Per cent.	CaO.	CaO × 32/56.	Total sulphur (calculated).	Total sulphur (gravimetric).
0.6	3.4	1.9	2.5	2.8
1.1	3.0	1.7	2.8	2.8
1.1	3.1	1.8	2.9	2.9
1.2	3.0	1.7	2.9	2.9
0.9	3.3	1.9	2.8	2.8
0.9	3.5	2.0	2.9	3.0
1.2	3.1	1.8	3.0	2.8
1.2	3.0	1.7	2.9	3.1
1.0	3.0	1.7	2.7	2.7
0.9	2.8	1.6	2.5	2.6
1.0	2.8	1.6	2.6	2.9
1.0	3.0	1.7	2.7	2.7
1.1	3.0	1.7	2.8	2.8
1.0	2.8	1.6	2.6	2.8
1.1	2.8	1.6	2.7	2.9
0.9	2.4	1.4	2.3	2.5
1.2	2.8	1.6	2.8	2.5
1.5	2.5	1.4	2.9	3.0
0.9	3.0	1.7	2.6	2.6
0.9	3.1	1.8	2.7	2.8
			Average, 2.73	2.80

after the volatilization of the false sulphur. The results are given in the following table:

Total S (gravimetric).	CaO.	CaO × 32/56.	Fixed S (gravimetric).	False S (new method).	Total S (sum of false and fixed sulphur).
2.3	3.0	1.7	1.6	0.7	2.3
2.3	3.0	1.7	1.8	0.6	2.4
5.9	0.9	0.5	0.5	5.5	6.0
2.5	0.6	0.3	0.4	2.2	2.6

These figures show that, under the prescribed conditions, the fixed sulphur calculated from the per cent. of lime is the same as that found by analysis, and that the total sulphur calculated from the false and fixed sulphur is the same as that determined by analysis.

Attempts to use the method for the determination of sulphur in raw ores have led to low results, either from failure to absorb the larger quantities of sulphur gases or from condensation of part of the sulphur trioxide in the cooled portions of the tube. As there is not the same demand for a rapid method in the case of raw ores as in that of the roasted, these attempts have not been continued.

There is every reason to believe that a similar method could be applied to the determination of sulphur in other roasted ores besides those of zinc, for example, pyrites cinders, copper ores, etc., where the sulphides and sulphates are easily decomposed by air and heat.

#### CONCLUSIONS.

1. The method is very rapid, less than ten minutes being required from taking the sample to completing the titration and calculation.
2. The results are sufficiently accurate for control work in the operation of the plant.
3. The operations are all so simple that they may be entrusted to boys with but little training in chemical manipulation.

## JAMAICA CAMPHOR.

By H. W. EMERSON AND E. R. WEIDLEIN.

Received November 1, 1911.

Up to the time of the Japanese-Russian war, the growth of the *Laurus camphora* outside of the control of the Japanese government for industrial purposes was unknown. On account of the existing high prices of camphor during the war, and the continuation of these high prices, afterwards, a stimulus for the extension of the camphor industry was furnished. It therefore became evident that we must soon depend upon synthetically prepared camphor or look for new fields in which the camphor can be cultivated. The former was naturally the first to supply the needs of the time, but the latter proposition was also taken up and is just beginning to show the great advantage it possesses.

Camphor has been prepared synthetically for a number of years. However, on account of the constantly increasing cost of the raw materials, pinene and crude turpentine, the synthetic preparation of camphor has never proved a paying undertaking.

The objects of this investigation are to determine the quality and amount of camphor and oil that can be obtained from leaves and twigs of camphor trees growing in Jamaica.

The method for determining the yield of camphor was to place weighed camphor leaves in a large galvanized iron can, about two feet high and one and one-half feet in diameter, and containing a false bottom of copper gauze, four inches above the stationary bottom. Steam distillation was carried on for about two hours, at the end of which time the leaves were exhausted. A number of experiments were carried on to determine the time necessary for the complete exhaustion of the leaves, and it was found that the odor of the distillate was an accurate indicator as to whether the leaves were exhausted or not. Long distillations caused a decomposition which gave the distillate a peculiar, unpleasant odor, which was very difficult to remove. The steam, together with the camphor in the vapor form, was led through a large condenser, connected with a two-liter balloon flask, which was surrounded by running water, and this in turn was connected with a second condenser, leading into a large bottle filled with alcohol. Almost all the camphor was condensed in the balloon flask, but a very little passed over and was condensed in the second condenser. The camphor, together with the oil, was collected on the water, as it is soluble in water to the extent of only one part to one thousand, and was separated from the water by filtration. The camphor in this form was a heavy brownish-white substance containing oil and other impurities.

There was sent to us a total of 787.5 pounds of leaves, twigs, trunk and roots and as we received it it weighed 620.25 pounds, making a loss of 21.2 per cent. during transportation.

Sack No. I was labeled "Green Leaves" and was marked "weight 149 pounds." It weighed when we received it 109 pounds, and it had lost 26.8 per cent. of its weight in transportation, due largely to drying.

TABLE I.—SACK NO. I.

Leaves. Grams.	Crude camphor and oil. Grams.	Per cent.
2,000	54.09	2.7
5,000	119.00	2.4
2,000	44.5	2.2
5,000	130.0	2.6
2,000	42.0	2.1
5,000	105.0	2.1
2,000	45.0	2.3
5,000	132.0	2.6
28,000	671.5	2.4

This was the best sack of green leaves and gave a little higher per cent. of camphor.

Sack No. II labeled "Green Leaves" weighed 135 pounds, but when we received it it weighed 99 pounds, a loss of 26.6 per cent.

TABLE II.—SACK NO. II.

Leaves. Grams.	Camphor and oil. Grams.	Per cent.
2,000	23.0	1.2 <sup>1</sup>
2,000	30.5	1.52 <sup>1</sup>
5,000	93.5	1.87 <sup>1</sup>
2,000	38.0	1.90
2,000	38.0	1.90
5,330	137.0	2.57
18,330	360.0	1.82

This was a sack of wettest leaves and the leaves were mildewed considerably, and this seems to decrease the percentage of camphor more than would be accounted for by the increased moisture. The mildew possibly causes the breaking up of the cells which contain the camphor and which seem to hold it quite effectively during ordinary drying.

Sack No. III weighed 82 pounds when shipped and 58 pounds when received, a loss of 29.3 per cent. during transportation.

Sack No. IV weighed 45.5 pounds when shipped and 33.25 pounds when received, a loss of 27 per cent. *Sacks Nos. III and IV were used together.*

TABLE III.

Leaves. Grams.	Camphor and oil. Grams.	Per cent.
1,500	30.0	2.0
2,000	39.5	1.98
2,000	39.0	1.95
2,000	46.5	2.33
2,000	40.0	2.00
2,000	41.8	2.09
2,000	48.0	2.4
140 (500 leaves)	3.0	2.1
2,000	62.0	3.1
2,000	59.5	2.98
2,000	60.0	3.00
19,640	469.3	2.38

The difference in the per cent. yield as noted in the leaves was largely due to the difference in moisture contained by them, which ranged from 25 per cent. in the leaves, on the outside of the sack, to 40 per cent. in the wet leaves in the center of the sack.

The weight expressed as crude camphor in the above three tables is a mixture of gum camphor, oil of camphor and water. The condensed camphor

<sup>1</sup> Omitting because of leaks and using only those in which there was no known loss of camphor gives an average of 2.27 per cent.

and oil of camphor was filtered through a cotton plug and the camphor containing the oil of camphor thus separated from the water. No attempt was made to separate the camphor contained in this saturated water. It was used in refilling the steam chamber and in moistening the leaves in the still.

#### SUMMARY OF THE CAMPHOR AND OIL IN THE GREEN LEAVES.

Leaves. Grams.	Crude camphor and oil. Grams.	Per cent.
19.640	469.3	2.38
9.350	213.0	2.27
27.950	671.5	2.40
56.940	1353.8	2.35

#### ON REMOVING THE OIL FROM THE CAMPHOR.

The mixture of crude camphor and oil of camphor, containing some water, was separated by placing it in cotton sacks in a hand press and subjecting to pressure. The fluid thus forced out consisted of oil of camphor and water. The residue was a white crystalline camphor. The camphor was purified, by mixing with charcoal and slaked lime, and sublimed. The oil was separated from the water by means of separating funnels and Table V gives the result of this purification.

#### CAMPHOR FROM GREEN LEAVES.

Per cent.
1.32 pure camphor
0.54 oil of camphor
0.38 water
0.11 loss in manipulation and impurities.
2.35

#### CAMPHOR FROM DRIED LEAVES.

Sack V contained when shipped 99 pounds and when we received it 94 pounds, a loss of 5 per cent.

Leaves. Grams.	Camphor and oil. Grams.	Per cent.
6,000	160	2.66
6,000	142.0	2.36
2,500	70.0	2.80
6,000	176.0	2.93
20,500	548.0	2.69

Sack VI weighed when shipped 77 pounds and when received 65 pounds, a loss of 15.6 per cent.

Leaves. Grams.	Camphor and oil. Grams.	Per cent.
2,000	38.0	1.90
5,000 <sup>1</sup>	70.0	1.40
2,000	48.0	2.40
5,000	100.0	2.00
5,000	133.0	2.66
2,000	54.5	2.73
21,000	443.5	2.08

<sup>1</sup> Omitting because of leak in the still 16,000 grams of leaves, 373.5 grams of camphor and 2.33 per cent. of oil.

Sack VII weighed when shipped 100 pounds and when received 95 pounds, a loss of 5 per cent.

TABLE VIII.—SACK NO. VII.

Leaves. Grams.	Camphor and oil. Grams.	Per cent.
5,000	125.0	2.50
2,000	44.0	2.20
5,000	135.0	2.72
2,000	48.0	2.40
5,000	126.0	2.52
2,000	55.0	2.75
5,000	140.0	2.80
2,000	154.0	2.70
2,500	70.0	2.80
30,500	798.0	2.60

#### SUMMARY OF RESULTS OF DRIED LEAVES.

Removing the oil and water from the crude camphor the three fractions gave the following results:

Leaves. Grams.	Camphor and oil. Grams.	Per cent.
20,500	548.0	2.69
16,000	373.5	2.33
30,500	798.0	2.60
67,000	1719.5	2.54

Grams.	Per cent.
600 grams gave 365 camphor	60.83
110 oil	18.33
120 water	20.00
5 loss	0.84

Grams.	Per cent.
500 grams gave 305 camphor	61.00
90 oil	18.00
95 water	19.00
10 loss	2.00

Grams.	Per cent.
600 grams gave 380 camphor	63.33
105 oil	17.5
105 water	17.5
10 loss	1.67

The 2.54 per cent. of crude camphor is made up as follows:

Per cent.
1.569 pure camphor
0.457 oil
0.482 water
0.031 loss in manipulation and impurities.
2.54

A comparison of the results between the camphor yield in the wet and dried leaves is unfortunately only of comparative value because, as noted before, the green leaves had dried more or less in shipment and the dried leaves were not thoroughly dried, but were assayed as sent to determine the best time to harvest the leaves. The results indicate that there is very little or no loss of camphor during ordinary weather drying, but that the cells of the leaves hold quite securely the gum camphor. This is important as it makes possible an economical harvesting of

the leaves. The leaves may be left to fall and can be gathered at convenient intervals.

The dried leaves contain proportionately less oil of camphor and more camphor than the green leaves. This is probably due to an accelerated oxidation of oil of camphor during the last stages of life in the leaves.<sup>1</sup> This also shows that the camphor is so well enclosed in the vegetable cells that it is not easily dispensed. This is further substantiated by the fact that the dead leaves still contain a considerable quantity of camphor.

The dead leaves weighed 12 pounds when shipped and 7.5 pounds when received, making a loss during transportation of 37.5 per cent. This marked loss in weight of the dead leaves is entirely unexpected.

2730 grams of dead leaves (leaves from a dead tree) yielded 38 grams of camphor or 1.39 per cent. This is less than the yield from green or dried leaves and this camphor contained very little oil of camphor.

#### CAMPHOR IN THE TWIGS FROM GREEN LEAVES.

Twigs.	Camphor and oil.	
Grams.	Grams.	Per cent.
2.275	24.15	1.06
2.800	22.00	0.81
2.800	38.00	1.36
4.000	51.00	1.27
10.550	138.00	1.30
<u>22.425</u>	<u>273.15</u>	<u>1.16</u>

Grams.	Per cent.
136.609 camphor	50 camphor
60.28 oil	22 oil
61.24 water	22.4 water
15.03 loss	5.0 loss

The green twigs then yielded:

Per cent.
0.580 camphor
0.255 oil of camphor
0.260 water
0.065 loss

#### TWIGS FROM DRIED LEAVES.

8770 grams of twigs yielded 0.84 gram of crude camphor and 46.2 grams of pure camphor = 0.5445 per cent. camphor.

#### CAMPHOR FROM WOOD.

1800 grams of wood yields 11 grams of camphor; 0.61 per cent of camphor.

The camphor thus obtained is a white crystalline substance. When first taken from the press it has a melting point of 175.5° C. and upon resubliming and crystallizing from petroleum ether it melts at 177° C. and boils at 204.5°. It is dextrorotatory 42.82°. Specific gravity 0.980.

The properties of camphor required by the U. S. P. are as follows:

Melting point.....	174° C.
Boiling point.....	204° C.
Specific gravity.....	0.990
Dextrorotatory.....	41°

The above analyses indicate this to be a good, pure gum camphor possessing all the properties required by U. S. P.<sup>1</sup>

The oil obtained from the green leaves was fractionated as follows:

Degree.	Per cent.
Boiling point... 158-165	11.2
165-173	14.5
173-178	9.285
178-190	10.20
190-195	5.45

Degrees.	Per cent.
Boiling point... 195-205	12.9
205-228	21.1
228-240	5.45
240-255	3.63
Residue,	1.82

15.2 per cent. camphor obtained from these fractions.

The oil from the dried leaves gave the following results:

Degrees.	Per cent.
Boiling point... 158-175	7
175-185	19
185-195	20
195-205	20
205-220	10
220-240	10
240-252	5
Residue,	9

After several fractionations the following fractions were obtained:

Boiling point...	Per cent.
158-170	9.3
170-175	10.3
175-185	21.7
185-195	7.0
195-205	24.4
205-215	6.3
215-230	7.7
230-235	2.7
235-240	1.3
240-260	2.3
260-280	2.3
Residue,	6.4

camphor separated

Boiling point...	Degrees.	Per cent.
150-195		41.5
195-220		41.5
220-245		8.5
Residue red oil,		8.5

The oil has a specific gravity of 0.915.

400 cc. of the oil obtained by combining fractions which distilled at the same temperature were redistilled with the following results:

Degrees.	Cc.	Per cent.	Specific gravity.
I. 158-165	20	5	0.844
II. 165-173	76	19	0.860
III. 173-178	43	10	0.870
IV. 178-192	56	14	0.899
V. 192-202	8	2	0.915
(Va) Camphor	101 grams	25	
VI. 202-215	26	6.5	0.924
VII. 215-228	20	5.0	0.928
VIII. 228-236	18	4.5	0.931
IX. 236-252	16	4.0	0.933
X. Residue	20	5.0	
		100.0	

<sup>1</sup> J. Soc. Chem. Ind., 21, 1036.

<sup>1</sup> Pharmacopoeia of the United States, Eighth Decennial Revision, p. 88.

V and VI are the results of six fractionations distilling between 192°-215° and freezing out successively.

55 grams of camphor  
26 grams of camphor  
14 grams of camphor  
6 grams of camphor

101

(Va)

Thus from 400 cc. of oil of camphor from the Jamaica leaves we obtained 101 grams of camphor or 25.25 per cent. of camphor.

Comparing the analysis of the oil obtained from Jamaica camphor with that as published in Schimmel<sup>1</sup> we have the following:

TABLE XXIII.

	Degrees.	Jamaica. Per cent.	Schimmel. Per cent.
Boiling point..	175-180	34	26-38
	180-185	14	30-44
	185-190	7	11-19
	190-215	31	10 camphor
	215	18.5	4 safrol and eugenol.

The last three constituents are valuable.

The oil as first obtained in pressing the camphor is a colorless liquid, which soon turns yellow on exposure to the air. The first fractions are colorless and transparent and have a specific gravity of 870-910. These distil between 150-195°. The chief constituents are pinene, phellandrene, cineol, dipentene and some camphor. On further fractionating, a yellow and a red oil are obtained. The camphor is separated out from the yellow oil and safrol from the red oil.

From this report, it is evident that the determinations are almost all made from the leaves which in all cases were richer in camphor than the wood. However, the percentage of camphor contained in the leaves, with the cheap labor and the good methods for obtaining the camphor and oil, which has been found to possess equal properties with the best camphor placed on the market, renders possible the industry of camphor in Jamaica. This can be accomplished by harvesting the leaves only, thus allowing the trees to grow and become larger, more vigorous, and consequently more valuable each year.

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## THE INDEX OF REFRACTION OF THE MIXED ACIDS OF FATTY OILS.

By W. B. SMITH.

Received November 1, 1911.

This study was begun because of the anomalous figures given by Lewkowitsch,<sup>2</sup> Allen<sup>3</sup> and Van Nostrand,<sup>4</sup> for the index of refraction of the mixed acids of rape oil and cocoa butter. Without doubt these

<sup>1</sup> Schimmel & Co., Allen's "Organic Analysis," Vol. II, Part III, p. 804.

<sup>2</sup> "Oils, Fats and Waxes," 1904, 563, 728, 196; 1909, II, 202, 481; I, 411.

<sup>3</sup> "Organic Analysis," 3rd Ed., 2, 137, 166; 4th Ed., 2, 124, 178.

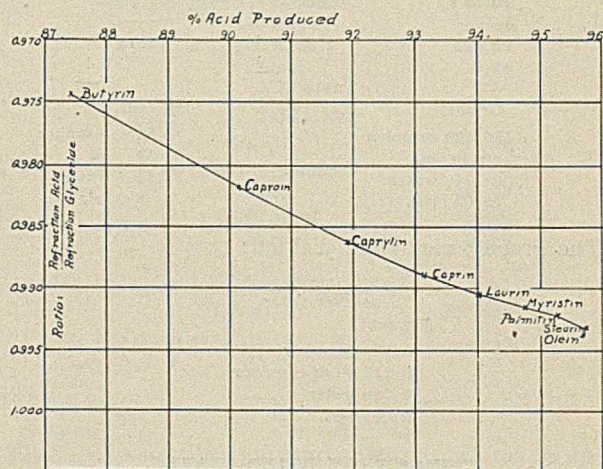
<sup>4</sup> Chemical Annual, 1909, 60, 63.

are typographical errors, 1.4991 instead of 1.4491<sup>1</sup> for rape oil at 60°, and 1.422 instead of 1.442<sup>1</sup> for cocoa butter. It seemed worth while, however, to find, if possible, a general formula for this constant and the following is offered as the solution:

TABLE I.—RATIO OF ACIDS TO GLYCERIDES.

Tri-glyceride.	Refractive Index.		Temp. °C.	"Refraction ratio" acid/glyceride.	Produced from glyceride.	
	Glyceride.	Acid.			Acid.	Glycerol Per cent.
Butyrim.....	1.43587	1.39906	20	0.9744	87.44	30.46
Caproin.....	1.44265	1.41635	20	0.9817	90.16	23.96
Caprylin.....	1.44817	1.42825	20	0.9862	91.91	19.58
Caprin.....	1.44461	1.42855	40	0.9889	93.14	16.67
Laurin.....	1.44039	1.42665	60	0.9905	94.04	14.42
Myristin.....	1.44285	1.43075	60	0.9916	94.75	12.74
Palmitin.....	1.43807	1.42693	80	0.9922	95.29	11.42
Stearin.....	1.43987	1.43003	80	0.9932	95.73	10.34
Olein.....	1.4636	1.4546	40	0.9938	95.70	10.41
Linolin.....	.....	.....	.....	(0.9935) ?	95.67	10.48
Linolenin.....	.....	.....	.....	(0.9935) ?	95.63	10.55

There have been collated in Table I the refractive indices of triglycerides and their corresponding acids, as given by Lewkowitsch, as well as the amounts of fatty acid and of glycerol produced on hydrolysis, and from these data has been calculated the ratio of the refractive index of the acid to that of the glyceride. By comparing these figures and from the curve



shown it is seen that this ratio depends upon the percentage of acid in the glyceride. The irregularities in the curve may be due to the differences of the temperatures of reading.

It is seen that the "refraction ratios" of stearin and olein are practically the same. As linolin and linolenin have nearly the same percentage composition as stearin and olein, there is no reason to doubt that their "refraction ratio" is not much different. Hence, it is reasonable to suppose that the refractive index of the mixed insoluble acids of the fatty oils may be closely calculated by multiplying the refractive index

<sup>1</sup> Thoerner, *J. Soc. Chem. Ind.*, 1895, 43.

Other errors noted: Index of refraction of black mustard oil, 1.4672 at 15.5°, *Lewk.*, 1904, 572; 1909, II, 214; Van Nostrand, p. 58; given by Tolman, *Bull.* 77, Bureau Chemistry, p. 45, as 1.4762.

Allen, 3rd Ed., p. 72, 4th Ed., p. 43, gives 0.000176 instead of 0.000365 as the factor for correction for temperature.

*Lewkowitsch*, 1904, 196; 1909, I, 411, should read for the refraction of the oils themselves: cocoanut oil, 1.441; lard, 1.4539; beef tallow, 1.4510; mutton tallow, 1.4501.



of the oil by 0.9938, the ratio of oleic acid to olein. A preponderance of laurin, myristin, or palmitin should lower this factor.

To test this hypothesis the published data was examined, and the best of it is given in Table II.

The greater number of these figures give "refraction ratios" not greatly different from 0.9938. Some, however, vary considerably from this, and consequently experimental data appeared desirable. A number of oils were procured from as reliable sources as was convenient. Rapeseed oil was extracted from the seed by ether and corn oil from yellow maize in the same way. The linseed, cod liver, peanut and olive oils and cocoa butter were purchased from trustworthy dealers, hence, although showing no evidences

in each case were read at the same temperature, and when necessary corrected to 40° by the use of the factor 0.000365, the error being negligible within short ranges of temperature. The results are shown in Table III.

Owing to the danger of oxidizing the acids if dried too long, the refractive indices of the acids are probably accurate to only about three units in the fourth decimal place. The oils themselves are more nearly correct.

The ratio for peanut oil is no doubt high because of the depression of the refraction of the oil due to the large amount (11 per cent.) of free acid present. Excluding this, the nine liquid oils range from 0.9933 to 0.9942, the average being 0.99376. Although

TABLE II.—PUBLISHED DATA.

Index of refraction.					
Oil.	Oil.	Acids.	Temp.	"Refraction ratio."	Authority.
Linseed.....	1.4660	1.4546	60	0.9922	Thoerner
Linseed.....	1.480	1.472	20	0.9946	Jensen
Cod liver.....	1.4621	1.4521	60	0.9932	Thoerner
Soy bean.....	1.4718	1.4650	27.5	0.9954	Oettinger and Buchte
	1.4728	1.4655	27.5	0.9950	Oettinger and Buchte
Rapeseed.....	1.4667	1.4491	60	0.9880	Thoerner
	1.4667	1.4991 (?)	60	1.0221	Allen
Castor.....	1.4636	1.4546	60	0.9939	Thoerner
Sunflower.....	1.4611	1.4531	60	0.9945	Thoerner
Poppyseed.....	1.4586	1.4506	60	0.9945	Thoerner
Cottonseed.....	1.4570	1.4460	60	0.9902	Thoerner
11 samples {	Max.....	1.4654	40	0.9941	Wagner and Clement
	Min.....	1.4647	40	0.9934	
	Av.....	1.4649	40	0.9937	
Sesame.....	1.4561	1.4461	60	0.9931	Thoerner
	1.4651	1.4559	40	0.9937	Utz
Almond.....	1.4555	1.4461	60	0.9935	Thoerner
	1.4674	1.4589	40	0.9942	Ross and Race
Apricot kernel.....	1.4674	1.4590	40	0.9943	Ross and Race
Peach kernel.....	1.4630	1.4558	40	0.9950	Ross and Race
Almond, Java.....	1.4602	1.4512	40	0.9938	Pactrovitch
Peanut.....	1.4545	1.4461	40	0.9942	Thoerner
Olive.....	1.4548	1.4410	60	0.9905	Thoerner
Cashew nut fat.....	1.4545	1.4459	60	0.9941	Smith and Clark
Lard (fat).....	1.4539	1.4395	60	0.9901	Thoerner
Lard, very oily.....	1.4541	1.4450	60	0.9934	Richardson
Lard, American.....	1.4594	1.4500	40	0.9936	Arnold
Lard, German.....	1.4578	1.4484	40	0.9936	Arnold
Lard, German.....	1.4582	1.4481	40	0.9931	Arnold
Chicken.....	1.4576	1.4499	50	0.9947	Ross and Race
Turkey.....	1.4565	1.4470	50	0.9935	Ross and Race
Rabbit.....	1.4587	1.4495	40	0.9937	Lewkowitzsch
Palm.....	1.4510	1.4441	60	0.9952	Thoerner
Tallow, beef.....	1.4510	1.4375	60	0.9907	Thoerner
Tallow, mutton.....	1.4501	1.4374	60	0.9912	Thoerner
Butter.....	1.445-1.448	1.437-1.439	60	0.9945-0.9938	Thoerner
Butter.....	1.4550	1.4470	40	0.9945	Arnold
Margarin.....	1.453	1.444	60	0.9938	Thoerner
Cacao butter.....	1.4496	1.442	60	0.9948	Thoerner
	1.4496	1.422 (?)	60	0.9810	Allen
Palmnut.....	1.4431	1.4310	60	0.9916	Thoerner
Cocoanut oil.....	1.4410	1.4295	60	0.9920	Thoerner

of adulteration are not guaranteed to be absolutely pure. The other oils were obtained from known sources.

Separation of the acids was performed by saponifying with alcoholic soda, boiling off the alcohol, dissolving in water, treating with dilute H<sub>2</sub>SO<sub>4</sub>, separating the oily layer and drying and filtering if necessary. Drying was curtailed as much as possible to avoid oxidation. The refractive index was determined on an Abbe-Zeiss refractometer with heatable prisms, a temperature of 40° being used except where the fats were solid at this temperature. The oil and acids

only a few samples were examined, the closeness of the result to the factor derived from Table I indicates that it is correct, while the variety of oils tested would show that it is of general application. The nine samples of solid fats range from 0.9941 to 0.9923, averaging 0.9932. The lower acids were evidently washed out of the butter. The other fats show a lower factor in proportion as they contain much palmitin, myristin or laurin.

The last column in Table III shows refractive indices of other observers calculated to 40°, excluding the incorrect figures on rape oil and cocoa butter. The

result obtained by Thoener on olive oil does not seem admissible, as the refractive index of oleic acid, which forms 75 to 90 per cent. of the fatty acids, is 1.4471 at 60°, or 1.4546 at 40°. If we take as a basis for calculation the index of refraction of linolic acid as approximately 1.4643 at 40° and the composition of the fatty acids of an olive oil as 10 per cent. palmitic acid, 84 per cent. oleic and 6 per cent. linolic, the refractive index of the mixture is about 1.4539 at 40°, while Thoerner's figure of 1.441 at 60° is about 1.4483, against the value on Table III of 1.4528.

TABLE III.—EXPERIMENTAL RESULTS.

Oil.	Index of refraction.		"Refractio ratio."	Refractive index mixed acids, other observers, calc. to 40°.
	Oil.	Acids.		
Linseed oil.....	1.4734	1.4635	0.9933	1.4619-1.4647
Cod liver oil.....	1.4711	1.4625	0.9942	1.4594
Soy bean oil.....	1.4682	1.4586	0.9935	1.4610
Corn oil <sup>1</sup> .....	1.4677	1.4583	0.9936	1.4585
Corn oil <sup>2</sup> .....	1.4675	1.4585	0.9939	1.4585
Rapeseed oil.....	1.4658	1.4572	0.9941	1.4564
Cottonseed oil.....	1.4637	1.4545	0.9937	1.4533-1.4567
Peanut oil.....	1.4633 <sup>3</sup>	1.4552	0.9947	1.4534
Olive oil.....	1.4619	1.4528	0.9938	1.4483
Lard.....	1.4590	1.4491	0.9932	1.4468-1.4500
Lard stearin (very soft)	1.4585	1.4487	0.9933	.....
Oleo oil.....	1.4578	1.4479	0.9932	(tallow 1.4447-1.4448)
Cocoa butter.....	1.4563	1.4475	0.9939	1.449
Butter (renovated).....	1.4544	1.4458	0.9941	1.444-1.447
Oleostearin.....	1.4549	1.4437	0.9923	(tallow 1.4447-1.4448)
Palmnut oil.....	1.4496	1.4392	0.9928	1.4383
Cocoonut oil.....	1.4494	1.4386	0.9925	1.4368
(80% cottonseed).....	1.4624	1.4527	0.9934	.....
(20% oleostearin)				

*Effect of Unsaturated Acids.*—The data given in this paper show that the iodine value and the higher refraction of unsaturated acids, due to double and triple linkages, do not effect this factor materially. As the iodine value and the refraction of the acids increase, those of the glycerides increase also. The difference between the refraction of the glycerides and of the acids, averaging 0.0092 for fatty oils, is so small in comparison with the total refraction that the absolute refraction has little effect on the factor.

The extremes of 1.5 and 1.4 would give  $\frac{1.4908}{1.5000} = 0.9939$  and  $\frac{1.3908}{1.4000} = 0.9934$ . This is, of course, true only of these oils. The relation between iodine value and this factor is shown in Table IV.

TABLE IV.—EFFECT OF UNSATURATION.

	Index of refraction.		Temp. Degrees.	Factor.	Iodine no. (Estimated.)	Per cent. unsaturated acids.
	Glyceride.	Acid.				
Olein.....	1.4563	1.4471	60	0.9937	86.2	100
Stearin.....	1.4472	1.4373	60	0.9932	0.0	0
Linseed oil... 1.4734	1.4635	40	0.9933	185	90	
Corn oil..... 1.4677	1.4583	40	0.9936	120	92	
Olive oil..... 1.4619	1.4528	40	0.9938	85	90	
Lard..... 1.4590	1.4491	40	0.9932	62	70	
Oleo oil..... 1.4578	1.4479	40	0.9932	50	55	
Cocoa butter. 1.4563	1.4475	40	0.9939	35	40	

<sup>1</sup> Extracted in laboratory.<sup>2</sup> Outside source.<sup>3</sup> Eleven per cent. free acid.

Here there is, with olein and stearin, a difference in refraction of 0.0091, of iodine value of 86.2, and in saturation of 100 per cent., yet the factor is about the same. With the oils given, which consist very largely of glycerides of acids of eighteen carbon atoms, but which differ widely in refraction, iodine value, and saturation, the variation in the factor is within the limits of error.

## CONCLUSIONS.

1. The values 1.4991, 1.422 and 1.441 for the refractive indices at 60° of the insoluble acids of rape oil, cocoa butter and olive oil, respectively, are not correct.

2. The relation between the refraction of the oil and the refraction of the acids depends upon the percentage of acid in the glycerides, being nearly independent of the total refraction and of the iodine value.

3. The refractive index of the insoluble acids of fatty oils should be close to the refractive index of the oil multiplied by 0.9938.

4. For solid fats this factor should lie between 0.992 and 0.994, according to the composition of the fat.

LABORATORIES U. S. BUREAU  
OF ANIMAL INDUSTRY.

THE CHLORINE CONTENT OF MILK.<sup>1</sup>

By PAUL POETSCHKE.

Received October 29, 1911.

In a previous publication<sup>2</sup> concerning the determination of sodium chloride in milk, the author gave preliminary consideration to the ratio existing between ash and sodium chloride as a means of detecting added salt. The present paper is the result of an extended study of this ratio.

All of the chlorine determinations recorded in this paper were made by the author's method. Briefly, this consists in removal of the fat and proteids with copper sulphate and sodium hydroxide. The filtrate is acidified with nitric acid and titrated by Volhard's method.

Total solids were determined with the aid of a special pipette<sup>3</sup> devised to deliver five grams of milk. The lactometer readings recorded in all of the present work were taken with the instrument described in the same paper on total solids. Fat was determined by the Babcock method. Incinerations for ash were made in a muffle, care being taken to prevent the platinum dishes from being heated to visible redness.

Richmond<sup>4</sup> states that the chlorides in milk can be titrated with silver nitrate, using potassium chromate as indicator, and that 10 cc. of milk took on an average 3.45 cc. N/10 silver nitrate with extremes of 3.35 and 3.6 cc. in nine samples.

It is well known that phosphates interfere with the determination of chlorine by this method. In the case of milk, a proper end point can not be obtained.

<sup>1</sup> Read before N. Y. Section, October, 1911.<sup>2</sup> THIS JOURNAL, 2, No. 5.<sup>3</sup> *Ibid.*, 3, No. 6.<sup>4</sup> *Analyst*, 34, 208.

Instead of changing to a reddish color when all of the chlorine is converted to silver chloride, a yellow color, gradually becoming deeper and developing into orange, is noted. A reddish color is observed only after a considerable excess of silver nitrate is present.

In Table I is given the results obtained by direct titration with silver nitrate using potassium chromate as indicator. The results obtained with the author's method are given for comparison.

The direct titrations with chromate indicator were made on 10 cc. of milk, with and without dilution. In each case the same amount of indicator was used and the titration continued until the first change to a deeper yellow was noted. The milk, containing the same amount of indicator, was used as a control to note the first change in color.

TABLE I.—SODIUM CHLORIDE IN GRAMS PER 100 CC. Direct titration with chromate indicator.

Number.	10 cc. undiluted.	10 cc. diluted with 90 cc. of water.	Author's method.
71,884	0.246	0.324	0.180
71,885	0.238	0.276	0.167
71,888	0.248	0.315	0.183
71,900	0.167	0.334	0.194

These results show that direct titration of milk with silver nitrate, using potassium chromate as indicator, is impossible. Richmond fails to give any data concerning the accuracy of the direct titration. The five experiments which he records, showing "salt added" and "salt found," may be correct in so far as the added salt is concerned, provided the titration is carried to the same end point as the original milk. This is no proof whatever that the chlorine in milk can be determined by direct titration.

Milk from individual cows may vary greatly from the normal. Certified milk offers an opportunity to study the mixed milk of healthy cows. Such milk is produced under the regulations of a medical or scientific milk commission or else inspected by law.

Table II gives the results obtained on ten samples of certified milk.

TABLE II.

Number.	Total solids.	Fat.	Proteids.	Lactometer at 60° F.	Ash.	Sodium chloride.	Ratio ash/sodium chloride.
57,025	12.50	3.60	3.28	115	0.781	0.181	4.31
53,989	12.18	3.60	..	111	0.730	0.158	4.62
53,990	12.24	4.10	..	105	0.725	0.205	3.54
58,037	14.40	4.90	..	121	0.739	0.111	6.66
67,526	13.05	4.60	2.66	105	0.693	0.154	4.50
64,304	14.01	5.50	3.25	104	0.740	0.200	3.70
68,808	12.74	4.00	..	112	0.745	0.143	5.21
71,617	12.54	3.90	3.22	110	0.790	0.195	4.05
a.	..	..	..	..	0.729	0.163	4.47
b.	..	..	..	..	0.759	0.186	4.08
Maximum	..	..	..	..	0.790	0.205	6.66
Minimum	..	..	..	..	0.693	0.111	3.54
Average	..	..	..	..	0.743	0.170	4.51

Table III gives the results obtained on twenty-two samples of market milk.

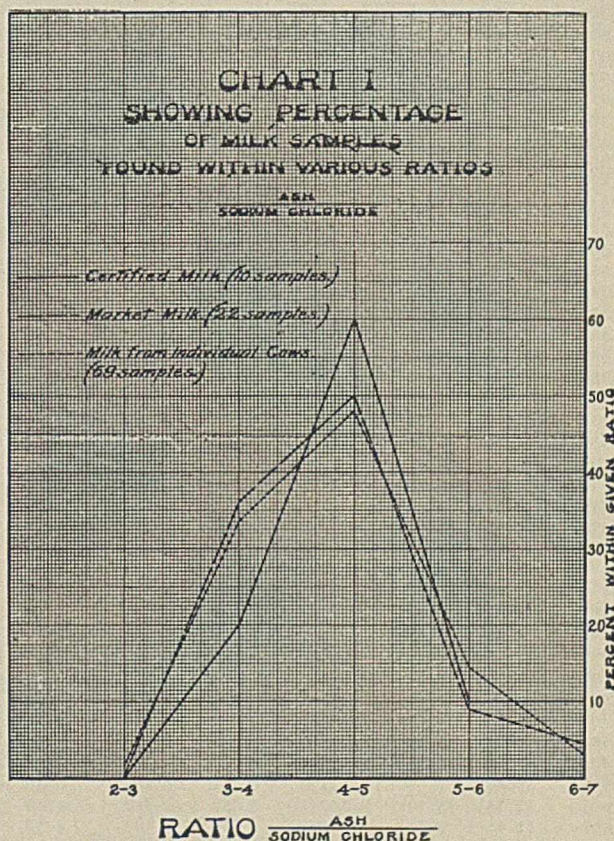
The sixty-nine samples of milk, recorded in Table V, are from individual cows. As far as it was possible to ascertain, these samples were taken under conditions which prevented their being watered or otherwise tampered with. It was necessary to preserve

TABLE III.

Number.	Total solids.	Fat.	Lactometer at 60° F.	Ash.	Sodium chloride.	Ratio ash/sodium chloride.
59,604	16.12	6.50	118	0.816	0.180	4.53
65,055	11.81	3.40	110	0.760	0.149	5.10
65,056	11.81	3.40	110	0.760	0.113	6.73
65,057	11.34	3.30	104	0.680	0.158	4.30
65,058	10.77	2.80	104	0.690	0.149	4.63
65,059	11.72	3.40	109	0.720	0.149	4.83
65,060	11.80	3.45	108	0.610	0.153	3.99
64,907	12.17	3.60	112	0.666	0.167	3.97
64,909	12.04	3.60	111	0.716	0.193	3.70
64,910	12.47	3.60	114	0.698	0.180	3.87
64,933	12.78	4.10	112	0.696	0.169	4.12
64,934	12.49	3.80	112	0.720	0.176	4.09
64,935	12.04	3.60	109	0.730	0.159	4.50
64,936	11.63	3.20	110	0.700	0.154	4.54
64,937	12.01	3.40	112	0.726	0.169	4.29
64,938	12.04	3.50	111	0.734	0.154	4.75
64,256	12.46	3.70	..	0.710	0.143	4.96
64,257	12.37	3.60	..	0.710	0.130	5.46
69,190	11.57	3.90	97	0.684	0.220	3.11
69,191	11.38	3.80	96	0.696	0.193	3.62
69,192	10.92	3.65	92	0.705	0.228	3.09
69,193	11.17	3.80	93	0.677	0.206	3.28
Maximum	..	..	..	0.816	0.228	6.73
Minimum	..	..	..	0.610	0.113	3.09
Average	..	..	..	0.709	0.168	4.34

these samples. Formaldehyde was added in the proportion of about 1 to 2000.

An experiment was made to determine whether formaldehyde in any way interfered with the determination of chlorine. A sample of milk was treated with formaldehyde (approximately 40 per cent.)



in the proportion of 1 to 1000 and 1 to 500. Chlorine was determined in the original and treated samples.

The results given in Table IV show that, even in this unusually large proportion of formaldehyde, no error is introduced.

TABLE IV.

Original sample	Sodium chloride.	
	Per cent.	
Original sample	a.	0.144
	b.	0.143
Treated with 1-1000 formaldehyde	a.	0.147
	b.	0.142
Treated with 1-500 formaldehyde	a.	0.144
	b.	0.144

TABLE V.

Number.	Total solids.	Fat.	Lactometer at 60° F.	Ash.	Ratio ash/sodium chloride.	
					Sodium chloride.	sodium chloride.
68,344	13.01	4.60	105	0.756	0.184	4.11
68,345	12.66	4.20	109	0.740	0.170	4.35
68,346	12.79	4.00	111	0.750	0.197	3.81
68,347	11.89	3.60	107	0.686	0.177	3.87
68,348	12.62	3.75	115	0.840	0.190	4.42
68,349	11.68	3.70	101	0.658	0.204	3.23
68,350	12.18	3.40	113	0.730	0.181	4.03
68,351	14.73	5.50	114	0.736	0.143	5.15
68,352	11.41	3.20	106	0.800	0.232	3.45
68,353	13.33	4.85	107	0.750	0.170	4.41
68,354	11.67	3.45	106	0.700	0.175	4.00
68,355	14.18	4.90	...	0.720	0.196	3.67
68,356	11.37	3.30	104	0.736	0.198	3.72
68,357	11.63	3.20	109	0.762	0.166	4.59
68,358	13.05	4.20	113	0.732	0.132	5.55
68,359	12.30	3.80	108	0.786	0.186	4.22
68,360	12.57	4.40	102	0.670	0.180	3.72
68,361	13.64	4.40	...	0.730	0.134	5.45
68,362	12.55	3.80	...	0.800	0.203	3.94
68,363	11.17	3.20	104	0.750	0.216	3.47
68,364	12.72	3.60	...	0.720	0.171	4.21
68,365	12.81	4.00	...	0.694	0.178	3.90
68,366	12.99	4.65	104	0.750	0.185	4.05
68,367	12.21	3.60	111	0.748	0.150	4.99
68,368	12.17	3.60	111	0.734	0.164	4.48
68,369	14.75	6.10	107	0.730	0.186	3.93
68,370	10.87	3.20	100	0.704	0.222	3.17
68,371	11.47	3.40	104	0.796	0.209	3.81
68,372	12.19	4.00	104	0.700	0.181	4.09
68,373	12.66	4.20	106	0.778	0.174	4.47
68,374	13.26	4.30	114	0.740	0.146	5.07
68,375	12.21	3.80	108	0.666	0.164	4.06
68,376	14.43	4.60	125	0.928	0.212	4.38
68,377	13.73	4.80	111	0.736	0.157	4.69
68,378	14.49	5.60	109	0.740	0.173	4.28
68,379	11.29	3.40	102	0.726	0.216	3.36
68,380	12.19	3.40	113	0.730	0.140	5.21
68,381	11.09	3.00	105	0.724	0.185	3.91
68,382	11.57	2.80	114	0.710	0.155	4.58
68,383	11.10	2.70	111	0.780	0.131	5.95
68,384	12.52	3.60	115	0.632	0.147	4.30
68,385	12.63	4.00	112	0.634	0.157	4.04
68,386	12.93	4.30	109	0.670	0.152	4.41
68,387	11.73	3.60	103	0.616	0.174	3.54
68,388	11.22	2.50	117	0.662	0.138	4.80
68,389	12.50	3.80	111	0.710	0.141	5.03
68,390	11.02	2.95	105	0.706	0.197	3.58
68,391	12.39	3.70	111	0.720	0.128	5.62
68,392	12.03	3.40	111	0.710	0.154	4.61
68,393	10.92	2.50	111	0.774	0.161	4.80
68,394	12.03	3.70	106	0.720	0.157	4.59
68,395	13.08	4.10	114	0.730	0.148	4.93
68,396	11.49	3.60	102	0.790	0.241	3.28
68,397	11.41	2.90	112	0.690	0.165	4.18
68,398	12.56	3.40	...	0.690	0.127	5.43
68,399	9.75	2.60	...	0.820	0.335	2.45
68,400	11.64	3.50	104	0.700	0.184	3.80
68,401	12.30	3.90	106	0.704	0.196	3.59
68,402	11.74	3.60	106	0.710	0.174	4.08
68,403	9.78	2.40	97	0.730	0.228	3.20
68,404	11.46	2.80	116	0.720	0.176	4.09
68,405	10.88	2.60	111	0.772	0.203	3.80
68,406	11.73	3.40	107	0.758	0.162	4.68
68,407	11.15	2.30	118	0.710	0.143	4.97
68,408	11.82	3.00	118	0.720	0.116	6.20
68,409	12.18	3.60	112	0.726	0.126	5.76
68,410	9.70	3.40	80	0.532	0.145	3.67
68,411	11.77	3.50	108	0.720	0.152	4.74
68,412	13.93	4.90	114	0.742	0.112	6.63
Maximum	...	...	...	0.928	0.335	6.63
Minimum	...	...	...	0.532	0.112	2.45
Average	...	...	...	0.728	0.173	4.33

Several samples of milk, found to contain large amounts of added salt, are given in Table VI. These samples were all very salty to the taste. Evidently the salt was added in these cases by persons who sought to satisfy a personal grudge against the dealer or consumer. The samples were received for examination for foreign substances.

TABLE VI.

Number.	Total solids.	Fat.	Lactometer at 60° F.	Ash.	Sodium chloride.	Ratio ash/sodium chloride.
64,908	12.23	3.40	118	0.780	0.415	1.88
49,383	12.10	3.50	117	1.140	0.615	1.85
67,819	14.84	5.80	...	3.160	2.610	1.21

One sample of milk containing both added water and salt is given below. The taste of this sample was not distinctly salty.

SAMPLE NO. 54,284.

	Per cent.
Lactometer at 60° F.	81
Total solids	8.00
Fat	2.00
Ash	0.65
Sodium chloride	0.239
Ratio ash/sodium chloride	2.71

Table VII gives the maximum, minimum and average results obtained on certified milk, market milk and milk from individual cows.

TABLE VII.

		Ash.	Sodium chloride.	Ratio ash/sodium chloride.
Certified milk, 10 samples	Maximum	0.790	0.205	6.66
	Minimum	0.693	0.111	3.54
	Average	0.743	0.170	4.51
Market milk, 22 samples	Maximum	0.816	0.228	6.73
	Minimum	0.610	0.113	3.09
	Average	0.709	0.168	4.34
Milk from individual cows, 69 samples	Maximum	0.928	0.335	6.63
	Minimum	0.532	0.112	2.45
	Average	0.728	0.173	4.33

Table VIII shows the percentage of milk samples found within various ratios of  $\frac{\text{Ash}}{\text{sodium chloride}}$ .

TABLE VIII.

Ratio ash/sodium chloride	2 to 3	3 to 4	4 to 5	5 to 6	6 to 7
Certified milk, 10 samples	0	20.0%	60.0%	10.0%	10.0%
Market milk, 22 samples	0	36.4%	50.0%	9.1%	4.5%
Milk from individual cows, 69 samples	1.5%	33.3%	47.8%	14.5%	2.9%

The results given in Table VIII, represented graphically in Chart I, show that 90 per cent. of the certified milk samples, 95.5 per cent. of the market milk samples and 95.6 per cent. of the samples of milk from individual cows be within the ratio of 3 to 6. Comparatively few samples fail to come within this range.

It is apparent that the chlorine content of milk is subject to wide variation and that the average chlorine content, expressed in terms of sodium chloride, is 0.17 per cent. The average ratio of  $\frac{\text{Ash}}{\text{sodium chloride}}$  is 4.5.

In conclusion I wish to acknowledge my indebtedness to my assistants, Messrs. Carl Marx and J. Schroff, for their assistance in the analytical work.

TEMPERATURE CORRECTIONS IN RAW SUGAR POLARIZATIONS.<sup>1</sup>

By W. D. HORNE.

Received November 23, 1911.

In polarizing raw sugars at temperatures other than the standard 20° C., errors are introduced that are of serious moment. Much has been written on the subject and temperature corrections have been worked out, but unfortunately they have not been generally adopted.

One of the best methods of effecting the necessary corrections is that described by Dr. C. A. Browne at the 7th International Congress of Applied Chemistry, to be described later; and the present paper is to present results on many hundreds of analyses, which entirely corroborate the reliability of this method of correction.

In the spring of 1909, when the New York Sugar Trade Laboratory was planned, I made some preliminary experiments on temperature corrections in my own laboratory to ascertain the relative results of polarizing at 20° and at higher temperatures, and later I have made continued close comparisons between the polarizations of the Sugar Trade Laboratory at 20° and polarization of the same sugars in other laboratories at ordinary room temperatures.

In the early experiments it was necessary, for polarizing at 20°: 1, that the solution of the normal weight of sugar should be made up to 100 cc. at 20° C.; 2, that it should be polarized at 20° C.; 3, that the polariscope should be at 20° C.

In order to accomplish these ends I cooled the solution of sugar in a flask with a thermometer in ice-water to 20° C., filling to the mark with a few drops of water at about this same temperature. The solution was then shaken and filtered at room temperature, which

polariscope. These caps fit tightly over the ends of the observation tube and each has a glass windowed end and an annular space inside filled with soda lime, which was found to be much better for this purpose than calcium chloride or caustic alkali.

The polariscope was kept at 20° C. by enclosing the working parts, including both polarizing and analyzing nichols, in a galvanized iron box covered with a 5/8-inch layer of cork board made by the Armstrong Cork Company, of Pittsburgh, and containing two 1/4-inch copper tubes, running along each side of the inside of the box, through which ice-cold water was circulated.

It was found advisable to pass water at 1° C. through this tube at the rate of 100 cc. per minute, which cooled the polariscope to 20° C. as shown by a thermometer passing through the box and covering, near the analyzer and compensating quartz wedges. The water issued at about 13° C. when the temperature of the room was between 23° and 31° C. The polariscope was maintained at 20° C. about an hour before polarizations were made and the observation tube when inserted at other than 20° quickly came to this standard, a change of as much as two degrees taking place in four minutes.

Cane sugars polarized at temperatures higher than 20° C. are subject to a temperature correction, which may be divided into two parts: 1, the temperature correction for pure sucrose, given by the formula  $P_{20} = P' [1.0 + 0.0003(t - 20)]$  and 2, the temperature correction as applied to pure levulose, the formula being  $P_{20} = P' - 0.00812L(t - 20)$ . By properly applying the combination of these formulas to a raw cane sugar polarized at temperatures other than 20°, it is possible to arrive at a result very close indeed to that which would be obtained when polarizing the sugars at 20°, as may be seen by the following set of

TABLE I.

			Calculated correction for sucrose alone.	Calculated correction for levulose.	Calculated correction for both levulose and sucrose.	Difference of calculated polarization from actual polarization at 20° C.
At 20.	At 26.5.	Diff.				
95.4	95.3	-0.10	+0.1868	-0.0330	+0.1538	+0.0538
At 21 C.						
95.5	95.25	-0.25	+0.1572	-0.0279	+0.1293	-0.1207
95.5	95.2	-0.30	+0.1571	-0.0279	+0.1292	-0.1708
95.3	95.15	-0.15	+0.1570	-0.0279	+0.1291	-0.0409
At 20 C						
95.3	95.15	-0.15	+0.1855	-0.0330	+0.1525	+0.0025
95.5	95.4	-0.10	+0.1862	-0.0330	+0.1532	+0.0532
95.3	95.2	-0.10	+0.1855	-0.0330	+0.1525	+0.0525
95.2	95.0	-0.20	+0.1856	-0.0330	+0.1526	-0.0476
Average						-0.0247

necessarily warmed it up a little, as the laboratory was usually above 20° C. This filtrate was put into a tubulated 200 mm. observation tube, containing a centrally located thermometer, and immersed in ice-water until the temperature fell a little below 20° C. (about 18° C.), the tube dried with a towel, two protecting caps containing granular soda lime to prevent condensation of atmospheric moisture on the cold end glasses slipped on, and the whole put into the

observations, on eight raw sugars, giving the polarizations actually obtained at 20° C. (the polariscope, the solution and containers all being at this same degree), as compared with polarizations of the same sugars at higher degrees, as indicated in Table I. These 95° sugars were assumed to contain 1.25 per cent. invert sugar on the average, and having calculated the corrections for the sucrose and levulose separately and having united all these results, it was found that these corrected polarizations were only 0.0247

<sup>1</sup> Paper presented at the meeting of the Association of Official Agricultural Chemists, Washington, November 22, 1911.

polariscopic degrees lower than the polarizations actually made at 20° C.

As the invert-sugar content of these sugars was not definitely known, a further investigation of 12 samples of raw sugar was made in which the invert-sugar content of each was accurately determined, giving the results shown in Table II.

By use of the formulas above given, the polarizations of all the raw sugars coming to two technical laboratories have been arranged in groups covering monthly periods, corrected for temperature and compared with the corresponding average polarizations obtained in the Sugar Trade Laboratory, when tested at 20° C.

TABLE II.

At 20°.	At 28°.	Diff.	Calculated correction for sucrose alone.	Invert sugar.	Calculated correction for levulose alone.	Combined corrections.	Difference of calculated polarization from polarization at 20°.
95.25	95.1	-0.15	+0.2282	1.33	-0.0432	+0.1850	+0.0350
86.80	85.7	-0.10	+0.2057	2.50	-0.0812	+0.1245	+0.0245
94.25	94.0	-0.25	+0.2256	1.45	-0.0471	+0.1785	-0.0715
94.3	94.1	-0.20	+0.2258	1.39	-0.0451	+0.1807	-0.0193
At 25°							
93.5	93.3	-0.20	+0.1400	1.84	-0.0374	+0.1026	-0.0974
94.7	94.55	-0.15	+0.1418	0.81	-0.0164	+0.1254	-0.0246
85.8	85.7	-0.10	+0.1286	1.77	-0.0359	+0.0927	-0.0073
94.7	94.6	-0.10	+0.1419	1.07	-0.0217	+0.1202	+0.0202
94.6	94.4	-0.20	+0.1416	1.45	-0.0294	+0.1122	-0.0878
At 27°							
87.6	87.6	-0.00	+0.1839	2.15	-0.0595	+0.1244	+0.1244*
95.3	95.15	-0.15	+0.2001	0.91	-0.0260	+0.1741	+0.0241
94.6	94.4	-0.20	+0.1998	1.48	-0.0421	+0.1577	-0.0423
							Average -0.0101
							Average without* = -0.0205

Having applied the formulas for correction of sucrose and levulose, the results obtained in the last column show the differences of the polarizations made at high temperatures and calculated back to 20°, from the polarizations actually made at 20°. The average of 12 such determinations gives us a result for the sugars polarized above 20° of only 0.0273 polariscopic degrees lower than the polarizations actually made at 20° C. If we omit the tenth sample in the above series, which seems to be irregular, we find that the average calculated polarization is only 0.0186 polariscopic degrees lower than the polarization actually made at 20°.

These observations are quite in accord with those made by Browne, on mixtures containing known amounts of sucrose and levulose in raw sugars and molasses and reported by him in the article above cited.

The results found are expressed in the following table, which gives in decimal fractions of a single degree polariscopic the variations of my laboratory "A" and laboratory "B" results after calculating to 20° C., from the results obtained in the Sugar Trade Laboratory when conducting the whole operation at 20° C.

Thus we see that the average discrepancy in this series of about one thousand samples is only +0.0148 polariscopic degree, but that if the levulose correction has not been applied this quantity would be increased to +0.0499.

The levulose correction is thus twice as great as the total experimental error and should by no means be ignored. The ideal method is, of course, to conduct the entire operation of polarizing sugars at 20°, but as this requires an expensive plant or somewhat increased work and time by the method outlined

Date. 1911.	Laboratory "A".				Laboratory "B."			
	Sucrose cor.	Levulose cor.	Total cor.	Diff. from Trade Lab.	Sucrose cor.	Levulose cor.	Total cor.	Diff. from Trade Lab.
March	+0.0800	-0.0140	+0.0660	-0.0070	+0.0424	-0.0059	+0.0365	-0.0096
April	+0.1290	-0.0236	+0.1054	+0.0140	+0.0239	-0.0036	+0.0203	+0.0200
May	+0.2002	-0.0303	+0.1699	+0.1190	+0.0990	-0.0180	+0.0810	+0.0710
June	+0.1977	-0.0575	+0.1402	+0.0436	+0.1832	-0.0355	+0.1477	+0.0117
July	+0.2235	-0.0576	+0.1659	+0.0273	+0.2373	-0.0580	+0.1793	-0.0170
Aug.	+0.2329	-0.0634	+0.1695	+0.0625	+0.1177	-0.0344	+0.0833	+0.0193
Sept.	+0.1379	-0.0373	+0.1006	+0.0047	+0.1666	-0.0520	+0.1146	-0.0267
Average corrections for both laboratories					+0.1486	-0.0351	+0.1135	+0.0148

In order to get a further comparison between polarizations made at 20° C., and those made at higher temperatures, and calculated to 20° C., I have made monthly comparisons between the polarizations of many hundreds of samples tested both at the Sugar Trade Laboratory and at other laboratories.

above, cases will arise where a temperature correction will be required. My contention is that if any such correction is to be applied it should be done in accord with a full appreciation of the significance of levulose as well as of sucrose and thus be made as correct as our present knowledge will allow.

# LABORATORY AND PLANT.

## AN ELECTRIC LABORATORY FURNACE.

By RAYMOND C. BENNER.

Received August 16, 1911.

In taking up the subject of methods of ignition, we must, of necessity, go back to the time of Bunsen. He would feel quite at home in this field, for, here, things have been at a standstill and we have remained about where he left us until within the last year or so, since which time more attention has been given to this important subject. In most places the Bunsen burner and air blast furnish the sole sources of heat for ignition purposes.

In order that a given compound may have a definite and constant composition for weighing, both the manner and temperature of ignition must be taken into consideration. In the case of many elements such as silicon and calcium, the form in which they are usually weighed is not altered by the highest temperatures obtainable with the Bunsen burner or the laboratory blast lamp. For such as these, this method of ignition may be used with perfect satisfaction.

Another factor, however, to be reckoned with, is the loss of weight undergone by the platinum crucibles when ignited in the open flame. By heating a platinum crucible weighing about 15 grams by means of gas, which contained considerable sulphur, the author has found this loss to be as high as 1 mg. per hour.

The question of temperature is likely to be the one of most importance, and one in which the analyst is most likely to err, since the various texts give but an indefinite idea as to the maximum temperature which certain precipitates will stand before undergoing decomposition, or, on the other hand, as to what temperature it is necessary to use in order to obtain a compound of definite composition. Treadwell's "Analytical Chemistry," N. Y., 1910, p. 167, says, in regard to the ignition of cadmium sulphate in a double crucible: "The outer crucible can be heated to the full red heat of the Teclu burner without running risk of decomposing the cadmium sulphate. It is, however, not necessary to heat it so strongly." In regard to the ignition of manganese sulphate, the following statement is found in Fresenius, "Quantitative Analysis", N. Y., 1896, p. 158: "It resists a very faint red heat, but upon exposure to a more or less intense red heat, it suffers decomposition."

Electricity makes the most convenient, cleanest way of obtaining heat for many laboratory purposes, but when platinum resistance furnaces are utilized, the likelihood of their burning out, as well as first cost, often makes their use prohibitive. With the advance in the study of alloys, it has been found that an alloy of nickel and chromium, called "Nichrome," possesses properties which make it an ideal wire for laboratory resistance furnaces for temperatures up to a maximum of 1100° C. This alloy which melts at about 2800° F. is not easily corroded by ordinary laboratory fumes and has a resistance of about 68 times that of copper. This high resistance makes it possible to use

electricity at 110 to 220 volts with very little, if any, external resistance in the circuit. This renders possible the construction of small laboratory furnaces for temperatures up to 1000° C. or 1100° C., not only at low cost, but with the additional advantage of economical operation because of the utilization of the currents ordinarily at hand. The furnaces most largely

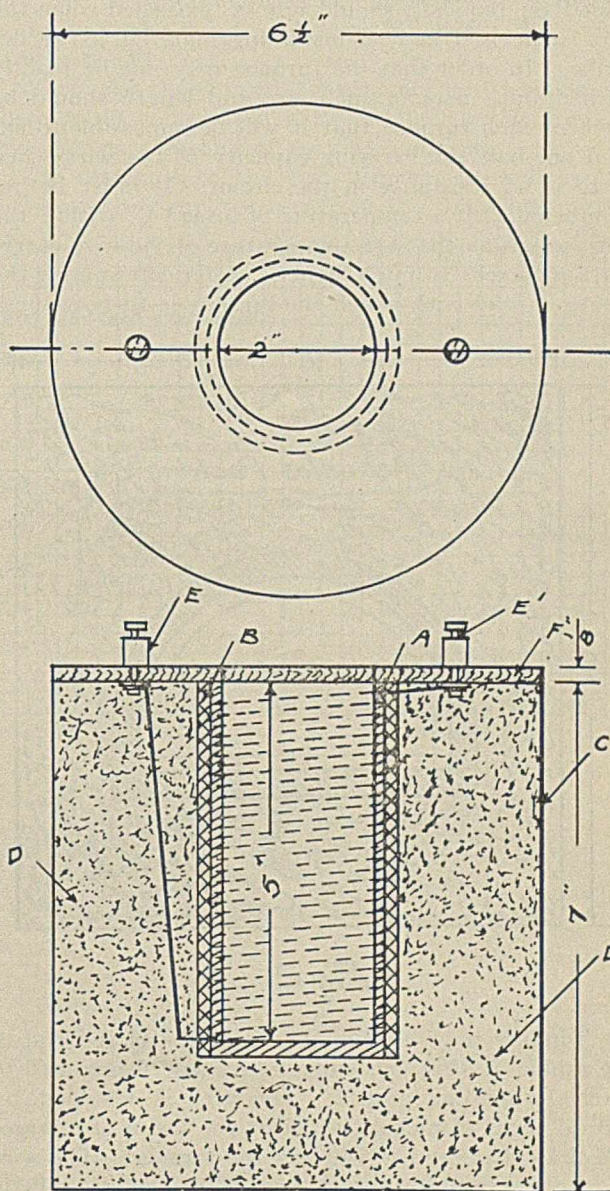


Fig. 1.

employed by us are of two types, but being made in the same general way, a detailed description of one will suffice. Somewhat similar combustion and treating furnaces have been described by Berry.<sup>1</sup> The furnace shown in Fig. 1 has for a foundation a small clay cup, such as is used in batteries. In the case of this furnace, it is 2 1/2" in diameter and 5" long, which size

<sup>1</sup> THIS JOURNAL, 2, No. 6.

is very convenient for individual use. When resting on end it has been used for making fusions and when resting on the side, for ignitions. This furnace can be made to hold four crucibles at a time by inserting a long piece of asbestos board to form a bottom. Around the clay cup A is wound the "Nichrome" wire, the size and length of which may be easily determined, when the size of the clay cup and the voltage of the current to be used are known. One half the carrying capacity of the air-cooled wire, as allowed by the manufacturers in their bulletin (this can be obtained on request), should not be exceeded when the wire is enclosed in heat-insulating material, as we describe. In order that the furnace may not be readily burned out, wire of such size and length should be used for each furnace that it will be impossible to exceed one-half the carrying capacity of the wire when there is no resistance in the circuit. It must be remembered that a temperature of  $1000^{\circ}\text{C}$ . within the furnace means that the temperature of the wire carrying the current is much higher. After determining the length of wire with which the furnace is to be wound,

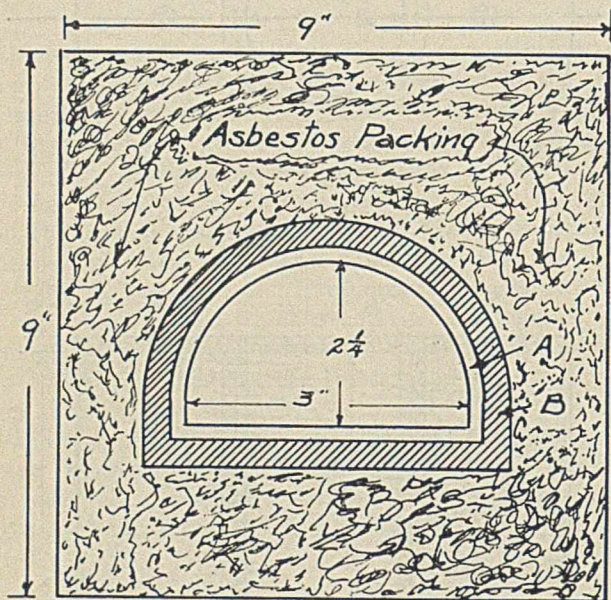


Fig. 2

the winding may be commenced at the closed end of the muffle A (Fig. 1), enough free end being left to reach the binding post E. The wire is fastened by several turns of nickel or "Nichrome" wire of a larger size than that used in winding the furnace. The wire on the outer end of the furnace is wound a little close in order that the temperature may be equalized where radiation is greater. Great care should be used to avoid kinking the wire, as this renders it brittle. When the winding is complete, the end is fastened in the same manner as before, leaving enough free end to reach the binding post E'.

After the winding has been properly executed, the problem of proper insulation and protection from the air is to be considered. The insulation in itself is not difficult, but to find a material which

will insulate, at the same time have no chemical action on the wires and yet protect them from the air at  $1000^{\circ}\text{C}$ ., is more or less of a problem. Of the mixtures which have already been tested, the one consisting of one part kaolin to three parts of ground quartz gives the best results. The kaolin and crushed quartz are mixed to a paste with water and plastered over the wire and cup, care being taken to fill all

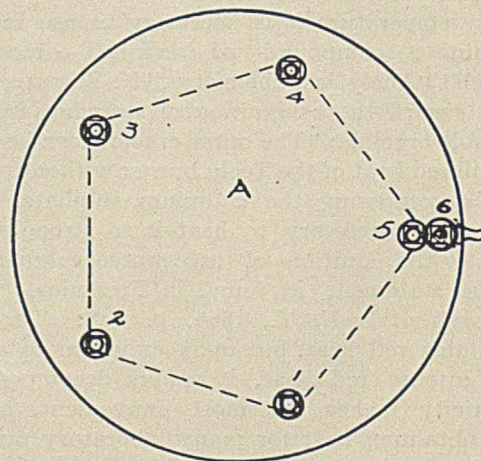
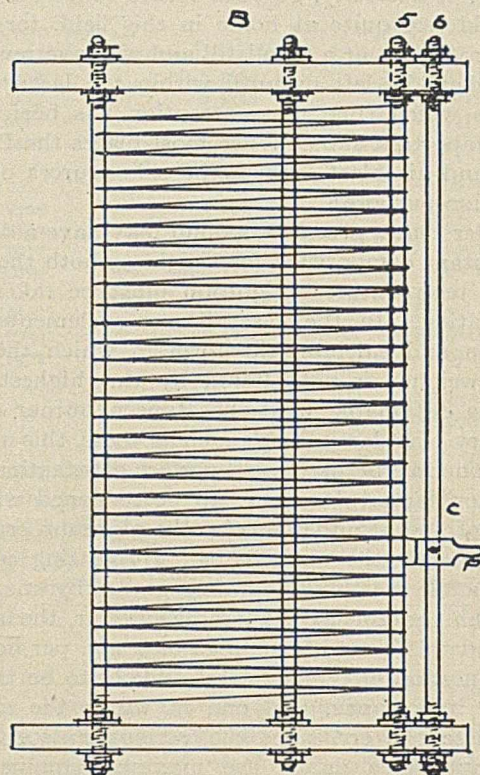


Fig. 3

space between the wires B (Fig. 1). This mixture is plastered on just thick enough to completely cover the wires. (If the coating is too thick it is likely to crack badly.) After drying and baking by means of the current, any imperfections are repaired and the baking repeated. This can be somewhat improved by painting with a dilute solution of water glass.

Now the furnace is ready for packing in the heat



insulating material D. This is done as shown in Fig. 1. The whole is then covered with a heavy piece of asbestos board F, and the binding posts E are placed as shown.

A furnace of the muffle type (Fig. 2) has likewise been constructed on the same general principles and has been found to be equally economical, as well as much more satisfactory when a large number of ignitions are to be made. This furnace is  $10\frac{1}{2}$ " long over all, while the muffle is  $5\frac{1}{2}$ " in length. The furnace is wired with No. 17 B. S. "Nichrome" wire. The same temperatures can be obtained in this furnace as in the one first described, but more care must be taken because of its increased size.

Without external resistance the highest temperature, to which it is safe to heat the furnaces, can be obtained when the furnace door is closed. Furnaces, constructed on the above plan and of the dimensions shown in the figures, as a rule, fulfil these conditions. Never-

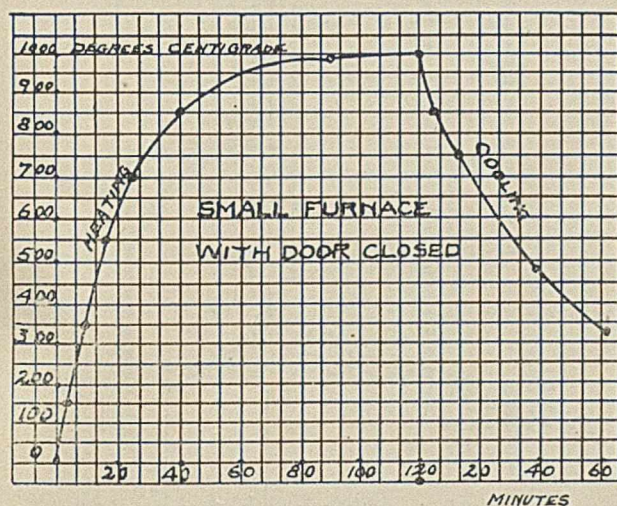


Fig 4

theless the highest temperature obtainable in each furnace should be determined and not allowed to exceed  $1000^{\circ}$  to  $1100^{\circ}$  C., if the life of the furnace is to be considered. In order that a constant temperature less than the maximum may be obtained, it is necessary to have external resistance. A very convenient resistance box, shown in Fig. 3, can be made by using six stove bolts of  $\frac{1}{4}$ " iron, about 12" long, threaded at both ends, each end being provided with two washers and two nuts. The ends of the resistance box may be made of either asbestos board or wood, wood being the more satisfactory for boxes, which are to be used for small currents which produce but small amounts of heat. For boxes which are to be wound with heavier wire and used for large currents, which develop sufficient heat to warp boards, asbestos, although not as stiff, is more satisfactory. The five iron rods, 1 to 5 (Fig. 3), should be wrapped with thin asbestos paper which has been painted with water glass solution. The water glass cements the paper firmly to the iron, so that no trouble will be experienced with its peeling off. Bolt 6 is left bare. When the water glass is dry, the bolts should be fastened in position on the boards as indicated by 1 to 6 in Fig. 3.

The slide contact is made of hard wood, the hole in the center being lined with copper by means of which contact is secured with the sheet of brass or "Nichrome" ribbon. The metal is kept in contact with rod 6, which passes through the metal lined hole, by means of a small screw. The brass or "Nichrome" sheet has enough spring to make a satisfactory contact, when pressed against the wire, where it winds about 5 by rotating around 6. This arrangement is quite satisfactory for currents up to 3 amperes, but for higher currents better contact is necessary and can be obtained by a thumb screw so arranged as to pass through C and press X against the resistance wire (Fig. 6).

When cleanliness and convenience are considered, there is no comparison between the electric furnace and gas as a means of making ignitions.

The first cost of the two furnaces as well as the resistance box can be seen from the following tables:

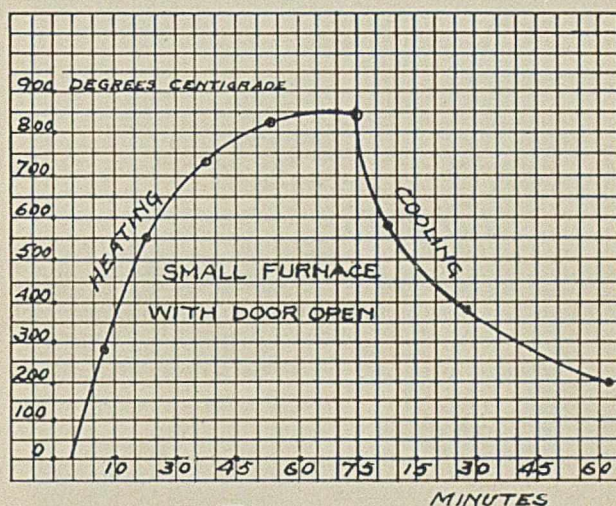


Fig 5

MUFFLE FURNACE.	
Muffle.....	0.50
Wire 65 ft. No. 17 B. S. "Nichrome".....	1.27
Labor at 50 cents an hour.....	1.00
Clay and quartz sand.....	0.10
Heat-insulating material (old pipe covering).....	0.00
	<hr/>
	\$2.87
CRUCIBLE FURNACE.	
Clay cup $2\frac{1}{2}$ " $\times$ $4\frac{3}{4}$ ".....	0.20
Wire No. 20 B. S. 32 ft. "Nichrome".....	0.35
Labor at 50 cents an hour.....	0.75
Clay and quartz sand.....	0.05
	<hr/>
	\$1.35
RESISTANCE BOX.	
Stove bolts.....	1.00
Wire 75 ft. No. 22 B. S. "Nichrome" with a resistance of about 70 ohms and a carrying capacity of about 6 amperes.....	0.80
Labor at 50 cents an hour.....	2.00
	<hr/>
	\$3.80

The cost of operation is as reasonable as the first cost. When used on a 110-volt circuit, with electricity at 10 cents per kw. hour, the muffle furnace, which takes 5 amperes, can be operated at a cost of 5 cents per hour and the crucible furnace, which takes 3 amperes, will cost 3 cents per hour. At the University

of Arizona, where gas is \$1.50 per 1000 cu. ft., it costs about 1.5 cents per hour to run a Bunsen burner. Therefore, when, as is the rule, several ignitions are to be made, the electricity is considerably cheaper than gas since 2 to 4 crucibles can be ignited in the smaller furnace and 4 to 8 in the large. In these electric furnaces, temperatures can also be reached in a porcelain crucible which are rather difficult to obtain by means of a Bunsen burner or blast lamp.

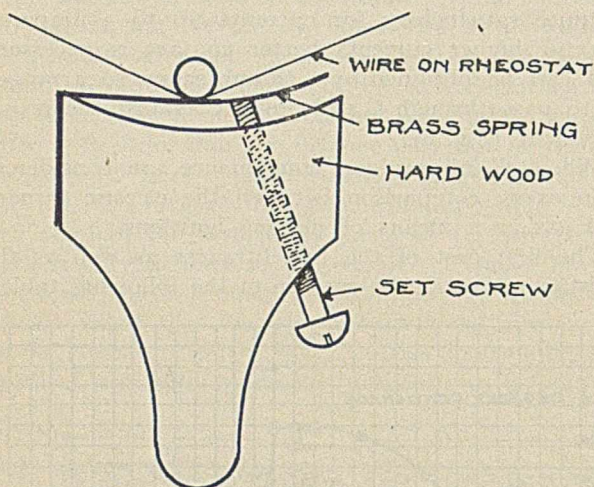


Fig 6

The heating and cooling curves (Figs. 4 and 5), taken with the door closed and open, show the rate at which the furnace will heat up and cool down. With ordinary usage, furnaces, made as described above, have a life of several hundred working hours and when one burns out it is only a matter of the expenditure of a few moments of time to renew the heating element.

UNIVERSITY OF ARIZONA,  
TUCSON.

### A STILL FOR ABSOLUTE ALCOHOL.<sup>1</sup>

By RALPH H. MCKEE.

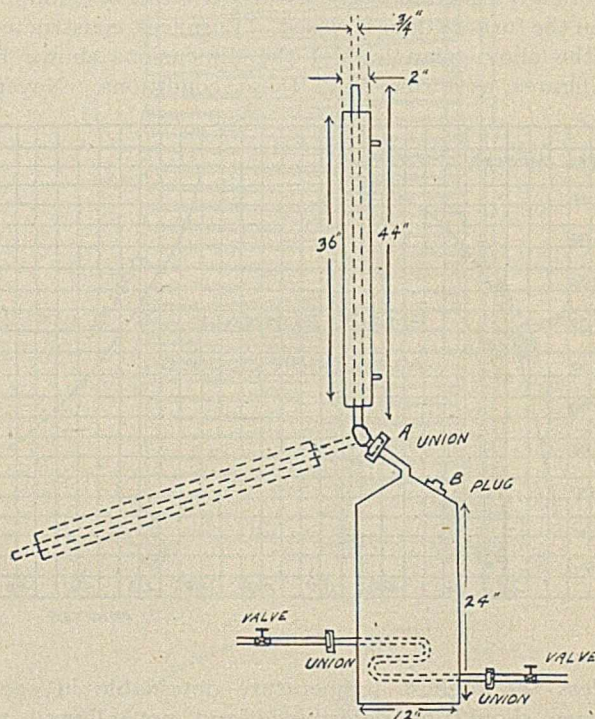
Received October 17, 1911.

In a laboratory, in which considerable organic work is carried on, the expense of absolute alcohol in a year's time is a distinct item. The still shown in the accompanying sketch has proven efficient in the preparation of absolute alcohol from ordinary 94 per cent. alcohol. It is made of sheet copper tinned on one side, with a half-inch bent brass pipe (32 inches within the still. A greater length would probably be an improvement), whose upper end is connected with the main carrying steam at about 40 pounds pressure and the lower end with the same steam trap used for our still for distilled water. An opening, B, four inches in diameter, is used for filling and emptying the still. It is closed by use of the top of a plumber's drum trap such as is commonly used in connection with bath tubs. The still was made

<sup>1</sup> Another form of alcohol still is described by Warren, *J. Am. Chem. Soc.*, 32, 698 (1910).

and installed by a local tinner at a cost of \$21.00, not including the steam trap.

In operation the still is charged with 35 pounds lump lime and 26 liters of ordinary 94 to 95 per cent. alcohol. A current of water is started through the condenser and steam turned on until the alcohol has been heated to boiling and then turned off. This takes about five minutes' time and the dehydration of the alcohol once started evolves sufficient heat to keep the still hot some hours. To insure the completion of the dehydration, the steam is turned on the next morning and the heating with the condenser vertical continued for six or seven hours. The union A is then loosened, the condenser turned down into the nearly horizontal position shown in the sketch,



the union tightened and the distillation and collection of the absolute alcohol begun. At this point it is necessary to wrap some kind of a blanket about the body of the still, otherwise, owing to air cooling, the distillation will be slow and incomplete. To clean, the unions are opened and the still removed to a convenient place and washed out by use of a hose inserted into the four-inch opening B.

The yield is somewhat over sixteen liters of alcohol of slightly better grade (99.8 per cent.) than shown by freshly opened bottles of "absolute alcohol" from two well-known German chemical houses. The cost of the product is about \$0.30 a kilo as compared with the ordinary purchase price of \$1.10 a kilo, both being on the "duty-free" basis granted to educational institutions. Laboratories which cannot avail themselves of "duty-free" straight alcohol may find it worth while to prepare, by this apparatus, water-free alcohol from the ordinary denatured article.

UNIVERSITY OF MAINE,  
ORONO.

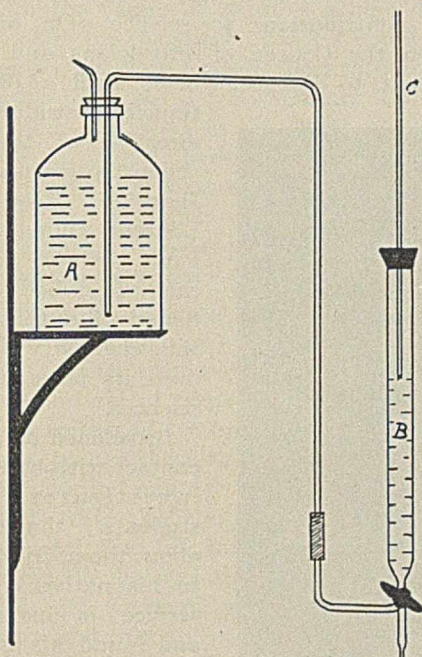
## AN AUTOMATIC PIPETTE.

By T. O. SMITH.

Received November 15, 1911.

Every chemist engaged either in routine work or in research involving a considerable number of determinations has learned to appreciate the value of any automatic or labor-saving device he may find available.

The automatic pipette here described is offered because of its simplicity, accuracy, ready adaptability to any volume required, and especially, because it is easily set up from materials and apparatus found in every laboratory.



The only materials needed are:

A—A bottle of a size adapted to the reagent which it is to contain.

B—A three-way stopcock burette.

C—A fine capillary tube long enough to reach from the zero reading on the burette to the top of the solution in the container.

D—Glass and rubber tubing to set up as shown in the illustration.

By raising or lowering the capillary tube *C*, the pipette may be adjusted to deliver any exact quantity within the limits of the capacity of the burette used.

It is obvious that if the capillary tube is drawn out as fine as possible and the burette and container brought near the same level, the accuracy of the pipette is practically that of the burette.

NEW HAMPSHIRE AGRICULTURAL EXPERIMENT STATION,  
DURHAM.

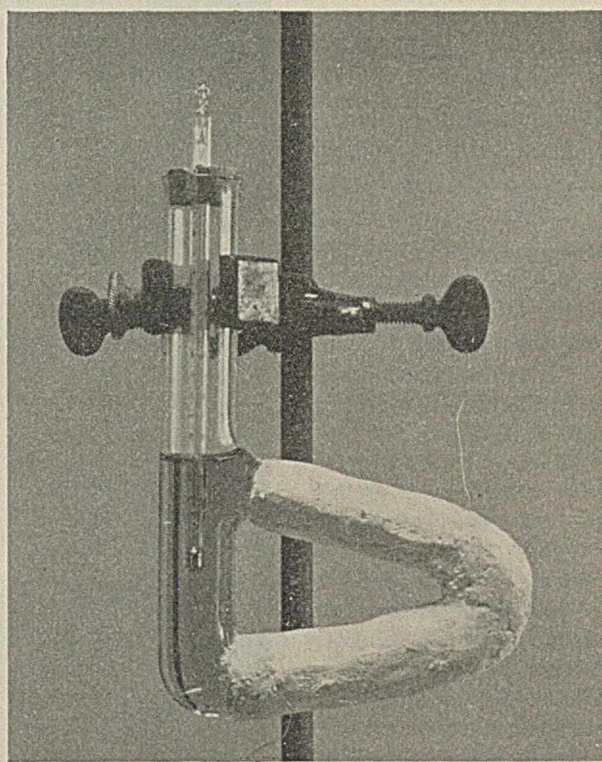
## A NEW MELTING-POINT APPARATUS.

By D. S. PRATT.

Received October 26, 1911.

In the course of some research work with organic compounds, it became necessary to have a melting-

point apparatus more accurate and more easily controlled than those ordinarily used. With all common types, the chief difficulty is in obtaining a uniform rise in temperature, and in duplicating the conditions at another time. The following simple piece of apparatus needs little explanation beyond the illustration. It is based upon the Thiele tube, with electricity supplanting the Bunsen flame, as heating agent. The lower half of the side arm is wound with resistance wire, such as fine manganin or nichrome. The whole arm is then enclosed with asbestos paste as shown in the cut. Using a lamp board, or rheostat, the current sent through the heating coil may be varied at will, and thus the rise of temperature very accurately controlled. By calibrating the instrument in terms of lamps and temperature it is only necessary to turn on the proper number of lamps and the rise of tempera-



ture will be exactly uniform each time it is used. The rate of change may be varied from an almost imperceptible rise, to a very rapid one, and the range is limited only by the fluid used.

In making a series of determinations it is very convenient to turn off, say, one lamp, while preparing the tubes, thus retaining the temperature within a few degrees of the melting point and saving much time usually spent in needless delay. For substances whose melting points vary with the rate at which they are heated, this device is especially convenient. It has been in use for over a year in the organic research laboratories of Cornell University, has given complete satisfaction and has supplanted the older types.

CORNELL UNIVERSITY,  
ITHACA, N. Y.

## ADDRESSES.

### ADDRESS AT THE UNVEILING OF THE BUST OF WOLCOTT GIBBS.<sup>1</sup>

By CHARLES E. MUNROE.

Received November 29, 1911.

[The unveiling of the Bust of Professor Wolcott Gibbs took place in Rumford Hall, Chemists' Building, New York City, on November 25th. The Bust was executed by Ernest Wise Keyser, and it will be appropriately placed in Rumford Hall among likenesses of other distinguished investigators in the field of chemistry.

The presentation address was made by Mr. Alfred Gibbs, representing the relatives and descendants of Professor Gibbs, and a review of his life work and history was given by Professor Charles E. Munroe.—EDITOR.]

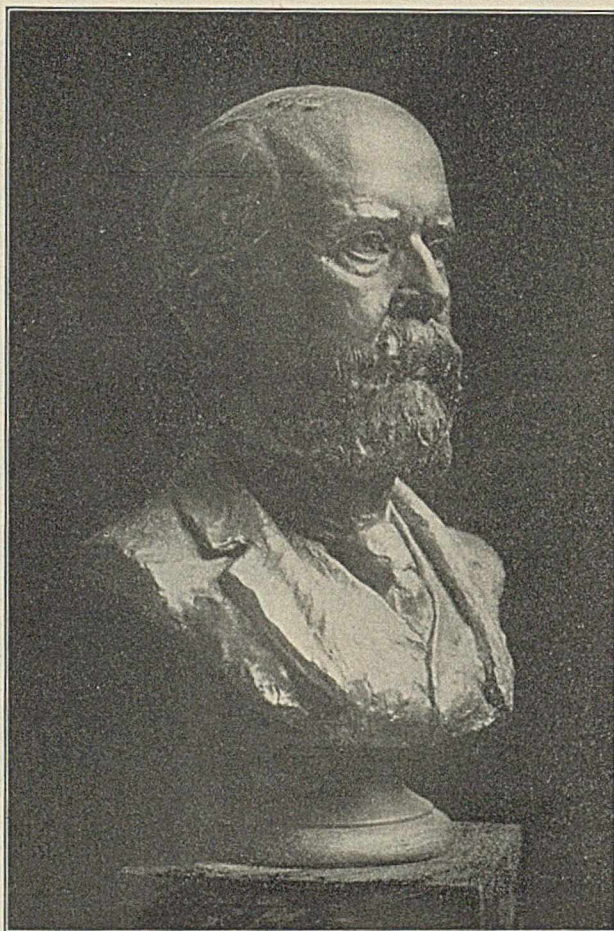
Because of the place of his birth and that where he was educated; because of the profession he chose and which he so highly adorned; because during the greater part of his mature life he applied his splendid talents and broad attainments to the realization of the hopes of the founder of the Royal Institution in his bequests to Harvard College and to the American Academy of Arts and Sciences; and because he was an academician and a club man, it is eminently fitting that the bust of Wolcott Gibbs should be unveiled in the Rumford Hall of the Chemists' Club of the City of New York.

For on February 21, 1822, he was born in this city of New York; in 1841 he received his baccalaureate degree from Columbia College of this city; in 1845 he received the degree of M.D. from the College of Physicians and Surgeons of this city; he chose chemistry as his profession; he was Rumford Professor of Harvard College and Harvard University for 45 years and a member of the Rumford Committee of the American Academy of Arts and Sciences for 30 years; he was founder, member and president of the National Academy of Sciences; he suggested and organized the Union League Club of New York and he promoted and supported other social organizations.

<sup>1</sup> Delivered in Rumford Hall, Chemists' Club, New York, November 25, 1911.

His education was, however, much broader and more comprehensive than that comprised in his satisfaction of the requirements for the degrees awarded him at Columbia College and the College of Physicians and Surgeons, for in the interim between his graduation from the first-named institution and his entrance on the second he served as laboratory assistant to Dr. Robert Hare, Professor of Chemistry in the University of Pennsylvania, then the most eminent chemist America had produced and to-day revered for his splendid contributions to science. On his graduation from the College of Physicians and Surgeons, Gibbs went to Europe where, until 1848, he continued his studies under the direction of the eminent chemists, Rammelsberg, Heinrich Rose, Liebig, Laurent, Dumas and Regnault, whose names are each inscribed upon the honor roll of those to whom the chemistry of to-day owes its place among the sciences.

Broadened by travel, by contact with these eminent investigators, and the students that gathered about them, Gibbs returned to his native country for service in his profession, and found his first opportunity in the delivery of a short course of lectures in a minor institution in Delaware, but very shortly after, in 1849, he was appointed professor of chemistry in the Free Academy, now the College of the City of New York, where he remained until 1863 when he accepted the Rumford professorship, requiring service in chemistry, in the Scientific School of Har-



vard College.

His term of service in New York was distinguished, for, while his duty to the college demanded only that he teach its students the elements of chemistry, he began in 1851 as associate editor of the *American Journal of Science*, the preparation of abstracts of foreign literature in chemistry, and he engaged in research with the result that in 1857, there was given to the world the first memoir on a notable and systematic research in chemistry from America when the Smithsonian Institution published the memoir of Gibbs and Genth on "The Ammonio-Cobalt Bases" which has ever since served as a model for the pres-

entation of results by investigators in chemistry. In 1861 his independent paper on the platinum metals appeared and, as Clarke says, "firmly established his reputation."

Gibbs was in New York when our Civil War came on and devoted as he was to his profession he was also a patriot. It will be recalled that in broadly fitting himself for his profession he had at the College of Physicians and Surgeons pursued that branch of applied chemistry styled medicine and qualified in it, hence he could serve his country best and most by the exercise of his special knowledge and attainments. When men are rushing to expose themselves as targets for the enemy it requires a high degree of courage to offer on the altar of one's country one's special talents in service outside the firing line. But this Gibbs did and the U. S. Sanitary Commission stands to-day as an epoch in the civilization of man. By its work it proved, perhaps, to be the greatest good for mankind that was realized from that dreadful period of labor in which a great nation was born. It has been a model for other nations that have subsequently, unfortunately, been engaged in war.

Not content with applying his acquired knowledge, especially in that branch of applied chemistry known as medicine, to the amelioration of the "horrors of war" and to the aid of those that conquered, Gibbs sought to organize and crystallize opinion and effort by bringing together those of influence in New York who favored active military operations against the seceders and thus the Union League Club, which met, to organize, in his home, was formed. And throughout his life he was an organizer, or member, of bodies of men through which, by investigation, consideration, and discussion, issues of moment in science were carefully wrought out, while social relations were conserved and promoted.

He was born and reared under conditions that could have bred an aristocrat. His father was in affluent circumstances. His progenitors had served their country and mankind in positions of importance. He inherited a competency. His associations from earliest youth were with the cultivated, intellectual and forceful. He was in person impressive and engaging. He was in taste and dress discriminating; but he was in his dealings with and estimates of man democratic.

As a prospective student I met him in 1868 and he looked to me god-like. It was my good fortune not only to be received as a student by him but later to become his assistant, and through other fortunate circumstances, such as being ordered to duty in Newport, where he resided after his retirement, to keep in contact with him quite up to the time of his death. I recall most vividly my first meeting with him for he embodied in the flesh all that I had ever imagined of man and though my relations with him were afterwards quite close this feeling and belief persisted and remains. He was above the average man in height, and his body was symmetrically developed with his stature so that he walked and moved with natural gracefulness. His head was

admirably proportioned and was covered with a splendid mass of curling black hair which matched the beard that covered his face. In clothing and person he was always decently fastidious, but ever the attracting features were his eyes, which were deep brown in color, lustrous and luminous; and his voice, which was full and rich, with a deliciously attractive and convincing overtone.

He gave the impression of mildness and fairness and continued association confirmed this first impression. Never have I met one who so avoided definitely judging his fellowman or who when forced to do so judged him more fairly and without prejudice, for his mind was filled with the contemplation of nature in a large way and of its processes, and he was endeavoring constantly to comprehend them and to record the results of his observations and tests for the benefit and use of mankind. He regarded his fellowman in the same broad and tolerant manner. In fact the definite impression of him which one received by close contact with him was largeness of vision; breadth of view; tolerance of differences in opinions, methods or manners; and sympathy, in a broad way, for mankind; and that he approached every issue, scientific or social, without prejudice, and with an entirely open mind.

If, in my attempt to portray Dr. Wolcott Gibbs from the image that abides with me, I have conveyed to you the impression that, through timidity or indolence, he sought to avoid strife, let me hasten to immediately correct this erroneous impression, for on the contrary he was intensely human and he met his troubles in a thoroughly human way but even then on a high plane.

Permit me to illustrate by an anecdote or two. Self-government by students is regarded in this country as a very modern and novel development. Dr. Gibbs introduced it at the outset of his coming to Cambridge. I do not know that he had not previously done so in the College of the City of New York. Eventually in my career as a student in his laboratory I succeeded to the first place in the governing body and I wore the resounding title of Chief of Police. During my administration a rebellion arose. The orders I gave were not obeyed and the fines I assessed were not paid. Having exhausted all the resources of authority at my command and the disorder having become a menace to all earnest students, after due warning, I resorted to the unheard-of expedient of reporting the recalcitrants to Dr. Gibbs. They were much amused when they were directed to report to this mild-mannered, sweet-tempered gentleman. I was not present at the interview. I never knew what occurred at that interview. The students never told me and Dr. Gibbs never referred to it. But what I do know is that when these students returned they said: "Munroe, you may order us to do what you wish; you may assess such fines as you please; but never again direct us to report to Dr. Gibbs;" and from that day until I left the laboratory discipline was complete.

Strange as it may seem Dr. Gibbs became, on com-

ing to Harvard, a storm center. President Hill called him because he had a vacant chair in chemistry to fill and he found in Gibbs the most eminent chemist in America. Gibbs accepted the position at Harvard because it seemed to offer the largest opportunity for usefulness in the field for which he was especially equipped. But his appointment thwarted the realization of the ambitions of others; it became a cause of dissension and the arraying of groups of men against each other. The situation had become acute as I entered upon the scene. In the regular performance of my duties I was unwittingly forced to know of it though then I knew not the reason for it or the extent of it. I was especially embarrassed to come upon Professors Gibbs and Cooke, when they were engaged in a gentlemanly, but very personal, altercation.

Unknowingly to me, out of this came my opportunity. While holding the position of private assistant to Dr. Gibbs I was appointed Assistant in Chemistry in the College under Professor Cooke. With the courtesy that prevails among gentlemen all these arrangements were ostensibly in the hands of Dr. Gibbs and it was from him that I received my instructions to make that visit to President Eliot at which I received notification of my appointment to the college. Naturally and most properly I reported to Dr. Gibbs that I had obeyed his instructions, and the results of so doing, and I can never forget his admonition. Knowing my loyalty to him, knowing that inadvertently I had become somewhat acquainted with the distressing situation he said: "Mr. Munroe I have been deposed and you are appointed to take my place. You know that my relations with Professor Cooke have not been entirely amicable, yet let me say that you can serve me best by serving him with entire devotion." Thus spoke the man in Wolcott Gibbs.

Were there time I should like to describe the laboratory at the Lawrence Scientific School and the manner in which it was directed by Dr. Gibbs. Fortunately this has been well recorded by Professor F. W. Clarke in his memorial Lecture before the Chemical Society of Great Britain and by Stephen Paschall Sharples in his description of the Lawrence Scientific School to the Cambridge Historical Society. I may say that were it to be investigated by an agent of the Carnegie Foundation, armed with a pad and pencil, it must have been condemned. I must further say that after completing my fortieth consecutive year of university teaching I should, if put under oath, state that, measured by pedagogical standards, it was unsound. But I must add that the results produced were splendid and that the students that survived the process went forth finely equipped to pursue their chosen professions. Dr. Gibbs' visits to us were infrequent but the impression he made in these conferences were such that he was an ever-living presence and a constantly present example. Mendenhall's remark that a student would prefer to be neglected by Rowland to being taught by another embodies the, thought I desire to convey concerning the re-

lation of Wolcott Gibbs as a teacher to his students. The pedagogue trains his pupils as the military sergeant drills the cowherd. But the educator educes from his student his best capacities in the line of his endeavor. He brings the within without. He reveals to the student the latter's own capacities. He preserves to the community that precious gift, individuality, but arouses, and enlivens, and controls it so that it may best serve the community in which that individual may be placed. It is impossible to formulate the manner in which this may be accomplished for the possibilities vary with each student to be taught and with him who teaches, and the teachers who comprehend this are rare, but such was Wolcott Gibbs.

It is said of Gibbs that he was not a "popular lecturer." I may say that this was most unfortunate for the populace. It has been my privilege to listen to a large number of those public speakers who have commended themselves to the public. As a youth I reported at length, for the newspapers, the lectures of Tyndall and Proctor. I served as demonstrator for Professor Cooke in that charming course of lectures at the Lowell Institute which appeared as "The New Chemistry." I sat at the feet of Edward Everett, Henry Ward Beecher, Wendell Phillips and Emerson. I was enthralled by Julia Ward Howe and Mary A. Livermore. Dr. Gibbs gave us but few lectures, but those were enriched by such a wealth of knowledge, graced with such diction, planned in so thoroughly logical and systematic a manner, and presented with such charming simplicity as to ever remain as almost unapproachable models.

For Dr. Gibbs was ever true to his best capacity (his proper sphere of usefulness to his fellows); namely research, and he continued this long after his retirement from the field of teaching. As one reviews his achievements in research one is amazed at the catholicity of his accomplishments. All recognize his numerous contributions to analytical chemistry; his application of the electric current to quantitative determinations being especially well known; but he covered the field from gravimetric, through volumetric, to gas analysis. It is also pretty generally known that his early investigations of the complex ammonium bases, and their compounds, were in his later life supplemented by researches into the constitutions of the complex inorganic acids. Organic chemistry claimed his attention. In 1853 he prepared an arsenical derivative of valeric acid. In 1868 he discussed the constitution of uric acid and its derivatives. In 1869 he described some products formed by the action of alkali nitrites upon them. In 1891 and 1892, with H. A. Hare and E. T. Reichert, he treated of the physiological action of definitely related chemical compounds. He produced memoirs on a normal map of the solar spectrum and on the wave lengths of the elementary spectral lines, and, in the study of interference phenomena, he discovered a constant, which he styled the interferential constant. The time allotted me is too brief to enable me to set forth the work of an investigator who at the age of

18 published a paper entitled a "Description of a New Form of Magneto-Electric Machine, and an Account of a Carbon Battery of Considerable Energy" and at 71 years of age published a method for the separation of the rare earths, further than to say that while Gibbs was an experimentalist rather than a theorist he published views on theoretical chemistry that have force to-day.

By virtue of his sympathy and breadth he became a pioneer in comprehending, assimilating and expounding the results of others, giving them always full credit. He was the first American to adopt and promulgate the conclusions of Cannizzario; so early as 1880 he appreciated the value of the researches of J. Willard Gibbs and was the prime factor in having the Rumford medal conferred on this immortal Yale physicist. I well remember his enthusiasm in those early days when speaking of the recently published, and now classic, memoir by Kekulé.

I fear the privilege you have afforded me to speak of my beloved master has tempted me to overstay the time allotted me and yet I feel I have but inadequately set forth the man and his achievements. In closing, permit me to quote from the admirable tribute paid him by Theodore W. Richards.

"The circumstances of his early academic life brought him in contact with but few students. This is the more to be regretted because his enthusiastic spirit, his tireless energy, his recognition of everything good, and best of all his warm human friendship which endeared him to all who knew him. Those who were thus fortunate, whether students or colleagues, will always devotedly treasure his memory; and his place as a pioneer in science in America will always be secure."

#### WOLCOTT GIBBS.<sup>1</sup>

Although I am unable to be present at the unveiling of the bust of Dr. Gibbs, I cannot let the occasion pass without some expression of my admiration for him; and in thinking the matter over, I have been struck with the contrast between his work and that of some of his followers in the study of the metal ammonia compounds, who seem to think that the main object of a research consists in providing oneself with a sufficiently flexible theory, and then fitting graphic formulas of the compounds to it with little or no attempt at experimental proof. Such researches, in which the pen is mightier than the test tube, had no attraction for Wolcott Gibbs. With him the test tube was supreme—in fact not only were his racks always full, but on his desk were many large beakers crowded with test tubes, each with a label sticking out of its mouth; and I remember few things so delightful and illuminating as seeing him flitting from one to another, dilating on the beauty of the crystals or precipitate, and pointing out its relation to past work and promise for the future.

In his obituary of van't Hoff, Emil Fischer said most good chemists follow their noses in research. This was eminently true of Wolcott Gibbs, and his scent

was fine enough to guide him unerringly through the most intricate trails, for the hardest subjects had a special attraction for him.

Few chemists have been his equal in the power of appreciating the suggestiveness of observations, of contriving new and varied conditions for experiment, and in shaping imagination.

His work, however, was exclusively experimental. In fact some of us wished that he would have drawn more inferences from his discoveries, but I think that he considered that his real function was that of the experimental pioneer; and certainly he converted many a tangled almost impenetrable thicket into a pleasant grassy path between well-trimmed shrubberies.

CHARLES LORING JACKSON.

HARVARD UNIVERSITY,  
November 22, 1911.

#### CHEMICAL INDUSTRIES IN SWEDEN.

By THOMAS H. NORTON.

Sweden is a land in which chemistry has played an important rôle from a comparative early date. Scheele and Berzelius were among the most noted pioneers in the formative period of the science, and no less than 20 of the known chemical elements have been discovered by Swedes. The Kingdom owes to three factors its past and present position in technical chemistry. The first is a fairly abundant and diversified mineral wealth. Swedish iron and copper once dominated the world's markets. While no longer leading in quantity, Sweden still occupies a foremost position with regard to the quality of its metal products, and the high technical standard of its mining and metallurgical equipment.

A second feature is the enormous extent of the country's forests. Once contributing the necessary charcoal for its choice product of iron, they now furnish the raw material for an enormous industry—the manufacture of cellulose in its varied forms.

The third factor is Sweden's abundant water power. The coal deposits of the land are unfortunately limited and of inferior quality. The annual output is 305,000 long tons. In earlier days the forests supplied fuel. Now that timber possesses a greater value, and the supply is more limited, it has been necessary to import coal from abroad to the extent of 3,000,000 tons annually.

With the advent of electric power and its economical transmission it has been possible to utilize the waterfalls on a most extensive scale. Electrotechnical industries have promptly availed themselves of this opportunity. The total available water power of the Kingdom is estimated at over 4,000,000 horse power. About one-ninth has thus far been harnessed for industrial purposes. Each year at present there is an additional increase of over 100,000 horse power. In 1911 it is estimated that this increase will be about 115,000 horse power. Owing largely to this rapid exploitation of her vast water power, Sweden is changing from a producer of raw materials to a predominantly

<sup>1</sup> Letter from Professor Jackson. Read at Unveiling of Gibbs' Bust.

<sup>1</sup> Special report to U. S. Consular Service.

manufacturing land. Modern industrialism is rapidly invading every field of national activity.

The chemical industry of the country is confined chiefly to inorganic branches. Apart from the wood pulp, and the products of the electrochemical branches, it is in most cases inadequate to meet the demands of the home market of a population of 5,500,000. It is, however, expanding steadily, more rapidly than the growth of population.

According to the census of 1900 there were 256 chemical factories, employing 2,644 workmen, with annual products valued at \$4,095,000. To these should be added 187 factories producing oils, tar, etc., with 2,434 workmen, and an output of \$5,650,000, and the match industry with 20 factories, 6,102 workmen, and products valued at \$2,665,000. Paper and allied branches were credited with 159 factories, 18,490 workmen, and an output of \$8,130,000. The metallurgical industries give employment to 16,876 workmen, and the products are valued at \$49,050,000. For purposes of comparison the statistics of other leading industries are here given: Mining, 13,187 workmen, output \$5,430,000; metals and machines, 56,222 workmen, output \$44,840,000; textiles, 37,187 workmen, output \$36,100,000.

The 256 establishments classified by the census as chemical works are subdivided as follows:

Character of products.	Factories.	Workmen.	Value of products.
Inorganic acids, bases, and salts	32	291	\$ 683,000
Fertilizers.....	72	921	1,402,000
Explosives.....	19	664	897,000
Colors and miscellaneous.....	133	768	1,113,000

For its supplies of most heavy chemicals Sweden is still to a great degree dependent upon other European countries.

Sulphuric acid is made largely in connection with the manufacture of superphosphate. In 7 factories, with 39 workmen, there was an annual output of 35,108 metric tons (metric ton = 2,204.6 pounds). Twenty years before the output was but 4,000 tons. The output nearly covers the domestic demand. Pyrites is used chiefly in the works, and is imported from Norway and Spain. There is also a considerable consumption of Sicilian sulphur.

There is but one factory for caustic soda and bleaching-powder. Both articles are imported chiefly, as is also the case with soda and its various by-products and derivatives. In the list of salts, however, there is one notable exception in the case of potassium chlorate. This compound, so important in the domestic match industry and in the manufacture of explosives, is now one of the leading staple products of Sweden and is extensively exported. The electrolytic process by which it is produced was devised in 1893 by Director O. Carlson, who started the first electrochemical establishment in Sweden. In 1900 the two factories employed 215 workmen, and the chlorate produced was valued at \$214,000. In 1909 imports of chlorate had sunk to 207 tons, and the Swedish factories were not only meeting the home demand but exporting annually 1,400 tons to other countries.

The industry uses potassium chloride from Stassfurt as its raw material.

Carlson's method is an advance upon preceding processes for the electrolysis of alkali chlorides, as he does not use diaphragms in the electrolytic bath and maintains a continuous operation. Cathodes of iron are employed, but the anodes, which were originally of carbon, are now of platinum-iridium gauze. Formerly the baths were kept slightly alkaline by additions of caustic potash or lime. Now they are kept slightly acid, as free hypochlorous acid favors the formation of chlorate. Small amounts of potassium bichromate are also added to prevent the reduction on the cathode of the potassium hypochlorite primarily formed. The process is noteworthy as the sole important instance where an electrolytic method has proved to be economically superior to an old, established process of chemical manufacture and has effectually displaced it. In Germany only one factory has tried to maintain its manufacture of potassium chlorate by the old process, using Deacon Chlorine. In England the old process is likewise being gradually abandoned.

#### FERTILIZERS.

Agriculture is highly developed in Sweden, one-half of the population being engaged in tilling the soil, and the demand for artificial fertilizers is extensive. Although there are large amounts of apatite in the country, it is impossible to compete with the raw phosphate brought from Florida, Algiers, and the Pacific. About 29,000 tons of Chile saltpeter are imported annually. Thomas slag is worked up very advantageously, as some of the Swedish iron ores are very rich in apatite and yield with the basic Bessemer process a very rich slag. Three factories grind this slag, producing 10,000 tons annually. As the demand for Thomas iron is limited, the production will not increase materially.

A few years ago Prof. Wiborgh devised a method for utilizing the phosphoric acid in the Swedish iron ores, which carry especially large amounts of apatite. These ores are finely ground and the resultant powder submitted to the action of powerful magnets. By this means the apatite is largely freed from the iron oxide. It is then fused with soda and the phosphoric acid is brought into the citrate-soluble form of tetracalcium phosphate. The product compares favorably with Thomas slag and even superphosphate in fertilizing efficiency. A factory was started at Svartön to manufacture the so-called Wiborgh phosphate, but after a short period the works were closed.

There are numerous factories for producing bone meal, as well as bone fat and glue and animal charcoal, and a few establishments for making poudrette. Herring guano is a staple article. At Gothenburg and its vicinity there are 17 factories engaged in extracting herring oil and preparing the residual press cakes for fertilizer. The annual product is valued at \$285,000, of which \$208,000 is represented by the



guano. This industry depends on the success of the annual herring catch.

In 1900 Sweden consumed 253,132 metric tons of artificial fertilizers, of which one-half were produced at home. In the course of a decade the industry has developed so rapidly that there is now a surplus of superphosphate available for export, amounting to 17,000 tons in 1909.

#### EXPLOSIVES.

The manufacture of explosives constitutes one of the leading industries of Sweden, largely owing to the genius and inventive ability of Nobel. In addition to dynamite his other leading invention, ballistite, a smokeless gunpowder consisting of a gelatinized mixture of nitroglycerin and guncotton, is manufactured. Originally the Nobel Co. prepared this powder from equal parts of the two constituents. It is now made from one part nitroglycerin and three parts guncotton. A small amount of diphenylamine is often added. Gelatinization is effected by means of a mixture of alcohol and ether (2 to 1).

Another distinctively Swedish explosive, Lamm's bellite, is manufactured to a limited extent. Its base is dinitrobenzene, and it belongs to the class of picrate powders. The growth of this branch of manufacture in Sweden has been remarkable. In 1870 there was one factory, employing 40 workmen, with an annual output of \$34,000. In 1900 there were 11 factories, with 394 workmen, and an output of \$724,000.

#### COLORING MATTERS—CARBIDE AND VANADIUM SALTS.

Small establishments manufacturing coloring matters produce pigments of domestic materials, lamp-black, ochers, zinc white, white lead, and cobalt blue, and prepare also, for use in solution or composition, the different organic dyestuffs, imported chiefly from Germany. There are 46 factories, employing 130 workmen, producing wares of an annual value of \$234,000.

Acetylene is widely used in Sweden, and the carbide industry was founded almost as early as in the United States. Two factories, one at Alby and the other at Trollhattan, with a total of 5,000 horse power, produce not only enough for the needs of the domestic market, but also export annually about 1,600 tons. The actual cost of production is given as 3 cents per pound.

Sweden has in the past supplied the world's demand for vanadium compounds, vanadium chloride for the aniline-black dyeing, and ammonium vanadate for use in steel-making. The exportation is now much less than formerly, for aniline black and copper sulphate and sulphide are more in use now as catalytic agents.

#### MATCHES.

The match industry is one of the most important in the Kingdom and has grown rapidly. Twenty factories produced matches valued at \$3,422,000 in 1909. Nine-tenths of the total product is exported. These Swedish matches are encountered all over the

world, despite a very keen competition. The export interests complain of the high protective duties in most countries. In France, Spain, and Greece matches are a Government monopoly.

The Swedish factory equipment contains many devices for replacing manual labor. Lagerman's "complete machine," introduced in 1892 at the Jonkoping works, is one of the most ingenious labor-saving devices known. The match splints, already cut into the proper form by other appliances, are fed regularly into one end of this machine and emerge at the other end a finished product, packed in boxes ready for shipment. One such machine produces hourly 3,600 boxes of matches.

Aspen wood is used almost exclusively in the match industry. It is easy to cut and porous enough to admit easily of impregnation with sulphur or paraffin. The domestic supply of aspen of good quality is now seriously reduced, and much is imported from Finland and Russia. Most of the chemicals used—phosphorus, antimony, sulphur, and paraffin—are imported. This was also formerly the case with potassium chlorate until the recent establishment of the Swedish electrolytic manufacture. In 1901 the Swedish Government forbade the sale in the Kingdom of matches made with yellow phosphorus. At present the manufacture of the old-fashioned matches is much less than formerly, although the Jonkoping works produce a considerable quantity in which yellow phosphorus is replaced by innocuous phosphorus sesquisulphide. In manufacturing the standard safety matches of Sweden now the splints are first soaked in paraffin, or in a concentrated solution of ammonium phosphate. The ignition mass for the heads consists of potassium chlorate, mixed with sulphur or antimony sulphide, gum, or dextrin in solution, and small amounts of powdered glass and ferric oxide. Potassium dichromate and manganese dioxide are occasionally added as oxidizers. The friction surface on the boxes is coated with a mixture of red phosphorus, antimony sulphide, and dextrin solution.

The largest factories in the Kingdom are the two at Jonkoping and the "Vulcan" at Tidaholm. Under their lead nearly all the other manufacturers have combined to prevent undue competition and facilitate the export trade. This latter is carried on largely through Hamburg and London, which serve as distributing points for much of the world's trade in matches. There has been a rapid shrinkage of the export of Swedish matches to Germany, partly as a result of recent legislation. In 1909 the export was 1,655 tons, and in 1910 but 44 tons.

#### WOOD PULP.

The manufacture of cellulose, or wood pulp, is another highly developed Swedish industry, based also primarily upon its forest wealth. The factory for grinding wood, established at Trollhattan in 1857, was the pioneer of the pulp industry. The preparation of chemical wood pulp was introduced in 1871, the soda process being used. In 1874 came the sulphite process, Francke using calcium bisulphite, and

Ekman the magnesium compound. Both of these methods are now utilized, but that of Ekman is used to a less extent on account of its greater cost; the product is, however, of a remarkable purity and gloss, and it is preferred as raw material for the finer qualities of paper. Swedish wood pulp ranks high, as the wood employed—*aspen* and *spruce*—is freer from resin than that available in most countries. The pulp factories of Sweden number 122, and in 1900 employed 6,910 operatives and produced 417,000 metric tons of pulp.

The exports of pulp reached 564,000 tons in 1909, as against 229,000 tons in 1900, and consisted of 372,000 tons of dry chemical pulp, 31,000 tons of wet chemical pulp, 46,000 tons of dry mechanical pulp, and 115,000 tons of wet mechanical pulp. The total value of the exports was \$15,372,000. Great Britain takes about 60 per cent. of the export, other heavy purchasers being France, Norway, Germany, Belgium, Denmark, and, recently, the United States.

There has been a serious overproduction of wood pulp during the past three years and prices have been much disorganized. Recently the wood-pulp associations of Sweden and Norway have agreed upon a systematic limitation of the production of mechanical pulp for 1911 and 1912. The prospective reduction during the period ending January 1, 1913, is fixed at 150,000 tons. No restriction is placed on the output of chemical pulp. As a result of this agreement prices of mechanical pulp have already been materially advanced.

#### PAPER.

The manufacture of paper is naturally closely allied with that of cellulose. In 1909 Sweden had 66 paper mills, employing 7,725 operatives, the production amounting to 250,000 tons, of which 137,510 tons, valued at \$7,346,000, were exported. The ratio of increase is about the same as that of cellulose. Of the paper exported, 80 per cent. goes to Great Britain and is used chiefly for newspapers.

Swedish filter paper for chemical purposes has maintained a world-wide reputation for a century. For a long period it was regarded as indispensable for the needs of the analyst, and the manufacturers enjoyed a practical monopoly. Rivals in other countries, especially in Germany, now compete in furnishing a filter paper carefully freed by treatment with hydrochloric and hydrofluoric acids from mineral matter. The remarkable success attained in earlier years by the largest Swedish firm was due to the use of the purest water available for manufacturing purposes and the choice of exceptionally pure raw material. The peculiar properties of the paper were largely secured by freezing it while in a moist condition. The formation of ice crystals rendered the feltlike mass sufficiently porous for the purposes of rapid filtration. This Swedish firm manufactures now a very choice grade of writing paper, made by simply dipping its filter paper into sizing.

#### ALCOHOL—NEW SOURCE OF SUPPLY.

Another industry, that of the manufacture of alco-

hol, is now linked with the great cellulose interests. The customary methods of distillation are well developed in the country. There were in 1900 131 distilleries, producing annually 450,000 hectoliters (11,887,650 gallons) of spirits containing on an average 50 per cent. of alcohol. Much of this is for use as a beverage. Of the total amount 62 per cent. is manufactured from potatoes, 35 per cent. from maize and grain, and 3 per cent. from beets and molasses. There were 429 distilleries in 1875 and only 131 at the close of the century.

The chemists of Sweden have experimented in a variety of ways to solve the problem of manufacturing alcohol from wood and thus find an additional source of revenue from the country's forest wealth. In the production of wood pulp by the sulphite method there is a waste product that the chemists have finally been able to utilize with profit in the production of alcohol. The profitable utilization of the exhaust lyes drawn off from sulphite boilers has always been a problem. For every ton of cellulose made by this process there are 10 tons of these lyes. They contain one-half of the weight of the wood originally introduced into the boilers. Among the matters present are dextrose and various other sugars, xylose, acetic acid, tannic acid, nitrogen compounds, resins, etc., as well as the calcium lignin-sulphonate, the chief product of the reaction. Of the sugars present most are susceptible of fermentation. They constitute about 1 per cent. of the lyes. The treatment of the liquid is comparatively simple. It is first neutralized with calcium carbonate, after which yeast is introduced. When the fermentation is complete the alcohol produced is secured in the ordinary manner. The process yields 6 gallons of alcohol (100 per cent.) for 1,000 gallons of lye. About 14 gallons are secured for every short ton of cellulose.

As may be imagined, methyl alcohol and other volatile compounds are contained in this crude alcohol. It is sold and used as denatured alcohol for heating and technical purposes. The excise rates in Sweden have been adjusted so as to aid this new industry. If the process were extended to all Swedish sulphite works, the annual output would be about 3,500,000 gallons. It is doubtful whether a sufficient demand exists for this grade of alcohol to furnish a home market for such an amount. German chemists are skeptical as to the possibility of producing the alcohol economically under existing conditions in the Empire, where, on the basis of the Swedish results, over 8,000,000 gallons could be produced annually.

In the attempts to manufacture alcohol directly from wood, investigation so far has been directed to the simple reaction involved in combining one molecule of water with one molecule of cellulose, and thereby producing dextrose, from which alcohol is obtained by fermentation. Chemically, by treatment with sulphuric acid under pressure, this change can be readily effected. From ordinary wood, alcohol amounting to 5 or 6 per cent. of the original weight can be thus secured. Pure cellulose yields 15 per cent. The problem is essen-

tially of an economic nature. It is claimed in Sweden that it has been solved, and that a method has been elaborated for utilizing the waste of the sawmills. Details are not given.

#### DISTILLATION OF WOOD.

The distillation of wood developed at an early day in Sweden, as was to be expected in a land so rich in forests. Swedish tar was exported to every land and given the preference for coating vessels and their rigging, walls, roofs, and roofing paper. It continues to enjoy its good repute, but exports are less in quantity than was the case half a century ago. There has been renewed activity during the past decade. The foreign trade in this article shows the fluctuations: 1875, imports 1,700 and exports 8,300 metric tons; 1900, imports 1,700 and exports 3,600 metric tons; 1909, imports 200 and exports 7,614 metric tons.

The industry is now centered in the northern section of the Kingdom and shipments take place chiefly from the port of Umea. Much of the tar is produced in a small way in old-fashioned tar hollows by individuals or families. There are some more modern establishments, where iron retorts are installed. The census of 1900 reported products from such works valued at \$58,000. As the exports alone of wood tar had a value of \$135,000, it is evident that the bulk of the output comes from small producers. There are now about 25 firms engaged in the business, most of them carrying it on in connection with extensive lumbering and wood-pulp interests.

The Skogens Kolaktieförlag, at Kilafors, is the leading company devoted exclusively to wood distillation. It uses annually for this purpose over 100,000 cubic meters of pine wood (cubic meter = 35.314 cubic feet). A good example of the diversity of interests carried on by a Swedish company is seen in the case of the Stora Kopparbergs Bergslags Aktieförlag, of Falun. This company is said to be the oldest industrial corporation in the world, having received a royal charter in 1347 after a previous existence of over a century. For two centuries, prior to 1750, it was the chief producer of copper in the entire world. Since then, as the copper supply diminished, it has devoted itself to iron, and is to-day the leading producer of iron and steel in Sweden, owning the largest iron works in northern Europe. During the past century it gradually increased its land-holdings until it now owns 750,000 acres of forest and has become the largest exporter of forest products in the Kingdom. At Skutskar it operates what is thought to be the largest sawmill in the world, and at Kvarnsveden it lately built a combined pulp factory and paper mill at a cost of \$760,000. Its annual dividends are 14 per cent.

The Kilafors Co. and a few others, notably the Skanaska Attikfabriken, of Perstorp, furnish a wide variety of products obtained from the destructive distillation of wood. Their lists contain the various charcoals, wood naphtha of 64°, methyl alcohol free from acetone, pyroligneous acid, technical and crystallized acetic acid, formaldehyde, creosote, carbon-

ates of creosote and guaiacol, turpentine, lampblack tar oil, carbolineum (for the impregnation of wood) pitch, etc. Little effort is made to go further in the isolation of the numerous other constituents of wood tar, or to use those separated for the manufacture of higher derivatives.

Forty years ago factories were started in Sweden for the purpose of utilizing the stumps of spruce and other conifers, by subjecting them to destructive distillation. Apart from tar, etc., it was sought to secure more particularly the crude wood oil, used in place of turpentine. This branch of manufacture has not been very successful, as it has not been possible to free the product from a pronounced burnt odor. At present six small factories carry on this distillation. The production of resin and turpentine is strangely limited for a country so rich in conifers as Sweden. There is an annual import of 400 tons of turpentine.

#### PRODUCTION OF COAL TAR.

There are 27 gas works in Sweden, but with the abundant water power and cheap electricity the industry does not expand rapidly. At Gothenburg the annual per capita consumption is 1,236 cubic feet. At Stockholm it is 3,088 cubic feet. Ammonia is collected in a few works in the form of the sulphate. The tar from the larger works is used for producing asphalt, carbolic acid, and creosote oils, chiefly for impregnating wood. The domestic supply of tar is insufficient, and there is an annual import of about 2,800 metric tons. One reason for the limited production of coal tar is the fact that Sweden possesses no coking coals and is forced to import coke from abroad. But little is done to carry on any systematic separation of the constituents of coal tar, apart from the carbolic acid.

A Swedish firm has introduced a novel and valuable feature into the treatment of coal tar. The Aktieförlaget Joh. Ohlsson's Techniska Fabrik, of Stockholm, has solved the problem of removing the carbon from coal tar. The black color of tar is due to the presence of a large amount of carbon in an extremely finely divided state. It constitutes about one-quarter of the weight of the crude material. As the liquid tar has about the same specific gravity as the solid carbon, the latter remains suspended. The firm in question has succeeded in completely isolating the carbon from the liquid constituents of tar, leaving the latter in the form of a clear, transparent, gold-brown liquid. Such a purified tar is evidently much better adapted for fractional distillation than the crude material, and should facilitate the separation of the hydrocarbons and other compounds with a high boiling point, an operation rendered difficult in the process of ordinary tar distillation, owing to the foaming caused by the fine carbon toward the end of the operation.

The carbon separated by the Ohlsson process is totally different from lampblack in its physical properties and is distinctly of a graphitic nature. When pressed into a compact form it furnishes admirable

electrodes for arc lighting and also for electrochemical work. It could probably be used with equal advantage in making crucibles, as it possesses marked superiority over retort graphite.

Details are withheld with regard to the method used for effecting the separation. In all probability light hydrocarbons are added to the tar to lower its specific gravity, and the actual separation is brought about by the use of centrifugals. The added hydrocarbons can be removed by distillation from the residual liquid tar. This process is worthy of careful study, owing to its possible bearing upon the isolation of the more complicated higher-boiling constituents of coal tar.

#### SUGAR.

Despite the northerly climate and various other unfavorable conditions, the production of beet sugar in Sweden has increased, until sugar importation has practically ceased. Beginning with 1 factory in 1876, there are now 20 in active operation, the largest producing 120 tons daily. The total annual production is about 130,000 tons. There are 9 sugar refineries employing 2,402 operatives. All factories employ the diffusion process. The industry is located entirely in the lower end of the peninsula. Seed is brought from other more favored lands. The average yield of sugar is very high, taking all factories into consideration, being 3.5 metric tons per hectare (2.47 acres). The average yield in France and Belgium is 3.2 tons and in Germany 4 tons. The industry has been fostered by a protective tariff, amounting at present to 1.82 cents per pound. The rate of duty has varied at different times, but has never been less than 1.6 cents. A helpful factor in establishing new factories has been the remission for five years of all taxes on such works when built at least 19 miles from any existing factory.

#### STARCH, GLUE, GELATIN AND YEAST.

Starch is manufactured on a somewhat extensive scale, chiefly from potatoes. There are 95 factories, producing a total of 7,580 tons annually. This meets the entire domestic demand. A large share of the product is changed into glucose and used for conserves. Glue and gelatin are likewise produced in nearly sufficient quantity to meet domestic needs. There are 13 factories, employing 223 workmen, and producing wares valued annually at \$300,000. Yeast is made in several factories, the annual output being 2,785 tons.

#### OILS.

In 1900 there were 14 oil factories, employing 159 operatives. The annual product was 11,736 tons of oil cake and 4,418 tons of linseed and rape oil, as well as small quantities of fish oil and train oil. In 1909 Sweden imported 16,904 tons of animal and vegetable oils, 872 tons of train oil, 684 tons of oleine, and 8,017 tons of fats.

One Swedish specialty in this branch deserves note. The "Collanoljefabriken" has produced for a number of years an oil very highly valued for treating leather

and rendering it waterproof. The composition and manufacture are kept secret, but it is supposed to be extracted from certain tropical plants. The oil is extremely viscous, can be drawn out in long strings, and repels water to a most pronounced degree. As a leather preservative it seems to render exceptional service.

#### MISCELLANEOUS.

The soap industry is highly developed. There are 42 factories producing annually 13,000 tons of soft soap and 3,700 tons of hard soap. Excellent toilet soap is manufactured in a few establishments. Imports of soap are relatively small. In some factories the production of scents is also extensive, the annual output being valued at \$200,000. This is about six times the value of the imports of scents.

The candle industry is so developed that it practically supplies the domestic demand. There are 3 factories, employing 312 workmen. The annual product, which includes also stearin, is valued at \$6,188,000. The imports are insignificant.

The domestic demand for varnish is very nearly covered by Swedish works. They number 15 and employ 91 workmen.

The manufacture of cement has grown rapidly of late years and is now an exporting industry. In 6 works 1,336 operatives are employed. The annual output of 126,000 tons has a value of \$1,130,000. About one-third of this is exported, chiefly to the adjacent countries.

There are Swedish concerns engaged in the manufacture of lactic acid, lanolin, fruit essences, cosmetics, and pharmaceutical preparations, and some of the last named are of international repute. Liquid carbonic acid is made in three factories, the total annual output being 415 tons.

#### MINING AND METALLURGY.

The mining and metallurgical interests are so closely connected with chemical industries that a few data are appended. In 1902 30,700 workmen were employed, of which 14,600 were employed in mining proper, including 10,500 in the iron mines.

The quantities of ores and minerals produced in 1902 were as follows, in metric tons: Iron ores, 2,897,000; lead and silver ores, 9,400; copper ores, 30,000; zinc ores, 49,000; manganese, 2,800; clays, 216,000; feldspar, 18,000; coal, 305,000. In 1908 the production of iron ore had increased to 4,713,000 metric tons, of which 3,444,000 were exported.

Metals were produced in 1908 as follows, in metric tons: Pig iron, 556,000; iron and steel bars, ingots, blooms, etc., 772,000; zinc, 274; lead, 277; copper, 2,807. Silver to the extent of 1,389 pounds and gold to the extent of 44 pounds were also produced.

Iron constitutes 95 per cent. of the value of the metallic output. Most of the companies engaged in the production of iron and steel are partners in the Järnkontoret, an association that represents

a peculiar valuable method of organization for mutual aid and protection.<sup>1</sup>

IMPORTATION OF CHEMICAL GOODS.

In the following table a detailed statement is given of the imports of chemicals, drugs, dyes, and raw material used in their manufacture, as furnished by the annual report of the Swedish customs administration for 1909.

Articles.	Metric tons.	Articles.	Metric tons.
Fats.....	8,017	Cosmetics.....	11
Oils, non-volatile.....	16,904	Perfumes.....	8
Petroleum.....	111,485	Soap:	
Train oil.....	872	Toilet.....	46
Camphor.....	14	Ordinary.....	260
Gums and resins.....	4,302	Albumen.....	3
Turpentine.....	412	Animal charcoal.....	186
Coal tar.....	2,782	Celluloid.....	22
Wood tar.....	200	Collodium.....	2
Barytes.....	529	Enamel material.....	102
Cryolite.....	57	Rennet extract.....	91
Fluorspar.....	672	Rubber, artificial.....	24
Magnesite.....	728	Varnishes.....	340
Pyrites.....	78,049	Medicines.....	137
Candles.....	46		

COLORS, ETC.

Alizarin.....	64	Lampblack.....	66
Annatto.....	11	Lithopone.....	960
Blacking.....	123	Paints.....	149
Bronze powder.....	8	Printers' ink:	
Bronze and chromium colors.....	110	Black.....	94
Catechu.....	207	Colored.....	22
Cinnabar.....	15	Quebracho wood.....	5,245
Coal-tar colors.....	687	Red lead.....	1,003
Colors in tubes and glass.....	13	Red ochre.....	542
Dyewoods.....	503	Sumach and extract.....	56
Gall apples.....	16	Tanning materials.....	6,286
Indigo:		White lead.....	510
Extract.....	37	Zinc, white.....	2,692
Synthetic.....	46	Other colors.....	2,563

CHEMICALS.

Alum:		Hydrochloric acid.....	2,134
Chrome.....	65	Iodine and iodides.....	4
Potassium.....	29	Lanolin.....	6
Alum cake.....	349	Lead acetate.....	61
Alumina.....	4	Lime.....	1,146
Aluminum sulphate.....	1,024	Lysol.....	1
Ammonia.....	13	Magnesium:	
Ammonium:		Chloride of.....	425
Carbonate of.....	137	Sulphate of.....	140
Chloride of.....	226	Naphthalene.....	74
Nitrate of.....	11	Nitric acid.....	89
Sulphate of.....	76	Nitric ether.....	5
Antimony.....	118	Nitrobenzene, mirban oil.....	46
Argols.....	19	Opium.....	1
Arsenic, white.....	13	Oxalic acid.....	38
Borax.....	365	Phosphorus.....	88
Bleaching-powder.....	3,018	Phosphoric acid.....	25
Boric acid.....	76	Potash, caustic:	
Bromine and bromides.....	10	Liquid.....	21
Carborundum.....	3	Solid.....	4
Cement.....	12,776	Potassium:	
Chloroform.....	6	Carbonate of.....	1,312
Citric acid.....	14	Chlorate of.....	207
Cobalt oxide.....	3	Chloride of.....	1,805
Copper sulphate (blue vitriol).....	104	Chromate of.....	87
Ferrocyanides.....	11	Cyanide of.....	4
Ferrous sulphate (green vitriol).....	945	Nitrate of.....	267
Formalin.....	5	Sulphate of.....	26
Glycerin.....	214*	Sulphide of (incl. Na <sub>2</sub> S).....	451
Graphite.....	442	Quinine preparations.....	1.3

Articles.	Metric tons.	Articles.	Metric tons.
Saccharin.....	4	Sulphur:	
Salicylic acid.....	4	Crude.....	23,255
Salt:		Flowers.....	59
Crude.....	2,174	Refined.....	3,521
Refined.....	24,143	Sulphuric acid:	
Soda, caustic:		Fuming.....	509
Solid.....	1,111	Ordinary.....	1,446
Liquid.....	8	Superphosphate.....	2,845
Soda ash.....	13,490	Tartaric acid.....	36
Sodium:		Thorium nitrate.....	1.7
Acetate of.....	36	Tin and lead oxides.....	58
Chlorate of.....	3	Tin salts.....	1
Chromate of.....	8	Water glass.....	405
Nitrate of.....	28,894	Zinc.....	8
Sulphite and hypo-sulphite of.....		Zinc chloride.....	50
Sulphate of.....	20,226	Other chemical preparations.....	1,444

It will be seen from the table that Sweden offers a fairly good market for a great variety of chemical products. Germany is the chief source of supply. The United States furnishes 78,143 tons of the petroleum, 4,314 tons of lubricating oils, 3,913 tons of molasses, 812 tons of fats, 1,157 tons of cottonseed oil, 1,543 tons of corn oil, and 284 tons of other oils.

EXPORTATION OF CHEMICAL GOODS.

In the following table are shown the Swedish exports in 1909 of chemicals and goods in the manufacture of which chemicals are used:

Articles.	Metric tons.	Articles.	Metric tons.
Fats.....	735	Wood pulp.....	564,000
Oils and olein.....	1,299	Ammonium sulphate.....	331
Coal tar.....	4,461	Calcium carbide.....	1,556
Wood tar.....	7,614	Cement.....	33,197
Turpentine, crude.....	191	Copper sulphate.....	182
Feldspar.....	16,575	Ferrocyanides.....	69
Rennet.....	61	Potassium chlorate.....	1,400
Soap.....	59	Sodium:	
Lampblack.....	392	Chlorate of.....	483
Red ochre.....	216	Sulphate of.....	598
Tanning extracts.....	196	Sulphuric acid.....	203
Matches (value, \$3,050,000).....	26,868	Superphosphate.....	17,245
Paper.....	137,510	Tin and lead oxides.....	60

THE FUTURE FOR SWEDISH PRODUCTS.

From the foregoing table it is evident that, apart from the magnificent array of its wares based upon wood as a primary material, Sweden has already begun to export in quantity several other products of its chemical industry. The chief representatives in this class are cement and superphosphate, supplied chiefly to adjacent countries, and the chlorates and calcium carbide, products of the recently established electrochemical works. It is likely that the exportation of the latter products will steadily expand and that Sweden will become a strong competitor in the world's markets for electrochemical products on account of the cheap water power. That its paper and pulp exports will continue to increase is likewise probable. While for the moment the match trade shows no evidence of a check, there is less confidence in its future growth. Tariffs, fiscal regulations, and an increasing power and efficiency among foreign competitors are threatening to restrict the field of operations very seriously. For the time being the

<sup>1</sup> A report covering the workings of the Järnkotoret, or Iron Institute, and the progress it has made in the reduction of iron ore in electric blast furnaces was published in Daily Consular and Trade Reports for July 19, 1911.

United Kingdom and the British Colonies are the best customers of the Swedish match trade.

#### EXPORTS TO THE UNITED STATES.

The values of the exports of chemical products to the United States in four years, 1907-1910, were as follows:

Articles.	1907.	1908.	1909.	1910.
Cement.....	\$ 41,000	\$ 7,000	.....	.....
Chemicals (chiefly caustic alkalis).....	6,000	4,000	\$ 7,000	\$ 13,000
Fusel and tar oil.....	.....	2,500	.....	5,400
Glue.....	.....	3,000	.....	.....
Matches.....	112,000	124,000	207,000	157,000
Paper.....	276,000	496,000	850,000	623,000
Wood pulp:				
Chemical.....	.....	.....	1,318,000	1,636,000
Mechanical.....	499,000	1,060,000	.....	4,500

The United States purchases Swedish bar iron to the amount of over \$1,000,000 annually, and about \$600,000 worth of wire rod, as well as smaller quantities of several other forms of the metal. The export of Swedish ferrosilicon to the United States was valued at \$5,000 in 1909, while in 1910 it had attained a value of \$51,000. Of much greater interest is the sudden increase of the American demand for Swedish iron ore. The value of the importations ranged from \$12,000 to \$32,000 during the period 1907-1909. In 1910 the figure reached \$983,000. For a number of years past America has purchased Swedish pig iron to an average annual value of \$90,000.

#### IMPORTANCE OF THE SWEDISH MARKET FOR CHEMICAL GOODS.

As the American manufacturers of heavy and finer chemicals approach the point of competing with Germany and Great Britain in the world's markets, they will find in the Swedish trade an important field. For the time being there is an opportunity to extend trade in colors, paints and pigments, blacking, varnishes, and a few similar articles, the value of which is already appreciated in Sweden. The consumption of quinine preparations is also relatively large.

The outcome of the Swedish studies in the electric smelting of iron ores may mean much for certain iron deposits in the United States where conditions are not unlike those existing in Sweden.

#### FUEL ECONOMY IN FACTORIES.<sup>1</sup>

By C. E. LUCKE.

Received November 13, 1911.

Maximum economy in the use of fuel as one of the primary raw materials in manufacturing is to be measured by the least consumption per unit of manufactured product, a statement ridiculously easy to make but most difficult of realization. The difficulties in realization arise from the almost infinite variety of combinations in the uses to which the heat liberated by combustion may be put, and the relation of heat quantities entering into each of the different uses

<sup>1</sup> Address delivered at the joint meeting of the A. C. S., S. C. I and A. E. S., New York, November 10, 1911.

of the heat, the difficulty in measurement of heat quantities, and the bulkiness of the records of the heat balance required to keep track of the heat disposition in the establishment. No one would think for a moment, in conducting the financial operations of a business, of dispensing with records of expenditures and income or fail to provide a more or less elaborate cost system for distributing the expense over the various parts of the apparatus used in production and for dividing cost of the products into the different elements that make it up. No one would think of attempting to economically manufacture a steam engine without providing for a regular systematic determination of the cost of the crank-shaft and in some instances for the distribution of the cost into elements of raw steel from which it was made, the operations of forging, machining, transporting and handling, and for the fixing of the overhead charges, all of which are determinable items, and to which may be added many others of lesser importance, carrying the process as far as the information is worth. Cost information is worth as much as the possible saving in cost of production to which the information leads, so that manufacturing managers must use and do use their best judgment in deciding how far they will detail their cost records, but it will be difficult to find one who would attempt to carry on his business without cost records as elaborate as the conditions warranted. It is quite the same with fuel economy, that is, so far as need of records is concerned but by no means the same in practice. There can be no rational attempt made toward fuel economy in a given system of manufacture using fuel without a knowledge of the distribution of the heat of that fuel throughout the establishment, and it is no more difficult to trace the heat throughout a system supplied with heat than it is to trace expenses for material and labor in a crank-shaft or fly-wheel relating each to the total expense incurred, though it is quite possible that the information may not be worth enough to warrant as complete a distribution of the expense of heat supply or as elaborate a system of records as would be warranted in the case of labor or materials entering directly into the factory product. However, there can be no question as to the absolute necessity for some heat accounting to determine the coal heat charge to be attached to each individual process using heat and to show sources and quantities of utilizable waste heat, however simple the system of the accounting may be, nor can there be any doubt as to the increasing justification for more and more elaborate records as coal cost forms an increasing fraction of the total cost of production. How many factories there are in which no more records of heat are kept than the cost of coal per month is impossible to state in the absence of statistics, but according to my own observations the number will be very large, probably in excess of three-quarters of the total establishments. It is not surprising, therefore, that maximum heat economy is so seldom attained or that managers are so constantly besieged and bewildered by, and so much dependent upon, the statement of promoters of all sorts of schemes

who promise to accomplish most impossible economies and yet whose absurd arguments cannot be answered because of absence of data. How many managers, having under their direction steam plants for power, know the cost of their draft sufficiently closely to be in a position to judge the value of a new draft system offered for their consideration? How many know even the cost of the power itself so as to be in a position to judge a new power system offered as a substitute? It is safe to say that those who have data of the above sort constantly up to date are far less in number than those who know the total cost of their product and its fractional part of overhead charges, labor and materials and yet every one will claim a desire for fuel economy.

Heat accounting is necessary. How much accounting depends upon the fraction of total production cost chargeable to fuel in one form or another and through these accounts economy may be affected most readily by attacking first the big losses or items of large heat waste. In this way and only in this way may the best results be obtained from a given installed system in operation or the relative value of a competing system judged, as a preliminary to a change of system. In many cases it will be found that managers believe they have records of their heat distribution and power cost but these figures are what their so-called operating engineer hands them. It is most curious how the engine room cost figures are so seldom subjected to critical analysis and checking to make sure that they include all the items that are properly chargeable to that account or to insure the correctness of the items that are reported. This is in striking contrast to the care with which so-called factory cost systems will be installed and operated by experts in the same establishment which is satisfied with the accounting of a skilled mechanic called an engineer for its power and heat cost.

Economy is then always a question of accounting, that is, correct accounting as elaborate as the conditions may warrant in every establishment. The attainment of maximum economy is, however, also largely a question of the operating system used. It is one thing to know for a given plant in operation where the heat losses are and how much they are; it is another thing to know how to reduce these losses in the existing system by modifying the detail of management or even altering connections or substituting new units, but it is quite a different thing to know when or how to change the system as a whole to an entirely different one or how to compare broadly existing with possible new systems.

In a general discussion of fuel economy, it seems as if the question of system ought to be put in the first place, especially if it can be shown that there is any fundamental and essential differences in the losses involved and hence in the net fuel economy of the system applied to a factory. It may be assumed that in a factory fuel enters directly or indirectly into four classes of services, all of which are essential to the making of the product:

1. Power.

2. Factory heating in cold weather.

3. Factory lighting.

4. Direct manufacturing processes, such as heating, cooling, evaporating, condensing, melting and drying.

In a given establishment the system involving all these processes may be self-contained or may be externally sustained. A system is self-contained when all four classes of service may be derived from the same coal supply. It is not self-contained when it is partly or wholly dependent upon external supplies as, for example, gas for lighting alone, electricity for lighting alone, or for power in addition, and perhaps for high temperature furnace work with coal for the other services. In all systems where coal is used in manufacturing, its heat is rarely applied directly to the service to which it is ultimately put, but its heat is usually transferred in part to steam or transformed in part to combustible gases, giving in the former case the steam system of heat distribution and in the second case the gas system. Where heat of coal is directly applied to such service as high temperature heat supply, that service can always be better done by gas derived from the coal in just about the same degree of gain in efficiency as in the gas over the coal-fired cook stove. There is one exception to this, however, and that is where fixed carbon enters as a necessary element in a thermal reaction, but these cases are so clearly exceptional as to need no comment.

The primary process in the use of heat is, therefore, the making of steam on the one hand, or the gasifying of coal on the other, and the direct loss encountered in either of these processes is the first factor for measurement and record in the heat-accounting system. This first step of conversion may be made to take place with an average efficiency of 65 per cent. to 75 per cent. for the steam system, depending on the coal, the goodness of the firing and the type of equipment, and an efficiency from 75 per cent. to 85 per cent. for the gas system, showing a clear gain of about 10 per cent. for the gas system on the average for this first step, with, however, a limitation and this limitation is the inability of the producer to handle as wide a range of fuel with equal success to that attainable in the boiler.

Steam once made is transmitted in pipes for short distances only and under high pressure with constant heat loss for every foot and involving high maintenance charges, on its way to engines for shaft power or electrical generation. The gas, however, may be transmitted at low pressure over very long distances to all the buildings of large establishments or to neighboring establishments several miles away or from a central gas generating station dozens of miles distant through pipes cheaper than those required for steam and with almost no maintenance cost in comparison. This pipe distribution cost is the next item in the accounting and without setting down numerical values is again plainly in favor of the gas system, which involves no heat loss whatsoever while the steam involves a large one and in which the pipe maintenance costs are hardly comparable at all.

The next step is the conversion into work, in the engine, of the heat carried by either the gas or the steam, and here the balance is again in favor of gas as the gas engine is a much more efficient heat converter than the steam engine in the small sizes found in the average factory and even in larger sizes also, though there to a lesser degree. Few factories would find any occasion to use engine units exceeding 1000 h. p., which size, operated condensing, whether of the turbine or compound Corliss sort, will yield thermal efficiencies less than 20 per cent., whereas, operated non-condensing, these efficiencies would drop to at least 15 per cent. A smaller steam engine of, say, 100 h. p., will be less efficient and the turbine would not be so good as the piston type, though the difference would not be so very great. For this size the efficiency that could be counted on would be about 15 per cent. operated condensing and not over 10 per cent. operated non-condensing. Compare these efficiencies with the sure and substantially constant thermal efficiency for the gas engine in all sizes, of 25 per cent., which indicates a margin in favor of the gas of at least 5 per cent. for large steam engines condensing, 10 per cent. for the smaller condensing and also for the large non-condensing and 15 per cent. for the small non-condensing steam engines. These efficiencies are not the best ever obtained but are about as good as one can count on obtaining in factory work. Of course, load fluctuations will affect the result but these figures are for a load equally favorable to the two classes of machines.

If electricity is generated and transmitted for lighting or for use in electric motors the efficiency of electrical generation, transmission and reconversion to power and light is the same for both systems. It is right here, however, that another item may be put on the credit side of the account in favor of gas and one that is not generally or sufficiently appreciated, arising from the possibility of eliminating some of the electrical equipment, if not all. If machines are to be driven in various distant parts of the establishment there is no reason why we should not do as has been done more often in Germany, and that is to set down small individual gas engines at the spot where power is required and transmit gas with practically no loss instead of generating electricity and transmitting it to electric motors with three losses. This would not only save the expense of the electrical equipment but would save all electrical losses, which are often considerable, especially when generator and motor work at unfavorable loads. This is a perfectly feasible proposition and will give substantially the same efficiency of conversion as is obtainable in a single large central engine, a fact that distinguishes the gas system more than anything else from the steam system. Concentration of many small steam engines into a single large one always results in steam economy, a fact which has led to constant concentration of steam systems into larger and larger central generating units, so that the steam may be regarded as a centralizing influence whereas, on the contrary, the constancy of gas engine

efficiencies in large or small sizes must characterize the gas system as a decentralizing influence in the interests of economy.

Take next the question of factory lighting of the self-contained system, which may be electrical with current supplied by either steam- or gas-driven generators and which can be only electrical for steam engine-driven generators but which may be something else when gas is available. There is no good reason for assuming that mantle lights of fair brilliance and moderate gas consumption cannot be produced to operate on weak producer gas for factory lighting and as soon as they become available, as they surely will when the demand becomes pressing enough, mantle gas lighting with producer gas will displace to a very large extent electric lighting in those establishments where the gas system is used for power and heat and gas transmission substituted, at least in part for electric. This sort of change while not feasible now, because of the lack of development of weak gas, mantle lights, will add another credit item in favor of the complete gas system and the possible gain in fuel economy for lighting should be incentive enough to produce these lights and warrant their quite general use.

Heating of buildings is almost universally accomplished by means of steam- or water-heated radiating surfaces at a temperature of 200 degrees Fahrenheit or thereabouts and there is no good reason for changing the system. The preparation of this low-pressure steam or moderately hot water can be done with equal efficiency by either the gas or steam system in cold weather, though the latter again receives a credit charge for all the rest of the year when heat is not required. For example, if the exhaust steam from steam engines is used it must be at approximately atmospheric pressure to give a high enough temperature, or live steam must be used either alone or added through reducing valves as make-up. In any case the heat put into the heating system gives about 100 per cent. effect in the warming of buildings, especially when drips are returned. This is also the case for the gas system as jacket water heat and exhaust gas heat can be combined for the same purpose and fresh gas drawn upon through thermostatic gas burners to supply any deficiency. By either method the effect is the same, full use of waste heat and 100 per cent. efficiency in the use of fresh supplies of heat in the warming of the building. During the time of no building heat requirement, which in this locality lasts for more than half the year, the waste heat of the steam engine is far in excess of that of the gas engine, especially if the former be non-condensing as is usually the case where exhaust steam-heating systems are in use so that measured over the whole year the gas system is again most favored.

There remains now to examine the possibilities of credit or debit items for the two systems in applying the heat to the direct manufacturing processes, which may be for convenience divided into low-temperature heating and high-temperature heating, the former including most of the evaporating and drying



processes and the latter roasting, baking, melting, etc. All low-temperature processes can be carried on by steam-jacketed chambers or steam coil apparatus, using live or exhaust steam, a fact that has led to the design and mechanical perfection of many appliances now regarded as standard and for which there is no gas-heated equivalent. There is no good reason, however, why equivalent apparatus should not be quite as effectively designed and with efficiencies over 90 per cent. for operation with gas burners but like the mantle light the need has not yet been strong enough to warrant their production. Such evaporators, concentrators, cookers and driers as fall in this class will, when gas-heated, offer still further simplification of piping and reduction of piping loss over their steam-heated equivalents and should be equally effective. High-temperature heating can be accomplished only by oil, piped illuminating gas or independent coal fires with the steam factory system while the gas system offers a possibility of producer gas-fired furnaces for practically all services and with almost perfect temperature control better by far than is possible with oil or coal and more economical because excess air and incomplete combustion can both be eliminated.

Thus, for the heating processes of manufacture, the gas system offers the same high-temperature source of heat, a gas fire, for all, while the steam system requires fresh fuel for the high temperatures, accomplishing the low-temperature service with live or exhaust steam, but the former is not an accomplished fact simply because no one is yet prepared to sell gas-fired appliances to take the place of steam-heated ones.

Comparing gas and steam systems broadly by summarizing the preceding brief analysis, it appears that the self-contained factory plant may be much more economically operated by the former than by the latter for all the services of power, lighting, building and process heating, but this is not all, for there is also in favor of the gas a very great simplicity in the heat balance determination for the gas over the steam so very great as to make it easier to keep up the operating economy nearer to the high possibility by quick and easy determination of wastes. For example, by means of a central gas meter and as many individual distributing meters as the separate consumptions of gas, the gas generated may be easily known as well as its distribution throughout the plant for all purposes by no more complicated procedure than the reading of meter dials which any good boy can do. Add to this recording calorimeters, and gas meter readings can be reduced by one multiplication to heat consumed by each part of the system. With such a ready check on consumptions and losses, it is sure that heat-accounting will be practiced because the cost of the account will be so small and, therefore, the plant may be and will be operated at somewhere near its good economy point. This is in most striking contrast to the case of the steam system where the possible economy is not only less but where in addition the realized economy is so much below its own maximum possibility simply

because of the difficulty of making the measurements necessary to a heat balance with reasonable accounting costs. In the first place it is impossible to continuously measure the heat supplied in steam generated because there is no reliable and cheap steam meter available and, even if there were, the heat equivalent of a pound of steam constantly varies with feed temperature, steam pressure and its wetness, or superheat. Pipe line losses can be determined only by collecting and weighing drips; steam consumptions of other appliances and their heat equivalents is a question of many recording steam meters, steam calorimeters, thermometers, pressure gauges and automatic water weighers. To keep up all the instruments of this sort that would be needed so that they will remain reasonably accurate and to make the calculations of combining the various readings to get a heat balance would in most cases cost more than the information would be worth though a partial record is always a paying thing when designed to include the big items.

Why is it then, in view of both the higher possible economies of the self-contained gas factory system for the four classes of factory service and the correspondingly easier and cheaper fuel-accounting that would follow its use, that there is to be found no such system in use? The answer is as complex as a detailed study of the systems, but a few facts stand out more prominently than others: First, there must be put the inertia of the human mind, which resists the mental effort necessary to bring about a change when things seem to be running fairly well in the older way. There is always a latent suspicion that the new thing may not work however clear a case is made out for it on paper, and even if the manager himself is convinced in the light of his trained ability for clear independent thought, he may not be able to convince his board of directors who are ordinarily incapable of thinking in terms of anything but past performances of the factory, however willing they may be to deal in the futures of stocks and bonds. So far there has been in this particular case some good reason for suspicion, not of the scientific engineering possibilities but of the business expediency of a general change because of the lack of standardization or mechanical perfection of some of the needed appliances. We have no producer gas mantles for lighting, no gas-fired evaporators, concentrators or driers, and most important of all, we have no universal gas producer capable of economically using any available solid fuel, but it is for us to make these things, and my main object in speaking on this subject is to stimulate their production by showing a motive. Let the gas appliance makers get to work on the problem of adapting apparatus for all the manifold services of lighting and heating to the use of weak producer gas and let us all, chemists and engineers, try to solve the gas producer problem. Progress in the perfection of appliances for using producer gas will stimulate the demand for the right sort of gas producer to generate it, and the appearance of the producer on the market will force

the creation of gas-burning industrial apparatus so that each reacts on the other. As soon as the industrial world concerned with fuel economy in manufacturing demands these things in a sufficiently strong and positive manner they will be produced.

### DEFLOCCULATION.<sup>1</sup>

By EDWARD G. ACHESON.

Received November 18, 1911.

It is with much diffidence that I come before you to speak on a subject that has not yet emerged from the embryonic state. My latest experimental researches had to do with it; I believe it will rapidly grow in importance in the scientific industrial world; and finally much work of a strictly scientific character remains to be done to reduce the fragmentary knowledge we now have of it to an exact science.

In my labors devoted to working out and developing industrial and commercial projects, I have upon several occasions found reactions, conditions and results that did not harmonize with the accepted theories and formulae of scientific men. Being an earnest believer in publicity, in order that any possible benefits that might accrue to the common welfare may the more quickly be enjoyed, I shall lay before you a detailed account of my experiments on the deflocculation of inorganic bodies. It will become very evident, as my story unfolds, that throughout the series of experiments described and the working hypothesis employed, I was wholly disregarding the prevailing theories, and that I unconsciously entered the field of colloids.

Having worked out the problems involved in the manufacture of graphite from coal and other carbonaceous materials, I undertook, in the summer of 1901, the introduction of this artificially made graphite into the crucible trade. My first efforts were devoted to the making of a satisfactory crucible of my graphite, using as a binding material American clays. Many failures were met with, and I found it difficult to locate the cause of the failure, whether with the graphite or with the clay. I soon learned that the manufacturers of crucibles in the United States invariably used, as a binding agent for the graphite in the crucible body, clays imported from Europe. I secured samples of these imported clays, and found them much superior to the American ones in plasticity and tensile strength.

Chemical analysis failed to disclose the cause of the physical differences existing in the clays. The question involved interested me greatly, and I decided to endeavor to determine what produced the variations. I found it generally stated in the books that residual clays were non-plastic, and sedimentary clays were more or less plastic. Here was the starting point. Plasticity was developed by or during the act of transportation from the point of formation to the final resting place of the clay. I did not believe there was anything in the simple act of the suspension

<sup>1</sup> Address delivered before the Society of Chemical Industry, London, November 7, 1911.

in water that would produce the effect noted, and therefore looked for the cause of the foreign matter carried by the water. It seemed that the most likely agents were the organic substances washed from the forests into the running waters. With this idea in view, I made a few experiments with those substances I thought likely to be found in the washings of vegetation. One of my early experiments was to treat kaolin with a solution of tannin, and I at once noticed less water was needed to produce a given degree of fluidity; also that the tensile strength and plasticity were increased.

"Tests were made on the increased tensile strength of clay, as the result of treatment with organic matter, and it was found that briquettes made of Harris kaolin and dried at 120° C. would break with a load of 5.73 kilograms per square centimeter, while the same clay, after treatment with 2 per cent. of catechu for a period of ten days, then formed into briquettes and dried at 120° C., would not break until the load was increased to 19.75 kilograms per square centimeter—an increase of more than 244 per cent.

I now began to wonder whether or not the effect I had discovered was known, as it might have much value to an industry of such colossal dimensions and antiquity as clay-working. Moreover, it would be amazing if it should not be known, in view of the tremendous amount of experimental work that had been done on that art. I searched for some record of the addition of organic matter to clay during its working, and only one instance could I find, that of the Egyptians in brick-making, as recorded in the fifth chapter of Exodus. The accepted theory of using straw fiber as a mechanical binding agent had never appealed to me. Straw, however, contains no tannin, and the effect I had found had always been produced with tannin, or a substance containing tannin. I procured some straw, boiled it with water, decanted the resultant reddish brown liquid, and mixed it with clay. The result was like that produced with tannin, and equal to the best I had obtained. It now seemed likely that the Egyptians were familiar with the effect I had discovered, and believing this was why they used straw in making brick and were successful in substituting stubble for the straw, I called clay so treated "Egyptianized Clay."

The effect of organic matter, as typified by tannin, in producing deflocculation and a resultant colloidal state of clay is very readily shown; for instance, I have here some powdered kaolin, a small quantity of which I will place in a test tube, add water, and after shaking, set aside. Another portion of the kaolin I will put into a beaker, and moisten with a water solution of tannin, to which a small amount of ammonia has been added. After a thorough mixture has been made, using a glass rod to stir with, to eliminate as much as possible any grinding action, I will add more water and divide the contents of the beaker between two test tubes. To one of the tubes containing tannin-treated clay I will add a little common table salt. The three tubes I will place here before you, that we may examine them later.

In the Summer of 1906 I succeeded in making artificially a high-grade graphite which I wished to make applicable to all kinds of lubrication. To meet the various demands, it would be necessary to have it remain diffused in liquids lighter than itself; for instance, water and petroleum oils. Recalling the effect of tannin on clay, which caused it to remain diffused in water, I treated a sample of my graphite with tannin, and found the same effect occurred. The graphite being black, it makes a better lecture demonstration than the clay I have shown you, and I will repeat my experiments, using graphite. I have here a sample of artificially made graphite, which has been disintegrated to a fineness that will permit it to pass through a sieve having 40,000 meshes to the square inch. I will introduce a small quantity of it into a test tube, add water and, after shaking, set aside. Another sample I will place in a beaker and moisten with a solution of organic matter, and after thoroughly stirring with a glass rod, I will add water and divide it between two test tubes, to one of which I will add table salt. These three tubes I will place beside those holding the clay, to be examined later.

The actual amount of deflocculating effect produced on the graphite in the beaker is very small indeed. In commercial work considerable mastication and time are required. I have here a bottle containing water having two- to three-tenths of 1 per cent. of its weight in deflocculated graphite; the deflocculation having been produced by a treatment similar to that I have applied to the graphite in the beaker, and a little of it being poured on a filter, you see the black liquid running into the test tube below the filter. The paper retained none of the graphite on its upper surface, all of it having passed into and through the paper. I will now add two or three drops of acid to the black liquid in the tube, and after warming over a spirit lamp, will throw it on another filter paper, and you now see a clear, colorless liquid descending into the tube below the filter. This is the water in which the graphite in a deflocculated condition was diffused; the graphite having been flocculated by the acid is now retained on the upper surface of the filter paper. The effect I have produced with the acid could have been produced with a solution of salt, lime water, or any one of that large list of substances known as electrolytes, even so weak an acid as carbonic acid if caused to bubble up through water carrying deflocculated graphite will cause flocculation and sedimentation.

Upon being deflocculated, the graphite is diffused through the water in a colloidal state, and I have samples of deflocculated graphite in water which have stood for more than two years without showing any settling, notwithstanding the graphite is two and two-tenths times heavier than water.

I have been able to obtain this same effect on natural graphite, amorphous alumina and silica, lampblack, and my manufactured product—Siloxicon, which is an amorphous body having the formula  $\text{Si}_2\text{C}_2\text{O}$ . The effect can be produced with a long

list of organic bodies; for instance, tannin or organic substances containing tannin, also with solutions containing the gum of the peach and cherry tree, or extracts from straw and grass. The off-fall from the barnyard proved to be very efficient. I speak of these organic substances as agents when used to produce deflocculation, and they act as protective colloids to the deflocculated body.

Some minutes have now passed by, and we will examine the tubes containing the clay and the graphite. We find the clay that had been mixed with plain water has settled. The mixture of clay, water, organic matter and salt has also cleared, while the tube containing the clay, water and organic matter remains muddy. In like manner the tube containing the disintegrated graphite in water has cleared; the second one containing water, graphite and organic matter remains black; while the third tube, which was set up the same as the second, but had a little salt added to it, has cleared. Apparently a great affinity existed between the organic and the inorganic substances introduced into the water. The inorganic body abstracted the organic from the water, and in doing so, was deflocculated. Each particle as it was thrown off was enveloped in an aqueous jelly of the organic agent, or at least such was the working hypothesis I followed to arrive at my results, and I find it difficult to think of this breaking up stopping short of the final subdivision with the resultant separation into individual molecules, or the smallest particles into which a body can be subdivided without loss of identity.

As I have already stated, I deflocculated clay in the year 1901 and graphite in 1906 and immediately afterwards a number of other bodies. I early understood I was producing colloidal conditions of these bodies, but not until the summer of the present year, 1911, did I read any treatise on colloids, being familiar with this state of matter only in a very general way. During the summer I procured a copy of the book "Colloids and the Ultramicroscope" as written by Dr. Richard Zsigmondy and translated into English by Jerome Alexander, of New York. I found the book extremely interesting, and at once wished to have a sample of my deflocculated graphite subjected to ultramicroscopic examination. Mr. Alexander kindly undertook the examination. He found the graphite in the deflocculated condition to be in a true colloidal state, the particles being in rapid motion, and he estimated their average size in linear dimensions to be 75 millimicrons. Seventy-five millimicrons are seventy-five millionths of a millimeter, and it would require slightly more than 13,000 of the particles to extend one millimeter. Now, the particles of disintegrated graphite, used as the crude material from which to produce deflocculated graphite, pass, as I have stated, through a sieve having 40,000 meshes to the square inch and their maximum linear dimensions are such that it would require 13 of them to extend one millimeter. Hence, the particle of disintegrated graphite is one thousand times greater in linear dimension than the deflocculated one. These

are figures that certainly test our powers of appreciation.

I have been asked, "Why don't you speak of the graphite as colloidal?" Knowing now that it is in a colloidal state, I speak of it as being colloidal, but when I am speaking of my process I am talking of a method of producing deflocculation. When does that process of deflocculation stop? Is it short of the final subdivision and the throwing off of the molecule? I think not. I believe we are here dealing with molecules. Their size may not agree with what they should be as computed in accordance with accepted theories, but, nevertheless, I cannot conceive the subdivision once started, in the presence of sufficient deflocculating agent, will stop short of the final, with the freeing of the molecule and the creation of the colloidal state.

How did all this I have been telling you come to happen? The following quotation aptly tells how:

"It's generally the fellow who doesn't know any better who does the thing that can't be done. You see the blamed fool doesn't know it can't be done, so he goes ahead and does it."

#### THE CHEMIST AND THE COTTON SEED OIL INDUSTRY IN AMERICA.<sup>1</sup>

By DAVID WESSON.

Received November 14, 1911.

Few industries have made greater progress during the past forty years than the manufacture of cotton seed oil and by-products of the cotton seed.

Without indulging too much in statistics, we need only compare the amount of seed crushed in 1872—52,000 tons—with the average annual crush of the present time—4,000,000 tons.

To put it another way, in 1872 4 per cent. of 1,317,000 tons were crushed, while the average crush of to-day is 66 $\frac{2}{3}$  per cent. of 4,000,000 tons. This put into dollars would mean that in 1872 the farmers received not over \$8.00 per ton from the mills or \$416,000 total, while at \$20.00 per ton for seed, a conservative price for to-day, the mills are paying annually \$80,000,000.

The growth of the industry is shown in the following table:

Year.	Seed worked.	Per cent. of crop.	50 gal. bbls. of oil.
1872	52,705	4	35,000
1880	235,404	9	175,000
1890	873,702	25	690,000
1900	2,479,386	53	1,920,000
1910	4,000,000	66	3,200,000

The rapid growth shown above is due to several causes, such as increase of population in the country and the increasing demand for edible fats in all parts of the world, but the chief cause has been the work of the chemist which has made the oil edible and has cheapened the cost of production, while the cost of the raw material, the cotton seed, has been constantly increasing.

<sup>1</sup> Presidential Address delivered before the Society of Cotton Products Analysts, Chemists' Club, New York, June, 1911.

When the speaker first saw crude cotton seed oil in the spring of 1884, the chemists employed by the oil mills and refineries could be counted on the fingers of one hand. If near chemists were included the fingers of both hands would have been sufficient.

Apparently the first chemists were employed in connection with the refineries, mill work being considered a mechanical problem pure and simple.

The refining methods at that time were very crude and purely empirical. Caustic soda solution was added in varying amounts according to the ideas of each individual refiner; in one case, for instance, the strength of the lye was judged by feeling a drop between the fingers.

The refining losses were tremendous. Oil was refined with 12-14 per cent. loss which now is easily handled inside of 8 per cent. The finished oil was as a rule tainted more or less by soap stock, and no two lots were alike. The refiner was entirely dependent upon the character of the seed for the color and flavor of his product. One season all oil might be prime, while next season most of the oil might be off.

White oils were produced in the early days by treating the yellow oil from alkali refining with sulphuric acid, followed by an alkaline wash. Some people used permanganate of potash or bichromate of potash and sulphuric acid. The resultant oil was white but not very edible.

In 1881-1882, fuller's earth was first used, making a white oil superior to that produced by what was called the acid treatments. It had a very poor flavor, but never the less large quantities were used in lard mixing.

The introduction of an improved method of refining the oil in 1900 has probably done more than any other factor to put the industry on its present basis.

By means of this refining method, which for trade reasons is kept secret, it is possible to turn out a perfectly neutral odorless product from the regular commercial oil, year in and year out, putting refined cotton seed oil so far as uniformity is concerned in the same class as granulated sugar.

The high standard of quality set by this process has made all large refineries improve their methods, and this has taken the oil from the soap kettle and put in on the table with consequent increase in value.

Now let us turn to the oil mill end of the business. The first systematic examination of mill products was begun in 1887, a year after the formation of the American Cotton Oil Trust. Samples from the different mills were examined regularly in the writer's laboratory, and the oil, ammonia and moisture determined in meal and cake. The hulls were also examined for meats and imperfect hulling. In those days the cake was sold on looks, soft yellow cake running 14 per cent. or so of oil being considered the best, while bright yellow meal, running about 10-12 per cent. of oil, was quite regular, though many mills did much better. Ten per cent. in cake was considered a fair basis for computing yields. I have before me analytical results from seven representative mills in Dec., 1888.

The lowest percentage of oil in meal was 8.75, while the highest was 13.37.

I also have here a table showing the yields of about fifty mills during the season of 1887-1888. The best oil production shown is 44.05 gallons with 783 lbs. of cake per ton, while the poorest is 33.69 gallons and 737 lbs. of meal or cake.

The highest cake production shown is 852.63, while the lowest is 636.38. These results compared with present methods calling for 900 lbs. of meal and not over 7 per cent. of oil left in the same show what an improvement has taken place in the work of the oil mills.

The change in mill results is mostly due to the chemist who by constantly testing the products has shown the losses, and made necessary constant improvements in machinery, and care in operating the same.

What the chemist has done for the oil mills as a whole can be figured in dollars and cents as follows: Old method of working made 800 lbs. of meal and left in it 10 per cent. of oil or 80 lbs. oil per ton of seed. Present methods make 900 lbs. of meal and leave 7 per cent. of oil or 63 lbs., a difference of 17 lbs. of oil saved per ton of seed worked. At 5 cents per lb. this means a saving of 85 cents per ton of seed, or \$3,400,000 on the crop, without taking increased cake into consideration.

If we compare the old refining methods with the present ones, we find instead of 12 per cent. loss, an average of 8 per cent., 4 per cent. of 3,200,000 bbls. worth 5 cents per lb., \$20.00 per bbl., or \$2,560,000.

The conversion of soap stock into fatty acids and glycerine has raised the value of the fat therein at least 3 cents per lb., or \$12.00 per bbl.

In refining 3,200,000 bbls. of oil, 7 per cent. would appear as fatty acids in soap stock—224,000 bbls., which, at \$12.00 per bbl., would be worth \$2,688,000.

By making the oil edible, \$2.50 per bbl. is a conservative figure to put on the increased value of the oil on 3,200,000 bbls. This amounts to \$8,000,000 a year.

If we sum up we find results as follows:

Improved mill work.....	\$3,400,000
Improved refining.....	2,560,000
Improved soap stock value.....	2,688,000
Improved flavor of oil.....	8,000,000
<hr/>	
Total annual increased value of oil mill products due to applied chemistry.....	\$16,648,000

We might go further into the various products made from the oil and soap stock, but this is sufficient to show, though somewhat imperfectly, the value of our work to the oil mills. On the other hand, the increased quality of the oil has increased the demand for it, and the supply of raw material, the seed, being limited, has gone in value from \$8.00 to \$20.00 per ton or an increase of \$12.00 per ton. 4,000,000 tons of seed at \$12.00 a ton is \$48,000,000 a year which goes to the farmer. In other words, chemistry applied to the oil mill business has done three times as much for the farmer as it has for the mills and refineries.

When we look back at our achievements, we feel something has been accomplished but we well know there is still a hard struggle before we get the business on a secure basis.

No sane miner would buy ore at \$20.00 per ton without having every car very carefully sampled and assayed. He would pay for no more than its value as shown by analysis. With cotton seed, we all know how little attention is paid to its intrinsic value, and how little care is taken to sample it. One per cent. oil in the seed means \$1.00 per ton difference in value, but who ever heard of seed being docked because it was poor in oil?

Meal is being more and more sold on analysis as it should be, but there is still much to be desired.

We have good methods for soap stock analysis, but we lack a strictly scientific and at the same time practical method for crude oil analysis. It is hoped we can some day in the near future obtain a method, the accuracy of which will be commensurate with the value of the product.

Let us continue our work, and, encouraged by the achievements of the past, endeavor to do better in the future, so we may all be proud of our membership in the Society of Cotton Products Analysts.

## NOTES AND CORRESPONDENCE.

### CORRECTION.

#### The Preparation and Properties of Metallic Cerium.

*Editor of the Journal of Industrial and Engineering Chemistry:*

In your Vol. 3, No. 12, December, 1911, pp. 880-897, appears an article by the writer on the "Preparation and Properties of Metallic Cerium." In justice to previous investigators, the writer would like to make the following corrections:

Bottom of page 881.—"The following year Hillebrand and Norton published . . . chlorides of cerium and potassium." They used sodium-potassium chlorides and cerium chloride.

Bottom of page 881.—"They succeeded in producing about 6 grams of cerium." Should read, "They succeeded in producing about 6 grams of cerium at a single operation and produced in all about 30 grams of the metal."

Page 886.—"Earlier work on the Electrolysis of the An-

hydrous Chlorides." The method there described is that given by Moissan in his *Chimie Minerale* and is somewhat different from the process used by Hillebrand and Norton.

Page 891.—Under the heading "Physical Properties of Metallic Cerium" should be inserted the following: "Some of the physical and chemical properties of cerium have been determined by Hillebrand (*Pogg. Ann.*, 156, 466 and 158, 71) and by Muthmann (see references 23 and 24)."

Page 893.—At the end of the last paragraph on the "Determination of the Melting Point" should be added: "The melting point determined by the writer corresponds fairly closely with the value given by Muthmann (623° C)."

Page 893.—At the end of the paragraph on "Specific Heat" should be added: "Dr. W. F. Hillebrand determined the specific heat of cerium by means of Bunsen's ice calorimeter (*Pogg. Ann.*, 158, 71). He gives the corrected value of 0.04479

for pure cerium, using about 2 grams of metal in a determination. The writer used more than 70 grams of metal in a determination using Joly's differential steam calorimeter, and obtained a corrected value of the specific heat for pure cerium equal to 0.05112. The atomic heat using Hillebrand's value is 6.28; using Hirsch's value, 7.17."

Page 893.—At the end of the paragraph on "Heat of Oxidation" should be added: "Muthmann (Liebig's *Ann.*, 331, 41) gives the heat of oxidation of cerium equal to 1603 cal. per gram."

Top of page 895.—At the end of the second paragraph under the heading "Alloys" should be inserted: "Muthmann and Beck (24b) have described some of the alloys of cerium with zinc, aluminum, magnesium and mercury."

Page 895.—Under "Aluminum Alloys" should be added: "Muthmann (Liebig's *Ann.*, 331, 47) describes the compound  $CeAl_4$ ."

Page 896.—Under "Mercury Alloys" should be added at the end of the paragraph: "Cerium will dissolve in mercury to the extent of about 15 per cent."

ALCAN HIRSCH.

BROOKLYN, December 6, 1911.

### THE MÉKER BURNER.

*Editor of the Journal of Industrial and Engineering Chemistry:*

The every-day tools of the chemist determine to a large extent the character of his work no less than those of the artisan do his. The success or failure of a great research problem in chemistry may depend as much upon the apparatus at hand as upon the imagination and skill of the worker. Possibly the leading fact in the history of science is this: that great trains of discoveries have depended more upon the invention of new apparatus than upon the development of the human brain. After all it is the attention to the details of equipment as well as the personal organization in a laboratory, which brings about perfect results. And so we sing the praises of the Méker burner. With this burner one can do almost the work of the blast-lamp, without the annoyance connected with the use of the latter. The flame is large and intensely hot and the highest temperature, strange to say, is reached at the base of the flame. For analytical work in crucibles it has no equal, nor is there any other device approaching it in excellence. It has no inner cone and platinum ware can be made to receive the full effect of the flame without danger of injury. "There is nothing new in the apparatus—no original idea involved," many a critic would say, "just a Bunsen burner with an abundant air supply and a piece of Davy safety-lamp gauze at the top." But not every burner so constructed will give a flame free from the destructive inner cone, and intensely hot at the base. Every point in the design must be carefully balanced to produce the perfect result. We do not know the inventor or the history of his invention, but a simple inspection of the burner in action indicates that this is no chance discovery, no day-dream or night-dream suddenly made concrete and perfect. Carefully thought out and wrought out by trial and experiment and repeated experiment is the perfectly simple Méker burner.

W. D. RICHARDSON.

### IMPORTS AND EXPORTS OF FARMERS' MATERIALS.

The agricultural interests of foreign countries buy nearly \$100,000,000 worth of American manufactures and other products for use in cultivating the soil, while about \$50,000,000 worth of foreign products are annually imported into the United States for use upon American farms. The foregoing summarizes certain information recently compiled by the Bureau of Statistics, Department of Commerce and Labor.

The exports from the United States of articles required in farming, the larger portion are composed of manufactures, such as agricultural implements, binding twine, barbed wire, and oil cake, while phosphate rock to be ground up and used as fertilizer, and clover, timothy and other grass seeds are among the articles exported in the natural state. In addition to the exports above enumerated as specifically for farm use, there are certain articles for which the agriculturalists of all other countries draw upon the United States but which cannot be classed as distinctively for farm purposes, such as wagons, carriages, traction engines, fiber bags, pumps, etc. In such cases the Bureau of Statistics has no means of determining the proportion of the exports intended for farm use and they are not, therefore, included in the \$100,000,000 worth of merchandise exported for agricultural purposes.

Taking up the principal exports of the class under discussion, agricultural implements head the list, with a probable total of \$40,000,000 in the year about to end, comprised of nearly \$20,000,000 worth of mowers and reapers, \$8,000,000 worth of plows and cultivators, \$2,500,000 worth of threshers, and the remainder miscellaneous farming tools and implements. These agricultural implements are exported to practically all parts of the world where man has adopted modern methods of cultivating the soil: the great wheat fields of Russia, in the vineyards of France, on the coffee plantations of Brazil, in the rice fields of China and Japan, and in all the rapidly developing countries of the New World, including Canada, Argentina, Cuba, and Mexico. The year's exports will range nearly \$8,000,000 to European Russia, \$6,000,000 to Argentina, and about \$5,000,000 to Canada, to about \$1,500,000 to Africa, while to British Australasia and Oceania will be sent a total of nearly \$2,000,000 to other Oceania and Asia about \$1,000,000, and Brazil about \$500,000 worth. Even the great manufacturing countries of Europe, the United Kingdom, Germany, and France, are represented in the year's exports of agricultural implements, the United Kingdom with a total of over \$1,000,000, Germany, \$2,000,000, and France, \$3,000,000.

Fertilizers, of which the exports will be about \$12,000,000 during the year, are chiefly sent to European countries, where long-continued use of the soil has largely depleted their natural fertility and rendered artificial fertilizers a necessity. For American phosphate rock and other fertilizers of that class Germany is the largest market, followed next by Netherlands and the United Kingdom. Another class of materials, used as cattle food, whose exports are considerable includes oil cake and oil cake meal made from cottonseed and linseed. Of these the exports amount to about \$15,000,000 per annum, being exported mostly to various countries of Europe.

Binding twine is another article of importance in wheat-growing countries. Of that article the year's exports will be about \$8,000,000, practically all destined for Russia and other European countries, Canada, and Argentina. American barbed wire exported, about \$5,000,000 per annum, goes chiefly to Canada, Australia, Argentina, British Africa, Mexico, Brazil, and Cuba. In addition to these articles, there are others largely if not exclusively required by farmers, such as windmills, with exports nearing \$2,000,000 per annum; and clover and other grass seeds, over \$1,000,000.

On the other hand, American farmers draw upon other countries for certain of their requirements, most of the imports, however, being articles in their natural state. The largest item shown in the imports of last year were fertilizers, including nitrate of soda, \$17,000,000; guano, manure salts, etc., \$10,000,000; potash salts, \$8,000,000; sulphate of ammonia, used in part as a substitute for manure and in part in the manufacture of alum, \$5,000,000; animals for breeding purposes, chiefly horses, nearly \$3,000,000; clover seed, \$3,000,000; and sugar beet seed, \$750,000.

### WHAT OUR RIVERS CARRY.

Colorado River discharges during an average year into the Gulf of California 338,000,000 tons of mud and silt as suspended matter. In addition to this the dissolved substances in the water include 4,550,000 tons of sodium chloride, or common salt; 3,740,000 tons of Glauber's salts; 4,000,000 tons of lime; 2,400,000 tons of gypsum; and 4,800,000 tons of Epsom salts. In spite of all this dissolved material the Colorado at its mouth is not considered to be a stream of unusually high mineralization for that region of the country. The reason is that the river also carries so enormous an amount of water that the dissolved salts constitute a comparatively small proportion of the total discharge. Other streams in the country contain dissolved salts in greater concentration—for example, the Elm Fork of Red River, in Oklahoma, discharges nearly 1,300,000 tons of common salt annually. Although this amount is not so great as that discharged by the Colorado it is much greater in proportion to the size of the area drained. The discharge of salt from the Colorado is equal to 20 tons annually to each square mile drained by the river, but the salt in Elm Fork of Red River is equal to 1,680 tons per square mile of area drained. The same river discharges annually 177,000 tons of magnesium chloride, 168,000 tons of Epsom salts, 690,000 tons of gypsum, and 54,000 tons of lime. These quantities, too, are considerably greater than those carried in the Colorado in proportion to the size of the drainage area.

Belle Fourche River, at Belle Fourche, S. Dakota, discharges 191,000 tons of gypsum, 79,000 tons of Glauber's salts, and 236,000 tons of Epsom salts. The mud and silt carried in suspension by this river amount to 1,100,000 tons. Milk River at Havre, Montana, discharges annually 41,000 tons of soda; Payette River, in Idaho, discharges 46,000 tons; Salt River at Roosevelt, Arizona, discharges 288,000 tons of salt and 170,000 tons of Epsom salts; and the Rio Grande discharges 245,000 tons of lime and 368,000 tons of Glauber's salts.

The foregoing are a few of the figures of incidental interest presented by Water-Supply Paper 274 of the United States Geological Survey, entitled "Some Stream Waters of the Western United States." The work reported by this volume is, however, of higher practical importance than the above statement would indicate. It is the result of an investigation of the quality of western stream waters made for the purpose of determining their availability for use for irrigation and other purposes. For a water-supply system the quality of the water available determines its usefulness quite as much as the quantity. Some waters contain ingredients that make it impossible to use them for irrigation unless certain precautions are taken in applying them to the land and in draining them off. Certain ingredients in water make it unavailable or destructive if used in boilers and the quality of water used in a manufacturing plant may very largely determine the quality of the product of manufacture. The report just published therefore can not fail to be of material value to the manufacturing and agricultural interests of the West; and the proper consideration of the information that it contains will prevent many costly mistakes in connection with the industrial development of that part of the country.

### CEMENT PRODUCTION TO 1910.

Ten years ago the production of Portland cement for the first time passed the 10,000,000-barrel mark, showing an increase of 2,600 per cent. over the production of 10 years previous, and the giant strides that had been made in the industry were widely remarked. Even this production was small compared with that of the present day. In 1910, according to the report on cement by Ernest F. Burchard, of the Geological Survey, the production of Portland cement reached the enormous total of

76,549,951 barrels, with a value of \$68,205,800. This is equivalent to 12,986,152 long tons, valued at \$5.25 a ton. It is an increase over the output for 1909 of 11,558,520 barrels, or nearly 18 per cent., and an increase in value of \$15,347,446, or more than 29 per cent. This increase alone is greater than the total output of Portland cement in 1900. In addition to Portland cement there was also produced last year 1,139,239 barrels of natural cement and 95,951 barrels of puzzolan cement, a total of 77,785,141 barrels.

The price of Portland cement in 1910 was as low as 73 cents a barrel in some places, the average for the United States being 89.1 cents a barrel. In 1890 the average price was over \$2 a barrel and as late as 1903 it was \$1.24 a barrel.

Mr. Burchard remarks that measured by the capital invested the cement industry is one of the world's three great extractive industries. In capital employed it apparently far outranks the gold-mining industry of the United States, including Alaska, as well as the copper industry. Only coal and iron stand ahead of it.

The principal constituent of Portland cement is limestone, and Mr. Burchard's report summarizes the most important limestone formations in all the States. The greatest of these are found in the eastern half of the United States, where there are enormous limestone deposits. The report is accompanied by a map showing the distribution of these limestones. The areas they cover comprise many thousands of square miles. The map also shows the location of the operating cement plants in the United States.

That the manufacture of cement is an American industry is shown by the fact that while our production last year was over 75,000,000 barrels our imports were only 306,863 barrels. Our exports were 2,475,957 barrels.

The following table shows the healthy growth of the American Portland cement industry and also the decrease in prices:

PRODUCTION OF PORTLAND CEMENT IN THE UNITED STATES 1880-1910.

	Barrels.	Average price per barrel.
1880.....	42,000	\$3.00
1890.....	335,500	2.09
1895.....	990,324	1.60
1900.....	8,482,020	1.09
1905.....	35,246,812	0.94
1909.....	64,991,431	0.81
1910.....	76,549,951	0.89

### WORLD'S PRODUCTION OF COAL.

The total coal production of the world in 1910 was approximately 1,300,000,000 short tons, of which the United States contributed about 39 per cent. According to the United States Geological Survey, it exceeded Great Britain, which ranks second, by over 200,000,000 tons. Great Britain's production in 1910 was less than 60 per cent. of that of the United States, and Germany's was less than half. The increase in both of these countries in 1910 over 1909 was comparatively small, whereas the increase in the United States was nearly equal to the entire production of France and was more than the total production of any foreign country except Great Britain, Germany, Austria-Hungary, and France.

The United States has held first place among the coal-producing countries of the world since 1899, when it surpassed Great Britain. In the 11 years since 1899 the annual output of the United States has nearly doubled, from 253,741,192 short tons to 501,596,378 tons, whereas that of Great Britain has increased only 20 per cent., from 246,506,155 short tons to 296,007,699 tons.

The following table shows the coal production of the principal

countries of the world in 1910, except those for which only the 1909 figures are available:

United States (1910).....	501,596,378
Great Britain (1910).....	296,007,699
Germany (1910).....	245,043,120
Austria-Hungary (1909).....	54,573,788
France (1910).....	42,516,232
Belgium (1910).....	26,374,986
Russia and Finland (1910).....	24,967,095
Japan (1909).....	16,505,418
Canada (1910).....	12,796,512
China (1909).....	13,227,600
India (1909).....	13,294,528
New South Wales (1909).....	7,862,264
Spain (1909).....	4,546,713
Transvaal (1910).....	4,446,477
Natal (1910).....	2,572,012
New Zealand (1909).....	2,140,597
Mexico (1909).....	1,432,990
Holland (1909).....	1,235,515
Queensland and Victoria.....	1,119,708
Italy (1909).....	611,857
Sweden (1909).....	272,056
Cape Colony (1909).....	103,519
Tasmania (1909).....	93,845
Other countries.....	5,236,903
Total.....	1,278,577,812

#### THE MINERAL PRODUCTION IN NORTH CAROLINA DURING 1908-10.

The North Carolina Geological and Economic Survey has just sent to the Public Printer a report on the mining industry in North Carolina for the past three years.

Of the metallic minerals, iron holds first place in the table of production and gold and silver next.

Of the non-metallic minerals, clay products, building stones, mica, and talc are prominent in their production in the order named. There has been a considerable decrease in the production of monazite, owing to the fact that thorium salts are imported cheaper than they can be produced in this country.

The table below gives the production of each mineral during 1908, 1909, and 1910:

Mineral.	1908.	1909.	1910.
Gold.....	\$ 97,495	\$ 43,075	\$ 68,586
Silver.....	668	324	4,888
Copper.....	2,560	29,186	17,845
Iron.....	76,877	107,013	114,237
Garnet.....	4,052	9,188	7,981
Millstone.....			
Mica { Sheet.....	114,540	122,246	193,223
Scrap.....	13,330	26,178	37,237
Precious stones.....	570	479	700
Monazite.....	37,224	46,928	10,104
Zircon.....		250	.....
Talc and Pyrophyllite.....	31,443	77,983	69,805
Mineral waters.....	27,163	20,558	21,389
Stone.....	824,927	850,807	920,027
Sand and gravel.....	2,070	13,358	13,406
Clay products.....	944,317	1,302,611	1,223,704
Miscellaneous <sup>1</sup> .....	109,880	133,642	145,314
Totals.....	\$2,307,116	\$2,783,826	\$2,848,446

#### CHIEF GEOLOGIST, GEOLOGICAL SURVEY.

The Geological Survey announces the appointment of Waldemar Lindgren as chief geologist, to succeed C. Willard Hayes, who recently resigned from the Survey. Mr. Lindgren has been a member of the Federal Survey since 1884 and has been in charge of its investigations in metalliferous deposits since

<sup>1</sup> Includes barytes, sand-lime brick, and kaolin productions.

1907. He is the author of some fifty reports published by the Survey and in addition has contributed between fifty and sixty articles to technical and scientific journals. Mr. Lindgren is a trained mining engineer and has a world-wide reputation as an authority on the geology of ore deposits.

#### NATION'S COAL PRODUCTION.

The Geological Survey has issued its annual coal chart, showing the production of coal by States from the year 1814-1910. The figures relate a wonderful history of growth. In 1814 a total of 22 tons of coal was produced in Pennsylvania. In 1815 the percentage of increase was good but still only 50 tons were taken out. By 1825 100,000 tons were mined in the two States producing. In 1850 the figure had reached 7,018,181 tons. In 1876 it was 53,280,000 tons. At the end of the century it was 269,684,027 tons. In 1905 it was 392,722,635 tons. In 1907, which it was supposed would remain the record year for some time, the production was 480,363,424 tons, but in 1910 the enormous total was reached of 501,596,378 short tons, a production larger by far than that of any other country in the world. The total production since 1814 has been approximately eight and a quarter billion tons.

#### CUBA IRON-ORE PRODUCTION.

The shipments of Cuban iron ores, according to statistics collected by the Geological Survey, show the output for 1910 of 1,417,914 long tons, as against 930,446 tons for 1909, the greatest previous production. In 1900 the production was 446,872 tons. The ores are obtained from deposits near Santiago. Cuba is now the fifth largest producer of iron ore in the world, being exceeded only by the United States, Germany, the United Kingdom, and France.

#### GREATEST IRON PRODUCING REGION IN THE WORLD.

The Mesabi Iron Range in Minnesota produced in 1910, according to the Geological Survey, 53<sup>3</sup>/<sub>4</sub> per cent. of the entire iron-ore production of the United States. The Lake Superior district, including Minnesota, Michigan, and Wisconsin, produced 81<sup>1</sup>/<sub>2</sub> per cent. of our total iron-ore production. Figures from other countries are not yet available for 1910, but this is probably a greater production than the entire year's output for any foreign country:

#### ILLINOIS COAL PRODUCTION.

Illinois has produced more coal than any other State except Pennsylvania, the total tonnage since 1833, when coal mining first began in the State, being 790,333,235 short tons, according to the Geological Survey. Last year the production was 45,900,246 tons and the State stood third, Pennsylvania producing 235,006,762 and West Virginia 61,671,019 tons.

#### COAL EXPORTS IN 1910.

The exports of coal from the United States during 1910, according to the United States Geological Survey, were 13,805,866 long tons, valued at \$41,470,792. Of this quantity 3,021,627 long tons, valued at \$14,785,387, was anthracite, and 10,784,239 long tons, valued at \$26,685,405, was bituminous coal.



The imports of anthracite amounted in 1910 to only 8,195 long tons, valued at \$42,244, and those of bituminous coal to 1,497,709 long tons, valued at \$3,975,561.

#### OIL IN ALASKA.

Petroleum has been found in Alaska, and while there has been practically no production, it is not impossible, according to the United States Geological Survey, that commercial pools may be found. Oil seepages occur on the west shore of Cook Inlet, on the east side of the Alaska Peninsula, and on Controller Bay, all close to tidewater, and hence capable of cheap development.

#### PURE FOOD AND DRUG LAW IN MONTANA.

The Pure Food and Drug Law enacted by the Twelfth Legislature of Montana was put into force on January 1, 1912. The administration of the law is made a duty of the State Board of Health. A food and drug laboratory has been provided for in connection with the Department of Chemistry of the Montana State College at Bozeman. The organization of the laboratory staff is as follows:

W. M. Cobleigh, State Chemist; C. E. Mollet, Director of Drug Analyses; Drury L. Weatherhead, Food Analyst; D. B. Swingle, Bacteriologist.

#### BUREAU OF MINES APPROPRIATIONS.

In the general estimates for appropriations for the fiscal year 1912, which begins July 1, 1912, Secretary of the Interior, Walter L. Fisher, has recommended the following items for the Bureau of Mines:

For the investigation as to the causes of mine explosions, methods of mining, especially in relation to the safety of miners, the appliances best adapted to prevent accidents, the possible improvement of conditions under which mining operations are carried on, the use of explosives and electricity, the prevention of accidents, and other inquiries and technologic investigations pertinent to the mining industry, \$360,000.

For the investigation, analyzing and testing of the coals, lignites, and other mineral fuel substances belonging to or for the use of the United States, \$135,000.

For the investigations into the treatment of ores and other mineral substances, with special reference to the prevention of waste in the mining and utilization of important mineral resources, \$100,000.

For the investigations of the coals of Alaska, with reference to their mining, transportation, and utilization, \$50,000.

#### CIVIL SERVICE EXAMINATION.

The United States Civil Service Commission announces an examination on January 17-18, 1912, to secure eligibles for assistant chemist in the Bureau of Chemistry, Department of Agriculture, at salaries ranging from \$1,200 to \$1,600 per annum.

The usual entrance salary is \$1,200, and only in exceptional cases of unusual qualifications, including considerable valuable experience, will a higher entrance salary than \$1,200 be paid. No assurance regarding promotion is given, but during recent years industrious men of average ability have received during the first five or six years of service promotions aggregating at the rate of about \$100 a year.

Qualified persons are urged to enter this examination as the opportunities for appointment are excellent. Of the thirty-eight persons who passed the assistant chemist examination held in April, 1911, the three highest on the list were appointed at \$1,440 per annum, and twenty-seven others were tendered appointment at \$1,200 per annum.

For detailed information interested inquiries are referred to the U. S. Civil Service Commission, Washington, D. C.

The International Petroleum Commission will hold its meeting January 16-22, 1912, in Vienna, with headquarters at the Engineers and Architects Club.

This commission will be divided into three parts, which will discuss the general and special phases of the petroleum industry. The first division will take up the general methods of analysis of crude oil, benzine, petroleum lubricating oils, paraffins and other products. The second division will discuss the scientific nomenclature, and the third will give its attention to the subject of storage, safety and transportation.

The entertainment committee has made arrangements for receptions, excursions to refineries and oil wells, etc.

Dr. F. W. Frerichs, President of the American Institute of Chemical Engineers, has been elected an honorary member of the Chemical Metallurgical and Mining Society of South Africa.

## BOOK REVIEWS.

*Handboek ten Dienste van de Suikerriet-Cultuur en de Reitsuikerfabricage op Java. Derde Deel. DE FABRICATIE VAN SUIKER UIT SUIKERRIET OP JAVA. Tweede druk. door H. C. Prinsen Geerligs. Director der Filiale Nederland van het Proefstation voor de Java Suiker Industrie, 1911, 512 pages. Price, bound f. 7.50. Edited by the United Sugar Experiment Station of Java. Amsterdam. J. H. de Bussy.*

The first edition of this work by Mr. Geerligs was reviewed in *THIS JOURNAL*, 1, 380. The importance of the volume to the cane sugar industry was recognized in all parts of the world; an English translation, published by the International Sugar Journal, appeared in 1909 and a Spanish translation in 1910. Those who have become familiar with this volume of the hand-

boek series will welcome the appearance of a new edition of the Dutch original.

The scope of the present volume is best stated by Mr. Geerligs in his preface: "While the purpose of this second edition is the same as that of the previous one, certain sections of the book, such as clarification of juice, boiling, and working up of massecuite, have been entirely revised in order to bring the subject matter fully up to date with the growing practice of making only first sugar and of avoiding after-products. The section of the book relating to calculation and interpretation of factory results has also been rewritten in order to include the latest observations of sugar house experience."

Comparing the second with the first edition of Vol. 3 of the

handbook the addition of some 10 new pages is noted in the part relating to the chemical composition and distribution of the constituents of the sugar cane. The second division of the book relating to processes of extraction, utilization of bagasse, defecation, filtration, evaporation, boiling, etc., has been entirely rewritten. The recent development in processes of sugar manufacture is shown by the fact that this part of the volume has been increased from 304-365 pages. This section of the book has also been improved by the introduction of over 40 cuts and photographs illustrating the latest improvements in sugar house equipment. A particularly valuable feature of the new edition is the comprehensive tables and charts which give the results of 130 Java sugar factories for the 1910 campaign.

The typography of the book conforms to that of the other volumes of the handbook and is excellent in every respect.

It is hoped that the English translation of Mr. Geerligs' book may also soon appear in a new edition and include the numerous improvements which have been noted in the original.

C. A. BROWNE.

**The Manufacture of Sulphuric Acid and Alkali.** By GEORGE LUNGE, Volume III, Third Edition, 764 pages. D. Van Nostrand Company, New York. Price, \$10.00.

Professor Lunge's books are too widely and favorably known to require detailed discussion. This third edition of Volume III is devoted to alkali making, and the chlorine industry in so far as they are strictly chemical processes. The electrochemical processes which have developed to such great proportions since the last edition (1896) are not included in this book, but are being treated in a separate volume, IV, by Professors Askenasy and Haber.

The third edition is more complete in its details and introduces much new matter. It is necessarily more bulky than the former editions and still includes many descriptions of processes of doubtful commercial value. It has been the consistent aim of the author, in this as in all volumes, to present as complete a record as possible of development of the industry. Students must know about the failures as well as the successes in an industrial development and will therefore find Professor Lunge's plan of selecting material very helpful.

The volume is divided into three general sections: (1st), Ammonia Soda Process; (2nd), Various Processes of the Alkali Manufacturing; (3rd), The Chlorine Industry. These subjects are treated under chapters including full references and illustrations of apparatus and plant arrangement.

A statistical appendix is a feature of the volume which is of value in establishing the relative importance of the branches of this industry. It is to be regretted, however, that the data here given is from six to fifteen years old. The exportation of chemicals from Great Britain (1738) in 1895 may fall far short of representing conditions to-day and the prices of soda crystals in 1889 have little value except to show that the ammonia soda was dropping.

A carefully compiled index will be greatly appreciated by us.

The volume is well executed and conforms to the style and binding of the previous editions.

The technical chemists have always received the publication of this author with hearty approval, and we should be doubly appreciative for this most recent addition to the works on this great chemical industry.

M. C. WHITAKER.

**Die Metallurgie des Wolframs mit besonderer Beruecksichtigung der Elektrometallurgie.** DR. HANS MENNICKE. 416 pages, 39 illustrations. Price, 17 Marks. Berlin: M. Krayn.

To many it will seem almost incredible that the metallurgy of tungsten requires so bulky a volume, yet the development of this metal has been so rapid within the last ten or fifteen years that it can no longer be classed as "rare." The book is intended primarily as a handbook and since it is the first of its kind it will be welcomed not only by the student but also by steel metallurgists and incandescent lamp manufacturers. Among the chapter headings are: Tungsten Ores; Reduction Methods; By-Products, including Molybdenum, Copper, Tin, Scandium, Titanium, Tantalum and Niobium; Manufacture of Sodium Tungstate, Tungstic Acid, Alloys of Tungsten, Tungsten Bronzes, Tungsten Filaments; Methods for Fireproofing Cotton and other fabrics; Analytical Methods. Although the chapters on tungsten steel and on tungsten filament manufacture are comparatively brief and the information based largely on patent literature, the chapters on the production of tungsten powder are particularly well treated, the author having had considerable experience in this line. A long list of references to patent and journal literature is appended.

C. G. FINK.

**Annual Tables of Constants and Numerical Data (Chemical, Physical and Technological).** Vol. I. Compiled and published by an International Commission, appointed by the Seventh International Congress of Applied Chemistry (see *Jour. Am. Chem. Soc.*, August, p. 105, of the Proceedings).

The first volume of the annual tables will contain all the constants and numerical data, collected by the Abstractors of the International Commission from more than 300 periodicals belonging to Chemistry, Physics and Allied Sciences, Pure and Applied, which were published during the year 1910. The tables will form a volume of about 400 pages, arranged in sections. The wide range of scientific and technical literature which is covered by the annual tables cannot fail to be of value to the scientific men, and will be a valuable addition to every library.

Subscriptions should be filed before January 15, 1911. Subscription blanks, terms of subscription and descriptive leaflets may be obtained from any one of the three American Commissioners: Dr. G. N. Lewis, Mass. Institute of Technology, Boston, Mass.; Professor G. F. Hull, Dartmouth College, Hanover, N. H.; and Professor J. Stieglitz, University of Chicago, Chicago, Ill.

## NEW PUBLICATIONS.

**Analysis, Qualitative.**— By F. DUPRÉ. \$1.50. Berlin, 1911. (German.)

**Casein, its Preparation and Technical Utilization.** By R. SHERER. 2d Ed. 8vo. \$2.00. The Oil and Colour Trades Journal, London.

**Chemical, Some— Problems of To-day.** By ROBERT KENNEDY

DUNCAN. Cr. 8vo., 253 pp. \$2.00. Philadelphia Book Co., Philadelphia.

**Constants, Tables of Physical and Chemical— and Some Mathematical Functions.** By G. W. C. KAYE. 8vo., 147 pp. \$1.25. Longmans, Green & Co., New York.

**Dipping Fluids, The Spontaneous Oxidation of Arsenical—.**

- By A. V. FULLER. U. S. Dept. of Agriculture, Bureau of Animal Industry, Circular, 182.
- Dyeing, Compendium of— Chemistry from the Scientific and Practical Standpoint. By A. SANSONE. L. 8vo., 326 pp. \$3.50. A. Hartleben, Vienna. (German.)
- Essential Oils: Semi-Annual Report of Schimmel & Co., October, 1911. (Translation.) Sm. 8vo., 154 pp. Schimmel & Co., Miltitz.
- Fertilizers. The Manufacture of Chemical Manures. (Translation.) By J. FRITSCH. 8vo., 350 pp. \$4.00. The American Fertilizer, Philadelphia.
- Gas, The Calorific Power of—. By J. H. COSTE. L. 8vo. \$1.50. Chas. Griffin & Co., London.
- Gypsum, Report on the— Deposits of the Maritime Provinces. By WILLIAM F. JENNISON. 171 pp. Canada Department of Mines, Ottawa.
- Inorganic, A Text-book of— Chemistry. (Translation.) By A. F. HOLLEMAN. 4th Ed. 8vo. \$2.50. John Wiley & Sons, New York.
- Inorganic, A Text-book of— Chemistry. By G. SENTER. 8vo. 567 pp. \$1.50. Methuen & Co., London.
- Meat, Text-book of— Hygiene. (Translation.) By RICHARD EDELMANN. 8vo., 392 pp. \$4.50. Lea & Febiger, Philadelphia
- Microbiology. By CHAS. E. MARSHALL. 12mo., 724 pp. \$2.50. P. Bakiston's Son & Co., Philadelphia.
- Molybdenum, Report on the— Ores of Canada. By T. L. WALKER. 64 pp. Canada Department of Mines, Ottawa.
- Oils, Chemistry of the Drying—. By W. FAHRION. 8vo., 298 pp. \$2.50. Berlin, 1911.
- Paint Technology and Tests. By HENRY A. GARDNER. 8vo. McGraw Hill Book Co., New York.
- Paint, The Use of— on the Farm. By PERCY H. WALKER. U. S. Dept. of Agriculture, Farmers' Bulletin No. 474.
- Perfumery, The Manufacture of—. By G. W. ASKINSON. 6th Ed. 8vo., 384 pp. \$1.25. Vienna, 1911.
- Pharmaceutical, The British— Codex, 1911. By PHARMACEUTICAL SOCIETY OF GREAT BRITAIN. 8vo., 1568 pp. \$3.00. The Pharmaceutical Press, London.
- Photography, Applied— in Science and Technics. By K. W. WOLF-CZAPEK. Vol. II. 8vo., 295 pp. Union Deutsche Verlagsgesellschaft, Berlin. (German.)
- Rubber, The Manufacture of— Goods. (Translation.) By A. HEIL AND W. ESCH. \$3.00. Maclaren & Sons, London.
- Silicates, The— from the Chemical and Technical Standpoints. By W. ASCH AND D. ASCH. 8vo. \$4.50. Julius Springer, Berlin. (German.)
- Soaps, Chemistry, Analysis, Technology of the Fatty Acids, Glycerin, Turkey Red Oils and—. By L. UBBELOHDE AND F. GOLDSCHMIDT. \$12.50. S. Hirzel, Leipzig. (German.)
- Soils, The Effect of Soluble Salts on the Physical Properties of—. By R. O. E. DAVIS. 38 pp. U. S. Dept. of Agriculture, Bureau of Soils, Bulletin 82.
- Spectrochemistry, Introduction to the Study of—. By G. URBAIN. \$2.00. Paris, 1911. (French.)
- Sugar, Analyses of— Beets, 1905 to 1910, together with Methods of Sugar Determination. By A. HUGH BRYAN. 48 pp. U. S. Dept. of Agriculture, Bureau of Chemistry, Bulletin 146.
- Technical, A Text-book of Practical Chemistry for— Institutes. By A. E. DUNSTAN AND F. B. THOLE. 8vo., 335 pp. \$1.00. Methuen & Co., London.
- Coal Carbonization. By V. B. LEWES. *Progressive Age*, Vol. 29, 1911, No. 24, pp. 1029-1035.
- Combustion Surface. By WM. A. BONE. *Progressive Age*, Vol. 29, 1911, No. 24, pp. 1037-1043.
- Concrete, Electrolytic Corrosion in Reinforced—. By BURTON MCCOLLUM, et al. *Proceedings of the American Institute of Electrical Engineers*, Vol. 30, 1911, No. 11, pp. 2248-2269.
- Cotton, A Study of— Fibers. By HANS WREDE. *Paper*, Vol. V, 1911, No. 12, pp. 13-15.
- Fertilizers: Inorganic Nitrogenous Plant Foods. By F. J. MACHALSKE. *American Fertilizer*, Vol. 35, 1911, No. 8, pp. 29-44.
- Illuminating Gas, Unstable Hydrocarbons in—. By E. C. JONES. *Progressive Age*, Vol. 29, 1911, No. 20, pp. 865-868.
- Leather, Report on— Analysis. By F. P. VEITCH. *Journal of the American Leather Chemists' Association*, Vol. 6, 1911, No. 12, pp. 565-579.
- Lignite, A Chemical Study of—: The Volatil Constituents. By G. B. FRANKFORTER AND A. P. PETERSON. *Journal of the American Chemical Society*, Vol. 33, 1911, No. 12, pp. 1954-1963.
- Lithopone White. ANON. *Oil, Paint and Drug Reporter*, Vol. 80, 1911, No. 21, pp. 281.
- Nitrate, The Accuracy of— Determinations. By S. SEYDEL AND L. WICHERS. *Zeitschrift fuer angewandte Chemie*, Vol. 24, 1911, No. 43, pp. 2046-2054.
- Oils, The Aromatic Grass—. ANON. *Bulletin of the Imperial Institute*, Vol. 9, 1911, No. 3, pp. 240-253.
- Oils, The Behavior of High-boiling Mineral— on Heating in Air. By C. E. WATERS. *Bulletin of the Bureau of Standards*, Vol. 7, 1911, No. 3, pp. 365-376.
- Oils, Progress in the Domain of Ethereal— and Perfumery Bodies. By F. ROCHUSSEN. *Zeitschrift fuer angewandte Chemie*, Vol. 24, 1911, No. 46, pp. 2185-2195.
- Papers, Testing Fastness to Ink in Writing—. By HANS WREDE. *Paper*, Vol. V, 1911, No. 10, pp. 11-12, 32.
- Plating, The Effect of Addition Substances in Lead— Baths. By F. C. MATHERS AND O. R. OVERMAN. *Advance Copy, Transactions American Electrochemical Society*, Vol. 20, 1912.
- Rubber, The Adsorption of Acids by the Colloids of Dialysed Hevea Latex. By WATSON CROSSLEY. *India Rubber Journal*, Vol. 42, 1911, No. 21, pp. 17-18.
- Rubber, Machines for Mining and Grinding—. By P. HOFFMANN. *Kunststoffe*, Vol. 1, 1911, No. 21, pp. 405-408.
- Sewage, The Different Conditions of— and their Causes. By GEORGE W. FULLER. *Engineering Record*, Vol. 64, 1911, No. 21, pp. 597-600.
- Steel, Chemical and Physical Reactions in— Production, Especially in the Electric Furnace. By T. GEILENKIRCHEN. *Zeitschrift fuer angewandte Chemie*, Vol. 24, 1911, No. 41.
- Steel, Electrolytic— and its Production. By W. RODENHAUSER. *Zeitschrift fuer angewandte Chemie*, Vol. 24, 1911, No. 48, pp. 2289-2302.
- Sucrose, Direct Determination of— in all Products Containing More or Less Reducing Sugars. By H. PELLET AND P. LEMELAND. *International Sugar Journal*, Vol. 13, 1911, No. 155, pp. 616-618.
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- Textiles, Effect of the Bleaching Process on the Weight and Strength of—. By S. H. HIGGINS. *Journal of the Society of Chemical Industry*, Vol. 30, 1911, No. 22, pp. 1295-1298.
- Vanadium, The Determination of— in Vanadium and Chrome-Vanadium Steels. By JOHN R. CAIN. *Bulletin of the Bureau of Standards*, Vol. 7, 1911, No. 3, pp. 377-392.
- Waters, The Chemical Examination of— and Trade Effluents. By J. A. S. MORRISON. *Journal of the American Leather Chemists' Association*, Vol. 6, 1911, No. 12, pp. 544-565.

#### RECENT JOURNAL ARTICLES OF INTEREST TO THE INDUSTRIAL CHEMIST.

Beer, Pasteurization of— in Cask and Bottle. By C. RUEHL. *Journal of the Institute of Brewing*, Vol. 17, 1911, No. 6, pp. 596-612.

## RECENT INVENTIONS.

Reported by C. L. Parker, Solicitor of Chemical Patents, McGill Building, Washington, D. C.

### PROCESS OF AND APPARATUS FOR MAKING CLAD METALS.

U. S. Patent No. 1,004,673, to John E. Monnot, New York City.  
Assignor to Duplex Metals Company, of New York City.

This invention relates to processes of and apparatus for producing clad metals, and it comprises a process of producing clad metals wherein a core or base of ferrous or like metal is given a dense, cohering, impervious coating of an unlike, high-melting, ductile metal by electro-depositing said unlike metal thereon in a molten state; all as more fully hereinafter set forth and claimed.

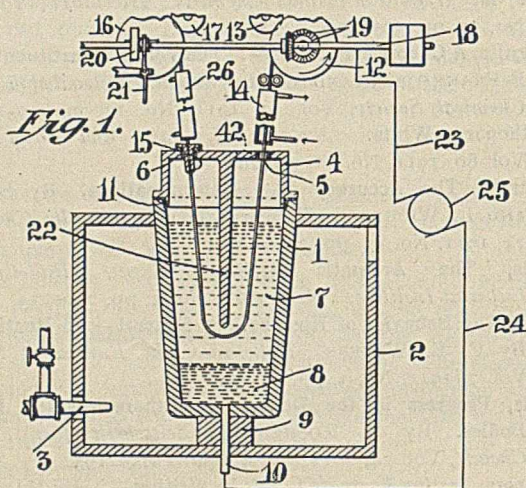
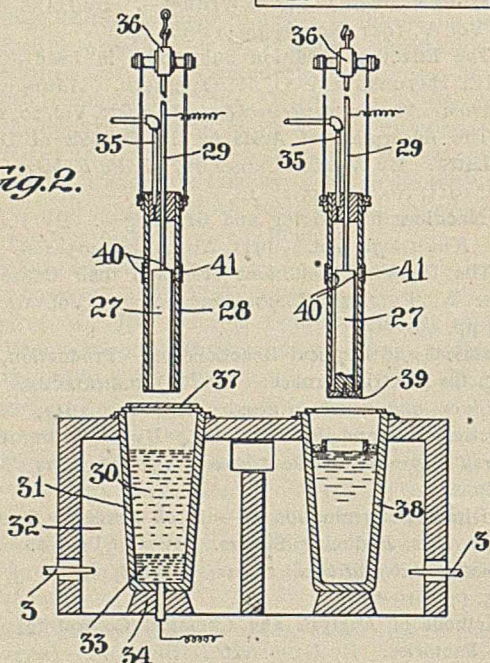


Fig. 2.



The process consists in immersing a high-melting metal body in a solution of a salt of an unlike metal and thereby producing a striking coating of the unlike metal, and then making such metal body a cathode in a bath of fused electrolyte containing a dissolved ductile high-melting different metal, such bath being maintained at a temperature above the melting point of such high-melting metal.

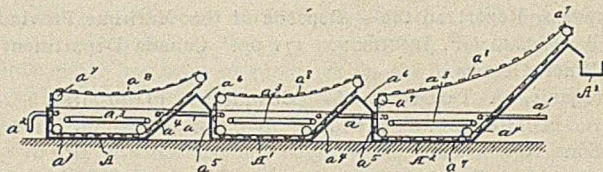
The accompanying illustration shows apparatus in which the process is carried out.

### METHOD OF PRODUCING CRYSTALS.

U. S. Patent No. 1,004,858, to Herbert H. Dow, Midland, Mich.  
Assignor to the Dow Chemical Company, of Midland, Mich.

This invention relates to the manufacture of chemical products of a crystalline nature.

The process consists in producing crystals by conducting the evaporation in two or more successive stages, and mechanically moving the precipitated crystals through the said stages in a



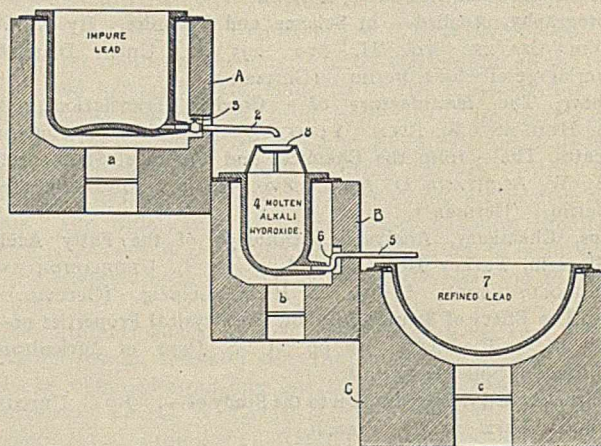
direction passing from a late stage of evaporation toward and through the earlier stages whereby the crystals formed in the later stages are washed in the purer liquor of earlier stages while evaporation is progressing therein.

The accompanying illustration shows apparatus in which the process is carried out.

### PROCESS OF REFINING LEAD AND LEAD ALLOYS.

U. S. Patent No. 1,006,323, to Leland E. Wemple, St. Louis, Mo.  
Assignor one-half to Hoyt Metal Company, of St. Louis.

This invention is a process for the removal of contaminating metals or foreign elements from pig lead and from alloys of lead and antimony, of lead and tin, and of lead antimony and tin, and particularly to the removal of arsenic antimony, or tin, from lead, and to the removal of arsenic from antimonial lead or antimony-lead alloys, and from lead-tin-antimony alloys without the removal of either antimony or tin from the alloys.



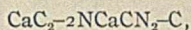
The process consists in bringing the lead, or lead alloy containing impurities, or foreign elements, while in a molten state, in intimate contact with the hydroxid of an alkali, such as hydroxid of sodium, or hydroxid of potassium whereby the foreign elements are attacked by said hydroxid while the metallic lead itself is practically unattacked, and therefore remains in the metallic state, the contaminating metals undergoing a chemical change which is regarded as oxidation or at least partial oxidation, combining with or becoming held mechanically by the hydroxid.

The process may be carried out in the apparatus shown in the accompanying illustration.

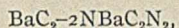
**METHOD OF PRODUCING NITROGEN COMPOUNDS FROM CARBIDS.**

U. S. Patent No. 1,006,927, to Albert R. Frank, of Halensee, near Berlin. Assignor to Societa Generale Per La Cianamide, of Rome.

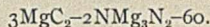
This invention is a method of producing nitrogen compounds from carbids, such, for instance, as cyanamids (carbodiimids), cyanids, nitrids, and other like compounds, as applied to the production of which the process would be illustrated by the formula



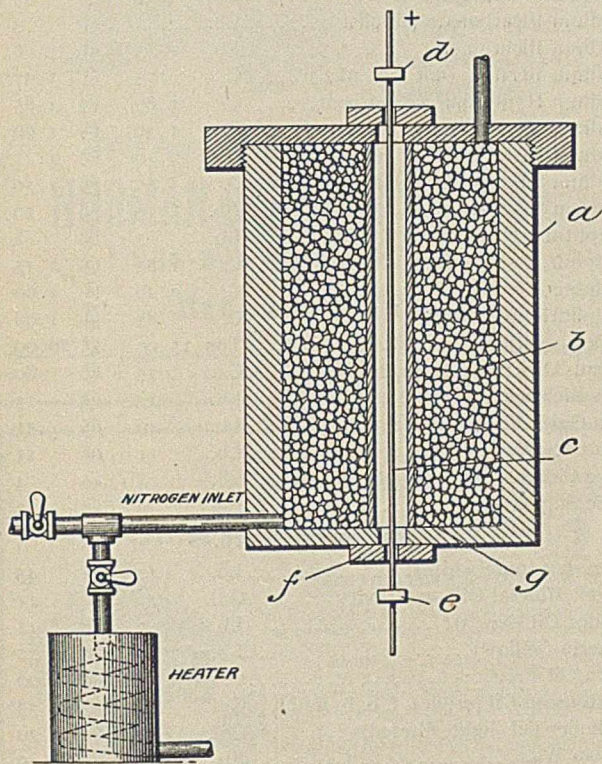
or



or



The process consists in bringing a relatively small part of the carbid mass in a finely divided condition into contact with heated nitrogen to heat said carbid to the reaction temperature, sup-



plying nitrogen as the reaction proceeds, thereby maintaining the temperature and continuing the reaction throughout the mass to be converted.

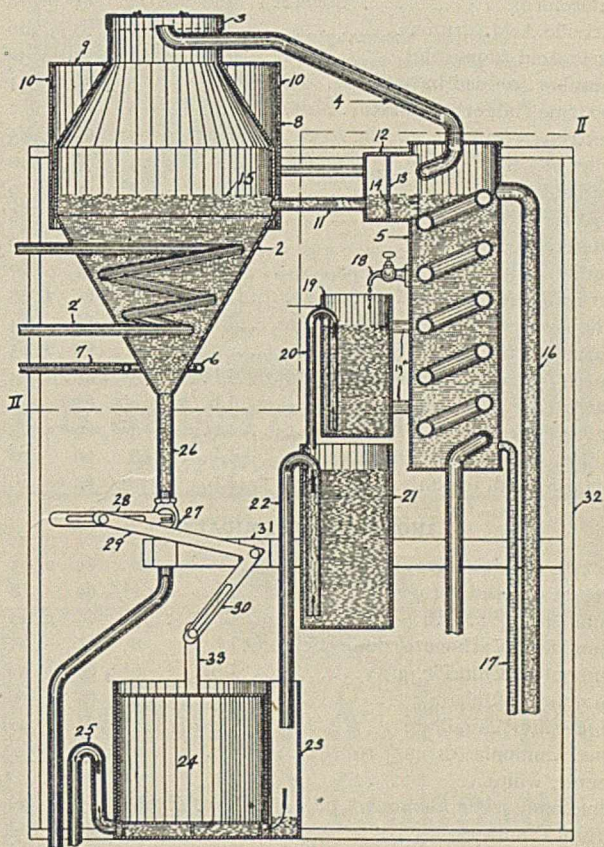
The accompanying illustration shows apparatus in which the process may be carried out.

**A SELF-CLEANING STILL.**

U. S. Patent No. 1,010,508, to W. H. McCune, Vandergrift, Pa.

This invention relates to an apparatus more particularly used for distilling water, with a mechanical arrangement for cleaning at intervals without substantial interruption of the process of distillation or the attention of an operator. It is planned to obviate the usual difficulties of interruption and loss of efficiency in distillation by so designing the apparatus as to clean automatically at regulated intervals.

The accompanying illustration is a sectional elevation, showing the distilling apparatus constructed and arranged in accord-



ance with this invention. The new and automatic feature of this device is shown in parts 19, 20, 21, 22, 23 and 24, and the opening and closing of the flushing valve is accomplished by means of a float. The frequency with which the retort is drained is regulated by the volume of water discharged from the chamber into the tank 19 by drip cock 18.

# MARKET REPORT.

## AVERAGE WHOLESALE PRICES OF STANDARD CHEMICALS, ETC., FOR MONTH OF DECEMBER.)

Full List of Weekly Quotations, see "Oil, Paint & Drug Reporter."

### ORGANIC CHEMICALS.

Acetanilid.....Lb.	20 <sup>1</sup> / <sub>2</sub> @	23
Acetone (drums).....Lb.	13 <sup>3</sup> / <sub>4</sub> @	15
Alcohol, grain (188 proof).....Gal.	2.56 @	2.58
Alcohol, wood (95 per cent.).....Gal.	50 @	52
Alcohol, denatured (180 proof).....Gal.	40 @	42
Amyl Acetate.....Gal.	3.00 @	3.25
Acetic Acid.....C.	1.75 @	1.90
Aniline Oil.....Lb.	10 <sup>3</sup> / <sub>4</sub> @	11 <sup>1</sup> / <sub>2</sub>
Benzoic Acid.....Oz.	11 <sup>1</sup> / <sub>4</sub> @	12
Carbon Tetrachloride (drums).....Lb.	7 <sup>3</sup> / <sub>4</sub> @	8 <sup>1</sup> / <sub>4</sub>
Carbon Bisulphide.....Lb.	5 @	7
Chloroform.....Lb.	23 @	28
Carbolic Acid (drums).....Lb.	19 @	20
Citric Acid (domestic).....Lb.	38 <sup>1</sup> / <sub>2</sub> @	39
Camphor (refined in bulk).....Lb.	— @	44
Dextrine (imported potato).....Lb.	6 @	7
Dextrine (corn).....C.	3.08 @	3.25
Ether (U. S. P., 1900).....Lb.	14 @	20
Formaldehyde.....Lb.	8 <sup>1</sup> / <sub>2</sub> @	9 <sup>1</sup> / <sub>2</sub>
Glycerine (dynamite).....Lb.	16 @	16 <sup>1</sup> / <sub>2</sub>
Iodoform.....Lb.	2.95 @	3.00
Oxalic Acid.....C.	7 <sup>1</sup> / <sub>4</sub> @	7 <sup>1</sup> / <sub>2</sub>
Pyrogallic Acid (bulk).....Lb.	1.25 @	1.48
Salicylic Acid.....Lb.	32 @	34
Starch (corn).....C.	2.00 @	2.15
Starch (potato).....Lb.	5 <sup>1</sup> / <sub>4</sub> @	5 <sup>1</sup> / <sub>2</sub>
Starch (rice).....Lb.	7 @	8
Starch (wheat).....Lb.	4 <sup>7</sup> / <sub>8</sub> @	5 <sup>1</sup> / <sub>4</sub>
Tannic Acid (regular in bulk).....Lb.	68 @	70
Tartaric Acid, crystals.....Lb.	30 <sup>1</sup> / <sub>4</sub> @	30 <sup>3</sup> / <sub>4</sub>

### INORGANIC CHEMICALS.

Acetate of Lime (gray).....C.	2.00 @	2.05
Acetate of Lead (brown, broken).....Lb.	7 <sup>3</sup> / <sub>4</sub> @	8
Alum (lump).....C.	1.75 @	2.00
Ammonium Carbonate, domestic.....Lb.	8 @	8 <sup>3</sup> / <sub>4</sub>
Ammonium Chloride, gray.....Lb.	6 <sup>1</sup> / <sub>8</sub> @	6 <sup>3</sup> / <sub>8</sub>
Aluminum Sulphate.....Lb.	90 @	1.75
Ammonium Vanadate.....Oz.	41 @	50
Aqua Ammonia (drums) 16°.....Lb.	2 <sup>1</sup> / <sub>4</sub> @	2 <sup>1</sup> / <sub>2</sub>
Arsenic, white.....Lb.	2 <sup>3</sup> / <sub>8</sub> @	2 <sup>5</sup> / <sub>8</sub>
Brimstone (crude, domestic).....Ton	22.00 @	22.50
Barium Chlorate.....Lb.	15 @	17
Barium Chloride.....C.	1.90 @	2.25
Barium Nitrate.....Lb.	5 <sup>1</sup> / <sub>2</sub> @	6 <sup>1</sup> / <sub>4</sub>
Borax, crystals (bags).....Lb.	3 <sup>1</sup> / <sub>2</sub> @	4
Boric Acid, crystals.....Lb.	7 @	7 <sup>1</sup> / <sub>2</sub>
Bromine, bulk.....Lb.	25 @	30
Bleaching Powder (35 per cent.).....C.	1.25 @	1.30
Barytes (prime white, foreign).....Ton	18.50 @	22.50
Blue Vitriol.....Lb.	4 <sup>1</sup> / <sub>2</sub> @	4 <sup>3</sup> / <sub>4</sub>
Calcium Chloride.....C.	65 @	90
Calomel, American.....Lb.	91 @	93
Chalk (light precipitated).....Lb.	4 <sup>1</sup> / <sub>2</sub> @	6
Cobalt Oxide.....Lb.	80 @	1.00
China Clay (imported).....Ton	11.50 @	18.00
Feldspar.....Ton	8.00 @	12.00
Fuller's Earth, powdered.....C.	80 @	85
Green Vitriol (bags).....C.	60 @	85
Hydrochloric Acid (18°).....C.	1.15 @	1.55
Hydrochloric Acid (22°).....C.	1.45 @	1.75
Iodine (resublimed).....Lb.	2.60 @	2.65
Lead Nitrate.....Lb.	8 @	8 <sup>5</sup> / <sub>8</sub>
Lithium Carbonate.....Lb.	60 @	63
Magnesite (raw).....Ton	10.00 @	11.00
Mercuric Chloride.....Lb.	82 @	85
Nitric Acid, 36°.....Lb.	3 <sup>7</sup> / <sub>8</sub> @	4 <sup>1</sup> / <sub>4</sub>
Nitric Acid, 42°.....Lb.	4 <sup>1</sup> / <sub>8</sub> @	5 <sup>1</sup> / <sub>4</sub>

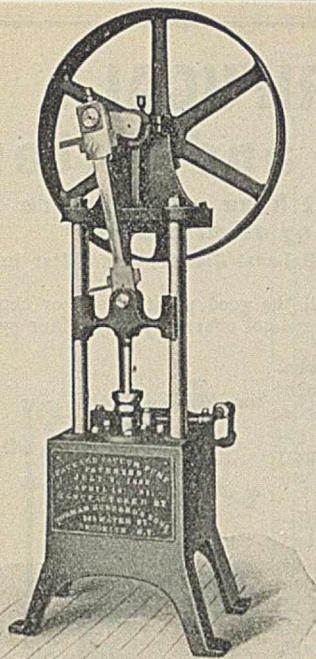
Phosphorus.....Lb.	45 @	1.00
Phosphoric Acid, s. g. 1.75.....Lb.	22 @	26
Plaster of Paris.....C.	1.50 @	1.70
Potassium Bromide.....Lb.	31 @	34
Potassium Permanganate (bulk).....Lb.	9 <sup>3</sup> / <sub>8</sub> @	10 <sup>3</sup> / <sub>4</sub>
Potassium Cyanide (bulk).....Lb.	20 @	24
Potassium Iodide (bulk).....Lb.	2.10 @	2.15
Potassium Chlorate, crystals.....Lb.	8 <sup>1</sup> / <sub>4</sub> @	9 <sup>1</sup> / <sub>2</sub>
Potassium Nitrate (crude).....Lb.	4 <sup>1</sup> / <sub>2</sub> @	5
Potassium Bichromate, 50°.....Lb.	7 <sup>3</sup> / <sub>8</sub> @	7 <sup>3</sup> / <sub>4</sub>
Pumice Stone.....Lb.	2 @	4
Quicksilver.....Lb.	62 @	64
Salt Cake (glass-makers).....C.	55 @	65
Silver Nitrate.....Oz.	36 @	38
Soapstone in bags.....Ton	10.00 @	12.00
Sodium Acetate.....Lb.	4 <sup>1</sup> / <sub>4</sub> @	5
Sodium Chlorate.....Lb.	8 <sup>1</sup> / <sub>4</sub> @	9 <sup>1</sup> / <sub>2</sub>
Sodium Bicarbonate (English).....Lb.	2 <sup>3</sup> / <sub>4</sub> @	3
Sodium Bichromate.....Lb.	5 <sup>5</sup> / <sub>8</sub> @	6
Sodium Bisulphite (not incl. pkg.).....C.	75 @	1.37 <sup>1</sup> / <sub>2</sub>
Sodium Hydroxide, 60 per cent.....C.	1.80 @	1.85
Sodium Hyposulphite.....C.	1.30 @	1.60
Sodium Nitrate, 95 per cent., spot.....C.	2.20 @	2.22 <sup>1</sup> / <sub>2</sub>
Sodium Silicate (liquid).....C.	65 @	1.50
Sodium Stannate.....Lb.	9 <sup>3</sup> / <sub>4</sub> @	10
Strontium Nitrate.....Lb.	7 <sup>1</sup> / <sub>4</sub> @	8
Sulphur, Roll.....C.	1.85 @	2.15
Sulphur, Flowers.....C.	2.20 @	2.60
Sulphuric Acid, 60° B.....C.	85 @	1.00
Talc (American).....Ton	15.00 @	20.00
Terra Alba (American), No. 1.....C.	75 @	80
Tin Bichloride (50°).....Lb.	13 @	13
Tin Oxide.....Lb.	49 @	51
Tin Chloride (36°).....Lb.	11 @	11 <sup>1</sup> / <sub>2</sub>
Zinc Chloride (granulated).....Lb.	4 <sup>1</sup> / <sub>4</sub> @	4 <sup>1</sup> / <sub>2</sub>
Zinc Sulphate.....Lb.	2 <sup>1</sup> / <sub>4</sub> @	2 <sup>1</sup> / <sub>2</sub>

### OILS, WAXES, ETC.

Beeswax (pure white).....Lb.	40 @	45
Black Mineral Oil, 29 Gravity.....Gal.	12 @	12 <sup>1</sup> / <sub>2</sub>
Castor Oil (No. 3).....Lb.	9 <sup>1</sup> / <sub>2</sub> @	11
Ceresin (yellow).....Lb.	9 @	20
Corn Oil.....C.	5.95 @	6.00
Cottonseed Oil (crude), f. o. b. mill.....Gal.	31 <sup>1</sup> / <sub>2</sub> @	33
Cylinder Oil (light, filtered).....Gal.	19 <sup>1</sup> / <sub>2</sub> @	20
Japan Wax.....Lb.	8 <sup>1</sup> / <sub>2</sub> @	9
Lard Oil (prime winter).....Gal.	75 @	83
Linseed Oil (double-boiled).....Gal.	74 @	88
Paraffine Oil (high viscosity).....Gal.	23 <sup>1</sup> / <sub>2</sub> @	24
Paraffine (crude 120 @ 122 m. p.).....Lb.	3 @	3 <sup>1</sup> / <sub>8</sub>
Rosin Oil (first run).....Gal.	36 @	50
Spindle Oil, No. 1.....Gal.	14 @	14 <sup>1</sup> / <sub>2</sub>
Sperm Oil (bleached winter) 38°.....Gal.	76 @	78
Stearic Acid (double-pressed).....Lb.	10 <sup>1</sup> / <sub>4</sub> @	10 <sup>1</sup> / <sub>2</sub>
Tallow (acidless).....Gal.	63 @	65
Tar Oil (distilled).....Gal.	30 @	31

### METALS.

Aluminum [No. 1 ingots].....Lb.	18 <sup>5</sup> / <sub>8</sub> @	19
Antimony (Hallet's).....Lb.	7 <sup>3</sup> / <sub>8</sub> @	7 <sup>5</sup> / <sub>8</sub>
Bismuth (London).....Lb.	— @	1.80
Copper (electrolytic).....Lb.	13.08 @	13.15c
Copper (lake).....Lb.	13.2 @	13.35c
Lead, N. Y.....Lb.	— @	4.45c
Nickel.....Lb.	40 @	50
Platinum (refined).....Oz.	48.50 @	49.00
Silver.....Oz.	54 @	56
Tin.....Lb.	— @	44 <sup>1</sup> / <sub>8</sub>
Zinc.....Lb.	6.22 @	6.37 <sup>1</sup> / <sub>2</sub> c



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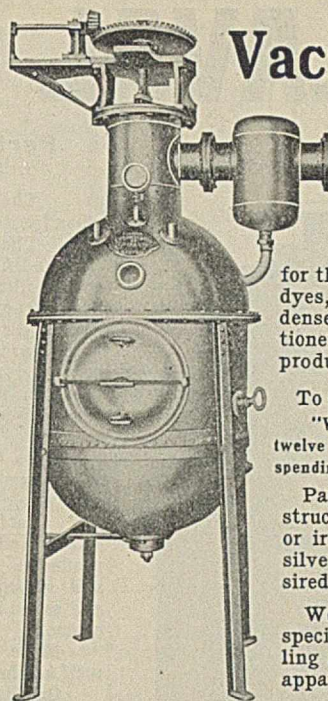
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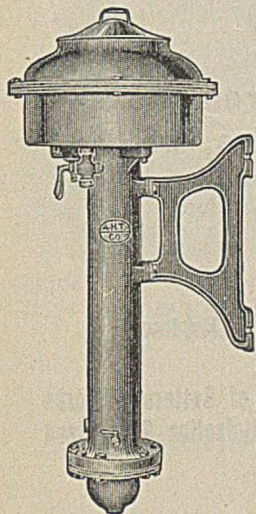
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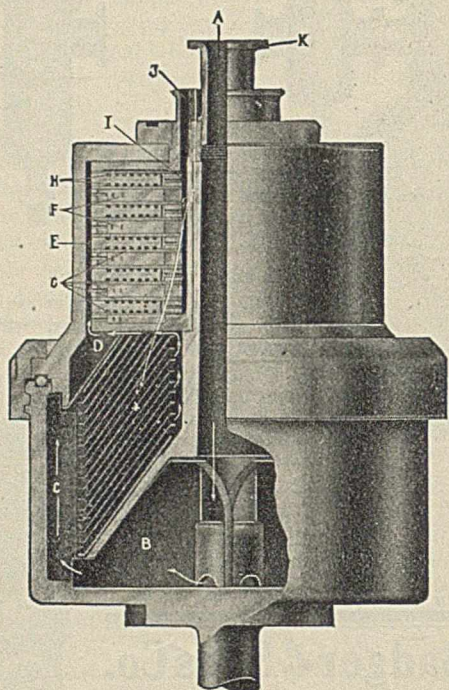
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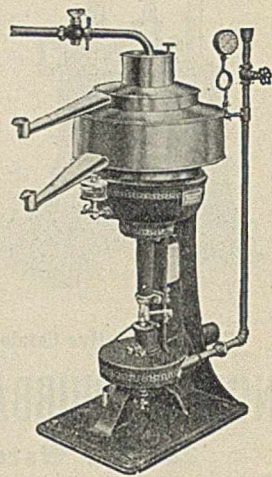
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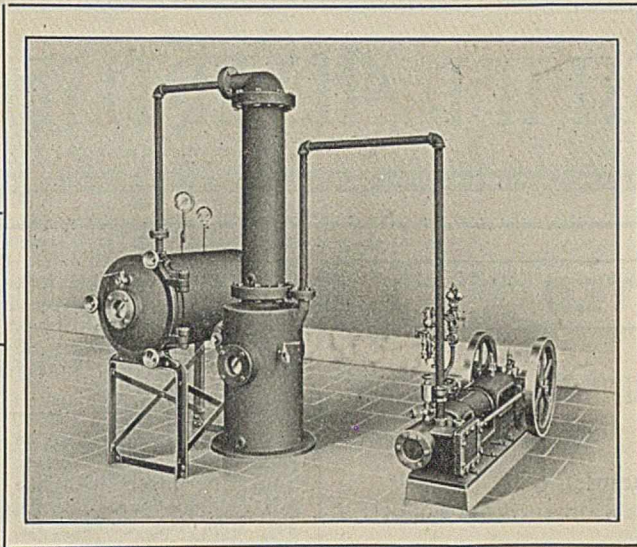
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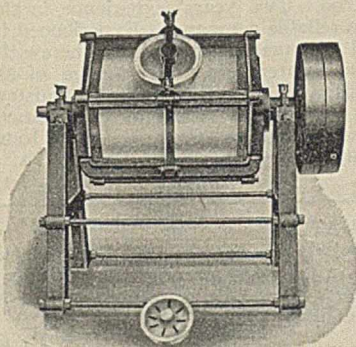


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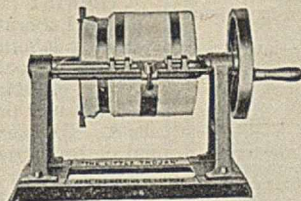
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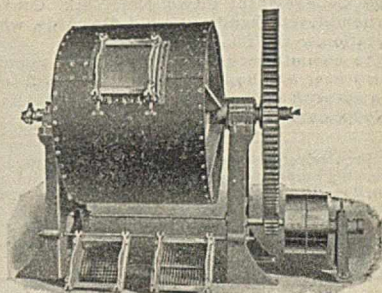


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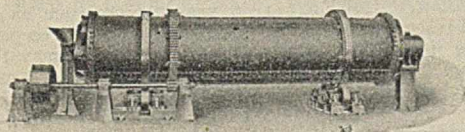
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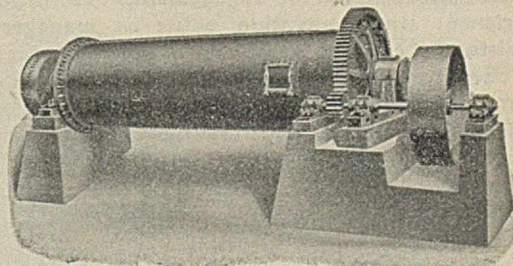
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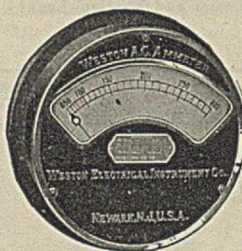
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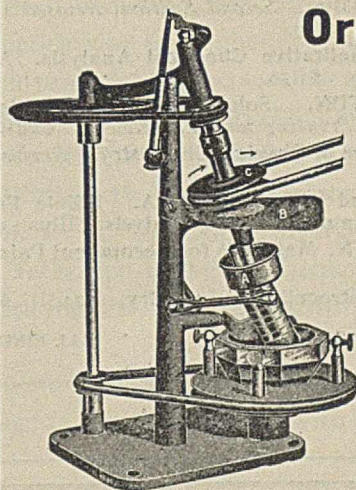
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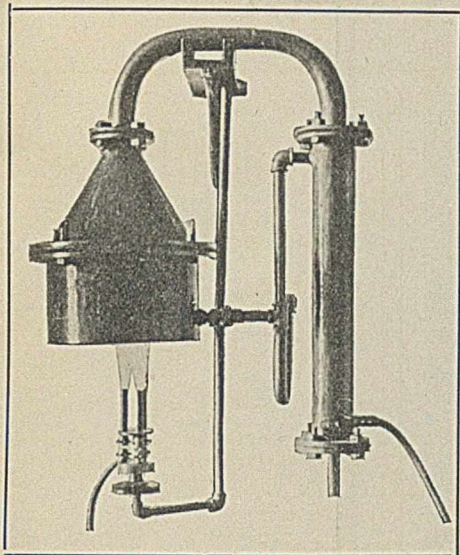
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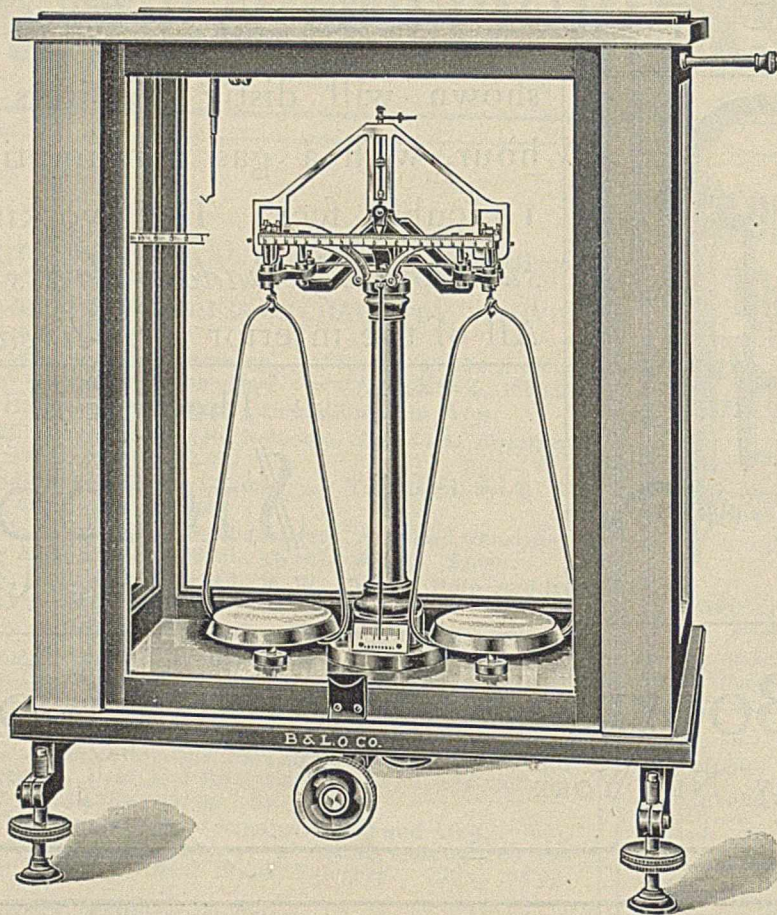
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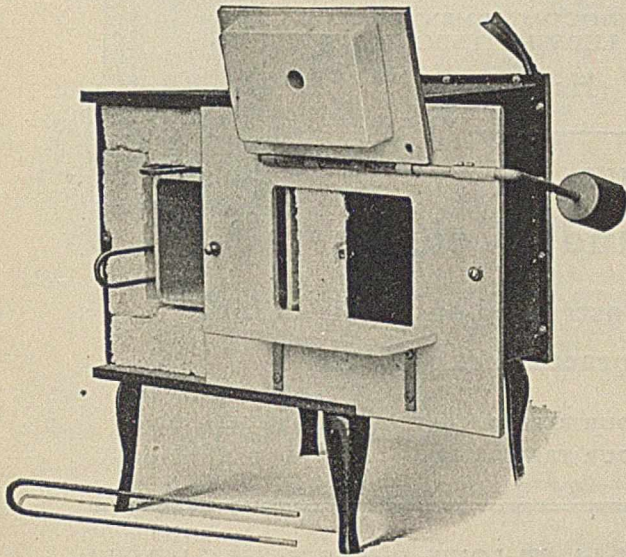
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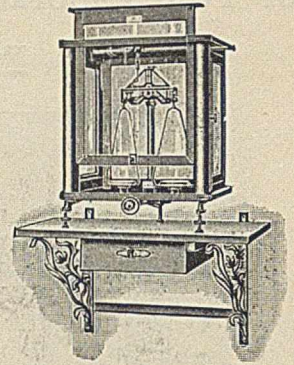
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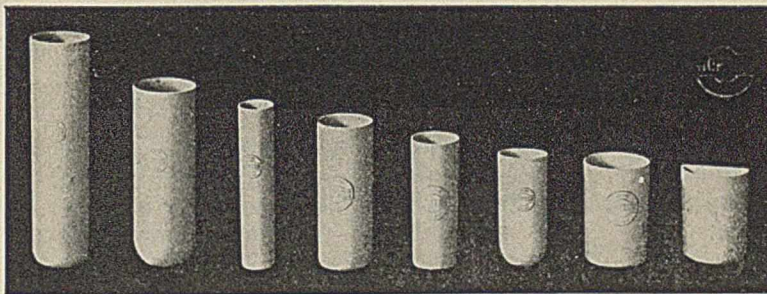
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