The Journal of Industrial and Engineering Chemistry

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EDITORIALS

THE CHEMICAL INDUSTRIES

After the war then what? What is to become of our newly developed chemical industries? These questions seem to be constantly on the minds of many. To-day we may be suffering for want of dyestuffs, potash, pharmaceuticals and a great host of other chemical products—but we are hustling at breakneck speed to remedy matters.

In our great rush in building plants to take care of our home demands there have necessarily been many costly blunders. Plants have been erected and torn down to be rebuilt on the experience of the first attempt. Old processes, old methods have been called into use, only to be discarded again and replaced by newer and better ones. The country is suffering a dearth of trained and experienced chemical engineers, but our country is young. We lacked many of the chemical industries because we have heretofore been able to obtain supplies cheaper abroad. The necessity of developing completely our home chemical industries was not existing before the war. Necessity still remains the mother of invention, and we are a race of inventors.

The development of certain industries is largely a matter of geography—just as the growing of oranges is largely a matter of geography. The location of the necessary natural resources determines the location of industries for utilizing them.

We are peculiarly fortunate in our deposits of coal, sulfur, oil, salt, phosphate rock, etc., etc. We lead the world in their production. Naturally we should lead the world in the industries depending on them and this includes the whole range of chemical industry.

We have had a wall more insurmountable than any tariff wall with absolutely no holes in it, for the last two years, which has resulted in a very acute famine in certain chemical products. This condition has given us a tremendous opportunity for building up some chemical industries we lacked, rounding out the cycle of our industrial activity.

The Exposition of Chemical Industries this fall will show in a measure what American Chemical Engineers have accomplished. It will be a Clearing House for ideas and plans for the future. Already there are more exhibits, and of greater variety, promised than have ever been brought together before. It will, without doubt, be the world's largest and most important exposition of strictly chemical industry, and will indicate clearly the position we shall aim to assume among the Chemical Industries of the world.

OUR RESPONSIBILITIES[1]

Added opportunity means increased responsibility. The holocaust now raging in Europe has forced opportunities upon American chemists and has correspondingly increased our responsibilities. The world in general and our own public in particular, has been thoroughly awakened to the important, though unobtrusive, part played by chemical operations in al-

[1] Address before the Chicago Section of the A. C. S., June 7, 1916.

most every phase of every-day life; chemistry and chemists have been drawn forth into public view and subjected to such inspection and examination as has never before been accorded them. Their past efforts have been scrutinized, their every-day vocations have been studied, and lessons have been learned therethrough that otherwise would not have been learned. In the enthusiasm of a newly acquired knowledge, imagination was by many given free rein; flights into the realms of fancy became the order of the day, and miracles more fantastic than any the alchemists ever claimed, were confidently heralded as accomplished facts, and echoed and re-echoed by our daily press.

PUBLIC CONFIDENCE NECESSARY

The admirable counsel of courage and caution by Professor M. C. Whitaker [THIS JOURNAL, **6** (1914), 794] is as fitting to-day as it was in October, 1914, when it was given; and its value and usefulness must inevitably grow and become greater in the difficult days ahead. Misguided enthusiasm may be as detrimental to progress as habitual and obstinate lethargy. Confidence is of slow growth; it is quickly checked by adverse happenings and by promises unfulfilled. Our industrial progress in chemistry to-day requires, more than anything else, solid and substantial public confidence in us. One unfulfilled, extravagant promise has potentialities for greater retardation upon the growth of that confidence than can be overcome by a dozen moderate, reasonable promises wholly redeemed and completely fulfilled. Constructive work is slow and of limited range of influence; destructive work is rapid and of far-reaching consequences; bad news travels far and fast.

RESPONSIBILITY TO THE PUBLIC

Our first responsibility is to check the super-enthusiastic, to put the bridle of reason upon unconstrained expectation, to bring back the public from its intellectual orgy to the stern reality that progress is not meteoric and that permanent, substantial progress is, like confidence, of exasperatingly slow growth; if all the chemists in the world could accomplish one real "thriller" a year, that would be a most satisfactory and satisfying record.

In spite of much diligent and convincing teaching to the contrary, there are many who still believe in the possibility of perpetual motion; there are many more who still believe that they can get something for nothing, and their misadventures in applying this doctrine to chemical enterprises do more harm over night than many earnest men can undo in many a month and year of hard and serious labor. We can have enthusiasm without extravagance, prudence without pessimism, courage without foolhardiness, caution without timidity, and confidence without bluster. Progress cannot be had without taking chances nor without failure, but there is a reasonable and an unreasonable kind of each, and it is our first responsibility to teach this to the public, however unwilling a pupil it may be or may profess to be.

This is not only our first responsibility but also the most difficult to discharge. Hosts of men and phalanxes of minds are daily contending with untold numbers of problems; the finished work of so many is so frequently being brought to public attention that consideration cannot be given to what may, for lack of a better expression, be termed "reports of progress;" progress alone does not arrest public attention nor excite public comment; conclusive, or at least moderately conclusive, achievements alone can engage the general public.

To bring the nature of this problem nearer home to us: How many of us chemists displayed breathless enthusiasm in the monthly reports that used to come up from Panama setting forth the number of cubic yards excavated? How many of us open our morning papers in haste to find out what the balance in the Federal Treasury was the day before? How many of us watched with nervous interest the daily progress of the bridge over Hell Gate? The only time we paid attention to Panama was when the Canal was opened; Hell Gate Bridge is not yet finished and has come in for but little comment, and the only time we take an acute interest in the Federal Treasury is when tax-paying time rolls around. That being so, by what stretch of the imagination can we expect the public to work itself up into a fit of excitement because some chemist has a new theory of solutions, or another has discovered a new chemical which he burdens with a baptismal appellation that exhausts all the letters in the alphabet and then some, or still another chemist has a new theory for the constitution of the terpenes? The public, however, will give attention to a new incandescent lamp that gives more light for less money, to a water purification process that really purifies, or to a new paint that stays put.

This being the situation, what are we going to do about it? This is a problem that can profitably engage the attention of each and every member of the American Chemical Society. How shall he make his thought bear fruit? At the Urbana meeting of our Society a committee on publicity was appointed to consider this very problem. Let each of our sections appoint a committee of one, two or three members to act for the section in connection with and under the direction of our parent society; when that parent committee has determined, with the help of sectional committees or otherwise, upon the course to pursue, then let all the members of each section and each sectional committee pitch in with a will and make that plan a success; with 49 publicity centers in 32 states of the Union and the District of Columbia, beneficial results ought to be forthcoming. But in this connection we too must bear in mind that progress will not only be difficult but also distressingly slow. We must not expect too much in a hurry and we must not lose heart.

RESPONSIBILITY TO PUBLIC OFFICIALS

Our second responsibility is to those who make our laws and shape our public policies. Questions of this kind involve so many elements that generally a dogmatic or categorical answer cannot be given; more often than not the answers are of necessity compromises and hence embrace both advantage and disadvantage. The best that our lawmakers and our public officials can hope to accomplish under those circumstances, and the most we should expect of them, is to select that course out of the large number usually possible, where the preponderance of advantage to the community or to the country is the largest. It is out of reason to expect no disadvantage at all. That being so, it is our privilege and our responsibility to see to it that in cases involving chemistry all the elements needful to the determining of the correct location of that preponderance are properly placed before those whose duty it is to decide and determine such questions for us. In such presentation of elements we should, to the very best of our ability, present every essential fact or consideration regardless of our own individual political or economic beliefs. Determining facts is one thing; drawing conclusions from facts or basing laws or public policy upon facts, is quite another thing; correct conclusions and correct public policies cannot be reached or arrived at if the underlying facts be wrong or incomplete in any essential. However much latitude we each and all of us do have and take in drawing our own conclusions from facts, that latitude ceases absolutely and instantly the moment we presume to present facts; facts are not always agreeable to ascertain or to state, but they are absolutely essential to a correct answer. No legislation and no public policy can be better than the reasoning which led to them, and this reasoning can be no better than the facts upon which it was based. A statement of facts is essential to all good legislation and all good public policy; such statement is not always easy to make, but it is always a distinctly worth-while thing to provide.

RESPONSIBILITY TO BUSINESS OF THE NATION

Our third responsibility is to the business of the nation. It is our duty to produce as many things in this country, from other things in or out of this country, as commercial conditions make possible. That we are doing so and have been doing so to the best of our ability and information is true beyond question. The one weak point in our armor is our information; that is reasonably complete as to production within the country, but it is unreasonably, unpardonably and woefully incomplete as to our business with other nations. European governments disseminate information better adapted and more useful to their own chemical industrials than does our own Government. Yet our Government is not to be blamed. It has done things in its own way and since we have not told the Government that we want and need better service and have not suggested how such better service could be obtained, why blame Washington? We ourselves are to blame and we should face the situation. The remedy is absolutely and wholly in our own hands, and it has been indifference, lack of time, or sheer indisposition to labor, that has kept us from telling Washington what we should have, in such a way as to make it clear that we really know what we want and are in

substantial agreement as to the remedy. Let no one think that it will be easy to get together a list of items that will satisfy the makers of and dealers in the products of and for our industry. Each member of the Central Committee appointed by the *American Chemical Society* at the Seattle Meeting in September of last year will have to be prepared to sacrifice two hundred or more hours of real hard labor to complete his share in the work; his work can be no better than the work of the various sections of the American Chemical Society, and when in next September these various sections get their allotments of work, as sent to them from that Central Committee, they will have to take their coats off in earnest and work hard to see to it that each have all their suggestions in on time. This word of warning of an impending avalanche of labor is given thus early that there may be no avoidable obstacle to prompt disposition of the work assigned. The Central Committee cannot get down to business until the sections have finished. When normal use of international trade channels is available, the information proposed to be asked for will be of the greatest help in preventing products of foreign make, and which can be made in this country, from gaining such a foothold that sudden drying-up of foreign sources of supply can seriously affect us. But this information when available will largely fail of its purpose if our makers of chemicals do not cut loose from the traditions of the past that have enchained substantially all our manufacturers of all kinds—namely, a sort of contempt for small outputs or tonnages. To be able to make a large number of different things, each of relatively small annual tonnage, or even poundage, is quite as essential to our national business life, as is ability to make large annual amounts of relatively few things. Times have changed and we will have to change with them. Also our industrials of all kinds, inclusive of our chemical industries, will have to learn that quality and price *do* count, and that there is no brand of patriotism that will, for long, withstand the lure of a profit. With individuals, patriotism is said to stop at the stomach; with business it may similarly be said to stop at the pocket-nerve.

RESPONSIBILITY TO EDUCATIONAL INSTITUTIONS

Our fourth responsibility is to our educational institutions. It is inconceivable that in the wealth of information and in the large ability of our university men there is not more opportunity for efficient, effective and valuable coöperation between them and our chemical industrials for the benefit of the nation. It is a knotty problem, there is no denying it, but the prospects of results of almost incalculable, potential national value seem so reasonable that it is worthy of the most serious attention of every chemist, be he university man, technical man, merchant or banker.

RESPONSIBILITY TO AMERICAN CHEMICAL SOCIETY

Our fifth responsibility, and the last I shall touch upon in this paper, but by no means the last of our responsibilities, is to the *American Chemical Society* itself. Our Society places on our desks four times each month publications which can challenge comparison with any similar publications in the world and can come out of such comparison with credit to themselves and honor to the American chemical profession; this is done at an annual cost to each member of about two-thirds the cost of the same service abroad. How is it done? The most of us take these publications as a matter of course, but we are indebted for these magnificent results to an untiring devotion, the unstinting labor, and the most generous loyalty of a relatively small number of our fellow-members; they give of their time, their strength and their ability far beyond their just share. Take love of labor and pride of profession out of the make-up of these men, and the quality and amount of their work would drop to such a point that the net result would not be creditable to us nor to our country. The real hard work of the *American Chemical Society* is performed substantially as a labor of love; that condition ought not be permitted to last one instant longer than it requires to remedy it. We will be asked in September next to provide the remedy. Let us not fail the *American Chemical Society!* She has served us and our country well!

<div align="right">B. C. HESSE</div>

25 BROAD STREET, NEW YORK CITY

ORIGINAL PAPERS

THE THERMAL DECOMPOSITION OF THE PROPANE-BUTANE FRACTION FROM NATURAL GAS CONDENSATE

By J. E. ZANETTI
Received June 10, 1916

Practically every fraction of petroleum and many of its individual constituents, from natural gas to the residues from the distillation of crude petroleum have been submitted to decomposition at high temperatures, so-called "cracking," with one notable exception: the fraction which contains propane and the butanes. The reason is not far to seek; it has not been until the last few years that the separation of this fraction from "wet" natural gas[1] and its use as a commercial product

[1] For description of this process of separation and analyses of various fractions see U. S. Bureau of Mines, *Bull.* **88** and *Tech. Paper* No. **10**.

under the name of "liquid gas," "gasol," etc., has become of commercial importance.

This fraction, which condenses along with the lower boiling pentanes, is usually allowed to escape in the air in the process of "ripening" and is the so-called "wild gasoline" which, because of its low boiling point, is useless for the purposes of "blending" with refinery naphthas. In some places, by further compression of the "wet gas" after the first gasoline condensate has been formed, this fraction is condensed together with ethane and put up in steel cylinders for use as an illuminant or for welding with oxygen.

For the study of the decomposition products, the "pyrogenetic reactions" of hydrocarbons, this fraction furnishes a very satisfactory material. Being

a gas at ordinary temperatures its rate of flow through a heating chamber can be regulated with nicety, no previous vaporizer is necessary as with higher boiling fractions, the formation of lower unsaturated compounds by splitting off of hydrogen as well as condensation of these to aromatics are exhibited, and finally, the *products formed, outside the aromatics, are gases, and any undecomposed propane or butane passes off* thus preventing the necessity of separating the aromatic from the undecomposed aliphatic, as is the case with higher boiling fractions.

The above reasons as well as the fact that this fraction constitutes mostly a waste product have led the writer to start a study of its decomposition products with a view to its possible utilization in other ways than as a lighting material.

A careful search of the literature has failed to furnish any work that has been published on the decomposition of propane or butane. Even Beilstein, that can usually furnish reference to the products obtained by passing vapors of almost any organic compound through "a red hot tube," furnishes no light on the subject.

The plan of the work consisted in:

1—The determination of the composition of the "cracked" gases with respect to "unsaturated" and hydrogen formation.

2—The determination of the amounts of tar formed (aromatics) and its composition.

3—The composition of the unsaturated.

4—The influence of catalyzers.

One difficulty arises with the investigation of the butane-propane fraction: being put up in a cylinder under pressure, on releasing it we have the reverse of ordinary distillation, the temperature being kept constant in this case, while the pressure changes. Under these circumstances the lower boiling fraction comes off first, the composition of the gas altering as the pressure drops. For this reason one must work within certain pressures in order not to change the composition of the gas between too wide limits. The pressure dropped 5 lbs. during the duration of these experiments and the percentage of butane rose, as one would expect. No appreciable difference could be noted either in the "unsaturated" or the hydrogen content, but the amount of tar increased somewhat with the increase of butane.

The apparatus employed[1] consisted merely of a heating chamber, the temperature of which could be accurately controlled, a coil to cool the gases and finally a device to settle the tar "fog." All who have worked with the decomposition of hydrocarbons at high temperature are familiar with the finely divided condition in which the liquid products deposit on cooling the gas, the so-called tar "fog." By far the largest proportion of the tar remains in suspension in this finely divided condition and requires considerable time for settling. On a large scale, as in gas manufacture, this "fog" is removed by various forms of scrubbers, but the writer is not aware of any device for use on a small scale.

[1] For full details see experimental part.

After many unsuccessful attempts to remove this "fog" by filtering the gases through numerous layers of glass wool the principle of a well-known patent[1] was finally adopted. This was the deposition of these finely divided particles passing the gases from the furnace between two conducting plates maintained at a high difference of potential. The results were very successful and whereas only a few drops of tar were obtained per cu. ft. of gas by relying solely on condensation, as much as 10 cc. per cu. ft. of gas were obtained by electrical deposition. There is, of course, no novelty in this device but it is surprising that it has not been employed before in small scale experimentation. The fact that almost all previous work on hydrocarbon decomposition on a small scale has failed to take into account this carrying off of most of the tar in finely divided form by the gas stream may necessitate some revision of earlier work. Thus Norton and Andrews,[2] without any tar depositing, passed the gases from the decomposition of hexane, iso-hexane and pentane through bromine for ab-
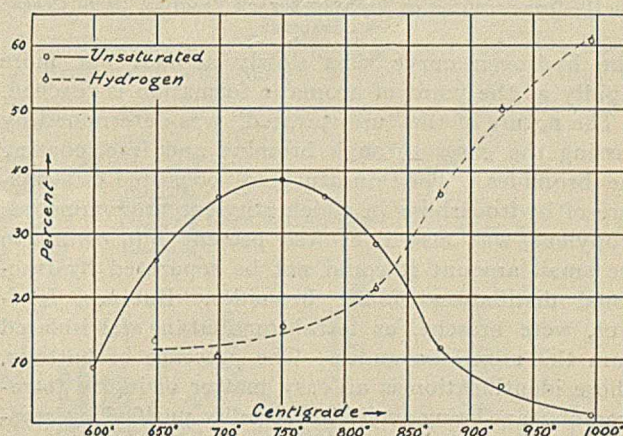
FIG. I—DECOMPOSITION OF PROPANE-BUTANE FRACTION
NO CATALYZER

sorption of the unsaturated, and obtained only a qualitative test for benzol with no mention of higher aromatics; they obtained no benzol from pentane. Haber[3] obtained by the decomposition of hexane "a little benzene." In view of the writer's experience with and without a tar-settling device, and taking into consideration that the higher hydrocarbons of petroleum distillates yield considerable benzol as well as toluol and higher aromatic homologues,[4] as well as the additional fact that the lower hydrocarbons investigated also give the same aromatics, it seems very unlikely that these few intermediate hydrocarbons should not, and the question should remain an open one, at least until the deposition of the tar "fog" should prove it otherwise.

The results of the decomposition of the gas at various temperatures are plotted in Fig. I. The rate of flow through the heating apparatus was kept constant throughout all these experiments, being maintained at 0.45 cu. ft. per hr. This rate was chosen

[1] See F. G. Cottrell, This Journal, **3** (1911), 542.

[2] *Am. Chem. J.*, **8**, 1.

[3] *Ber.*, **29**, 2691.

[4] For a very complete review of the literature on this subject see Egloff and Twomey, *Jour. Phys. Chem.*, **20** (1916), 121.

as being sufficiently slow to permit the gases to be heated to the high temperature. The per cent of "unsaturated" increases gradually till a maximum is reached in the neighborhood of 750°. Beyond 750°, when the aromatic formation begins, the "unsaturated" decrease and become practically nil near 1000°.
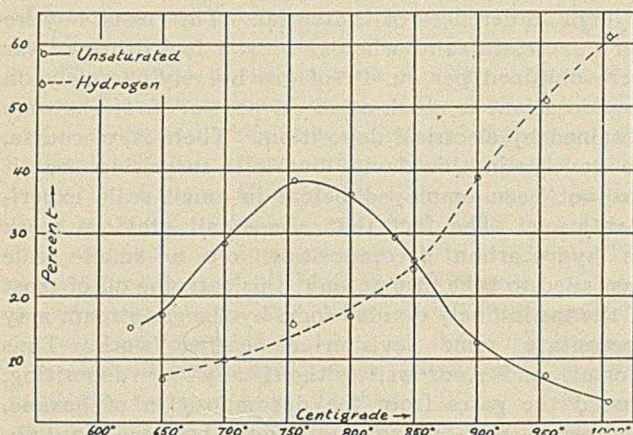
Fig. II—Decomposition of Propane-Butane Fraction, Using Copper as Catalyzer

The hydrogen curve rises slowly at first but more rapidly as the point of aromatic formation is reached.

The nature of the "unsaturated" was determined by passing the gases through bromine and fractionating the bromides. The "unsaturated" consisted of a mixture of hydrocarbons in which ethylene predominated; propylene was also doubtless present but owing to the small amount it could not be separated by fractional distillation of the bromides. Butenes, however, were present, as tetrabromobutane was isolated from the mixed bromides. The presence of butenes, whose identification is an easy matter owing to tetrabromobutane being a solid and readily purified by crystallization, has been reported before as a decomposition product of the lower hydrocarbons.[1]

Benzol, toluol, and naphthalene were shown to be present in the tar. In the intermediate fractions other homologues are doubtless present, but owing to the small amount of material could not be isolated.

It is interesting to note that aromatic formation has taken place here from hydrocarbons of lower carbon content than benzene and that the formation of aromatics is coincident with a lowering of the content of "unsaturated," the principal constituent of which is ethylene.

By using copper as a catalyzer a curve of the same nature is obtained as may be seen in Fig. II. The aromatic formation takes place at the same temperature and the amounts at the rate of 0.45 cu. ft. per hr. are the same.

The use of iron as catalyzer gives an altogether different result (Fig. III). The "unsaturated" content drops suddenly at about 725° and the hydrogen rises very rapidly. Coincident with these changes is the copious deposition of finely divided carbon. There is no tar formation noticeable beyond a very slight amount of bluish "fog." It is of interest to note the rapidity with which the reaction forming carbon and hydrogen is catalyzed above 725°. Below that temperature the decomposition of the gas proceeds in much the

[1] Norton and Andrews, *Loc. cit.*, p. 8.

same manner as in the two previous cases with the exception that the unsaturated content is slightly lower and that the maximum is reached at about 725° instead of 750°. There can be no question of simple surface action. Copper and iron gauzes of the same mesh, cut into equal sizes, were used in both cases and the results are so widely different as to leave no doubt that we are dealing here with a specific catalytic action.

With nickel as catalyzer the results were even more divergent from the decomposition curve of the gas without catalyzer. Here there was practically no "unsaturated" formed, the highest per cent being 4.9 at 630° whereas the hydrogen per cent rose suddenly between 540 and 580° till a maximum of 83.2 per cent was reached at 700°; above that temperature no readings could be taken as the carbon deposited plugged up the tube in a few minutes. No curve is plotted for the decomposition with nickel as catalyzer but the results are given in Table II. The formation of hydrogen increases so rapidly and is so much affected by such slight changes as a slight diminution or increase in temperature as well as the deposition of carbon on the catalyzer that no curve could be drawn through the points obtained though they indicate a rapid decomposition rate. The results obtained are similar to those of Buganadse[1] who by passing crude Russian petroleum over nickel obtained a gas analyzing 72 to 75 per cent hydrogen.

EXPERIMENTAL

MATERIAL—The material used was so-called "liquid gas" bought in the market in a steel cylinder under 100 lbs. pressure. It was obtained by condensation from natural gas. The pressure quickly dropped to 42 lbs. where it maintained itself for a considerable time, falling to 37 towards the end of the experiments presented. At the beginning of this work the analysis of the gas by explosion with about nine volumes of oxygen gave results that indicated it to be composed
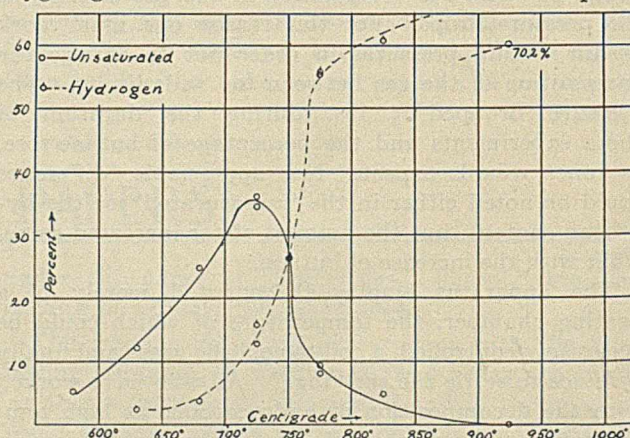
Fig. III—Decomposition of Propane-Butane Fraction, Using Iron as Catalyzer

of propane with 3 per cent butane. Towards the end the proportion of butane increased considerably as would be expected from the decrease in pressure. The results of the analyses are shown in Table I. It is to be noted that the proportion of CO_2 increases with time, being always higher than that required for

[1] *J. Russ. Phys. Chem. Soc.*, **1910**, 195; see also Brooks, Bacon, Padgett and Humphrey, THIS JOURNAL, **7** (1915), 182.

pure propane, but never reaching that required by a mixture that would contain more than 20 per cent butane.

TABLE I—ANALYSES OF GAS DURING COURSE OF EXPERIMENTS

TIME SAMPLED:	FIRST WEEK			EIGHTH WEEK		
SAMPLE No.:	I	II	III	I	II	III
Gas Sample, cc.	9.0	9.3	9.4	10.1	10.2	9.8
Contraction, cc.	30.7	30.7	31.0	33.2	32.6	31.3
Volume CO_2, cc	30.2	30.6	30.7	34.0	33.4	32.1

The gas showed no "unsaturated" when these were determined by shaking with bromine water. When shaken with fuming sulfuric in a gas pipette for the customary 3 min. a decrease in volume of 3 to 4 per cent took place. As it was shown later that this difference between the absorption by bromine and by fuming sulfuric decreased as gases that came from a higher and higher temperature were analyzed, it was evident that this absorption was due to the direct absorption of the propane and butane by the fuming acid. The gas showed no CO_2 or hydrogen.

APPARATUS—The apparatus used is shown diagrammatically in Fig. IV. Since the pressure used was atmospheric, the gas was released from the cylinder into a gas holder and then passed through a meter graduated to 1/1000 of a cu. ft. and provided with a water gauge. The gas passed through two tall drying towers filled with granulated calcium chloride and then into the heating chamber, which consisted of a quartz tube

were led in through the middle openings, the end of the copper coil reaching just below the rubber stopper: they passed out through one of the side openings, the exit tube reaching to within $1/4$ in. of the bottom of the bottle. In front of this exit tube was a copper plate connected by means of a fine wire to one pole of an induction coil. Through the opposite opening passed a carefully insulated wire attached to another copper plate or a piece of copper gauze and connected to the other pole of the induction coil. The gases coming from the cooling coil had then to pass between the two plates before passing out, and if a sufficient difference of potential was maintained between the two plates the "fog" completely settled in the bottle, the gases passing out perfectly clean. The difference of potential sufficient for these experiments was furnished by an induction coil powerful enough to give a 2-in. spark, the current being supplied by a set of six storage cells. This tar separator was also kept immersed in ice water throughout the duration of a determination, the object being to keep the vapor pressure of the tar as low as possible.

The samples for analysis were taken at the exit of the tar separator. When samples were not being taken, a water trap was placed at this exit to prevent air

FIG. IV—APPARATUS USED FOR CRACKING "LIQUID GAS"

1—Gas Cylinder   2—Gas Holder   3—Meter   4,5—Drying Towers   6—Heating Apparatus
7—Cooling Coil   8—Tar-Settling Chamber   9—Induction Coil   10—Storage Battery
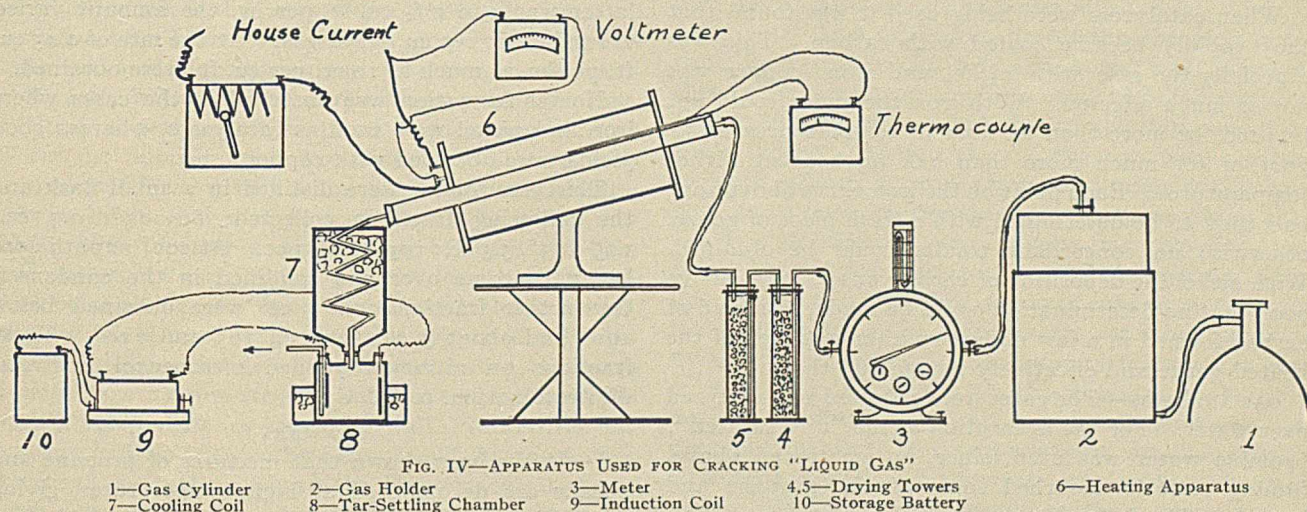
30 in. long, $3/4$ in. internal diameter, glazed on the inside, heated in an electric furnace capable of giving a temperature of 1200° C. The current was controlled by means of a rheostat and the temperature determined by means of a platinum-platinum-iridium thermocouple attached to a millivoltmeter carefully standardized. By means of the rheostat the temperature could be controlled within 5°. Corrections were made from the temperature read off on the millivoltmeter by reference to a correction curve. These varied from 20 to 26° in the range of temperatures employed.

After passing through the heating chamber the gases were led through a copper coil made of 3 ft. of $1/8$-in. copper pipe. This coil was kept immersed in ice water throughout the duration of a determination. But little tar condensed in the coil, most of it passing along with the gases in the form of a fine "fog." To remove this "fog" the gases were passed into a 250 cc. Woulfe bottle provided with three openings. The gases

from passing into the apparatus. When the gases were being absorbed in bromine for the determination of the nature of the "unsaturated," a gas-washing bottle with bromine and water was placed beyond the water trap.

The catalyzers were copper, iron and nickel, 40-mesh wire gauzes cut into uniform sizes 12 in. × 4 in. and rolled so as to fit the quartz tube snugly. These rolls were pushed into the tube far enough so that they would be entirely within the heated area. Copper wool, copper and iron filings were also used, but for the sake of uniformity only the gauzes were used in the experiments presented.

PROCEDURE—The *modus operandi* was as follows: The gas was led through the meter at a measured rate, in the case of these experiments the rate being 0.45 cu. ft. per hr. The rate was determined with a stop watch, the flow of gas being adjusted by slightly opening or shutting the intake cock of the meter. The gas was passed through the cold apparatus to remove

TABLE II—ANALYSES OF GASEOUS PRODUCTS OBTAINED WITH AND WITHOUT CATALYZERS

No Catalyzer			Copper as Catalyzer			Iron as Catalyzer			Nickel as Catalyzer		
Temp. °C.	Unsat- urateds Per cent	Hy- drogen Per cent	Temp. °C.	Unsat- urateds Per cent	Hy- drogen Per cent	Temp. °C.	Unsat- urateds Per cent	Hy- drogen Per cent	Temp. °C.	Unsat- urateds Per cent	Hy- drogen Per cent
600	8.9	....	625	15.0	....	580	5.3	....	510	0.0	0.4
650	25.7	13.0	650	16.9	6.8	630	12.2	2.3	540	2.1	4.6
700	35.9	10.9	700	28.4	9.9	675	24.8	3.8	580	3.0	45.8
750	38.5	15.2	755	38.4	15.5	725	34.5	12.5	630	4.9	54.0
785	35.8	....	800	36.3	16.7	725	36.1	15.8	640	3.9	75.1
825	28.4	21.4	835	29.4	....	750	26.8	27.1	680	2.6	68.4
875	11.8	37.0	850	25.8	24.3	775	9.4	54.8	700	1.9	83.2
925	5.7	50.5	900	12.6	38.9	775	8.1	55.6	...	...	....
995	1.3	62.9	955	7.4	51.0	825	4.8	60.7	...	...	....
...	....	....	1010	3.2	61.0	975	0.0	70.2	...	...	....

the air and the furnace quickly brought up to the desired temperature by putting the full current through it. As the temperature was reached the current was regulated so that the reading of the voltmeter corresponded with the desired temperature. The temperature was closely watched during the experiment and regulated by means of the rheostat. Once adjusted it kept fairly constant within 5°. The gas was then passed through the heated tube for half an hour before a sample was taken for analysis. This time was found to be the minimum necessary to insure concordant results. After the sample of gas had been taken and while it was being analyzed, the temperature was quickly brought up to the next point and the gas passed through for another half hour before the next sample was taken.

When catalyzers were being used it was found that they rapidly became coated with carbon. This was especially the case with nickel and iron; copper was not so much affected. With iron the gauze could not be used for more than an hour at temperatures below 700° or for much more than half an hour at higher temperatures. Each point on the iron curve above 700° had then to be determined with a fresh piece of gauze, otherwise no concordant results could be obtained. With nickel the deposition of carbon was so rapid above 700° that no determination could be made, as a wad of carbon formed in a few minutes within one inch of the heated gauze and effectively plugged up the tube.

GAS ANALYSIS—The gases were collected and analyzed over water. For the absorption of the "unsaturated", bromine water was used, since, as mentioned above, fuming sulfuric absorbed some of the propane and butane. The following are typical results:

Temperature....................	Room	650°	700°	750°	800° C.
Per cent absorbed by bromine.....	0.0	14.2	23,4	36.6	34.5
Additional per cent absorbed by sulfuric acid..................	4.0	4.0	2.1	0.3	0.1

These showed that as long as any undecomposed propane or butane was present some was absorbed by the fuming sulfuric acid, and as the amount of these decreased with increasing temperature, the amount absorbed by the sulfuric acid decreased also. The hydrogen was determined by combustion over copper oxide at 275° C. The results of the analyses are shown in Table II.

THE UNSATURATED—The gases coming between 800 and 850° were passed through bromine till the latter became colorless. After drying, 40 cc. were distilled fractionally in a small distilling flask. After four fractionations the following fractions were obtained: 125–129°, 5 cc.; 129–131°, 12 cc.; 132–142°, 6 cc.; 143–144°, 2 cc. Thick fumes of HBr came over above 145° so that the distillation was stopped. On standing,

the residue in the flask solidified into a mass of dark colored crystals. Drained from about 3 cc. of mother liquor and crystallizing three times from alcohol they crystallized in small, colorless prismatic needles melting at 116°.

The fraction 129–131° gave a specific gravity of 2.30 at 20°.

AROMATICS—No aromatic formation was observed below 700°. At that temperature the "fog" began to make its appearance, at first of a bluish tinge, growing darker with increasing temperature till it became dark brown at about 850°. The tar deposited between 800 and 900°, was dark in color and quite fluid. It showed a specific gravity of 0.985 to 1.000. Above 900° the tar became quite thick and on standing solidified.

The amount formed varied with the rate. If the latter was 1 to 1½ cu. ft. per hr. the amount varied around 5 cc. per cu. ft. of gas. At the rate of 0.45 cu. ft. per hr. as much as 10 cc. per cu. ft. were obtained.

No tar formation was observed in the cases where iron and nickel were used as catalyzers, whereas good yields were obtained with copper.

Thirty cc. of tar were distilled in a small flask and the following fractions collected: 80–125°, 9.5 cc.; 125–173°, 5.5 cc.; 175–200°, 3 cc. At 200° naphthalene began to come over and solidified in the condenser. On further fractionation 6 cc. were obtained below 100° and about 3 cc. between 105 and 110°. These fractions on nitration yielded nitrobenzol and 2,4-dinitrotoluol, m. p. 69° C.

SUMMARY

I—It has been shown that mixtures of propane and butane are decomposed at high temperatures, giving ethylene, butene and other lower unsaturated compounds, hydrogen and aromatics.

II—The percentage of "unsaturated" increases with increasing temperatures to a maximum in the neighborhood of 750° C., then decreases with increasing temperatures. The percentage of hydrogen increases with increasing temperatures. The aromatic formation begins at about 750 and is coincident with an increase of the rate of hydrogen formation.

III—The action of copper, iron and nickel as catalyzers has been studied. Iron and nickel prevent aromatic formation and favor the decomposition of the hydrocarbons to free carbon and hydrogen.

IV—The electrical deposition of tar "fog" in gases from high temperature decomposition has been found to work very satisfactorily for small scale experiments.

Further work upon these topics is now in progress in this laboratory.

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AN INVESTIGATION OF COMPOSITION FLOORING[1]

By R. R. Shively[2]

Received April 17, 1916

In 1867, Sorel[3] found that a very hard material might be produced by mixing magnesia and a solution of magnesium chloride and then allowing the mixture to stand for some time. Sorel concluded that the compound which was formed possessed the following formula: $MgCl_2.5MgO.12H_2O$, or $MgCl.OH.4Mg(OH)_2$. $12H_2O$. The investigations of Sorel initiated considerable inquiry, mostly by French and German chemists, but there has been a diversity of opinion regarding the exact composition of the product.[4]

During the past twenty-five years Sorel cement has been extensively employed with success as a cementing material for so-called "composition flooring." According to European practice, the magnesia, mixed with various "fillers," such as sawdust, sand, terra alba, infusorial earth or asbestos, and colored with different inorganic colors, is made into a "mortar" by the addition of a solution of magnesium chloride; the material is then applied to the floor and leveled, and, after standing several hours, it is carefully troweled. After 8 or 10 hrs. the cement sets and the surface of the flooring is then usually treated with a coat of oil, after which the composition is ready for use.

It has been found that suitable preparations of this type make ideal compositions for floor, base, wainscot and stairs. When carefully prepared, the composition is permanent, fireproof and waterproof, and it can be applied upon wood, concrete or iron base in a plastic form, giving a smooth, continuous surface. The wainscot may also be made from the same material, thus forming a perfect seal between the base and wainscot and giving a floor devoid of cracks. Any color shade may be produced with the exception of pure white, and very attractive effects may be obtained by using borders of different colors, a mosaic design, or a narrow strip of another color around the floor a short distance from the wall. Moreover, the mixture is little affected by heat or cold, having a very low heat conductivity, and the floor produced therefrom does not impart a feeling of coldness to the feet; furthermore, its absorption of water is very small and it is accepted by insurance companies as fireproof.

The composition is usually applied in two coats: an under fibrous coat, about 5/16 in. thick, and an upper coat containing less fiber, from 5/16 to 1/2 in. thick. The under fibrous coat imparts elasticity to the material and renders the floor noiseless and pleasant to the tread. In fact, this composition does not give rise to the fatigue experienced from the use of stone, cement, terrazo or other hard floors. Furthermore, even when troweled to a very smooth surface, this composition does not produce a slippery floor.

These advantages, along with the facts that the composition costs less than tile, marble, etc., and that it will outwear eight or ten applications of linoleum, have made it a very popular floor material in Europe, especially in Germany and England.

Composition flooring of the general type described above was introduced into the United States a short time after it came into favor abroad. American manufacturers have, however, experienced considerable difficulty in the use of Sorel cement. Some have attributed this to the differences in the climatic conditions of Europe and America, the contention being that the sudden changes of temperature encountered in the United States are not experienced abroad, particularly in England and Germany. Others have expressed the opinion that the principal cause of the failure in this country is attributable to the mixtures employed: the assertion has been made that the mixtures in use here have been neither scientifically nor even carefully prepared. Inasmuch as many of the floors examined by the author are as perfect as could be desired, he is inclined to favor the latter explanation. If the trouble is due to the sudden climatic changes, why should one floor be affected and not another in the same locality?

In 1911—that is, at the time when the author became interested in this field—in New York City alone there were at least twenty firms engaged in the composition-flooring business, and the competition was very keen indeed. All of the companies were cutting prices in order to secure business and many of the firms were putting down the cheapest mixtures which they could conceive. In fact, everything possible was being done to minimize the cost of the product, without much regard for the requirements of a correctly made mixture. The components of the mixtures were weighed and mixed by ordinary laborers, so that it was quite impossible for the mixtures to be of a definite consistent composition. Consequently, all of the manufacturers were experiencing decided trouble with their floors. To illustrate, the color frequently "went bad;" some floors would crack or bulge, and at no time could they be assured of a satisfactory floor. As a result, many floors had to be relaid. Then, too, the cost of magnesite was constantly increasing, labor costs were high, and the selling price was so low that it was impossible to make a reasonable profit out of the business.

At the time of this investigation, the red mixture in use by the donors of the Hydrolith Fellowship was supposed to contain:

Ingredients:	Magnesite	Sawdust	Red color	Clay	Terra alba	Infusoria earth
Percentages	50	9	10	11	10	10

Some very good floors were made from this composition and again some could not have been worse. In some cases the floors expanded, while in other cases they contracted and cracked badly. Then, in certain of the floors, the color faded decidedly, so that in a short time the appearance became very unattractive.

EXPERIMENTAL

The investigation conducted by the author at the Mellon Institute of Industrial Research included

overcoming the change in color, obviating the expansion and contraction, and reducing the cost in any legitimate way.

The first experiments carried out were for the determination of the correct amount of magnesite and magnesium chloride to be used, the effect of different fillers, and the results of working samples in various ways. Red colored mixtures were used in this series, which consisted of 1000 samples. All of the materials entering into the compositions were carefully weighed, made up into 500-g. samples and thoroughly mixed before the addition of the magnesium chloride solution, the strength and amount of which were also very accurately determined.

The results of this series of experiments showed that with the fillers employed it was necessary to have present not less than 8 or 9 per cent of magnesium chloride and from 20 to 30 per cent of commercial magnesia. When an extremely hard and durable flooring composition was desired, still larger amounts were required. In samples where more than 1 part of magnesium chloride was employed to 3 parts of magnesia, there was always a tendency for the excess of magnesium chloride to come to the surface.

In a number of experiments, terra alba was the only filler employed with the exception of sawdust; in others infusorial earth was used, while in still others clay was the only filler besides sawdust. These tests were carried out in parallel, *i. e.*, each day samples were made containing the same percentage of magnesium chloride and magnesite with the same percentage of each of the different fillers. This was done for the purpose of studying the relative effect of each filler and to ascertain whether all acted in the same manner. The only noticeable differences were in the samples in which clay and infusorial earth were employed. Since these substances are lighter than terra alba, it was found that the samples covered more space than those in which terra alba alone was used. It was also learned that, on account of the greater bulk of these substances, they hardened more slowly and invariably gave less hard samples than those in which terra alba alone was employed. The terra alba samples hardened first, those containing infusorial earth next, and the ones with clay last. Clay quite appreciably retarded the settling of the cement.

Determinations were made of the least amount of color which could be used to obtain the desired shade. Varying amounts of Venetian red were employed, and it was ascertained that 10 to 12 per cent were necessary. Determinations were also made of the greatest amount of sawdust which could be used to obtain a floor which would not be liable to expand or contract and would yet be of sufficient hardness. It was found that samples containing 6 to 8 per cent of sawdust were very hard and did not impart the proper resistance or elasticity to the floor; but that samples with 10 to 12 per cent, according to the other fillers used, were very satisfactory. Samples containing a higher percentage of sawdust hardened very slowly and those with from 16 to 20 per cent of this material were always quite soft after drying. It was

found, however, that this large amount of sawdust could be used with advantage in the under fibrous coat.

Following the completion of this preliminary work, the author superintended the laying of a floor of 550 sq. ft., following the formula which had given the best results in the laboratory experiments, the composition being:

Ingredients:	Magnesium chloride	Magnesia	Sawdust	Red color	Clay	Terra alba
Percentages.....	10	40	10	12	20	8

The materials were carefully weighed and thoroughly mixed, the dry mixture was then incorporated with the calculated amount of magnesium chloride solution and water was added until the proper consistency was attained. The mixing was continued until all lumps had disappeared and the mixture was then applied over metallic laths. It hardened slowly, but, when hard, it gave a perfect floor which exhibited no physical changes after the expiration of 6 months.

A second series of experiments was taken up in order to determine the effect of different fillers upon the hardness, color and surface of the floor and the effect of various combinations of fillers. It was thought that such an investigation would supply information which would enable one to select the materials giving best results, and that if there was no difference in the results obtained by the employment of various fillers, the cheapest thereof could be selected.

It was found that in samples containing small percentages of magnesium chloride and oxide, the hardening occurred much more slowly than when a greater percentage of the actual cementing material was used. In samples containing high percentages of terra alba, infusorial earth or clay, the difference in the time of setting was quite noticeable: without exception, the samples containing terra alba hardened first, those containing infusorial earth next, and those filled with clay last. In samples with higher percentages of cement, this difference was not so pronounced, but still it could be observed. In all experiments wherein these ingredients were added as the only mineral filler, the terra alba samples were hardest, and there was little, if any, difference between the clay and the infusorial earth samples. It is, of course, a question as to whether this hardness will cause a floor to wear longer than a floor with more resilience which gives, rather than wears. If, however, such hardness is desirable, it can be attained by using greater percentages of cement with the clay and infusorial earth. In none of the samples could any apparent objection be found to any of the fillers in reference to their effect on the surface. All samples, whether containing but one filler or all three, appeared to give the same finish. It was found, however, that when the samples contained a large percentage of clay or infusorial earth, it was necessary to use more color.

Regarding the amount of magnesite and magnesium chloride necessary, the results of this series of experiments showed clearly that never more than 40 per cent of magnesite or 12 per cent of magnesium chloride could be employed. In tests where more than these percentages were used, a very hard floor was obtained, whereas the smaller amount gave a sufficiently hard

composition. The only samples in this series of experiments which showed a tendency to develop physical defects were those which contained high percentages of magnesite and magnesium chloride. In every case when the percentage of magnesium chloride was more than one-third that of the magnesite, there was a tendency for the excess chloride to come to the surface.

In a third series of experiments an effort was made to study the relation of the mass of composition to the volume of magnesium chloride solution sufficient to make a mixture of the proper consistency for laying. This was considered important, since, in preparing the mixture, the workmen invariably used a sufficient amount of magnesium chloride solution of a certain degree Baumé regardless of the actual magnesium chloride contained therein. For example, it was customary to use a 21° Bé. solution in summer and an 18° Bé. solution in winter. Accordingly, solutions of these strengths were used in the experiments. The solid constituents of the mixture were made up to 250 g. and to these samples enough magnesium chloride solution was added to obtain the proper consistency for laying. From the amount of solution required, the actual weight of the magnesium chloride used was calculated, and from this the percentage of magnesium chloride in the finished product was computed.

The average amount of magnesium chloride solution required for 32 samples of this composition was 178 cc. with a maximum of 225 cc. and a minimum of 160 cc. The average amount of magnesium chloride added in per cent was 11.1 for the 18° Bé. solution and 13.3 for the 21° Bé. solution. The maximum percentage required for the 18° Bé. solution was 13.8 and the minimum 10.2. It was found that mixtures containing a large amount of clay or sawdust required enough of the magnesium chloride solution in mixing to bring the percentage of magnesium chloride above the 1:3 limit; in such samples it was found that there was always a tendency of the floor to warp on standing.

These experiments demonstrated that, in order to obtain the best results, the strength of the magnesium chloride solution should be known and just sufficient of it should be added to secure the correct percentage of magnesium chloride with enough additional water to obtain the proper consistency.

In connection with the third series of experiments, slate was used as the coloring material, but it was found to give very uncertain results. Talc was also tried as a filler and it was found that, when used in moderate amounts, it aided greatly in giving the floor a luster when properly troweled; moreover, the tests showed that it slightly decreased the absorption of the material. When too much talc is used, however, it constantly comes to the surface, especially after the sample is washed and allowed to dry slowly. Some experiments which were made to determine the largest amount of sawdust that could be used, showed that the greatest that could be safely and successfully employed was 15 per cent. In these samples, it was necessary not to have an excess of magnesium chloride,

for bad warping occurred when too much was used. Sand was also experimented with as a filler and it was found that a very hard composition resulted with a large percentage of sand. While it is difficult to obtain sand of such fineness as to permit of the production of a very smooth surface, it aids in producing a hard durable floor.

The results of ten other experimental series may be briefly noted. Efforts to find a substitute for Venetian red established the fact that while hematite of desired color would cost, when using 25 per cent of color, at least two-thirds as much as Venetian red, the color obtained was one that did not meet with general approval and twice as much was required to produce the same color intensity as that imparted by the prepared color. In connection with reducing the cost of the color, about 30 samples were made in which aniline dyes were used: none of these retained their color for any length of time.

In one series in which varying amounts of sawdust and asbestos were used, it was found that when used in too great quantities, asbestos interfered somewhat with the troweling; the results obtained indicated that not more than 5 per cent of asbestos should be employed, since greater amounts interfered with the production of a good surface.

A series of experiments was carried out for the purpose of studying the effect of adding oil to the composition and then comparing the absorption of samples made from different fillers. The samples were all made on the same day and an attempt was made to trowel each sample in the same manner. In the samples in which oil was used, the oil was thoroughly mixed therewith, and then these samples were brought to the proper consistency by the use of a calculated amount of concentrated magnesium chloride solution and water. The samples were allowed to stand one week and then a part of each sample was used for an absorption test. The samples were weighed, immersed in water for 24 hrs., and reweighed. These samples were then placed aside for 4 mos., weighed, immersed in water, and then reweighed. The absorption was not proportional to the amount of oil used, but, after the samples had stood for some time, they all absorbed more water, with the exception of those in which linseed oil was used. These showed no indication of oil on the surface, while those in which hard mineral oil was used possessed an oily appearance. The results indicated that the magnesium linoleate acted as a binder between the particles, thus lowering the absorption. The samples in which a small amount of oil was used were sufficiently hard; but, when a large amount of oil was employed, several months of standing were necessary for the samples to become satisfactory in this respect. The samples without oil faded badly after the absorption test, while little change could be observed in the color of the samples which contained oil. Invariably, the samples containing oil set more slowly.

Experiments were conducted with the object of reducing the cost of the composition by employing other materials with reduced amounts of magnesite.

Portland cement-magnesite mixtures had a bad color, absorbed a large amount of water and shrank on drying; in general, they resembled more closely the cement than the composition floors. Plaster of Paris gave compositions which dusted badly, lacked hardness, and set too quickly. Likewise, gypsum which had been heated to 400° C. gave samples which set too quickly and were no harder than the samples made from ordinary plaster of Paris. Samples prepared with the addition of a cement made of molecular quantities of magnesia, magnesium sulfate and plaster of Paris, said to form a white, quick-setting strong cement, also set quite quickly and were very hard, but the material rubbed off easily.

In the effort to find the material which would be a suitable binder for fillers or one that could well be used as a surface material, a number of experiments were made with asphalt, "Redmanite," gelatin-glycerol mixtures, "Galalith," viscose and vegetable resins. No results of commercial importance were obtained.

Finally, it may be mentioned that in 1912 a section of a floor, 12 ft. by 16 ft., was laid in a factory room which was in constant use; this floor was put down in the center of the room and was subjected to a great deal of wear. It was scrubbed every day with alkaline cleansing material and hot water. At the end of 7 mos. the floor was reported to be entirely set and as good as terrazo. Further experience warranted the conclusion that the flooring, the composition of which was identical with that laid in 1911, noted above, was more desirable than a floor constructed from a hard, nonpliable, highly conductive substance like terrazo.

PRACTICAL CONCLUSIONS

The successful working of composition flooring of the type considered does not depend entirely upon the chemical composition, but also upon the mechanical manipulation. The exercise of constant care is necessary in laying the floor and consequently only skilled workmen should be employed in practice; in particular, composition flooring should not be over-troweled, and the necessary precautions should always be taken in laying floors over surfaces which are subjected to heat. The composition should be dependent upon the uses to which the floor is to be put; but a formula is given which, when suitably modified along the lines indicated and expertly used, will comply with the conditions usually encountered in practice.

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EMULSIFIED OR CUT PETROLEUM

By Charles K. Francis

A large quantity of emulsified or "cut" oil is produced from many wells in the Cushing pool of Oklahoma. Some of the pipe lines have refused to accept this oil claiming that it contains too much sediment which does not separate well on standing, and the refineries complain that it has a bad action on the stills.

It is the author's intention to present part of the details of an investigation which was undertaken for the purpose of determining the nature of these emulsified oils and to suggest methods for properly treating them.

The oil selected for special study was produced from the Bartlesville sand and had a specific gravity of 0.8279 (39.1° Bé.). After 10 min. in the centrifuge, which made 1450 r. p. m., the total residue appeared to be 1.6 per cent. Subsequent examinations proved that this residue contained but a very small amount of solid matter, the larger part being water. The solid substance consisted of minute particles which, when measured under the microscope, were found to vary from $1/400$ mm. to $1/20$ mm. in diameter, or from 98 millionths to 19 ten-thousandths of an inch.

A portion of the oil was filtered and washed with casing head gasoline, gravity 84° Bé. This treatment left an insoluble residue, equivalent to 0.505 per cent, consisting of 0.277 per cent nonvolatile mineral matter and 0.228 per cent organic and volatile substances.

Analysis of Residue from Cut Oil from the Bartlesville Sand

Substance	Per cent Based on	
	Residue	Oil
Sand	2.69	0.007
Gypsum	13.35	0.037
Calcium chloride	7.60	0.019
Magnesium chloride	0.91	0.002
Salt	75.45	0.209
Total	100.00	0.274

There is but a trace, less than 0.5 per cent, of water in this oil, so the quantity of salts present is more than sufficient to saturate it.

The common method for testing an oil for suspended matter, at the well or while in the stock tank, is to place some of it on a piece of glass and then look through it while holding it to the light. An experienced gauger can by means of this simple method quite accurately estimate the quality of an ordinary oil, but the test gives no positive information concerning the nature of any sediment which may be detected. Sometimes particles of water and gas may be thought to be mineral matter; indeed, such mixtures often have the appearance of suspended mineral matter. Oils holding water and gas in suspension soon settle, whereas oils truly cut or emulsified have been allowed to stand for weeks without clearing satisfactorily.

The nature of any substance held in suspension in petroleum may be quickly determined by means of a microscope. Good oils are represented in Figs. I and II—the predominating objects are water and gas; but, under the same magnification of the microscope, a cut oil, that is, emulsified, will appear as shown in Figs. III and IV. The mineral matter in these oils is chiefly common salt. In Fig. V several of the more prominent crystals of this substance are indicated by an X beneath. This photograph illustrates how the material shown in Fig. III changed through evaporation while exposed to the air for a few minutes, and brings out the character of the mineral matter. In the lower right-hand corner of the photograph a crystal of salt may be seen encased in a globule of water.

The analysis and the microscopical examination confirm each other as to the nature of the solid material present in this oil. The amount of mineral residue,

o.2 per cent, is surprisingly small when compared with the 1.6 per cent found by means of the centrifuge, which is so extensively used for making this test. A residue of o.2 per cent would not be sufficient to cause the oil to be rejected by the average gauger representing the pipe line, but the chemical properties of the salts are very serious if their behavior when

Many experiments have been made to devise a method which will cause emulsified petroleum to separate. A patented process has been described[1] by A. T. Beasley which is being used in California, consisting of a combination of electricity and hot water. "The action of the electricity is to create a very strong electrostatic field in which the infinitely small particles
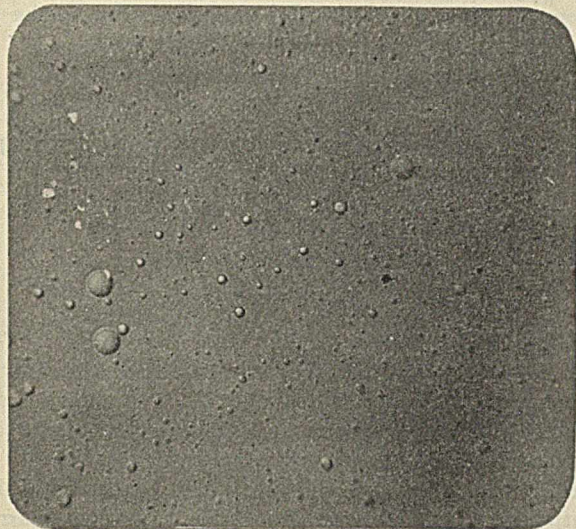
FIG. I × 83

FIG. II × 372

GOOD OIL—SHOWING WATER AND GAS
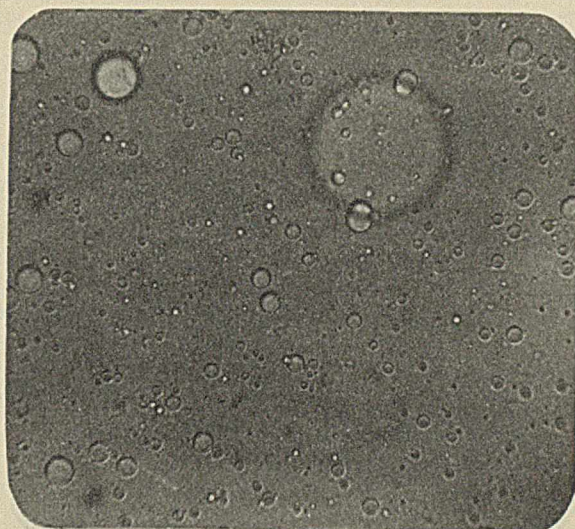
heated is considered. The well-known action of chlorides, especially magnesium chloride, in boiler waters may be used to illustrate this:

$$MgCl_2 + H_2O = MgO + 2HCl$$
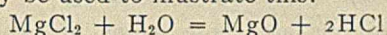
Hydrochloric acid (HCl) may be formed in the same way, certainly to some extent, when calcium chloride is heated. The scale forming and corrosive action

of water will be formed into chains from electrode to electrode. If the voltage be sufficiently high the fine films of nonconducting oil between the water particles will be punctured, bringing the entire chain together in the form of one comparatively large drop. This drop is now free water and is deposited." The system is composed of three units—a water trap,
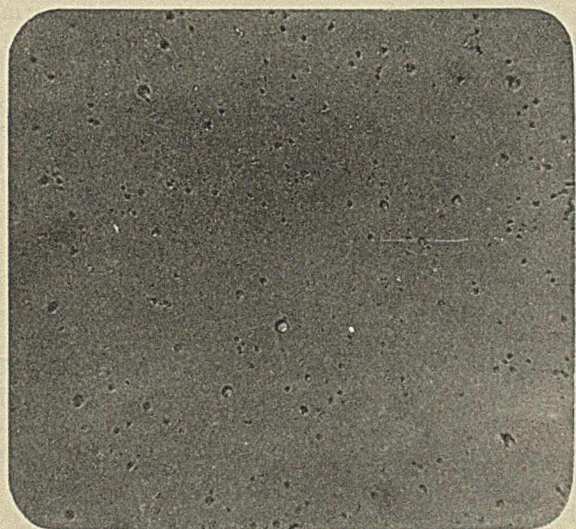
FIG. III × 83

FIG. IV × 372

EMULSIFIED OIL—SHOWING WATER AND MINERAL MATTER
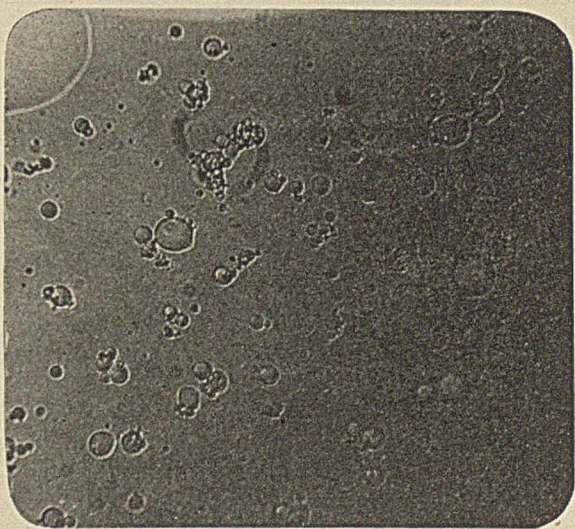
of the group of salts reported in the analysis has often been a grave matter in steam boilers; so then, oils containing them would in a short time seriously injure the condensing tubes and the stills in the refinery. On a basis of 4000 bbls. of oil treated each day, the amount of salt accumulating daily would be 2400 lbs., about 240 lbs. of calcium chloride and 24 lbs. of magnesium chloride.

an electrical treater, and an automatic oil and water separator. The oil in the treater is maintained at a temperature which may vary with the viscosity of the oil and the weather conditions, by means of a steam coil. No doubt the heat has much to do with the breaking down of the oil globule when this method is used.

[1] Oil Age, April, 1911; see Redwood, 1, 318.

In the Oklahoma fields it is the common practice to treat the emulsified oils with water, the temperature of which is maintained at 80 to 125° F., depending upon the quality of the oil and the weather conditions. This treatment is conducted in a tank (Fig. VI), placed between the flow tank and the stock tanks. It is simply one unit in addition to the usual
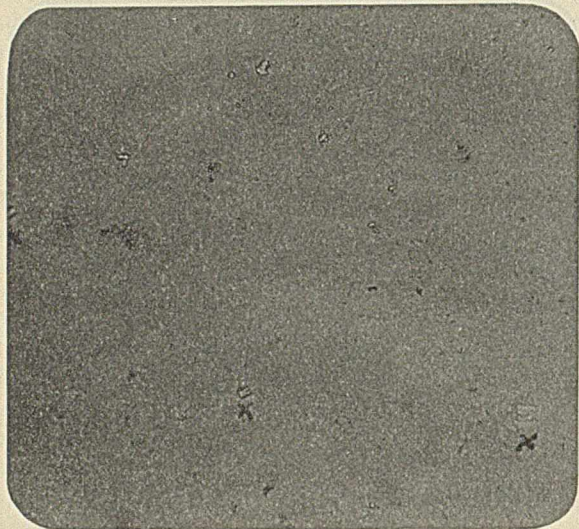
FIG. V × 83
SALT IN EMULSIFIED OIL

equipment on a lease. The illustration indicates all the essential features of this method. The flow of oil is continuous, being controlled so that a constant stream is distributed upward through the warm water, passing out to the stock tanks.

Many samples of crude oil from the Cushing field, which have been examined here, have had an initial boiling point of 130° F.; hence this treatment, even

FIG. VI—TREATING TANK FOR EMULSIFIED OIL

when conducted at the higher temperature, should not seriously affect the gravity of the oil.

There is a large quantity of salt in some wells in the Cushing pool, and it is this substance, as noted above, which is mainly the cause of the emulsified oils. In the warm water treatment, the slight ex-

pansion causes the breaking of the oil film; then the fine particles of salt are dissolved by the extra water present and carried to the bottom of the stock tank within a short time after the treated oil is delivered there. Some salt is removed by the water during the treatment, consequently the water in the treating tank should be renewed occasionally or it may become saturated with the salt and have no action on the oil being passed through it.

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THE DECOMPOSITION OF HYDROCARBONS AND THE INFLUENCE OF HYDROGEN IN CARBURETED WATER GAS MANUFACTURE

By M. C. WHITAKER AND E. H. LESLIE

(*Concluded from our previous issue*)

THE DECOMPOSITION OF PARAFFIN HYDROCARBONS

Figs. 9 to 14 indicate the proportions of the various components obtained when the oil was decomposed alone. Fig. 9 shows that at 621° C. the gases are composed of about 10 per cent hydrogen at all oil rates above 5 cc. per minute. The reactions which contribute to the formation of this hydrogen, named in order of their probable importance, are:

$$(1) \quad C_2H_6 \rightleftharpoons C_2H_4 + H_2$$
$$(2) \quad C_2H_4 \rightleftharpoons C_2H_2 + H_2$$
$$(3) \quad CH_4 \longrightarrow C \quad + 2H_2$$
$$(4) \quad C_2H_2 \longrightarrow 2C \quad + H_2$$

The conditions are those which are known to be favorable to the condensation of acetylene. The methods for the determination of acetylene are so unsatisfactory that no effort was made to determine the proportion of acetylene in the gases formed. Other investigators have found the amount small in similar experiments.

The rapid increase in the proportion of hydrogen with decreasing oil rates below 5 cc. per minute is thought to be due to a marked increase in the extent to which reactions (3) and (4) take place. At 723° C. only a slightly larger proportion of hydrogen is formed than at 621° C., indicating that reactions (3) and (4) do not become rapid up to this temperature. A very marked increase in the hydrogen production takes place when the temperature is elevated to 825° C., chiefly due to an increase in reactions (3) and (4), as is evidenced by the very large amount of free carbon which was liberated and which tended to stop the furnace tube. No trouble was occasioned by this carbon deposition at 621 or 723° C.

Fig. 11 shows the proportion of illuminants, or components removed by 20 per cent fuming sulfuric acid, in the oil gases made at the three temperatures. The percentage of illuminants is highest in the gases made at 621° C. at all oil rates, and remains practically constant at 52 per cent at all oil rates above 10 cc. per minute. The proportion of illuminants formed at 723° C. is higher than at 825° C. except possibly at high oil rates. It is thought, however, that the proportion of illuminants at high oil rates would not be greatly different at any of these temperatures.

The fact that the proportion of illuminants is lower, the higher the temperature at moderate to low oil rates, is due to the secondary reactions of these hydrocarbons. Ethylene is decomposed into carbon and methane to some extent. Condensation to naphthenes takes place. At these low oil rates the proportion of hydrogen present is considerable, and the higher the temperature the higher this percentage. Hence it

faster speeds of such reactions as $C_2H_4 \longrightarrow C + CH_4$ and $C_2H_6 + H_2 \rightleftarrows 2CH_4$.

It might reasonably be expected that the percentage of saturateds in the gases would be greater at 825° C. than at 723° C., but it can be seen from Fig. 10 that this is not the case. At low oil rates the proportion of saturateds is lower at 825° C. than at 723° C. This is chiefly due to the fact that the reaction
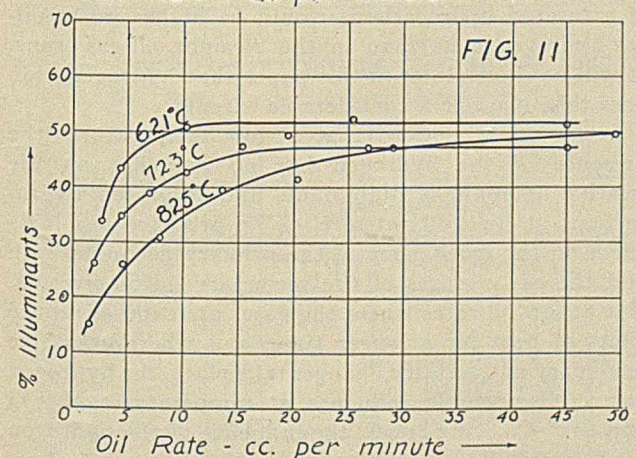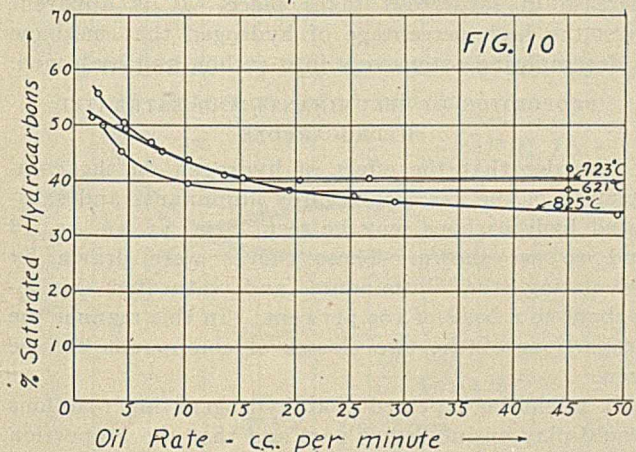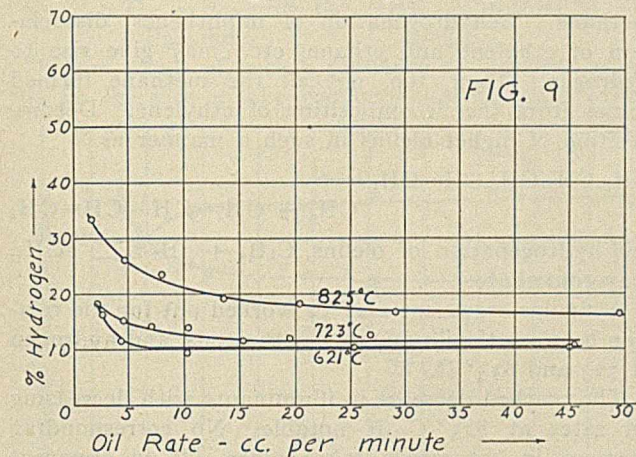
would be expected that hydrogenation reactions such as $C_2H_4 + H_2 \rightleftarrows C_2H_6$ and $C_2H_4 + 2H_2 \rightleftarrows 2CH_4$ would take place.

The relation between the proportions of saturated hydrocarbons formed at different temperatures is shown in Fig. 10. It can be seen that the proportion of saturateds in the gas is higher at 723° C. than at 621° C. at all oil rates. This no doubt is due to the
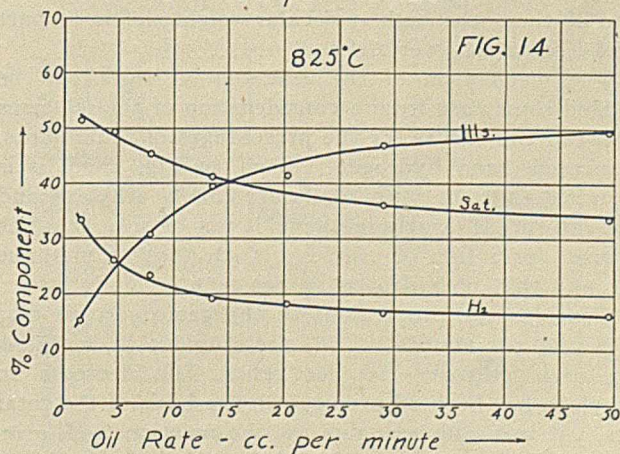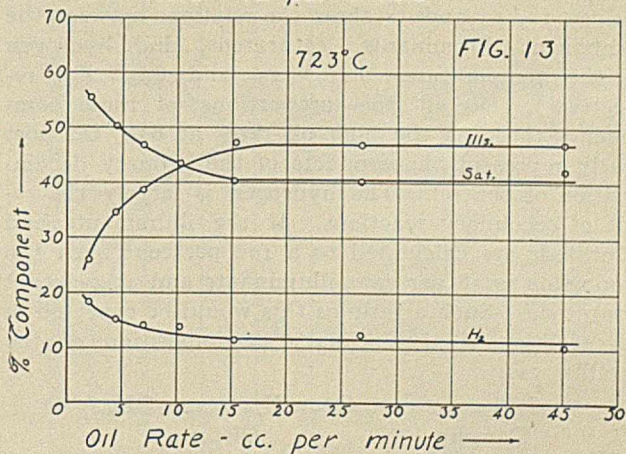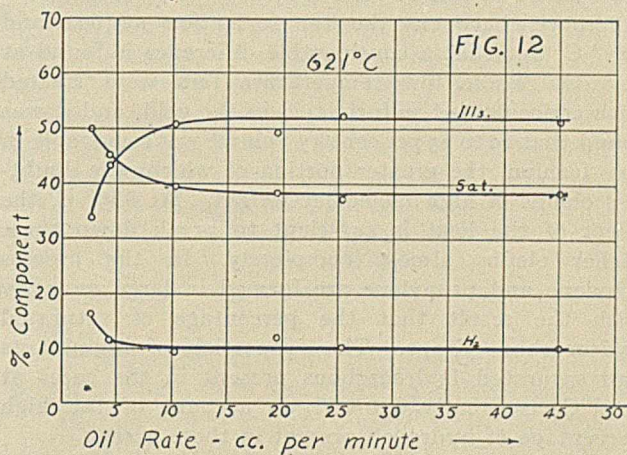
$CH_4 \longrightarrow C + 2H_2$ has an appreciable velocity at this temperature. As the oil rate increases it would be reasonable to expect that the effect of this reaction would be less and less. However, the divergence between the 825° C. curve and the 723° C. curve becomes greater the faster the oil rate. This state of affairs is apparently due to the superimposed effect of another reaction. When the high paraffin hydro-

carbons first break down under these conditions the chief products are low molecular weight paraffin hydrocarbons and high molecular weight olefins. At 621 and 723° C. a considerable proportion of the high molecular weight olefins formed pass on through the tube into the tar. The tars were larger in amount the lower the temperature, and when the tar curves are studied it will be seen that there is not a marked difference between the tar formation at 621 and 723° C. but that a considerable difference is found at 825° C. These low temperature tars were treated with concentrated sulfuric acid in the cold, and it was found that 20 to 25 per cent by volume was removable in this fashion, the greater portion of which was doubtless olefins of high molecular weight. At 825° C. the effect of the heat is sufficient to break down these higher olefins almost completely. In the process ethylene and propylene are formed in large quantity with the result that the percentage of saturated hydrocarbons drops. The proportion of illuminants and saturated hydrocarbons present in the gases at 825° C. is greatly lowered on account of the high percentage of hydrogen present in these gases.

Figs. 12, 13 and 14 show the relations between the component illuminants, saturateds, and hydrogen at the temperatures 621, 723 and 825° C., respectively. Of all the proportions of these components those at the high oil rates at 621° C. most nearly represent the products of the primary decomposition of the oil. The hydrogen is largely the result of secondary reactions. If the illuminants and saturateds are calculated to a 100 per cent basis the proportion is 58 per cent illuminants and 42 per cent saturateds. Such a ratio as this would be expected in the reaction if the primary decomposition of the paraffin was:

$$C_nH_{2n+2} \longrightarrow C_{n-1}H_{2n-2} + CH_4$$
$$\text{Paraffin} \qquad\qquad \text{Olefin}$$
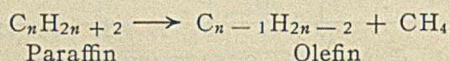
and if then, the high molecular weight olefins in part broke down to lower olefins.

The mechanism of the reactions at work can be judged somewhat from a consideration of the relations between the curves for the percentages of illuminants, saturateds, and hydrogen. Thus, in Fig. 12, if it is assumed that the illuminants are chiefly ethylene and the saturateds largely methane, it can be seen that the normal formation of ethylene is 52, that of methane 39, and that of hydrogen 10 per cent.

Consider the proportions in the gas at an oil rate of 2.5 cc. per minute. They are ethylene 34, methane 50, and hydrogen 16 per cent. The decrease in ethylene has been 18 per cent on the basis of the total gas. If this had been due to the reaction $C_2H_4 \longrightarrow C + CH_4$ the methane should have increased 18 per cent on the basis of the total gas. In fact it increases only 11 per cent.

The hydrogen increases 6 per cent on the total gas basis. If this were due to the reaction $CH_4 \longrightarrow C + 2H_2$ a 3 per cent decrease in the methane should have taken place. But as has been seen, other reactions also give rise to hydrogen. Assuming, however, that the above reaction was the sole change of this sort, only 14 per

cent out of the 18 per cent increase in methane which should have been found, had the ethylene reacted entirely with formation of carbon and methane, would be accounted for. Hence the ethylene must be removed in other ways, for example, by the condensation to naphthenes.

It is not probable that the 6 per cent increase in the hydrogen is entirely due to the decomposition of methane. Dehydrogenation of naphthenes, dissociation of ethylene and ethane, etc., may give rise to hydrogen. Then, too, not all the methane formed comes from the decomposition of ethylene. Decomposition of higher olefins in such a manner as

$$CH_3.CH_2.CH_2.CH{=}CH_2 \longrightarrow$$
$$CH_4 + CH_2{=}CH{-}CH{=}CH_2$$
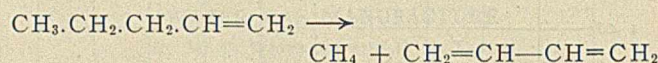
and hydrogenation of olefins, $C_2H_4 + 2H_2 \rightleftarrows 2CH_4$, may contribute.

A similar argument may be worked out for the relation between the illuminants, saturateds, and hydrogen at 723 and 825° C.

The marked decrease in illuminants with decreasing oil rates at 825° C. is notable. No corresponding increase in saturateds takes place. It is apparent from the high percentage of hydrogen that methane is decomposing extensively into carbon and hydrogen.

PROPORTION OF ILLUMINANTS AND SATURATED HYDROCARBONS

In order that the effect of hydrogen on the composition of the gases as regards illuminants and saturated hydrocarbons may be seen, Figs. 15, 16, 17, 18 and 19 are shown. These curves were drawn by calculating the illuminants and saturated hydrocarbons to a basis of 100 per cent. In this manner the proportions of the two classes of compounds can be seen.

It might be expected that hydrogenation reactions would play an important part and that the proportion of saturated hydrocarbons would be higher in the oil-gas-hydrogen runs than in the straight oil-gas runs. It will be shown later in this paper that hydrogenation does take place to a considerable extent.

It should be noticed that at low temperatures the presence of the hydrogen has no influence on the relative amounts of illuminants and saturated hydrocarbons at high oil rates; i. e., its presence has little effect on the mechanism of the primary decomposition and the early stages of the secondary decomposition. But at low oil rates where the gases are exposed to the effect of heat for a longer time, and where extensive secondary and tertiary changes take place, the hydrogen has a considerable influence at temperatures of 723 and 825° C. At 621° C. the influence of the hydrogen is not marked.

It will be remembered that the first reaction undergone by a paraffin hydrocarbon when it is thermally decomposed is that which gives rise to a high molecular weight olefin and a low molecular weight paraffin. It is probable that the higher the molecular weight of an olefin the more readily it is hydrogenated.

If the high molecular weight olefins are hydrogenated, paraffins would be formed. These would

again decompose into long chain olefins and low molecular weight paraffins. This sequence of reactions may be represented as follows:

$$C_nH_{2n+2} \longrightarrow C_{n-1}H_{2n-2} + CH_4$$
$$\text{Paraffin} \qquad\qquad \text{Olefin}$$

$$C_{n-1}H_{2n-2} + H_2 \longrightarrow C_{n-1}H_{2n}$$
$$\text{Olefin} \qquad\qquad \text{Paraffin}$$

$$C_{n-1}H_{2n} \longrightarrow C_{n-2}H_{2n-4} + CH_4$$
$$\text{Paraffin} \qquad\qquad \text{Olefin}$$
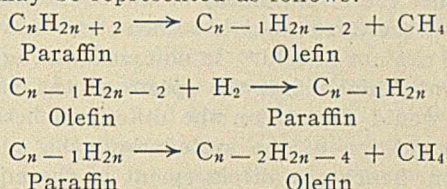
The net result of such a sequence of reactions would be an increase in the proportion of paraffins in the

FIG. 15
Ills.
Sat.
Oil Gas
Oil Gas + H₂
621°C   1H₂ : 2 Gas

FIG. 16
Ills.
Sat.
Oil Gas
Oil Gas + H₂
723°C   1H₂ : 2 Gas

FIG. 17
Ills.
Sat.
Oil Gas
Oil Gas + H₂
723°C   2H₂ : 1 Gas

$2H_2 : 1$ Gas (Figs. 17 and 19), the proportion of saturated hydrocarbons is greater in the hydrogen-oil-gas runs. It is probable, therefore, that the above reactions take place in all cases but that the effect of the hydrogen at low oil rates on the extensive secondary changes is so great that the result of the hydrogenation is masked.

Apparently the low molecular weight olefins are not hydrogenated to a large extent, for were this the case the proportion of saturated hydrocarbons present would be greatly increased. This is not the case.

The increase in the proportion of olefins in the hydrogen-oil-gas runs at low rates of oil feed may be accounted for in two ways:

(1)—The effect of the hydrogen may be to increase those reactions which give rise to olefins.

(2)—The effect of the hydrogen may be to retard those reactions which tend to remove or destroy the olefins.

The largest proportion of the ethylene and propylene present comes from the direct splitting up of high

FIG. 18
Ills.
Sat.
Oil Gas
Oil Gas + H₂
825°C   1H₂ : 2 Gas

FIG. 19
Ills.
Sat.
Oil Gas
Oil Gas + H₂
825°C   2H₂ : 1 Gas
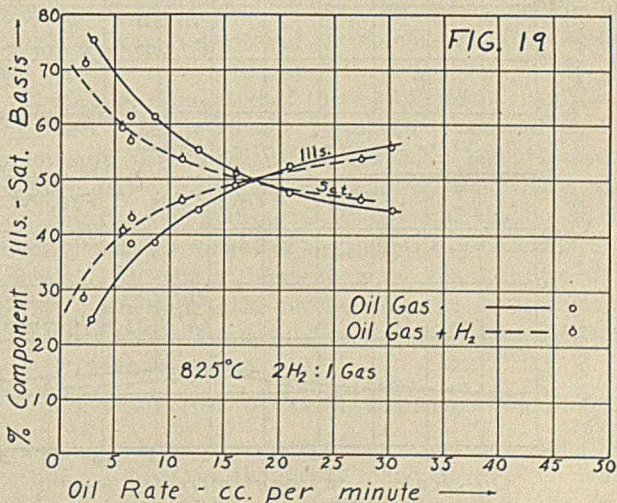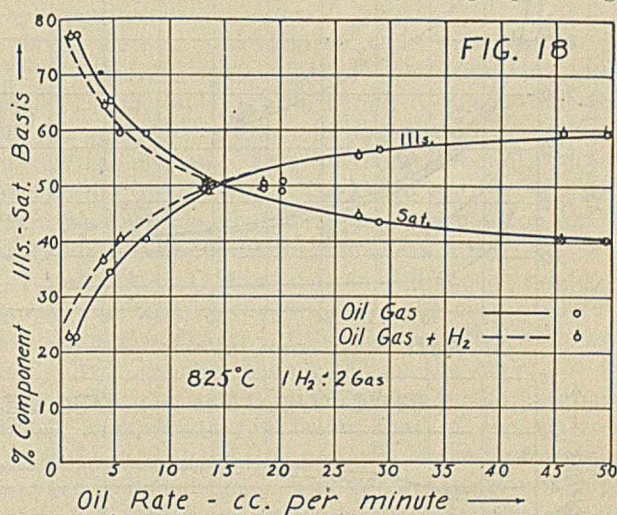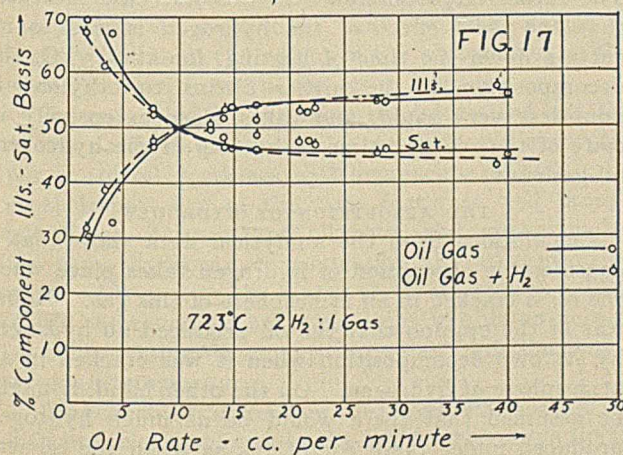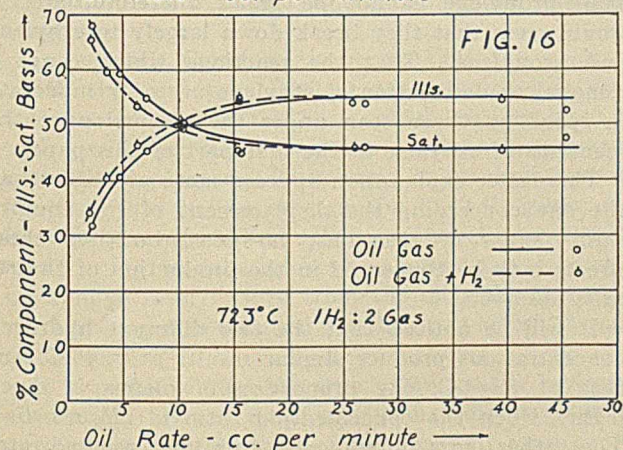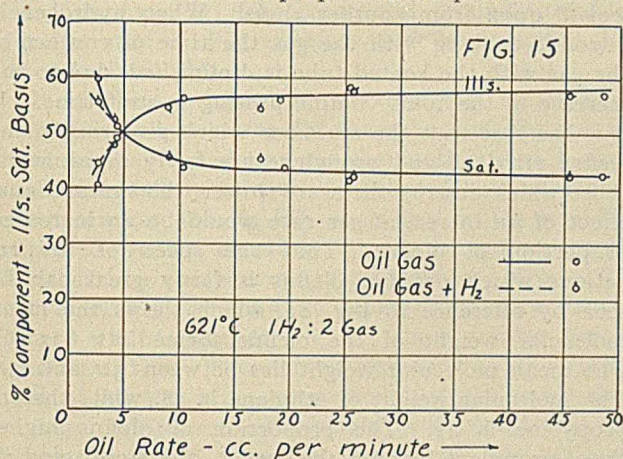
gas. But as can be seen in the figures, the proportion of paraffins is less in the cases of the hydrogen-oil gases. However, at high oil rates where secondary reactions are not so important, and in the series of runs at 825° C. where the concentration ratio was

molecular weight olefins. If an olefin of fairly high molecular weight may be used to illustrate, this reaction may be represented:

$$CH_3CH_2CH_2CH{=}CH_2 \longrightarrow CH_3CH{=}CH_2 + CH_2{=}CH_2$$
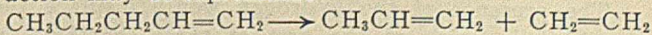
It will be noticed that the result is an increase in

volume. The equilibrium point of the reaction would therefore be shifted by a diminution of pressure in such direction as to favor the production of ethylene and propylene. The introduction of hydrogen has the same effect as a reduction in pressure, and would therefore have a similar effect on the equilibrium.

Chief among the reactions which remove olefins

FIG. 21

FIG. 22

FIG. 23

FIG. 24

such as ethylene and propylene are condensation and decomposition. These reactions may be represented as $3C_2H_4 \longrightarrow C_6H_{12}$ and $C_2H_4 \longrightarrow C + CH_4$.

The presence of the hydrogen would displace the equilibrium point of the first of these reactions in favor of the ethylene. The latter reaction, in so far

as the influence of volume relationships on the equilibrium point is concerned, would not be affected.

Whether the displacement of the equilibrium points in any of these reactions is sufficient to be worthy of mention can not be said. In no case is the equilibrium condition attained, but the speeds of the various reactions would depend on the difference between the actual condition of the system and the equilibrium condition; hence any displacement of the equilibrium point would be important.

The increase in the proportion of olefins may be looked upon from another angle. When hydrogen is introduced along with the gas the time of contact of the gas with the heated tube is diminished, due to the increase in the total volume passing in unit time. If it is the case that the reactions which give rise to the olefins ethylene and propylene are fairly rapid, while those which destroy them are slower, the summational effect of an increased gas rate would be an increased proportion of olefins. That the speed of the reactions which produce olefins is fairly great can be seen by reference to Fig. 22, which shows the mean molecular weight of the olefins formed at 825° C. The mean molecular weight lies between 30 and 34. The molecular weight of ethylene is 28, while that of propylene is 42. The proportion of olefins higher than propylene cannot be great, therefore, and it would seem that they break down largely to ethylene and propylene. That the reactions which cause a removal or destruction of ethylene are only moderate in speed has been seen under the discussion of the reactions of ethylene in the first part of this paper.

This latter explanation appears more probable than the one concerning the displacement of the equilibrium points of reactions; however, both of these effects may be concerned in the production of the results observed.

It will be noticed that the two different hydrogen concentrations produce similar results at 723° C. but that at 825° C. the proportion of olefins is much higher when the concentration ratio is $2H_2 : 1$ Gas. The rather large difference in this last case indicates that the chief effect of the hydrogen is due to its cutting down the time of heating, for at 825° C. the decomposition of the higher olefins to ethylene is doubtless very rapid, and takes place extensively in spite of the reduced time of heating in the hydrogen-oil-gas runs.

THE ABSORPTION OF HYDROGEN

Calculations from the analytical data show that a considerable absorption of hydrogen takes place when the oil is cracked in an atmosphere of this gas. Haber was of the opinion that the oil produced no hydrogen by its own decomposition when it was cracked in an atmosphere of hydrogen. On the other hand, it might be assumed that there would be as much hydrogen produced under these conditions as when the oil was cracked alone. These two assumptions offer two bases on which the absorption of hydrogen may be calculated.

(I)—If no hydrogen is produced by the cracking of the oil, the hydrogen absorption per cc. of oil would
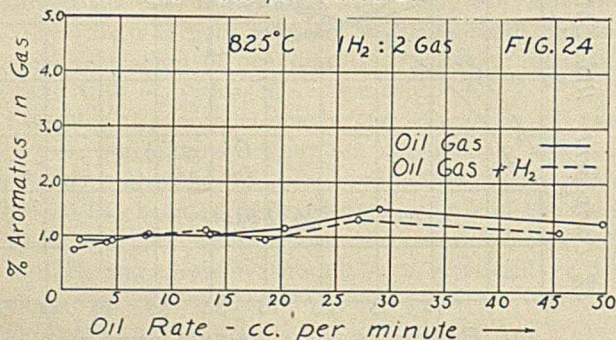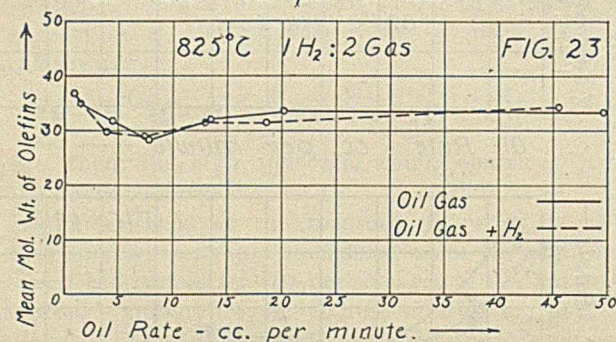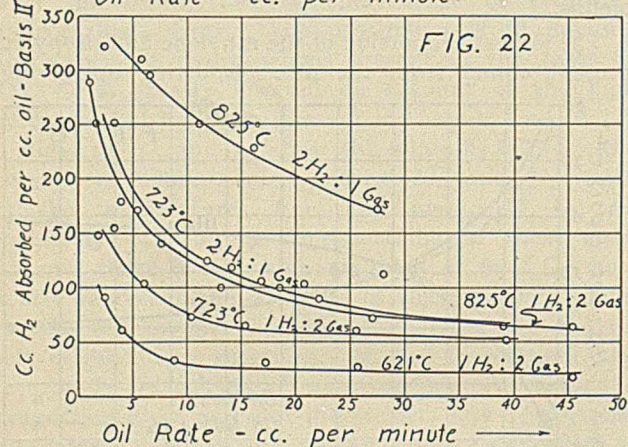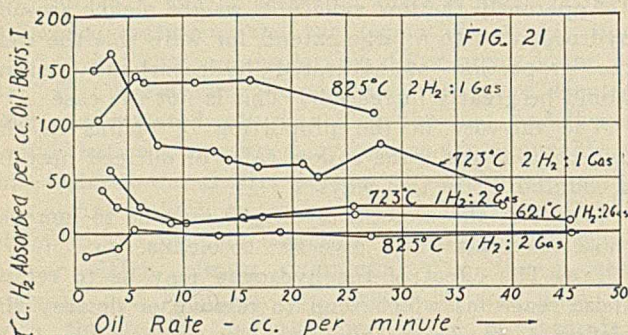
be equal to the difference between the hydrogen added and that present in the final gas divided by the total number of cc. of oil.

(II)—If the oil produces as much hydrogen as when cracked alone, the difference between the hydrogen added plus that normally produced from the oil at the particular oil rate and the hydrogen in the final gas represents the absorption. This divided by the total number of cc. of oil gives the absorption in cc. per cc. of oil.

Fig. 21 shows the absorption per cc. of oil calculated on basis (I) for the several temperatures and concentrations. Fig. 22 shows these absorptions calculated on basis (II).

It will be seen that the curves of Fig. 22 are much smoother and more regular than those of Fig. 21, not because they are drawn more smoothly, but because the points fall on smoother curves. The general form of the curves, too, in Fig. 22 is that which would be expected from a consideration of the curves for the hydrocarbon components formed per cc. of oil. The curves of Fig. 21 show no general similarity to each other, while those of Fig. 22 show similar general characteristics. The curves representing the formation of all the other components of the gases show regular variations, and it would be expected that this regularity would extend to the curves for hydrogen absorption.

In general, therefore, it seems that the basis on which the curves of Fig. 22 are calculated is more nearly correct than the basis which assumes that no hydrogen is produced from the oil when it is decomposed in hydrogen. This view is strengthened by the fact that in the case of the 825° C. gases with hydrogen concentrates $1H_2 : 2$ Gas, the absorption curve in Fig. 21 falls below the 0.0 line, *i. e.*, hydrogen must have been formed from the oil since there was more hydrogen in the final gas than was added through the meter.

It is probable that the true value for the hydrogen absorptions for any set of conditions falls between the two values as calculated from the two limiting assumptions. It is thought that the true values are slightly less than the values of the absorptions as they would be read from the curves in Fig. 22.

The curves of Fig. 22 show the interesting fact that at any particular temperature the hydrogen absorption per cc. of oil decreases with increasing oil rate. The great importance of the time factor is well brought out here. At constant oil rate, and approximately the same hydrogen concentration the absorption per cc. of oil is greater the higher the temperature. There would, however, be an upper limit to this on account of the excessive decomposition of all hydrocarbons at elevated temperatures.

The effect of increasing the concentration of hydrogen is clearly shown in Fig. 22 for the curve for the $2H_2 : 1$ Gas runs is above the curve for the $1H_2 : 2$ Gas runs at both 723 and 825° C. The speed of hydrogenation reactions is greater the higher the concentration of hydrogen.

It is interesting to note that the curve for the $1H_2 : 2$ Gas runs at 825° C. falls below the curve for the $2H_2 : 1$ Gas runs at 723° C. This shows that the increasing temperature is tending to cause dehydrogenation reactions or hydrocarbon dissociations to a marked degree at 825° C. The effect of hydrogen in greater concentration in reversing these dissociations is clearly brought out when the position of the $2H_2 : 1$ Gas curve for 825° C. is considered in its relation to the $1H_2 : 2$ Gas curve at this same temperature.

MEAN MOLECULAR WEIGHT OF THE OLEFINS

In Fig. 23 the mean molecular weight of the olefin hydrocarbons in gases made at 825° C. in oil-gas runs and in hydrogen-oil-gas runs with the concentration ratio $1H_2 : 2$ Gas can be seen.

It should be kept in mind that the molecular weight of ethylene is 28 and that of propylene is 42. From the position of the curves it can be seen that approximately one-third of the olefins is propylene. The curves lie very close together, and it is impossible to say just what the influence of the hydrogen is on the formation of the olefins.

If the method of calculation of the molecular weight of the olefins, as explained under the discussion of the analytical methods, is considered, it is apparent that all the analytical errors pile up and are brought out in this calculation. This no doubt accounts for the irregularity in the curve, and also for the fact that there is no consistent difference in the position for the oil-gas runs and the hydrogen-oil-gas runs.

It was thought that certain differences might be brought to light by the curves for the mean molecular weights of the olefins. If the higher olefins were more easily hydrogenated than ethylene the curve for the mean molecular weight of the olefins in the hydrogen-oil-gas runs would fall below that of the oil runs. If, on the other hand, the presence of the hydrogen, on account of its causing a more rapid passage of the gas through the tube, resulted in a less extensive decomposition of the higher olefins, the curve for the hydrogen-oil-gas runs would lie above that for the oil-gas runs.

It may be thought that these two effects are balancing each other with the result that the curves are practically the same. It would have been desirable to have carried out a similar series of runs with a high concentration of hydrogen, but the calculation of the mean molecular weight of the olefins can be made only when the per cent of benzene in the gas is known, and, as has been noted, the method for the determination of benzene was found only as this experimental work was drawing to a close.

THE FORMATION OF AROMATIC HYDROCARBONS

Fig. 24 shows the percentage of aromatic hydrocarbons present in the gases made at 825° C. when oil is cracked alone or in hydrogen when the concentration ratio is $1H_2 : 2$ (*Oil Gas + Tar Gas*). The method of determining these percentages has been described under the analytical methods.

The percentage of aromatics appears to increase slightly with increase in oil rate. Whether this is

actually the case or not cannot be definitely stated. The exact opposite would be expected. It is thought that the apparent increase may be due to the freezing out of high molecular weight hydrocarbons of other types than the aromatic compounds. High molecular weight paraffins and olefins are present in greater

FIG. 25

Oil Gas
Oil Gas + H₂

621°C   1 H₂ : 2 Gas

Oil Rate - cc. per minute. ——

FIG. 26

723°C   1 H₂ : 2 Gas

Oil Gas
Oil Gas + H₂

Oil Rate - cc. per minute. ——

FIG. 27

723°C   2 H₂ : 1 Gas

Oil Gas
Oil Gas + H₂

Oil Rate - cc. per minute. ——

proportion in the gases made at high oil rates than in those made at low oil rates.

The smaller proportion of aromatic hydrocarbons present in the gases made at low oil rates may possibly be due to the removal of benzene to form compounds such as diphenyl, naphthalene and anthracene, which pass largely into the tars.

The hydrogen apparently has little effect on the formation of aromatics at low oil rates, but decreases the aromatic formation somewhat at higher oil rates.

This is possibly due to the retarding effect which the presence of hydrogen would have on the formation of aromatics or hydroaromatics by condensation reactions. Less gas is formed from the oil at high oil rates than at low oil rates, and as a consequence the concentration of hydrogen is greater at high oil rates than at low oil rates.

TARS

The tars were collected from the tar drip and the volume measured. This volume divided by the volume of the total oil used and multiplied by 100 gives the percentages of tar formed. Figs. 25 to 29 show these tar percentages for both oil-gas and hydrogen-oil-gas runs plotted against the oil rate at the temperatures indicated. It should be mentioned that at low oil rates these percentages are not accurate. The low-oil-rate tars are heavy and viscous, and as a result do not run down through the condenser as easily as the lighter high-oil-rate tars.

As far as can be judged from the curves in Figs. 25, 26, and 27, for temperatures of 621 and 723° C., there is no marked regular difference between the tar formation in the oil-gas runs and the hydrogen-oil-gas runs.

At 825° C. the percentage of tar in the oil-hydrogen runs is consistently less than in the oil runs except at low oil rates where, as has been mentioned, the tar percentages as shown mean little. This difference is more marked in the curves of Fig. 27 where the concentration ratio was 2 Hydrogen : 1 (Oil Gas + Tar Gas) than in the curves of Fig. 28.

In general, two classes of compounds are contained in the tar: (1) unchanged or partially changed oil; (2) synthetic hydrocarbons which are the products of extensive change. It may be thought that hydrogen, on account of its decreasing the time of contact of the hydrocarbon vapors with the heated tube, would tend to increase the proportion of tar since the decrease in the time of heating would cause a less extensive decomposition of the oil vapors. On the other hand, this decrease in the time of heating would also diminish the extent to which synthetic reactions resulting in the formation of tarry products would take place. Also the percentages of the hydrogen would retard these reactions, since they are all reactions which result in decrease of volume. Apparently these effects are balanced at temperatures of 723° C. or below. At 825° C., however, the percentage of tar is less. This leads to the belief that synthetic reactions are responsible for a considerable proportion of the tars at temperatures in the neighborhood of 825° C.

Fig. 30 shows clearly the effect of temperature on tar formation, and also the effect of increase of oil rate at constant temperature. The proportion of tar increases with increasing oil rate and most markedly so, at temperatures of 621 and 723° C. The largest proportion of these tars at moderate to high oil rates is undecomposed oil, as shown by distillation and treatment with concentrated sulfuric acid. This is also indicated by the fact that a temperature change from 621 to 723° C. produces no great difference

in tar formation, and also by the fact of the very rapid increase in the percentage of tar with increasing oil rate.

At 825° C. the proportion of tar does not increase greatly with increasing oil rate, indicating that these tars are largely composed of synthetic products,

which is further substantiated by other physical characteristics, such as distinct aromatic odor and their reactions with concentrated sulfuric acid.

All the tars were strongly fluorescent.

FORMATION OF ILLUMINANTS, METHANE, AND ETHANE, AND THE OBTAINING OF PARTICULAR END-PROD-UCTS FROM A PARAFFIN HYDROCARBON OIL

In Figs. 31 to 35 the cc. of illuminants, methane and ethane formed from 1 cc. of oil are shown plotted against the oil rate at the temperatures and hydrogen-

gas concentration ratios indicated. It can be seen from Fig. 31 that at 621° C., with the exception of the illuminants in the case of the straight oil-gas runs, the number of cc. of all these hydrocarbons formed from 1 cc. of oil increases with decreasing oil rate. There would be a limit to this, however, for, were the oil rate

made low enough, a very extensive decomposition of the hydrocarbons would set in.

Fig. 31 shows that the illuminants are the most easily decomposed of the gaseous hydrocarbons. The curve for the illuminants in the straight oil-gas runs has a maximum due to the fact that though the longer time of contact of the oil vapors with the heated tube at low oil rates causes a more extensive formation of ethylene and other illuminants, an oil rate is reached where extensive decomposition of these

hydrocarbons takes place, which more than overcomes the more rapid illuminants formation at low oil rates. Condensation and hydrogenation, which are more extensive at low oil rates, are important in this connection also.

No maximum is observed in the illuminants curve for the hydrogen runs, doubtless because the time of contact of the oil vapors with the heated tube is less at any particular oil rate than in the case of the oil-gas runs. The curve for the illuminants in the hydrogen-oil-gas runs, for this reason also, is always below that for the illuminants in the oil-gas runs, except at low oil rates.

Less methane is formed per cc. of oil in the hydrogen-oil-gas runs than in the straight oil-gas runs except at low oil rates. That the time of contact here is sufficient so that extensive hydrogenation takes place is clearly brought out from a consideration of Fig. 22 in connection with Fig. 31. At all oil rates the decreased time of contact of the gases with the heated furnace tube, on account of the absorption of hydrogen, results in a lower formation of methane.

The relationships for ethane are much the same as for methane, and for the same reasons.

Figs. 32 and 33 show the relationships between the hydrocarbons at 723° C. at hydrogen concentration ratios of $1H_2 : 2\ Gas$ and $2H_2 : 1\ Gas$, respectively. More of each of the components is formed per cc. of oil at 723° C. than at 621° C. The maximum in the illuminants curve falls at a higher oil rate than at 621° C. as would be expected, since the higher temperature would cause a more rapid decomposition, condensation, and hydrogenation of the illuminants to take place. At 723° C. the maximum on the illuminants curve for the hydrogen runs can be seen clearly. It is interesting to note that this falls to the left of the maximum on the curve for the oil-gas runs. The decreased time of heating on account of the hydrogenation admixture is responsible for this.

The effect on the illuminants of increasing the concentration of hydrogen is clearly brought out in Figs. 32 and 33. The maximum on the curve for the illuminants in the hydrogen-oil-gas runs at the higher hydrogen concentration falls at a slightly lower oil rate than the maximum on the curve for the lower hydrogen concentration. The divergence between the illuminants curves for the oil-gas runs and the hydrogen-oil-gas runs is greater both at low and high oil rates at the higher hydrogen concentration than at the lower hydrogen concentration on the time of contact of the gases with the heated tube surfaces.

The relationships in the case of the methane and ethane are exceedingly interesting. The formation of methane is less in the hydrogen-oil-gas runs than in the oil-gas runs at moderate to high oil rates, due to the decreased time of contact of the gases with the heated surfaces, and this effect is more pronounced at the higher hydrogen concentrations, as would be expected. As the oil rate decreases the hydrogenation effect becomes important and the proportion of methane formed from 1 cc. of oil is greatest in the case of the hydrogen-oil-gas runs. The curve for

methane in the hydrogen-oil-gas runs crosses the methane curve for the oil-gas runs. This crossing is at a higher oil rate with the higher hydrogen concentration, showing clearly the effect of the increase of concentration of hydrogen on the hydrogenation reactions. The formation of methane is slightly greater in the hydrogen-oil-gas runs than in the straight oil-gas runs when the hydrogen concentration ratio is $1H_2 : 2\ Gas$. This difference is slightly greater at low oil rates than at high oil rates.

When the hydrogen concentration ratio is $2H_2 : 1\ Gas$ the ethane formation per cc. of oil is less in the hydrogen-oil-gas runs than in the oil-gas runs at high oil rates. This is the effect of the decreased time of contact due to the admixture of a larger volume of hydrogen. But at low oil rates the formation of ethane is much greater in the hydrogen-oil-gas runs as can be seen in Fig. 33. The ethane curve has a maximum, too, which is interesting because it shows that at low oil rates the reactions of the hydrocarbon ethane itself have an important part to play.

Figs. 34 and 35 show the relationships between these hydrocarbons at 825° C. A much less pronounced decrease in the formation of illuminants with increasing oil rate is due to the fact that the temperature of 825° C. is sufficient to promote actively the formation of illuminants. The maxima on the illuminants curves for the oil-gas runs fall at the higher oil rates, as would be expected when the higher temperature is taken into consideration. The illuminants curves for the hydrogen-oil-gas runs are entirely above the illuminants curves for the oil-gas runs. This is again a result of the decreased time of heating when hydrogen is admixed. The effect is most marked when the higher concentration of hydrogen is used.

The curves for methane in the hydrogen-oil-gas runs fall above the curves for methane in the oil-gas runs at all oil rates at 825° C. This is due to two effects: *first*, the less extensive decomposition of the methane into carbon and hydrogen due to the decreased time of heating in the hydrogen-oil-gas runs; *second*, the increased rate of hydrogenation reactions such as $C_2H_4 + 2H_2 \rightleftarrows 2CH_4$. The effect of hydrogen in reversing the reaction $CH_4 \rightleftarrows C + 2H_2$ is probably not important, as has been brought out in the discussion of the methane equilibrium and the reactions of methane in the first part of this paper.

The effect of the greater concentration of hydrogen on methane production can be seen clearly by comparing Figs. 34 and 35. The divergence between the hydrogen-oil-gas and the oil-gas methane curves is greatest when the hydrogen concentration ratio is $2H_2 : 1\ Gas$, and this is practically true at low oil rates where hydrogenation reactions are most important.

More ethane is formed when hydrogen is mixed with the vapors of the oil than when it is not added. This is doubtless due to the combined influence of the hydrogen in diminishing the decomposition of the ethane and to its effect in hydrogenating the olefins. These effects are particularly marked when the concentration ratio is $2H_2 : 1\ Gas$.

It may have been noticed that the curves for the oil-gas runs made at the same temperature do not coincide exactly, since the carbon tube used carbonizes somewhat and becomes of smaller internal diameter, thus decreasing the time of contact of the gas with the tube and consequently altering the composition somewhat.

The effect of temperature on the hydrocarbon products of decomposition of an oil can be seen very

higher temperatures in promoting the decomposition of the long chain olefins to ethylene and propylene.

It is interesting to note the position of the maxima of the curves for the illuminants at the several temperatures. These maxima indicate where the balance between the reactions of formation and the reactions of decomposition falls.

Within the temperature range studied, the formation of methane is greater the higher the temperature. At low oil rates the difference in the methane produced by a 100°-temperature rise is greater in the range from 621 to 723° C. than from 723 to 825° C., since at low oil rates 723° C. is a sufficiently high temperature to break down the original oil extensively.
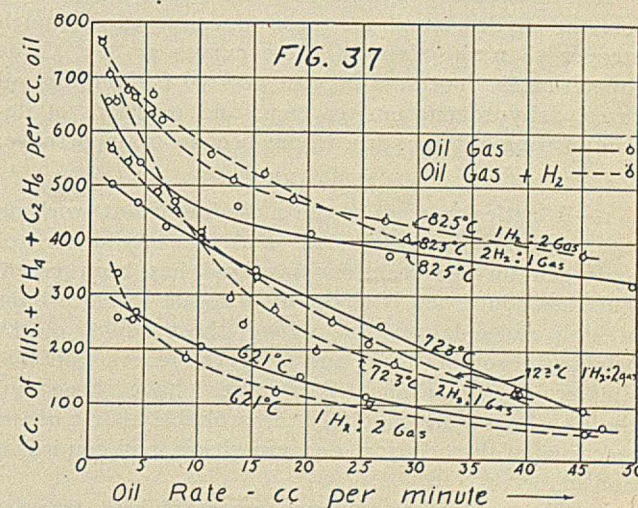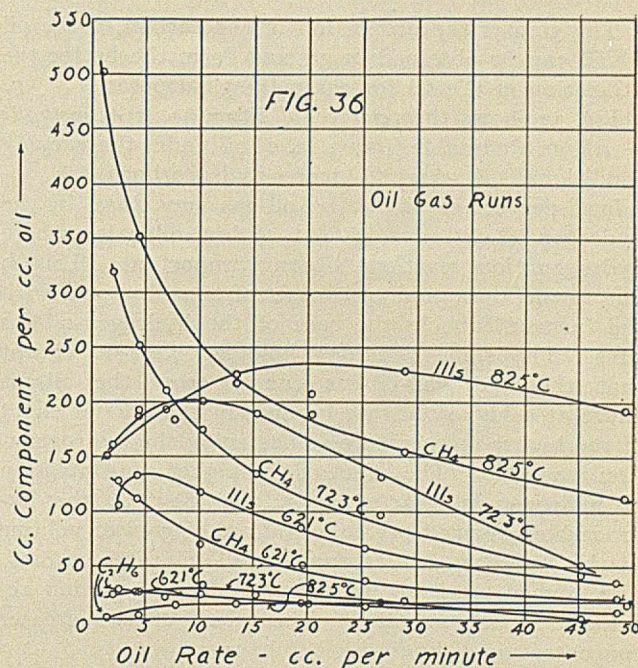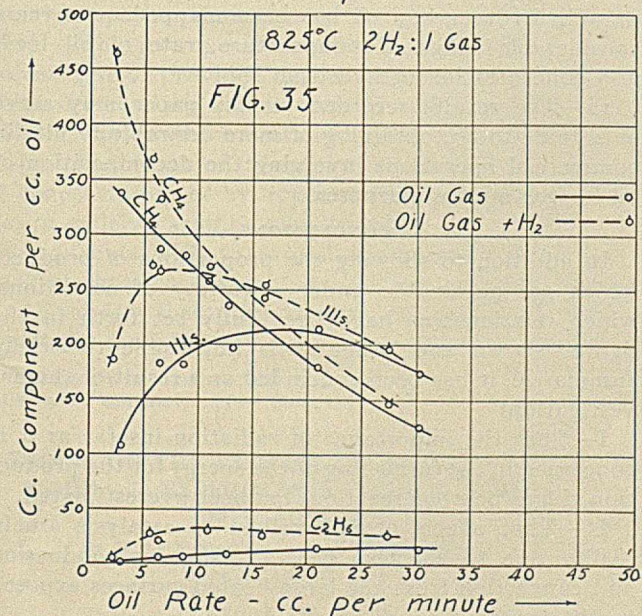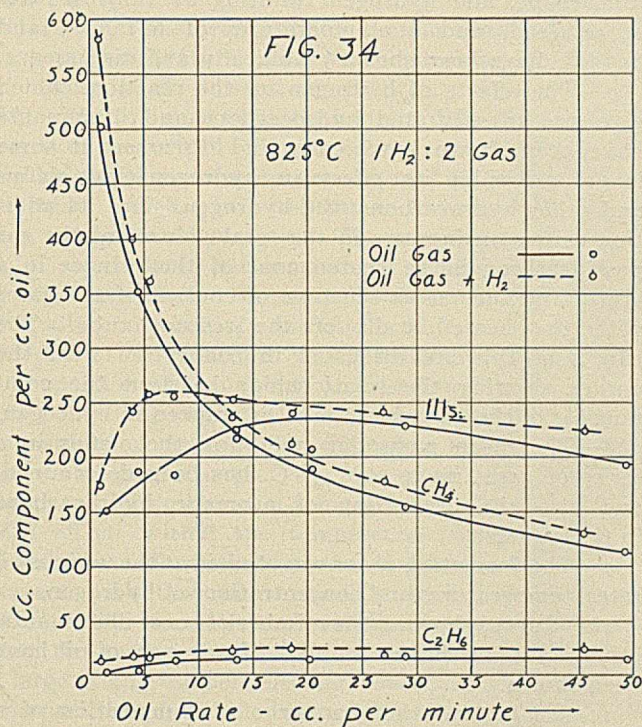
clearly from Fig. 36. Within the temperature range studied, the cc. of illuminants per cc. of oil increases with temperature, with one exception. At low oil rates there are more illuminants formed at 723° C. than at 825° C. The effect of the higher temperature in increasing the speed of the reactions which decompose ethylene more than overcomes the effect of the

The methane increase between 723 and 825° C. is largely due to the decomposition and hydrogenation of olefins $C_2H_4 \longrightarrow C + CH_4$ and $C_2H_4 + 2H_2 \rightleftharpoons 2CH_4$, as can be seen from a consideration of the illuminants curve for 825° C.

At high oil rates a temperature of 825° C. is necessary to form methane largely, as can be seen from the

position of the curve for methane at 621, 723 and 825° C.

The formation of ethane per cc. of oil is not large at any temperature studied, as shown in Fig. 36. The primary decomposition of the oil therefore involves chiefly a splitting off of methane rather than ethane or higher paraffin. The decomposition and dissociation of ethane are clearly shown by the falling off of the ethane curve as the oil rate decreases at a temperature of 825° C. On the other hand, a temperature of 825° C. is necessary to cause an extensive formation of methane and ethane per cc. of oil at high oil rates.

TOTAL HYDROCARBONS OBTAINABLE FROM THE OIL

The greater the proportion of the carbon of the oil which can be obtained in gaseous form the better the utilization of the oil for gas-making purposes.

Fig. 37 shows the *total cc. of illuminants + methane + ethane* obtainable from 1 cc. of oil under the varying conditions. At 621° C. more hydrocarbons are obtained per cc. of oil in the oil-gas runs than in the hydrogen-oil-gas runs, except at low oil rates where hydrogenation reactions become important. This is due to the lower time of contact of the oil vapors with the furnace tube in the case of the hydrogen-oil-gas runs. The same relations hold at 723° C. except that the hydrogen-oil-gas curves cross the oil-gas curve at a higher oil rate because of the greater effect of the higher temperature in hastening the hydrogenation reactions. The effect of the higher concentration of hydrogen is clearly shown. At 825° C. the hydrocarbons formed per cc. of oil are of greater volume in the hydrogen-oil-gas runs at all oil rates studied. The higher temperature promotes hydrogenation reactions at all oil rates. The effect of the greater concentration of hydrogen can be seen.

It is interesting to note that the slope of the curve for the hydrogen-oil-gas runs at 723° C. is much steeper than the slope of these curves at 825° C. at low oil rates, doubtless because at 825° C., and low oil rates, dehydrogenation reactions and reactions of decomposition of the hydrocarbons become of importance.

SUMMARY[1]

1—A critical review of the most important work on hydrocarbon decomposition and the influence of hydrogen on the reactions involved has been given. This has concerned itself with: *first*, the hydrocarbons of high molecular weight; *second*, the products of the primary decomposition; and *third*, the reactions of the simpler hydrocarbons. Summaries have been included which state concisely the probable course of the reactions of dissociation, decomposition, and condensation involved.

2—The subject of the mechanism of heat transfer in gas machines has been discussed.

3—Difficulties in the measurement of the true temperature of a gas have been pointed out.

4—In the experimental work a paraffin oil was thermally decomposed alone and in hydrogen at temperatures of 621, 723 and 825° C. Concentra-

[1] Summaries 1, 2 and 3 refer to the printed dissertation.

tions of hydrogen approximating 1*H₂* : 2 *Oil Gas* and 2*H₂* : 1 *Oil Gas* were those studied. It has been shown what results may be expected in the decomposition of a hydrocarbon oil when temperature, rate of oil feed, and concentration of admixed hydrogen are carefully controlled.

5—The relationship between the rate of oil feed and the rate of gas generation has been brought out.

6—The proportions of illuminants, saturated hydrocarbons, and hydrogen resulting at varying rates of oil feed, and at temperatures of 621, 723 and 825° C. have been shown graphically and discussed.

7—The effects of hydrogen on the reactions which give rise to saturated hydrocarbons and illuminants have been shown graphically and discussed at some length. Besides its effect in hydrogenating olefins and other hydrocarbons, the hydrogen, since its addition causes an increase in the total volume of the gas passing through the heated zone of the furnace in a given time, decreases the time of contact of the gases with the heated walls of the resistor tube. The effects of this are discussed in connection with the curves showing the relationships between the components of the gas when the oil is cracked in hydrogen.

8—The mean molecular weight of the olefins in a series of gases made at 825° C. has been determined, and also the proportion of aromatic hydrocarbons in these gases.

9—The formation of tar was studied at the various oil rates, temperatures and concentrations of hydrogen.

10—Curves showing the number of cc. of illuminants, ethane and methane obtainable from 1 cc. of oil have been shown.

11—In general the manner of decomposition of a paraffin hydrocarbon oil has been mapped out over a considerable range of temperature, rate of oil feed, and concentration of hydrogen.

12—The results recorded in this paper may serve as a guide to the obtaining of more desirable results in commercial operations involving the decomposition of oil for gas-making purposes.

CONCLUSIONS

In addition to showing the proportions of products which are obtainable under a variety of conditions, which relationships have been fully set forth in the figures shown and which it is impossible to briefly summarize, it has been concluded as a result of this investigation:

I—That the importance of radiation insofar as it is concerned in the furnishing of the energy for the production of hydrocarbon reactions has been overestimated.

II—That effects often ascribed to catalysis are in reality due to effective heat transfer by conduction and convection from the large heated surfaces exposed to the gases.

III—That the equilibrium condition is not attained in a hydrocarbon system when an oil is decomposed by heat under conditions analogous to those of carbureted water-gas manufacture.

IV—That the course of the changes involved in the breaking down of a hydrocarbon oil may be roughly traced.

V—That hydrogen is produced from an oil even when the cracking takes place in hydrogen.

VI—That considerable absorptions of hydrogen take place when an oil is cracked in an atmosphere of hydrogen, and this absorption is greater the higher the concentration of hydrogen, the higher the temperature (within the range studied), and the lower the oil rate.

VII—That propylene and higher olefins constitute approximately one-third by volume of the illuminants of the gas.

VIII—That the proportion of tar increases with decrease in temperature, and with increasing oil rate, particularly at the lower temperatures.

IX—That no marked and consistent difference in the amount of tar formed when an oil is decomposed alone or in hydrogen at temperatures of 723° C. or below is noticeable. At 825° C. less tar is formed when the oil is cracked in hydrogen. The tars formed below 723° C. are in large part unchanged or partly changed oil, whereas those tars formed above 800° C. are essentially composed of synthetic products.

X—That the reactions which result in decreasing the proportion of illuminants are the most rapid.

XI—That the presence of hydrogen during the decomposition of an oil has the effect of increasing largely the proportion of the carbon of the oil appearing as hydrocarbons in the gas.

XII—That within the temperature range studied the volume of illuminants produced per volume of oil increases with the temperature with one slight exception. The formation of methane is greater the higher the temperature. The formation of ethane is not large at any temperature and therefore the primary decomposition of an oil involves chiefly a splitting off of methane rather than ethane or higher homologues.

XIII—That a temperature of 823° C. is desirable in decomposing an oil provided that too great opportunity for extensive secondary and tertiary change is not given.

XIV—That with correct design of apparatus, and proper adjustment of temperature, rate of oil feed, and concentration of hydrogen it is possible to obtain gases of widely varying compositions.

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STUDIES ON THE EXTRACTION OF ROSIN FROM WOOD. I—EXPERIMENTS USING A PETROLEUM SOLVENT

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PURPOSE OF WORK

From a technical viewpoint, the process of extracting rosin from wood with chemicals offers a promising possibility for the utilization of "fat" stumps and other waste resinous wood. In the simple steam distillation process only the volatile constituents of the wood are recovered and this method is no longer industrially feasible on this account. In the destructive distillation process, the products are charcoal, tar, and a turpentine more or less contaminated with products from the destructive distillation of the rosin and wood substance. This turpentine, at best, does not bring as high a market price as steam-distilled wood turpentine, although the recent introduction of temperature-controlled processes has removed this objection to a large extent. Compared with these two processes, the so-called solvent or extraction process affords the recovery of wood turpentine and pine oil comparable in quality and value with the oils from steam distillation and also a medium grade rosin whose market value, under normal conditions, is practically equal to the combined value of charcoal and tar from the destructive distillation process. However, the market value is less likely to fluctuate for tar and charcoal than for rosin. Strictly speaking, the extraction and distillation processes are not comparable, because the products are quite different. These processes represent the two types which taken together cover most of the possible products to be obtained from resinous wood.

Of the several different processes proposed for treating wood, the destructive distillation method, which is by far the oldest, is also at the present time apparently the best established from the standpoint of profitable commercial operation.[1] The principal difficulties that have been encountered in the extraction process have been: (1) an unstable market price for rosin, and (2) high cost of operation, due largely to an excessive loss of solvent. In attempting a solution of these difficulties, there are then at least two lines of attack which may give this process a better opportunity for commercial success: (1) obtaining another product which would not be subject to very great market variations, and (2) decreasing the operating cost. The use of the extracted wood as a raw material for paper pulp has been suggested several times as a possible solution of the problem of obtaining another product. Extraction by the usual method requires wood so finely divided (shredded wood) to yield a high proportion of the rosin that the extracted material is not suitable for pulp. If the wood is large enough for pulp, the yield of rosin is decreased. Considering the high operating costs as due largely to the loss of solvent, it would seem that the problem here is largely mechanical, and its solution should not offer very great difficulties.

With these ideas in mind, it was felt that a careful study should be made of some of the fundamental operating variables of the process.

The experiments were carried on at the Forest Products Laboratory, Madison, Wisconsin. The material consisted of longleaf pine stumps from Louisiana, donated by the Long-Bell Lumber Company of Kansas City, Missouri. Acknowledgment is made to Mr. S. D. Wells, Engineer in Forest Products, of the section

of pulp and paper, for making the experiments on the suitability of extracted chips for pulp.

DESCRIPTION OF PROCESS

About fifteen patents have been granted in the United States for extraction processes and apparatus since 1909, and one or two plants were built and had been operated before that time. The different patents vary chiefly in the treatment of the wood before and after applying the solvent, in the manner of applying the solvent, and in the solvent itself. In general, the process, as carried out in the commercial plant, consists in first steaming the finely divided wood to recover the volatile constituents (turpentine and pine oil), followed in some cases by evaporating out of the wood as much of the condensed steam as possible. The solvent is then applied to the wood to dissolve out the rosin. After extraction, the wood is treated to recover the solvent adhering to, or absorbed by the chips. Evaporation of the solvent leaves the rosin as a residue, the solvent being recovered for subsequent extractions.

EXPERIMENTAL

APPARATUS—The apparatus used in the experiments was an extraction battery composed of three retorts, A, B, and C, shown in Fig. I. The chips were placed in the perforated baskets, D, each retort holding from 30 to 35 lbs. of wood. The battery was so arranged that the solvent could be forced by means of a pump, E, to a storage tank, F, from which it flowed by gravity into any one of the retorts and from that retort to each one below it. Each unit was independent of the others so that different operations could be carried on at the same time. Each retort was also connected to a vacuum pump, G, through a condenser, H, and receiver, I, and equipped with an open coil for direct steam and closed coils for heating the solvent.

As it was desired to boil the solvent in contact with the chips the apparatus was so arranged that this could be done without decreasing the solvent by evaporation. This was accomplished by connecting the vapor outlet line of each unit to a condenser, J, and receiver or trap, K, placed above the battery. The trap was connected to the bottom of each retort and thus allowed the vaporized solvent, after it was condensed, to flow continuously back into the retort as in a reflux condensing apparatus. By connecting the top of each retort to the top of the trap, the vapor conditions were equalized and the solvent was prevented from backing up. With these "equalizers" open, the solvent in any retort could be boiled under pressure for any desired length of time with a constant reflow of the condensed vapors.

The simple still, L, was used for the evaporation of the saturated solvent to recover the rosin. The still was equipped with a steam jet and was connected through the receiver, M, to the vacuum pump to facilitate the removal of water and heavy oils from the rosin.

SOLVENT—Since a petroleum distillate has been most frequently used commercially as a solvent there was selected for the experimental work, a special gasoline furnished on a guarantee to boil between 70 and 150° C.

It was found, however, that the material contained at least 20 per cent boiling under 70°, a portion boiling as low as 30°.[1] The solvent was, therefore, fractionated in a laboratory column (4 in. in diameter), the fraction below 70° C. being removed. By distillation in a Hempel column the solvent used gave 4 per cent boiling below 70° and 95 per cent boiling below 150°.

EXTRACTION—It was desired to make the laboratory study as comparable as possible with extraction in a continuous commercial extraction battery. In an extraction system of this type, all units receive exactly the same treatment and in no case is fresh solvent run on to fresh material, but the solvent most nearly saturated with the substance being extracted is run on to fresh material and the most nearly spent material
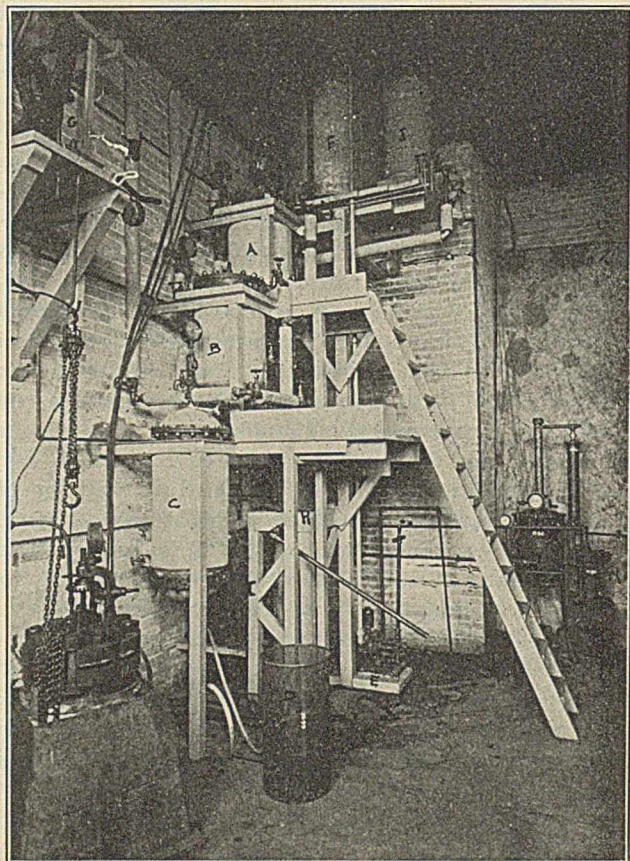
FIG. I—EXTRACTION BATTERY

receives a final treatment with fresh solvent. This condition was approximated in a laboratory test, providing a system of four washes for each unit by employing six solvents. Three of these were distilled at the end of each run and three were used for the next run without recovering the rosin. The method may be better explained diagrammatically by referring to Table I.

Solvents I, II and III in each run were distilled for the recovery of rosin, having extracted the rosin from the wood in four retorts, while Solvents IV, V

[1] In purchasing solvents of this type commercially, the importance of contracts calling for extremely rigid specifications is indicated by this experience. With such a large proportion of a very low boiling fraction in the solvent it would be impossible to prevent a high solvent loss.

TABLE I

RETORT	─Run 1─			─Run 2─		
	1	2	3	1	2	3
SOLVENT	I					
	II →	II				
	III →	III →	III			
	IV →	IV →	IV →	I		
		V →	V →	II →	II	
			VI →	III →	III →	III
				IV →	IV →	IV
					V →	V
						VI
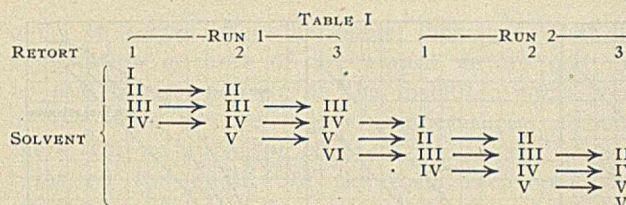
and VI become Solvents I, II and III, respectively, for the next run. Solvent I, in Retort 1; II, in Retort 2; and III, in Retort 3, represent the action of nearly saturated (theoretically) solvents on wood which has not been previously extracted. Solvent IV, in Retort 1; V, in Retort 2; and VI, in Retort 3, represent the final extraction of nearly spent chips with fresh solvent. Before beginning the first run, a preliminary run was necessary in order to secure Solvents I, II, and III, for the first test. After the retorts were charged in these tests, they were not opened until the dried extracted chips were removed.

The procedure used in conducting any test was briefly as follows: The chips were first steamed under 30 lbs. pressure[1] until no more turpentine and pine oil were obtained; the retorts were then connected to the vacuum pump from 30 to 60 min. to remove as much of the moisture as possible, steam being kept in the closed coils to facilitate evaporation. The solvent was then introduced until the chips were just covered and was then brought to boiling by means of the steam in the closed coils. The reflux system was open so that the vaporized solvent would condense, and flow back into the retort. In case the extraction was conducted under pressure the valves connecting the retort to the reflux were closed until the desired pressure in the retort was reached. The line connecting the retort with the reflux was then opened and the pressure controlled by the amount of steam passed into the closed coils. After extraction the solvent was transferred to the next retort, or in case it was the last extraction for that solvent, it was passed through a condenser which delivered it cold to the container. When the extraction was made under pressure, it was better to relieve the pressure before drawing out the solvent; this was readily done by turning off the steam and allowing condensation to continue in the reflux. When the solvent was drained out after the last extraction, that remaining in the chips was removed by steaming first at atmospheric pressure until no more solvent came off, and finally at 30 lbs. pressure for a few minutes. To insure that all the solvent was removed, the retort was then connected to the vacuum pump for about one hour. In only a few cases, however, was any more solvent recovered in this last step.

As soon as each solvent was removed after final extraction it was thoroughly stirred, and a sample taken. The weight of each solvent was taken before it was used in any run and after its final use in that run. The intermediate weights when a solvent passed from one retort to the next were not taken. In other words, the total amount of wood in the three units

of the battery was considered as the charge for the run rather than that in each retort as a separate unit. Since only three solvents were redistilled in any run and these contained some rosin from a previous run, it was not possible to determine the rosin yield from the actual rosin recovered by the redistillation of the solvent. The yields were determined by an analysis of the solvents. The total yields from several runs checked very well with the analyses.

ANALYSES

The efficiency of any extraction was based on a comparison of the amount of rosin in the solvents, as determined by an analysis, and the amount of rosin contained in the charge, as determined by an analysis of an average sample of the wood.

WOOD—A sample of the charge was taken by quartering the wood in each retort until the desired amount (about $1/2$ lb.) was obtained. A portion of the sample from each retort was then ground to the fineness of sawdust and a moisture determination made by the xylol method,[1] generally employed in estimating moisture in wood which contains a volatile oil. An equal portion of the finely ground sample from each retort was extracted in a Soxhlet with chloroform. On drying the extracted sample to constant weight at 110° C. the weight of the wood free from moisture, rosin, and volatile oil was obtained. The extract from the Soxhlet was then evaporated in an oil bath kept at 150° C. After the chloroform had all distilled off a small jet of steam at slightly reduced pressure was passed through the rosin. This was continued for $1/2$ hr. and the residue was then dried by using a much higher vacuum for $1/2$-hr. intervals, without the steam, until the rosin showed a loss of less than 0.2 per cent for two successive treatments. Continued heating caused some decomposition, so that drying could not be carried to constant weight. The final residue was then taken as rosin. Since moisture and rosin and the extracted wood were determined the volatile oil was estimated by difference. The method is not entirely satisfactory, but since it is not practicable to determine volatile oil in so small a sample, and thus determine rosin by difference, it was felt that rosin could be determined as the non-volatile chloroform extract. Duplicates checked within 0.003 to 0.005 per cent and errors that were large enough to be of consequence to the experiment fell back on the sample itself. It is, furthermore, not strictly correct to compare the petroleum extraction with the chloroform control since there were probably small portions of the rosin that were insoluble in petroleum but were soluble in chloroform.[2] The extraction efficiencies were, therefore, lower than if based on the total petroleum soluble rosin present in the wood.

EXTRACTION—The yield of rosin in the tests was determined, as stated above, by an analysis of the different extracts. The amount of rosin in a weighed portion (about 200 g.) was determined in practically the same manner described for estimating the rosin

[1] THIS JOURNAL, **4** (1912). 789; also F. S. *Bull.* **109**, "Distillation of Resinous Wood with Saturated Steam."

[1] F. S. *Circ.* **134**, "Estimation of Moisture in Creosoted Wood."
[2] Allen, "Commercial Organic Analysis," chapter on Colophony.
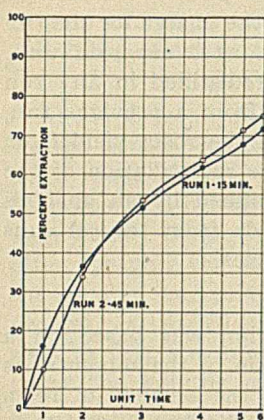
FIG. II—EFFECT OF TIME
Chip, 3/16 in.
Pressure, 0 lbs.
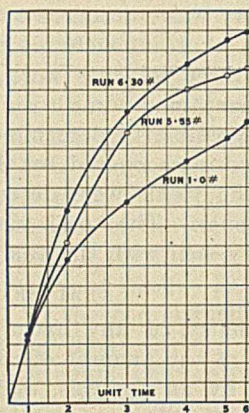
FIG. III—EFFECT OF PRESSURE
Chip, 3/16 in.
Extraction, 15 min.
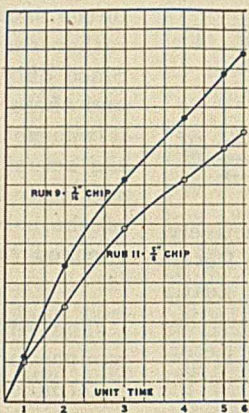
FIG. IV—EFFECT OF SIZE OF CHIP
Pressure, 30 lbs.
Extraction, 15 min.
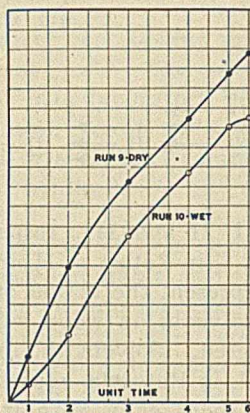
FIG. V—EFFECT OF MOISTURE
Chip, 3/16 in.; Pressure, 30 lbs.; Extraction, 15 min.
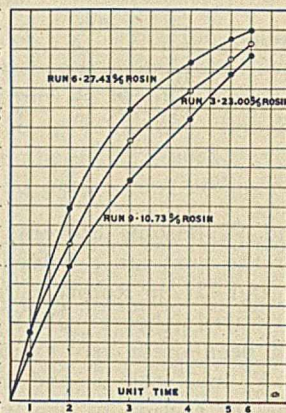
FIG. VI—EFFECT OF ROSIN CONTENT
Chip, 3/16 in.; Pressure, 30 lbs.; Extraction, 15 min.

in the chloroform extract, except that the residue was not steam-distilled because no appreciable amount of high boiling oils, such as pine oil, was left in the wood and rosin after the chips were thoroughly steamed.

SCOPE OF EXPERIMENTS

The experiments in this first series included a study of the more important fundamental operating variables on the efficiency of the extraction: (1) time, (2) pressure, (3) size of material, (4) moisture, and (5) effect of rosin content on the percentage yield of rosin, based on the amount originally present. The tests also included a preliminary study of the suitability of the extracted material for paper pulp. Two sizes of chips, averaging 3/16 in. and 5/8 in. with the grain of the wood, were selected for the tests. The 3/16-in. chip is larger than the usual commercial chip used in the extraction process, but is considered the minimum size for making pulp. The material was prepared in a semi-commercial pulp chipper. The wood in each case comprised the whole of the stump including the outer bark. All of the stumps had the roots removed and were practically free from earth and sand.

TABLE II—EFFECT OF VARIOUS FACTORS ON EXTRACTION OF ROSIN

EFFECT ILLUSTRATED	Run No.	TIME Min.	CHIP In.	PRESSURE Lbs.	% ROSIN IN WOOD	ROSIN YIELD Per cent
TIME	1	15	3/16	0	29.92	72.6
	2	45	3/16	0	25.10	75.0
	3	15	3/16	30	23.00	91.60
	4	45	3/16	30	25.92	93.70
	11	15	5/8	30	12.90	68.5
	12	45	5/8	30	12.23	68.9
PRESSURE	1	15	3/16	0	29.92	72.6
	6	15	3/16	30	27.43	94.3
	5	15	3/16	55	29.35	85.3
	2	45	3/16	0	25.1	75.0
	4	45	3/16	30	25.92	93.7
SIZE OF CHIP	9	15	3/16	30	10.73	88.8
	11	15	5/8	30	12.9	68.5
	4	15	3/16	30	25.92	93.7
	12	45	5/8	30	12.5	68.9
MOISTURE, High	9	15	3/16	30	10.73	88.2
Low	10	15	3/16	30	10.7	72.1
ROSIN CONTENT	7	15	3/16	0	13.32	70.8
	1	15	3/16	0	29.92	73.6
	9	15	3/16	30	10.73	88.8
	3	15	3/16	30	23.00	91.6
	6	15	3/16	30	27.43	94.8

RESULTS

EFFECT OF TIME—Fig. II, based on the results of Runs 1 and 2 (Table II), shows the progress of the ex-

traction. The curve is obtained by plotting the percentages of rosin recovered as ordinates, and the time the solvent was in contact with the wood as abscissas. Referring to Table I, which shows the procedure used in the test, it is seen that in any run Solvent I is in contact with the wood only for the unit time, which is 15 min. for Run 1. Solvent II, however, is in contact with the wood twice as long as Solvent I, Solvents III and IV three times as long as Solvent I, and Solvents V and VI for the same time as Solvents II and I, respectively. The units on the abscissa, therefore, represent the progress of extraction. Since each solvent was analyzed before its first and after its last application, the proportion of the total rosin extracted by each solvent in any run could be determined.

It will be seen that the effect of increasing the time from 15 to 45 min. is slight for the three sets of conditions used, and for practical purposes 15 min. is evidently the maximum time necessary. In Runs 3 and 4, which gave the highest yields, the efficiency was increased only 2.1 per cent when the time was increased threefold. In Fig. II it will be noticed that in the 15-min. extraction a larger proportion was recovered with the first two solvents than when the time was 45 min. This same condition was shown in corresponding curves for Runs 3 and 4 and 11 and 12, but no logical explanation of this was made evident during the experiment.

EFFECT OF PRESSURE—The progress of the extraction for Runs 1, 5 and 6, shown in Fig. III, is typical of the effect of pressure. The figure shows that the rosin recovered was increased from 72.6 per cent to 94.3 per cent by increasing the pressure from 0 to 30 lbs. At 55 lbs. pressure there is an apparent break in the curve, and a decided decrease in per cent recovery as compared with 30 lbs. pressure. Since, in a study of the penetrance of creosote in longleaf pine heartwood at the Forest Products Laboratory,[1] a decided decrease was noted as the pressure was increased from 50 to 75 lbs., the cause of the decreased efficiency of extraction may be a physical

[1] U. S. Dept. of Agric., *Bull.* 101, "Relative Resistance of Various Conifers to Injection with Creosote."

one. It seems more probable, however, that the explanation is more of a chemical nature and may be due to an increase of the insoluble constituents of the rosin at the higher temperatures, although there was no apparent effect of the higher temperatures on the quality of the rosin recovered. The effect of increasing the pressure in the 45-min. extraction was practically the same as for the shorter time.

EFFECT OF SIZE OF CHIP—Fig. IV shows the curve of extraction for Runs 9 and 11. Increasing the size of the chip to $5/8$ in. decreases the efficiency of extraction from approximately 90 to about 70 per cent, and the result is practically independent of the time of extraction. In the studies of the penetrance of creosote in longleaf pine heartwood referred to above, it was also shown that the ratio of longitudinal to radial penetration was 26 : 1, and of longitudinal to tangential was 100 : 1. For practical purposes the action of the solvent is, therefore, almost entirely in the longitudinal direction, which is also the same direction as most of the resin ducts, that is, with the grain in the wood. The decrease in the rosin removed from the longer chips is then undoubtedly a question of decreased penetration of the solvent into longer resin ducts. Since giving the solvent a longer time to penetrate does not increase the yield, as seen in Runs 11 and 12,[1] it seems safe to predict that additional pressure is necessary to penetrate to the center of the longer chips. A continuation of those studies will determine the accuracy of this supposition.

EFFECT OF MOISTURE—After steaming the chips for the removal of volatile oil preparatory to extraction, it was the usual procedure to pull a vacuum of about 20 in. on the retort for $1/2$ hr., the retort being kept hot by means of steam in the closed coils in order to remove as much as possible of the excess moisture caused by steaming. In general, the water removed in this way was equivalent to about 30 per cent of the dry weight of the wood, but the ratio of this amount to the total moisture in the chips after steaming was not determined. As several patents call attention to this feature of the process it was desired to determine its effect. A run was made, therefore, in which the vacuum was omitted and the solvent was run directly on to the chips after steaming. The results of this run compared to a similar run in which the vacuum was used are given in Table II and the progress of the extraction for the two runs is shown in Fig. V.

The effect of removing a comparatively small amount of moisture by the vacuum treatment is more pronounced than might be supposed, since the efficiency is increased over 15 per cent. It will be noted in the curve that the extraction is most retarded at the start; and it is, therefore, quite possible that the excess of moisture is removed by the first wash, after which the extraction proceeds more normally. When the water has once boiled off with the gasoline vapor

[1] The more pronounced effect of increasing the time used in Run 4 to that used in Run 9 may be largely explained by the higher rosin content of the wood used in Run 4, as will be shown in the discussion to follow.

and, on condensing in the reflux, flows back into the bottom of the retort, it probably does not interfere to any extent with the extraction. By taking the condensed solvent from the top of the trap in the reflux line it would be possible to get rid of this excess water in a short time, since the moisture could be drawn off at the bottom of the trap and thus be removed from the system. With such an arrangement, increasing the time of extraction should then give almost as high efficiency as when the chips are dried by the vacuum unless, in addition to removing the surface moisture, the vacuum has some mechanical action of opening up the ends of the exposed resin ducts and thus facilitates the penetration of the solvent. It is desired to test this point in future experiments.

EFFECT OF ROSIN CONTENT—These tests on the effect of different variables were made on samples of quite different rosin content, one lot being comparatively rich and varying from 23 to 30 per cent rosin, and another lot being comparatively low in rosin and varying from $10^{1}/_{2}$ to $13^{1}/_{2}$ per cent. An analysis of the data (Table II) from runs made under the same extraction conditions but with chips of different rosin content is of interest.

Runs 3, 6 and 9, made at 30 lbs. pressure and 15 min. extraction, are shown graphically in Fig. VI. It is seen from the table that a higher percentage of rosin is recovered from woods of greater rosin content and the higher recovery is evident throughout the progress of the extraction as shown in the figure. This is probably due to the fact that there is a larger proportion of the rosin which has saturated the cells surrounding the resin ducts proper in the richer wood and this rosin is, therefore, made more accessible to the action of the solvent. The wood containing 13.5 per cent rosin is about as low in rosin as could be extracted commercially.

SOLVENT LOSS

The apparatus used in the test was not found to be entirely suitable for the study of the loss of solvent. Because of errors in construction the solvent collected in pockets in the piping and it was, therefore, not recovered until another run. There were also noticeable leaks at several points. In general it was apparent that extraction under pressure tended to increase slightly the amount of solvent retained in the chips but there was no difficulty in recovering this solvent on subsequent steaming. With evident leaks in the apparatus, increasing either time or pressure

TABLE III—SOLVENT LOSSES IN VARIOUS RUNS

Run No.	Chip In.	Pressure Lbs.	Time Min.	Solvent Loss Per cent	Rosin Yield Per cent
7	3/16	0	15	2.38	72.6
2	3/16	0	45	6.87	75.0
3, 6, 9 (Av.)	3/16	30	15	3.46(a)	91.5
4	3/16	30	45	4.30	93.7
5	3/16	55	15	3.74	85.3
11	5/8	30	15	3.46	68.5
12	5/8	30	45	3.85	68.9

(a) Varied from 3.16 to 3.79.

would, of course, increase the solvent loss. The amount lost in several of the runs is shown in Table III. While the data do not show clearly the effect of the different time and pressure conditions on sol-

vent loss, it may be noted that a longer time seemed to give a greater increase in solvent loss than a higher pressure.

It was possible to determine approximately the proportion of this loss that occurred in the redistillation of the solvent and this averaged about 10 per cent of the total. The solvent loss will be taken up in detail in a later study.

FORMATION OF A PRECIPITATE

An interesting point that came up in the experiments was the discovery of a precipitate in the solvent removed from the chips, especially after extracting wood of high rosin content. Yaryan[1] calls attention to a black pitchy substance which precipitates out of the rosin solution and which he says is due to the action of fire on the stumps or lightwood. The precipitate was at first dark brown, later becoming black; it was quite sticky, and was first thought to be some constituent of the rosin insoluble in petroleum. Further examination indicated that it was largely basic ferric acetate which carried down a small amount of rosin with it. It was no doubt formed by the attack of the iron retorts by the free acetic acid occurring in the old wood, the acetate of iron formed becoming the insoluble basic salt at the high temperature of the boiling solvent. In some of the runs the precipitate amounted to as much as 1 per cent of the dry weight of the wood.

SUITABILITY OF THE EXTRACTED CHIPS FOR PULP

Only preliminary experiments have been made with the extracted chips. It was desired first to determine the quality of pulp that could be obtained from the $3/16$-in. chip although it seemed that this size would give too short a fiber length for good pulp. By using the sulfate process the unscreened chips from Runs 6 and 9 were cooked in a semi-commercial digester. The time of cooking was 1 hr. in getting up the pressure, and 2 hrs. at 100 lbs. pressure. The cooking liquor contained 15.7 lbs. NaOH and 7.5 lbs. Na_2S per 100 lbs. of wood. The pulp yield was 43.2 per cent of the dry chips. Two beater treatments were made on the "half stuff," the duration of beating being 5 hrs. in each case. In one run the beater roll was hard down during the last hour, while in the second run the stock was much more dilute and the roll was set so as to give a comparatively light brush. The pulp was run over the machine and the sheets from the two beater treatments gave the following strength tests. The figures from what may be considered a No. 1 Kraft are given in the following table for comparison.

Paper	Strength Factor Points per lb.	Schopper Test Breaking Length in Meters	Folding Test Times	Weight per Ream Lbs.
Beater Run No. 1	0.72	5425	800	40
Beater Run No. 2	0.65	4730	367	38
No. 1 Kraft	1.00	6000	1500	40

While Run 1 was stronger, Run 2 gave a softer sheet. Both were a very fair grade of No. 2 Kraft. Further tests will be made by using the larger chips, but since the small chip gives such promise of being

[1] U. S. Patent 934,257.

suitable for pulp, they are more desirable, since it is possible to get so much higher yields of rosin from them by extraction.

CONCLUSIONS AND THEIR COMMERCIAL APPLICATION

The results of these experiments seem to indicate that a solution of the problem of obtaining another product in the extraction of resinous wood by the solvent process may be found in using a larger chip, whose minimum size is $3/16$ in., and extracting under a maximum pressure of 30 lbs. by using four washes of 15 min. each. A closed type of battery in which the vaporized solvent can be returned continuously to the retort is advantageous for extracting under pressure. Extraction under pressure for a short time is shown to give much higher yields of rosin than no pressure for a longer time.

As noted in the results given above, the yields by petroleum extraction are based on control analyses made by using chloroform as solvent. It was observed that certain constituents of the rosin are insoluble in petroleum but are soluble in chloroform. Since it is also shown that increasing the time of extraction has no appreciable effect at 30 lbs. pressure, and since 94.8 per cent were recovered under those conditions, it would seem possible that practically all of the petroleum-soluble rosin was extracted at this pressure. It is probable, therefore, that a lower pressure would be as efficient as 30 lbs.

Chips as small as $3/16$ in. are apparently suitable for paper pulp as they gave a good grade of No. 2 Kraft by the sulfate process. Chips of this size could probably be most advantageously cooked commercially in a rotary type of digester. A plant extracting wood yielding about 250 lbs. or 1.09 bbls. of "F" grade rosin per ton (89 per cent yield of 14.0 per cent rosin), which is about the minimum for practicable commercial operation, would produce extracted chips suitable for making about 750 lbs. of pulp per ton of wood. A 50-ton extraction plant would then supply a 20-ton pulp mill, or since an extraction plant might require about three-quarters of its extracted chips as fuel in case it were not advantageous to buy other fuel, a 200-ton plant would have chips for fuel and still could supply a commercial pulp mill of practicable size.

It is difficult to give cost figures to cover the variety of contingencies met with commercially, but the following estimates of average practice show clearly the effect on the net returns of obtaining extracted chips as an additional valuable by-product.

COST ESTIMATE PER TON ON 200-TON EXTRACTION PLANT

Cost of Operation	Prices for past 15 yrs.	Returns at Average	
Wood	$ 2.25	6 gals. turpentine and pine oil at 40 c.	$ 2.40
Solvent: $7\frac{1}{2}$ gals. at 12 c.	0.90	250 lbs. rosin at 1.5 c.	
Fuel: $3/4$ ton extracted chips at no cost	0.00	($3.77\frac{1}{2}$ per net bbl. of 230 lbs.)	3.75
Cooperage	0.65		
Superintendence and Labor.	0.87		$6.15
Selling and shipping, including freight	0.72	Cost of operation	5.92
Taxes, insurance and depreciation over 15 years	0.35	Net profit	$0.23
Repairs and miscellaneous	0.18	Selling price 250 lbs. extracted chips at $1.00 per ton	0.25
	$5.92		
		Net profit with chips as by-product	$ 0.48

The plant would pay 5.5 per cent on an invest-ment of $250,000 without the chips and 11.5 per cent assuming the chips to be worth $1.00 per ton for pulp, which is a low estimate since the material would be in condition for immediate pulping.

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SOME STUDIES OF SOAP SOLUTIONS

By VICTOR LENHER AND MARY V. R. BUELL
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The chemical analysis of a soap shows only the con-stituents of which it is composed. The results indi-cate the content of water, fatty acid as soap, un-saponifiable matter, alkali (combined or free), glyc-erin, salt, filler, etc. No satisfactory method has been proposed for the determination of the real value of a soap, namely, its cleansing power. From the time of Chevreul, one of the first to study soap, to the present day, considerable literature has accumula-ted on the cleansing power of soap. Some of the work is experimental, and some is purely speculative, but with all the thought and energy which has been ex-pended on this subject it must be confessed that we do not possess sufficient data to explain satisfac-torily the phenomena ordinarily exhibited by soap solutions, nor can the chemist tell the laundry how much soap is required to remove a definite amount of dirt or wash a given weight of soiled clothing.

Various phases of the question as to how soap acts as a detergent have been studied at different times. These studies are well summarized by Bancroft in his papers on emulsification, now running in the *Journal of Physical Chemistry.*

The various methods which have been suggested from time to time for the evaluation of soaps, have depended largely on the use for which the soap is in-tended. Hillyer[1] divides soaps into two classes: those used with cold or lukewarm water, such as toilet soaps; and those used with boiling water, as laundry soaps. His method for determining the de-tergent value of soap is by the Traube stalagmometer method for determining the surface tension of a soap solution against a kerosene which he arbitrarily adopted as a standard. The number of drops formed in a given length of time is regarded as a measure of the amount of soap in solution, the emulsifying power of the soap, and its cleansing power. The num-ber of drops is referred to a curve obtained by running standard solutions of neutral sodium palmitate through standard kerosene and the relative efficiency of the soap is thus approximated. In testing soaps intended for use with hot water, the whole of the apparatus is surrounded by a water bath which is kept at the de-sired temperature.

Luksch[2] attempted to determine the efficiency of a soap by coloring pieces of chiffon of different sizes and washing them under the same conditions in a washing machine. The results varied 10 per cent, but were sufficiently accurate to show the difference between different soaps. He also studied the weaken-

[1] *J. Am. Chem. Soc.*, **25**, 1256.
[2] *Seifensieder-Ztg.*, **40**, 413, 444.

ing effect of the soaps on the fibers by making tensile strength determinations.

SURFACE TENSION—In most of the theories which have been brought forth to explain the detergent value of soap, surface tension has played an important part. In order to study the relation between surface tension and concentration, a number of experiments were made by means of the Traube stalagmometer. While the Traube stalagmometer method for surface tension determinations possesses certain objections, these errors are of minor rather than major character. It was found to suffice in this work, inasmuch as the results are comparative.

A bulb was blown in the stalagmometer so that 5 cc. would be the quantity delivered, and a device used whereby the pressure could be regulated. The whole was surrounded by a water bath and the outlet al-lowed the drops to form in the air or in a given liquid. In the measurements made by this method, the num-ber of drops were counted and the time taken when a definite volume of liquid was allowed to flow out through the pipette.

Sodium oleate solutions were prepared from pure sodium hydroxide and very pure oleic acid. When soap solutions are made to form drops under the sur-face of an oil or of a liquid immiscible with water, the number of drops formed is much greater than when an equal volume of water is made to form drops under similar conditions, that is, the surface tension be-tween oil and water is much greater than the surface tension between oil and soap solution, or, the surface tension is inversely proportional to the number of drops formed. The relation between concentration of soap solutions and their surface tensions toward some liquids immiscible with water is shown in the accompanying curves. In all cases the temperature was controlled at 25°.

The first point on each of the curves is not as accurate as the others because at the $N/10$ concentration hydrolysis is fairly rapid. The significance of the points is that they show the general direction of the curves. The error of the observations is considered to be approximately 0.5 per cent. Theoretically, the point of minimum surface tension should be at the point at which the maximum number of drops is formed. In all of the cases investigated, with the single exception of the experiment with air, the sur-face tension was lower with the concentrated solutions than with the dilute solutions, and the general direc-tion of the curves is the same.

It has been observed that the surface tensions of sodium oleate solutions toward air are increased with the dilution. The actual surface tensions of various concentrations of sodium oleate solution toward air were found by the Traube stalagmometer method to be at 25° as follows, expressed in dynes per centimeter:

$N/10$	$N/20$	$N/40$	$N/80$	$N/160$	$N/320$	$N/640$	$N/1280$
28.20	26.21	25.33	24.17	24.77	24.91	25.55	41.28

By the same method water showed 71.78 toward air. It is interesting to note that with $N/10$ to $N/320$ sodium oleate there is such a small variation in the

surface tension, especially when the surface tension is so different from that of water. The solutions in this range of concentration are the ones which foam so well. When a dilution of $N/640$ is attained the solutions commence to take on a milky appearance. The fact that the foaming is so much more profuse between $N/80$ and $N/320$ affords evidence to the view that in these concentrations we have the soap acting as an emulsifier to produce the foam which is an "emulsion of air in soap solution."

While the above observations were taken at 25°, a study was made at temperatures of 20, 22, 24 and 25°, and it was found that within this range of temperature the surface tension measurements if plotted as a curve form a straight line, showing that the rate of flow is directly proportional to the temperature.

EMULSIFYING ACTION OF SOAP

Following the lines of our modern methods of preparing emulsions, the emulsifying power of soap was studied. Emulsions of kerosene and water were made by shaking kerosene with an excess of soap solution in a wide-mouthed, glass-stoppered bottle. After the mixtures had stood for a few minutes, the excess of soap solution separated out at the bottom of the bottle, leaving all of the kerosene emulsified in the upper layer. These emulsions remained permanent for months. If insufficient soap is present when one attempts to make an emulsification of this character, either no emulsification or only partial emulsification takes place.
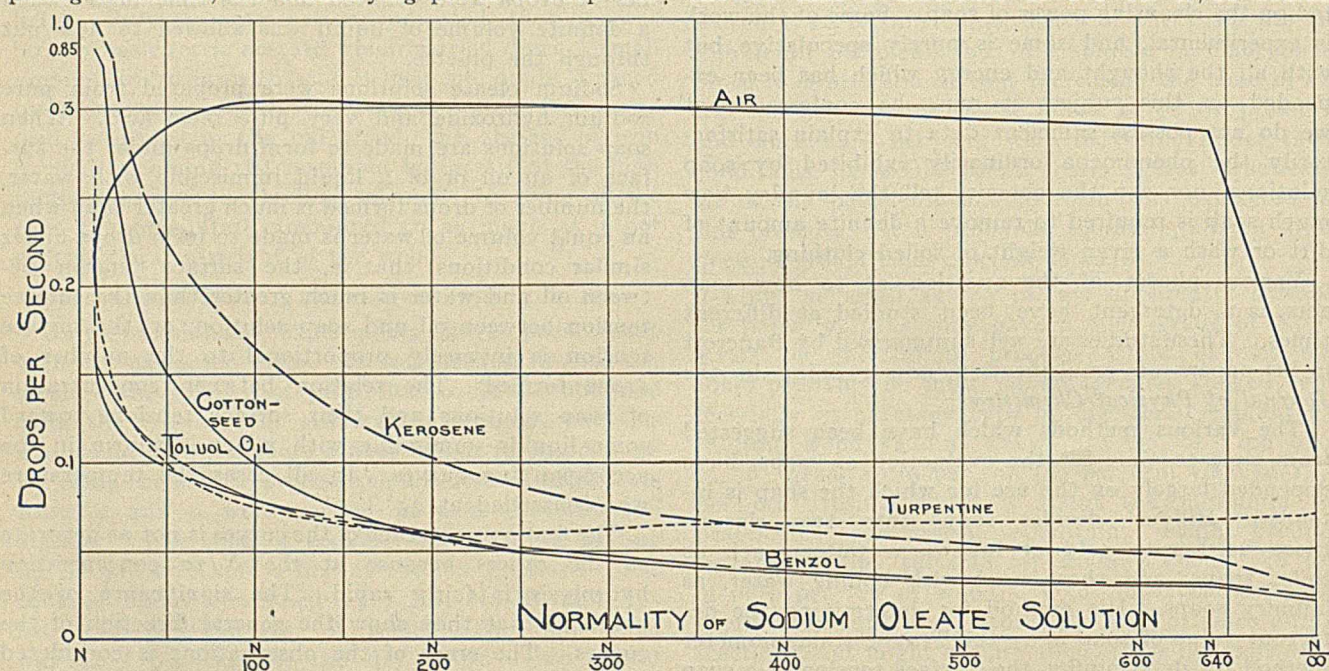
In order to study this emulsifying action of the soap with water and immiscible liquids, it is noted that, as with other emulsions, simply a uniform shaking, such as given by a shaking machine, is insufficient. A shaking machine, even when running at high speed, gives unsatisfactory results. In fact, a shaking machine does not give as satisfactory results as does the jerky double motion given by hand. The apparatus found to be best adapted for the purpose was a thin stirring paddle made of German silver in which a large number of holes were cut. The paddle was electric motor-driven and the jar in which it was revolved was immersed in a constant temperature bath.

Whenever possible, equal volumes of water and immiscible liquid were used and the amounts of soap varied. In the first experiments, the object was to ascertain the minimum quantity of soap which would act as an emulsifier under definite conditions. An emulsion was considered satisfactory if there was no visible separation of either constituent at the end of 5 min., the time of stirring being 5 min. This standard is not so arbitrary as it might seem at first sight, because in most cases if the emulsion separates at all, it does so in the first half-minute. This is especially true of the emulsions produced at the lower temperatures. A test of the accuracy of the method showed that a difference of 0.1 cc. $N/20$ sodium oleate solution, corresponding to 0.0015 g., could be readily detected. In the results given, the accuracy has not been worked out to such a high degree, but is approximately 0.5 cc. $N/20$ sodium oleate, or, 0.0075 g. The experiments were conducted at 20° and the water used was highly purified.

Substance	Water Cc.	Sodium Oleate Necessary for Emulsification Grams
50 cc. Turpentine	50	0.6331
50 cc. Carbon Tetrachloride	50	0.1140
50 cc. Chloroform	50	0.7824
50 cc. Benzol	50	0.9804
2.5 g. Vaseline	50	1.5200

It is obvious that entirely different amounts of soap are necessary to emulsify under the same conditions the same volume of different substances which are immiscible with water. The experiment with vaseline can hardly be compared with the others, nor is its accuracy so great on account of the physical condition of the vaseline, but is introduced simply to show its behavior under these conditions.

EFFECT OF CONCENTRATION ON EMULSIFICATION

The effect of the relative amount of water present in emulsions with turpentine was observed.

TURPENTINE Cc.	WATER Cc.	$N/20$ SODIUM OLEATE NECESSARY Cc.	TOTAL VOLUME OF WATER Cc.	SODIUM OLEATE NECESSARY Gram
25	0	30	30	0.4560
25	25	18	43	0.2736
25	50	8	58	0.1216

It follows from these results that weak solutions of sodium oleate are more economical in the emulsification of turpentine with water than the more concentrated solutions. It is, therefore, apparent that the volume of water in which a given amount of soap is dissolved is an important factor in its detergent effect.

EFFECT OF TEMPERATURE ON EMULSIFICATION

TEMPERATURE	SUBSTANCE	WATER Cc.	SODIUM OLEATE NECESSARY Gram
20° C.	50 cc. Chloroform	50	0.7828
50°	50 cc. Chloroform	50	0.4636
20°	50 cc. Olive Oil	50	0.6384
100°	50 cc. Olive Oil	50	0.3040

It is evident from these observations that a definite amount of sodium oleate will emulsify more of these materials at high temperatures than at low temperatures. The emulsions produced are, however, not so stable at high temperatures as the low temperature emulsions. The emulsions were, however, permanent for 5 min. in all cases. For this kind of a study no objection can be raised to emulsification of this character, inasmuch as in the actual process of washing, as soon as the emulsion is formed, it is washed away. It is in general true that sodium oleate solutions wash more efficiently at high temperatures than at low. Soaps which are relatively high in oleic acid content are often spoken of as cold water soaps. From this it might be inferred that they wash better in cold water than in hot. This is not the case. It is true that they wash better in cold water than the stearate or palmitate soaps, which have only a slight detergent action in cold water. In general, the oleates wash better in hot water than in cold.

EFFECT OF AGE ON SOAP SOLUTION

Strong solutions of sodium oleate decompose on standing with the formation of a precipitate which in chemical composition is an acid soap. This precipitate is relatively insoluble and cannot be entirely brought back into solution by boiling. Experiments were made to find the relative emulsifying power of fresh $N/10$ sodium oleate solutions, and of $N/10$ solutions in which this precipitate had formed. The solutions were thoroughly shaken in order to insure the precipitate being uniformly suspended through the solution.

SUBSTANCE	WATER ADDED TO MAKE UP TO Cc.	AGE	SODIUM OLEATE NECESSARY Gram
50 cc. Chloroform	50	Fresh	0.7828
50 cc. Chloroform	50	3 mo	0.7904
50 cc. Chloroform	50	4½ mo.	0.7965

From these experiments it is evident that the emulsifying power of sodium oleate solutions is impaired only very slightly by the hydrolysis, and a soap solution which has stood for a long time washes appar-

ently as well as water in which the soap has been freshly dissolved.

COMPARATIVE EMULSIFYING POWERS OF THE SODIUM SOAPS WHEN USED HOT

A comparison of the emulsifying powers of pure sodium oleate, stearate and palmitate has been made. $N/20$ solutions of the three soaps were prepared from the pure fatty acids and pure sodium hydroxide. The solutions of the stearate and palmitate were prepared hot. Both the stearate and palmitate solutions solidify to a jelly on cooling. It is obvious that when a sufficient concentration is necessary to emulsify an immiscible liquid with water, it is necessary that these soaps be used at a high temperature. Such liquids as benzol, carbon tetrachloride and chloroform as were used at lower temperatures were not serviceable at 100°. Highly inflammable liquids could not be used in our apparatus; hence olive oil was selected as the most satisfactory liquid for the purpose. The end-points are not as definite at high temperatures as at 25°. The oil was well emulsified by very small quantities of soap, but a considerable excess of soap is necessary to render the emulsions permanent for 5 min.

TEMPERATURE	OLIVE OIL Cc.	WATER TO MAKE UP TO Cc.	REQUIRED FOR EMULSIFICATION
100° C.	25	25	0.1672 g. Sodium Oleate
100°	25	25	0.1390 g. Sodium Palmitate
100°	25	25	0.2250 g. Sodium Stearate

In this comparison it appears that at 100° sodium palmitate emulsifies olive oil more efficiently than the oleate or the stearate. These data are very suggestive as to the relative detergent values of the three soaps at the boiling temperature, but more experiments should be made along these lines with various substances before it is possible to generalize.

SUSPENSIONS

In studying the action of soap solutions with suspended matter, such as ferric oxide and manganese dioxide, concentrations of sodium oleate from $N/20$ to $N/1280$ were used. 250 cc. of the sodium oleate solutions were shaken up with the sifted minerals and allowed to stand from 5 days to 3 weeks. The results with the two minerals were of the same order, and though it was impossible to make accurate quantitative measurements, it was noted that the maximum suspension invariably occurred with about $N/320$ solution.

Further studies on soap solutions are in progress in this laboratory.

UNIVERSITY OF WISCONSIN, MADISON

VANILLA EXTRACT

By J. R. DEAN AND J. O. SCHLOTTERBECK
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(Concluded from our previous issue)

SECOND-GRADE EXTRACTS

The conclusion reached in regard to the use of alkali was entirely in regard to a first-class extract where the aroma of the extract is of the greatest importance. It is often desirable, however, to prepare second-

grade extracts, either from second-grade beans or from the dregs left in the manufacture of the highest grade product. Here it would seem that the use of a little alkali might prove of value. These secondary extracts are never of as high a quality as the first-grade extracts and are usually incorporated into substitutes or imitations with coumarin or tonka extract. By the careful use of alkali it would be possible to obtain a nicely colored product and, although its flavor would not be equal to a pure extract, the addition of vanillin, coumarin or tonka extract, would tend to make up this lack of flavor. Such an extract could not be sold as a pure extract of vanilla and it is not a very difficult problem to prove that such an extract is not official.

It is doubtless certain that there are a great number of such extracts on the market, some of which are represented to be straight extracts. Their flavors differ greatly, but some are fairly good and are comparable with some straight extracts. There is a difference, however, and it will usually make itself appar-

The behavior of the ash indicated that an alkali had been used on exhausted beans and that the extract had not been made from prime vanilla beans. The flavor of this extract was fairly good, but it was far from being equal to a pure extract of vanilla.

SUGAR, SAND AND GLYCERIN

Sugar and sand, independently and together, packed with the beans in the percolator, tend to make possible a more uniform pack and reduce the chances of the menstruum forming canals through the pack, leaving part of the beans untouched. We made several extracts with both sugar and sand and each separately to see if either would be of any more service than the other in yielding a more complete extraction, but it was found that they are of about the same value.

In Table I we report the results obtained from extracts made with both, together and independently. In some of these, especially those made from undried beans, there were noticeable variations, but the variations were not constant and often conflicting. This

Beans	Percentages Alcohol	Vanillin	Lead No.	Extract Color Red	Yellow	Filtrate Color Red	Yellow	Ratio of Red to Yellow Extract	Filtrate	Remarks
Mexican	60	0.19	0.70	33.0	103.0	1.8	7.1	1 : 3.1	1 : 3.9	Alcohol and sand
	60	0.19	0.65	35.0	103.0	2.0	8.6	1 : 2.9	1 : 4.3	Alcohol and sugar
	60	0.16	0.69	41.0	123.0	2.0	8.8	1 : 3.0	1 : 4.4	Alcohol and glycerin(b)
	35	0.17	0.82	31.0	75.0	1.8	6.6	1 : 2.4	1 : 3.7	Alcohol and sand
	35	0.17	0.79	28.0	75.0	2.0	7.5	1 : 2.4	1 : 3.8	Alcohol and sugar
	35	0.18	0.80	40.0	90.0	2.0	7.5	1 : 2.3	1 : 3.8	Alcohol and glycerin
Bourbon	60	0.19	0.61	34.0	108.0	1.9	8.2	1 : 3.2	1 : 4.3	Alcohol and sand
	60	0.19	0.57	39.0	119.0	2.4	10.0	1 : 3.1	1 : 4.2	Alcohol and sugar
	60	0.20	0.58	43.0	133.0	2.0	9.6	1 : 3.1	1 : 4.8	Alcohol and glycerin
	35	0.17	0.71	30.0	73.0	1.8	7.0	1 : 2.4	1 : 3.9	Alcohol and sand
	35	0.17	0.66	31.0	78.0	2.2	8.0	1 : 2.4	1 : 3.6	Alcohol and sugar
	35	0.19	0.68	50.0	110.0	2.4	8.4	1 : 2.2	1 : 3.5	Alcohol and glycerin
Tahiti	60	0.11	0.43	13.0	40.0	0.6	2.6	1 : 3.1	1 : 4.3	Alcohol and sand
	60	0.11	0.44	15.0	40.0	0.6	3.1	1 : 2.7	1 : 5.2	Alcohol and sugar
	60	0.11	0.43	24.0	53.0	0.7	3.3	1 : 2.2	1 : 4.7	Alcohol and glycerin
	35	0.09	0.39	15.0	30.0	1.2	4.0	1 : 2.3	1 : 3.3	Alcohol and sand
	35	0.09	0.44	15.0	33.0	1.3	4.4	1 : 2.2	1 : 3.4	Alcohol and sugar
	35	0.10	0.40	15.0	33.0	0.9	3.3	1 : 2.0	1 : 3.6	Alcohol and glycerin
1st Grade	..	0.20	0.49	24.0	77.0	1.5	6.1	1 : 3.2	1 : 4.2	....
2nd Grade	..	0.20	0.53	31.0	92.0	1.7	7.2	1 : 3.1	1 : 4.4	....
3rd Grade	..	0.19	0.55	32.0	104.0	1.8	7.6	1 : 3.3	1 : 4.3	....
4th Grade	..	0.17	0.58	35.0	113.0	2.1	8.7	1 : 3.2	1 : 4.1	....
5th Grade	..	0.15	0.59	35.0	111.0	1.9	7.5	1 : 3.1	1 : 4.0	....
20 to 23 cm. long	..	0.20	0.52	30.0	99.0	1.8	7.6	1 : 3.3	1 : 4.3	....
15 to 19 cm. long	..	0.18	0.55	32.0	100.0	1.7	7.2	1 : 3.2	1 : 4.3	....
10 to 14 cm. long	..	0.18	0.56	33.0	104.0	2.1	8.4	1 : 3.2	1 : 4.1	....

(a) U. S. Dept. of Agr., Bur. of Chem., *Bull.* **152**, p. 157.　(b) The beans in the glycerin extracts were rubbed up twice their weight of sand. The glycerin extracts contained 20 per cent glycerin.

ent on a close comparison with a pure extract. The chief difference lies in the delicacy of the flavor and in the way in which these extracts deteriorate with age.

The analysis of some of these extracts compare very well with the analyses of a pure extract in regard to vanillin and color, and often even in the lead number value. The total acidity of an extract made with alkali is usually lower and the alkalinity of the ash is usually higher than that of a pure extract. These cheap and alkali-made extracts yield an ash more quickly than do the pure extracts and the nature of the ash is often very different.

We had occasion to examine an extract that was called "Extract of Vanilla," "Non-Official," and which was claimed to have been made from prime vanilla beans. The vanillin content was normal, about 0.18 per cent; the lead number was low, about 0.300; the color was normal and the extract was perfectly clear. The total acidity, however, was only half of that of a pure extract and it was found that the solids of the extract burned with great ease, leaving an ash that contained a very large amount of iron oxide.

was especially so in regard to the dried bean extracts where about the same results were obtained from three extracts made in all three ways. Thus in extract No. 16, the lead number was 0.8034 and both sugar and sand were used; in extract No. 17, the lead number was 0.8011 and only sand was used; and in extract No. 24, the lead number was 0.8095, and only sugar was used. The color values of all of these three extracts were about the same. The samples containing the sugar were a little cloudy and this was found to be the case in every extract made by packing the beans and sugar together in the percolator, and would, it seems, prove that sugar should never be packed with the beans but should be added to the percolate.

Glycerin, however, does tend to increase the color of the extract and should be added to the menstruum. In Table IV we have copied, in part, the results obtained by the Bureau of Chemistry of the Department of Agriculture, in their work on vanilla extract relative to the use and value of 35 and 60 per cent alcohol, with and without sugar, sand and glycerin.[1]

[1] U. S. Dept. of Agr., Bur. of Chem., *Bull.* **152**. 158.

These results agree with ours in that glycerin is of value in making a darker extract. And, also, that there is no preference between sand and sugar.

Glycerin also serves to give the desired "body" or gravity to the extract and imparts a sweet taste that is often desired. Some manufacturers use both sugar and glycerin in their extracts, the glycerin being added to the menstruum and the sugar to the percolate.[1]

GRADE AND LENGTH OF BEANS

In Table IV we have also included the results obtained by the above observers on extracts made from vanilla beans of different grades and lengths. These figures show that, in general, the highest grade beans of all the various varieties contain the most vanillin, decreasing as the grades become lower. The lead numbers and color values, on the other hand, were found to increase as the grades decreased.

The vanillin content of the longest beans was found to be higher than that of the shorter beans. The lead numbers and color values were the same here as with the different grades of beans, being increased as the beans were shorter. These differences, although slight, point to a small degree of inferiority of the shorter beans.[2]

THE SOLVENT ACTION OF VANILLA EXTRACT ON CERTAIN METALS

The solvent action of vanilla extract on metals is of twofold interest to the manufacturer. In the first place the deterioration of the containers used to manufacture and store the extract is of great importance from a straight economical viewpoint and the presence of some metals in the extract is of no lesser importance from the viewpoint of their toxic effects. It is well known that vanilla extract has quite a solvent action on some metals,[3] especially copper, and that the food authorities object seriously to the presence of copper, lead, etc., because of their toxic properties. It was for the purpose of determining the relative solubility of certain metals in vanilla extract that the following experiment was carried out:

Pieces of pure tin, copper, aluminum, zinc, lead, nickel and silver 1 cm. sq. were placed in 25 cc. portions of vanilla extract and allowed to remain at room temperature for two months. At the same time a piece of gold foil and a piece of "Monel metal" wire were treated in the same way. The original weights of all the metals were known. After the two months of exposure to the extract, the metals were again weighed in order to see how much of the metal had gone into solution during that period of time from a surface of 2 sq. cm. in 25 cc. of the extract. This equals a unit square surface to 12.5 units of volume or 4.68 sq. in. to one gal. The results are recorded in Table V.

The object in trying out gold and silver was partly to make the experiment more complete, but chiefly

to see if it would be practical to use either gold- or silver-plated containers in connection with vanilla extract. Either of these two metals would be ideal if the cost of the plating is not prohibitive and if the containers are not subjected to sufficient friction to quickly wear away the rather thin coating of the metal.

The next best metal was found to be tin and is no doubt the most practical of all. Tin dissolves to some extent but, according to our experiments, at a much slower rate than any of the other metals, excepting gold and silver.

TABLE V—MILLIGRAMS OF METAL DISSOLVED BY EXPOSURE OF 2 SQ. CM. OF SURFACE TO 25 CC. OF VANILLA EXTRACT FOR TWO MONTHS

Gold	Silver	Tin	Aluminum	Lead	Monel metal	Nickel	Zinc	Copper
0.0	0.0	1.8	3.2	3.6	4.8	5.8	7.2	8.0

Aluminum has the peculiar property of becoming pitted when it is exposed to vanilla extract. Both of the samples of aluminum which we experimented with were soon eaten through, one on the edge and the other well towards the center of the square. This pitting property of aluminum would make it unfit for use in connection with vanilla extract if the same result is obtained where the surface of the metal is exposed to the extract on only one side, as would be the case in a container.

The color of the samples of extract which contained the pieces of copper were very different from the samples containing the other metals; there was a suggestion of green in the color which made them very unsightly. We accidentally obtained this same thing in a much magnified form in some extracts prepared by suspending the chopped beans in copper wire baskets in the upper portion of the menstruum. At the end of one month these extracts had a greenish caste and were a vivid green at the end of two months. Half of the extracts were prepared from undried beans and half from dried beans; and curiously enough, the dried bean extracts were the first to show the green colors. The rate of solution of copper would make its use undesirable and all copper vessels which are used in connection with vanilla extract should be heavily coated with pure tin and watched closely in order to prevent exposure, due to the wearing away of the tin.

The sample of extract which contained the piece of nickel seemed to be darker than the rest and had a blackish appearance very similar to that produced in vanilla extract by metallic iron.

There was nothing of interest except the solvent action of the extract in the cases of the other metals. It is certain that copper, aluminum, lead and zinc should not be used from their solubility, and copper and nickel are undesirable from the color viewpoint as well as from their solubility. The sample of "Monel metal" did not alter the color of the extract and, in view of the fact that this sample was in the form of a wire which exposed a great amount of its surface, it would seem that this alloy might prove of service. It is noted for its non-rusting ability which would make it very easily kept clean and bright.

[1] Special attention is called to this article on vanilla extract, for it is by far the most complete to be found in the literature on this subject. Especial attention is given by it to the analytical data obtainable from vanilla extracts made in various ways.

[2] U. S. Dept. of Agr., Bur. of Chem., *Bull.* **152**, 156.

[3] 1914 Minutes of the F. E. M. A. Convention, p. 42.

AGING VANILLA EXTRACT

The aging of vanilla extract is a part of the manufacture of the extract that seems to have received all too little attention. Most manufacturers agree that aging is beneficial but there is little agreement as to either the period of aging or the temperature at which it should be done. In fact, the literature contains nothing on this subject beyond the simple fact that the flavor is improved if the extract is aged.

It is well known that whiskey is greatly improved by aging and a long study has been made on this subject. And, from the similarity in the alcoholic content and the fact that both of these substances owe their chief value to their aroma, it seems logical to consider the problems as being closely related, and to apply, as best we can, the facts learned from this investigation on whiskey.[1]

In part, the conclusions of this investigation are as follows:

A chemical change takes place in whiskey when stored in wood, for three or four years in which acids, aldehydes and esters are formed and which reach a state of equilibrium at this age which is maintained thereafter, any improvement of the flavor after this time being largely due to a concentration of the reduction of the volume. This reduction of the volume is also largely due to loss of water and accounts for the increase of the alcoholic content of whiskey after being stored. It is their opinion that the water passes through the wooden barrels, by a process of osmosis, at a much greater rate than does ethyl alcohol, and that the same selective action is shown with the acids, aldehydes, and esters. It is evident that the changes taking place in the spirit is dependent on the storage of the spirit in porous receptacles as none occur when the spirit is placed in glass, tin, or even barrels, the inside of which has been covered with paraffin or glue. It is also evident that the condition of the outside of the barrel, as to moisture, temperature, etc., will have a decided influence on the rate of osmosis.

REDUCTION OF VOLUME

They also found that whiskies which had been aged at low temperatures suffered a comparatively low loss in volume but at the same time they were not matured to anything like the same degree as those which were stored at higher temperatures and which lost considerable of their volume.

REDUCTION IN VOLUME OF WHISKIES DURING STORAGE

VOLUME	BOURBON (a)		RYE (b)	
	Gal.	Loss—Per cent	Gal.	Loss—Per cent
Original............	47.4	....	46.7	....
First year..........	43.9	8	42.8	8.4
Second year........	Not given	....	40.5	14.0
Third year.........	40.5	15	37.7	19.0
Fourth year........	38.2	22	35.2	27.0
Eighth year........	32.5	31.5	24.2	48.0

(a) Stored at 80° F. in summer and 45° in winter.
(b) Stored at 80 to 85° F. during entire year.

It was found that the greater part of the chemical changes had taken place at the end of the first year and, with the exception of only a few cases, the remaining changes were small as compared with that of the first year. The reduction of the "new spirit" taste and the production of color are two items of

great importance in whiskey aging that would not enter into vanilla extract aging at all and, since the larger amount of the chemical changes take place in the first year, it would seem that a one year's aging would be the most profitable. From the above evidence it would also seem that the extract ought to be aged at room temperature and in wooden barrels, that are not painted, in order to permit the process of osmosis.

METHODS OF MANUFACTURING VANILLA EXTRACT

The methods of manufacturing vanilla extract are very numerous and varied in character. They range from plain percolation to hot maceration under the influence of strong agitation and extend over periods of time varying from twenty-four hours to one year. Each different method contains some good and some bad features, producing extracts that vary quite a little in their physical and chemical properties, as well as in their flavoring strengths. There is no one method which all manufacturers agree in calling the best, in fact, favorable comment can be obtained for most any of the methods, some preferring one and some another. Because of this great difference of opinion in regard to this question, we think that it will not be amiss to give a short description of some of the methods that are employed.

MACERATION AND PERCOLATION—This is possibly the oldest and best known of all of the methods used but its value seems to be very questionable. It is the method employed in the U. S. P. and the one commonly used by the smaller producers since it is by far the most convenient.

The method consists of a maceration of the beans in desired menstruum (65 per cent in the 8th U. S. P.) which is followed by percolation of the finished product. The period of maceration varies from twenty-four hours U. S. P., to one or more years, according to the personal opinion of the operator. The method of packing the beans in the percolator also varies very greatly. Some pack the beans alone, some use sugar either with or without a preliminary maceration, and some use various amounts of sand, with or without sugar. The packing of the chopped beans is the most important part of the whole process, for upon it depends whether or not a good degree of extraction will be obtained. This method usually results in a clear and bright extract, but one that, as a rule, does not contain much color. It is also claimed that it is not possible to extract completely the whole of the beans in the percolator due to the rather coarse subdivision of the beans which makes a firm packing impossible, and which tends to allow the menstruum to form canals instead of passing through the whole of the pack. As in all other methods, an increase in temperature seems to increase the color and other extractive matter.

CIRCULATORY DISPLACEMENT—This is a modification of the above method where the chopped beans are held in the upper portion of the menstruum instead of in the lower. The usual method is to place the chopped beans in a perforated basket, suspended from the sides of the containing vessel in such a way

[1] Crampton and Tolman, *J. Am. Chem. Soc.*, **30** (1908), 98.

that the top of the basket is just below the surface of the menstruum, and, it is claimed, that a much better result will be obtained if the basket is removed from the menstruum once or twice during the process and allowed to drain before being replaced.

The principle of this method is the circulatory motion of the menstruum due to the always increasing gravity as the soluble constituents are dissolved. In this way the bean is repeatedly exposed to the menstruum because as the menstruum in contact with the beans becomes charged with soluble matter, its gravity increases and it sinks, forcing fresh menstruum up in its place. This action will continue in this way until a state of equilibrium is reached throughout the entire container when the extract can be drawn off in a perfectly clear and bright condition. This method is slow in action, requiring at least three months, but it is without doubt one of the best known and produces an extract that is very rich in delicate aroma as well as high in physical constants.

The containers should be made of oak and be well painted on the outside to prevent undue evaporation. The basket should be of oak or of heavily tinned metal and the outlet should be about an inch above the bottom of the container to allow the separation of any solid matter that might have passed through the small holes in the basket. By this method the extract is made and partly aged at the same time and need not require any attention from the time it is started until it is finished. The method is usually employed at room temperature but, obviously, higher temperatures could be used by storing the containers in heated rooms.

AGITATION—This method is in reality a maceration process in which the chopped beans and the menstruum are kept in motion by means of a mechanical agitator and, as in all other methods, the time of maceration and the temperature are regulated to suit the various conditions. This method has the advantage of exposing the beans to the solvent action of the menstruum over and over, producing a highly colored product, especially where the menstruum is kept warm. The one big disadvantage of the method is the separation of the finished product from the rather fine pieces of the dregs. This is done either by pressing out the extract or by transferring the whole to a percolator and percolating. The latter, however, is to be preferred in order to reduce the evaporation of the alcohol as far as possible. The containers used are usually of metal and should be kept well covered with tin. Where the extract made in this way is made cold and aged, it is comparable with that made with the circulatory displacement method but it seems positive that an extract made with a hot menstruum is inferior because of the lack of the delicate aroma and excessive amount of inert extractive matter which seems to cover up the delicate flavor so noticeable in the extract prepared by cold methods.

FRACTIONAL SOLUTION—This method differs from those given above only in that the menstruum is added in fractions, one after the other. The chopped beans are agitated or macerated with one-third or one-

fourth of the total amount of the menstruum for the desired length of time and then drawn off as finished extract. The partly extracted beans are then treated again in the same manner until the total amount of extract is obtained from the weight of beans taken. If the different amounts of menstruum are allowed to remain in contact with the beans long enough to become saturated, this method will no doubt produce the greatest extraction possible but only long macerations will suffice, even if more menstruum is to be added, because the extractive matter in vanilla beans is so difficultly soluble, except vanillin, that it requires a rather long time to effect their solution and it is possible that the menstruum would be withdrawn before it has accomplished its purpose.

AGITATION AFTER MACERATION—In this method the chopped beans are allowed to macerate with a portion of the menstruum, say one-half, for the desired period of time and the liquid then drawn off as finished extract. The resulting partly extracted beans are then agitated with the remaining portion of the menstruum heated to about 100° F. until the desired color is obtained and the dregs are exhausted. This liquid is then separated from the dregs and added to the first portion to form the complete extract.

It would seem that there are two objections to this method and two serious ones. In the first place, the maceration is accomplished by allowing the chopped beans to stand in the menstruum without motion and it is certain that the beans settle to the bottom and become surrounded by a layer of saturated menstruum and slow diffusion must be depended upon to bring the unsaturated menstruum in contact with the incompletely extracted beans. And this is so slow that it is doubtful if a very great portion of the menstruum is ever in contact with the beans. The second objection is the hot after-treatment which is believed to be detrimental to a first-class extract of vanilla.

It is unfortunate that we did not have the necessary apparatus to prepare extracts by all of the different methods so that we could report on their relative values from a physical constant standpoint. We did have an opportunity, however, to watch the progress of an experiment made with the circulatory displacement method and analyzed a sample of the extract at the end of three and six months. The results are given in Table I, extracts Nos. 33 and 33A. These results show that the extraction was about complete at the end of the three months but it was found that the extract had a much finer flavor at the end of six months than it did at the end of three months, and will no doubt improve in this respect for sometime. The analytical data obtained from extracts made from these same beans, but with heat and agitation, are not obtainable now, but it is known that they did not exceed those of the circulatory displacement extract to any great extent even in regard to the color.

QUESTIONS AND ANSWERS

In order to get the opinions of different manufacturers on some of the most important questions rela-

tive to vanilla extract, we sent out a list of such questions to twenty members of the Association for their answers. These questions were largely ones that could not be answered by laboratory experiments and which seemed to us as being of interest to the entire membership of the Association. We have received twelve sets of answers and wish to take advantage of this opportunity to thank ·these members for their kind coöperation in the preparation of this report.

A composite of the questions and the number of like answers received for each question is given below. The letters in the parentheses refer to the sections of the different questions; the numbers refer to the number of manufacturers who gave like answers to the same questions.

QUESTION—Do vanilla beans improve with age?
Answers: Yes, 11. No, none. Doubtful, 1.

QUESTION—If so, is the improvement due to (*a*) a change in the beans, or (*b*) is it due to concentration caused by loss of moisture?
Answers: (*a*), 4. (*b*), 4. (*a* and *b*), 2. Doubtful, 1.

QUESTION—What do you consider the best method of grinding vanilla beans?
Answers: Sausage grinder, 1. Chopping machine, 9. Both, 1. Doubtful, 1.

QUESTION—Do you ever dry your bean before extracting it, and if so, to what extent?
Answers: Yes, 3. No, 9. Two or three remove only the excess moisture; the other dries to constant weight at 70° C.

QUESTION—In your estimation should vanilla extract be made by (*a*) maceration, (*b*) percolation, (*c*) agitation, or by some combination of two or more of these?
Answers: (*a*), 1. (*b*), 1. (*a* and *b*), 6. (*a*, *b* and *c*), 1. (*c*), 1.

QUESTION—In your opinion, what is the best menstruum for the complete extraction of vanilla?
Answers: 40 per cent, 1. 50 per cent, 5. 52.5 per cent, 1. 57 per cent, 1. 60 per cent, 3.

QUESTION—Do you prefer glycerin to sugar in vanilla extract?
Answers: Yes, none. No, 9. Use both, 3.

QUESTION—Does sugar interfere in the percolation of the extract?
Answers: Yes, 5. No, 3. Doubtful, 3.

QUESTION—Will either one increase the color?
Answers: Yes, 3. No, 8. Doubtful, 1.

QUESTION—Will either one prevent baking-out or freezing-out of the flavor?
Answers: Yes, 3. No, 4. Doubtful, 4.

QUESTION—When sugar is used do you add it before or after percolation?
Answers: Before, 3. After, 7.

QUESTION—Does aging vanilla extract improve its flavor?
Answers: Yes, 12. No, none.

QUESTION—If so, will it improve in glass as well as in wood?
Answers: Yes, 2. No, 8. Doubtful, 2.

QUESTION—Should the extract be aged at, (*a*) room temperature, or (*b*) at reduced temperature?
Answers: (*a*), 7. (*b*), 2.

QUESTION—How long do you think that the extract should be aged?
Answers: Six months, 4. One year, 4. Longer the better, 3.

QUESTION—In, your experience what is the average loss due to evaporation when wooden containers are used?
Answers: 1 to 3 per cent, 2. 3 to 6 per cent, 1. 11 per cent, 1. Doubtful, 5.

QUESTION—Does the hot process impair the flavor of the finished extract?
Answers: Yes, 6. No, 1. Yes, if over 100° F., 1. Yes, if over 110° F., 1. Doubtful, 2.

QUESTION—Does the hot process yield a stronger extract than the cold process?
Answers: Yes, 5. No, 5.

This leaves but little doubt in regard to questions Nos. 1, 3, 4, 7, ·9, 12 and 14, and shows an almost equal division of opinion in regard to the other questions. It is regretted that more data are not available in regard to the loss in volume of the extract on aging. We are, however, of the opinion that about the same loss will result here that has been found in the case of whiskey where the extract is stored under like conditions, namely, about 8 per cent in the course of a year. This question seems to have received all too little attention by manufacturers and it would seem to be a question that is worthy of great consideration.

CONCLUSIONS

1—Vanilla beans are improved on aging where the aging is conducted under proper conditions.

2—Vanilla beans should be chopped and not ground.

3—Vanilla beans can be dried without material loss of flavor if the drying is carefully carried out at room temperature (60 to 70° F.).

4—Extracts made from dried beans have higher color and lead number values than those made from the same but undried beans.

5—Where the beans are not dried the menstruum should be regulated to suit the moisture present in the beans.

6—The physical constants are not greatly affected by the various amounts of alcohol in the menstruum as long as the amount of alcohol is sufficient to produce a clear extract. The color values are slightly increased and the lead number values are slightly decreased by an increase of alcohol up to a maximum of about 65 per cent.

7—The flavor of an extract prepared with 60 per cent alcohol is superior to that made with 50 per cent alcohol.

8—The resins of vanilla beans are of no value as flavoring agents but are of value as fixatives for the flavoring compounds present and as coloring matter for the extract.

9—Alkaline menstrua produce extracts that are higher in color and lead number values but the alkali impairs the natural flavor of the beans.

10—Short maceration and percolation will not produce the maximum extract. The hot process produces an inferior extract, one that is lacking in delicate aroma. The most desirable extract is produced only by long maceration at room temperature as in the circulatory displacement method.

11—Sugar does not increase the color of the extract and should be added to the percolate. Glycerin tends to increase the color and should be added to the menstruum before percolation or maceration.

12—Vanilla extract should not be left in contact with any metal that is not completely covered with tin.

13—Vanilla extract is decidedly improved by aging. The period of the aging should be at least one year and should be carried out at room temperature (60 to 70° F.) and in unpainted, porous, wooden barrels.

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RELATIVE OIL YIELD OF FLORIDA ORANGES

By S. C. HOOD
Received April 22, 1916

In connection with experiments carried on to determine the commercial possibilities in the manufacture of orange oil from Florida cull oranges, it seemed advisable to determine how early in the shipping season the oil was present in commercial quantities, and also to secure some data on the relative oil yield of different varieties and from various localities. In the fall of 1915 a series of tests was made beginning at the opening of the shipping season of the early varieties and extending until well into the season for all except the very late varieties. The writer is indebted to Mr. M. G. Mastin of the Bureau of Chemistry for the material used in this study.

VARIETIES SELECTED

The Parson Brown was selected as being the principal commercial early variety, and although mature enough to ship at the time the experiments were begun, yet the color was bright green. At the close of the work this variety was well colored. Another variety of very early maturity is the Ilsworth Early; and on November 12th it was fully mature and beginning to show a slight yellow color. The Homasassa was taken as the type of the early mid-season orange, and on the date of the first tests was still green in color, although fairly well matured. The Pineapple being one of the leading mid-season varieties, was taken as the type of this group, and samples from three widely separate localities were secured. These were still immature and green in color when the experiment began. Since the common seedlings are still extensively grown in Florida, and there are wide differences in the fruit, samples were taken from eight

TABLE I—Percentages of Oil in Florida Oranges

		Percentages Calculated on Weight of Fruit							Percentages Calculated on Weight of Peel						
Variety	Locality	Nov. 1–6	Nov. 7–13	Nov. 14–20	Nov. 21–27	Nov. 28 Dec. 4	Dec. 5–11	Dec. 12–18	Nov. 1–6	Nov. 7–13	Nov. 14–20	Nov. 21–27	Nov. 28 Dec. 4	Dec. 5–11	Dec. 12–18
Parson Brown	Lake Weir	0.41	....	0.35	....	0.50	....	....	2.74	....	2.05	....	3.18	....	....
Ilsworth Early	Windermere	....	0.43	....	0.24	....	0.40	0.36	....	2.51	....	1.52	....	2.35	1.91
Homasassa	Florence Villa	....	0.51	0.49	0.35	0.58	0.51	0.50	....	2.63	2.56	2.00	3.26	2.94	2.67
	Palatka	....	0.40	....	....	0.25	....	....	....	2.44	....	....	1.62	....	....
Jaffa	Palatka	....	....	....	....	....	....	....	....	2.13	....	....	1.74	....	....
Carlton Seedless	Windermere	....	0.42	....	0.30	....	0.41	0.41	....	2.47	....	1.78	....	2.42	2.24
Pineapple	New Smyrna	0.13	....	0.14	....	0.14	....	0.15	1.00	....	1.14	....	1.20	....	0.99
	Palatka	....	0.17	....	....	0.30	....	....	....	1.42	....	....	1.83	....	....
	Florence Villa	....	0.30	0.32	0.35	0.32	0.35	0.41	....	2.00	1.88	2.30	1.95	2.21	1.84
Seedlings	Plant City	0.39	....	....	....	....	0.37	....	2.02	....	....	....	....	2.27	....
	New Smyrna	0.25	....	0.28	....	0.27	....	0.36	1.70	....	2.17	....	1.95	....	2.18
	Lakeland	0.21	....	0.17	....	0.14	....	....	....	1.55	....	1.34	....	0.83	....
	Tampa	....	0.16	....	0.14	....	....	0.27	....	1.00	....	0.88	....	....	1.43
	Ozona	....	0.11	....	0.25	....	....	0.34	....	0.92	....	1.45	....	....	1.91
	Orlando	....	0.32	....	0.25	....	0.41	....	....	1.91	....	1.52	....	2.51	....
	Wauchula	....	0.40	....	0.35	....	....	0.36	....	2.53	....	2.74	....	....	2.15
	Geneva	....	0.44	....	....	0.41	....	0.47	....	....	2.40	....	2.50	....	2.68
Valencia	Florence Villa	0.38	0.44	0.50	0.31	0.46	0.35	0.53	2.46	2.58	2.50	1.95	2.60	2.00	3.04
	Oakland	0.32	0.36	....	....	0.43	....	....	....	2.20	....	....	2.76	....	....
	Windermere	....	0.46	....	0.35	....	....	0.24	....	2.47	....	2.00	....	....	1.41
	Manatee	....	....	0.37	....	0.38	....	0.41	....	....	2.10	....	2.38	....	2.27

localities. On November 1st, all of these were livid green in color, but at the close of the tests they were mature and well colored. Since the Valencias do not mature until the latter part of the winter, the tests were not continued on this variety to maturity. Most of the samples of this variety were only partially grown at the beginning of the tests, and at the close were of full size, but still very immature and green in color. Each series of samples was gathered from the same tree at intervals of one to two weeks from November 1st to December 18th.

METHODS

Each sample consisted of 12 oranges picked from the tree set apart for these tests. Usually 1 to 3 days elapsed between the date of picking and the date of the tests. The oranges were peeled with a knife and care was taken to remove all of the peel without cutting the pulp cells. Care was also taken to prevent breaking of the strip of peel and consequent loss of oil. The fresh peel was then finely ground in a food chopper and the ground material distilled with water in a Remington still. The colorless oil secured was taken as the total oil content. Since this oil consisted almost entirely of limonene, it does not represent the actual oil content of the peel, but the loss of the oxygenated constituents would be fairly constant in each sample, and this method may be used as a relative means of the total oil determination. The percentages, Table I, were calculated on both the weight of the peel and the weight of the fresh fruit.

It will be noted from Table I that in the case of most samples there was a considerable increase in oil content as maturity advanced, but that there is a high percentage of oil in the peel of the fruit sometime prior to the change in color. This is especially marked in the case of the Valencias. It will also be noted that there is a very wide difference in the oil content of the same variety from different localities. From this preliminary investigation it is not possible to determine whether this variation is due to soil and climatic conditions, or to the degree of care which the trees have received. From the appearance of the fruit as received, however, it is believed the latter is the case. Especially in the case of seedlings this difference was marked, and fruit showing by its form and texture that the trees were highly fertilized and

well cared for, showed a higher oil content. There was a marked drop in oil content in most samples during the two weeks from November 14th to 27th. During this period there was an excessive rainfall throughout the entire state. Table II gives the rainfall records of the Weather Bureau in the localities where samples were secured during the period of these experiments.

TABLE II—Rainfall in Inches in Localities where Orange Samples were Gathered

Names in Parentheses are Weather Bureau Stations Nearest to Place where Orange Samples were Taken

	Nov. 7–13	Nov. 14–20	Nov. 21–27 Dec. 4	Dec. 5–11	Dec. 12–18
Lake Weir (Lynn)	....	1.93	0.26	0.16	....
Windermere (Clermont)	0.03	3.46	0.11	0.10	0.26
Florence Villa (Lucerne Park)	....	1.82	....	0.58	....
Palatka (Satsuma Heights)	0.04	0.67	0.10	0.05	....
New Smyrna	0.10	2.34	....	0.01	0.09
Plant City	....	3.15	0.20	0.30	....
Lakeland	....	2.18	....	0.12	....
Tampa	....	2.89	0.09	0.30	....
Ozona (Tarpon Springs)	....	3.77	0.63	0.65	....
Orlando	....	2.22	0.02	0.05	0.20
Wauchula (Fort Meade)	....	3.59	....	0.15	....
Geneva (Sanford)	0.08	1.85	....	0.14	....
Oakland (Clermont)	0.03	3.46	0.11	1.10	0.26
Manatee	....	2.62	0.10 0.26	0.66	....

In some cases no observations are available from the places where the samples were taken, and the records of the nearest observer are given. It will be noted that this decrease in oil content immediately follows the period of rainfall, and that where the rains extended over a longer period, the oil content was retarded in its increase.

EFFECT OF RUST MITE

It has long been believed that the typical brown coloring of the orange skin known as "russet" following the presence of large numbers of the rust mite, *Eriophyes oleivorous* Ashm., is due to the puncturing of the oil cells. In order to determine if this insect injury decreased the oil yield of the fruit, tests were made on the fruit of the same tree at two stages of maturity. The first test was made on November 26th while the fruit was immature and the second on January 25th after the oranges had reached full maturity. Each sample consisted of 12 bright and 12 russet fruit. The results are given in Table III.

TABLE III—Oil Yield of Russet and Bright Fruit of Same Tree

PER CENT OIL IN:	NOVEMBER 26 Fruit	Peel	JANUARY 25 Fruit	Peel
Bright Fruit	0.20	1.25	0.40	2.26
Russet Fruit	0.10	0.60	0.40	2.68

These results indicate that early in the season there is a lower oil content on the fruit infected by rust mite,

but as the orange matures a normal amount of oil is formed. The results secured in the commercial tests on large lots of bright and russet fruit also indicate that an equal amount of oil may be secured from each kind of fruit.

CONCLUSIONS

These preliminary results show that there is a wide variation in the oil yield of Florida oranges under different climatic and cultural conditions, and that the question of variety is likely to have some bearing on the commercial production of orange oil. The oil content has not reached its maximum until the oranges are fully mature, but the oil is present in commercial quantities before the fruit are ready for harvest.

The occurrence of heavy rainfall during the season of harvest will cause a considerable decrease in the oil content. The presence of rust mite does not decrease the percentage yield of oil of the mature fruit, but may show some effect early in the season.

BUREAU OF PLANT INDUSTRY, WASHINGTON

A NUMERICAL EXPRESSION FOR COLOR AS GIVEN BY THE IVES TINT PHOTOMETER

By OTTO KRESS[1] AND G. C. McNAUGHTON[2]
Received May 27, 1916

The matter of a numerical expression of a definite shade of color is one on which various investigators have expended considerable energy with varying degrees of success. In almost countless phases of commercial and experimental work it is extremely desirable that there be a means by which an exact shade of color may be communicated to others or so recorded that this shade may be matched or referred to when the original is not available. One readily comprehends the importance and practicability of an instrument which would give an accurate reading of color.

So far as the authors know, there are but two instruments on the market, which would appear to be practicable from a scientific and a possible mill standpoint, for measuring the depth of color of either liquids or solids. These instruments are known as the Ives Tint Photometer and Colorimeter. The tint photometer, being the more simple instrument, was used in these experiments.

The original purpose of the experiments following was, (1) to determine if color indications as shown by the Ives tint photometer could be employed as a means of measuring the progress of beating, and (2) to observe the relation between the numerical expressions for depth of color as read from the instrument and the relation of the shades as noted by the eye.

The tint photometer (Fig. 1) consists essentially of a form of telescope into which light from two sources is reflected, by means of a mirror (A), into two apertures, and focused by a special lens into two semicircular fields. Each semicircle is uniformly illuminated by passing the light from the apertures, or slits, through a rotating wheel (B) mounted with lens,

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the object of which is to blend the light. The amount of light reflected through the left-hand aperture is controlled by a shutter actuated by a long lever (C) that moves over a scale (D) so divided into 100 divisions or parts that at zero reading the sliding shutter is entirely closed and at 100 divisions is wide open.

By means of a thumb screw (E) the second aperture may be adjusted but through much narrower limits. The base of the eye-piece tube is equipped with a sliding carrier (F) in which are mounted red, green, and blue color screens, as well as one of colorless glass. The light under which the tests are made is received through a south window covered with tracing cloth.

In using the instrument two magnesia blocks are first placed directly beneath the reflecting mirror and in front of a vertical mirror (G). The lever operating the shutter is set at 100, to give full opening of the left aperture, and the right-hand aperture adjusted by the thumb screw until the two halves of the field are of uniform intensity. This adjustment made, the mag-
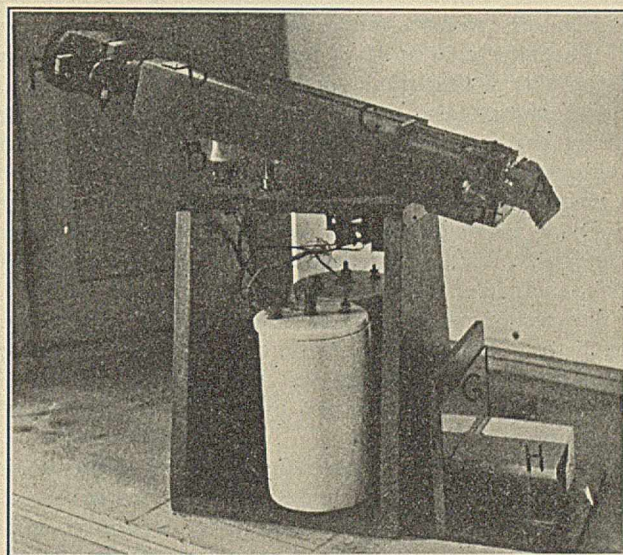
FIG. 1

nesia block on the right is removed and the paper (H) to be tested is substituted. Using each of the color screens successively (i. e., red, green, and blue) the amount of light entering the left aperture is decreased until it is of the same intensity as that entering the right aperture from the paper sample, or until the two halves of the field appear of one luminosity. The position of the lever when this matched condition of fields is obtained with each screen indicates numerically the proportion in which red, green, and blue light are reflected by the paper under test.

For example, the white standards show 100 parts each of red, green, and blue, while a certain sample of an undyed sulfite paper gives readings of 77 parts red, 71 parts green, and 67 parts blue. The sum of the three colors in the case of the white standard is 300, while in the case of the paper it is but 215. For purposes of convenience it has been assumed that in the latter case there exists a darkening effect of 85 parts of black (in 300). This factor of "parts black" which
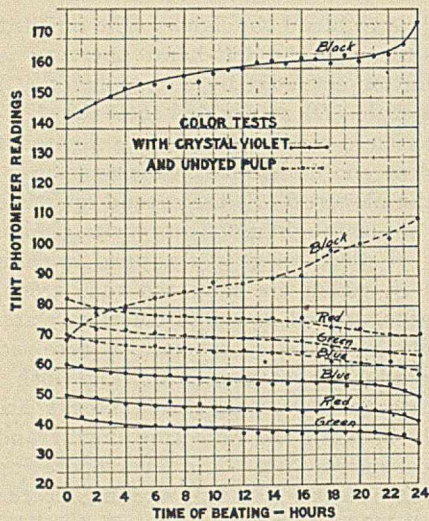
FIG. 2
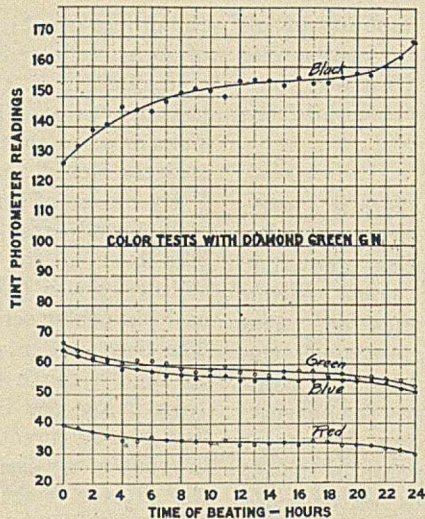
FIG. 3
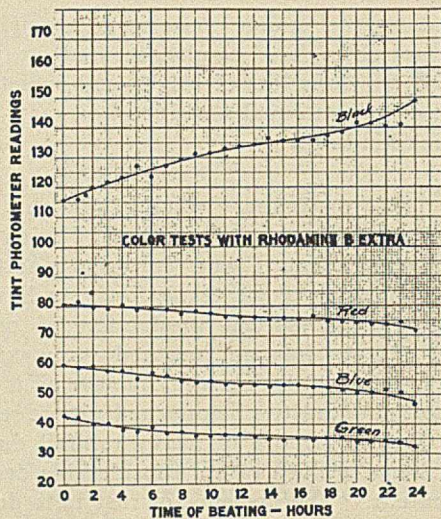
FIG. 4

is secured, not by direct reading of the instrument, but by the subtraction of the sum of the three colors from 300, immediately expresses the relative brilliancy of the color examined although it conveys no idea of the tint if the other three readings are not given. The authors have found no difficulty in checking within one division (or part) the values secured in previous tests. The inventor of the tint photometer claims that the personal equation becomes practically negligible in operating this instrument, and that even a marked color-blindness does not affect the readings of the instrument.

The tests were divided into two groups, (1) where depth of color was secured by prolonged beating effect, using the same amount of dye, and (2) where depth of color was obtained by different amounts of dye employed.

In the first group unbleached spruce sulfite pulp was beaten in an experimental Marx beater of 50 lbs. capacity. The beater tub is of concrete construction, with a specially formed trough for rapid circulation. The roll is 24 in. in diameter by 18 in. face, with a cluster filling of iron bars, and was operated throughout the experiments at a peripheral speed of 2,000 ft. per min. A very light treatment was given in order that the comparatively short stock used would not be badly cut before the end of the operation. The treatment extended over a period of 24 hrs. Representing by zero the position of the roll above the bed plate when a very faint hum could be heard as the roll turned in the empty beater, the following table shows the manner in which the roll was gradually lowered. Even at the end of 24 hours the stock was

Time at which roll was lowered	Position of beater roll
At start.	+0.10″
At end of 2 hrs.	+0.05″
3	+0.01″
17	+0.002″
20	+0.000″
21	—0.002″
22	—0.004″
23	—0.006″
24	roll raised

but slightly shortened and as it still was rather far from pergamyn stock it is probable that greater differences in color tests would have been secured had

the stock been submitted to a more severe brush after the first few hours of beating.

In this group of tests a sample of the stock in the beater was removed every hour. For the various dyeings, pulp samples of 3 g. (dry) weight were employed which were secured as follows: A large sample of about 3.3 per cent density was pressed by the palm of the hand on a 70-mesh screen until no water dripped from it (about 12 per cent dry) and then thoroughly mixed. After determinations of moisture content of the pressed stocks were made, amounts equivalent to 3 g. (dry) were weighed out and bottled with 100 cc. distilled water. This method presents the difficulty of obtaining a homogeneous mixture of the pressed stock, but was by far the best method available.

When ready for the dyeing, the pulps were washed out of the bottles with 250 cc. of water into enamel-ware cups $3^{1}/_{2}$ in. in diameter and $3^{5}/_{8}$ in. deep. In these the stock was agitated by a mechanical stirrer for a total of 20 min.—color, sizing and alum being added in the order named.

In the cases of the red and green dyes, respectively, 0.5 g. of Rhodamine B Extra and 0.5 g. of Diamond Green G. N. were dissolved with the addition of a little acetic acid in a liter of water and a volume equivalent to 0.1 per cent of the dry weight of the stock used for each sample. This gave dyeings of 1 lb. per 1000 lbs. of stock. The strength of the Crystal Violet was but half of the others. Dilute solutions of size and alum were standardized and volumes equivalent to 1 per cent of size (calculated as rosin) and $1^{1}/_{2}$ per cent of alum employed.

The finished stocks were made into hand sheets on a special circular mold $6^{3}/_{4}$ in. in diameter, pressed between the press rolls of an experimental paper machine and dried between sheets of blotting paper on one of the driers. During the drying the hand sheets were turned frequently to avoid the burning of the color to the surface, and so cause an unevenness in depth of color on the two sides of the sheet.

The wire side of the sheet was in every case used for the color tests with the tint photometer. In making these tests all sheets of one color were ob-
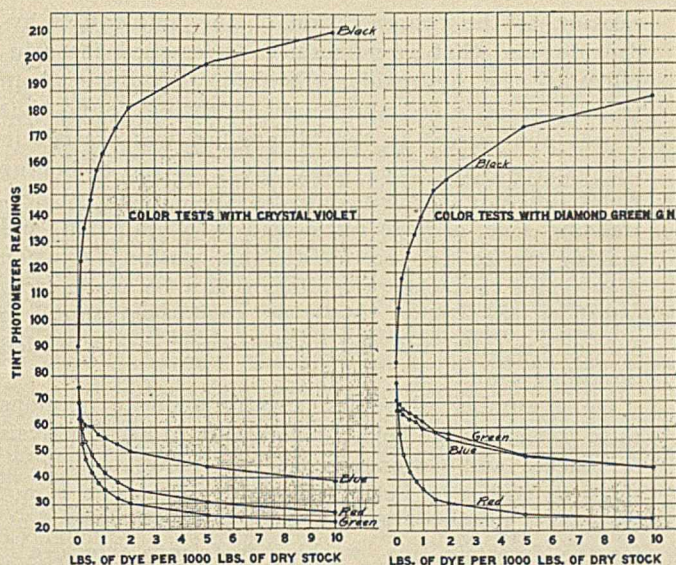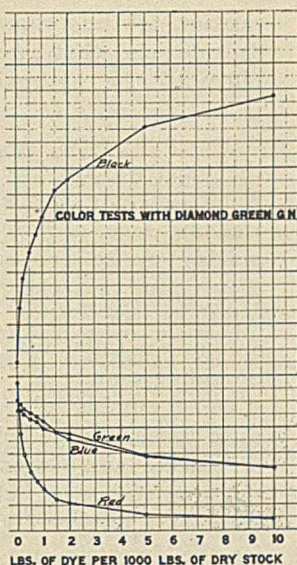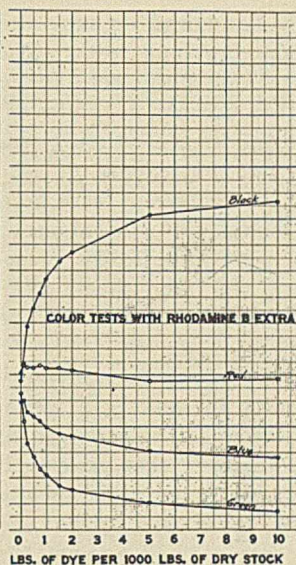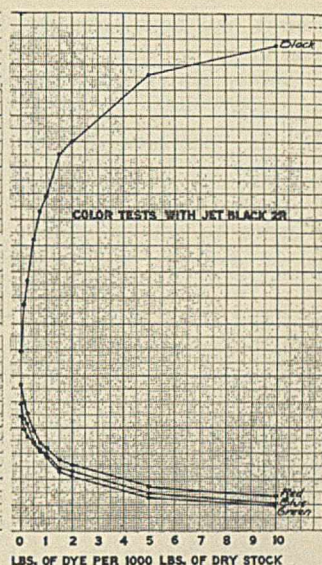
FIG. 5   FIG. 6   FIG. 7   FIG. 8

served in consecutive order and continuously in order to avoid any great variation in lighting conditions.

The results of the tint photometer observations are shown graphically in Figs. 2, 3, and 4, in which the readings of parts red, green, and blue, as given by the instrument, are plotted against the duration of the beating treatment. The calculated "parts black" also have been plotted in these figures. When comparing the depth of tint as determined by the tint photometer with the ratings exhibited to the eye, it was noted that an almost identical arrangement could be made. However, as the instrument does not distinguish as readily as does the eye between very slight differences of shade, it is necessary to be very exact in the adjustments. Reference to the curves in Figs. 2, 3, and 4 will give the reader an idea of the rather small differences in color between consecutive papers. While it will be noted that many of the values obtained do not fall upon the smooth curve, this fact is not exceedingly serious, since the paper samples when viewed by the eye do not show an absolutely regular progressive darkening that should be expected. Such irregularities would occur if error had been made in the rather involved method of sampling the stock, and where such a number of samples were taken it is not improbable that errors were occasionally introduced. Slight variations from 3 g. (dry) weight would cause the stock to assume a deeper or lighter tint, depending on whether the samples were too light or too heavy. Unfortunately, in trimming the colored samples to fit the special paper holder that comes with the instrument, the clippings were discarded—consequently the authors had no means of establishing the experimental error.

The accelerated hydration and the corresponding darkening of the stock, when the brushing became more severe near the end of the beating treatment, are clearly shown in all of the figures. Fig. 2 is especially interesting in that curves for the undyed beaten stock have been plotted on the same scale as that used for the blue stock.

As is well known, the longer the stock is beaten, causing increased hydration, the less dyestuff will be required to produce the same depth of color. This is well shown in all of the curves, as the same amount of dyestuff per 1000 lbs. of stock was used, and the increased depth of color is due solely to hydration.

That a color may be expressed in numbers—even though the numbers allow a slight variation in shade—is far more satisfactory than to attempt a description of the color. For example, a red may be on the yellowish cast tending to approach the orange, or may be on the bluish cast tending towards the violet. The addition of any other primary color such as green would, in the case of red, not form a greenish red but would be a step towards darkness. It is absolutely impossible to record scientifically either a shade of red or the intensity of a red shade, by merely describing it as either a *light or dark bluish red* or a *light or dark orange* shade. The addition of color in all practical dye work must be considered as a step towards darkness, and it is common practice in the mill to avoid mixtures of colors if brilliant shades are desired, especially in view of the fact that the practical dyes used are never of a pure shade.

A second series of color tests was made in order to observe the behavior of the Ives tint photometer with various colors and intensities of colors. It will be remembered that in the first series difficulty was met with, due to an almost exact similarity of many of the test papers. In this second group of experiments all of the papers were made from one mass of pulp that was removed from the beater after a light brush and tinted with various amounts of dye. The colors employed were "Diamond Green G. N.," "Rhodamine B Extra," "Crystal Violet," and "Jet Black 2 R," which were made up to strengths of 0.5 g. dye in 1 liter of water, sufficient acetic acid being used to prevent hydrolysis, and volumes of each used equivalent to $1/8$, $1/4$, $1/2$, $3/4$, 1, $1^1/2$, 2, 5, and 10 lbs. of dye per 1000 lbs. of dry stock. As before, the 3 g. (dry) samples of the stock were secured from the wet stock,

after it had been squeezed by hand and its moisture determined as described in the previous tests. The manner in which the color, size and alum were added and mixed was similar to that formerly employed, with the exception that the total time of stirring was increased to 30 min., and that an excess of size and alum (2 and $2^1/_2$ per cent, respectively) were used in order to set the heavy shades. Up to about 2 lbs. of dye the back-waters were practically colorless. The method of preparing the hand sheets from the dyed stock was identical with that used in the previous series.

The gradations in shade of these papers were very distinct, and offered no difficulty in making a visual selection of the sheets in the order of the strength of dye employed. The tint photometer also gave uniform readings of color which are shown graphically in Figs. 5, 6, 7, and 8 where "parts red, green and blue" and also "parts black" are plotted against the strength of dye used.

While we are not prepared to say just how the readings of the three colors should be interpreted or how matching a shade could be facilitated from them, it is very evident to us that with this instrument a shade or tint may be given a definite numerical value.

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NOTES ON THE DETERMINATION OF ALUMINUM

By C. F. SIDENER AND EARL PETTIJOHN
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The determination of aluminum in the form of aluminum oxide, after precipitation with ammonium hydroxide, is frequently made. As ordinarily carried out, there are several points in the determination that might give rise to variations in results. The purpose of this piece of work is to determine the best procedure with regard to some of these points.

In looking up the literature on the subject of this precipitation, considerable difference of opinion was found. The ordinary directions are to boil until the liquid just smells of ammonia, though it was very early discovered[1] that at this point ammonium chloride may have hydrolyzed to such an extent with volatilization of ammonia that the solution is actually acid. In this case, of course, aluminum hydroxide would be redissolved and quantitative results could not be obtained.

L. Blum[2] worked on this precipitation and found that ammonium chloride was decomposed in solution at 100° C. with loss of ammonia; he advises filtration when the liquid is still quite strongly ammoniacal. He also states, and many others verify his statement, that ammonium hydroxide dissolves a certain amount of freshly precipitated aluminum hydroxide, which amount is considerably lessened by the presence of ammonium salts. C. F. Cross,[3] using ammonium hydroxide solutions of varying strength, found that aluminum hydroxide was dissolved at the moment of precipitation, that the amount dissolved bore no

relation to the concentration of the ammonium hydroxide, and that ammonium salts lessened the solubility of the hydroxide. Penfield and Harper,[1] in testing for dissolved aluminum hydroxide, found that it was not present in the filtrate but was present in the wash water. They tried using, instead of boiling water, an ammonium nitrate solution, made by neutralizing 2 cc. of pure nitric acid with ammonia, and making up to 100 cc. With this washing solution they claim that the precipitation can be made from solutions containing larger or smaller amounts of ammonium salts, and that no very great care is needed in adding ammonia.

On a number of points, exactly opposite opinions as to procedure are held. Hillebrand, in his "Analysis of Silicate and Carbonate Rocks," states that, contrary to the usual belief, aluminum hydroxide is not volatile in presence of ammonium chloride, and that, for the determination, washing free from chlorides is unnecessary. Washington, in his "Rock Analysis," and Mahin, "Quantitative Analysis," take the opposite view, and say that aluminum hydroxide must be washed free from every trace of chloride or low results will be obtained. With regard to boiling the solution for precipitation, the same difference of opinion exists. Washington advocates boiling (for not more than 1 min.) with a slight excess of ammonia, while Handy[2] boils for 20 min. under the same circumstances. For washing the precipitate, ammonium nitrate is frequently recommended, Treadwell, Penfield and Harper, Washington and others making use of it. Renz,[3] however, found that when aluminum hydroxide was precipitated with ammonium hydroxide in presence of ammonium nitrate, a small quantity remained in solution. Fresenius, Handy and several others advise the use of hot water.

There is a similar difference of opinion as to the ignition of the precipitate. Allen and Gottschalk[4] found that the highest heat of the blast lamp was required to effect complete dehydration: precipitates heated to 1100° C. and cooled for half an hour were still hygroscopic. Handy[5] also considers the oxide very hygroscopic and advises intense ignition. Carnelley and Walker,[6] on the other hand, obtained complete dehydration by gradual heating to 850° C.

The points which this paper covers are as follows:

1—To determine whether the precipitate need be washed free from chlorides before ignition.

2—To determine whether an excess of ammonia has any solvent effect on the freshly precipitated hydroxide.

3—To determine whether water or ammonium nitrate solution is more suitable for washing the precipitate free from chlorides.

4—To determine a satisfactory time for ignition to constant weight.

An aluminum chloride solution was made up from

[1] Z. anal. Chem., 2, 394; 1, 69.
[2] Ibid., 27, 19.
[3] Chem. News, 39, 161.

[1] Am. J. Sci., 20, 9, 181.
[2] J. Am. Chem. Soc., 18, 766–82.
[3] J. Chem. Soc. (London), 2 (1903), 729.
[4] Am. Chem. Jour., 24 (1900), 292–304.
[5] J. Am. Chem. Soc., 18, 766–82.
[6] J. Chem. Soc. (London), Trans., 53, 87.

sheet aluminum and hydrochloric acid. The ammonium chloride used volatilized without residue, as did the ammonium hydroxide. The ammonium hydroxide solution was filtered into a ceresin bottle: the specific gravity of the solution was 0.955.

In all of the precipitations made with these solutions, the following general conditions were maintained: The volume of the solution from which the precipitation was made was 250 cc. and in every case 25 cc. of the total volume was a 10 per cent solution of ammonium chloride. All precipitations were made in porcelain casseroles, the solution being boiled for 1 min. and filtered hot. The precipitates were ignited moist, in platinum crucibles: the ignited precipitates, after cooling somewhat in the air, were left in the desiccator for 20 min. before being weighed: the crucibles were covered while in the desiccator and on the balance. Twenty-five cc. of aluminum chloride solution were used in every case. The filtrate and wash water from each determination were evaporated separately and tested for the presence of aluminum.

STRENGTH OF SOLUTION

The strength of the aluminum chloride solution was first determined in terms of the oxide. Two methods were used: (1) direct evaporation of the solution and ignition of the residue; and (2) evaporation and ignition after adding enough ammonium hydroxide to precipitate the aluminum hydroxide.

TABLE I—STRENGTH OF ALUMINUM CHLORIDE SOLUTION.

(1) Direct Evaporation of the Chloride		(2) Evaporation after Addition of Ammonium Hydroxide		
0.2303	0.2303	0.2300	0.2302	0.2303

The results in Table I tend to show that the same value is obtained if the chloride solution made ammoniacal is evaporated as if the pure chloride is evaporated. This result would hardly be expected if aluminum chloride is volatile under the given condition, since the amount of aluminum present as chloride is much less in the first case than in the second. A slight loss might, however, occur in both cases so that a second series of test determinations was made. The amount of aluminum in terms of aluminum oxide was determined in a sample of Kahlbaum's crystallized aluminum sulfate. A second series was run parallel to this one, using some of the

TABLE II

	PER CENT Al$_2$O$_3$ IN Al$_2$(SO$_4$)$_3$		EFFECT OF NH$_4$Cl ON Al$_2$(OH)$_6$			
No.	Sample Grams	Per cent Al$_2$O$_3$ Found	Sample Grams	NH$_4$Cl Gram	NH$_4$OH Cc.	Per cent Al$_2$O$_3$ Found
1	0.5841	17.24	0.5336	0.3	5	17.17
2	0.6435	17.17	0.7723	0.3	5	17.17
3	0.7018	17.17	0.9590	0.3	5	17.18
4	0.9150	17.18	1.1417	0.3	5	17.18
5	0.9301	17.22	0.9561	0.5	5	17.24
6	1.0571	17.17	1.2990	0.5	5	17.21
7	1.2964	17.24	1.6290	0.5	5	17.20

same sample, but, after solution in a little water, adding some ammonium chloride and enough ammonium hydroxide to precipitate the aluminum as hydroxide.

The results, in Table II, show that there is no loss by volatilization of aluminum chloride when the precipitated hydroxide is ignited in presence of ammonium chloride.

EFFECT OF VARYING AMMONIUM HYDROXIDE

The next point taken up was the effect on the precipitation of varying the amount of ammonium hydroxide present. Water was used to transfer the precipitate from the casserole to the filter paper, as small an amount as possible being used. In no case did the wash water show a test for aluminum, while the filtrate from those determinations giving low results did give a test for aluminum. The results appear in Table III.

TABLE III—EFFECT OF EXCESS OF AMMONIUM HYDROXIDE IN PRECIPITATION OF ALUMINUM HYDROXIDE.

Very Slight Excess	10 cc. Excess	15 cc. Excess	20 cc. Excess
0.2303	0.2281	0.2294	0.2288
0.2302	0.2271	0.2289	0.2305
0.2298	0.2282	0.2294	0.2302
0.2297	0.2304	0.2297	0.2287
0.2299	0.2304	0.2274	0.2291
0.2305	0.2283	0.2282	0.2265

The uniformity of results in the first column is very evident, as compared with that of the other three, although the uniformity there is not as great as desired. An excess of ammonia has a decided solvent effect, not proportional to the excess of ammonia added. The variation in results obtained in each of the other three columns must be due to very slight variations in procedure, such as time of filtering. The results in Column 1 check those obtained in evaporating the solution in a platinum crucible, as was to be expected.

EFFECT OF WASHING PRECIPITATE FREE OF CHLORIDES

On the effect of washing the precipitate free from chlorides, the results in Table IV were obtained.

TABLE IV—EFFECT OF WASHING ALUMINUM HYDROXIDE FREE FROM CHLORIDES BY VARIOUS WASHES

Hot Water	1 Per cent NH$_4$NO$_3$ Solution	2 Per cent NH$_4$NO$_3$ Solution	3 Per cent NH$_4$NO$_3$ Solution
0.2285	0.2277	0.2262	0.2263
0.2281	0.2286	0.2263	0.2276
0.2280	0.2286	0.2262	0.2282

For the precipitations, 5 cc. of ammonium hydroxide were added, about 2 cc. of this being in excess. In no case was aluminum hydroxide found in the filtrate. In the first series, in all three cases, the wash water gave a test for aluminum hydroxide. In the second experiment the precipitate obtained was filtered off, ignited, and found to weigh 0.0022 g., which brings the total weight of the precipitate up to the value 0.2303 g. formerly obtained.

In every experiment the wash water was tested for aluminum and it was found to be present, so that the final conclusion was that ammonium nitrate solution, as used, was unsatisfactory for washing purposes.

EFFECT OF BLASTING IN VARIOUS WAYS

For heating the precipitates to constant weight an ordinary blast lamp was used. The amount of air used and type of lamp might change the results obtained. The first precipitates were blasted for 10-min. periods until constant weight was obtained.

Typical results appear in Table V. The weights given indicate the weight in grams of crucible and precipitate.

It is seen that three 10-min. periods or two 20-min.

periods served to effect a constant weight. Slight differences are undoubtedly produced by the size, weight and thickness of the crucible.

Table V—Time Required in Heating Alumina to Constant Weight

Time Min.	No. 1	No. 2	No. 3	No. 4	Heated in 20-Min. Periods 20 min.	40 min.	60 min.
10	36.9620	29.6670	21.0587	29.6647	29.6538	29.6530	29.6527
20	36.9615	29.6653	21.0570	29.6638	33.7635	33.7627	33.7625
30	36.9605	29.6643	21.0569	29.6638	33.7760	33.7760	......
40	36.9605	29.6643	.....	.....	36.9621	36.9602	36.9602
					29.6730	29.6730	29.6730

One precipitate was given a special series of blastings to determine whether or not the alumina was hygroscopic after the blasting. The results (Table VI) indicate that this change in weight was very largely

Table VI—Showing Hygroscopic Property of Alumina

	Weights
Constant weight obtained (cruc. + ppt.)	21.0598
Left 6 days in the desicator................	21.0636
Blasted 10 min.............................	21.0611
After 24 hrs. blasted 20 min................	21.0605
After 24 hrs. blasted 20 min................	21.0604
After 24 hrs. blasted 20 min................	21.0602
Blasted a second 20 min....................	21.0598
Left covered in the balance case 24 hrs......	21.0624

due to the precipitate, since a platinum crucible treated in much the same way did not change appreciably in weight.

CONCLUSIONS

I—Boiling for 1 min. completely precipitates all aluminum present, and longer boiling may lead to a re-solution of part of it.

II—Aluminum hydroxide need not be washed free from ammonium chloride before ignition.

III—The excess of ammonia present when precipitation is made should be as small as possible and never more than 1 or 2 cc. in 250 cc. of solution.

IV—Aluminum hydroxide freshly precipitated is soluble to a slight extent in water and to about the same extent in dilute ammonium nitrate solution.

V—The precipitated hydroxide, when large, must be blasted 40 min. to insure its being reduced to a constant weight.

VI—The ignited alumina is strongly hygroscopic.

Note—Since this paper was written a paper has been published by W. H. Daudt, This Journal, 7 (1915), 847, confirming what we have found to be true regarding the presence of ammonium chloride at the time of ignition.

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THE ANALYSIS OF SOAP POWDERS

By Louis Rosenberg and Victor Lenher
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During recent years great progress has been made in those industries which supply cleansing materials. The advent of the modern washing powder has brought into the household a convenient, economical cleanser. The powders which to-day are in common use divide themselves into three general classes:

I—So-called Washing Powders, composed of trisodium phosphate, borax, or a mixture of various proportions of sodium carbonate and bicarbonate: it is obvious from the composition of these powders that their main efficiency lies in their ability to soften the water used.

II—Soap Powders proper, a mixture consisting of various proportions of sodium carbonate and soap: the soaps used are commonly prepared from cottonseed soap stock and have as a filler sodium carbonate, which itself is a water softener of great value.

III—Scouring Powders, which contain an abrasive and soap either with or without the addition of sodium carbonate.

In this article are described various experiments which have been conducted on the soap powders proper. The basic scheme used is that given by Leffmann in Allen's "Commercial Organic Analysis." During our studies, Dr. Leeds published a scheme for the analysis of soap, which is quite similar to the one used by us for the analysis of soap powders. Additions and modifications to the older schemes were made as the work progressed, and it is to aid the soap analyst that important details are described.

Our purpose in presenting this paper is to offer our experience to those interested in the analysis of soap powders, and we do so because the particular problems we have found are not discussed in the form that is desirable for those who have similar problems to solve.

MOISTURE

The ordinary methods for determining water in soap are not satisfactory when applied to washing powders. Experiments have repeatedly demonstrated that heating a 10-g. sample at 105° C. until the weight becomes constant is worthless, for the odor of decomposing soap is usually observed before this temperature is reached. When heated for 1 hr. to 105°, most of the soap powders give off a strong odor of decomposed soap, hence the determination is of little value. Further, the soap powder upon heating to 105° frequently forms lumps which prevent complete drying: even if a weighed glass rod is used to break up the lumps, it is difficult to keep the sample from caking. In powders which contain as high as 30–40 per cent water, spattering frequently occurs. Leffmann's suggestion for soap—that the drying be done on a sand bath—is useless with many powders, inasmuch as even with the greatest care, decomposition takes place. The methods in use for the determination of moisture in a high-grade stearic acid soap are unsatisfactory, as they frequently fail entirely when applied to soap powders containing cottonseed soap stock or low-grade tallow soap.

The method of Fahrion, in which the sample is heated with three times its weight of oleic acid until a clear solution is obtained, indicating the removal of the water, was found to be inapplicable, as difficulty was experienced in determining when the solution became clear. The recommendation of Fahrion, to heat the sample (to which has been added oleic acid) in a platinum dish over a free flame, was also tried with the soap powders, but even with the greatest care decomposition of the soap took place. On a sand bath the powder soon decomposed. Sample 1, containing actually 37.8 per cent of water, was used in experimenting with oleic acid in order to test this method for moisture. Six samples of 2 g. each were

mixed with 6 g. of anhydrous oleic acid. On heating, the odor of decomposing soap was noticed from all. The two samples showing the clearest solution and the least odor indicated 56.5 per cent and 60.3 per cent water, respectively, or an error of 50 to 60 per cent more water than they actually contained.

The method recommended is one in which the heating can be done at low temperature. A vacuum drying oven is admirably suited for this purpose. The various powders examined in our studies were heated between 60 and 65° at a pressure of 60 mm. until the weight became constant. In the 2-g. samples used, constant weight was attained only after 10 hrs.' heating, and in some cases 20 hrs. of heating were required. No odor was observed and the samples did not cake. The results (percentages) were as follows:

No. 1, 37.85 and 37.82; No. 2, 23.75 and 23.99.

The recommendations commonly given to heat for 12 hrs. are insufficient. No. 3, for example, required 20 hrs.' heating in the vacuum oven before constant weight was attained.

PETROLEUM ETHER EXTRACTION

The usual procedure of extraction with petroleum ether, with a Soxhlet extractor, was used in order to determine the unsaponified fat, unsaponifiable matter from the fat and free fatty acids. A 10-g. sample of the soap powders frequently required as much as 15 hrs. of extraction with petroleum ether. The solvent was subsequently evaporated and the residue weighed. All of the powders examined were found to contain less than 1 per cent of unsaponified matter.

FATTY ACID DETERMINATION

The determination of the fatty acids in the soap powders is, of course, of the greatest importance, inasmuch as it means the soap content of the powder. Of the various methods which have been suggested for the determination of the fatty acid content in soaps, the most important are: (1) Direct weighing; (2) petroleum ether extraction; (3) potassium soap; (4) volumetric. Fendler and Frank[1] have shown the inaccuracies of all of these methods.

In the direct weighing method, a piece of weighed paraffin or stearic acid is added to the liberated fatty acids, and the cooled solidified mass is subsequently weighed. The results are frequently very high. Extraction of the fatty acids with petroleum ether and evaporation of the solvent commonly gives low results. The variation in the results depends largely on length of drying of the fatty acids obtained. In one instance the percentage of fatty acids was reduced nearly 60 per cent by long drying. Fendler and Frank preferred for accuracy the potassium soap method, but inasmuch as the potassium soap must be dried for 20 to 28 hrs. on a water bath, the method is rather too time-consuming for ordinary commercial analysis. Volumetric methods can be said to give only fair results.

The cake method is a simple, rapid method; but unless due precautions are taken, discordant results

[1] Z. angew. Chem., **22** (1909), 252–61.

are obtained. The inaccuracies of the cake method, according to Noerdlinger, are attributed to the stearic acid used to collect the fatty acid. If the stearic acid is previously heated to 160° the inaccuracies are avoided. In our experiments the cake method was adopted as the most satisfactory procedure, and was carried out as follows:

A 2-g. sample of the powder is placed in a tall, narrow, 200-cc. beaker 4 in. high, and the sample dissolved in hot water. An excess of $N/2$ nitric acid is then added to separate the fatty acids. The solution, stirred continuously, is heated for $1/2$ hr. in boiling water, and a weighed portion of 4 g. of stearic acid, previously heated to 160°, added. The solution is stirred repeatedly and warmed for another $1/2$ hr., after which the beaker and contents are allowed to cool. The stirring rod, which should be about 1 in. longer than the beaker, is allowed to remain in the beaker. The fatty acid and the stearic acid on cooling solidify, and can be easily lifted out of the beaker by means of the stirring rod. The liquid in the beaker can be filtered in order to collect any solidified fat which has not adhered to the cake, but usually this amount is small. The circle left by the cake around the beaker can be readily scraped off and added to the cake. The cake is then wiped with filter paper and placed upon a weighed cover glass, allowed to dry for several hours, after which it is weighed. The weighed cake less the weight of the stearic acid added gives the amount of fatty acid in the sample.

If the melted fat is allowed to cool rather slowly, a more uniform cake is formed, and the cake can be more easily removed from the beaker; whereas if the cake be cooled quickly, the rapid solidification causes moisture to be enclosed in the cake. If cavities are formed, these are likely to enclose the solution. A 200 cc. beaker of the tall, narrow type is a convenient size, inasmuch as a cake suitably fitted to the balance pan is formed. Furthermore, the addition of mineral acid to a washing powder commonly causes much effervescence, due to large percentages of sodium carbonate: the use of a tall beaker lessens the danger of overflow during this sudden effervescence.

TOTAL ALKALI DETERMINATION

Should the percentage of total alkali be required, the excess of standard acid required to liberate the fatty acid can be titrated back with $N/2$ alkali. In washing powders, the total alkali reported does not have the significance that it does in soap. The presence of sodium carbonate marks the amount of alkali combined as soap. A total alkali determination cannot be used as a means for determining the relative merits of different powders, since the powder with the highest per cent of sodium oxide may be the one which contains the lowest per cent of soap. A total alkali determination is unnecessary, unless it is desirable to check the individual alkali determinations.

SODIUM CHLORIDE DETERMINATION

In our hands the gravimetric method is preferable over the volumetric method for the determination of chlorides in soap powders.

ALCOHOL EXTRACTION

In order to separate the soap content of the powders from such fillers as sodium carbonate, sodium silicate, borax, etc., it is necessary to make an extraction with alcohol. Due to the fact that soap powders contain a high percentage of water, it is preferable to use absolute alcohol. If, however, the analysis is to be made on a sample which has been dried in the vacuum oven, 95 per cent alcohol is satisfactory.

A 2-g. sample of the powder can be extracted in a 250-cc. beaker with 100 cc. of alcohol for $^1/_2$ hr. The alcohol becomes yellow colored and a white residue of the inorganic salts, such as sodium carbonate, sodium silicate, and borax, separates out and can be filtered off and washed with alcohol. In this extract, phenolphthalein will show the presence of caustic alkali. No free alkali was found in any of the powders examined. On the other hand, all of the soap powders showed a trace of free fatty acid. The alkali combined as soap is found by adding a large excess of water to the alcoholic solution and boiling off the alcohol, decomposing the soap with an excess of $N/2$ nitric acid and titrating back with $N/2$ alkali. The alkali found is reported as Na_2O. No distinction need or-

DETERMINATION OF SODIUM CARBONATE AND SILICATE

The residue from the extraction with alcohol is dissolved in water and diluted to exactly 250 cc. in a volumetric flask. The residue from all of the samples analyzed was completely soluble in hot water. In one or two powders a slight residue remained upon the filter paper, but the amount was too small to warrant further examination. Aliquot portions of 50 cc. are taken for the silica determination. The solution is acidulated with hydrochloric acid and evaporated to dryness, as in the ordinary method for the determination of silica. The acidification and evaporation are repeated. The soluble matter is extracted with hot water and the residue when dried and ignited is weighed as SiO_2. The percentage of silica was calculated to sodium silicate ($Na_2Si_4O_9$). In the soap powders analyzed the amount of silica found was so small that it was probably present as an impurity rather than as a real constituent of the powder. Borax was not found to be present in any of the powders analyzed. In the absence of borates and with less than 1 per cent of sodium silicate, a fair determination of the sodium carbonate can be made by direct titration with $N/2$ hydrochloric acid, using methyl orange as the indicator.

TABLE I—SCHEME OF ANALYSIS

Weigh out a 2-gram sample.   Dry in vacuum oven at 65° C.   Loss in weight = Moisture.   Extract with petroleum ether.

EXTRACT	RESIDUE consists of Soap and Mineral Constituents. Extract with 95 per cent alcohol.
Contains Uncombined Fat. Evaporate the ether, dry at 110° C. and weigh.	EXTRACT consists of Soap and free Alkali. (1) Add 2 or 3 drops of phenolphthalein, and titrate with $N/10$ acid; the amount used corresponds to free caustic alkali: calculate as Na_2O. (2) Next add a large excess of water, boil off the alcohol, decompose with excess of N HNO_3 and boil: add 4 g. stearic acid, boil again, and cool. SOLUTION contains Soda combined as Soap. Titrate excess of acid back with $N/2$ NaOH. N acid used corresponds to Na_2O combined as Soap. CAKE contains Fatty Acid. Remove cake, dry and weigh.	RESIDUE consists of Na_2CO_3, NaCl, $Na_2Si_4O_9$, Na_2SO_4, $Na_2B_4O_7$, Starch and Insoluble Matter. Dissolve in hot water, dilute to 250 cc. and filter: divide the filtrate into five equal parts. 1—Na_2CO_3: Titrate with $N/2$ HCl. 2—NaCl: Precipitate with $AgNO_3$ and weigh as AgCl. 3—$Na_2Si_4O_9$: Add HCl and evaporate to dryness; repeat. Add water, filter, dry, ignite and weigh as SiO_2. Calculate to $Na_2Si_4O_9$. 4—Na_2SO_4: Precipitate with $BaCl_2$ and weigh as $BaSO_4$. Calculate to $BaSO_4$. 5—Borates: If present, determine by glycerol or mannitol method. RESIDUE Consists of Starch and Insoluble Matter.

dinarily be made between the sodium and the potassium in soap powders. The alcoholic extract should be diluted to at least 250 cc. before titrating in order to get a satisfactory end-point.

A rapid method for analysis of soap powders is based on the alcoholic extraction procedure: A 2-g. sample is extracted with absolute alcohol, the solution filtered and the filtrate tested with phenolphthalein for free alkali. If the solution contains free alkali it is titrated with $N/2$ nitric acid: the alcohol is then evaporated off, leaving a residue of dry soap, which, if it has not been necessary to add nitric acid, can be weighed and reported as percentage of total soap. To determine the alkali combined as soap, the dry residue of soap is dissolved in water and the solution titrated with $N/2$ nitric acid, using methyl orange as the indicator. The residue from the alcoholic extraction, which consists of sodium carbonate, sodium silicate, borax, etc., can be tested by the methods given below. The percentage of total soap plus sodium carbonate, etc., deducted from 100 per cent will give the approximate amount of water, or, an accurate water determination can be made in the vacuum drying oven.

Should sodium bicarbonate be present in the powder, the treatment of the residue insoluble in petroleum ether and in alcohol, would necessarily need to be materially modified.

SCHEME OF ANALYSIS

The condensed scheme of analysis outlined in Table I is suggested for soap powders. It is based on the solubility of soap in alcohol and the insolubility of the inorganic constituents. It possesses the advantage that but one sample need be taken for analysis, and the soap itself is separated at once from the other constituents of the powder. In the simple scheme, as given, such substances as fluorides, rosins, glycerin, sugar, and sodium bicarbonate are not provided for. Our experience with the powders examined showed them to be of simple composition.

DISCUSSION OF RESULTS

The analyses of a number of representative soap powders are given in Table II. Their composition in a number of cases differs materially from that given for the same powder by Bragg[1]. This difference is doubtless due to the introduction of the continuous

[1] *Chemical Engineer,* **18** (1913), 73.

drying apparatus which has changed the formula used by a number of the larger manufacturers. The continuous drying apparatus allowed the manufacturer to

TABLE II—TABULATION OF RESULTS (PERCENTAGES)

Sample No.	Moisture	Fatty Anhydride	Na₂O as Soap	Total Soap	Na₂CO₃	Na₂SiO₃	NaCl	Unsaponified Matter	Total Per cent
1	37.82	18.39	2.63	21.02	39.37	0.45	0.55	0.69	99.90
2	22.06	19.44	3.58	23.02	53.53	0.99	0.75	0.24	100.59
3	23.75	21.63	2.71	24.34	50.35	0.72	0.35	0.85	100.36
4	20.86	18.93	2.65	21.58	55.38	0.90	0.86	0.45	100.03
5	18.52	20.34	2.68	23.02	56.45	0.36	1.32	0.74	100.41
6	43.38	16.74	2.23	18.97	36.30	0.72	0.34	0.87	100.58
7	30.40	21.46	2.86	24.32	40.00	0.99	0.72	0.79	97.22

increase the water content of the product and to lessen the amount of soap. Previously this addition of water resulted in an inferior caked powder.

The determinations made show that the soap powders consist essentially of sodium carbonate and soap. This should be the case from the method of manufacture employed, i. e., the soap stock, caustic alkali and soda are incorporated and the product prepared similar to that of soap. After hardening it is ground into the powder form in which it appears on the market.

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THE DETERMINATION OF CHROMIUM AND VANADIUM IN STEEL BY ELECTROMETRIC TITRATION

By GEORGE LESLIE KELLEY AND JAMES BRYANT CONANT
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The method to be described includes the solution of the steel in acid, the oxidation of the elements in question under suitable conditions, and the titration of the solution with ferrous sulfate using the electrometric method of determining the end-point. This method of titrating chromates has been described recently by Hildebrand[1] and by Forbes and Bartlett,[2] and has been applied by the authors to the determination of vanadium.[3] The apparatus is in all respects identical with that described elsewhere for the determination of vanadium and therefore will not be dealt with here.

DETERMINATION OF CHROMIUM IN STEEL

It was hoped that by the application of the electrometric method to the determination of chromium, an accurate and rapid method could be devised for the determination of this element. To this end the oxidation of chromium salts to chromates was undertaken after the methods suggested by Walters,[4] Rich[5] and Tusker.[6] These methods were selected because of their simplicity and because they involve no filtering. In these methods ammonium persulfate is used to oxidize chromium and manganese to chromate and permanganate, sometimes with the addition of silver nitrate. Hydrochloric acid, added in small amounts to the hot solution, serves to reduce the permanganic acid, but is without action on the chromates. On trying the different modifications of this method which have been published, extremely variable and unsatisfactory results were obtained. This experience

[1] J. Am. Chem. Soc., 35 (1913), 869.
[2] Ibid., 35 (1913), 1527.
[3] Ibid., 38 (1916), 341.
[4] Met. Chem. Eng., 12 (1914), 310.
[5] Ibid., 13 (1915), 239.
[6] Chem.-Ztg., 39 (1915), 122.

led us to undertake a more careful study of the conditions necessary to make the method work with certainty.

It seemed possible to us that the following errors might interfere with the successful working of the method: (a) Incomplete decomposition of ammonium persulfate, (b) incomplete oxidation of chromium, (c) reduction of part of the chromate by hydrochloric acid during the breaking up of permanganic acid, and (d) incomplete reduction by the hydrochloric acid of manganese in the higher states of oxidation. (a) and (d) would tend to make the results too high, while (b) and (c) would have the opposite effect; thus it might be possible for some of the errors to exist undetected because of counterbalancing. We therefore planned to test each point separately.

DECOMPOSITION OF AMMONIUM PERSULFATE

Eight solutions of 300 cc. volume, each containing 0.0102 g. of chromium as chromate and 65 cc. of sulfuric acid (sp. gr. 1.20), were heated to boiling and 5 g. of ammonium persulfate added. The solutions were then boiled in groups of two for 5, 10, 15 and 30 min., after which they were cooled and titrated with ferrous sulfate, using the electrometric end-point. The potassium dichromate solution had been standardized against ferrous sulfate electrometrically and this in turn had been compared with standardized potassium permanganate solution.

From the experiments shown in Table I, it is evident that 5 min'. boiling of such a solution does not

TABLE I—THE DECOMPOSITION OF AMMONIUM PERSULFATE
0.0102 G. Chromium Present in Each Case

Time of Boiling (min.)	5	10	15	30
Chromium Found (g.)	0.0159	0.0101	0.0095	0.0096
	0.0196	0.0117	0.0094	0.0082

completely remove the ammonium persulfate and the oxidizing agents into which it decomposes. Even 10 min. is insufficient in some instances. Boiling for a much longer time has the apparent effect of reducing some of the chromate, for the values are uniformly lower than the expected values. It is not improbable that this reduction goes on from the first, but its effect upon the titration is masked by the fact that other oxidizing agents have not been removed. This may be explained as due to the breaking down of the ammonium persulfate in acid solution to give Caro's acid and hydrogen peroxide, both of which reduce chromium. We believe that any method depending upon ammonium persulfate alone for its oxidizing action can give correct results only by a balancing of errors. This opinion is supported by other experiments in which the sulfuric acid mentioned above was changed to higher and lower concentrations, and in which it was partly and completely replaced by nitric acid.

DECOMPOSITION OF AMMONIUM PERSULFATE IN THE PRESENCE OF SILVER NITRATE

Marshall[1] suggested the use of ammonium persulfate and silver nitrate in dilute nitric acid as an oxidizing agent for manganese. Experiments parallel

[1] Chem. News, 83, 16.

to those shown in Table I were tried, using the same volume of solution and the same amounts of ammonium persulfate and potassium dichromate, but adding in every case 10 cc. of a solution of silver nitrate which contained 1.3 g. of $AgNO_3$ in a liter. The sulfuric acid (sp. gr. 1.20) was replaced by nitric acid (sp. gr. 1.13) as indicated in Table II.

TABLE II—DECOMPOSITION OF AMMONIUM PERSULFATE IN PRESENCE OF SILVER NITRATE

Initial Volume, 300 cc.   0.0102 G. Chromium Present in Each Case

Acid Used	60 cc. HNO_3	60 cc. H_2SO_4	60 cc. HNO_3
Time of Boiling	5 min.	30 min.	30 min.
Chromium Found (G.)	0.0103	0.0103	0.0101
		0.0101	0.0101

The variations found in the experiments recorded in Table II are within the experimental error of the method and show that in the presence of silver nitrate the course of the decomposition of ammonium persulfate is very different from that which it follows when this salt is absent. This follows from the fact that no oxidizing materials, except the chromium, were present after 5 min.' boiling, and 30 min.' boiling did not result in the reduction of any chromate. The presence of nitric acid does not appear to be necessary, but since in the analysis of steel the iron in the sulfuric acid solution of the sample is most conveniently oxidized with nitric acid, the effect of nitric acid was determined.

THE REDUCTION OF OXIDIZED MANGANESE WITH HYDROCHLORIC ACID

Solutions containing 0.005 g. of manganese as manganous sulfate, an amount corresponding to 0.5 per cent of manganese in a 1-g. sample of steel, were treated with nitric or sulfuric acid, as specified in Table III, and made up to a volume of 300 cc. The

TABLE III—EFFECT OF SMALL AMOUNT OF COLD HYDROCHLORIC ACID ON TITRATION OF MANGANESE

HNO_3 (Sp. Gr. 1.13)	H_2SO_4 (Sp. Gr. 1.20)	CHROMIUM Taken	CHROMIUM Found	Oxidizing Materials Found in Terms of Chromium
3 drops	300 cc.	0.0010	0.0010	0.0000
...	60 cc.	0.0069	0.0070	0.0001
60 cc.	..	0.0081	0.0081	0.0000
60 cc.	..	0.0081	0.0081	0.0000

solutions were heated to boiling and 10 cc. of the silver nitrate solution added, followed by 5 g. of ammonium persulfate. After boiling 7 min., 1 cc. of dilute hydrochloric acid (1 acid : 2 water) was added, and boiling continued for 5 min. In all cases the solutions had a red color after the addition of ammonium persulfate; and they became colorless after the addition of hydrochloric acid. To detect oxidizing substances left in the solution, after cooling, a measured amount of potassium dichromate was added and the solution titrated with ferrous sulfate as usual.

Under the conditions of the experiments shown in Table III, the oxidation and reduction of manganese were entirely satisfactory for the purposes of the method, inasmuch as no unreduced manganese compounds were left in the solution after the treatment with hydrochloric acid.

Subsequent work on the method as finally established for the analysis of steel showed that the oxidation of manganese by ammonium persulfate was by no means as simple as might be supposed. On the addition of the oxidizing agent to the hot solution, the colors resulting after a few minutes' boiling would vary from a light pink, a deep red, a murky brown and finally an actual precipitate of manganese dioxide, depending on the amount of manganese present. On the addition of hydrochloric acid to such solutions, those having a pink or red color would grow yellow (in the presence of chromium) or colorless, in the course of 2 min. The dark brown solutions would lose their color and turbidity only after boiling 5 to 7 min., while solutions containing precipitated manganese dioxide failed to clear up, except on prolonged boiling with a larger amount of hydrochloric acid. In spite of the fact that large amounts of manganese cause the solution to become murky and even to precipitate manganese dioxide, it has been found possible to determine Cr in a 1-g. sample of steel when the amount of manganese corresponded to 3 per cent. This is possible for two reasons: (*a*) When such a solution is obtained in the presence of the other products of solution of steel there is less tendency for the manganese to precipitate as oxide; and (*b*) if the acid concentration is kept reasonably high (60 cc. of sulfuric acid of sp. gr. 1.20 to 300 cc. of solution) a very high percentage of manganese is necessary to cause precipitation. However, the latter condition may be carried too far, for if the concentration of acid amounts to as much as 100 cc. of sulfuric acid of this strength, both the chromium and manganese oxidize with greater difficulty and may not oxidize at all.

THE OXIDATION OF CHROMIUM AND THE EFFECT OF BOILING WITH HYDROCHLORIC ACID

In order to test the usefulness of the method for the oxidation of chromium, solutions containing varying quantities of potassium dichromate and 2 cc. of $N/10$ potassium permanganate solution were reduced with an excess of ferrous sulfate in the presence of 30 cc. sulfuric acid (sp. gr. 1.40). The excess of ferrous iron was oxidized by heating with 15 drops of concentrated nitric acid. The solution, now containing a known weight of chromium with some ferric salt and manganese, was diluted to 300 cc. and treated with 10 cc. of dilute silver nitrate solution and 5 g. of ammonium persulfate as usual. After boiling 10 min., 5 cc. of hydrochloric acid (1 to 3) were added and the solution boiled 5 min. longer. The solution was then cooled and titrated electrometrically.

TABLE IV—DETERMINATION OF CHROMIUM IN THE PRESENCE OF MANGANESE

0.0022 G. Manganese Added in Each Case

Chromium Taken (G.)	0.0102	0.0203	0.0404
Chromium Found (G.)	0.0101	0.0203	0.0403

The few figures given in Table IV are sufficient to illustrate that the oxidation of chromium by ammonium persulfate and silver nitrate is a quantitative process and that hydrochloric acid effectively reduces the manganese oxidized at the same time without reducing the chromium.

OBSERVATIONS CONCERNING THE ELECTROMETRIC TITRATION OF CHROMIUM

The apparatus and method used was identical with that used by Forbes and Bartlett,[1] except as

[1] *Loc. cit.*

noted in our paper on the determination of vanadium. In titrating pure chromate solution with ferrous sulfate, we noted the anomalous rise first observed by Forbes and Bartlett. Thus on setting the needle of the galvanometer on the middle of the scale and adding ferrous sulfate, a retrograde motion of the needle began at once, continuing until it had covered about seven divisions on the scale. The movement became less and less as more ferrous sulfate was added until just before the end-point was reached, when it remained stationary until a slight excess of ferrous sulfate caused it to move rapidly in the direction opposite to that in which it had been moving. As soon as enough dichromate solution had been added to cause the needle to return to its stationary position the titration was considered completed.

It was noted early in the course of these experiments that the anomalous rise in potential either was absent or was much less marked in solutions which had been treated with an oxidizing agent, such as ammonium persulfate. During the titration of the solution of a sample of steel which had been so oxidized, the needle remained within a few divisions of the original point during the addition of the first portions of ferrous sulfate, and would remain absolutely stationary during the addition of the last portions, when an excess would produce a decided movement. Ferric iron likewise seemed to inhibit this phenomenon. In one instance, a solution of chromium sulfate, after oxidation with potassium permanganate, was boiled with ammonia to decompose the permanganate. The solution so obtained, when filtered to free it from manganese dioxide, showed no anomalous rise on titrating. Our limited investigation of this point leads us to believe that amounts of oxidizing agents other than chromate, which are too small in amount for detection otherwise, are effective in suppressing this anomalous behavior of the cell.

THE EFFECT OF FERRIC IRON ON THE ACCURACY OF THE TITRATION

Having noted that ferric salts interfered with the anomalous rise, we became interested to know how these salts would affect the titration if they were present in large amounts. Accordingly, solutions containing 1, 2 and 3 g. of ferric iron were prepared and known quantities of chromium added. Each solution had a volume of 250 cc. and contained 25 cc. of sulfuric acid (sp. gr. 1.40) and 0.0253 g. of chromium as chromate. The ferrous sulfate solution was of such strength that one drop was equivalent to 0.00005 g. of chromium. In Table V the

TABLE V—EFFECT OF FERRIC IRON ON THE TITRATION

Grams Iron Added as Ferric Alum	Gram Chromium Taken	Found	Galvanometer Changes after Successive Drops FeSO₄					Total (2 cc.)	Anomalous Rise before End-Point
			1	2	3	4	5		
None	0.0253	0.0253	0	1	3	4	4	12	7 divisions
1	0.0253	0.0252	0	0.5	2	3	3	8.5	2 divisions
2	0.0253	0.0252	0	0.5	2	2	3	7.5	None
3	None	None	...	...	None				1 division
None	0.0051	..	0	2	2.5	3	3	10.5	Several divisions

change of reading of the galvanometer is shown for each drop of ferrous sulfate added immediately before and after the end-point. After the final throw of the needle, enough potassium dichromate solution was added to cause the needle to return to the end-point and two drops additional.

The experiments recorded in Table V indicate that the end-point is as sharp for large amounts of chromium as for small amounts (cf. electrometric titration of vanadium[1]), but is much affected by the presence of ferric salts, especially as regards the anomalous rise. That the amount of change for a given increment of ferrous salt would be less in the presence of a large amount of ferric iron would be expected from a consideration of the ferric-ferrous potential, although we should not expect the differences to be so large as those found. The expression,[2]

$$\pi = 0.988 + \frac{RT}{F}\ln\frac{Fe^{+++}}{Fe^{++}}$$

gives the value of the potential which is established when the first drop of ferrous iron in excess is added. A given amount of ferrous iron will obviously produce a greater change in the value of π when the concentration of Fe^{+++} is small than when it is large. The effect upon the anomalous rise cannot be so readily explained. The differences observed in the above experiments show that there might be a tendency to add more ferrous sulfate when titration is made in the presence of large amounts of ferric salt, but if the titration is carefully made the end-point is the same.

THE SENSITIVENESS OF THE END-POINT

It seemed possible that while our method of noting the potential change was simpler than that used by Forbes and Bartlett, perhaps it was less sensitive. To test this point we diluted our solutions to ten times the volume and made titrations with these. The original solutions of potassium dichromate contained 0.001 g. of chromium per cc. The diluted solution therefore contained 0.0001 g. per cc. In working with this and an equivalent solution of ferrous sulfate we had no difficulty in making duplicate titrations on 30 cc. of solution within 0.30 cc. This corresponds to an error of 0.000033 g. It would thus appear that our apparatus was sufficiently sensitive for practical purposes.

ACID CONCENTRATION AND THE EFFECT OF CHLORIDES

Numerous experiments on the titration of chromium in steel failed to show any effect of temperature on the accuracy or position of the end-point. The acid concentration should be quite high, particularly in the presence of chlorides, as it prevents irregular movements of the needle. Our experiments on the titration in the presence of chlorides showed that small amounts were without effect, intermediate quantities gave irregular results, but cold solutions containing large amounts of hydrochloric acid gave excellent titrations.

THE TITRATION OF CHROMIUM AND VANADIUM

Since chromium and vanadium often occur together in steel, the fact that any device for oxidizing chromium

[1] Loc. cit.
[2] Peters, Z. physik. Chem., **26** (1898), 205.

oxidizes the vanadium at the same time, made it necessary to adapt the method to the determination of both elements. When the procedure employed for the analysis of plain chromium steel is applied to the analysis of a solution containing chromium as chromate and vanadium as vanadate, both are reduced by titration with ferrous sulfate. The end-point obtained under these circumstances is less sharp than the one obtained when chromium alone is titrated. The end-point observed is really the vanadium end-point, the characteristics of which have been described elsewhere.

To test the accuracy of this titration, known quantities of chromium as chromate solution were mixed with known quantities of vanadium. These mixed solutions were reduced with excess of ferrous sulfate and oxidized with nitric acid, silver nitrate and ammonium persulfate under the conditions described above. The solutions were then boiled with a small amount of hydrochloric acid. After cooling to 20° C. they were titrated electrometrically with ferrous sulfate in a volume of 250 cc. The chromium was assumed as the amount taken, the difference being taken as vanadium.

TABLE VI—TITRATION OF SOLUTIONS CONTAINING BOTH CHROMIUM AND VANADIUM

Chromium Assumed (G.)		0.0203	0.0101	0.0203	
Vanadium Taken (G.)	0.0255	0.1000	0.0255	0.1000	0.0255
Vanadium Found (G.)	0.0254	0.1001	0.0252	0.1005	0.0247

This titration gives the oxidizing value of both chromium and vanadium. As the percentage of vanadium in steels is often quite low, it is convenient to take a 3-g. sample for the vanadium determination, of which an aliquot portion may be used for the total chromium and vanadium determination.

THE APPLICATION OF THE METHOD TO THE DETERMINATION OF CHROMIUM AND VANADIUM IN STEEL

PROCEDURE IN THE ABSENCE OF VANADIUM—A 1-g. sample of steel is dissolved in 60 cc. of sulfuric acid (sp. gr. 1.20), evaporated until salts separate, diluted to a volume of 60 cc. and while still hot oxidized with dilute nitric acid. Evaporation until salts separate is recommended here as a device for breaking up carbides: the completeness with which this has been accomplished may usually be judged by an examination of the diluted solution, for, if carbides remain undestroyed, a more or less well marked turbidity, possibly accompanied by clearly visible, dark particles, will be noted: when this method fails to break up carbides, a more vigorous attack may be had by dissolving in 60 cc. of dilute hydrochloric acid (sp. gr. 1.15) after which the solution should be oxidized with nitric acid; this solution, however, must be evaporated with 60 cc. dilute sulfuric acid to the appearance of fumes to give complete separation of hydrochloric acid. However prepared, the solution (containing the equivalent of 60 cc. sulfuric acid of sp. gr. 1.20) should be diluted to a volume of 300 cc. and heated to boiling. To the boiling solution are then added 10 cc. of a solution of silver nitrate containing 2.6 g. per liter and 5 g. ammonium persulfate, conveniently used in the form of a solution containing 50 g. of the salt in a volume of 250 cc.

The solution is boiled vigorously for 10 min., after which 5 cc. dilute hydrochloric acid (1 : 3) are added and the boiling continued for 5 min. After cooling to a convenient temperature and the addition of a little more sulfuric acid, the solution is ready to titrate electrometrically.

It is generally possible to tell if the process has failed at any point by certain readily noted signs. Thus the failure to break up carbides may be noted by examining the solution. If the solution contains chlorides which would interfere with the catalysis of the ammonium persulfate oxidation by the silver nitrate, a precipitate of silver chloride will appear. If the oxidation of the chromium is unsatisfactory, the deep red color of the permanganic acid will fail to appear. If the decomposition of the permanganic acid with hydrochloric acid is incomplete, the solution will not have a clear yellow color. The chief pitfalls into which the analyst may fall are, (1) incomplete breaking up of carbides of chromium, (2) incomplete oxidation of chromium due to high concentration of acid, precipitation of silver or the presence of too little ammonium persulfate, (3) failure to remove all of the products of the decomposition of the oxidizing agent (which may be accomplished by boiling 10 min.), and (4) failure to remove the chlorine liberated by the action of hydrochloric acid on the permanganic acid for which 5 min. boiling is necessary.

PROCEDURE IN THE PRESENCE OF VANADIUM—A 2 or 3 g. sample of steel is dissolved as in the above procedure, using up to 100 cc. of dilute sulfuric acid where this is necessary. The solution is diluted to 200 cc., heated to 80° C., and after the addition of 5 g. of sodium phosphate, is titrated with $N/10$ potassium permanganate until the first "gray" color appears. The solution is then cooled to 10 to 20° C. by adding ice, and more sulfuric acid is added, when it is ready for the electrometric titration with ferrous sulfate. After the titration, the stirrer and the electrodes are rinsed into the solution, which is then made up to definite volume and a portion representing 1 g. of the steel is taken for the chromium determination. This portion is diluted to 300 cc., heated to boiling and otherwise treated as described under the procedure for chromium, except that before the titration with ferrous sulfate, the solution should be cooled thoroughly. The amount of chromium present is calculated from the amount of ferrous sulfate used, as corrected for the amount of vanadium present and the amount of chromium used in finding the end-point in this and the previous titration.

When tungsten is present the precipitate of WO₃ makes it difficult to deal with the carbide present. The addition of 5 g. of sodium phosphate, as suggested by Wdowiszewski,[1] by keeping the tungstic oxide in solution, makes this much easier.

The fact that potassium permanganate will oxidize chromic salts in hot acid solution causes the end-point obtained by oxidizing with this reagent to be somewhat fleeting. Usually the end-point is visible for 2 min. In general, there is very little difficulty in find-

[1] *Chem.-Ztg.*, **34**, 1365.

ing the end-point if the solution is hot (70 to 80° C.), fairly dilute (200 cc.) and the chromium content is not too high. The addition of some sodium phosphate prevents interference from the yellow color of the ferric iron, but green chromic salt is the main cause of difficulty. The first change of color which appears (from green to "gray") should be taken as the end-point. If too much permanganate is added, a few drops of ferrous sulfate may be added and the end-point sought again.

TABLE VII—ANALYSIS OF SAMPLES OF STEEL FOR CHROMIUM AND VANADIUM

Expt. No.	Sample	Sample Weight G.	Per cent Mn Added	Per Cent Chromium			Per Cent Vanadium		
				Present	Added	Found	Present	Added	Found
1	Private Standard	1	0.50	1.46	..	1.46	..	..	..
2		1	0.80	1.46	..	1.49			
3		1	1.50	1.46	..	1.47			
4		1	3.10	1.46	..	1.44			
5		1	..	1.46	..	1.44			
6		1	..	1.46	..	1.45			
7		1	..	1.46	..	1.40			
8		1	..	1.46	..	1.45			
9		1	..	1.46	..	1.44			
10		1	..	1.46	..	1.44			
11		1	..	1.46	..	1.43			
12		2	..	1.46	..	1.46			
13		2	..	1.46	..	1.46			
14		3	..	1.46	..	1.47			
15		3	..	1.46	..	1.47			
16		3	..	1.46	..	1.47	..	0.13	0.13
17		3	..	1.46	..	1.47	..	0.26	0.26
18		3	..	1.46	..	1.47	..	0.53	0.52
19		3	..	1.46	..	1.46	..	1.04	1.03
20		2	..	1.46	..	1.47	..	1.57	1.55
21		2	..	1.46	..	1.46	..	2.55	2.58
22		2	..	1.46	2.08	3.54	..	2.55	2.58
23		2	..	1.46	3.60	5.08	..	2.55	2.58
24	Bureau of Stds. No. 32	1	..	0.89	..	0.92			
25	32	1	..	0.89	..	0.93			
26	32	1	..	0.89	..	0.92			
27	32	1	..	0.89	..	0.93			
28	31	1	..	3.51	..	3.51			
29	31	1	..	3.51	..	3.56			
30	31	1	..	3.51	..	3.54			
31	31	1	..	3.51	..	3.56			
32	34	1	..	0.01	..	0.01			
33	34	1	..	0.01	..	0.003			
34	34	1	..	0.01	2.53	2.55			
35	34	1	..	0.01	2.53	2.55			
36	10(b)	1	..	0.005	..	0.007			
37	10(b)	1	..	0.005	2.53	2.54			
38	10(b)	1	..	0.005	2.53	2.54			
39	16(a)	1	..	0.008	..	0.007			
40	30	3	..	1.35	..	1.37	0.21	..	0.20
41	30	3	..	1.35	..	1.36	0.21	..	0.20
42	30	3	..	1.35	..	1.36	0.21	..	0.20

The results given in Table VII were obtained by using the procedure given above. Besides the standards issued by the Bureau of Standards, a standard prepared in this laboratory, having a chromium content of 1.46 per cent, was analyzed with and without varying amounts of added chromium, manganese and vanadium.

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POMACE WINES: THEIR COMPOSITION AND DETECTION[1]

By JOHN R. EOFF, JR.
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Desiring some information on the composition of various types of American wines, the laboratory of the Internal Revenue Bureau undertook in 1913–14 the collection and analysis of a large number of these wines, and it particularly desired to ascertain the composition of the so-called pomace wines, at that time being rather extensively manufactured in certain sec-

tions of the East. The result of the investigation of the pomace wines has been very gratifying and it has enabled us to recognize this product with assurance. Having received from various sources urgent and repeated requests for the information obtained from this work the author feels this demand a sufficient reason for its publication. For the benefit of those who are not acquainted with the manufacture of pomace wines a brief outline of the processes used is included in this paper with the hope that it will aid in interpreting the results.

I—GRAPE POMACE

For the purposes of this article, grape pomace may be defined as the residue of the grape after the juice has been partially or thoroughly removed, before or after fermentation. Pomace may be divided into two main divisions—white and red.

(a) WHITE POMACE—This pomace is almost invariably that from the Catawba grape. The grapes are ground and the juice pressed from the skins, seeds and pulp, the juice so obtained being fermented separately. The residue (pomace) can never be pressed entirely free of juice and often contains as much as 8 per cent sugar. Sometimes the pomace is immediately used for pomace wine, but oftener it is packed tight into barrels and allowed there to ferment. The length of time the pomace is kept in barrels before use varies from a few weeks to a year or more.

(b) RED POMACE—The principal grape from which this pomace is derived is the Concord. To a lesser extent Concord and Ives mixed furnish this type of pomace, and though in this investigation there is no record of them, the writer has seen used the heavier colored varieties such as Norton and Clinton. The red pomace is obtained in three ways: (1) In making red wines the grapes are ground and the whole mass—juice, skins, pulp and seeds—is allowed to ferment, and at the proper stage the new wine is pressed off and the pomace packed in barrels; (2) instead of pressing off the wine the pomace is sometimes allowed to drain and, without pressing, immediately used for pomace wine: this procedure is infrequent; (3) in the manufacture of red grape juice the fruit is crushed, the mass heated to about 150° F., and then pressed.

II—POMACE WINES

For the purposes of this article, pomace wine may be defined as the product obtained by the alcoholic fermentation of sugar solution upon grape pomace, accompanied by the usual cellar treatment. The Commissioner of Internal Revenue[1] has held that the manufacture of pomace wine is prohibited by Section 3282, U. S. Revised Statutes, "except in a building or on premises of a distillery duly authorized according to law."

GENERAL METHOD OF MANUFACTURE—A quantity of pomace ranging from 650 to 1500 lbs., according to quality of pomace and product desired, is placed in open fermentation vats of sufficient capacity, and upon this are run 700 to 800 gals. of "sugar water," the mash being yeasted or not, as conditions require. The "sugar water" is prepared from cane or corn sugar

and contains in the neighborhood of 20 per cent sugar. Ammonium chloride is often added to the mash to facilitate fermentation, especially when old, dry pomace is used. The time allowed for the fermentation to proceed on the pomace varies from 4 to 16 days, after which the wine is drawn off and, if the fermentation is not complete, allowed to finish in closed casks. The wine is then almost immediately clarified and placed on the market, often blended with other wines, or it is aged in cellars for varying periods. The red pomace wines are often artificially colored, placing malvae flowers in the fermentation vats being a common method of coloring.

III—COLLECTION OF SAMPLES

In order that the investigation might deal with a product made in a manner identical with that found upon the market, an officer of the Internal Revenue Bureau was detailed to collect samples of pomace wines from three wine cellars engaged in the commercial manufacture of this article. He was instructed

V—DEDUCTIONS

In the following discussion it is presumed that an analyst desiring to establish pomace origin in a wine is sufficiently familiar with wines and wine analysis to enable him to recognize to what general type his sample belongs, and the characteristics of this type, for without such knowledge it were a hopeless undertaking to attempt to arrive at an intelligent conclusion. To illustrate this, unfortified pure dry Eastern wines will rarely contain less than 0.2 g. total tartaric acid in 100 cc., and in any wine which contains a less amount of total tartaric acid and which belongs to this general group one may be suspicious of pomace origin. There is another class of dry, or practically dry, wines whose tartaric acid content is normally about 0.1 g. in 100 cc., or less—the dry sherries. A novice might confuse the two; one with experience, never.

NITROGEN—Should the analysis show a figure for this element below 10 mg. per 100 cc., there is strong indication of pomace origin, and should the figure

TABLE I

Lab. No.	Collection No.	Samples Taken	Analyzed	Wine Cellar No.	Kind of Sugar	Sugar Solution Gallons	Degrees Balling	Temp. °F.	Amount Pomace Lbs.	Condition of Pomace	Days on Pomace	NH₄Cl Oz.	Kind of Pomace
WHITE POMACE WINES													
61869	3	10/28/13	1/5/14	1	Cerelose	800	19.4	92	1021	Fresh	6	None	Catawba
61870	4	10/28/13	1/5/14	1	Cerelose	1600	19.3	91	2010	Fresh	6	None	Catawba
61871	5	11/1/13	1/5/14	1	Cerelose	1600	19.0	92	2134	Fresh	4	None	Catawba
61872	6	11/6/13	1/5/14	1	Cerelose	800	19.2	86	920	Fresh	5	None	Catawba
61877	11	11/12/13	1/5/14	1·	Cerelose	1600	19.4	70	1896	Fresh	5	None	Catawba
61880	14	11/22/13	1/5/14	1	Cane	800	18.7	90	951	Fresh	7	None	Catawba
61884	18	12/3/13	1/14/14	1	Cane	800	18.9	86	953	Fresh	7	None	Catawba
61873	7	11/3/13	1/5/14	2	Cane	700	19.3	65	693	Old, dry, pale	12	About 30	Catawba
61875	9	11/12/13	1/5/14	2	Cane	700	19.3	65	693	Old, dry, pale	12	About 30	Catawba
61876	10	11/13/13	1/5/14	2	Cane	800	19.4	70	1056	Old, dry, pale	14	About 30	Catawba
61879	13	11/22/13	1/5/14	2	Cane	700	19.5	70	781	Old, dry, pale	14	About 30	Catawba
61883	17	12/1/13	1/14/14	2	Cane	700	19.2	70	700	Old, dry, pale	14	About 30	Catawba
61885	19	12/3/13	1/14/14	2	Cane	700	19.2	70	700	Old, dry, pale	14	About 30	Catawba
61887	21	12/8/13	1/14/14	2	Cane	700	19.3	75	728	Fresh; poor color	16	28	Catawba
63032	32	12/17/13	2/20/14	2	Cane	800	19.2	70	874	Fair	13	32	Catawba
63035	35	12/27/13	2/20/14	2	Cane	800	19.2	70	1052	Old, dry, pale	15	32	Catawba
63039	39	1/8/14	2/20/14	2	Cane	700	19.2	73	843	Old, dry, poor	13	28	Catawba
61882	16	11/29/13	1/14/14	3	Cerelose	800	19.0	72	641	Moist, fair	8	24	Catawba
61890	24	12/8/13	1/14/14	3	Cerelose	800	24.2	72	938	Moist, fair	14	24	Catawba
61894	28	12/12/13	1/14/14	3	Cerelose	800	19.2	70	665	Moist, fair	8	24	Catawba
63031	31	12/13/13	2/20/14	3	Cerelose	700	19.2	70	1270	Moist, fair	5	24	Catawba
63034	34	12/22/13	2/20/14	3	Cerelose	800	19.0	70	705	Moist, fair	10	24	Catwaba
63038	38	1/6/14	2/20/14	3	Cerelose	800	24.6	70	691	Moist, fair	13	24	Catawba
RED POMACE WINES													
61886	20	12/8/13	1/14/14	1	Cane	800	18.8	90	1490	Good	6	None	Concord Grape Juice
61896	30	12/13/13	1/14/14	1	Cane	800	18.7	80	1508	Good	5	None	Concord Grape Juice
63033	33	12/22/13	2/20/14	1	Cane	800	18.7	82	1528	Good	5	None	Concord Grape Juice
61874	8	11/8/13	1/5/14	2	Cane	675	20.0	70	1078	Dry; poor color	12	About 30	Concord and Ives
61878	12	11/19/13	1/5/14	2	Cane	800	19.4	70	1440	Dry; poor color	14	About 30	Concord
61891	25	12/9/13	1/14/14	2	Cane	700	19.2	70	886	Fresh; poor color	14	28	Concord
61895	29	12/13/13	1/14/14	2	Cane	700	19.5	70	904	New, dry, poor color	14	28	Concord and Ives
63037	37	12/30/13	2/20/14	2	Cane	800	19.3	70	1201	Dry; poor color	13	32	Concord and Ives
63036	36	12/29/13	2/20/14	3	Cerelose	800	19.2	70	1023	Fair; part wet; part dry	11	24	Concord and Ives

to observe personally the various steps in the manufacture and to make full records of them. This he has done most painstakingly and thoroughly, and through the courtesy of the wine makers, has collected many details. For the purposes of this Bureau it was of especial importance that the wine should be collected at the time it was drawn from the pomace, so most of the samples were secured as that stage. In case of fermentations not having been completed, the samples were to be sterilized directly after bottling. For wines of this character the conclusions drawn from the following analyses will hold for older wines of the same type.

IV—HISTORY AND ANALYSES OF POMACE WINES

In Table I is included a brief history of the pomace wines collected, and the analyses of them appear in Table II. For comparative purposes the latter table also contains the analyses of two pure Catawba wines made in the fall of 1913.

fall to 5 or below it is almost a foregone conclusion that one is dealing with a pomace wine. Nitrogen in the form of ammonia or nitrate has never been observed in any wine analyzed by the author.

CHLORINE—This constituent is of peculiar interest. Its determination not only aids in detecting spurious wines, but often furnishes evidence of their manner of manufacture. Natural wines are invariably low in chlorine content, the great majority containing less than 5 mg. in 100 cc. Some California wines have been known to exceed double this amount, but as a general rule any chlorine content above 10 mg. may be viewed with suspicion. The excess chlorine may be due to either of two causes, or to both: (1) the use of ammonium chloride as a fermentation accelerator; (2) the use of corn sugar solution in the manufacture of the wine. Since it is rarely the custom to add ammonium chloride to a normal wine fermentation, its presence not being necessary, one is justified in con-

TABLE II—ANALYSES OF POMACE WINES WITH ANALYSES OF TWO PURE WINES FOR COMPARISON

Grams per 100 cc.

Laboratory No.	Alcohol Vol. %	Extract	Nonsugar Extract	Reducing Sugar as Invert	Glycerol	Acidity: Total as Tartaric	Acidity: Volatile as Acetic	Acidity: Fixed as Tartaric	Tartaric Acid: Total	Tartaric Acid: Free	Tartaric Acid: Cream Tartar	Tartaric Acid: Combined (a)	Tannin and Coloring Matter	Pentosans	Volatile Esters	Ash	P_2O_5 Total	P_2O_5 Sol.	P_2O_5 Insol.	N	Cl	K_2O	Na_2O	CaO	MgO	SO_3	Alkalinity Total	Alkalinity H_2O-Soluble	Alkalinity H_2O-Insoluble	Polarization V. 100/100 200 mm. 20°C.	% P_2O_5 in Ash	Specific Gravity 15.6°C.
WHITE POMACE WINES																																
61869	9.37	6.56	2.73	3.83	0.517	0.435	0.032	0.394	0.237	0.00	0.12	0.14	0.028	....	....	0.288	0.0114	0.0014	0.0100	....	0.0094	0.0584	0.0268	0.0210	0.0094	....	16.8	10.4	6.4	+14.7	4.0	1.0127
61870	8.94	6.67	2.65	4.02	0.491	0.443	0.032	0.402	0.239	0.00	0.13	0.13	0.028	....	....	0.276	0.0096	Trace	0.0096	....	0.0094	0.0585	0.0236	0.0184	0.0093	0.0270	17.2	7.0	10.2	+14.8	3.5	1.0136
91871	8.04	8.40	2.58	5.82	0.465	0.409	0.031	0.370	0.230	0.00	0.14	0.12	0.033	....	0.023	0.250	0.0080	Trace	0.0080	....	0.0093	0.0902	0.0154	0.0110	0.0109	0.0232	7.6	3.6	4.0	−8.8	5.5	1.0214
91872	8.40	6.80	2.36	4.44	0.528	0.401	0.031	0.362	0.230	0.00	0.16	0.09	0.033	....	....	0.256	0.0078	Trace	0.0078	....	0.0096	0.0817	0.0126	0.0072	0.0061	0.0021	16.4	8.6	7.8	+17.9	3.2	1.0148
61877	8.66	7.58	2.55	5.03	0.401	0.377	0.029	0.341	0.206	0.00	0.18	0.06	0.026	....	....	0.258	0.0076	Trace	0.0076	....	0.0087	0.0852	0.0171	0.0160	0.0120	0.0030	15.8	9.4	6.4	+19.7	2.9	1.0175
61880	8.95	7.13	1.61	5.52	0.369	0.386	0.041	0.335	0.216	0.07	0.07	0.09	0.038	....	....	0.110	0.0076	0.0018	0.0058	....	0.0160	0.0698	0.0160	0.0126	0.0080	0.0171	13.2	8.8	4.4	−17.8	6.9	1.0154
61884	9.95	5.97	1.62	4.35	0.438	0.386	0.056	0.315	0.185	0.02	0.17	0.07	0.033	....	....	0.103	0.0060	0.0008	0.0052	....	0.0032	0.0537	0.0032	0.0080	0.0056	....	10.8	6.0	4.8	−17.1	7.7	1.0098
61873	12.38	1.39	1.32	0.07	0.600	0.315	0.076	0.220	0.185	0.07	0.07	0.11	0.033	....	....	0.147	0.0060	0.0008	0.0050	....	0.0178	0.0791	0.0170	0.0107	0.0056	0.0178	10.8	6.0	4.8	−0.8	5.8	0.9893
61875	12.45	1.57	1.51	0.06	0.602	0.278	0.070	0.220	0.185	0.01	0.17	0.00	0.028	....	....	0.160	0.0116	0.0032	0.0084	....	0.0056	0.0507	0.0039	0.0112	0.0056	0.0065	15.0	9.6	5.4	+0.3	7.2	0.9900
61876	12.30	1.57	1.50	0.07	0.604	0.342	0.083	0.238	0.108	0.01	0.14	0.09	0.040	....	....	0.180	0.0098	0.0080	0.0018	....	0.0060	0.0887	0.0046	0.0060	0.0070	0.0178	19.0	10.8	8.2	+0.1	9.8	0.9901
61879	11.87	3.07	1.45	1.62	0.560	0.383	0.071	0.294	0.191	0.00	0.06	0.06	0.048	0.060	....	0.111	0.0026	0.0011	0.0026	....	0.0034	0.1013	0.0038	0.0100	0.0058	0.0031	13.6	9.0	4.6	−7.8	2.3	0.9964
61883	12.18	1.70	1.53	0.17	0.522	0.469	0.059	0.395	0.285	0.02	0.17	0.00	0.060	0.024	....	0.150	0.0122	0.0042	0.0080	....	0.0058	0.0826	0.0032	0.0108	0.0101	0.0025	18.0	12.6	5.4	+0.1	8.0	0.9908
61885(b)	10.86	11.47	1.65	0.73(c)	0.474	0.522	0.115	0.306	0.287	0.08	0.24	0.17	0.024	0.060	....	0.224	0.0176	0.0080	0.0098	....	0.0192	0.0869	0.0388	0.0084	0.0106	0.0031	28.0	22.0	6.0	+29.3	6.5	1.0299
61887	11.32	3.26	1.68	1.58	0.587	0.521	0.132	0.356	0.273	0.05	0.20	0.06	0.058	....	0.058	0.156	0.0050	0.0022	0.0022	....	0.0027	0.0771	0.0031	0.0098	0.0058	0.0027	15.2	10.4	4.8	−7.4	8.1	0.9977
63032	12.16	1.99	1.46	0.53	0.522	0.431	0.074	0.338	0.222	0.07	0.07	0.17	0.093	....	0.022	0.130	0.0050	0.0028	0.0046	....	0.0028	0.0690	0.0040	0.0102	0.0056	0.0015	16.0	10.4	5.6	−2.5	3.8	0.9919
63035	11.35	2.40	1.82	0.58	0.564	0.604	0.160	0.404	0.278	0.20	0.20	0.24	0.033	....	0.030	0.173	0.0176	0.0050	0.0094	....	0.0025	0.0265	0.0051	0.0108	0.0102	0.0094	16.0	10.4	5.6	−2.8	8.0	0.9944
63039	11.86	1.76	1.55	0.21	0.604	0.506	0.110	0.368	0.266	0.07	0.07	0.26	0.009	....	0.033	0.154	0.0138	0.0052	0.0098	....	0.0108	0.0815	0.0051	0.0094	0.0106	0.0125	15.4	10.6	4.8	−0.8	8.9	0.9913
61882	10.58	3.23	2.45	0.78	0.598	0.360	0.060	0.285	0.129	0.00	0.07	0.20	0.031	....	0.052	0.242	0.0106	0.0008	0.0094	....	0.0105	0.0966	0.0031	0.0106	0.0102	0.0109	10.4	3.6	6.8	+8.5	4.4	0.9984
61890	13.08	4.37	3.20	1.17	0.782	0.469	0.072	0.379	0.198	0.02	0.08	0.12	0.062	....	0.056	0.309	0.0144	0.0008	0.0144	....	0.0170	0.0836	0.0693	0.0150	0.0109	0.0190	12.2	4.2	8.0	+11.3	4.6	1.0001
61894	9.75	2.97	2.20	0.77	0.608	0.375	0.037	0.328	0.161	0.02	0.10	0.08	0.055	....	0.056	0.228	0.0098	0.0004	0.0094	....	0.0146	0.0923	0.0670	0.0150	0.0146	0.0232	11.0	4.2	6.8	+9.6	4.3	0.9984
63031	10.97	3.10	2.50	0.60	0.637	0.398	0.079	0.299	0.155	0.08	0.08	0.09	0.045	....	0.044	0.245	0.0101	0.0003	0.0101	....	0.0101	0.0506	0.0668	0.0150	0.0101	0.0154	11.6	4.2	7.4	+7.1	4.1	0.9975
63034	11.46	3.26	2.50	0.76	0.615	0.424	0.056	0.354	0.195	0.01	0.09	0.09	0.029	....	0.030	0.242	0.0098	0.0004	0.0094	....	0.0080	0.0593	0.0590	0.0162	0.0080	0.0167	12.2	5.0	7.2	+8.4	4.3	0.9975
63038	14.11	4.44	3.00	1.44	0.775	0.450	0.055	0.381	0.225	0.03	0.11	0.11	0.043	....	0.072	0.281	0.0168	0.0010	0.0158	....	0.0104	0.0973	0.0750	0.0152	0.0104	0.0166	13.0	5.6	7.4	+11.5	6.0	0.9993
RED POMACE WINES																																
61886	9.28	7.63	2.01	5.62	0.452	0.420	0.053	0.354	0.186	0.04	0.10	0.07	0.114	....	....	0.118	0.0105	0.0007	0.0098	....	0.0036	0.0501	0.0043	0.0138	0.0080	0.0255	9.8	5.2	4.6	−18.0	9.0	1.0169
61896	10.88	3.64	1.81	1.83	0.460	0.450	0.038	0.402	0.188	0.04	0.09	0.07	0.119	....	....	0.114	0.0090	0.0024	0.0066	....	0.0036	0.0462	0.0037	0.0124	0.0074	0.0270	9.6	5.0	4.6	−6.7	8.0	0.9997
63033	10.66	4.00	1.69	2.31	0.455	0.435	0.040	0.385	0.188	0.05	0.09	0.06	0.107	....	0.023	0.105	0.0058	0.0004	0.0054	....	0.0025	0.0470	0.0031	0.0110	0.0109	0.0232	7.6	3.6	4.0	−4.8	5.5	1.0014
61874	12.41	2.71	1.76	0.97	0.650	0.518	0.088	0.408	0.263	0.01	0.23	0.07	0.040	....	....	0.134	0.0050	0.0010	0.0040	....	0.0027	0.0893	0.0027	0.0072	0.0061	0.0021	12.4	7.6	4.8	−4.4	3.7	0.9944
61878	12.11	2.12	1.76	0.36	0.657	0.536	0.118	0.388	0.279	0.07	0.26	0.048	0.033	....	0.050	0.166	0.0120	0.0040	0.0080	....	0.0039	0.0992	0.0040	0.0114	0.0072	0.0030	17.2	12.4	4.8	−1.0	7.2	0.9924
61891	11.92	1.86	1.71	0.15	0.528	0.488	0.115	0.344	0.272	0.06	0.26	0.048	0.048	....	....	0.158	0.0082	0.0024	0.0058	....	0.0042	0.0791	0.0047	0.0107	0.0080	0.0026	19.2	13.8	5.4	−1.0	5.2	0.9916
61895	11.63	3.10	1.71	1.39	0.520	0.529	0.110	0.391	0.281	0.02	0.23	0.048	0.036	....	0.053	0.158	0.0080	0.0016	0.0064	....	0.0058	0.0822	0.0040	0.0112	0.0107	0.0110	17.6	12.4	5.2	−6.3	5.1	0.9968
63037	12.50	1.99	1.74	0.25	0.572	0.480	0.071	0.323	0.201	0.02	0.30	0.23	0.036	....	0.028	0.201	0.0116	0.0028	0.0088	....	0.0241	0.0791	0.0076	0.0110	0.0080	0.0017	22.8	16.0	6.8	−0.8	5.8	0.9915
63036	11.15	3.12	2.45	0.67	0.594	0.398	0.080	0.298	0.225	0.01	0.04	0.11	0.041	....	0.079	0.237	0.0179	0.0003	0.0176	....	0.0045	0.0846	0.0601	0.0192	0.0116	0.0154	13.0	5.6	7.4	+6.0	7.6	0.9974
PURE CATAWBA WINES																																
63041	11.60	3.23	1.98	1.25	0.516	0.724	0.065	0.643	0.444	0.21	0.15	0.11	....	0.064	0.033	0.125	0.0154	0.0040	0.0114	0.0246	0.0033	0.0540	0.0051	0.0131	0.0067	....	15.6	8.2	7.4	−6.5	12.3	0.9973
63042	11.31	3.31	2.05	1.26	0.505	0.735	0.050	0.672	0.446	0.23	0.13	0.11	....	....	0.021	0.121	0.0192	0.0060	0.0132	0.0213	0.0028	0.0531	0.0055	0.0151	0.0122	....	14.4	7.0	7.4	+6.9	15.8	0.9979

(a) Combined with alkaline earths.   (b) A clarified, finished pomace wine, ready for marketing.   (c) Sucrose = 9.09.

cluding that it is added in case of poor fermentation, such as is the case with sugar solution over old, dry pomace. In this event the chlorine will be close to 20–30 mg. per 100 cc. When the chlorine figure approaches 100 mg. per 100 cc., with a corresponding increase in the content of soda, one has practical proof that corn sugar has been used in the preparation of the wine and, with the aid of other determinations, that it is pomace wine. Polarization at 87° C. after inversion is useful in substantiating the presence of corn sugar or corn-sugar residue. The practice of using corn sugar for sweetening or gallizing wines, once considerably in vogue, has greatly decreased and it may often be disregarded.

SODA—The ranges and interpretation of this figure are similar to those for chlorine.

TOTAL TARTARIC ACID—Pomace wines are low in total tartaric acid as compared with wines with which they are likely to become confused. A figure of 20 cg. per 100 cc. or under is suspicious, and any unfortified wine falling below 10 cg. of this constituent may usually be deemed spurious. The author has been informed of some supposedly pure unfortified European wines whose tartaric acid content is below 10 cg. per 100 cc., but the history of the wines proved to be incomplete.

FIXED ACID AS TARTARIC—Any figure below 50 cg. per 100 cc. is ground for suspecting pomace origin.

ASH—For white dry wines an ash of 20 cgs. per 100 cc. or over indicates pomace. For red wines this figure is of but general value.

ALKALINITY OF ASH—The alkalinity of the water-soluble ash of pomace wines often falls as low as 8 cc. $N/10$ HCl per 100 cc. wine or under, which is unusual for American wines. When the alkalinity of the water-insoluble ash exceeds the alkalinity of the water-soluble ash the fact is very characteristic of pomace wines.

NONSUGAR EXTRACT—Any figure below 1.5 g. per 100 cc. for white wines and 2.0 g. per 100 cc. for red wines is suspicious.

PENTOSANS—For white wines, any figure below 50 mg. per 100 cc., and for red wines, below 100 mg. per 100 cc., is suspicious.

PER CENT P_2O_5 IN ASH—Having this figure below 10 is almost a constant property of pomace wines.

NEUTRALIZATION TEST—This test, which is communicated by Dr. B. G. Hartmann, of the Bureau of Chemistry, has been found very valuable as an aid in the detection of white pomace wines: "Straight wines or gallized wines, when neutralized with sodium hydroxide, darken slightly and acquire a brownish pink tint; pomace wines acquire a brownish color and very often contain a sediment after standing."

ADJUSTMENT OF ASH AND NONSUGAR EXTRACT—In cases where corn sugar is indicated in the manufacture of the suspected wine it is advisable to adjust the figures for ash and nonsugar extract to those of true grape material, i. e., make allowance for the effect of the corn-sugar residue on these constituents. The corrections to be made can be determined by referring to the analyses of pomace wines given above. Necessarily these corrections will be more or less arbitrary, but the results obtained will be of value in differentiating between pomace wines and wines gallized with corn sugar.

The following determinations are of secondary importance in judging pomace origin, but they are of value in sustaining interpretations of the cardinal determinations given above:

FREE TARTARIC ACID—Pomace wines contain little or no free tartaric acid.

CREAM OF TARTAR—Below 15 cg. per 100 cc. is suspicious.

SULFURIC ACID (SO_3)—Below 5 mg. per 100 cc. is suspicious.

MAGNESIA—For white wines, any striking variation from 10 mg. per 100 cc. is suspicious.

In searching for pomace origin in wines the determination of any single constituent never closes the problem, and it is equally true that few, if any, pomace wines will show all the peculiarities of this product. The organoleptic examination is of great aid in making the final decision and the color of the wine is often helpful.

In conclusion, the author wishes to express his sincere appreciation to Mr. D. W. Campbell, of Sandusky, Ohio, for the untiring interest shown in the collection of the wines represented in the above analyses. Thankful acknowledgment is also extended to several colleagues in the Internal Revenue Laboratory for timely aid in the analysis of the wines: to Mr. W. V. Linder for determining nitrogen, volatile acids and pentosans in Samples 61882–96; Mr. J. M. Doran, total acid and tannin, Samples 61882–96, and lime and magnesia, Samples 61869–80; Mr. P. Valaer, specific gravity and tannin, Samples 63031–42.

CHEMICAL COMPOSITION OF ALFALFA AS AFFECTED BY STAGE OF MATURITY, MECHANICAL LOSSES, AND CONDITION OF DRYING

By C. O. SWANSON AND W. L. LATSHAW
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This paper is a partial report on the chemical work done on alfalfa cut at the time of budding, one-tenth bloom, full bloom, and at seed formation, in an experiment carried on jointly by the departments of chemistry, agronomy, and animal husbandry at the Kansas Experiment Station.[1] This paper discusses one phase of the work, viz., the chemical composition as affected by variations in maturing and curing.

The alfalfa was cut from duplicate and triplicate 0.1 acre plots. As soon as cut, a sample was taken from the green material and spread in an attic room to dry. When the cut hay in the field was dry enough to stack, it also was sampled: this sample was weighed and taken to the same attic room to dry thoroughly. After the green sample was partially wilted a small sub-sample was taken and the relative amount of

leaves and steam was determined on an air-dry basis. Samples of alfalfa cut at the same stages of maturity were also obtained at the time of feeding. These five different kinds of samples will be designated as: *sampled when cut, sampled when stacked, leaves from the material sampled green, stems from the material sampled green,* and *sampled when fed.*

This experiment has been carried on for two years. The summer of 1914 was moderately dry, while 1915 was unusually wet. By analyzing the samples from each cutting, data were obtained in regard to the change in composition during each of these seasons. It was found, however, that the stage of maturity so strongly influenced the composition that it more than offset any effect due to the time of season. Therefore, only the averages calculated on a 10 per cent moisture basis, for each stage of maturity, are given in the following tables. There were from 4 to 6 cuttings on the first three stages, and 3 cuttings on the last stage.

TABLE I—COMPOSITION OF ALFALFA CUT AT DIFFERENT STAGES OF MATURITY—SAMPLED WHEN CUT

YEAR	PORTION ANALYZED	STAGE OF MATURITY	Ash	Crude Protein	Crude Fiber	N-free Extract	Ether Extract
1914	Whole Plant	Bud	10.53	19.65	22.50	35.06	2.36
		1/10 Bloom	9.59	18.38	23.58	35.41	2.93
		Full Bloom	8.79	16.30	25.01	36.07	3.77
		Seed	7.54	14.97	26.53	37.37	3.54
	Leaves	Bud	10.78	26.17	13.64	36.01	3.27
		1/10 Bloom	10.52	26.16	14.06	37.40	4.06
		Full Bloom	9.11	22.10	13.66	39.32	5.72
		Seed	8.70	21.25	14.55	39.66	5.48
	Stems	Bud	8.78	12.57	33.54	33.92	1.23
		1/10 Bloom	7.97	10.63	35.12	32.97	1.33
		Full Bloom	7.62	9.72	36.33	35.83	1.22
		Seed	7.12	10.22	36.41	34.86	1.39
1915	Whole Plant	Bud	10.24	19.94	26.86	31.29	1.67
		1/10 Bloom	9.18	16.12	30.80	32.08	1.82
		Full Bloom	8.76	15.70	30.90	32.62	2.03
		Seed	8.08	14.48	31.56	33.95	1.93
	Leaves	Bud	10.63	27.30	15.58	32.91	3.56
		1/10 Bloom	10.64	24.60	18.04	33.44	3.29
		Full Bloom	10.01	22.70	15.91	37.78	3.60
		Seed	9.49	22.21	17.28	37.91	3.11
	Stems	Bud	8.77	13.04	36.62	30.58	0.99
		1/10 Bloom	6.74	10.80	40.60	38.85	0.96
		Full Bloom	6.38	9.83	43.37	29.15	0.97
		Seed	7.07	8.91	43.21	29.79	1.02

The percentage of feeding constituents in the whole plant, leaves, and stems for the two separate years is given in Table I. The ash and protein decrease regularly as the plant matures; crude fiber and nitrogen-free extract increase. Because of the large amount of chlorophyll in the ether extract, the figures do not give any significant data. The ash content of the leaves is uniformly greater than of the stems. The greatest difference between the leaves and stems, however, is in the content of protein and crude fiber. The leaves contain from 2 to $2^1/_2$ times as much protein as the stems, while the stems contain over $2^1/_2$ times as much crude fiber as the leaves. The samples of 1915, in comparison with those of 1914, have a slightly lower ash content, but a considerably higher content of crude fiber. These differences are most pronounced in the stems, and are probably due to the larger growth in 1915.

In 1914 alfalfa was cut from a larger field at the same stages of maturity. This was cured, baled, and stored in a barn, being sampled at the time of feeding. A small amount was taken from each bale on opening, and placed in large bags. In this way three to five composites were obtained from each stage of cutting. These composites were analyzed separately, and the average figures are given in Table II. In 1915, no samples were taken at the time of feeding, but instead the average figures for the samples obtained at the time of stacking are given in Table II. A comparison of the figures in Table II with those in Table I shows that there is less ash and crude protein, and more crude

TABLE II—COMPOSITION OF ALFALFA CUT AT DIFFERENT STAGES OF MATURITY—SAMPLED AS HAY

YEAR	STAGE OF MATURITY	Ash	Crude Protein	Crude Fiber	N-free Extract	Ether Extract
1914, when fed	Bud	9.47	18.27	24.30	35.98	2.67
	1/10 Bloom	8.56	15.96	24.34	38.19	2.86
	Full Bloom	7.53	14.62	27.48	37.70	2.64
	Seed	7.28	13.38	28.14	38.84	2.36
1915, when stacked	Bud	10.21	18.01	26.17	34.00	1.61
	1/10 Bloom	8.76	15.46	31.53	32.72	1.53
	Full Bloom	8.25	14.76	31.64	33.56	1.79
	Seed	7.56	13.24	34.87	32.79	1.54

fiber and nitrogen-free extract in the alfalfa sampled as hay. This difference is largely due to mechanical loss of leaves.

The relative amount of leaves and stems in the material sampled when cut was determined by taking a handful selected from different parts of the large sample as soon as the alfalfa was wilted, and by separating this sample into leaves and stems. These were allowed to dry under the same conditions as the rest of the sample, and the relative percentage by weight was determined. The averages of all the cuttings are given in Table III. In 1914 there was a larger percentage of leaves in the first three stages, but in 1915 the percentage of stems was larger in all of the stages. In both years the proportion of leaves decreased and the proportion of stems increased as the plant matured.

TABLE III—RELATIVE PER CENT OF LEAVES AND STEMS IN ALFALFA SAMPLED WHEN CUT

YEAR	Bud Leaves	Bud Stems	1/10 Bloom Leaves	1/10 Bloom Stems	Full Bloom Leaves	Full Bloom Stems	Seed Leaves	Seed Stems
1914	57.57	42.43	56.40	43.61	51.54	48.22	43.50	56.51
1915	47.89	52.11	41.54	58.46	37.65	62.35	36.58	63.42

The exact amount of leaves lost in the process of haymaking is impracticable to determine directly. However the approximate amount can be calculated if the chemical composition of alfalfa leaves and of alfalfa hay containing all of the leaves is known, as well as the composition of the hay when there is a loss of leaves due to handling. The errors involved depend on the correctness of the assumptions made. Since the greatest difference in composition between the leaves and the whole plant is in protein, the figures for protein are used in the calculations. It is assumed that there is no loss of total protein in the process of curing, and that change in composition is solely due to mechanical loss of leaves which contain a large amount of protein. One source of error is in the determination of the relative amount of leaves and stems. The amount of material used in this determination is small in comparison with the total amount of hay under consideration. As this determination was made on a number of samples, however, the errors were largely eliminated in the average. The method of calculation was as follows:

Let x = number of pounds of leaves lost.

    a = per cent of protein in alfalfa sampled when cut.

    b = per cent of protein in alfalfa sampled as hay.

    c = per cent of protein in the leaves.

Make the calculation on the basis of 100 lbs. air-dry

hay. Then $100 - x =$ weight of hay after loss of leaves. The formula for calculation would then be: $.b(100 - x) + .cx = a$. Substituting the known values for a, b and c, as obtained in the chemical analysis of any of the samples under consideration, as for bud stage samples of 1915, where $a = 19.94$, $b = 18.01$, $c = 27.30$, the equation becomes $0.1801 (100 - x) + 0.2730 x = 19.94$ (per cent loss of leaves by weight). $x = 20.77$. Using this method of calculation, and the figures for the percentage amount of leaves given in Table III, Table IV is produced presenting the percentage loss of leaves and crop.

TABLE IV—PERCENTAGE LOSSES OF LEAVES AND CROP

MATURITY	Bud		1/10 Bloom		Full Bloom		Seed	
YEAR	Leaves	Crop	Leaves	Crop	Leaves	Crop	Leaves	Crop
1914......	25.04	14.35	19.67	11.00	23.85	12.72	25.77	11.67
1915......	20.77	9.95	7.22	3.00	11.84	4.46	13.82	5.06

The figures in Table IV show that the loss of leaves is an important matter. The relative amount of leaves was larger in 1914,[1] the drier year, and the loss of leaves was also greater than in 1915.

The total amount of nutrients produced per acre was one factor to be determined in the present experiment. The pounds of nutrients per acre in the total alfalfa crop as grown, as well as in the leaves and stems, may be obtained by using the data of yield per plot at time of stacking, as obtained by the agronomy department (see Table V), the figures for dry matter in the samples taken at this time, the figures in Table IV for loss in handling, and the figures for composition in Table I. In 1914 the largest amount of all nutrients was obtained in the bud stage. In 1915 the largest amounts were obtained at full bloom.

TABLE V—POUNDS OF NUTRIENTS PRODUCED PER ACRE IN THE TOTAL CROP, LEAVES AND STEMS, 10 PER CENT MOISTURE BASIS

YEAR		STAGE OF MATURITY	Ash	Crude Protein	Crude Fiber	N-free Extract	Ether Extract
1914	Whole Crop	Bud	817.01	1483.66	1815.57	2674.92	186.75
		1/10 Bloom	664.35	1235.92	1734.61	2399.51	251.00
		Full Bloom	534.61	967.17	1589.52	2167.56	218.50
		Seed	331.40	674.55	1310.24	1786.94	134.82
	Leaves	Bud	487.06	1145.44	582.41	1501.87	147.07
		1/10 Bloom	405.43	946.55	556.12	1422.32	167.22
		Full Bloom	281.07	693.07	429.39	1258.41	159.95
		Seed	182.21	423.12	286.00	804.76	93.93
	Stems	Bud	329.95	338.22	1233.16	1173.05	39.68
	(by difference)	1/10 Bloom	258.92	289.37	1178.49	977.19	83.78
		Full Bloom	253.54	274.10	1160.13	909.15	58.55
		Seed	149.19	251.43	1024.24	982.18	40.89
1915	Whole Crop	Bud	878.31	1710.30	2303.80	2683.76	143.25
		1/10 Bloom	1066.34	1767.20	3376.50	3516.62	199.52
		Full Bloom	1165.08	2088.10	4109.71	4338.46	269.99
		Seed	792.31	1419.88	3094.71	3329.07	189.25
	Leaves	Bud	436.63	1121.35	639.95	1391.65	107.21
		1/10 Bloom	484.55	1120.28	821.54	1522.86	149.80
		Full Bloom	501.25	1136.69	766.99	1891.82	180.29
		Seed	340.40	796.66	619.83	1359.82	111.55
	Stems	Bud	441.68	588.95	1663.85	1292.11	36.04
	(by difference)	1/10 Bloom	521.79	646.92	2554.96	1993.76	49.72
		Full Bloom	663.83	951.41	3313.02	2546.64	89.70
		Seed	451.91	623.22	2474.88	1969.25	77.70

The change in composition due to mechanical loss has been shown in the preceding paragraphs, but no attempt was made to determine the total loss of any chemical constituent. That there are losses of chemical constituents during the process of haymaking has been shown by Fleishmann,[2] these losses depending on the conditions of drying. He found that the greatest losses occur under unfavorable weather conditions and that the losses of chemical constituents fall most

heavily on the carbohydrates, and to a much less extent on the ether extract. Fleishmann found also that there were very important changes in the form of phosphorus and nitrogenous compounds. These changes, to an important extent, also occurred under favorable conditions of drying. Headden[1] found that great losses of nutrients, due to chemical and bacterial action, occurred under unfavorable drying conditions. Honcamp[2] also found that losses of chemical constituents occur to some extent even under favorable conditions of drying in the sun. He also found that the digestibility of hay depends to an important extent on the method of curing. This has been shown by other investigators.[3]

That the amount of pure protein as determined by Stutzer's method is decreased by slow drying was found in the present investigation. The nitrogen by Stutzer's method[4] was determined on all the samples taken at the time of cutting and at the time of stacking. The results are summarized in Table VI. The green alfalfa samples taken at the time of cutting were usually brought to the chemical laboratory in the evening. The temperature in the attic room where they were spread to dry was always high in the summer. Nevertheless the drying here was much slower than in the open field where there was a free movement of air, and usually sunshine.

TABLE VI—AVERAGE FIGURES SHOWING RELATIONS BETWEEN TOTAL AND PURE (STUTZER'S) PROTEIN IN ALFALFA SAMPLES WHEN CUT, SAMPLED AS HAY, AND IN THE LEAVES AND STEMS

MOISTURE-FREE FOR 1914

YEAR	ALFALFA SAMPLED	STAGE OF MATURITY	Percentages Total	Pure	Protein Diff.	Pure Protein in % of Total
1914	When Cut	Bud	22.27	15.96	6.31	71.96
		1/10 Bloom	20.62	15.33	5.08	74.55
		Full Bloom	18.34	14.77	3.58	80.55
		Seed	17.16	14.40	2.77	84.00
	As Hay	Bud	20.01	17.24	2.77	86.09
		1/10 Bloom	18.82	15.76	2.86	83.50
		Full Bloom	16.22	13.97	2.25	86.04
		Seed	14.71	12.50	2.21	84.95
	Leaves	Bud	28.55	21.63	6.93	75.79
		1/10 Bloom	26.94	21.04	5.90	78.21
		Full Bloom	25.21	20.42	4.79	80.98
		Seed	23.94	19.86	4.08	82.88
	Stems	Bud	14.12	9.56	4.56	68.62
		1/10 Bloom	12.09	8.68	3.40	73.08
		Full Bloom	11.25	8.34	2.92	74.24
		Seed	11.32	8.93	2.39	79.64

TEN PER CENT MOISTURE BASIS

1915	When Cut	Bud	19.90	12.83	7.07	64.41
		1/10 Bloom	16.73	11.28	5.46	66.55
		Full Bloom	15.38	10.89	4.49	70.79
		Seed	14.80	11.21	3.59	75.67
	As Hay	Bud	17.16	13.09	4.07	75.92
		1/10 Bloom	15.25	11.33	3.92	74.22
		Full Bloom	14.85	11.26	3.59	75.78
		Seed	13.79	10.42	3.37	75.55
	Leaves	Bud	26.59	19.23	7.76	72.30
		1/10 Bloom	24.50	18.18	6.34	74.17
		Full Bloom	22.58	17.09	5.49	75.20
		Seed	22.45	17.34	5.11	77.16
	Stems	Bud	12.81	7.29	5.52	56.86
		1/10 Bloom	10.91	6.33	4.58	58.05
		Full Bloom	9.68	6.11	3.57	63.12
		Seed	9.05	6.80	2.25	75.07

A study of the figures in Table VI brings out these facts:

1—The per cent of pure protein in the total protein is less in the samples cured in the shade than in those cured in the sun, or open field. The greatest difference occurs in the samples from the bud stage, and there is a gradual decrease to the seed stage, where

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[1] The figures for protein used in the calculations, 1914, are found in Table VI.

[2] Die Landw. Vers. Stat., 76, 237–447.

[1] Colorado Experiment Station, Bull. 110.

[2] Die Landw. Vers. Stat., 86, 215–275.

[3] Ibid., 75 (1911).

[4] Bureau of Chemistry, Bull. 107.

there is no significant difference; that is, the protein in the younger plants was more profoundly affected by this condition than that from the seed stage, which was very little changed.

2—The differences between total and pure protein are greater in the alfalfa cured in the shade than in that cured in the sun or open field; that is, the proportion of pure protein decreases during the process of drying. This is most noticeable in the earlier stages. The hay cured in the sun contains a larger per cent of pure protein than that cured in the shade.

In comparing the figures obtained on the samples for 1914 with those of 1915, important differences are noticed. The per cent of pure protein in the total protein is less in every instance in the samples of 1915, and differences between the alfalfa cured in the shade and that cured in the sun are less. The more unfavorable drying conditions of 1915, with the necessity of curing under cock covers, allowed a prolongation of the vital activity of the protoplasm, resulting in protein cleavage. The per cent of pure protein in the total protein is from 6 to 9 per cent less in the alfalfa dried in the shade in 1915 than in the same lot in 1914, while the 1915 samples dried in the open field show about 9 per cent less than the 1914 samples. The larger, heavier plants of 1915 took longer to dry. The stems, which dry the slowest, show the greatest differences in comparing the samples of the two years, and the leaves, which dry the fastest, show the least difference.

These facts point to a very profitable line of investigation in regard to the chemical changes which take place under different conditions of haymaking. These changes no doubt include not only the proteins, but also the carbohydrates, fats, and phosphorus compounds. The feeding value of hay is affected not only by mechanical losses due to handling, and the changes due to bacterial action, but also by chemical changes which are little known or noticed.

SUMMARY

I—The alfalfa cut in the bud stage had the largest ash and crude protein and the smallest crude fiber and nitrogen-free extract.

II—In each successive stage the crude fiber and nitrogen-free extract increases, and the crude protein and ash decrease. In pounds per ton the alfalfa cut in the earlier stages has more of crude protein and less of crude fiber.

III—The total amount of any or all nutrients produced per acre depends to a large extent on the yield, as shown by the fact that in 1914 the greatest amount of nutrients was obtained in the bud stage, while in 1915 the full bloom gave the greatest amount of total nutrients.

IV—The leaves and stems differ in content of ash, ether extract, and nitrogen-free extract, but the greatest difference is in the per cent of crude protein and crude fiber. The leaves contain over $2^1/_2$ times as much protein as the stems, while the stems contain over $2^1/_2$ times as much crude fiber as the leaves.

V—In harvesting and handling there is a large loss of leaves, which loss affects the composition of the hay in an increase of crude fiber and a decrease of crude protein.

VI—The alfalfa cured in the sun has a larger pure protein content as determined by Stutzer's method, than that cured in the shade. This difference is so great as to more than offset the influence of the loss of leaves. The differences in respect to pure protein content were most pronounced in the alfalfa cut in the earlier stages.

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THE VOLATILE OIL OF CALYCANTHUS OCCIDENTALIS

By CHARLES C. SCALIONE
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Calycanthus occidentalis, or *Butneria occidentalis*, also popularly known as spice-bush, belongs to the family *Calycanthaceae*, 4 or 5 species of which are found in the United States. The species named is limited to northern California and southern Oregon and is rarely found in large patches, but usually scattered along stream banks or river bottoms or on the lower slopes of hillsides. It is an unarmed shrub, which sometimes reaches a height of 10 ft. It bears an abundance of ovate to oblong lanceolate leaves, somewhat scabrous and of a bright green color, and dark red flowers from 1 to $1^1/_2$ in. long. The leaves, bark and wood are all strongly aromatic; the flowers less so.

Considerable interest has been attached to some of the other species of this genus on account of their physiological action.

An alkaloid was discovered in the seeds of *C. glaucus C. fertilis*, by R. C. Eccles,[1] in 1888, and named by him Calycanthine. This was confirmed by H. W. Wiley[2] in 1889. H. M. Gordin,[3] in a number of papers, has shown the presence of two alkaloids, calycanthine and isocalycanthine.

The volatile oil of Floridus has been studied by Miller[4] and his co-workers and found to consist of pinene, cineol, borneol, bornyl acetate, salicylic acid, possibly linalool and some other esters besides the bornyl acetate. The cineol predominated.

EXPERIMENTAL

Four hundred pounds of leaves and twigs from Oriental, California, that had been kindly supplied by officials of the United States Forest Service, at San Francisco, were submitted to steam distillation under a pressure of 4 lbs. The yield of oil obtained from the united distillates amounted to about 0.27 per cent of the weight of the original material.

In transit considerable decomposition of the leaves had taken place and the water distilling over with

[1] Eccles, *Proc. Am. Pharm. Assoc.*, **36**, 84 and 382.
[2] Wiley, *Am. Chem. J.*, **11**, 557.
[3] Gordin, *Proc. Am. Pharm. Assoc.*, **52**, 345; **53**, 224.
[4] Miller, *J. Am. Chem. Soc.*, **26**, 2182.

the oil was strongly alkaline. After removing the oil the alkaline distillates were treated with an excess of hydrochloric acid and evaporated to dryness on the water bath. The residual salts were found to have an odor very suggestive of aliphatic amines; they were crystallized from absolute alcohol and the chlorine determined in both the alcohol-soluble salt and the alcohol-insoluble salt. The former determination gave 37.48 per cent chlorine, which corresponds with the theoretical percentage of chlorine in trimethylamine hydrochloride (*i. e.*, 37.17 per cent). The alcohol-insoluble salt gave 66.18 per cent chlorine which corresponds with the percentage of chlorine in ammonium chloride (*i. e.*, 66.37 per cent). A distillation on some fresh leaves failed to show either of these constituents and whether the trimethylamine and ammonium hydroxide were due to the decomposition of the protein matter alone or whether they were derived from the protein material plus some easily decomposable alkaloid has not been definitely settled. Qualitative determinations with phosphomolybdic acid demonstrated the presence of basic bodies in the leaves. These bodies failed to respond to tests for tannins: they were probably alkaloidal in character. The total nitrogen content was found to be 3.75 per cent by weight of the dry leaf. This nitrogen content is abnormally high if due to protein material alone.

A second sample of the plant, which was grown in a garden at Berkeley and weighed some 30 lbs., was separated into twigs and leaves and the two portions distilled separately while still fresh. The yield of oil from the leaves was about 0.15 per cent and from the twigs 0.37 per cent.

PHYSICAL CONSTANTS OF THE OIL

The oil had a greenish yellow appearance, a bitter taste, and a camphoraceous odor. The constants were as follows:

Density at 25° C....... 0.9295	Free acid.............. 0.05
Rotation............. +7° 28′	Saponification value..... 54.3
Index of refraction N_D^{20} 1.4713	Acetyl value.......... 33.5

The oil is soluble in all proportions in 90 per cent alcohol, and in 15 to 16 volumes of 70 per cent alcohol by volume.

CHEMICAL EXAMINATION OF THE OIL

ALDEHYDES AND KETONES—The oil did not respond to decolorized magenta or sodium bisulfite solution. When tested with phenylhydrazine a distinct cloudiness was produced. This would indicate the presence of a ketone that does not respond to the magenta or bisulfite test, such as camphor.

FREE ACID—The free acid value of the oil was so low that it was impossible to identify the acid.

COMBINED ACIDS—One hundred grams of oil were saponified with alcoholic potassium hydroxide by heating on the water bath with a reflux condenser for half an hour. Water was added to the mixture and the oil layer separated out. After distilling off

the excess of alcohol on the water bath the alkaline solution was extracted with ether to remove any adhering oil. The remaining solution was evaporated to a small volume, acidified with sulfuric acid and distilled with steam. The distillate was extracted with ether. The ether was evaporated spontaneously, leaving a reddish brown crystalline residue. When tested with ferric chloride a purple color was formed, indicating salicylic acid.

The aqueous portion remaining after the ether extraction was neutralized with sodium carbonate. An aliquot of one-tenth of the original solution was concentrated to a small bulk and precipitated in three fractions with silver nitrate. Fraction 1 yielded 1.0560 g. of Ag salt; Fraction 2, 0.5431 g. of Ag salt; Fraction 3, 0.2114 g. of Ag salt. A perceptible reduction of the silver nitrate showed the presence of formic acid. On ignition the following percentages of silver were obtained from the silver salt: Fraction 1, 65.85 per cent Ag, Fraction 2, 53.1 per cent Ag, Fraction 3, 39.9 per cent Ag. Acetic acid yields 64.6 per cent Ag, butyric acid 55.3 per cent Ag, valerianic 51.6 per cent Ag, and capric 38.67 per cent Ag.

The combined acids consisted mainly of acetic and salicylic with traces of formic, capric and possibly butyric, or valerianic, or a mixture of the two.

FRACTIONATION OF THE OIL

The oil was fractionated with a 3-bulb Le Bel fractionating column, the results being given in the table.

TABLE I—FRACTIONATION OF THE OIL OF CALYCANTHUS OCCIDENTALIS SHOWING PHYSICAL PROPERTIES OF EACH FRACTION

Fraction No.	Fractionation Temperatures °C.	Per cent Distilled over	Specific Gravity 15° C.	Specific Rotation at 15° C.	Index of Refraction 15° C.
1	154–170	8.51	0.8810	+16.42°	1.4590
2	170–180	54.83	0.9119	+ 5.85	1.4585
3	180–190	9.47	0.9215	+ 6.56	1.4650
4	190–200	2.48	0.9290	+ 9.52	1.4665
5	200–220	8.75	0.9635	+36.42	1.4817
6	220–over	10.22	0.9365	+18.85	1.4793
7		5.01	0.9171	...	1.4884

Fractions 5, 6 and 7 were made under reduced pressure at 150–180°. The boiling points of these fractions were redetermined at atmospheric pressure.

From the fractions in Table I it will be seen that this oil contains 5 groups of constituents, the first distilling between 154–170°, the second between 170–190°, the third between 190–220°, the fourth between 220–230°, and the fifth over 230° C.

CHEMICAL EXAMINATION OF THE FRACTIONS

PINENE—Fraction 1 consisted mainly of pinene. The nitroso chloride was prepared according to the method of Wallach[1] and was found to melt at 102.3°; the pure nitroso chloride melts at 102–103°. The medium value of the specific rotatory power indicates that although the pinene is mainly of the dextrorotatory form some of the laevo-rotatory form is also present. A quantitative estimation of pinene in the original oil was made by distilling off a fraction between 155 and 170° C. and placing a portion of this fraction in a cassia flask and adding a 50 per cent

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[1] Wallach, Liebig's *Ann.*, **245**, 251; **253**, 251.

resorcinol solution, which absorbed the cineol. From the residual volume it was estimated that about 8.3 per cent of the original oil consisted of pinene.

CINEOL—Fractions 2, 3 and 4 combined and refractionated between 175 and 180°. After twice refractionating, the portion boiling at 176° was collected. The physical constants of this fraction and of the melting point of the iodol show that this fraction is mainly cineol.

	Fraction	Pure Cineol
Boiling point	176–177°	176°
Density at 20° C	0.930	0:929
Index of refraction at 20° C	1.4561	1.4559
Melting point of iodol	111.2°	112°

A quantitative determination by the resorcinol method gave 60.32 per cent cineol by weight of the original oil.

BORNEOL AND CAMPHOR—Fractions 4 and 5 were combined and redistilled under vacuum. They had a strong camphoraceous odor and on freezing crystals separated out which were very difficult to remove from the adhering oil. An attempt to prepare the semicarbazone by the method of Tiemann[1] gave no results.

Phenylhydrazine produced a distinct cloudiness, showing the presence of a ketone. When the fraction was heated to vaporization, and the vapor permitted to cool on a microscopic slide, a white sublimate was obtained which was found to consist of two distinct substances, one crystallizing in plates, the other in smaller irregular aggregates. This would point toward a mixture of borneol and camphor. A portion of this fraction was oxidized with Beckmann's chromic acid mixture, the acid neutralized and the mixture distilled with steam. The semicarbazone was prepared from the product and was found to have the melting point 235.8°: camphor semicarbazone melts at 236–238°. This fact, as well as the evidence of a ketone in the original oil, points to a mixture of borneol and camphor. If the acetyl value be considered due to borneol then there would be 9.21 per cent borneol present. This value is a little high, as some of the sesquiterpene alcohols present combined to form the ester with acetic anhydride, thus raising this constant.

METHYL SALICYLATE—Fraction 6 had a strong pleasant odor of esters. Since salicylic acid was found in the original oil, methyl salicylate was suspected. This fraction was treated with cold $N/5$ potassium hydroxide solution in order to form the soluble potassium methyl salicylate salt. After extracting the remaining oil the alkali solution was neutralized with dilute acid and again extracted with ether. On spontaneous evaporation of the ether a small quantity of oil remained, having the characteristic odor of methyl salicylate. Such a small quantity was obtainable that the index of refraction alone was determined and found to be 1.5298. The index of refraction of pure methyl salicylate is 1.536 to 1.538 at 20° C. A further amount of the oil was saponified with concentrated hot alkali solution. The solution was acidified and extracted with ether. The ether extract yielded

a few crystals which gave the characteristic color reactions for salicylic acid. From these data and from the boiling point of the fraction in which this substance appears it can be safely concluded that a small quantity of methyl salicylate was present in the oil.

LINALYL ACETATE—The remainder of Fraction 6 had a very pleasant bergamot odor. When saponified with alcoholic potash the saponification number was found to be 277.9. The alcoholic solution was diluted and the excess alcohol distilled off on the water bath. The solution was acidified and extracted with ether. Silver nitrate was added to the solution; the per cent of silver in the silver salt obtained was found to be 64.12 per cent Ag: acetic acid combines with 64.46 per cent Ag. The empirical formula for the ester would then be $C_{10}H_{17}OOC.CH_2$. The ether extract from this solution was evaporated spontaneously and the residual oil refractionated. The greater proportion distilled between 197 to 198°. The specific gravity was found to be 0.8793; the index of refraction at 20° C. was 1.4691. If from this data the molecular index of refraction is calculated it is found to be 48.77. The theoretical molecular index of refraction for a compound of formula $C_{10}H_{17}OH$ with two double bonds is 48.86. This would indicate linalool. The only other alcohol having this structure is geraniol which has a higher boiling point and a greater specific gravity than linalool. The presence of linalool was confirmed by preparing the phenyl urethane derivative which had a melting point at 65.4°: linalool phenyl urethane melts at 65 to 66°.

SESQUITERPENE ALCOHOLS—These remained in the fractions boiling above 230° C. Owing to the small quantity of material and due to the fact that considerable polymerization had taken place, it was impossible to determine what they were. They constituted less than 5 per cent of the original oil.

SUMMARY

The volatile oil of *Calycanthus occidentalis* is made up of the following constituents in the proportion given:

	Per cent
Pinene (*d* and *l*)	8.30
Cineol	60.32
Borneol	9.21
Camphor	...
Methyl salicylate	...
Linalyl acetate	18.99
Sesquiterpene alcohols	...

In the above estimation of the percentages of constituents present, the pinene and cineol were determined directly. The borneol was calculated from the acetyl value: this is perhaps slightly in error, due to the presence of sesquiterpene alcohols. The linalyl acetate was estimated from the saponification value, and is slightly in error, due to a small amount of methyl salicylate.

In conclusion, the writer wishes to thank Professor W. C. Blasdale, of the Chemistry Department of the University of California, for valuable suggestions in carrying out this investigation.

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[1] Tiemann's *Ber.*, **27**, 1, 815.

LABORATORY AND PLANT

COMMENTS ON THE KREBITZ PROCESS OF SOAP-MAKING AND GLYCEROL RECOVERY[1]
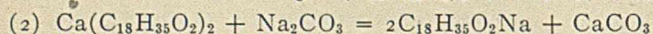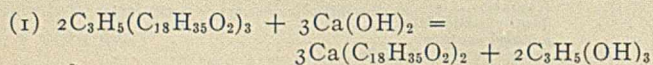
By G. A. Wrisley
Received May 6, 1916

The process of soapmaking by boiling fats and oils (glycerides) with caustic soda is, in general, considered the most practical process since it yields soaps of uniformly good quality, color and hardness, and at the same time a good amount of glycerol may be recovered by comparatively simple means. The yield of glycerol from the soap lye runs from 60 to 80 per cent of the total amount formed by the saponification of the fats and oils. The loss depends upon the few or many washes that the soap receives. However, it seems that no matter how careful one is to make the washes and changes on the soap, there is always some glycerol, $1/_2$ to 1 per cent, left in the soap. It has also been demonstrated that in evaporating down waste lyes and handling so much salt which cannot be perfectly dried and freed from glycerol, that there is recovered but about 90 per cent of the glycerol that is in the soap lye. Hence even after the glycerol is in the soap lye, there is a loss in its recovery.

With the continually advancing price of fats, oils, and other raw materials, it is becoming more necessary for the soap manufacturer to obtain a large glycerol yield; in fact it is essential that he should recover all the glycerol liberated by the saponification. Then, too, the grade of fats and oils obtainable for soapmaking is lower, because of increased utilization of the better grades for edible purposes, so that the soap manufacturer must strive to produce a good quality soap from a poorer grade of material.

The Krebitz process, here discussed, offers the possibility of recovering the theoretical yield of glycerol, while at the same time the caustic lime exercises some purifying action, especially in the case of a low-grade material. The Allen B. Wrisley Company, of Chicago, is the first in this country to work this process on a large scale with good results.

The Krebitz process is based on two simple chemical reactions:

(1) $2C_3H_5(C_{18}H_{35}O_2)_3 + 3Ca(OH)_2 =$
$$3Ca(C_{18}H_{35}O_2)_2 + 2C_3H_5(OH)_3$$
(2) $Ca(C_{18}H_{35}O_2)_2 + Na_2CO_3 = 2C_{18}H_{35}O_2Na + CaCO_3$

In practice a batch of 10,000 lbs. of fat and oil may be conveniently handled. In a rather shallow tank 1200 to 1400 lbs. of lime are slaked with 3700 to 4500 lbs. of water, and the mass is heated, if necessary, to about 70° C. Then the fat and oil are run in while the entire mass is stirred vigorously. With live steam the mass is heated up slowly to 90–92° C., and taking about one-half hour to gain that temperature, with constant stirring, a thorough emulsion is obtained. The vessel is then covered to prevent loss of heat. Two or three hours after covering the mass is likely to boil and swell considerably. After it has stood 8

to 12 hrs., it has the appearance of a solid porous mass which is still sufficiently warm and soft to allow digging out the lime soap. This lime soap is dropped through a trap door in the bottom of the tank into a hopper and then to the mill where it is ground to the size of fine corn meal.

With the lime and water there will result between 15,000 and 16,000 lbs. of lime soap from 10,000 lbs. of fat and oil. The finely ground soap is carried on a conveyor and allowed to fall loosely into a circular, hopper-shaped tower, a capacity of about 25,000–30,000 lbs. being necessary. Four washes, about 40,000 lbs. of water, are required to leach out the glycerol from such a batch. The first wash water contains 10 to 12 per cent of glycerol, and is sent to the glycerin works. The second, third, and fourth waters are put on a fresh batch, only the last wash being made with fresh water. In this way, there is no cause for evaporating glycerin water containing less than 10 per cent of glycerol. When the plant is working satisfactorily the water evaporated averages 15 per cent of glycerol.

After the glycerol has been obtained, the lime soap is carried by a conveyor and introduced slowly into the soap kettle containing a boiling solution of soda ash. After the lime has been replaced by the soda, a stage which can be noticed by the nonappearance of small lumps on the paddle, a small amount of caustic soda is added, and shortly after the soap is salted out. The contents of the kettle are allowed to rest, when $CaCO_3$ settles out at the bottom as a heavy sludge, and the soap gathers on top, a salt solution containing an excess of alkali forming an intermediate layer.

Although Krebitz claims that the lime sludge will settle out and occlude or entangle but 4 to 7 per cent of soap, it has been impossible, so far, to prevent the occlusion of less than 9 to 12 per cent of soap. Of course, this soap must be recovered. At first this constituted quite a serious problem, because the lime sludge could not be filtered without leaving 2 to 3 per cent of soap in the lime cake. Attempts to wash out the soap by a series of washings and filtration were unsuccessful, because so much soap was present as to fill up the pores of the filter cloth, making filtration slow and unsatisfactory. Attempts to boil up the sludge with a little water and resalt out the soap were unsuccessful, because on salting out, the lime sludge settled to the bottom carrying most of the soap with it again. By continued experiment, it was found that the addition of enough water with heat and vigorous agitation caused the soap to go into solution, and on allowing the mass to settle the lime sludge, occluding but 3 to 5 per cent of soap, went to the bottom, the soap rose to the top, and could be pumped off and so recovered. The lime sludge could then be easily filtered, and only 1 to 1.5 per cent of soap is lost in the lime cake. This loss, though small, may perhaps still be reduced.

The quality and color of the finished soap obtained

by the Krebitz process compares favorably with the soap produced by any other process. The amount of lime found in the finished soap depends on the number of washes or changes made in the kettle. Usually there was never less than 0.2 per cent of lime and it may run as high as 0.5 to 0.7 per cent lime. The process, however, claims that only a few thousandths of one per cent should remain in the finished soap.

At the present time a yield of about 95 per cent glycerol is being obtained by this process. This, compared to the yield of 60 to 80 per cent obtained by the recovery of glycerol from the soap lye in the case of soapmaking by boiling the glycerides with caustic soda, would give the Krebitz process a decided advantage. Moreover, we must recall that soap lyes contain at best but 5 to 8 per cent glycerol, depending on the way in which the changes of soap lye are worked up, and at the same time hold in solution much common salt, some caustic soda, soda ash, and organic impurities; whereas the Krebitz waters contain 12 to 15 per cent glycerol and are comparatively free from impurities, thereby making purification more simple and giving less water to handle and evaporate. Soda ash is cheaper than caustic soda, and since soda ash is used in place of caustic soda another material saving is made.

Without doubt, the Krebitz process would be preferred at this time, because of the high price of glycerol, but were glycerol 15 to 20 cents per lb. instead of 55 to 60 cents per lb. there might be a shadow of doubt as to whether it would be preferred to the soap-lye process taking into consideration the manifold operations prior to the soapmaking proper, and the work necessary to recover all the entangled soap from the lime sludge.

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THE DETERMINATION OF BENZOL IN COKE-OVEN GAS BY BURRELL'S VAPOR APPARATUS

By Louis C. Whiton
Received April 18, 1916

The method for determining benzol and its homologues in gas as proposed by the Laboratory of Gas Investigations, U. S. Bureau of Mines,[1] has been tried in this laboratory with a view to determining the efficiency of benzol scrubbers utilizing coke-oven gas. The extraction of benzol, toluol and solvent naphtha has become at once such a profitable and important addition to a by-product coke oven's equipment that a rapid method of determination of scrubber efficiency seems imperative. The customary method of absorption of the vapors requires from 12 to 24 hrs. time and in this period many dollars worth of benzol and toluol may be lost, due to faulty operation of the scrubbers. Furthermore, especially when starting up a plant, conditions may have automatically been improved, without the operator's knowledge, toward the end of the test, which would mean that a change in operation as indicated by the test would perhaps be a disadvantage rather than otherwise.

The method as described in This Journal did

[1] This Journal, **7** (1915), 669.

not give the details of operation, which are extremely essential if accuracy is to be expected when determining such small traces of benzol vapors as are contained in debenzolized coke-oven gas.

Briefly, the apparatus as described by Burrell and Robertson, of the Bureau of Mines, consists essentially of a bulb about 6 in. long with a slight constriction in the center. The upper portion of the bulb contains glass wool mixed with phosphorus pentoxide to extract all the moisture from the gas. Connected with this bulb is a mercury manometer and an entry and exit tube with a ground glass 3-way cock. The air is first evacuated by means of a vacuum pump, the gas to be tested is sucked in and the 3-way cock turned when the gas is under barometric pressure. The entire bulb is then placed in liquid air or in a mixture of CO_2-snow mixed to a consistency of slush with alcohol or acetone. It is allowed to remain there 10 min., the temperature being approximately $-78°$ C. The benzol freezes out at this temperature and the CO_2, O_2, CO, H_2, CH_4, N_2, and C_2H_4, which have a high vapor pressure at this temperature, are evacuated by means of the vacuum pump, the cock turned to seal the system and the apparatus brought up to room temperature. The partial pressure of the benzol vapor is registered on the manometer and this result divided by the barometric pressure times 100 indicates the per cent benzol.

There were several difficulties which had to be overcome in order to term the above a practical method for commercial work.

1—The readings on the manometer ranged from 10 mm. for benzolized gas to 1 mm. for debenzolized gas. With an ordinary manometer it is difficult to read much closer than 1 mm., which was beyond an allowable error.

2—In a vertical position it was impossible to surround the apparatus with the CO_2-alcohol slush with economy in the use of the material which withal, as a commercial proposition, is expensive.

3—The P_2O_5 in the glass wool soon ceased to be a good dehydrator and with the suggested construction of the apparatus it was impossible to charge it again with P_2O_5.

These difficulties were overcome to a certain extent in the following manner:

I—The mercury manometer was connected by means of a short, heavy piece of rubber tubing and was placed at a 10 to 1 slant, *i. e.*, 10 on the hypothenuse to 1 of altitude. This was placed on a firm gauge stand and leveled with spirit levels both ways. The gauge then gave a reading ten times the actual pressure. Thus it was possible to determine the pressure within 0.1 mm. which was sufficiently accurate.

II—The bulb was laid horizontally in a trough with a notch at the end through which the stem of the apparatus could pass. In this position it could be more easily surrounded with carbon dioxide slush without danger of breakage of the bulb and with greater economy in respect to the slush. This trough was 8 in. long, $2^1/_2$ in. deep and $2^1/_2$ in. wide, with a hinged cover and double walls with the air evacuated. It was

found difficult to make this vacuum chamber absolutely tight so a petcock was soldered on and frequently the vacuum was attained again by means of the pump. This trough saved considerable cost in CO_2 and alcohol, preventing the former from being melted by the heat in the room to a considerable extent and enabling one to collect all of the alcohol after the determination by allowing the CO_2 to vaporize off.

III—The P_2O_5, which soon ceased to be active in respect to water vapor, necessitated cutting the tube leading from this bulb and connecting the two pieces by means of rubber tubing. In this way fresh P_2O_5 and glass wool could be easily inserted.

Several other points of operation may be of interest. An ordinary Geryk vacuum pump, single stroke, was used and found effective. It might be well to state the well-known method of making CO_2-snow economically. A cylinder containing 50 lbs. CO_2 at high pressure is inverted to an angle of about 45° and the snow which issues forth when the valve is opened quite wide is caught in a canvas bag about 12 × 18 in. in size. The snow keeps well in a cardboard box.

It was found that more constant results were obtained by bringing the bulb up to room temperature after immersion in the freezing mixture, by means of inserting it in water for 10 min. A more careful control of the temperature can thus be maintained.

Although this article is intended only to give certain suggestions for the operation of Burrell and Robertson's apparatus it might be of interest to show some results obtained with it using coke-oven gas, to supplement the results obtained at the Bureau of Mines on Pittsburgh illuminating gas.

	Gas A (duplicates)		Gas B (duplicates)	
Bar. pressure, mm............	742.0	742.0	741.0	741.0
Partial press. benzol vapors....	5.4	5.2	1.8	1.9
Per cent benzol in gas.........	0.729	0.702	0.243	0.256

These are typical determinations made by this machine. Benzol scrubber efficiency does not call for absolute benzol determinations, comparative results before and after scrubber being sufficient. Therefore it is not necessary to have the manometer as carefully leveled as otherwise. Efficiency of scrubbers with the above results averaged would be determined thus:

$$\left(1.00 - \frac{1.85}{5.3}\right) \times 100 = 65.1 \text{ per cent}$$

The extreme results on the above tests would indicate 66.7 and 63.7 per cent efficiencies, which is as good as can be expected with this apparatus.

The result using this method as a determination of absolute benzol, toluol and solvent naphtha is somewhat lower than that obtained by absorption methods and averaged 24 per cent less than the amount obtained in actual practice. Benzol it is known has a sufficiently low vapor pressure at —78° C. to assert that it is completely condensed; about toluol there appears to be some doubt, and about solvent naphtha (a mixture of xylol and higher homologues of the benzene series) there appears to be little known and with reason as its composition varies in different plants.

With an actual recovery during the period of one month of 2.20 gals. per net ton of coal carbonized of benzol + toluol + solvent naphtha, the average of many results determined by this apparatus indicated an amount equal to 1.70 gals. per ton coal or 22.7 per cent low. The recovered hydrocarbons in practice consisted in the following:

HYDROCARBON:	90% Benzol	Crude Toluol	Solvent Naphtha
Per cent recovered......	67.5	19.3	13.2

This would seem to indicate that all of the benzol, part of the toluol and little if any of the solvent naphtha was condensed at the temperature of —78° C.: but this is merely a suggestion as it has not been as yet thoroughly investigated.

In conclusion it might be added that this method is more difficult of operation than it appears. The apparatus is delicate and the conditions as stated above must be rigidly adhered to in order to obtain consistent checks. As a method for determining benzol scrubber efficiency it is of some value; as a method of absolute determination of benzol, toluol and solvent naphtha it is of doubtful value, giving results apparently 24 per cent lower than those obtained in actual practice. Furthermore, the operation of the method requires a man skilled in handling such apparatus.

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CHATTANOOGA, TENNESSEE

AN AUTOMATIC PIPETTE[1]

By ALEXANDER LOWY

Received March 28, 1916

Fig. 1 shows the entire pipette with the modified stopcock adaptable to any given volume. Fig. 2 shows the position of the stopcock while the liquid is being drawn up. Line AB is the mark of graduation. H shows the chamber below stopcock and G the chamber above the stopcock. F is a cylindrical bore through the stopcock connecting chambers G and H during the process of suction. E is a cylindrical bore ending back of F at an angle of 90°, of which C is a continuation and ends at a slight elevation at D. Fig. 3 shows the position of stopcock after it has been turned clockwise 90° to that shown in Fig. 2. D shows place closed by thumb. In this position channel C is connected with chamber H through opening E. In this position channel G is shut off from channel H.

With suction applied at end of chamber G (Fig. 2), the liquid is drawn up through chamber H until it just passes the graduated mark AB. The thumb is then placed on D so as to close opening D air-tight. The stop-cock is then rotated clockwise through 90° thus bringing opening E (the continuation of tube C and D) to line of graduation AB and directly in contact with the upper opening of chamber H. The thumb is then released. Air pressure now forces the exact

[1] Patent applied for.

measured volume of liquid out of chamber H. Any excess liquid drawn up beyond line AB will be entrapped in either bore F, or in bore F and in chamber G. This excess is returnable by revolving the stopcock counter-clockwise through $90°$.

The advantages of this pipette may be summarized as follows:

1—It enables the operator to automatically control an exact measured volume of liquid drawn into the pipette.

2—It obviates the necessity of adjusting, maintaining and manipulating the exact volume of the liquid once it has passed the graduation mark, placed

C. C,
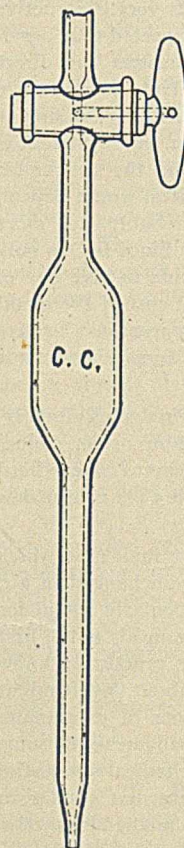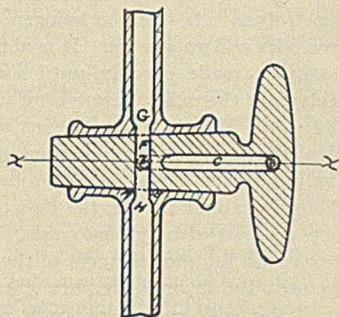
FIG. 2

Section at Y-Y

FIG. 1
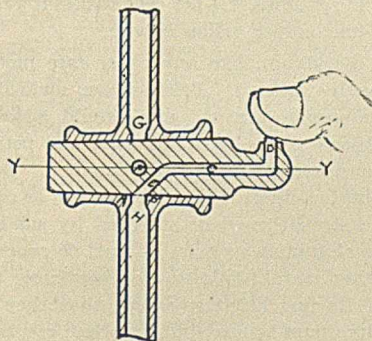
FIG. 3 — Section at X-X

where the stopcock meets the lower end of the valve of the pipette.

3—It permits the discharge of the exact measured volume of liquid from the pipette.

4—It is exceedingly easy to manipulate.

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THE DETERMINATION OF AIR, WATER VAPOR AND NITROUS OXIDE IN MIXTURES OF THESE THREE CONSTITUENTS[1]

By G. A. BURRELL AND G. W. JONES
Received May 19, 1916

The authors of this report had occasion recently to examine some samples of nitrous oxide (dentists' "laughing gas") for the presence of water vapor and

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

air. The mixture was first liquefied by means of liquid air, the air being then withdrawn with a Töpler mercury pump, and measured. The residual gas was subjected to a temperature of $-78°$ C., the nitrous oxide being then withdrawn and measured. Finally the partial pressure of the water vapor was measured. This analysis is one of the many gas analyses that can be performed by means of the apparatus shown in Fig. I.

The apparatus is first exhausted of its air by means of a Töpler mercury pump, and the sample of "laughing gas" is introduced at atmospheric pressure. Next the bulb A is immersed in a Dewar flask containing liquid air. After about 10 min. the air is withdrawn from the mixture through the pump and measured. The vapor pressure of the air at the temperature of liquid air is of course very high, and the air can be readily removed from the mixture. The vapor pressure of nitrous oxide is 1 mm. at a temperature of $-144.1°$ C.,[1] hence its pressure at the temperature of liquid air is practically negligible.

After the air has been removed and measured, the bulb A is immersed in a mixture of solid carbon dioxide and acetone. This mixture gives a temperature of $-78°$ C. The bulb A is exposed to this temperature for about 10 min., and the nitrous oxide is withdrawn through the pump and measured. The normal boiling point of nitrous oxide is $-88.7°$ C.[2] so it can be readily removed from the mixture at a temperature of $-78°$ C. On the other hand, the vapor pressure of water is practically nil at a temperature of $-78°$ C. so that it freezes and remains in the bulb A.

After the nitrous oxide has been withdrawn and measured, the Dewar flask containing the solid carbon dioxide and acetone is removed from around the bulb A. The frozen water vapor then vaporizes and exerts its partial pressure on the mercury in the manometer tube C. This pressure is, of course, proportional to the percentage of water vapor present.

The results of the analysis of three samples of gas taken from the same tank follow:

FIG. I

Apparatus for the Determination of Air, Water Vapor and Nitrous Oxide in Mixtures of the Three

PERCENTAGE ANALYSES OF NITROUS OXIDE

CONSTITUENT	AIR	N$_2$O	H$_2$O	TOTAL
Sample 1	2.0	95.9	2.0	99.9
Sample 2	2.1	95.6	2.0	99.7
Sample 3	2.0	96.2	2.0	100.2

BUREAU OF MINES, WASHINGTON

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[1] G. A. Burrell and I. W. Robertson, "The Vapor Pressures of Sulfur Dioxide and Nitrous Oxide at Temperatures below Their Normal Boiling Points," *J. Am. Chem. Soc.*, **37** (1915), 269.
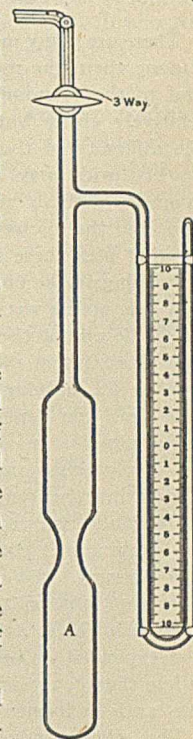[2] G. A. Burrell and I. W. Robertson, *Loc. cit.*

ADDRESSES

THE METALLURGY OF THE RARER METALS[1]

By J. W. Richards

Received June 14, 1916

There are many metals which may be called "the rarer metals." Among them the most interesting by far, to the metallurgist and to the economist, are those metals whose compounds are relatively cheap but which command a high price because of the difficulty of their reduction. These are the metals whose market price may at some time be reduced one-half, three-quarters, perhaps nine-tenths, by improved methods of reduction, and the discussion of how this might be accomplished and to what uses these metals at such low prices might be put, is interesting to the border of fascination.

If this article were being written thirty years ago, aluminum would be one of the metals to be discussed. It is now out of that class, but in 1886 it was one of the rarer metals, selling at $10 per lb., although its ores were "as common as dirt." At that time you could buy a ton of bauxite ore containing 0.6 ton of alumina or 0.3 ton of aluminum, for $5.00, while the 0.3 ton of aluminum was being sold for $3,000, at wholesale. Or, eliminating the purely chemical work, 0.6 ton of chemically purified alumina could then have been bought for $50, while the aluminum it contained was worth on the market 60 times that amount. Such were the metallurgical conditions in the aluminum industry thirty years ago, and so attractive were they to experimenters and inventors that the genius of the profession expended its best talents on the problem, with the result, in less than ten years, of reducing the market price to less than one-tenth its former figure.

The silicon industry furnishes another example in point. Silica is the most abundant and the cheapest material in nature, yet silicon was selling in 1900 as a chemical curiosity at *over $100 an ounce*. Imagine the stirring up which my metallurgical wits received, when in 1902, Mr. Tone, at Niagara Falls, showed me a barrel full, 100 lbs. perhaps, of silicon made in his electric furnace, and asked me what uses it could be put to. At the present time, 10 c. per lb. is a good market price for silicon, which is often sold by the carload.

It is such seeming fairy tales as these which constitute the fascination of those metals which are abundant in nature but whose high cost rests on the difficult and costly methods of reduction employed. Such opportunities exist for our rising generation of chemists and metallurgists to make themselves famous and, incidentally, rich—but they will find their fame more of a reward than their riches.

Among the metals at present of high price, but which by improved metallurgical processes might be made very cheaply, are beryllium, boron, magnesium, calcium, strontium, zirconium, molybdenum, barium, titanium, chromium and cerium (mixed metals of the cerium group).

BERYLLIUM

Commencing with one of the light, alkaline-earth metals, its ore is not so rare as is ordinarily supposed. Most non-mineralogists link it with beryl, and think of the latter as a very pretty and very expensive gem—the emerald. But the emerald is only the clear green or aquamarine stone, while massive beryl, looking like massive green quartz, is much more common, and is even abundant in some localities. The beryl crystals of Acworth, N. H., are sometimes as large as a barrel, and the massive beryl at this locality is quarried like feldspar. Its composition is: SiO_2, 67; Al_2O_3, 19; and BeO, 14 per cent.

It is difficult to say what the price of this material would be

if it was desired by the ton, but it should not be expensive. When chemically treated, both the Al_2O_3 and the BeO which it contains could be separately obtained.

Up to the present, no one has succeeded in isolating the metal except by reducing a halide salt of beryllium by potassium or sodium (Bussy, Wöhler, Debray, Menier, Reynolds, Nilson and Petterson, Kruss and Moraht), or by electrolysis of double chloride, bromide, or fluoride of beryllium and sodium or ammonium (Borchers, Warren, Lebeau, Liebmann).

These methods are tedious and costly for two reasons: *First*, they require the conversion of the beryllium into an anhydrous halide salt, which is a difficult chemical operation; *second*, the electrolysis sets free the halogen, which is very destructive to electrodes and apparatus. It is not to be expected that beryllium can be made cheaply until someone masters the direct electrolysis of the oxide, dissolved or suspended into a more stable melted salt. This is by no means an impossibility; a similar solution was found for the aluminum problem, and systematic, determined search would in all probability find the answer for beryllium. In such a case, the cost of the metal would depend only on the cost of beryllium oxide, being probably not more than 20 c. per lb., plus the cost of the oxide. Since BeO is only 36 per cent Be, it would require 3 lbs. of oxide to give one of metal; if the oxide cost 10 c. per lb., the total cost of the metal should not exceed 50 c., by such a suppositious process. Dealers in rare chemicals will charge you, at present, $300 per oz. for a specimen of it.

Lebeau has produced beryllium bronzes by reducing directly, in the electric furnace, a mixture of beryllium oxide, copper oxide and carbon: 0.5 per cent of beryllium makes copper hard and sonorous, 1.5 per cent makes it yellow, and 5 per cent makes a fine, golden yellow bronze.

Beryllium, like any other rare metal, must find uses which justify its cost. Being white, malleable and unchanged in air, its specific gravity, 1.64, would make it particularly useful for objects where great lightness and permanence in air is the first consideration and cost secondary. So far we practically know nothing about its tensile strength or rigidity, about how it might be strengthened or stiffened by small additions of magnesium or aluminum or even of zinc, or copper, or manganese, or some other metal. We do not yet know the mechanical properties of its fine bronzes, except that they are somewhat similar to aluminum bronze, but in what respects they might be superior or perhaps unique, is unknown. Finally, the metal may easily possess special properties, now unknown, which may render it particularly useful for some specific purpose. Its specific heat, for instance, is the highest of any useful metal, and its latent heat of fusion must be abnormally high, possibly 300 calories, and its latent heat of vaporization probably higher than that of any known element, except carbon or boron. Such characteristics might give it special uses in electrical instruments, or for physical apparatus, where its cost would not exclude its use. Altogether, beryllium is a metal which will well repay extended metallurgical research and minute physical and chemical study of its many unique properties.

MAGNESIUM

The metallurgist has been coquetting with magnesium for half a century, and has not, as yet, made a fraction of the progress which he should have made. We can get its oxide cheaply and in abundance, its salts are not very difficult to prepare, we know their properties to a considerable extent, we know almost all the properties of the metal which bear on its isolation, and yet the industry lags and halts as if there were no such thing as modern metallurgy. To prepare by tedious methods the anhy-

drous double chloride, and then to electrolyze it about as Matthiesen did fifty years ago, is nearly all that can be said with certainty about its present metallurgy. At any rate, using magnesium oxide costing a few cents per lb., the metal sells for about as many dollars per lb., and yet there is a great scarcity of the metal.

Dr. W. M. Grosvenor, in a recent paper before the American Electrochemical Society, summarizes the present methods of production and the uses of the metal. Happily he also suggests the great field open for improvement, especially for radically new metallurgical methods of production. With magnesium in its salts costing less than 8 c. per lb., he places the actual cost of the metal at $1.00 per lb. leaving over 90 c. per lb. for the cost of its extraction. "Brethren, these things ought not so to be." Speaking with the enthusiasm born of past achievements in electrometallurgy, along entirely analogous lines, a modern, up-to-date attack on this problem ought to result in producing magnesium at 25 c. per lb.

The point of attack should be undoubtedly to reduce the oxide directly. The halogen salts are hygroscopic, and the halogen is destructive of the reducing apparatus. It is almost certain that proper research will enable the electrometallurgist to feed magnesium oxide (MgO) directly into an electrolytic bath of fused salts, and take magnesium or magnesium alloy from it. The pure metal will float on almost any fused salt, but its alloy with heavier metals may be made such as to sink, and many of its alloys have immediate useful applications.

Dr. Grosvenor makes suggestive remarks about the reduction of magnesia by carbon. If the boiling point is only 1200° C., then a process of reduction similar to that of zinc oxide might be practicable if we could (1) find a retort material which will stand the temperature required (1800 to 2000° C.), and (2) condense the vapors without contact with air. Fortunately, magnesium does not form carbide at high temperatures, so that its reduction is simpler by that much. Dr. Grosvenor speaks of a chemical process which will use cheap raw materials, a moderate amount of fuel, and give a fair efficiency of reduction. With all expenses added, he estimates a cost not over 35 c. per lb. This may be true, but whether it materializes or not, he and his colleagues have the right vision of the possibilities, I may even call them the probabilities, of this field; they are truly "absolutely fascinating."

While the world war lasts, with its enormous demand for magnesium for military purposes, the price will remain in the dollars per lb. But experience in this line is being rapidly accumulated, and improvements are undoubtedly rapidly succeeding each other, although keen competition is keeping them secret as far as possible. After the war's close, with normal industrial conditions reappearing, magnesium will undoubtedly sell at a price which will take it out of the class of the rarer metals and put it among the common ones. As the price goes down its industrial uses will increase in geometric proportion, and instead of production being expressed in thousands of lbs. per year it will reach thousands of tons. This will be another of the by-products of the great war's stimulus to metallurgical industry.

The possibilities held out to the metal industry by reasonably cheap magnesium are extremely interesting. The stiffening of magnesium to produce strong alloys with specific gravity not over 2, has not been properly studied. It is quite possible that alloys analogous to *Dur-alumin* may be discovered, as strong as soft steel and only 30 per cent of its weight, which will find extensive use in aeroplanes and dirigibles. Such alloys may also largely displace aluminum alloys, which are used by thousands of tons annually in the automobile industry, with a saving of one-third in weight, which will compensate for a higher first cost. The metallurgical uses of magnesium will also be greatly extended by its lower price, such as for deoxidizing brass, bronze,

nickel and Monel metal, since it is a much stronger deoxidizer than aluminum. In fact, aluminum has blazed the way into numerous uses for which magnesium, as soon as it becomes cheaper, will compete and replace its older sister. With supplies of magnesium ore as plentiful as those of aluminum ore, and the metallurgist awake to his responsibilities and producing the metal cheaply, there will inevitably be a large future for magnesium as one of the common metals of every-day life.

CALCIUM, STRONTIUM, BARIUM

These form a trio of highly interesting elements, common enough in nature, but all scarce and of high price because of the metallurgists' lack of efficient and cheap methods of reduction. With burnt lime, CaO, one of the cheapest of common materials, strontium sulfate, a mineral found in considerable abundance, and barium sulfate, so common as heavy-spar that it is used as an adulterant for some cheap paints, the metallurgist is again faced with the demand for cheap methods of reduction. And yet, although calcium is sold at a few dollars per lb., strontium and barium cost several dollars per oz. Here is a twofold need: *First*, cheap production; *second*, a thorough study of these metals to find out their specific properties and their particular uses. Which should be undertaken first is an interesting topic for discussion. Historically, the metallurgist has usually produced the metal first and then studied its properties and possibilities; at present, with these metals already at hand, metallurgical activity might be greatly stimulated by extensive studies of the properties, alloys, and chemical uses of these elements. Our present information in this direction is fragmentary and partly unreliable as far as it goes. A Carnegie research scholar, or even the Bureau of Standards, by disclosing to us some of the unknown properties of these elements, might stimulate the experimenter to renewed efforts to find cheaper methods of reduction.

CALCIUM, at the present time, is the best known of these three elements. The method of electrolyzing its fused chloride and lifting the metal away from the surface, as an irregular stick, has been fairly successful, and since the chloride is not difficult to dehydrate, the whole operation is not very expensive. Calcium is therefore, at present, perhaps a *semi-rare* metal, which could be produced much cheaper even by present methods if made on a large scale to fill a large demand. The method of production is easily susceptible of minor improvements, and the chlorine is a valuable by-product to the manufacture. The principal hitch at present is in finding the uses for a large production of calcium. Here is where extensive study of the properties and possible uses of calcium would greatly stimulate the metallurgical industry. With a specific gravity of 1.85, its possible alloys with other light metals should be exhaustively studied; quite possibly some of them are strong, resistant to air and water, perhaps even to acids. Calcium tarnishes easily in the air, and magnesium also, but it is quite possible that some alloy of the two does not tarnish, and may have valuable mechanical properties. Another large possible use is as chemical purifying agent in melting and casting metals. Calcium-silicon-aluminum alloy has already found application as a deoxidizing agent in steel, because while aluminum oxide and silicon oxide and their combination with each other are infusible at steel-melting temperature and therefore are eliminated slowly from the metal, calcium oxide forms with these an easily fusible slag, which easily rises out of the molten metal. It is quite possible that a small addition of metallic calcium may in a similar manner reduce the amount of sulfur and phosphorus in steel, because it is either as calcium sulfide or calcium phosphate that these elements are eliminated in refining steel. Other metals and alloys, whose properties are damaged by sulfur or phosphorus, may be similarly refined or improved. The alloys of calcium with copper, tin, bronze, brass, Monel metal, and other commercial alloys have not been studied; until they are, no

one knows how many useful mixtures may exist with particular properties of industrial value. The question of adding calcium to the light, stiff aluminum alloys, for instance, is worthy of attention, but has not been touched.

STRONTIUM is a silvery white, very soft metal, with properties similar to calcium, density 2.54. Its ores cannot be called rare minerals, and it is a rare metal, therefore, only because of the difficulty of its isolation. It is chemically very active, and electrochemically extremely hard to manage. It has about the same specific gravity as its fused salts, so that it neither rises nor sinks in any of them quickly; it seems also to redissolve in its melted salts with great velocity, so that very high current density is required to obtain any metal at all. Its surface tension appears to be abnormally high, so that it separates out in more or less minute globules which are highly indisposed to running together into one mass. What an attractive subject this forms to the electrochemist who really wants to meet difficulties and taste the joys of overcoming them. And then, one may well ask: "To what purpose?" Here again we do not know, but we can feel confident that in the innumerable list of possible combinations of metals strontium might have properties different from any other element, which would lead to its employment on a large scale. These are all questions of the future which form the undiscovered country open to the investigator and chemical pioneer. We may well thank our stars that the world of science still holds unexplored areas to tempt the adventurous investigator and to reward the *wanderlust* of the metallurgical pioneer.

BARIUM is common in its compounds and almost unknown in itself. Under electrolytic conditions where calcium comes out *en masse*, and strontium separates as small globules, barium is obtained only as a fine powder. Its density is 3.75, but its salts are heavier than the corresponding strontium and calcium salts, so that the fine powder may sink or swim, largely according to the temperature. And yet the fused barium salts are very decent and manageable salts, easily obtained anhydrous and melting cleanly. Their electrolysis, however, is nearly the most difficult task that the electrometallurgist can take up. But it should be taken up and mastered, because a metal so common in its compounds could be obtained in large quantities if uses for it were developed, and if our modern electrometallurgists are worthy successors of Faraday, Bunsen and Castner, they should vigorously attack the problem of its cheap extraction. Even if their end was not reached, they would be whetting their metallurgical wits upon the finest of chemical whetstones, and their experiences would be of value to themselves as well as to other lines of electrometallurgy.

BORON

As an intermediate element, semi-metal, cheap and abundant in its compounds, yet almost unknown as an element, boron is very interesting. Although quoted at several dollars per oz. yet there are possibilities of it being made for x cents per lb., where x may be anything over 25. It is only the prophetic prescience of the enthusiastic metallurgist, however, that can discern the goal in the distant future.

Boron occurs in nature as its oxide, Sassolite, containing 31 per cent of boron, as borax, containing 11.5 per cent of boron, and as colemanite, containing 18.9 per cent of the metal. These sources are comparatively abundant, the oxide being found in volcanic districts, borax near dried up lakes, and colemanite (calcium borate), being literally a waste product of the borax mining, for which uses have only recently been developed.

Gay-Lussac and Thenard, Wöhler and Sainte-Claire Deville reduced the oxide by potassium or sodium, other chemists by phosphorus, magnesium, aluminum and calcium, while Duncan reduced boron chloride gas by hydrogen. Chemically, its reduction does not appear to be of extraordinary difficulty. Electrochemically, Davy electrolyzed fused boracic acid, Gore potas-

sium boro-fluoride, and Faraday fused borax. All describe having isolated boron. Quite recently, Weintraub has decomposed boron chloride by hydrogen in a high-tension arc, and obtained purer boron and in larger quantity than any previous investigator. His classical paper is in the *Transactions of the American Electrochemical Society*, 16 (1909), 165.

The properties of the pure boron obtained by Dr. Weintraub are exceedingly interesting. Its fusing point is extremely high, between 2000 and 2500° C.; it was fused in a boron nitride crucible under the pressure of its own vapor. Its boiling point is near to its fusing point; it has considerable vapor tension as low as 1600° C. It has a conchoidal fracture, and is nearly as hard as the diamond. At room temperature it is electrically almost a nonconductor, but at 500° C. its conductivity has increased 2,000,000 times; at 1000° C. its conductivity is of the order of that of the metals. Applying moderate voltages to a cold piece, it soon warms up and makes itself a good conductor. Such extraordinary properties suggest its use for many interesting electrical contrivances, which are enumerated by Dr. Weintraub.

Other uses of boron, which have not yet been thoroughly investigated, are in the formation of boron steels and boronized copper. The former were investigated by Guillet, in France, with irregular results; in some respects, at times, the effects were similar to that of vanadium in the famous vanadium steels, at other times the results were different. The uncertainty may have been due to irregular composition of the ferro-boron alloy used. A great amount of investigation should be done on this line, first in making a reliable quality of ferro-boron, and second in using it systematically in various qualities of steel. The question of boronized copper is in a still greater state of uncertainty. Boron or even boron sub-oxide (B_7O, or perhaps, boron saturated with B_2O_3) added to melted copper enables a perfect copper casting to be obtained, of practically 100 per cent electrical conductivity; a trade product sold as boronized copper has similar effects in producing sound copper castings. These results are only the beginning of an extensive field of investigation of the effects of small amounts of boron on metals and metallic alloys. They will certainly lead to important metallurgical discoveries and improvements.

In metals and alloys boron can act chemically, as a purifying or refining agent to remove oxygen, nitrogen and perhaps sulfur, phosphorus, and dissolved oxides, while in larger amount it acts metallurgically as an alloying element. In the latter respect it forms true alloys, such as the ferro-boron alloy, which is an article of commerce. The possibilities of extensive use in molten metals and alloys are great, but mainly dependent upon systematic metallurgical research in properly equipped laboratories.

The production of the metal and its alloys also needs expert attention. For use in steel, a good uniform quality of ferro-boron is needed, and the manufacture of this in satisfactory uniform quality has not yet been mastered. Some years ago, the Pacific Coast Borax Company offered a prize of $500 to be awarded by the American Electrochemical Society, for a practical electric furnace method of producing ferro-boron directly from calcium borate (Colemanite), a waste product of the borax mines. A few years later the prize money was returned by the Society to the Company, as not having been earned, although several attempts were made for it. For use in copper, brass and bronze, cupro-boron alloy answers as well as having pure boron. One method of making this is being tried, and the product is giving some very satisfactory results. Improved and more certain means of getting boron into copper are needed, and could probably be found by a moderate amount of careful investigation. As for pure boron and the fascinating possibilities dependent on its remarkable properties, Dr. Weintraub's method makes the product, but at considerable expense, and a cheaper, easier method is a great *desideratum*. If such is found, boron

will certainly occupy an important place among the useful metals.

CHROMIUM

This element is also common and abundant in nature, and rare and expensive as a metal. Chromite, containing 34 per cent of chromium, costs normally $20 to $25 per ton, while the ferro-chromium alloy produced from it sells at $100 to $550 per ton, according to the percentage of chromium and carbon contained. But pure chromium, carbon-free, is produced only by reduction of chromium oxide by aluminum, and commands 75 c. per lb. The use of chromium in steel is rapidly extending to all varieties of extra hard and high-speed steel, but the use of pure chromium is limited by the high cost of its production and our lack of knowledge of how to handle it and of its possible useful effects. For example, chromium electroplating is white and durable, and for many purposes may be superior to nickel and almost equal to platinum plating, but the technique of always getting perfect plating has not been satisfactorily mastered. Cobalt-chromium alloys have been made which have some of the remarkable properties and uses of high-speed tool steel (Stellite alloy of Mr. Haines). This is an excellent example of totally unexpected and valuable physical properties being discovered by systematic investigation. These alloys, however, must be made from pure chromium, and not from ferro-alloy. How many other remarkable alloys yet remain to be discovered by patient and intelligent investigation, no one knows or can even guess.

As for the methods of reduction, ferro-chromium alloy carrying high carbon (6 to 8 per cent) is produced quite cheaply in crucibles, cupola furnaces, blast furnaces, or electric furnaces. Low-carbon ferro-chromium commands three to five times as high a price, because of the difficulty of decarbonizing the raw product. It is very much to be hoped that tests will be made in the electric shaft-furnace to see if it is not possible to produce directly from the ore a low-carbon product. The thing has been done in the case of pig iron, producing a low-carbon product which is called pig steel; there is no inherent impossibility in similarly mastering the conditions for producing directly the low-carbon ferro-chromium. The present prices of the two products, $100 and $500 per ton, respectively, would warrant great efforts in that direction.

Similarly, chromium is not a difficult metal to reduce to the metallic state, but it is a difficult question to find the proper flux, and to keep carbon out of it. Goldschmidt reduces it by aluminum; electrolysis of its fused salts is difficult because of their high melting points. If electrolysis of aqueous solutions of chromium salts could be satisfactorily controlled, so as to produce heavy deposits, this might open the door at once to cheap and pure chromium. Electrolysis of molten relatively fixed salts in which chromium oxides are dissolved (similar to the Hall aluminum bath) is not a hopeless proposition. The chromium would be plated solid, however, on chromium cathodes, since the working temperature would be below the melting point of chromium.

The metallurgy of chromium is full of attractive possibilities, and the usefulness of pure chromium in the field of alloys is only beginning to be scratched; the scratching, however, is proving very much "worth while."

TITANIUM

Our friend, Mr. A. J. Rossi and the Titanium Alloy Manufacturing Company of Niagara Falls are the *alpha* and *omega* of the titanium industry. Nearly 15 years ago my first introduction to Mr. Rossi was in the historic barn, at Niagara, the cradle of so many of the Niagara Falls industries. Mr. Rossi was running the electric furnace and Mrs. Rossi was in the little laboratory, making the necessary analyses.

Everyone knows of the enormous masses of titanic iron ore in northern New York and Canada, which contains so much iron and so little sulfur and phosphorus, that every blast furnace would be glad to get it if it was not for the titanic oxide which makes it so unworkable that you could not give it to them. Mr. Rossi tried first to extract the iron only, throwing away the titanium in the slag, but that was not profitable. He then turned to producing ferro-titanium alloy, for use in steel and cast iron, and by indomitable perseverance has made that a success. A finely written booklet of over 100 pages, published by his company and to be had for the asking, tells the whole story, so why take up time to rehearse it here? Get the booklet and read it. In addition to titanium treatment of steel, to deoxidize and denitrogenize, this company also makes a specialty of titanium-treated aluminum bronze, also of titanium-treated bronzes and brasses of various compositions.

With titanic iron ore carrying 10 to 15 per cent of titanium as cheap as iron ore, and if iron-free material is required, rutile, 60 per cent titanium, at 5 to 7 c. per lb., there is no lack of cheap raw material. If uses are found for pure titanium, however, some other than the electric furnace must be used to reduce it, because, in absence of iron, titanium carbide would result. The methods for producing pure titanium are, like its prospective uses, still in the future, but they nevertheless are worth study and work. One can buy titanium metal now at about the price of silver, but if the problem were properly faced it could probably be made as cheaply as chromium.

MOLYBDENUM

Molybdenum sulfide, MoS_2, 60 per cent molybdenum, looks almost exactly like graphite, and is about as widely distributed. Normally, it can be purchased as 90 per cent concentrates at 25 c. per lb. This would make the raw material for 1 lb. of molybdenum cost nearly 50 c.; the selling price of the metal is about $2.00. This leaves a large margin to pay for reduction. In fact, $1.00 per lb. of contained molybdenum ought to return the reduction works a good profit.

However, the principal need of the molybdenum industry is a better utilization of its sources of raw material. The deposits have not been, in general, properly prospected or opened up, and then not properly worked. They are usually low-grade propositions, with 5 to 10 per cent of molybdenite disseminated through hard rock. This calls for careful study of crushing and concentrating methods, so as to minimize waste and loss. In most cases, the actual treatment falls far short of this, and possibly half the molybdenite in the ore is lost. The producers of the concentrates are being paid high prices for their material, but the market is limited and dull. If molybdenite were sold cheaper, there is little doubt that ferro-molybdenum, 50 to 85 per cent molybdenum, could be sold at half its present price, and the uses of molybdenum in steel correspondingly increased. New uses have also been found, such as the molybdenum wire so useful in electric resistance furnaces. This wire is scientifically very useful in that it resists the alloying action of many liquid metals even at very high temperatures. Dr. C. G. Fink, of the Edison Lamp Works, has studied these notable physical and chemical properties, and has described them in the *Transactions of the American Electrochemical Society*, 17 (1910), 229.

ZIRCONIUM

We may mention this element, which, as metal, is rare enough, but whose oxide has recently been found in considerable abundance. The familiar mineral Zircon is the silicate, containing nearly 50 per cent of zirconium, but it is found only in limited amount. In the last ten years the oxide Baddeleyite has been found in large quantities in Minas Geraes, Brazil, running 75 to 95 per cent pure, giving 50 to 75 per cent of the metal. This source is now so common that it sells at 4 to 5 c. per lb., and is being used in large quantities as a refractory material, on account of its high melting point (2000° C.), high resistance to all kinds of slags, low thermal conductivity and low coefficient of expansion.

The metal, however, is almost an unknown quantity. It has been obtained by the action of potassium or sodium on the anhydrous fluorides. Fused, it is white, density 6.4, melting point 1500° C., hard enough to scratch quartz. Ferro-zirconium has been made in the electric furnace, and used in small amounts in steel, with rather indefinite results. And yet, if some very useful properties of zirconium were discovered, the metal could undoubtedly be prepared at a reasonable price—only a fraction of the $5.00 per oz. now asked for it as a chemical curiosity. How it is to be obtained cheaply is one more of the interesting questions confronting the metallurgical pioneer.

CERIUM

There is a peculiar interest attaching to this metal and its close associates, from the fact that hundreds of tons of fairly rich cerium material is lying on the waste heaps of the incandescent mantle factories. The thorium ore used by these factories is Monazite—a phosphate of the cerium earths plus thorium silicate. On extracting the thoria, the residue is worthless for mantle fabrication. It is known as "Commercial Cerium Carbonate," and contains cerium, lanthanum, neodymium, praseodymium, samarium, gadolinium, yttrium, ytterbium, a little thorium, and considerable alkalies, iron, phosphoric acid and silica. By treatment with acids, precipitation of the rare earths, and ignition, a chocolate-brown mixture of oxides of the rare earths is obtained, in which cerium oxide predominates, and which contains nearly 50 per cent of metallic cerium. When this mixture is reduced directly, without further separation, to the metallic state, an alloy of the rare earth metals is obtained which is known as mixed metal (Mischmetal), or impure or commercial cerium. The composition of mischmetal naturally varies, but may be taken as approximately 30 to 50 per cent of cerium, 15 to 25 per cent of lanthanum, 10 to 15 per cent of the didymiums, up to 20 per cent of the yttrium metals, and 1 to 5 per cent of thorium.

An immense amount of laboratory and practical work has been expended on the production of mixed metal and of purer cerium. Professor Muthmann and his students in Munich, and Alcan Hirsch in the United States deserve particular mention for their articles, in *Liebig's Annalen* (1902 to 1910), and *Transactions of American Electrochemical Society*, **20** (1911), 1–102, respectively. Dr. Auer von Welsbach was the pioneer in the commercial manufacture and use of mischmetal. Annoyed by the sight of heaps of the cerium residues around his mantle factories, he experimented with their reduction to mischmetal and with the possible uses of the latter. Finding that it gave off sparks when scratched he conceived the idea of using it in automatic lighters, but found that it sparked far too feebly and unreliably to be practical. He then thought that if he purified the cerium it might give sparks more freely, but on making the purest cerium he found it to spark less than the impure metal. Turning in the opposite direction, he took mischmetal and added to it alloying metals not of the rare earth class, and found that they increased the sparking property. Iron, for instance, when increased to 30 per cent gave an alloy with remarkable spark-giving properties, such as make it most efficient and reliable in automatic lighters. This has formed the basis of the "pyrophoric alloy" industry, since although other metals have similar effects the 30 per cent iron alloy is probably the best sparking alloy, for general use, so far made.

The electrolysis of fused cerium salts, double chlorides or double fluorides, to give the melted mixed metal is carried on on a large scale in Austria at Treibach, in Germany near Berlin, and was commenced in the United States, in 1916, near New York City, by Mr. Hirsch and his associates. The technique is not easy to master, and all the works keep their operations as secret as possible. The principal difficulties are the re-solution of deposited metal, metal fog, and scattering of the metal as fine globules or "metal-mush" through the electrolyte, making it difficult to unite the metal to one melted mass. Since the latter trouble is largely due to surface tension, a study of this property, particularly how it can be diminished, might help in overcoming the difficulty. As an example of the opposite effect, the cupelling of lead on a bone-ash muffle depends absolutely, for its success, on the surface tension of the molten lead. But, metallic tellurium in quite small percentage *decreases* the surface tension of the lead so greatly that the metal *wets* the cupel and cupellation is rendered impossible. Arsenic, on the other hand, *increases* the surface tension of melted lead, and is therefore purposely added, about 0.25 per cent, to lead being made into lead shot, in order to make rounder shot. A physical study of such effects on cerium might well assist in overcoming the scattering of the metal in globules in the electrolytes. Another direction in which improvement might be made would be the careful study of the eutectics of mixtures of cerium salts with barium salts, so as to find an electrolyte of lower melting point in which the losses by re-solution of deposited metal would be less than they are at present. Another possible improvement would be the finding of an electrolyte which would dissolve cerium oxide or the mixed oxides directly, and give metal by electrolysis. Such a bath has been discovered for aluminum oxide, and a long experimental search for a similar bath for the cerium oxides would be amply justified.

The possibility of using the 30 per cent iron alloy as a melted cathode, and enriching it in cerium by electrolysis, might be considered. If we had the fusing point curve of cerium-iron alloys we could draw some useful conclusions in this direction. The addition of small amounts of other metals to the bath, so as to produce other useful cerium alloys directly, might facilitate the electrolytic operation. Further experience with the process, particularly by those familiar with the electrolysis of molten baths for sodium, magnesium or aluminum, will very probably lead to considerable improvements and reductions of cost.

The uses of cerium and particularly of its alloys are sure to increase, and may attain considerable proportions. It has been proposed by Borchers as an addition in small quantity to aluminum, to improve its properties. But the large use will always be the pyrophoric alloys, which have so largely replaced matches. Before the European war, over 3000 workers were employed in Austria in this industry of pyrophoric alloys and automatic lighters. Mistakes were made in the early days of the industry, and some alloys put into lighters which crumbled to pieces by the time the apparatus reached Australia, but continual improvement was being made, until a satisfactory substitute for matches was attained. The improvement most needed in the pocket lighting apparatus is to be able to dispense with alcohol or similar liquid; a wick impregnated with a *solid* combustible which can be ignited by a pyrophoric alloy, would give a great impetus to this art.

The author apologizes for making this paper so largely critical and prophetic. If it has sounded too prospective in tone, the excuse must be that it is inherent in the subject. Some of these elements are among the most common in nature, yet they remain so rare in practical life that they are still chemical curiosities. But the chemical curiosities of one age have frequently become the chemical commonplaces of the next age, and even within a generation some of us have seen this happen, under our own eyes. Let us then be foresighted, forehanded, and anticipate possible developments a little by such a general review as has just been given, so that when some wonderful changes and improvements are made in the next few years we may have the satisfaction of knowing that we glimpsed their shadows sometime before they arrived, and may even have assisted in hastening their arrival.

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RESEARCH[1]

By Arthur L. Walker

Research does not necessarily mean delving into abstruse problems that it is impossible for the average man to understand, neither does it mean work that is entirely governed by university regulations. Research is diligent investigation that will as a result improve our knowledge and condition whether it is in abstruse science or in the nature of an advance in the Arts which will add to the world's welfare and the usefulness of mankind. It covers a multitude of fields and may be carried on in numerous ways.

I remember a few years ago when I attended the initiation ceremonies of our sister chapter at Yale, George Borup was one of the initiates who had accomplished his work about as far away from the center of civilization as it is possible to get. But what could be more useful than the diligent investigation which helped to discover a way to an unknown portion of the world, in this case the North Pole, which had been the goal of many explorers and the cause of much suffering for nearly a century.

A practical engineer approaches the study of a new problem, whether it is the examination of a property, the design of a plant, the development of the science, or a study of conditions with the idea of improvement, in a true scientific spirit. He obtains facts, physical, chemical, economic, etc., and he must assemble these facts in a logical manner in order that deductions and conclusions may be arrived at upon which he can base his recommendations, his designs, or the theories which he uses as a foundation for the improvement.

The wonderful development which in the last few years has been made in lead smelting operations is due to careful investigations of the theory of smelting operations and the chemistry involved therein, as well as to the study of the theory of roasting ores preparatory to blast furnace operations. This work extended over a period of many years and was conducted at several plants in the Far West, Southwest, and in Mexico. It is gratifying to know that the investigator who conducted this work is a graduate of our School of Mines and this society has seen fit to recognize his services by admitting him into Sigma Xi this evening. I refer, of course, to Arthur S. Dwight.

In another field of work our colleague, Robert M. Raymond, has distinguished himself as an investigator of the conditions which surround mine development and mining operations. His success has won for him international fame of a high order, and we are glad to welcome him as one of our *Brothers in Zealous Research.*

While it can be truly said that the university is not the only place in which research can be conducted, it must be admitted that conditions which surround university life and university work are such that it should be the place where work of an investigative nature can be most successfully and effectively accomplished.

There never has been a time in the history of the world when the systematic investigation of problems, the solution of which will give us what we want or what is really necessary, has attracted so much attention. Never until the present has there been a time in the history of this country when the question of systematic investigation has commanded any attention at all from the people at large.

The various nations of the world are realizing that in order to protect themselves they must not lay on their oars and let others do the work of investigation which may result in the manufacture of what all the world needs. They too must work and improve and manufacture all products that they are dependent on, in order to be prepared for any unforeseen emergency, and be independent when that time arrives.

[1] Initiation address before the Society of Sigma Xi, Columbia University, April 18, 1916.

In this country we are beginning to awake to a realization of the extent of our dependence on others, and our attention is now brought to the fact that we should prepare to manufacture what we need. There are reasons why it has not been deemed necessary, in the past, for us to consider this question. The wonderful natural resources of the country have been conducive to lax methods; but we now begin to realize that these resources should be conserved, the products which have formerly gone to waste should be utilized, and that a study should be made as to how we can manufacture that which even the wealth of our resources does not provide.

While we as a nation have not considered the question of research as carefully as we should, and there has been a lack of the spirit of research as it is understood from the university standpoint, there has been *no* lack of diligent investigation, which has resulted in the enormous improvements which have added to the world's progress and the comforts of mankind.

Among the leading nations it is an undeniable fact that that nation which is supposed to have conducted the greatest amount of pure research is the nation which has shown the least amount of initiative in connection with the great discoveries of the world. And it can also be said, without boasting, that our own nation, which, up to the present, has done the least amount of pure research, has contributed more than any other to the great discoveries of the world. And as we look at the list of nations we see that the amount of pure research undertaken and original discoveries made are inversely proportional. This is an important consideration, something which should be thought of now and remembered in the future.

All of our large manufacturing establishments are either operating research laboratories on a large scale or are making plans for laboratories which will give them facilities for conducting research. Our universities are establishing laboratories on very much more extensive lines than heretofore thought of, and the question of a very large research laboratory for the government is being discussed. This wave of enthusiasm is quite recent, but if the work is properly conducted and coördinated it is bound to result in great benefit to this country, and to the world at large.

It has been said that a university cannot compete with the industrial laboratories of our large corporations. Probably this has been more or less true in the past, as the situation is such that the investigator comes in actual contact with the operations at the industrial corporations and thereby sees a great deal which aids him in the work he is performing. But a greater reason than this is that in most of our university laboratories the investigators are so tied down by routine work that they cannot devote a sufficient amount of concentrated time to investigation. On the other hand, the works laboratories are at a great disadvantage in that there is a large amount of duplication in spite of the fact that, at the present time, there is a freer interchange of ideas than ever before. The advantages which the university laboratories should have compared with the works laboratories are: (1) complete absence of secrecy; (2) the universal character of the work which can be done; (3) the facilities open to all; (4) the reduction of the expense of investigation; (5) the fact that unsuccessful work accomplished is more apt to be recorded and the data open for future reference.

In the advance copies of papers to be presented at the meeting of the American Electrochemical Society, in Washington, during the end of this month, on the subject of industrial research, some of the most distinguished scientists have expressed their opinion from professional, university, government, and corporation standpoints. The keynote of these expressions is "coöperation," but how coöperation can best be obtained is not definitely determined or agreed upon.

Undoubtedly all of our research laboratories at the present

time, whether they be in university or industrial plants, are operating under great disadvantages, either for lack of space or facilities in the case of the university laboratories, or for lack of time in the case of industrial laboratories. Will not these difficulties be properly solved by the creation of an industrial development laboratory under the auspices of some large university like Columbia? With the proper technical staff unhampered by excessive academic duties, work could be conducted in such a laboratory with a thorough knowledge of what is being done in the field.

Professional men with problems to solve could have them investigated, large corporations who have not the time to undertake investigations, and small concerns which have neither the requisite time nor the money for research could confidently avail themselves of the facilities offered by such a laboratory, and even the government might find it advantageous to utilize it for certain classes of work.

The magnitude of an undertaking of this character is enormous, but so would be the beneficial results obtainable in a proper industrial development laboratory, conducted on scientific, professional, and business lines. As Dr. Whitney facetiously suggests, to this laboratory could be brought "the poorest furnace slag to be quickly tried for farm fertilizer, tested in cements, made into glass, ground into paints, calendered into writing paper, blown into thermal insulation, turned into asbestos, put into dynamite, or injected into medicine."

No matter how much we may progress in research or industrial development work, let us remember that this work should not be controlled in such a way that the initiative the people of this nation now possess is made subservient to every-day plodding and investigation work.

COLUMBIA UNIVERSITY
NEW YORK CITY

CURRENT INDUSTRIAL NEWS

COAL-MINE FATALITIES IN THE UNITED STATES, 1870 TO 1914

Detailed information concerning coal-mine fatalities in the United States from 1870 to the close of 1914 is given in *Bulletin* 115 recently issued by the Bureau of Mines, Department of the Interior. In this bulletin more than 52,000 fatalities at coal mines have been classified by cause of accident, State, and calendar year. For the first time all of the States are placed on a calendar-year basis from the beginning of inspection in each State to the close of 1914. This report is a diagnosis of the mine-accident hazard and contains information that will be used as a basis of the study of accident prevention by operators, State and Federal officials and insurance companies.

Part I of the bulletin is devoted to the coal mines of the United States, in which the United States is treated as a unit by calendar years and by causes. It contains detailed information concerning accidents due to falls of roof, haulage, explosives, and electricity; a complete list of mine disasters in which five or more men have been killed since 1839; complete production figures, number of men employed and data relating to mining methods. There is also a study of mine accidents as related to machine mining, in which four groups of mines are maintained, namely, those in which 1 to 20 per cent of the coal is machine mined; 20 to 40 per cent machine mined; 40 to 60 per cent machine mined; and 60 per cent and over machine mined. These groups are tabulated by States and by years covering a period of 18 years, and include the number of men employed; number killed and fatality rates per 1,000 employed for each group of machine mines according to the foregoing classification.

The bituminous coal mines of the United States are treated independently of the anthracite so that fatality rates for the bituminous mines may be obtained.

Part II takes up the mine-accident question by States. Under each State is given a brief paragraph on the area and distribution of the coal fields; character of the coal beds; mining methods; reportable accidents and the organization of the inspection service. Data are given showing the production, number of men employed, number killed, number of machines in use in each State, and the percentage of coal mined by hand or shot off the solid, as well as the amount of coal mined per man, per day and per year. There is also under each State a list of the mine disasters that have occurred therein.—A. H. FAY.

QUALITATIVE AND QUANTITATIVE ESTIMATION OF GUM ARABIC

The most characteristic qualitative test for gum arabic is the precipitate with basic lead acetate. Mixtures of copper sulfate and caustic soda and of neutral ferric chloride and alcohol are of value as confirmatory tests. Basic lead acetate is conveniently made by the prolonged boiling of 40 g. crystallized lead acetate dissolved in 250 cc. water, with excess of litharge. The quantitative method adopted makes use of an alcoholic copper acetate-ammonia solution made up as follows: 50 g. copper acetate are dissolved in water, excess of ammonia added and the solution made up to 1000 cc., using water and alcohol in such proportions that the final solution contains 50 per cent alcohol. For each determination a 50-cc. portion of a gum arabic solution representing 0.25 g. of gum arabic was pipetted into a beaker, an equal volume of alcohol added and then 25 cc. of copper reagent, with constant stirring. The precipitate was allowed to settle, filtered on tared paper, washed with 50 per cent alcohol containing ammonia, then 70 per cent and finally 95 per cent alcohol. It was afterwards dried to constant weight at 105° C., ignited and the ash weighed. The amount of ash is deducted from the original weight and the difference called "net gum arabic." The amount of moisture in the gum originally must be allowed for; this is determined by drying in a current of hydrogen at 105° C. The method seems to give accurate results.—A. McMILLAN.

ZINC ORES

The last two numbers of the Bulletin of the Imperial Institute, London, says the *Mining World*, contain an interesting paper on the known occurrences of zinc ores in all parts of the world. Although containing a good deal of information of a familiar character, the paper gives a good and complete account of all well-known sources of supply. Of special interest at the present time is the information in regard to the deposits within Germany itself and in adjacent countries from which Germany could, in future, draw supplies in the event of her being cut off from the mines in allied countries. The paper concludes with notes on the valuation and smelting of zinc ores and the chief products yielded.—M.

MOTORS AND CAPSTAN CONTROL GEARS

The *Times Engineering Supplement* gives a résumé of the dimensions and other particulars of small-power motors for both continuous and single-phase alternating current from a list published by the British Thomson-Houston Company, of Rugby. The machines are made in two types. In one, standardized for 0.02 to 0.5 h. p., the continuous current motors are shunt wound up to 0.1 h. p., and compound wound up to 0.5 h. p.; and those for alternating current are of the split-phase, induction type with rotating field, the primary winding being placed on the rotor and connected direct to the line while the secondary winding is on the stator. The machines of the second type,

which are made in two forms, one for alternating and the other for direct current and are rated between $1/200$ and $1/15$ h. p., are designed with series characteristics and are suitable for cases in which the exact load to be connected is known. The same firm is also issuing a list describing control gears for electric capstans and winches which are designed to ensure that the motor is started up in the quickest time compatible with safety. The gear includes a pedal switch and an accelerating unit consisting of a series of contactors or electrically operated switches. The pedal switches, which do not carry the main current, but when depressed close the operating coil circuit of the first or shunt contactor, causing it to close and complete the motor circuit through the starting resistance, are made in two forms. One is intended for service in unexposed positions and the other for places where the switch is likely to be exposed to heavy rain or sea water or to be injured by vehicles passing over it. In the second form, the plunger passes through a water-tight gland and the pedal portion, when not in service, can be disengaged from the operating mechanism and dropped down flush with the ground. The switches are made in various capacities up to 25 h. p. and for circuits up to 550 volts.—M.

KIESELGUHR FROM AUSTRALIA

According to a report in the *Mining Journal*, there is at present a large demand in the United Kingdom for kieselguhr (infusorial earth) for use in the manufacture of dynamite, for metal cleaning and for sugar refining. The Imperial Institute, London, lately suggested to the Australian authorities that various samples of Australian diatomite should be forwarded for technical examination. Samples were sent from Victoria, New South Wales, Western Australia and Tasmania. Those from Victoria and New South Wales proved to be of most promising quality. In particular, samples from the Lillicur deposits, northwest of Ballarat in Victoria, found special favor.—M.

EUROPEAN BEET-SUGAR PRODUCTION

According to an estimate of the European beet-sugar production for 1915–16, the total yield is put down at 5,190,387 tons. Germany is credited with 1,500,000 tons, Austria with 1,011,400 tons, France with 140,000 tons, Belgium with 100,000 tons, the Netherlands with 230,000 tons, and Russia with 1,588,872 tons.—M.

ELECTRICAL CHARGING-WAGONS FOR COKE OVENS

According to the *Journal of Gas Lighting and Water Supply*, an installation of electrically driven wagons for charging coke ovens has been applied to a battery of 60 coke ovens, which can be kept charged by one man instead of 10 or 12 by the old system. The coal bins are not in a direct line with the axis of the track over the charging hoppers but through small curves down to 5 meters radius. The charging-set comprises 3 wagons of 3 tons capacity each. Each wagon is 4 meters long by 1.2 meters wide and 1.7 meters high and is divided into two parts with the usual drop bottoms. Between the two hoppers is the driving motor of 4 k. w. capacity which, together with the starting and the other switch gear, is enclosed in a dustproof compartment. Power is taken from an overhead trolley wire and the motor is controlled by a lever. After the three wagons have been filled at the coal bins, they are sent along in succession under their own power towards the ovens, the operator following on the last. When the wagons reach the coke-oven platform, the motors are switched off automatically and breaks applied by means of stops projecting from the track. These stops are placed by the operator at the particular oven it is desired to charge. The wagons are brought into the correct position and emptied. The operator then sends them along to the coal bins on their own power, closes the top of the filled oven and arranges the stop at the next oven to be filled.—M.

NEW SOURCE OF POTASH

In a recent number of the *Journal of the Society of Chemical Industry*, Mr. R. H. Ellis gives the results of an analysis of banana stalks. It was found that the ash contained no less than 45.9 per cent potash while soda was practically absent. The dried matter of the stalk is as rich in potash as kainit. The author calculates from his analysis that one ton of banana stalks would yield 188 lbs. of dried material containing 13.7 per cent of potash [K_2O] or 54 lbs. of ash containing 47.5 per cent or 25 lbs. of pure potash. If one considers the very large quantity of banana stalks at present classed as waste material, it is evident that municipal authorities should investigate whether this material could not be advantageously used as a source of potash.—M.

RESEARCH IN ILLUMINATING APPLIANCES

The Illuminating Engineering Society, London, says *Nature* (Vol. 97, No. 2429), has been considering the encouragement of researches of special utility at the present time and, at the annual meeting held recently, a report was submitted by the Committee on Research. A number of problems were mentioned and among these the following were included: researches in the quality of glassware required for illuminating purposes; the study of lighting appliances (globes, shades, reflectors, etc.); investigation of the conditions of illumination required for various industrial processes. Attention is also directed to the need for a series of standard colors of specified tints and reflecting value, the standardization of so-called artificial daylight, and the prescription of a standard method of testing the permanence of colors, all of which problems are of interest in relation to the dyeing and coloring trades. The list submitted includes some thirty sections and it would appear that the study of the subjects will afford work for many years to come.—M.

QUEENSLAND SHALE OIL

At last, says *Gas and Oil Power*, the shale beds in Western Australia are to be exploited. A first-class man and a modern plant have been secured from America so that the enterprise starts under good auspices. The derrick is the largest in Australia and is said to be better than any other oil derrick in the world. The cost of the experiment will be about $100,000 and the bore is a state monopoly. Originally the Ronia bore, which was put down in search of oil or natural gas, was begun as long ago as 1907 but, after a depth of 3000 feet had been reached, it was found that the spot located was not the right one. The new bore is about half a mile from the original hole. It was begun in November last and put down some 300 to 400 feet when the official opening took place. The expert says that he expects to strike oil at about 4000 feet.—M.

RAMIE FIBRE EXPORTED FROM KUIKIANG

Ramie fiber, says a consular report, is produced chiefly in Hupeh province though some is grown in the province of Kiangsi, notably in the Juichang district. The first crop is cut at the end of June and between then and November, two other crops are taken from the same roots, one in July–August and another in October–November. The fiber is divided into three qualities according to its length: (1) 30 to 40 in.; (2) 20 to 30 in.; (3) below 20 in.; this latter is purchased by the Japanese. The fiber is tied up in bundles bound together by a rope of ramie. It receives no treatment except in sorting for length but shippers at Hankow and Shanghai use hydraulic presses for treating the fiber. Japan, Belgium and France are the principal consumers, and shipments are made by Chinese dealers on account of foreign firms. The quantity exported in 1914 amounted to 100,883 cwts., being 15,024 cwts. less than that exported in 1913. The falling off is attributed to diminished shipments owing to the outbreak of the war and the absence of German buyers for the European market.—M.

PAPER PULP INDUSTRY

According to the *Paper Maker*, exhaustive trials of New Zealand timbers—mainly from the West Coast of the South Island—have recently been made and samples sent out of the Dominion to be tested by experts in Canada and elsewhere. These have satisfactorily proved that New Zealand timbers are eminently fitted for conversion into paper pulp and then into the paper used for newspapers. A company is about to be floated, which has secured the water rights of a large watershed as well as many thousands of acres of virgin bush covered with suitable timber, and much is looked for by experts from this new industry. Similar experiments on a substantial scale have been instituted by the Victorian (Australia) State Ministry regarding the production of wood pulp. The government printer uses, at least $400,000 worth of paper annually and the demand for private consumption is also considerable. There is, then, ample opportunity for the opening up of a large industry if it can be shown that Australian woods can produce the raw material.—M.

UTILIZATION OF WASTE PRODUCTS—IRON SLAGS

A fruitful direction, says *The Times Engineering Supplement*, in which national economy can be effected is the application of discarded refuse to industrial purposes. History affords numerous examples of the accumulation of enormous quantities of practically indestructible waste products forming unsightly heaps which scientific progress has ultimately shown to possess an intrinsic value. Among such collections are the great banks of slag produced in the manufacture of iron. Many unsuccessful attempts have been made to use up this slag in the manufacture of bricks, slabs, and cement but, at last, a practical method adopted in Germany for some years is being introduced and developed in England. A brick-making plant is working in full swing in South Wales and another one to utilize the slag banks in the neighborhood of Cleator Moor is being built. Again, the modern methods of road construction required to meet the demand of motor traffic have provided an increasing market for tar macadam for the manufacture of which broken slag is peculiarly suitable, and molded slag blocks are used both in Great Britain and in the United States in the construction of pavements.—M.

RUSSIAN BENZOL PRODUCTION

According to the *Oil and Color Trade Journal*, the cessation of the importation of benzol from Germany and other countries and the pressure of the strong demand of the Russian chemical industry have increased the production of this article in Russia during the last year and a half. The quantity produced in 1915 was a little over 9647 long tons, against 806 for 1914 and 113 in 1913. At the beginning of the war in the Donetz basin, which was the chief and practically the only source of this product hitherto in Russia, only 3 installations with 358 coking furnaces out of 1,082 at work were provided with benzol-producing equipment. During the first year of the war new concerns gradually sprung up with benzol factories and, at the end of last year, there were over 1,200 furnaces fitted for production of benzol and about 800 more were under construction. The quantity produced, however, does not nearly supply the demand of the market, which grows with the increase of chemical industry. The quantity produced in 1915 does not correspond to the capacity of the Russian equipment, some elements being able to work only up to 60 per cent of their capacity through want of labor, fuel, etc. In the Kuznetz basin there are large reserves of coking coal, and furnaces are being built with benzol-producing equipment. It is, however, questionable whether the demand can be supplied even with these latter furnaces producing material at an early date.—M.

DRY-POWDER FIRE EXTINGUISHERS

A committee was recently formed, says the *Journal of Gas Lighting and Water Supply*, one of the members of which was Dr. J. A. Harker, F.R.S., of the National Physical Laboratory, London, to conduct experiments to test the value of dry-powder fire extinguishers as compared with water and other first-aid appliances for extinguishing fires caused by bombs. The broad conclusions arrived at may be given from the report which has been issued. The substances tested were a particular form of dry-powder extinguisher, water and liquid extincteurs. In one case, a supplementary test was carried out with sand. It seemed that, while none of the agents employed had any material effect on the burning of the bomb itself, the spread of the fire caused was greatly limited and in some cases totally extinguished by the application of water whereas, after the application of the dry powder, the fire continued to burn and seemed to be checked only temporarily. Dry-powder extinguisher generally contains as its main constituent bicarbonate of soda, which on being heated gives off carbon dioxide but, according to the report, it is regarded as doubtful if the quantity produced is sufficient to have any material effect in the case of small fires. Water was found to be far more effective and it appeared that a given volume of water applied as a jet from an extincteur was more effective than the same amount applied in buckets. The application of sand was, weight for weight, found to be less effective than the dry powder. In view of the results, the committee are confident that by far the best extinguishing agent is a plentiful supply of water applied in the manner most convenient.—M.

HIGH-EFFICIENCY LAMPS

The new high-efficiency gas-filled lamp introduces variables not hitherto encountered in the photometry of incandescent electric lamps. On account of the comparative broadness of the filament spiral and the dissymmetry of the filament mounting, says the *Engineer*, there is considerable irregularity in the distribution of the light about the vertical axis. Consequently, when the lamp is rotated, as is commonly done in rating lamps at the factory, the light as seen in the photometer flickers so excessively as to render accurate measurements of candle power practically impossible without the use of an auxiliary apparatus. However, as is sometimes done, if two mirrors inclined to each other be placed at the back of the lamp, the flickering is so much reduced as to permit accurate candle power measurements even at very low speeds of rotation.—M.

OIL-PRODUCING NUTS AND SEEDS

The report of the committee on "edible and oil-producing nuts and seeds" of West Africa just issued affords, says *Nature*, an interesting glimpse of the way in which the British Government has changed its attitude towards science and industry since the war broke out. The exports of oil seeds from British West Africa in 1913 were valued at $35,140,000 and of this Germany took no less than $18,345,000 chiefly in the form of palm kernels, the crushing of which for oil and cake she had practically monopolized. With the outbreak of war, the West African exporters were deprived of their best customer and a new British industry in the crushing of palm kernels has been set on foot. The committee, which has issued the report, makes four recommendations with a view to keeping the industry in British hands and two of these are to be put into force immediately. The first is the imposition of an export duty of $10 per ton on all palm kernels exported from West Africa to ports outside the British Empire; the second is a recommendation for further coöperation between the West African Branch of Agriculture and the Imperial Institute, London, with a view to continuing investigations on the palm oil.—M.

SPANISH CHEMICAL AND COLOR IMPORTS

The *Oil and Color Trade Journal* gives the following figures for the imports in chemical and color materials into Spain for the years 1914 and 1915, the figures denoting quantities in metric tons:

	1915	1914
Alkaline carbonates, borates and silicates.............	3,213	2,022
Caustic soda and potash.............................	135	53
Mineral manures, ammonium sulfate.................	123,353	100,277
Superphosphate of lime and slag....................	116,897	63,013
Indigo and cochineal...............................	70	184
Dye extracts (vegetable)...........................	3,653	5,852
Colors, artificial, dry..............................	1,221	339

—M.

BORNEOL IN AMBER

In a paper read recently before the French Academy of Sciences, M. Reutter gives an account of his studies on several samples of amber. Of the samples examined, all were found to contain free borneol, the alcohol present in rosemary and spike lavender oils. The residue from ambers of Italian origin—one from Sicily, the other from Bologna—was found after extraction with ether and alcohol to be amorphous while that from German amber treated in the same way, was crystalline. Amber of Italian origin contains 1 to 16 per cent succinic acid whereas German amber contains 65 to 80 per cent of the same acid.—M.

SICILIAN SULFUR PRODUCTION

The figures of the Sicilian Sulfur Consortium giving the production of sulfur for the month of February, 1916, which are the latest official statistics, show a marked decline in the output. Sales, on the other hand, exceed those of the previous year, thus producing a reduction in the stocks. The production for February was 16,865 metric tons as against 24,799 for the corresponding month of 1915, while shipments for the month were 53,023 tons as against 35,121 in 1915. There were marked increases in the exports this year to the following countries: France, Great Britain, Greece, Turkey, Spain, Portugal and South America. Stocks in hand at the end of February were 258,766 tons as against 365,330 tons last year, being the lowest for sulfur stocks in Sicily since the consortium took over the industry.—M.

DETECTION OF CADMIUM

A 20 per cent solution of ammonium perchlorate in solution of ammonia of sp. gr. 0.90 is a sensitive reagent for cadmium which it precipitates as a white, crystalline double perchlorate of formula $Cd(ClO_4)_2.4NH_3$. In applying the test to the mixed sulfides of Cu, Cd and Bi as precipitated in the 2nd group, the precipitate is dissolved in nitric acid and the solution treated with excess of ammonia. Bismuth hydrate is filtered off and the blue filtrate treated with the perchlorate reagent which precipitates the cadmium even when five times as much copper is present.—M.

TRADE OPENINGS IN SOUTH CHINA

The British Consul at Kiung-chow (Hoi-how) reports that generally speaking the whole of the import business of Kiung-chow is done through importing firms in Hongkong, the center of supply for the South China market. Firms, therefore, who desire to place their products on the market and are not in a position to send out travelling agents of their own to push their sales direct, should endeavor to get into touch with importing firms in Hongkong through the Chamber of Commerce there or through the firm's own home offices. There is a good demand in South China for hardware, small lamps, fancy globes and cheap novelties of every description. The Germans have hitherto been very active in the following (among other) lines: sulfate of soda, brass electroplated lamp bowls, lamp glasses, burners, lamp frames (gilded), wicks, hanging lamps, imitation gold varnish, soaps (laundry and toilet) and plate glass (silvered and unsilvered).—M.

INDIAN STATISTICS

Vol. I of agricultural statistics for India, 1913–14, which deals with British India, shows a noteworthy steadiness of agricultural operations during recent years. In the last decade, the total area cropped—the areas sown with rice, millets, wheat, sugar, cotton, jute and oil-seeds—suffered slight fluctuations. The cropped area, which has been irrigated and the area devoted to food-crops have both increased, the former by 30 per cent. In the whole of India 80 million acres are sown with rice—an area which is ten times the acreage of Japan. The area under wheat, which measures 29 million acres, is only exceeded by the wheat acreage of the United States, while that devoted to the production of cotton, *viz.*, 25 million acres, covers a space equal to about two-thirds the cotton area of the United States. About one-eighth of the India area is cropped more than once a year. The exceptions to the general increase found are indigo and opium, the area for the cultivation of which has declined by about one-half since 1909. Recently the area devoted to cinchona was increased by one-tenth, this increase being due to a great extension of the cultivation in Bengal, the acreage having declined in Madras, which is the other chief growing district. Nearly half of the sugar cane is produced in Agra where the area under cultivation is being increased. One-third of the cropped area in Madras and the United Provinces, one-half in the Punjab and three-fourths in the district of the Sind depend upon irrigation, canals, tanks or wells for their water supply.—M.

BOARD OF TRADE

During the month of June, the British Board of Trade received inquiries from firms in the United Kingdom and abroad regarding sources of supply for the following articles. Firms which may be able to supply information regarding these things are requested to communicate with the Director of the Commercial Intelligence Branch, Board of Trade, 73 Basinghall St., London.

Aluminum plates for domestic use
Asbestos pulp
Asbestos, metallic yarn, three-ply
Asbestos sheets
Balances, pocket, cheap
Balls, hollow or solid, in brass or copper, $^1/_2$ in. to $^5/_8$ in. in diameter
Boxes, tin, round, with mirror top
Boxes, pill, cardboard
Brushes, tooth, xylonite, transparent
Candles in 1 lb. packets
Cane pulp
Capsules, metallic
Casein for glueing three-ply wool
Cases, cigarette, tortoise shell
Casks for soft soap in 28-lb. and 56-lb. sizes
Catgut, raw
Celluloid, bangles and rings
Cement, white for manufacture of tiles and Mosaic

Chemicals:
    Acetic anhydride
    Alum
    Arsenate of copper
    Benzyl alcohol
    Beta-naphthol-7-sulfonic acid
    Borax
    Calcium phosphate
    Magnesium acetate
    Oxalic acid
    Sodium phosphate and permanganate
    Tetrachlorethane
    Triacetin
    White zinc oxide
China and earthenware: cheap household goods; dinner, tea and toilet sets
Cork dust
Cutlery: Penknives
    Pocket knives
    Razors, ordinary
Drugs: Saponine
Dyes: Aniline for soap, dark brown, nut brown, maroon, purple, dark green, navy blue, pink, leather stains
Electrical apparatus, 2- and 3-cell flash lamp batteries

Filters, wine, vacuum apparatus, similar to Seitz make
Glass and Glassware:
    Amber disinfecting bottles
    Fruit preserving bottles
    Square screw cap bottles
    1-oz. bottles for boot cream
Gelatine, leaf, thin for process printing
Handles, bone and metal, for shaving brushes
Hinges, cabinet, small, $^1/_4$ in. to $^1/_2$ in.
Kapok
Locks, small

Machinery and Plant for:
    Making condensed milk
    Cutting corks
    Making capsules
    Making dominoes
    Extracting tapioca
    Making paraffin wax candles
    Making ice
    Electrolytic production of hydrogen
    Dating and stamping cans
    Crushing seeds
    Making buttons
    Refining oil
Ore, Caledonian, chrome
Paper, carbon
Paper, steel blue
Paper, tissue, pink
Pomades
Prussian blue
Rings, gas mantle, magnesia or fire-clay
Rivets, boot electro-brassed
Starch: Dextrine
    Fecula
Tubing, voice, flexible, metallic, as used in steamships
Tar, pine
Twine
Whale-bone, thin
Willows
Wire ropes, cast steel, flat, width, 90 × 100 mm.; thickness, 18 × 16 mm.; breaking strain, 55 tons

—M.

SCIENTIFIC SOCIETIES

A MASTER'S COURSE IN CHEMICAL ENGINEERING

By William H. Walker

The splendid new buildings of the Massachusetts Institute of Technology, located in Cambridge on the banks of the Charles River, were formally dedicated last month, and will be occupied at the opening of the school year next September. The greatly increased facilities for both instruction and research in all departments makes possible the execution of a number of plans for improvement which have been under consideration at the Institute for some time. Of these none is more important than a new Master's Course in Chemical Engineering, including a School of Chemical Engineering Practice, which is now fully organized.

It is unnecessary to rehearse to the readers of THIS JOURNAL the rapid rise, within the last decade, of Chemical Engineering as a profession. The curve of progress has turned even more sharply upward since the beginning of the European war, and the recognition of the importance of the chemically trained man in industry grows more generous every day.

The current journals are full of pertinent articles on the urgent necessity of preparedness—both military and industrial— and we need here but call attention to the fact that while the economic conditions which must obtain when the war is over are still unknown to us, there will under any condition be a most urgent call for men to maintain that place in the world's industry and commerce which the United States is now rapidly assuming.

To meet this demand there must be provided men of the broadest possible training. They must be well grounded in science, trained in the application of its principles to daily problems, have acquired engineering points of view and angles of approach, and have developed business perspective with relation to chemical industries. In short, there must be produced the *potential* chemical engineer. When a man has acquired a solid foundation in science, has become an accurate observer, an exact and logical thinker, and has developed a love for the application of this knowledge to the investigation and solution of the many problems which industrial practice presents, he has done marvelously well; but he still lacks much that goes to make the successful chemical engineer.

While it is true that the resourcefulness in applying theory to practice, the training in the solution of industrial problems, the general business perspective and self-reliance, which in time complete the man's education and make him a *creative* engineer is obtained in its entirety only by years of experience, there is much which other engineering branches have accomplished to render more quickly available the scientific education of the embryonic engineer. Without well-equipped mechanical and electrical laboratories it would be a long and difficult step to pass from the small apparatus and light load of a physics laboratory to the heavy responsibilities of a power plant. But in operating and testing the commercial-sized units with which mechanical engineering laboratories are easily equipped, a measure of self-confidence and an appreciation of responsibility are obtained not otherwise possible. In addition to all this the student translates for himself the fundamental principles of physics as exemplified in the laboratory into the application of these same principles in units and processes of commercial size and value.

But analogous facilities have never been available for instruction in Chemical Engineering. The important and ofttimes controlling difficulties inherent in carrying on a chemical process on a manufacturing scale, are not present when this same process is conducted in a chemical laboratory. To duplicate the apparatus which has been designed and built for factory operation is possible, and in many cases well worth while. But in order to study its performance the apparatus' must be operated—a process for which it was designed must be carried out. This procedure is obviously expensive and at best limited in its scope. A student cannot acquire the self-reliance necessary to operate a high pressure digester holding 25,000 gallons of acid by experimenting upon one of a few liters capacity. He cannot learn how to meet difficulties incident to handling a filter press, an electric furnace, a multiple-effect evaporator, a rotary kiln, and so on, if he has access only to such apparatus as the laboratory can provide. That experience which means power to execute comes only from contact with commercial-sized apparatus operating under the conditions imposed by practice.

Neither can the student obtain that training in the application of the principles of science to the problems of chemical industry on which successful industrial research so largely depends, without an opportunity to live with chemical processes which are being conducted on a large scale. It is true that the research chemist must be able to visualize the hitherto unaccomplished fact, but, on the other hand, the undreamed accomplishments of the future, while using methods and equipment now unrealized, will also undoubtedly, to a large extent, employ the apparatus and the processes of to-day, although along new and varied lines. A familiarity with what does and does not succeed in present practice is the best foundation for that soundness of judgment so essential in every research worker, and most especially in those who are to direct and guide the activities of our industrial research laboratories.

In view of the difficulty of developing the type of engineer outlined without greater breadth of method and of contact than is possible in our educational institutions alone, it is generally recognized that the industries themselves have a duty to perform in this regard, and that without their coöperation educational accomplishment must remain imperfect. On the other hand, no satisfactory scheme involving industrial coöperation with educational institutions has hitherto been proposed, and the conception and inauguration of such a scheme cannot but represent an educational advance of the first order. Within the last few years a comprehensive plan of great promise for the more adequate training of chemical engineers along these lines has been originated and developed by Mr. Arthur D. Little, a member of the Corporation of the Institute of Technology, and chairman of the Visiting Committee for the Department of Chemistry. The plan has been enthusiastically adopted, and is based upon the idea of a close coöperation between the Institute and certain selected manufacturing organizations in representative industries. It involves radical changes in the Institute curriculum for chemical engineering, and the location of a part of the Institute's instructional activity in the manufacturer's plant. As Mr. Little points out in his report to the Corporation of the Institute, "any chemical process on whatever scale conducted, may be resolved into a coördinated series of what may be termed 'unit-actions,' as pulverizing, mixing, heating, roasting, absorbing, condensing, lixiviating, precipitating, crystallizing, filtering, dissolving, electrolyzing, and so on. The principles underlying each of these unit-actions are the same, however different the materials operated upon may be. Thus in a gas absorbing system, the laws of countercurrent absorption which control the action of an ammonia scrubber do not differ from those involved in making sulfite digester acid. The number of these basic unit operations is not very large and relatively few of them are involved in any
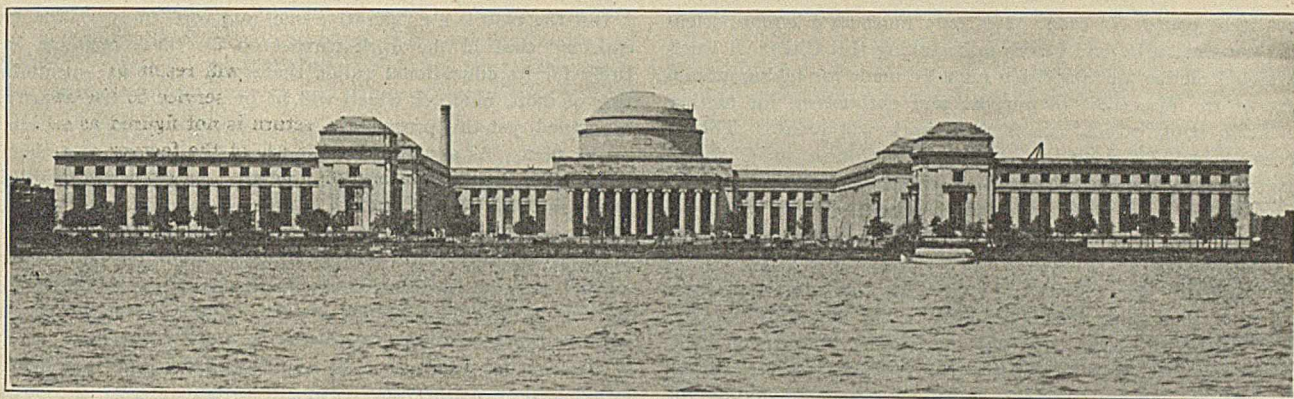
New Buildings of the Massachusetts Institute of Technology

particular process. The complexity of chemical engineering results from the variety of conditions as to temperature, pressure, concentration, etc., under which the unit-actions must be carried out in different processes, and from the limitations, as to materials of construction and design of apparatus, imposed by the physical and chemical character of the reacting substances. It is possible to so select a relatively few industries that there will be represented by them all of the important unit-actions of chemical industry."

Experience has shown that when proper provision is made, the performance of a piece of apparatus may be studied without materially interfering with its output. Thus the principles of multiple-effect evaporation or of countercurrent lixiviation can best be understood by making properly designed tests upon a working plant involving these unit-actions. In other words, it is possible, under careful regulation, to use a manufacturing plant as a chemical engineering laboratory, and not cut down its production or influence adversely the quality of product. Some of the advantages of such a method of engineering practice over a laboratory equipped only for instruction, are:

1—A first-hand knowledge of the machines with which the plant is equipped, operating under conditions imposed by practice, the processes involved being considered as unit-actions; the factors which control their efficient performance; and a general knowledge of the cost of chemical apparatus in its relation to cost of installation and operation.

2—A study of the unit operations of the plant and the process which is being carried on, as examples of the application of the principles of science to industry; their interpretation in terms of physics, chemistry and mechanics.

3—A general knowledge of modern methods of factory management and control, obtaining thereby some familiarity with the problems presented by the human element in industry.

4—The education which comes from taking part in the work of the plant; in acquiring some degree of self-confidence in handling industrial processes and large sized apparatus.

To carry into execution Mr. Little's idea, a School of Chemical Engineering Practice has been organized, which will increase in scope as experience is acquired. For the present it consists of five Stations selected to furnish opportunity in specific fields of broadest general importance in Chemical Engineering. Each station will include a small instructional laboratory, drafting room, and conference room and will be provided with a projection lantern, such special library, drawings, and models as may be needed. It will be in charge of a director, who will be a member of the Institute faculty.

STATION A—Located with the Eastern Manufacturing Company, Bangor, Maine. Here will be studied:

(1)—That most important application of the principles of electrochemistry to reactions taking place in solution, namely, the decomposition of common salt, with the formation of hydro-

gen, caustic soda and chlorine. The unit operations here available for study are electrolysis, multiple-effect evaporation with separation of a crystalline precipitate, caustic soda purification and concentration; absorption of gas of low partial pressure; agitation of both heavy and light liquids; and sedimentation of sludge.

(2)—The manufacture of bleached spruce pulp by the sulfite process. This is a high pressure-temperature reaction under careful analytical control, which involves most interesting equilibria.

(3)—The manufacture of poplar pulp by the soda process. In this process a study of the losses of soda through the cycle of operations is a most instructive one. It involves precipitation, sedimentation, filter press separation, systematic lixiviation and washing, and a determination of the soda lost in furnace flue gases by the Cottrell electrostatic separation principle.

(4)—The boiling, bleaching, and beating of rags for paper stock, and the manufacture of bond and ledger paper. This is both beater and tub sized, and is partly drum and partly loft dried. The opportunities for chemical engineering study here are obvious.

STATION B—Located at Everett, Mass., with the New England Gas and Coke Company. Without going into detail, the opportunities here offered are readily appreciated. The Company operates large by-product coke ovens and water-gas plants, with all the apparatus incident to the recovery of naphthalene, benzol, toluol, tar, ammonia, etc. Every opportunity is here presented to study those high temperature reactions of hydrogen with various hydrocarbons which have become so important in recent years.

STATION C—Located at Niagara Falls with the Carborundum Company. High temperature electrochemistry is here represented in all its phases. Powerful electrical furnaces of both the resistance and arc types are in operation making a variety of products. The apparatus for pulverizing, with accurate separation, is here seen at its best, systems of sieving and screening, hydraulic classifying, and pneumatic separation being in constant operation.

STATION D—Located at Stamford, Conn., with the American Synthetic Color Company. At this plant will be studied the chemical engineering of organic chemistry. Fractional distillation, sulfonation, alkali fusion, nitration, reduction, crystallization, with all the necessary apparatus, are but a few of the operations here available.

STATION E—Located near Allentown, Pa., with the Atlas Portland Cement Company. Large scale operations involving crushing and grinding in many types of machines are here offered. The chemistry of combustion and furnace control is nowhere studied to better advantage than in the mammoth kilns of this plant.

To obtain the greatest good from such splendid opportunities it is clear that this school must be a part of a consecutive and

consistent course of study. Not only must an adequate scientific foundation be laid before undertaking this Chemical Engineering Practice, but provision must be made for taking advantage of the directness of purpose and enthusiasm for further scientific study which the factory work will create. This is accomplished by providing a Master's Course in Chemical Engineering of five years' duration, including the entire summer between the fourth and fifth years. The first three years are identical with those of the four-year Bachelor's Course in Chemical Engineering as now offered at the Institute. At the end of the third year the student will elect either to finish the regular course of four years, receiving therefor the Bachelor's degree, or to spend two years and the intervening summer in study and receive both the Bachelor's and Master's degrees. From those students who elect this Master's Course, including the School of Chemical Engineering Practice, the faculty will select those whose attainments and character, as shown by their previous record of scholarship and by other information, are such as to indicate that the course can be creditably pursued.

At the middle of the fourth year the men so selected will be divided into five groups, and one group sent to each of the five stations for a period of six weeks. (Experience may prove that this is too short a time to accomplish the greatest good.) At the end of each six-week interval the groups will change stations, so that by September 1st each group will have occupied each station. After from three to four weeks' vacation these men will all return to the Institute for the fifth year of advanced work—graduating the following June. It must be kept in mind that the students going out into these industries do not go as employees of the industry, but as students in the School of Chemical Engineering Practice. It is intended that the work in the plant shall be wholly educational, and the men are to be under the control and direction, not of the plant organization, but of the director of the station—a member of the educational staff of the Institute. While it is intended to give the men as much industrial experience as possible, having them take shifts in the ordinary routine of the factory, such work will be directed throughout to secure the maximum educational result, and the interpretation of the experiences and results of work in the plant will be accomplished by conference and drawing room exercises which will take up no small fraction of the time spent in each station. It is expected in this way to avoid the weakness and inefficiency of previous schemes of coöperation by making no attempt whatever to have the student an economic asset to the industry, but planning his whole activity for its educational return to the student himself. An educational feature of great potential value to the industries which we shall hope to develop at these stations, and for which their equipment will be especially well adapted, is a type of extension course for the benefit of the foremen and selected employees. In these courses will be taken up the theory of the processes employed, and a study of the plant from the point of view of current practice in analogous industries. It is believed that these courses will react for greater efficiency not only among the employees, but the students making up the school.

To secure this high educational efficiency from the contact of the student with the industry, it will be noted that the student has completed substantially the equivalent of a four years' course in Chemical Engineering before entering the School of Practice. This will make possible the full appreciation of the significance of every fact and the interpretation of every experience in the light of fundamental principles.

The work of the fifth year is advanced in character, broad in scope, and almost wholly elective, the purpose being to take advantage of the students' enthusiasm and to allow him to specialize in the line in which he has found by experience that he has the greatest aptitude and interest.

But the plan is a coöperative one, and while it is confidently expected that in the work carried on by these students entirely for its educational value, there will result an accumulation of data, much of which will be of service to the industry concerned, yet this prospective return is not figured as an asset of the plan. As a return for the use of the factory as a chemical engineering laboratory the Institute proposes to establish and direct for each Company concerned, a research organization devoted entirely to the solution of its individual problems. While a laboratory will be maintained at each station, the extensive research facilities of the Institute will be available for work of a special character, and the faculty of the Institute will function as a Consulting Staff.

The earning power of industrial research is now too firmly established to require any argument to demonstrate the possibilities for coöperative service which the plan possesses. It is earnestly hoped that in it may be found an effective method by which science may be more closely linked to industry, for the lasting benefit of both.

MASSACHUSETTS INSTITUTE OF TECHNOLOGY,
BOSTON, July, 1916

FIFTY-THIRD (ANNUAL) MEETING AMERICAN CHEMICAL SOCIETY, NEW YORK CITY, SEPTEMBER 25 TO 30, 1916

A meeting of the American Chemical Society will be held in conjunction with the Second National Exposition of Chemical Industries, September 25 to 30, inclusive. A Council meeting is called for Monday afternoon and Monday evening. A general meeting follows on Tuesday morning, and on Tuesday afternoon it is hoped to have a public meeting in the large hall at the City College, with addresses by the President of the Society and by prominent men bearing upon "Chemistry and the National Welfare." On Tuesday evening a general "get-together" meeting or smoker will be held by the New York Section, complimentary to the parent Society, to which visiting chemists will be invited. On Thursday evening the Electrochemical Society will give a smoker, to which the members of the American Chemical Society will be invited, and on Friday evening a subscription banquet will be held in one of New York's large hotels.

Meetings of Divisions will be held on Wednesday, Thursday, Friday and Saturday mornings.

One of the special features of the meeting will be general conferences on special subjects in which the chemists of the country are now interested. The idea of these conferences is to have some important topics such as:

Glassware and Porcelain	Oils and Motor Fuels
Steel Alloy Metals	Convertibility of Plant
Paper and Its Utilization	Medicinal Chemicals
Dyestuffs and Their Relation to Munition Factories	
Industrial Alcohol, Acetone and Formic Acid	

The discussions will be started by well-known specialists in these lines. No set program is planned for these conferences, but it is believed from past experience that chemists interested in these various lines will get together, and many interesting points will be brought out which will be of mutual interest. The topics for these conferences have not as yet been determined upon, and suggestions are desired from members of the Society. These suggestions will all be placed before the Program Committee, and some six or eight topics selected therefrom. It is anticipated that two conferences will be in session each afternoon at the same time, one in the lecture hall of the Grand Central Palace, where the Second National Exposition of Chemical Industries will be held, and one in the lecture hall of the Chemists' Club.

The Divisions of Biological Chemistry, Physical Chemistry, and Industrial Chemistry will hold a joint symposium on colloids on Wednesday and Thursday mornings. On Wednesday morning the symposium will be of a theoretical nature, in which

the Industrial Division will not take part. On Thursday morning the symposium will be composed of industrial applications of colloid chemistry. A symposium on occupational diseases is also planned and is to take up part of one of the morning sessions of the Industrial Division.

REVISION OF OUR CHEMICAL STATISTICS

By Bernhard C. Hesse

In This Journal, **7** (1915), 58, it has been pointed out that our chemical statistics need revision; a general plan or outline of plan therefor was then presented. At the Seattle Meeting of the American Chemical Society in September, 1915, a committee was appointed to devise some practical and practicable way of obtaining such revision and of putting it into useful and serviceable effect. The organization of that committee was not completed until June, 1916. The reason for this delay is that service upon this committee entails a large amount of very difficult and most tedious labor, and men hesitate to assume such added responsibilities. Successful execution of this plan calls for coöperative thinking and planning on the part of all members of the A. C. S., of all our other technical and chemical societies and associations, and of all those engaged in chemical pursuits of any industrial or commercial aspect in this country.

Inspection of the lists of European countries for the past twenty years, so far as they relate to chemicals and allied materials, shows that these lists have been very greatly expanded and indeed much more so than our own.

In general, each European country seems to arrange its list with a view of telling its own people the most about other peoples and of telling other peoples as little about their respective affairs as is convenient, particularly in those items which are capable of high diversification. For example, the German list divides 120,000 short tons or $54,250,000 (= 21.5 c. per lb.) of coal-tar dyes into 4 items only; photographic chemicals totaling 3,080 short tons or $2,000,000 (= 32.5 c. per lb.) are lumped into one item; and 1,320 short tons or $5,300,000 (= $2.00 per lb.) synthetic pharmaceuticals also take up but one item. These 6 items total 124,400 short tons or $61,550,000 and average $495 per ton (= 24.7 c. per lb.) and cover fully 1800 different articles of manufacture, only few of them closely related; 123 short tons of various alkaloids and their salts of a total value of $1,750,000 or $14,220 per ton (= $7.11 per lb.) make up one export item.

On the other hand, Switzerland, in its lists, is more diversified as to its imports from Germany than Germany is in its list of exports to Switzerland; in fact, Switzerland seems to enter and report trade-marked or proprietary synthetics and the like as such; our own lists do this in isolated cases only.

German statistics for the calendar year are published within the first 5 weeks of the succeeding year; our own reports are two or three months later.

On its face, it seems plausible that if we are now to diversify our domestic chemical industry rapidly and with the fewest false steps and commercial or industrial calamities, we should be advised in some authoritative manner as to the nature of our domestic market as it is now and may develop to be in the future. If under these conditions we do not properly develop our markets and our domestic manufacture we at least cannot reasonably raise the defense that we did not do better because we had no way of determining what our country's needs were and therefore could not intelligently plan our independence from foreign supply-sources. I am encouraged in a belief in this utility and benefit by the rather large number of inquiries that have been made of me personally, orally and by letter, within the past 20 months, for information as to the domestic

consumption of this, that or the other imported article and in only the fewest instances have I been able to dig up an answer that filled the bill. It seems to me that there can be no debate on the general proposition that the more we know about our market requirements the more likely are we to build up a domestic industry. Add to this the fact that at comparatively little added expense our Federal Government, through one or more of its Departments, can gather and promptly publish such added information, and it seems to me to be conclusive that the chemists of this country should get together, make up a list of such specific added items concerning which they need information and indicate such further diversifications of items as promise to them the greatest help in the future. We can rest assured that the number of different chemical materials and products that this country will use and continue to use will be not at all diminished in the future but, quite on the contrary, will be greatly and very likely rapidly increased. The longer we put off such diversification of our lists the harder the task of future diversification will become; it will certainly not become any easier. Such a request for added information has nothing in common with asking for a subsidy or a bonus or added tariff protection; it is simply a request to be better informed as to the requirements of the public so that we may more intelligently work towards our nation's industrial independence of foreign supply-sources; in so helping us the public will be helping itself.

In presenting to the proper authorities a request for added information we must make as reasonably sure as possible that whatever is presented is not half-baked but is as mature as circumstances and conditions will permit, that it meets with and embodies the views of the great majority of those likely to be affected by this information and that there is a reasonable stability to the list; such request should be in as definite, final form as possible.

Clearly, the constructing of such a request is by no means whatever the duty of any one man or of any small group of men; it is a duty in which every person in the country engaged in the making, selling and distributing of articles of this class fully shares; in fact it *is* everybody's business. The committee of the American Chemical Society can act merely as a clearing-house for requests and then put them into a form that will be likely to receive the sympathetic attention and enlist the active coöperation of the officials who must supervise the details of gathering and publishing such material.

As Chairman of this special committee of the American Chemical Society, I have prepared a consolidated list (List I) of articles used or produced in or by chemical industries as they appear in the most diversified commercial reports of Austria, France, Germany, Great Britain, Italy, Sweden and Switzerland; italicized entries are to be found in the most diversified U. S. lists; black entries are additional items in our own U. S. lists as given in *Monthly Summary of Foreign Commerce of the United States* and *Quarterly Statement of imported merchandise entered for consumption in the United States*. In noting these entries it must be remembered that not all those entries appear separately; in many cases they are separated, but in the majority of cases they are lumped in with a large number of other items and segregation into values is not feasible and generally impossible; this is also true of List III.

In working with List I it must be borne in mind that in few cases only has any alphabetical arrangement been attempted for the reason that in the original lists little or no regard to alphabetical arrangement is had; the items seem to be arranged according to some commercial connection, real or assumed, between the various items. Further, that extreme niceties as to logic of classification cannot be considered by those who, in the final event, must gather the information; that repetition *must* be avoided and no material can be reported more than once.

List I—Articles Produced in or by Chemical Industries: Taken from Most Diversified Lists Issued. European (Roman Type); Imported Merchandise Entered for

I—Malt, Oil Fruits, Industrial Plants, Fruits & Plant Juices
Beans: Soya, vanilla
Malt, excl burnt & ground
Rapeseed
Castor beans
Poppy & sunflower
Linseed & meal
Cocoa, crude
Palm kernels
Licorice root
Kopra
Hemp seed
Hops
Madderroot, Quercitron & other dye plants
Sugar-beet-seed
Opium
Strychnine & its salts
Kino
Aloe & other fruit & plant juices

II—Vegetable Products for Industrial or Medicinal Uses
Cinchona bark
Rhubarb root
Gentian root
Chocolate
Curcuma
Cudbear
Caraway, Iceland moss & other crude lichens; Tamarinds, stick-cinnamon; berries, leaves, flowers, etc., for medicinal uses.
Insect powder & flowers
Tea for caffeine
Vegetable waxes in natural state
Wood for wood pulp, etc.
Wood charcoal & powder, charcoal briquettes
Wood, flour & excelsior
Roots for distillation. Roots for medicinal use

III—Dyewoods & Tanning Materials
Logwood
Fustic, Brazil wood
Cam-wood
Orchil
Ground or fermented dyewoods
Quebracho & other woods, in blocks; ground
Algarobilla, Dividivi & other n. s. p. f.
Valonia
Nutgalls
Myrabolans
Sumach
Catechu (brown & yellow) *Gambier* (crude or refined)
Oak bark
Coniferous barks
Mimosa, *mangrove*, maletto & other tan-barks
Oak, pine & *chestnut*-extracts
Nutgall-extract
Quebracho-extract
Sumach-extract
All other tanning extracts

IV—Resins, Lakes, Varnishes & Putties
Lakes, dry or pulp
Turpentine resins
Kauri & other copals
Damar, akaroid & other resins, olibanum & other soft resins; gum-resins
Gum-lac
Gum, chicle
  guayule
Shell-lac
Acacia, ocajon, cherry, cutera & bassora-gums
Extract Persian berries
Tragacanth
Scammony
Oil varnishes, Bird-lime ex linseed oil
Spirit varnishes. Shellac-putty
Lacquers (non-spirit) asphalt varnishes, *coach varnishes*
Sealing waxes
Putty & putties n. s. p. f.
Asbestos paints, asbestos pigments, asbestos putty

V—Caoutchouc & Camphor
Caoutchouc, crude or refined
Gutta percha, crude or refined
Balata, crude or refined
Insulating compound
Rubber, scrap or waste
Caoutchouc substitutes
Camphor & manna

VI—Animal & Vegetable Fats & Oils
Hog lard
Lard, neutral compounds & substitutes
Oleomargarine
Goose-fat, *beef marrow*, etc.
Grease: enfleurage, soluble

Prime beef tallow
Beef & mutton tallow
Bone-fat, fat-waste, stearin-pitch
Fish, whale & seal oil
Fats, etc., from fish, whale & seal
Horse fat, deer-tallow, etc.
Rape oil
Linseed oil
Bean-oil
Peanut-oil
Sesame oil
Olive oil
Lavat & sulfur oil
Cottonseed oil
Wood tar oils
Castor oil
Oil.: Almonds (sweet)
  Birch tar
  Cajeput
  Chinese nut
  Cinnamon
  Citronella
  Cod
  Cod liver
  Eucalyptus
  Hempseed
  Origanum (red & white)
  Poppyseed
Beech, bone, *corn,* poppy, sunflower and other fatty oils
Fat-oils in tins, bottles, etc.
Cocoa butter
Cottonseed stearin
Palm oil (palm butter, -fat, etc.)
Palmkernel oil & fat
Cocoanut oil, fat & tallow
Vegetable tallows
Vegetable ivory
Olein
Margarine
Edible vegetable tallow
Oil Cake: Corn
  Cottonseed
  Cotton & linseed
  Mustard
  Peanut
  Rapeseed
  Soya bean
Oil cake meal
Oleostearine

VII—Animal Products
Extract, meat
Egg-yolk
Egg-albumen
Bees & other insect. waxes, honeycombs
Gelatine
Spermaceti
Fish glue, *agar-agar*
Glue
Glue-size
Glue-waste
Bones, horns, hoofs, etc., not for cutting purposes
Horn-waste for fertilizer
Galalith
Oil cloth
Linoleum
Bone & other animal blacks; bone-ash
Dried blood

VIII—Starch, Sugar
Potato starch green or dry
Rice starch
Corn, wheat & other starches
Starch gums, dextrins, burnt starch, paste, adhesives & gluten-meal
Beet sugar
Beet-Sugar: Flats, sticks & cubes
  Ground & lump
  Ground refined
  Loaf & brown
  Confectioners'
  Raw, solid or liquid
Cane sugar
Cane sugar, raw, solid or liquid
All other solid & liquid sugars
Sugar (cane & beet) tank bottoms, syrups, etc., up to 1000
Sirups & molasses
Glucose (syrup)
Starch, fruit & other fermentable sugars, burnt sugar
Caramel, solid & liquid
Grape sugar
Invert sugar
Maple, sugar & syrup
Milk sugar

IX—Alcohol, Vinegar, Yeast & Mineral Water
Alcohol, in barrels
Alcohol in bottles
Vinegar of all kinds
Methylated spirits
Wine yeast
All other yeasts
Mineral waters, artificial, etc., incl., bottles

X—Mineral & Fossil Raw Materials
Alunite
Clay, common, potters, fire & pipe
Kaolin & china clay
Burnt clay, chamotte, Fayence, etc.
Yellow, ochre, bole, Sienna & Verona earths
Other earth-colors, artificial oxide iron, crude
Chalk, white, crude
  Purified
Graphite, crude, ground & washed
Pumice, Tripoli, crude, ground, washed or in bricks or tile
Emery, crude, ground or washed
Mineral abrasive, polishes, & cleansers, crude, ground, or washed
Enamels & glazes
Kieselguhr, quartz-sand, firestones
Lime, slaked, limestone or lime mortar
Magnesia
Magnesite, native or burnt
Witherite, native or burnt, strontianite
Lime phosphates (Apatite, phosphorite, coprolite, etc.)
Gypsum; gypsum-superphosphate
Puzzolan, etc.
Portland & Roman cements
Gypsum cement
Keene's cement
Ground lime; tripolith
Asbestos, crude
Venetian chalk, crude, ground or burnt
Talc, crude, ground or burnt
Mica, crude
Barytes & Coelestite
Feldspar, ordinary
Fluorspar, crude; *Cryolite,* natural
Borocalcite & Tincal
Monazite-sand
Mantles (gas, oil, etc.)
Fuller's & other earths & bituminous shale

XI—Ores, Iron, Slags
Antimony ore & matte
Antimony regulus
Arsenic ores
Bauxite, concentrates
Lead-ores
Cerium-ore
Chrome-ores
Cobalt-ore
Iron-ores
Gold-ores
Corundum
Copper-ores inclusive of pyrites cinders
Copper regulus
Kainite
Kieserite
Manganese-ores
Nickel-ores
Platinum-ores
Pyrites (Pyrrhotite, marcasite, etc.)
Silver-ores
Tungsten ores
Zinc-ores
Zinc sulfide
Tin-ores
Uranium, molybdenum & other ores n s. p. f.
Uranium oxide
Gas-purifying masses containing iron or manganese; slags, slag felts, slag wools; ferrocyanide, sludge; ashes, pyrites-cinder, etc.

XII—Fossil Fuels
Anthracite, bituminous & cannel coal
Lignite
Peat, peat-coke & peat fuels
Coke
Coal—briquettes
Culm
Lignite—briquettes
Coke-like residues & artificial fuels
Carbon electrodes
Incandescent lamps, carbon or metal filament
Carbons, formed & crude; vegetable carbon

XIII—Mineral Oils & Other Fossil Raw Materials
Asphaltum & bitumen
Lubricating & vaseline oils
Crude petroleum; pitch & natural liquid asphalt
Heavy gasolines, turpentine substitutes

Gas oils
Dead oils
Refined petroleum
Crude gasoline
Benzine, gasoline, ligroin, petroleum ether & similar refined light-oils
Lignite, peat and shale-oils & other not otherwise specified mineral oils, tars & pitches
Paraffin, crude & refined
Soft paraffin
Solid asphalt & asphalt bricks
Asphalt-mastic, pitch, resin & wood cement
Ozocerite & mountain-pitches, crude
Refined ozocerite, ceresin
Pitch & black wax
Pitch-like petroleum residues heavier than water
Marine glue pitch
Shale-pitch; lignite tar, peat tar, wood tar, birch tar
Oil & water-gas tar
Gas liquor

XIV—Coal Tar Oils & Products
Coal tar
Carbolineum
Coal tar pitch
Benzol, toluol, cumol, etc.
Anthracene, carbolic, creosote & other heavy coal-tar oils & asphalt-naphtha
Solvent-naphtha
Naphthalene
Anthracene
Phenol, crude or refined
Lime carbolate
Pyridine bases
Cresol, crude carbolic, 100 per cent
Arseniate
Anilin: Oil & salts
Dimethylanilin
Benzyl chloride
Naphthol, naphthylamin
Naphthol compounds
Anthraquinone, *nitrobenzol, toluidin, resorcin, phthalic acid & other coal-tar products*

XV—Waxes
Prepared bees, & other insect waxes
Prepared *vegetable waxes;* vegetable wax waste

XVI—Soaps and Fat Products
Ordinary soft soap; oils & fluid fats, Turkey-red oil; liquid Creolin & like cleansers; soap substitutes in barrels
Hard soaps; Creolin & like hard cleansers; soap substitutes & evaporated lyes not elsewhere specified
Cake soaps; liquid soaps, soap powders, papers & other soap substitutes
Soap: Castile
  Medicinal
  Toilet, perfumed
  Toilet, unperfumed
Stearin, palmitin
Tapers, candles & night-lights
Phonograph plates & rolls of wax or ceresin
Crude glycerin
Refined glycerin
Soap waste lyes
Paraffin ointment, *vaseline, vaseline ointment, lanolin & lanolin preparations*
Rosin-soaps
Washing powder & fluid
Degras
Axle-grease
Other lubricants made from fats or oils
Shoeblacking, solid
Yellow shoe polish
Polishes made of fats, oils, soaps; alumina soap, artificial polishes
Stearin, palmitin, etc., formed

XVII—Chemical & Pharmaceutical Products
Abraum salts:
  12-15 per cent K₂O
  More than 15-19 per cent K₂O
Abraum salts n. s. p. f.
Acetanilid
Acetates, all other n. s. p. f.
Acetone oil
Acetphenetidine
Acid: Acetic
  Acetylsalicylic
  Arsenic
  Arsenious
  Benzoic & sodium benzoate
  Boric
  Chlorsulfonic

U. S. (Italics); Additionals Contained in "Monthly Summary of Foreign Commerce of the United States" and "Quarterly Statement of Consumption in the United States" (Black)

Acid (*Continued*):
  Chromic
  Citric
  Formic
  Glycerophosphoric
  Hydrochloric (nitrohydrochloric)
  Hydrofluoric
  Lactic
  Nitric
  Oxalic
  Phosphoric
  Prussic
  Pyrogallic
  Salicylic & sodium salicylate
  Sulfuric:
    Non-fuming
    Fuming
    For fertilizers
    Spent
Tannic & gallic
Tartaric
Valerianic
Acid, Anhydrides
  Acetic
  Sulfuric
Alkali & other metals not specified
Alkaloids, all other, their salts & compounds
Alum: soda, potash or ammonia chrome, iron or copper
Aluminum: Acetate
  Chloride
    Hydrate, artificial
    Oxide, artificial
    Sulfate
    Sulfite
    Sulfocyanide
Ammonia water
Ammonium: *Carbonate*
  Chloride
  Fluoride
  Nitrate
  Perchlorate
  Persulfate
  Phosphate
    Sulfate
Amyl acetate
  nitrite
Anilin compounds, not dyes
Antimony: Fluoride
  Oxalate
    Regulus or metal
  All other compounds n. s. p. f.
Antipyrene
Antitoxins
Arc carbons
Argols, crude or refined (soda or potash)
Artificial & natural mineral water salts
Artificial balsams, extracts & waters, not perfumes
Arsenic: Metal
    Sulfide
  Compounds, all other n. s. p. f.
Aspirin
Baking powder
Barium: Chlorate
    Chloride
  Hydrate
  Oxide
  Nitrate
    Peroxide
  Sulfide
Beetroot potash
Benzoates n. s. p. f.
Bismuth salts
Borax
Bromine
Bromides of potash, soda, ammonia
Bromides n. s. p. f.
Bromoform & Iodoform
Bromine, organic compounds n. s. p. f.
Caffeine
Calcium: *Carbide*
    Chloride & liquors of
    Nitrate (air)
Tartrate
Carbon bisulfide
Carbon tetrachloride
Carborundum
Carborundum discs
Carborundum paper
Carborundum, other manufactures of
Carbides n. s. p. f.
Carnallite, 9–12 per cent K₂O
Casein & non-edible casein preparations
Chemical products, n. s. p. f. for photographic, cleansing & other uses
Chloral hydrate
Chlorates n. s. p. f.
Chlorine compounds, organic n. s. p. f.
Chloroform

Chrome: Acetate
  Chloride
    Oxide (hydroxide)
Chromous chloride
Cobalt oxide
  Cobalt salts n. s. p. f.
Cocaine, crude & refined
Cocaine, salts of
Cellophone
  Collodion & celloidin
  Copper sulfate (copper & ferrous sulfate)
  Creosote, wood-, peat & tar
  Cryolite
  Cyanides (potash or soda)
  Disinfectants
Ecgonine
  Egg albumen, dried & powdered; albuminous materials
  Epsom salts
  Ethyl bromide
  Ethyl chloride
  Ethyl iodide
  Ferrosilicon with 25 per cent Si or more
  Fertilizer salts inclusive of potash fertilizer with 30 per cent K₂O
Filter-tubes
  Fluorides
  Fluosilicates
  Formaldehyde
  Formaldehyde-sulfoxylates
  Fruit & plant juices containing ether or alcohol
Gases, Compressed: Acetylene
    Hydrogen
    Nitrogen
    Oxygen
    All other
  Liquefied: Acetylene
    Ammonia
    Carbon dioxide
    Sulfur dioxide
    All other
Gelatine
Gelatine capsules
Glue: *Crude*
  Other than egg
  For printing rolls
Gold: Chloride
  All other salts of
Green: *Schweinfurth & similar*
Guaiacol carbonate
Hydrogen peroxide
Hypochlorites n. s. p. f.
Industrial chemicals n. s. p. f.
  Inorganic
  Organic
Inorganic chemicals for medicinal use n. s. p. f.
Insecticides
Iron: Acetate
  Chromate
  Lactate
  Mordant
  Sulfate
  Sulfide (artificial)
Iodine
Iodides of *potash*, soda & ammonia
  All other
Lactates n. s. p. f.
Lead: *Chromate*
  Nitrate
  Oxide (litharge) with or without gold & silver
  Peroxide
  Red oxide of
  Sugar of
  Sulfate
  All other salts n. s. p. f.
Licorice extracts
  Crude or refined
  Medicinal
Lime: *Acetate & gray acetate*
  Chloride of & bleach liquors
  Citrate of
Magnesia: Carbonate, artificial
  Chloride & liquors of
Manganese chloride
Medicinal & pharmaceutical preparations, n. s. p. f.
Mercury: Metal & alloys thereof
  Chlorides
  Metalloids n. s. p. f.
  Methyl-ethyl ketone
  Morphine & its salts
Nickel: *Oxide*
  Sponge
  Sulfate
Nicotine
Nicotine, salts of
Nitrates of *thorium*, cerium & rare earths
Nitrates n. s. p. f.
Opium alkaloids
Organic chemicals for medicinal use, n. s. p. f.
Oxides n. s. p. f.
Perchlorates

Persulfates
Phenolphthalein
Phosgene
Phosphorus, red & yellow
  Phosphorus hydride
  Phosphorus sesquisulfide
Photographic chemicals n. s. p. f.
Photographic dry plates & films
Potassium (or Potash):
  Arsenate
  Bitartrate
  Carbonate
  Caustic, solid or liquor
  Chlorate
  Chloride
  Chromate & bichromate
  Magnesium sulfate
  Manganate & *permanganate*
  Nitrate
  Oxalate
  Phosphate
  Prussiate yellow or red (sodium)
  Sulfate
  Sulfide (soda)
  Sulfocyanide
  Salts, all other n. s. p. f.
Proprietary medicines
Quinine, its salts & compounds
Rochelle salts
Saccharine
Salicylates n. s. p. f.
Salicine
Salol
Salt
Salt brines & bitterns
Santonin
Saponine
Selenium sludge
Sheep-dip
Silk, artificial
  Nitro
  All other
Silver: Bromide
  Chloride
  Nitrate
  All other salts
  All other compounds
Sodium (or Soda): Acetate
  Aluminate
  Arsenate
  Ash, crude and refined; boiler scale preparations containing soda ash; washing soda
  Bicarbonate
  Bisulfate (nitre cake)
  Caustic solid or liquid
  Chlorate
  Chromate or bichromate
  Crystals (soda)
  Hydrosulfite
  Hyposulfite
  Nitrate
  Nitrite
  Peroxide
  Phosphates
  Silicofluoride
  Sulfate (salt cake)
  Sulfhydrate
  Sulfide
  Sulfite & bisulfite
Strontium: Carbonate (artificial)
  Chloride
  Oxide
Sulfides, all other n. s. p. f.
Sulforicinates
Sulfur, Spence metal
Sulfurchloride
Synthetic or artificial foods or nutrients
Tartar emetic
Terpene hydrate
Thorium, cerium & zirconium, all other salts of
Thymol
Tin, chlorides of
  Oxides of
  All other salts of n. s. p. f.
Tungsten dioxide
Urea
Water glass (potash or soda) liquid
Water glass (potash or soda) solid
Welsbach mantles, scrap
Zinc: *Chloride*
  Sulfate
  Salts, all other n. s. p. f.
  All other chemicals & pharmaceutical products

XVIII—Dyes & Dye Wares
Cochineal
Lac-dye
Animal kermes, *cochineal*, carmine, sepia
Bleacher's blue
Coal-tar dyes
Aniline dyes
Azo dyes
Sulfur dyes
Azuline, fuchsine & rosein dyes
Naphthalene dyes
Anthracene dyes other than alizarin red

Alizarin red
Indigo vegetable & synthetic
Indigo carmine & lakes of indigo & indigo carmine
Food & butter colors
Prussian blues, chrome-green & zinc-green
Ultramarine & ultramarine lakes
Cobalt oxide
Litharge
Orange mineral
White lead
Blanc fixe
Zinc oxide (white)
Zinc dust
Lithopone
Zinc oxide (gray)
Red cinnabar
Copper oxide
Logwood extracts
Extracts of fustic, Brazil wood, Quercitron & other dye woods
Chalk, white, washed, etc.
Iron oxide, native & artificial (also yellow ochre)
Umber, Sienna & other n. s. p. f. earth colors
Dry, not prepared printing ink lampblack
Blanco polish
Bronze & metal colors
Chrome colors
Copper pigments & other n. s. p. f. pigments & lakes, dry or pulp
Colored printing inks; cheese colors, orseille extract, chlorophyl & other n. s. p. f. prepared colors
Printing ink, black
Lead, zinc, lithopone, iron oxide ground in oil, in tins or otherwise packed for retail; all other prepared colors
Smalts
Colors in color boxes, water-colors, etc.
Ink, ink-powder
Graphite, formed for retail
Earth, Van Dyke
Zinc chromate
Lead & colored pencils or crayons; chalk crayons
All other dyes & dye wares

XIX—Ethers & Alcohols
Acetic ether
Amyl ether
Butyric ether
Ethyl ether
Ether, nitrous
Ethers of all kinds; cognac-oil
Fusel oils; amyl, butyl, propyl alcohols
Crude wood alcohol
Crude acetone
Refined *acetone, wood alcohol* & water solutions of formaldehyde
Acetaldehyde, paraldehyde

XX—Volatile Oils, Artificial Perfumes, Toilet Preparations
Wood tar oil, caoutchouc oil; animal oil
Turpentine oil, pine-needle oil, *rosin spirit*
Orange, lemon, bergamot & other citrus volatile oils
Camphor, anise, clove, laurel, elderberry, *peppermint, rosemary,* lavender & other volatile oils; menthol & menthol pencils
Terpineol, heliotropin, vanillin, cumarin, anethol, *benzaldehyde,* & other perfume materials
Synthetic perfumes
Odoriferous fats, ointments & pomades
Eau de cologne
Other ether & alcohol containing toilet waters, vinegars & preparations
Ether & alcohol containing mouth & hair washes
Toilet waters
Powders, cosmetics, tooth & n. s. p. f. powders

XXI—Artificial Fertilizers
Artificial guano, ground bones & flesh, animal manure
Natural guano
Bone-meal
Thomas phosphate meal
Superphosphates, etc.
Calcium cyanamide

XXII—Explosives, Ammunition, Combustibles
Guncotton, *collodion cotton*
Gunpowder
Cordite

List I—(concluded)

Dynamite & other explosives
Fulminates
Picric acid
High explosives
Propellants
Primers & the like; flobert munitions
Loaded cartridges
Matches, wood & paper
Matches, wax
Fireworks: antimony, magnesium or zinc torches
Pitch torches: fuses, etc.

XXIII—Wood Fiber. Chemical Paper

Mechanical pulp
Straw, esparto & other fibers; paper-stock
Tracing, blue-print, gelatine, fly, ozone & test papers
Sulfate pulp
Sulfite pulp
Chemical pulp: bleached & unbleached
Papier maché
Sensitized paper
Photographic paper

XXIV—Fire-Brick & Clay Products

Firebrick; rectangular under 5 kg. per piece
Firebrick; rectangular over 5 kg. per piece
Other fireproof materials of clay or clay-like materials; crucibles, muffles, capsules, tubes, cylinders & other hollow-ware except retorts; plates other than stone; crucibles of magnesia cement or soapstone
Earthenware: Vitrified & non-vitrified
Retorts
Graphite crucibles, tuyeres, etc.
Porcelain chemical ware

XXV—Metals

Barium
Bismuth
Calcium
Cobalt
Magnesium
Pewter
Potassium
Fine gold; alloyed gold, crude or cast; gold bars
Gold ashes, scrapings or broken gold
Platinum, iridium, osmium, palladium, rhodium, ruthenium, unalloyed crude or cast
Fine silver, alloyed silver, crude or cast; silver bars
Silver scrapings

Pig iron
Scrap steel
Scrap cast-iron
Ferro-aluminum, -chrome, -manganese, -nickel, *-silicon, & other non-malleable iron-alloys*
  -Cerium
  -Molybdenum
  -Phosphorus
  -Titanium
  -Tungsten
  -Vanadium
Aluminum crude in plates
Aluminum bronzes
Lead, crude, lead scrap
Zinc: *crude & scrap*
  Drawn, rolled
  Dust
Tin, *crude & scrap*
Nickel: *Metal* & coins
  Alloys
Copper, crude
Copper coins, scraps, etc.
Copper rods, sheets, vessels & hollow-ware
Brass & similar alloys & scrap
Bronze & similar alloys & scrap
Yellow metal
Babbitt metal
Radium & its salts
Rhodium
Ruthenium
Selenium & its salts
Sodium
Tantalum
Titanium
Type-metal
Antimony, crude
Chrome, *cadmium, tungsten* & other non-noble metals adapted for fabrication, crude & scrap

XXVI—Industrial Wastes

Animal
Vegetable
Iron
Glass
Tobacco
Wax
Cotton
Lead
Dried fruit
Dyehouse
Linen, hemp, jute
Caoutchouc
Nickel
Silk
Soapmakers'
Wool
Sugar
Beet pulp
Fertilizer
Papermakers'
Leather & hides
All other

Those who use the lists in their final form must be prepared to perform some mental labor in spelling out from such records the precise information they may want in any specific instance.

List I is based upon official documents (except U. S.) published before the outbreak of the present war.

On May 10, 1916, the British Government prohibited the exportation of a number of chemical substances from Great Britain. This list appeared in the May 31, 1916, issue of the *Journal of the Society of Chemical Industry*, pp. 620–622. It is more diversified than any single Government Commerce Report that I have found; it may give valuable hints as to those things that in time of stress assume an importance to a nation's life that is not accorded them in times of peace; since such list may foreshadow further expansion of commerce reports such parts as are not contained in the foregoing are now given as List II. Study of this list may very well suggest proper additions to our own commerce or similar reports.

The Tariff Act of the U. S. recites certain specific chemicals and also classes of chemicals and these may also suggest proper additions; some of the relevant entries in the Tariff Act of October 3, 1913, are given in List III. The Treasury Department makes returns under each of the heads of List III and this information might be kept separate and published or at any rate so much of it as may thus serve a useful purpose; it is to this point that special attention should be directed since it

promises the easiest and most practicable mode of expansion for all concerned.

The labor expended in getting up the foregoing lists has been considerable and it has been exceedingly tiring work; it has resulted in bringing into relatively small compass information contained in four different languages and itself widely scattered; in spite of reasonable care I have, no doubt, overlooked some items and duplication may not have been avoided, but this should in no wise deter everyone interested from taking off his coat and getting down to work to see, firstly, if he himself and his interests are supplied with proper information and, if not, just what further information may be needed; then repeat that process for his section of the country, and then for the country at large, and forward that information in a letter (preferably typewritten) to the secretary of the nearest section of the American Chemical Society or American Electrochemical Society. Such letters should be written, well spaced, on paper of the ordinary business letter size (8 or $8^1/_2$ by 11 inches) and *on one side of the paper only;* the intention is to have these original letters suitably bound and appropriately indexed, and to have them accompany the final recommendation when, and if, it is presented to the officials in Washington. The work of the members of the local sections of the A. C. S., the A. E. S. and of this special committee, will be greatly expedited if each such letter were to be accompanied by seven (7) carbon copies to serve as working copies for the various committee members, and such effective coöperation will be more than repaid in speed and certainty. The final recommendation of this special committee must be backed up, as far as possible, by individual letters, otherwise the recommendations might very properly be looked upon as the personal opinions of only a few; that would not be fatal but it would be disadvantageous.

This special committee itself cannot possibly bring its plans to the attention of all interested; it must depend upon the coöperation of the members of the A. C. S. and the A. E. S. to bring this matter to the attention of local boards of trade, chambers of commerce or similar coöperative business organizations, and to the attention of individual concerns and corporations. Each member has his work to do, and this special committee counts with entire confidence upon a full performance.

Persons and corporations not members of the A. C. S. or the A. E. S. need feel no hesitancy whatever in taking active part in

List II—Exportations (Chemicals) Prohibited by British Government, May 10, 1916

Aeroplane dope
Capsicum
Capsicum-oleoresin
Carbon, Brazilian
Aluminum nitrate
Amidol & mixtures containing it
Ammonia perchlorate
Ammonia sulfocyanide
Anti-tetanus serum
Belladonna & its preparations
Belladonna alkaloids, their salts & preparations
Bismuth nitrate
Calcium sulfide
Cantharides
Chromium chlorate
Chromium nitrate
Cobalt nitrate
Copper suboxide & mixtures containing it
Cresol & nitrocresol
Cyanamide
Diethyl-barbituric acid (Veronal)
Dyes, coal-tar, all articles containing them
Emetine & its salts
Ergot of rye
Ether, formic
Gentian preparations
Green oil
Halogen derivatives of aliphatic hydrocarbons

Henbane & its preparations
Hexamethylene tetramine & its preparations
Hydrobromic acid
Hydroquinone & mixtures containing it
Ipecacuanha root
Manganese peroxide
Methyl aniline
Methyl salicylate & its preparations
Metol & mixtures containing it
Naphthalene compounds & preparations
Neosalvarsan
Nitrotoluol
Novocain & its preparations
Nux vomica & its preparations
Radium compounds
Salipyrine
Salvarsan
Senna leaves
Stramonium leaves & seeds
Sulfonal
Theobromine—sodium salicylate
Triphenyl phosphate
Trional
Trioxymethylene
Xylol, its preparations & compounds
Scheelite
Selenium
Vanadium
Mineral jellies

this work; write your suggestions to the secretary of the section of the A. C. S. or the A. E. S. nearest to you; there are 54 sec-

LIST III—CHEMICAL ENTRIES IN U. S. TARIFF ACT OF OCT. 3, 1913. (TREASURY DEPARTMENT RETURNS ARE MADE UNDER HEADINGS USED BELOW)

ACIDS: Acetic
Arsenic
Arsenious
Boracic
Carbolic
Chromic
Citric
Fluoric
Formic
Gallic
Hydrochloric
Hydrofluoric
Lactic
Nitric
Oxalic
Phosphoric
Phthalic
Prussic
Pyrogallic
Salicylic
Silicic
Sulfuric
Tannic and Tannin
Tartaric
Valerianic
Acetic anhydride
Acetine
Alcohol, wood
Alkalies, alkaloids & medicinal mixtures
Alumina hydrate
Alum
Alum cake
Patent alum
Sulfite of alumina
Aluminous cake
AMMONIA: Carbonate
Muriate
Liquid anhydrous
Sulfate
Nitrate
Perchlorate
Apatite
Argols
Crude tartar
Cream tartar
Rochelle salts
Calcium tartrate
BALSAMS: Copaiba
Fir
Canada
Peru
Tolu
BARIUM: Chloride
Dioxide
Carbonate, precipitated
Blacking, polishing powders & creams
Bleaching powder
Caffein
Calomel
Corrosive sublimate
Chalk, precipitated
Chloral hydrate
Salol
Phenolphthalein
Urea
Terpin hydrate
Acetanilid
Acetphenetidine
Antipyrine
Glycero-phosphoric acid
Acetylsalicylic acid
Aspirin
Guaiacol carbonate
Thymol
Chloroform
Carbon tetrachloride
Benzol
Naphthol
Resorcin
Toluol
Xylol
Toluidin
Xylidin
Cumidin

Benzidin
Binitrobenzol
Binitrotoluol
Tolidin
Dianisidin
Naphthylamin
Diphenylamin
Benzaldehyde
Benzyl chloride
Nitrobenzol
Nitrotoluol
Naphthylamin—sulfoacids
Naphthol—sulfoacids
Amidosalicylic acid
Binitrochlorbenzol
Diamidostilbendisulfoacid
Metanilic acid
Paranitranilin
Dimethylanilin
Cobalt, oxide of
Collodion
ETHERS: Sulfuric
Amyl nitrite
Amyl acetate
Ethyl acetate
Ethyl chloride
All other ethers & esters
EXTRACTS OF: Nutgalls
Persian berries
Sumac
Logwood
Chlorophyll
Saffron
Safflower
Formaldehyde
Fusel oil
Amylic alcohol
Gelatine
Glue
Glue-size
Isinglass
Agar-agar
GLYCERINE: Crude
Refined
GUM: Amber
Arabic
Senegal
Camphor, crude, refined or synthetic
Chicle, crude
Dextrine
Burnt starch
British gum
Ink & ink powders
Iodoform
Potassium iodide
Licorice extracts
Licorice root
Coca leaves
Lime citrate
MAGNESIA: Calcined
Carbonate of
Sulfate (Epsom salts)
Menthol
OIL: Cod
Seal
Herring
Whale
Sperm
Wool grease
Turkey red
Linseed
Poppy seed
Rape seed
Peanut
Hempseed
Almond, sweet
Almond, bitter
Sesame
Bean
Olive
Orange
Lemon
Peppermint
Amber
Ambergris

OIL (continued):
Anise
Bergamot
Camomile
Caraway
Cassia
Cinnamon
Cedrat
Citronella
Lemon grass
Civet
Fennel
Jasmine
Juniper
Lavender
Aspic
Limes
Neroli
Origanum, red & white
Thyme
Valerian
Morphine sulfate
All other opium alkaloids
Cocaine
Ecgonine
Barytes
Blanc fixé
Satin white
BLUES: Prussian
Berlin
Chinese
Ultramarine
BLACK: Bone
Ivory
Vegetable
Gas
Lamp
CHROME: Yellow
Green
Ochre
Sienna
Umber
Spanish Brown
Venetian Red
Indian Red
Colcothar
Litharge
Orange, mineral
Red Lead
White Lead
Lead acetate
Lead nitrate
Varnishes
Vermilions
Whiting & Paris White
Zinc oxide
Lithopone
Zinc sulfide (white)
Zinc chloride
Zinc sulfate
Enamel paints
Paints
Colors
Stains
Pigments
Crayons
Smalts
Ceramic fluxes
Glass fluxes
POTASH: Bicarbonate
Chlorate
Chromate
Bichromate
Nitrate
Permanganate
Red prussiate
Yellow prussiate
SALTS OF: Bismuth
Gold
Platinum
Rhodium
Silver
Tin
SOAPS: Perfumed toilet
Medicinal
Castile
All other

Vanillin
Vanilla beans
Tonka beans
SODA: Benzoate
Chlorate
Nitrite
Bicarbonate
Caustic
Phosphate
Hyposulfite
Sulfide
Sulfite
Chromate
Bichromate
Yellow prussiate
Borate (borax)
Carbonate
Sal
Sulfite crystals
Talcum
Ground talc
Steatite
French chalk
Arsenic
Sulfide of arsenic
Cinchona bark
Bauxite
Beeswax
Bismuth
COPPER: Sulfate
Acetate
Sub-acetate
BONE: Dust
Meal
Ash
Crude
Borates of soda, crude
lime, crude
Bromine
Cadmium
CALCIUM: Acetate, brown or gray
Chloride, crude
Carbide
Nitrate
Cement, Roman, Portland & other hydraulic
Cerium ore
Chromate of iron
Chromium, hydroxide (crude)
Anthracene
Anthracene oil
Naphthalene
Phenol
Cresol
Cobalt & ore
Cochineal
Sulfate of iron
Cryolite
Cudbear
Divi-divi
Fulminates
Gambier
Greases & fats for soap-making
Guano
Basic slags
Cyanamide
Lime-nitrogen
Gunpowder & all explosives
Gutta percha
India rubber, crude, scrap & refuse
Indigo
Iodine
Iridium, osmium, palladium, rhodium & ruthenium
Iron ore
Kierserite
Kainit
Casein
Lard
Asphalt, bitumen
Litmus
Madder
Magnesite

Manganese oxide & ore of
Myrabolans
Oleostearin
Orchil
OILS: Birch tar
Cajeput
Coconut
Cod
Cod liver
Cottonseed
Croton
Ichthyol
Juglandium
Palm
Palm kernel
Perilla
Soya bean
Olive oil
Chinese nut
Nut
Kerosene
Benzine
Naphtha
Gasoline
Paraffine
Spermaceti
Whale
ORES OF: Gold
Silver
Nickel
Platinum
Paris green
London purple
Phosphates, crude
Phosphorus
Photographic films
Platinum metal & manufactures of
Plumbago
POTASH: Crude or black salts
Carbonate
Sulfate
Hydrate
Nitrate
Muriate
Quinine sulfate & all cinchona alkaloids
Radium & its compounds
Selenium & its salts
Salt
Santonin
Sheep-dip
SODA: Arsenate
Cyanide
Sulfate (salt cake)
Bisulfate (niter cake)
Ash
Silicate
Nitrate
Soya beans
STRONTIUM: Oxide
Carbonate
Strychnine & its salts
Sulfur
Pyrites
Talcum, steatite, French chalk
Tanning extracts:
Quebracho
Hemlock bark
Oak
Chestnut
Nuts
Nutgalls
Terra alba
Tin ore & metal
Tungsten ores
Turpentine, Venice & spirits
Uranium oxides & salts
Valonia
Wax, vegetable or mineral
Witherite
WOODPULP: Mechanical
Chemical
Bleached
Unbleached
Matches

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tions in 32 states of the Union and the District of Columbia. There are no formalities and there is no need to stand on ceremony.

In addition to the foregoing list helpful suggestions may be obtained by consulting the weekly import and export lists that appear in the *Oil, Paint and Drug Reporter* of New York, and the daily lists of manifests of incoming steamers as they appear in the *Journal of Commerce and Commercial Bulletin* of New York; also the weekly and annual summaries of British foreign and domestic chemical trade as they appear in *The Chemical Trade Journal* may offer useful suggestions.

List IV gives the name and address of the secretary of each local section of the A. C. S. and the A. E. S., arranged alphabetically by states.

Should there be any changes in local secretaries from those given, the person addressed as indicated can be confidently counted on to forward such communication to his successor.

WORKING SCHEDULE OF COMMITTEE

The present plan is for this special committee to begin its preliminary list November 1, 1916; complete it November 15, 1916; publish this provisional list in the JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY, December 6, 1916, and in *Metallurgical and Chemical Engineering*; begin its final draft February 14, 1917; close the final recommendations February 28, 1917; and present its recommendations to the proper authorities in Washington before March 15, 1917; this will leave $3\frac{1}{2}$ months for Washington to make its decision and arrange for the

List IV—Names and Addresses of Secretaries of the Local Sections of the American Chemical Society and of American Electrochemical Society.

A. C. S.

Alabama

W. H. Beers, Jr.
2114 First Avenue
Birmingham, Ala.

California

B. S. Drake
5830 Colby Street
Oakland, Cal.

Henry L. Payne
223 West 1st Street
Los Angeles, Cal.

Connecticut

R. J. Marsh
C/o R. Wallace & Sons Mfg. Co.
Wallingford, Conn.

G. S. Jamieson
121 Linden Street
New Haven, Conn.

District of Columbia

E. C. McKelvey
Bureau of Standards
Washington, D. C.

Georgia

J. S. Brogdon
701/2 Peachtree Street
Atlanta, Ga.

Idaho

H. A. Holaday
University of Idaho
Moscow, Idaho

Illinois

D. K. French
2005 McCormick Building
Chicago, Ill.

Geo. D. Beal
University of Illinois
Urbana, Ill.

Indiana

H. W. Rhodehamel
643 East 32nd Street
Indianapolis, Ind.

Iowa

W. G. Gaessler
Iowa State College
Ames, Iowa

Perry A. Bond
Cedar Falls, Iowa

Kansas

W. B. Smith
24 Federal Building
Kansas City, Kans.

Kentucky

P. L. Blumenthal
Agricultural Experiment Station
Lexington, Ky.

A. M. Breckler
C/o Janes & Breckler
Louisville, Ky.

Louisiana

F. W. Liepsner
310 Customs House
New Orleans, La.

Maine

L. M. Burghart
University of Maine
Orono, Me.

Maryland

F. M. Boyles
C/o McCormick & Company
Baltimore, Md.

Massachusetts

E. B. Spear
Massachusetts Institute of Technology
Boston, Mass.

Michigan

James H. Bogart
925 Y. M. C. A. Building
Detroit, Mich.

H. H. Willard
802 Monroe Street
Ann Arbor, Mich.

Minnesota

Sterling N. Temple
1758 W. Blair Street
St. Paul, Minn.

Missouri

George Lang, Jr.
3601 Salena Street
St. Louis, Mo.

Albert G. Loomis
518 College Avenue
Columbia, Mo.

Nebraska

H. M. Plum
Department Agricultural Chemistry
Lincoln, Neb.

New York

J. A. Bridgman
Morse Hall
Ithaca, N. Y.

C. F. Roth
52 East 41st Street
New York City

Ray H. White
730 Buffalo Avenue
Niagara Falls, N. Y.

H. H. Tozier
26 Jones Avenue
Rochester, N. Y.

A. J. Salathe
Union College
Schenectady, N. Y.

R. S. Boehner
Syracuse University
Syracuse, N. Y.

North Carolina

F. E. Carruth
West Raleigh, N. C.

Ohio

E. K. Files
University of Cincinnati
Cincinnati, O.

A. F. O. Germann
Adelbert College
Cleveland, O.

W. J. McCaughey
O. S. U.
Columbus, O.

Oregon

Norman C. Thorne
841 Brooklyn Street
Portland, Ore.

Pennsylvania

Geo. C. Beck
325 Wyandotte Street
South Bethlehem, Pa.

J. H. Graham
113 W. Manheim Street
Germantown, Pa.

W. C. Cope
U. S. Bureau of Mines
Pittsburgh, Pa.

Rhode Island

Robt. F. Chambers
Brown University
Providence, R. I.

South Carolina

A. C. Summers
University of South Carolina
Columbia, S. C.

South Dakota

H. I. Jones
Dakota Wesleyan
Mitchell, S. D.

Tennessee

Paul C. Bowers
9 Garland Avenue
Nashville, Tenn.

Vermont

C. E. Burke
Burlington, Vt.

Virginia

W. A. Burrows
1000 East Carey Street
Richmond, Va.

Washington

H. L. Trumbull
University of Washington
Seattle, Wash.

Wisconsin

H. G. McAllister
404 Alhambra Building
Milwaukee, Wis.

L. F. Augspurger
University of Wisconsin
Madison, Wis.

A. E. S.

New York

J. M. Muir
239 W. 39th Street,
New York City

A. T. Hinckley
C/o National Carbon Co.
Niagara Falls, N. Y.

Pennsylvania

S. S. Sadtler
39 South 10th Street
Philadelphia

H. C. Ray
University of Pittsburgh
Pittsburgh, Pa.

Wisconsin

O. P. Watts
University of Wisconsin
Madison, Wis.

gathering of the added information, if any, decided upon with the beginning of the fiscal year 1917–18.

Delay, or failure in getting suggestions to the local sections will hamper the special committee. Points of view cannot be considered unless communicated. Everybody's coöperation is not only necessary but most cordially invited; if our list is not the best and most useful in the world to-day we must strive to make it so.

25 Broad Street
New York City

DEATH OF SIR WILLIAM RAMSAY

Sir William Ramsay died at his home, Beechcroft, at Hazlemere, Bucks, England, on July 23rd. He was born in Glasgow October 2, 1852. He received his early education in Glasgow Academy and University, and served as assistant in technical chemistry in the Young Laboratory as early as 1872. He later became assistant in Glasgow University, where he remained until 1880, at which time he became professor of chemistry in University College at Bristol. He later joined the faculty of the University College, London, as professor of chemistry, which position he occupied until 1913, when he retired as emeritus professor.

Sir William Ramsay has for many years held an undisputed position among the foremost scientists of the world and his scientific achievements are well known to the chemical profession. He was personally known to a large body of American chemists through his frequent visits to this country where he lectured at many of the leading universities and before the notable international meetings of the technical societies.

Of the many honors which he has received, probably the most notable is that of the Nobel prize in chemistry, which was awarded to him in 1904. Sir William was created a Knight in 1902. He was also a Commander of the Crown of Italy, a Knight of the Prussian Order Pour le Merite, an officer of the French Honor Legion, and an honorary member of the Royal Academies of twelve countries.

RESOLUTIONS ADOPTED ON THE DEATH ON ELTON FULMER

The following resolutions have been adopted by the Northern Intermountain Section of the American Chemical Society on the death of Professor Elton Fulmer, of the Washington State College, Pullman, Washington:

Whereas, Professor Elton Fulmer has suddenly been taken from our midst by death, be it

Resolved, That we, the members of the Northern Intermountain Section of the American Chemical Society, express our deep appreciation of his work as pioneer and leader in the development of chemistry in the Northwest, his sterling character, and his devotion to high ideals.

Be it further Resolved, That we express to the family our deep sense of loss in the taking away of a fellow worker and friend, and extend to them our heartfelt sympathy.

(Signed)   J. Kostalek
J. S. Jones

NOTES AND CORRESPONDENCE

ON EFFICIENCY OF AIR DRYERS

Editor of the Journal of Industrial and Engineering Chemistry:

I have read with considerable interest the letter of Prof. W. K. Lewis on the Efficiency of Air Dryers,[1] wherein he refers to a discussion of this subject in the columns of the *Journal of Metallurgical and Chemical Engineering* in the years 1909 and 1910. Inasmuch as I was guilty of starting some of the discussion referred to, in the issue of April and May, 1910, I beg permission to say a few words further on the subject.

The discussion arose from a published report[2] of a test of a drying machine wherein an "efficiency" of 85 per cent was claimed by the late Mr. W. B. Ruggles.

I criticized the use of the word "efficiency" in the sense in which it was employed, and I take this opportunity to say that my criticism was directed solely to what I regarded as a misleading use of a scientific term, and that I very clearly recognize the valuable services of the late Mr. Ruggles to the practical art of drying.

Mr. Lewis finally arrives at the conclusion that it is desirable to express the results attained by drying machines in terms of "performance" rather than "efficiency," "Performance" as defined by Mr. Lewis being the actual heat consumption in terms of B. t. u. per lb. of water evaporated. With this I heartily concur and it really differs in no way from what I contended was the correct definition of "efficiency" in 1910. Such a statement of performance is exact and definite, and precisely expresses what a drying machine accomplishes towards the purpose for which it is designed, *viz.*, the evaporation of water by expenditure of heat. Further, the form of statement as proposed by Mr. Lewis makes it possible directly to compare the operation of two or more machines of different types. The "performance" as defined by Mr. Lewis is indeed a direct statement of energy expended and useful work performed thereby.

The difficulty pointed out by Mr. Lewis of stating the "efficiency" of multiple-effect evaporating apparatus is a real one and we may as well boldly face the fact that such apparatus (multiple-effect) really has an efficiency as heat using machinery of greatly over 100 per cent actually, and its theoretical "efficiency," as Mr. Lewis shows, is infinity. This is really only another way of stating the doctrine of conservation of energy. Ordinarily our utilization of energy is confined to one transformation from kinetic to potential or *vice versa*. Multiple-effect apparatus is one instance (practically almost a singular instance) where we are able to repeat the change cyclically. Hence a definite amount of heat can be made to evaporate an indefinite amount of water. Of course in practice the physical limits are somewhat sharply drawn at five or six cycles, but theoretically there is no limit to the quantity of water that can be vaporized by a given amount of heat.

Such, however, is not the case with "air dryers," which is the titular heading of Mr. Lewis' letter and which was the subject matter of the discussion of several years since, referred to by him.

The consumption of heat in an "*air dryer*" for evaporative purposes is a perfectly definite thing and just as definite as in the case of a steam boiler. We have to deal only with one possible transformation of heat from sensible to latent form and the work that can thereby be performed (as evaporation) is perfectly definite.

It is perhaps well to define an "air dryer," and by the term I understand a machine utilizing artificial heat to evaporate water from some solid substance, wherein air at substantially atmospheric pressure is usually the medium by which heat is

[1] This Journal, **8** (1916), 570.
[2] *J. Met. and Chem. Eng.*, March, 1910.

conveyed to the material to be dried and *always* the medium by which the vapor produced is removed from the material or machine. I believe this definition covers all practical types of atmospheric drying apparatus. It will be noted that it does not cover vacuum drying apparatus; but in all practical vacuum drying apparatus the evaporative possibilities of a given amount of heat are also definite and limited. The theoretical possibility of indefinitely repeated utilization shown by Mr. Lewis applies only to multiple-effect machines. As far as I am aware multiple effect has not been applied to drying, but only to the evaporation of solutions.

We see, therefore, that either in "air dryers" or in vacuum dryers (single-effect) the quantity of water that can be evaporated by a given amount of heat is quite definite—not only practically but as a theoretical matter, and hence it is quite possible to state the performance of all practical dryers as a percentage "efficiency."

It requires practically 1120 B. t. u. to evaporate 1 lb. of water from average normal temperature (I speak of course in round numbers) and any air-drying machine or vacuum dryer that evaporated 1 lb. of water for an input of 1120 B. t. u. would have an efficiency of 100 per cent.

In practice there are various losses, due to radiation, to heating the dried material to the discharge temperature and to heating the outgoing air to the discharge temperature—this last being usually the greatest. All the losses are unavoidable; all can be greater or less according to the type and perfection of the machine and on their balance depends the efficiency of the machine. My contention in the discussion of several years since was that any drying machine should be charged with the total heat supplied, whether in the form of steam or from direct combustion of fuel, and credited solely with the heat usefully expended in evaporation of water. This view is, I believe, entirely in accordance with Mr. Lewis.

The second example given by Mr. Lewis to demonstrate that there is no definite efficiency in air drying, appears to me quite irrelevant. The case is given of spreading material under a shed and allowing air to blow over it. Manifestly there is in this case no expenditure of artificial energy and it can have no relation to efficiency of fuel because fuel is in no way concerned. Nevertheless energy is expended and quite definitely; and the only reason we are not able to determine the efficiency of such a system is the impossibility of determining the factors. Could we determine with any accuracy the amount of air coming in contact with the material under such circumstances and the amount of (low-grade) heat brought into the system, also the drop in temperature and the increase in saturation (both of which would be very small), we should have in hand all the data necessary to determine the efficiency of the system.

Mr. Lewis' letter really treats of much more than the efficiency of air dryers and goes into the field of evaporation in general. While this is of equal interest, it appears to me that the two things are quite distinct, although they do indeed involve the common factor of evaporation of water. Nevertheless the other factors are so different that they had best be treated independently.

Mr. Lewis has himself clearly shown one reason for separate treatment, in pointing out that a theoretically perfect multiple-effect evaporator has an unlimited efficiency. I do not think this has been scientifically shown before, but we see in trade customs a dim realization that the standards applicable to drying machines, or in general single-effect machines, are not equally applicable to multiple machines. It is quite a common custom for manufacturers of drying machines to claim for their

machines a certain "efficiency," as a percentage. As I remember, this is never done with multiple-effect evaporators, but instead a certain evaporation per pound of steam supplied is specified. This it will be seen is practically identical with the "performance" proposed by Mr. Lewis.

As regards the last portion of Mr. Lewis' letter, treating of the evaporation of solutions, I frankly confess that I utterly fail to understand. He assumes a solution boiling under a definite pressure, p, which is less than the pressure, p_o, of the pure solvent at the same temperature. The vapor from the boiling solution is to be compressed isothermally and "can be introduced into the coils of the same effect from which the vapor itself came and there condensed at a temperature *differentially higher* (italics are mine) than that of the solution on the other side of the heating surface. The heat of condensation will thus be available for the evaporation of more water from the original solution."

Now the temperature of the vapor is that of the pure solvent boiling at the assumed pressure and is *lower* than that of the boiling solution, owing to the lowering of the vapor tension by the substance in solution. This vapor is compressed isothermally, that is, it remains at the same temperature, but it is required to give up its "heat of condensation" (? latent heat) to a solution at a temperature higher than its own.

Unless I have misread this passage, it looks like a flagrant violation of the second law of thermodynamics, and indeed involves the direct transfer of heat from a lower temperature to a higher.

I hasten to say this is not criticism and that my failure to understand the mechanism of the described process is undoubtedly due to my personal weakness in the science of thermodynamics.

BAYONNE, NEW JERSEY   W. E. WADMAN
June 24, 1916

ACTIVE AND LATENT SOIL ACIDITY *vs.* IMMEDIATE AND CONTINUED LIME REQUIREMENT

Editor of the Journal of Industrial and Engineering Chemistry:

Recently there appeared an article in THIS JOURNAL, 8 (1916), 572, entitled "Immediate and Continued Lime Requirement *vs.* Active and Latent Soil Acidity," by W. H. MacIntire, of the University of Tennessee. In this article the inference is made that the present writer in a report in THIS JOURNAL, 8 (1916), 341, has not given sufficient credit or prominence to MacIntire's recognition of his so-called "Immediate and Continued Lime Requirement" as given in THIS JOURNAL, 7 (1915), 864. In regard to the statements that are made and the questions that are raised the writer deems it appropriate to state as follows:

Although possibly unknown to MacIntire, the writer published an article in *Science*, 42 (1915), 505, in which the distinction is made between *active* and *inactive* soil acidity. This article appeared in the issue of October 8, 1915, and MacIntire's article, in which he gave the expressions *immediate* and *continued lime requirement*, appeared also in the October, 1915, number of THIS JOURNAL. In the *Science* article, the writer used the term *inactive*, for which was later substituted the term *latent*, since that seemed more desirable.

It is well to note further that Veitch, in an excellent discussion of the nature of soil acidity in the *Am. Chem. Jour.*, 26 (1904), 659, used the terms *active* or *actual acidity* and *inactive* or *negative acidity* to designate different kinds of soil acidity. Veitch states that the total *active* and *inactive* acidity is estimated by his lime-water method. This distinction of Veitch is thus plainly a different one than that made by the writer in THIS JOURNAL, 8 (1916), 341, since the lime-water method does not estimate even all the acidity that is designated by the

writer as *active* acidity, to say nothing of the acidity designated as *latent* acidity.

Possibly other references could be given in which a recognition has been made of differences in the *activity* of the acids causing soil acidity. One of the most significant facts in regard to the various methods proposed for determining soil acidity is that the results by any one method are seldom the same as by any of the others. These differences have been recognized and attributed by some to differences in the nature and activity of the acids causing soil acidity. The writer used the term *active* acidity and recognized differences in *activity* of soil acids in *Wis. Sta. Bull*. 249, p. 15. This bulletin appeared eight months before MacIntire's publication in which was used the expression *immediate lime requirement*.

Perhaps the expressions chosen by MacIntire are superior to those chosen by the writer. If such is the case the criticism may be made that the writer should have changed his expressions in the subsequent publications after MacIntire's article appeared. The writer is as yet not convinced of this superiority, and even if this superiority is granted other difficulties arise as follows:

Determinations made lately indicate that MacIntire's method for his so-called *immediate lime requirement* does not indicate the same amount of acidity as the method given by the writer for *active acidity*. The methods thus clearly do not indicate the same point in the complete neutralization of the soil acids. The writer would thus have been open to serious criticism had he stated, subsequent to the publication in *Science*, that the method proposed measures the so-called *immediate lime requirement* as defined by MacIntire. In fact, the writer wishes to state definitely that the method proposed does not measure this *immediate lime requirement* as apparently defined by MacIntire.

Whatever practical or scientific meaning MacIntire may wish to convey by the expression, *immediate lime requirement*, it may be well to state clearly that the method (in which is used a solution of $Ba(OH)_2$) proposed by the writer for *active acidity* does not, and never was intended to indicate, in any way, the amount of lime that should be used under practical field conditions. It simply indicates the amount of acid substances present which are sufficiently active to react quickly with bases like $Ba(OH)_2$ or $Ca(OH)_2$. The expression, *amount of active acidity*, has no connection or relation to the expression, *lime requirement*, in the sense in which the latter expression has been used in the past.

The writer confesses that he does not clearly understand what MacIntire means by *continued lime requirement*. It has not been possible to find a description of a laboratory method for the determination of this so-called *continued lime requirement* and hence had the writer appropriated the expression *continued lime requirement* in place of *latent acidity*, or stated that the two expressions mean the same thing, he would again have been open to serious criticism.

Further than mentioning that MacIntire had described a method in which a solution of $CaCO_3$ in carbonated water is used, the writer purposely avoided either favorable or unfavorable comments. Since MacIntire did not present data, the relation of *immediate lime requirement* to *active soil acidity* could not be intelligently discussed at that time. The field was left clear, and as far as the present writer is concerned, MacIntire should have little trouble in getting full recognition for the merits of his work.

MacIntire states that he is unable to find in the writer's article in THIS JOURNAL, 8 (1916), 341, any data which indicate that soil acidity is due to true acids and not selective ion adsorption by colloids. The writer believes that this is entirely possible. For further explanations the reader is referred to an article by the writer entitled "The Cause and Nature of Soil

Acidity with Special Regard to Colloids and Adsorption," *Jour. Phys. Chem.*, **20** (1916), 457.

In the past several theories have been advanced regarding the nature of soil acidity; numerous methods have been proposed for determining a part or the whole of the acidity; and various names have been proposed for designating this or that part or kind of the acidity. MacIntire has seen fit to use the terms *immediate* and *continued lime requirement* for designating what he believes are certain soil conditions; the writer has seen fit to use the terms *active* and *latent* soil acidity for what he believes designate certain other soil conditions; and undoubtedly investigators in the future will see fit to use still other terms for designating what they may think are certain kinds of soil acidity, for it seems quite certain that no one as yet has a monopoly on the ideas regarding soil acidity or names that are suitable for designating certain aspects or phases of this subject. It seems reasonable to believe that investigators of this subject in the future will be as versatile and ingenious as those of the past.

In conclusion, the writer wishes to state that he has always entertained a high regard for the work of MacIntire and his associates and hopes this may continue in the future.

MADISON, WISCONSIN   -E. TRUOG
  June 20, 1916

NOTE REGARDING DETERMINATION OF ALKALINITY OF ASH IN ANALYSES OF ORGANIC MATERIALS

It is frequently recommended that the alkalinity of ash of various food materials be determined in order to gain an idea as to the nature of the ash and the probability of adulteration of the sample.

In this connection there is room for considerable error in that sodium chloride may be dissociated, the chlorine driven off and the sodium appear at the end as sodium carbonate. Indeed, if care is not taken to avoid too high temperature or too prolonged heating all the chlorine may be thus driven off and the alkalinity of the ash is then no criterion as to organic combination of elements.

That chlorine is lost is shown by the following results obtained on corn syrup:

| | | PER CENT CHLORINE IN ASH | |
| | Per cent chlorine | Ashed with | |
Sample.	by titration.	excess Na$_2$CO$_3$	Ashed straight
No. 1	0.180	0.180	0.065
No. 2	0.185	0.185	0.070
No. 3	0.190	0.190	0.071

In order to observe the effect of ashing pure granulated cane sugar to which a small percentage of salt had been added the following tests were made: Ten grams sugar were ashed with 0.025 g. salt (1 cc. of solution containing 2.5 g. NaCl in 100 cc.).

A—Straight ignition at lowest heat practicable.

B and C—Charred, extracted with hot water, char ashed, extract added, evaporated and heated to redness.

D—A few drops of sulfuric acid added and then sample ignited as A.

TEST:	A	B	C	D
Ash (added NaCl)	0.25%	0.25%	0.25%	0.25%
Ash found	0.18	0.20	0.22	0.24
Alkalinity of ash as Na$_2$CO$_3$	0.12	0.20	0.19	0.03

These results illustrate the point made above, *viz.*, that too much weight should not be placed on alkalinity of ash when it is possible that chlorides of the alkalies may have been present in the original material.

CLINTON SUGAR REFINING COMPANY   A. P. BRYANT
  CLINTON, IOWA, April 24, 1916

PERSONAL NOTES

The Illuminating Engineering Society will hold a lecture course at the University of Pennsylvania, Philadelphia, from September 21 to 28, inclusive, immediately following the Annual Convention of the Society, to be held in that city.

This is the second time in its history that the Illuminating Engineering Society has decided to conduct a series of lectures, the first having been held in the fall of 1910, in coöperation with the Johns Hopkins University, Baltimore. Whereas the 1910 course emphasized the *science* of illumination, the new course will emphasize the *art* of illumination. This course will include about twenty lectures by men selected on account of their qualifications, to deal authoritatively with the several phases of the subject. The preliminary list of lecture subjects will appear in the September issue of THIS JOURNAL. Associated with the lectures will be an exhibition, at the University of Pennsylvania, of the latest developments in illuminating appliances, together with novel applications of light. In connection with the lecture course there will be organized an inspection tour which will include visits to manufacturing establishments, laboratories, lighting companies and notable installations. The price of tickets, which include admission to all lectures and functions connected therewith, has been fixed at $25.00. The *Chairman* of the Sub-committee on Publicity of the Illuminating Engineering Lecture Course, is Mr. H. K. Mohr, Philadelphia Electric Company, Tenth and Chestnut Streets, Philadelphia.

The 1916 Directory of the American Chemical Society was issued July 1st. Members of the Society can secure copies by request to the Secretary, accompanied by twenty-five cents, and the statement that "The Directory will not be loaned or disposed of to any firm or individual for advertising purposes." This issue is revised to June 1, 1916, and shows a membership of 8057. The officers of the Society, of the divisions, and of the local sections are listed separately on the first pages. Corporation members, honorary members, and life members are grouped separately immediately following the officers.

The National Fertilizer Association elected the following officers at their 23rd Annual Convention held in Hot Springs, Va.: *President*, Horace Bowker, New York City; *Vice-President*, W. D. Huntington, Baltimore; *Treasurer*, Irvin Wuichet, Dayton, Ohio (reëlected); *Secretary*, W. G. Sadler, Nashville, Tenn. (reëlected); *Assistant Secretary*, John D. Toll, Philadelphia.

The United States Civil Service Commission announces the following open competitive examinations: *Assistant Physicist* (Male) *Qualified in Physical Metallurgy*, on August 23, 1916; *Junior Chemist*, Departmental Service, and *Laboratory Apprentice* (Male), Bureau of Standards, on September 13, 1916; *Laboratory Assistant* (Male), Bureau of Standards, on October 11, 1916. Further information may be had from the U. S. Civil Service Commission, Washington, D. C.

The Thirtieth General Meeting of the American Electrochemical Society will take place this fall, in New York City, in the week of the Second National Exposition of Chemical Industries. Sessions will be held at the Chemists' Club, 52 East 41st Street, on September 28, 29 and 30, 1916.

Mr. F. L. Craddock has been appointed manager of the New York office of The Pfaudler Company, 110 West 40th Street, to succeed Mr. George M. Kendall, a notice of whose death appeared in the July issue of THIS JOURNAL. Mr. Craddock has long been associated with The Pfaudler Company, both as erection engineer and as salesman, and enjoys a wide acquaintance among the trade.

Mr. R. B. Kilmer has been appointed manager of the Chicago office of The Pfaudler Company, 1001 Schiller Building. Since casting his lot with The Pfaudler Company, Mr. Kilmer has been continually associated with the Chicago office and is well known to the Middle Western trade.

The following officers were elected by the American Society for Testing Materials at their annual meeting held recently in Atlantic City, N. J.: *President*, A. A. Stevenson, Philadelphia; *Vice-President*, S. S. Voorhees, Washington, D. C. *Executive Committee:* W. H. Bassett, Waterbury, Conn.; John Brunner, Chicago; G. W. Thompson, New York City; and F. E. Turneaure, Madison, Wis.

Mr. Ralph L. Fuller, vice-president of Harshaw, Fuller & Goodwin, manufacturers of chemicals, has been elected president of the Cleveland Chamber of Commerce. Mr. E. R. Grasselli, second vice-president and treasurer of the Grasselli Chemical Company, is vice-president of the Chamber.

Prof. Frank H. Thorp, Massachusetts Institute of Technology, has resigned his assistant professorship of industrial chemistry and expects to devote himself in the immediate future to private business.

We learn from *The Engineering and Mining Journal* that Dr. Henry M. Howe, emeritus professor of metallurgy in Columbia University, has been appointed honorary vice-president of the Iron and Steel Institute of Great Britain. This distinction is held by only two other persons: Georgio E. Falck, of Italy, and D. Tschernoff, of Russia.

The University of Cincinnati, on June 19th, conferred the honorary degree of doctor of science on Prof. John Uri Lloyd, known for his contributions to chemistry and pharmacy.

In order to improve increased manufacturing space for the well-known Lee puncture-proof pneumatic tires, the Lee Tire & Rubber Company has just placed a contract with John W. Ferguson Company, of New York City and Paterson, N. J., for another building at their Conshohocken plant. The building measures 80 ft. by 120 ft. in plan and will for the present contain but two stories. The foundations and reinforced concrete columns on the first floor are designed, however, for several additional stories, with the idea of probable expansion in view. H. M. Haven and W. W. Crosby, of Boston, drew up the plans and specifications.

Dr. A. D. Emmett, assistant chief in animal nutrition, of the Illinois Agricultural Experiment Station at the University of Illinois, has accepted the position of research biological chemist in the Research Laboratory of Parke, Davis & Company, Detroit, Michigan. Dr. Emmett will assume his new duties on September 1st.

Richard K. Meade, chemical, mechanical and industrial engineer, Baltimore, announces that owing to need for increased facilities, he has removed his offices from 202 North Calvert Street to The Law Building, Courtland Street, Baltimore.

Frank H. Probert, a graduate of the Royal School of Mines in London, and for the past twenty years engaged in consulting mining engineering practice, has been appointed professor of mining in the University of California, as successor to the late Prof. Samuel Benedict Christy.

Prof. Arthur Green, F.R.S., lately of the tinctorial chemistry chair at Leeds University, England, has accepted the post of director of the research department of Messrs. Levinstein, Ltd., aniline dye manufacturers, Blackley, Manchester.

The Chemical Society, London, has removed from its list of honorary and foreign members the names of enemy aliens.

The employees of the Joseph Dixon Crucible Company, who joined the National Guard prior to its being called by the President and subsequently sworn into the Federal Service in June, 1916, will receive full pay while away doing military duty. These men will have their places kept open for them. This provision holds good until future developments make it necessary to change or modify it.

Herman & Herman, Inc., New York manufacturers and jobbers of chemicals, announce the removal of their offices, on account of increased business, to more spacious quarters, in the Robinson Building, 6 Church Street, cor. Liberty Street, New York City.

Mr. Henry Brunner, of Messrs. Brunner, Mond & Company, Ltd., died at his residence in Liverpool, England, on June 17th at the age of 78 years.

The new chemical laboratories recently erected in connection with Oxford University, England, were opened at the beginning of June.

Charles W. H. Kirchhoff, of New York City, editor-in-chief of *The Iron Age* from 1889 until 1910, died on July 22nd at his summer home, North Asbury Park, New Jersey, in his sixty-fourth year. Mr. Kirchhoff was the special agent of the U. S. Geological Survey for the collection of statistics of the production of lead, copper and zinc from 1883 to 1906. He was prominent for years in the mining industry, being at one time president of the American Institute of Mining Engineers. He was a member of the American Iron and Steel Institute, the Iron and Steel Institute of Great Britain, the American Society of Mechanical Engineers, the Verein Deutscher Eisenhuettenleute, the Century and Engineers' Clubs of New York City, and an honorary member of the Franklin Institute.

Dr. Leo H. Baekeland, of Yonkers, N. Y., and Prof. Julius Stieglitz, of the University of Chicago, were given the honorary degree of doctor of chemistry by the University of Pittsburgh at its recent commencement.

The Annual General Meeting of the Society of Chemical Industry was held in the hall of the University Union, Edinburgh, Scotland, on July 19, 1916.

Dr. William Simon, professor of chemistry at the College of Physicians and Surgeons, Baltimore, died at Eaglesmere, Pa., on July 19, 1916. He was born in Germany in 1844, and came to this country in 1870. The following year he opened the first chemical laboratory for instruction in Baltimore. Dr. Simon was a professor of chemistry in the Maryland College of Pharmacy from 1872 to 1902, in the College of Physicians and Surgeons, Baltimore, since 1880, and in the Baltimore College of Dental Surgery since 1888.

The American Institute of Mining Engineers extend an invitation to the members of the American Chemical Society to accompany them on their trip to the Southwest in connection with the Arizona Meeting of the Institute to be held on September 18–25, 1916. It is planned to make a special train tour of the Arizona Mining Districts and Grand Canyon. A pamphlet containing the itinerary and other information may be obtained from Mr. Bradley Stoughton, *Secretary*, American Institute of Mining Engineers, 29 West 39th Street, New York City.

The Hoskins Manufacturing Company announce that the address of their Boston office has been changed from 613 Unity Building, 185 Devonshire Street, to 445 Tremont Building, 73 Tremont Street. The office will continue to be in charge of Mr. J. E. Hines.

The Sarco Company, Inc., New York, have appointed Newton-Johnson, sales engineers, as sales representative in Wisconsin and the northern peninsula of Michigan.

GOVERNMENT PUBLICATIONS

By R. S. McBride, Bureau of Standards, Washington

NOTICE—Publications for which price is indicated can be purchased from the Superintendent of Documents, Government Printing Office, Washington, D. C. Other publications can usually be supplied from the Bureau or Department from which they originate. Commerce Reports are received by all large libraries and may be consulted there, or single numbers can be secured by application to the Bureau of Foreign and Domestic Commerce, Department of Commerce, Washington. The regular subscription rate for these Commerce Reports mailed daily is $2.50 per year, payable in advance, to the Superintendent of Documents.

PUBLIC HEALTH SERVICE

Bread as a Food. Carl Voegtlin, M. X. Sullivan and C. N. Myers. Public Health Reports, **31** (April 14, 1916), pp. 935–43. The changes in the vitamine content of bread as related to the nutritive value are discussed with reference to the occurrence of pellagra. The following conclusion is reached: "The fact that the above-mentioned influences, which have undoubtedly reduced the vitamine content of the diet, made themselves felt a relatively short time before the rapid increase in the pellagra incidence in this section of the country, furnishes considerable evidence in favor of the vitamine-deficiency theory of pellagra."

Establishments Licensed for the Propagation and Sale of Viruses, Serums, Toxins, and Analogous Products. Public Health Reports, **31** (April 14, 1916), pp. 943–8. This is a list of establishments holding licenses under the Treasury Department for traffic in the above-mentioned articles.

Some Fallacies Regarding Phenol. Martin I. Wilbert. Public Health Reports, **31** (April 28, 1916), pp. 1046–51. This is a review with reports of observations on the influence of ethyl alcohol on the germicidal and on the toxic properties of phenol. "In conclusion, it may be again noted that the experimental work clearly shows that the addition of ethyl alcohol to phenol not only increases the solubility of phenol in water, but also increases rather than diminishes the antiseptic value of the resulting solution. Ethyl alcohol can be used to advantage as a substitute for glycerine in making antiseptic solutions of phenol.

"The experiments with animals clearly show that the addition of ethyl alcohol to solutions of phenol in water does not, in any way, inhibit the toxic action of phenol, but rather tends to facilitate absorption and thus hasten death."

The Effect of Alcohol or Glycerin on the Toxicity of Phenol as Shown by Inoculations into White Mice. Liston Paine. Public Health Reports, **31** (April 28, 1916), pp. 1052–4. This is an abstract of a report by this author.

Pellagra: Laboratory Examinations in Connection with the Disease. J. R. Ridlon. Public Health Reports, **31** (May 19, 1916), pp. 1231–41.

Public Health Administration in Florida. Carroll Fox. Public Health Reports, **31** (June 2, 1916), pp. 1359–1407. This is a description of the administrative methods, personnel, arrangements, and activities of the Florida State Board of Health. It is of interest to administrative officials particularly.

Cyanide Gas for the Destruction of Insects. R. H. Creel and F. M. Faget. Public Health Reports, **31** (June 9, 1916), pp. 1464–76. This work has special reference to mosquitoes, fleas, body lice, and bedbugs and it also discusses the procedure for making cyanide fumigation.

SANITARY LEGISLATION

Manufacture or Storage of Acid Drinks and Foodstuffs in Zinc Lined and Galvanized Metal Containers. Regulation of the Indiana State Board of Health, adopted July 2, 1915. It specifically prohibits the use of zinc lined or galvanized metal containers with citric, tartaric or vegetable acids which will dissolve zinc.

Adulterated or Misbranded Drugs. New York City Department of Health regulation, adopted October 26, 1915, amending Section 116, which defines the terms "drugs," "adulterated," and "misbranded."

Names of Ingredients of Proprietary Medicines to be Registered. Regulation of the New York City Department of Health, adopted October 26, 1915, amending Section 117, which prescribes rules for the registration of composition of proprietary medicines.

Regulation for the Registration of Proprietary Medicines. New York City Department of Health regulation, as amended December 21, 1915. This gives further rules as to the registration of proprietary goods.

Production, Care and Sale of Milk and Cream. The following regulations on this general subject have been adopted recently including in each case definitions of the minimum quality of milk, cream, skimmed milk, and other milk products which are required in the localities affected. The page numbers in parenthesis refer to pages of Volume **31** of the Public Health Reports:

1—Hackensack, N. J. Regulation of the Board of Health, adopted December 28, 1915. (1115).

2—Leominster, Mass. Regulation of the Board of Health, adopted September 29, 1915. (1116).

3—Jackson, Mich. Ordinance, adopted April 27, 1915. (1222).

4—Lackawanna, N. Y. Ordinance, adopted February 1, 1915. (1276).

5—Kansas City, Kansas. Ordinance 13208, adopted July 29, 1915. (1338).

6—New York City Department of Health Regulation, adopted December 21, 1915, amending Section 155 of the Sanitary Code. (1347).

7—Portland, Oregon. Ordinance 30491, adopted May 26, 1915. (1507).

8—Portland, Oregon. Ordinance 30554, adopted June 10, 1915, defines buttermilk. (1512).

9—Sacramento, California. Ordinance 208, as amended August 19, 1915. (1515).

GEOLOGICAL SURVEY

Relation of the Wissahickon Mica Gneiss to the Shenandoah Limestone and Octoraro Schist of the Doe Run and Avondale Region, Chester County, Pennsylvania. Eleanora F. Bliss and Anna I. Jonas. Professional Paper 98-B, from Shorter Contributions to General Geology, pp. 9–34. Published May 20, 1916.

Mineralogic Notes. Series 3. Waldemar T. Schaller. Bulletin 610. 164 pp. "The results of the smaller, independent pieces of mineralogic research work carried out by the writer in the chemical laboratory of the United States Geological Survey from July 1, 1911 to December 31, 1913, are here assembled."

The following articles varying from a few pages to 35 pages each make up this bulletin: Koechlinite (bismuth molybdate); inyoite and meyerhofferite, two new calcium borates; kycubute, a new mineral; a dimorphous form of variscite; the crystallography of variscite; schneebergite; romeite; the natural antimonites and antimonates; velardenite, a new member of the melitite group; the melitite group; the composition of cebollite; the crystallography of thaumasite; the chemical composition of tremolite; new occurrences of some rare minerals; gigantic

crystals of spodumene; the probable identity of mariposite and alurgite; the amblygonite group of minerals—fremontite (=natramblygonite); the crystallography of fremontite; the chemical composition of nephelite; large crystals of bloedite; alunite from Marysvale, Utah; the composition and relations of custerite; the composition of hodgkinsonite; crystals of pisanite from Ducktown, Tenn.; the refractive indices of strengite; the calculation of a mineral formula.

Contributions to Economic Geology, 1915. Part I—Metals and Nonmetals Except Fuels. F. L. RANSOME AND HOYT S. GALE. Bulletin 620. 360 pp. This bulletin is made up of 17 short articles, most of which have been previously announced in THIS JOURNAL.

Geology and Coal Resources of Castle Valley in Carbon, Emery, and Sevier Counties, Utah. CHARLES T. LUPTON. Bulletin 628. 86 pp.

Natural Gas Resources of Parts of North Texas. Bulletin 629. 125 pp. This bulletin includes the following three articles which report on work done in coöperation with the cities of Dallas and Fort Worth:

"Gas in the Area North and West of Fort Worth," by EUGENE WESLEY SHAW; "Gas Prospects South and Southeast of Dallas," by GEORGE CHARLTON MATSON; "Notes on the Gas Fields of Central and Southern Oklahoma," by CARROLL H. WEGEMANN.

Geology and Underground Water of Luna County, New Mexico. N. H. DARTON. Bulletin 618. 186 pp. This report includes a discussion of the mineral resources, the quality of water, and the general geological conditions of the district. Rock and mineral analyses from the region are also reported.

The Caddo Oil and Gas Field, Louisiana and Texas. GEORGE CHARLTON MATSON. Bulletin 619. 62 pp. Paper, 30 cents. The history, physiography, geology, and oil and gas resources of this region are discussed in detail.

A Reconnaissance in Palo Pinto County, Texas (with Special Reference to Oil and Gas). CARROLL H. WEGEMANN. Bulletin 621-E, from Contributions to Economic Geology, 1915, Part II, pp. 51-9. Corrected edition published April 17, 1916. This report includes a discussion of the history and development, topography, stratigraphy and report on tests for oil and gas. It recommends an exploration at a particular point by the drilling of a well. According to the results on this well the general conditions in the region can be interpreted.

The Atlantic Gold District and the North Laramie Mountains. A. C. SPENCER. Bulletin 626. 85 pp. This is a group of papers on conditions in Freemont, Converse, and Albany Counties, Wyoming.

Ground Water in San Joaquin Valley, California. W. C. MENDENHALL, R. B. DOLE AND HERMAN STABLER. Water Supply Paper 398. 310 pp. and numerous plates. The third section of this report (by R. B. Dole) which occupies over 100 pages is on the quality of water found in this region. It discusses in considerable detail the important question of quality, describes methods for collection of water samples and methods for field and laboratory assay, gives standards for water classification, and discusses the importance of various characteristics and methods of purification of water for different applications, such as boiler use, potable water, and others. Much of the discussion is of general interest to anyone concerned with the testing, purification, or industrial application of water supplies. Many analyses of water from this district are reported.

Ground Water in the Hartford, Stamford, Salisbury, Willimantic and Saybrook Areas, Connecticut. HERBERT E. GREGORY AND ARTHUR J. ELLIS. Water Supply Paper 374. 150 pp. and numerous plates and maps. This report is the result of an extended investigation made jointly by the United States Geological Survey and the Connecticut Geological and Natural History Survey for the purpose of obtaining information as to the quantity and quality of water available in various parts of the State of Connecticut for municipal and private uses. The data presented are largely of local interest and include many analyses of water from these districts. The general method of attack upon the problem is of wider interest.

Chromic Iron Ore in 1915. J. S. DILLER. Separate 1, from Mineral Resources, 1915, Part I, pp. 1-6. Published April 10, 1916. "The production of chromic iron ore or chromite in the United States in 1915 amounted to 3,281 long tons valued at $36,744, a gain of 2,690 tons and of $28,029 over 1914. From 1880 to 1915, inclusive, the average annual production has been 1,229 tons, and the average price per ton has been $14.82.

"The total imports of chromic iron ore in 1914 were 74,686 long tons, the maximum since 1908, valued at $655,306. The imports in 1915 as compared with those in 1914 increased 1,769 long tons in quantity and $124,755 in value, so that the average price per long ton increased from $8.77 to $10.20 in 1915.

"A fair price for imported ore in December, 1915, was $25 to $35 a ton for 50 per cent ore in full cargoes of about 7,500 tons. The retail prices ran as high as $45 a ton, and one lot of 2 tons was imported at $125 a ton. This was ground and packed in casks.

"The price at which chromic iron ore was sold on the Pacific coast during 1915 ranged from $11 to $18 a ton f. o. b. cars at point of shipment. On ore that contains more than 50 per cent chromic oxide a premium of 50 cents a ton is paid for each additional per cent unit of chromic oxide. The silica permissible in 50 per cent ore is limited to 8 per cent, and a penalty of 50 cents a ton is imposed for each additional unit in excess of that quantity.

"The price of bichromate of soda on January 1, 1915, was $4^3/4$ to 5 cents a pound, varying with the quantity, and for bichromate of potash about 10 cents a pound. There has been a continuous advance in the price of these commodities since the spring of 1915, caused by the increase in the prices of raw materials and the uncertainty of receiving them and also by the unprecedented demand for bichromates, a demand due to the unusual conditions abroad. Domestic manufacturers are shipping bichromates freely to foreign countries, and owing to the lack of coal-tar dyes there is a large temporary increase in the use of bichromates in the textile industry at home.

"The price of chrome brick fluctuated during 1915 from $50 to $90 a thousand for standard sizes, and on account of freight is usually $2.50 to $3 a ton higher at inland cities than at the seaboard."

Contributions to the Hydrology of the United States. NATHAN C. GROVER. Water Supply Paper 375. 180 pp., and numerous plates. This is a consolidation of seven separate articles relating to water supply conditions. Only those portions in which water analyses are reported are of chemical interest. Such analyses are given only for the following districts: Sacramento Valley, California, Paradise Valley, Arizona, and Lasalle and McMullen counties, Texas.

Fuel Briquetting in 1915. C. E. LESHER. Separate 1, from Mineral Resources, 1915, Part II, pp. 1-6. Published May 6, 1916. "Little change was recorded in the fuel-briquetting industry in 1915. The manufacture of this type of fuel is still in its infancy in the United States and probably many years will elapse before it assumes large proportions. The production in 1915 amounted to about 222,000 short tons, valued at about a million dollars, and although there was a decrease, as compared with 1914, of 11 per cent in quantity and 10 per cent in value, the output exceeded that of all years prior to 1914."

Sand-Lime Brick in 1915. JEFFERSON MIDDLETON. Separate 2, from Mineral Resources, 1915, Part II, pp. 7-8. Published May 4, 1916. "The increase in the quantity of sand-lime brick

sold in 1915 over 1914 was 7,014,000 bricks, or 4 per cent, and the increase in value was $76,592, or 7 per cent."

Fuller's Earth in 1915. JEFFERSON MIDDLETON. Separate 3, from Mineral Resources, 1915, Part II, pp. 9–12. Published May 6, 1916. "The fuller's earth industry continued to show progress in 1915, the product marketed increasing in both quantity and value. The increase in quantity was 6,920 short tons, or 17 per cent, and in value $85,573, or 21 per cent, over 1914, the year of largest production and value prior to 1915. Imports of fuller's earth decreased in 1915, compared with 1914, 22 per cent in both quantity and value. Almost from the inception of the industry in this country the domestic production and value and the average price per ton have exceeded those of imports. In 1915 the quantity of domestic earth marketed was more than twice as great as that imported, the value of the domestic output was more than three times that of the imports, and the average price per ton of domestic earth was $2.37 (nearly a third) more than that of the imported earth.

"Fuller's earth obtains its name from its original use in fulling cloth. Little domestic earth is now used in this country for that purpose, the principal use being in bleaching, clarifying, or filtering fats, greases, and oils. It is also used in the manufacture of pigments for printing wall papers, for detecting certain coloring matters in some food products and as a substitute for talcum powder."

Geology and Ground Waters of Northeastern Arkansas. LLOYD W. STEPHENSON AND ALBERT F. CRIDER. Water Supply Paper 399, with supplement discussing the Chemical Character of the Water by R. B. DOLE. 309 pp. The interesting section of the publication from the chemical standpoint is the supplement which includes a discussion of the characteristics of waters which are desirable for various uses, describes the treatment of the characteristic water of this region, and tabulates numerous analyses of these supplies.

Petroleum Withdrawals and Restorations Affecting the Public Domain. MAX W. BALL. Bulletin 623. 427 pp., with numerous large maps. Paper, $1.10. This is a summary with accurate copies of orders of withdrawal, restoration, modification and classification, together with certain correspondence bearing on these questions, as affecting the policy of petroleum lands in the Public Domain.

Some Manganese Mines in Virginia and Maryland. D. F. HEWETT. Bulletin 640–C, from Contributions to Economic Geology, 1916, Part I, pp. 37–71. Published June 22, 1916. The minerals and nature of deposits with suggestions as to quantity, treatment, and handling of these ores in this district are discussed in considerable detail.

Molybdenite and Nickel Ore in San Diego County, California. F. C. CALKINS. Bulletin 640–D, from Contributions to Economic Geology, 1916, Part I, pp. 73–82. Published July 8, 1916. This report discusses the occurrence of molybdenite from the standpoint of further prospecting. It suggests the possibility of further development that will warrant commercial operations, but prospectors are warned against being too optimistic because of the rich showings at certain points on two accounts *first*, because of the apparent strong showing of small deposits; and *second*, because of the uncertainty as to the continuity of deposits.

The section on nickel ore discusses the character of ore showing probable origin and mineral characteristics of the material found in this region. The direction in which to look for further ore bodies is suggested, but the uncertainty of the problem is clearly set forth.

Ozokerite in Central Utah. HEATH M. ROBINSON. Bulletin 641–A, from Contributions to Economic Geology, 1916, Part II, pp. 1–16. Published June 13, 1916. This bulletin includes a discussion of the history of the discovery and development of ozokerite with sections on its composition and properties, tests, form and content of deposits, origin, and statistics of comparison with foreign fields, imports, production, etc. Methods of concentration and the uses of this mineral are also set forth.

A Reconnaissance of the Archean Complex of the Granite Gorge, Grand Canyon, Arizona. L. F. NOBLE AND J. FRED. HUNTER. Professional Paper 98–I, from Shorter Contributions to General Geology, 1916, pp. 95–113. Published July 7, 1916.

Gold, Silver, Copper, Lead, and Zinc in the Eastern States in 1915. Mineral Resources of the United States, 1915, Part I, pp. 7–20. Published June 7, 1916. "This section of the Mineral Resources report deals primarily with the mines of the Eastern States from which gold, silver, copper, lead, and zinc were produced in 1915. For convenience in the collection of statistics the zinc and copper mines of eastern Tennessee are grouped with the Eastern States.

"The total value of the output of gold, silver, copper, lead, and zinc in the Eastern States was $29,968,372 in 1915, a notable increase of $18,530,665, or nearly 158 per cent, over the production in 1914. The total value is distributed among the various metals as follows: Gold, $224,250; silver, $51,990; copper, $3,285,229; lead, $32,775; zinc, $26,374,128.

"The great increase in value is due largely to the increased output of the zinc mines, and, of course, to the increase in price of all the metals except gold and silver."

The report is principally devoted to a review, by states, of production and mines statistics.

Asbestos in 1915. J. S. DILLER. Mineral Resources of the United States, Part II, pp. 13–18. Published May 24, 1916. "The United States marketed a domestic production of 1,731 short tons of asbestos in 1915, valued at $76,952. As compared with the production of 1914, this represents a gain of 484 tons, or 39 per cent, in quantity, and of $57,987, or 306 per cent, in value. The great increase in value was due to the high grade of the material produced.

"The average annual production of asbestos in the United States (for the last 26 yrs.) from 1890 to 1915, inclusive, has been 1,470 short tons, ranging from a minimum of 50 tons in 1893 to a maximum of 7,604 tons in 1911. In 1911 and 1912, when there was a large production in Vermont, the total value of the output exceeded that of the production in 1915. But on account of the high grade of the Arizona asbestos the average value per ton in 1915 was $44.46, against $15.77 and $19.98, respectively, in 1911 and 1912.

"The principal source of the asbestos sold in the United States is in Quebec, Canada, where also prices are controlled. The average price in New York has been gradually rising, especially for the higher grades, and the rise continues. Early in April, 1915, No. 1 crude was selling as high as $500 a ton, and No. 2 at $300.

"The principal source of the world's supply of asbestos is Canada. The United States, being Canada's nearest neighbor and having every facility for intercourse, has naturally become the greatest importer of raw material from Canada and the largest manufacturer of asbestos products.

"Unmanufactured asbestos is admitted to the United States free, but manufactured asbestos is dutiable at from 25 to 40 per cent *ad valorem*.

"The imports of manufactured asbestos from Canada in 1915 were nearly 34 per cent greater than in 1914; but it is not certain how much of the imported material remained in the United States for consumption and how much was reshipped to other countries.

"It is of interest to note that practically all of the imported unmanufactured asbestos came from Canada, but that manufactured products of asbestos were imported from 10 countries."

Slate in 1915. G. F. LOUGHLIN. Mineral Resources of the United States, 1915, Part II, pp. 19–31. Published May 25, 1916.

"The depression which began to affect the slate industry in October, 1914, continued throughout the greater part of 1915, and the total value of slate sold in the latter year decreased 13 per cent. For the first time since 1901 the total value of slate sold fell below $5,000,000. Roofing slate decreased 5 per cent in quantity and 10 per cent in value. For the first time since 1898 the number of squares sold was less than 1,000,000 and for the first time since 1900 their value fell below $4,000,000. Mill stock decreased nearly 15 per cent in quantity and 17 per cent in value, the lowest value since 1908. Blackboard material and school slates ("other uses") decreased nearly 31 per cent."

Fluorspar in 1915; with a Note on Cryolite. ERNEST F. BURCHARD. Mineral Resources of the United States, 1915, Part II, pp. 33–41. Published June 5, 1916. "The year 1915 was a record year in the fluorspar industry in the United States, the output exceeding that of the next highest year, 1912, by more than 20,000 tons or nearly 18 per cent.

"The total quantity of domestic fluorspar reported to the Survey as sold in 1915 was 136,941 short tons, valued at $764,475, an increase in quantity of 41,825 short tons and in value of $194,434, representing nearly 44 per cent of the quantity and about 34 per cent of the value of the product marketed in 1914. The average price per ton for the whole country, all grades of fluorspar, gravel lump, and ground considered, was approximately $5.58 in 1915, compared with $5.99 in 1914, a decrease of nearly 7 per cent. This value represents the selling price on board cars or barges at railroad or water shipping points; and with reference to the product from Colorado, New Mexico, and New Hampshire, the price reported for much of the spar includes the cost of a long wagon haul—$1.50 to $3 a ton. In Illinois the principal producing mines are near river transportation and many of the mines reporting from Kentucky are near a railroad, so that the cost of long wagon hauls has not entered to an important extent into the reported value of the fluorspar from those States.

"The imports of fluorspar into the United States entered for consumption in 1915 were 7,167 short tons, valued at $22,878, as compared with 10,205 short tons, valued at $38,943, in 1914. This represents a decrease of nearly 30 per cent in quantity and of about 41 per cent in value. The price assigned to the imports in 1915 averaged $3.19 a ton, as compared with $3.82 a ton in 1914, a decrease of 63 cents a ton or about 16.5 per cent. The imports of fluorspar in 1915 were equivalent to about 6.3 per cent of the domestic production of gravel spar, as compared with nearly 13 per cent in 1914.

"The market for the bulk of the fluorspar sold in the United States depends on the steel industry, and the demand for fluorspar fluctuates with the rise and fall in the production of steel. Gravel spar is consumed as a flux in basic open-hearth steel furnaces and to a smaller extent in other metallurgical operations. In both 1914 and 1915 the sales of gravel spar constituted between 83 and 84 per cent of the total marketed output of domestic fluorspar. Fluorspar is used also as a flux in blast furnaces, iron foundries, and in silver, copper, and lead smelters; in the manufacture of fluorides of iron and manganese for steel fluxing; in the manufacture of glass, enameled, and sanitary ware, and of hydrofluoric acid; in the electrolytic refining of antimony and lead; and in the production of aluminum. In the last process fluorspar is reported to be fused with bauxite and soda ash into a so-called artificial cryolite, to which more bauxite is added, and from this mixture aluminum is extracted in the electric furnace.

"No cryolite is produced in the United States, the entire supply used in this country being imported from Greenland.

"The quantity of cryolite reported to have been imported for consumption in the United States in 1915 was 3,940 long tons, valued at $82,750, as compared with 4,612 long tons, valued at $94,424, in 1914. The average price per ton declared in 1915 was apparently $21 as compared with $20.47 in 1914. Cryolite is now imported free of duty."

BUREAU OF STANDARDS

Determination of Volatile Thinner in Oil Varnish. E. W. BOUGHTON. Technologic Paper 76. 6 pp. Paper, 5 cents. "It has been shown that any of the methods described above give results which, though only approximate from a scientific standpoint, are satisfactorily accurate for practical purposes. The volatilization of the thinner, in some cases turpentine and in others mineral spirits, from drying films of freshly prepared varnishes was practically complete in 2 hrs. at room temperature (30 to 35° C.)."

Regulation of Electrotyping Solutions. ANONYMOUS. Circular 52, 2nd Ed. 42 pp. Paper, 10 cents. "The principal new information in this edition is that relating to the study of copper electrotyping baths, from which certain specific recommendations have been made. There are also included in this edition tables giving the density of acid copper sulfate solutions and tables showing the weight and thickness of the copper deposited from such solutions by definite current densities in specified periods of time."

CONGRESSIONAL COMMITTEES

The following Congressional Committee reports contain material of chemical interest:

Uniform Standards of Classification for Cotton. House Report 777, to accompany House Bill 15913 which proposes to authorize the Secretary of Agriculture to establish uniform standards of classification for cotton, etc.

Mineral Resources of Certain National Forests. House Report 772, 1 page. This report is to accompany House Bill 12885 which proposes to authorize the Secretary of Agriculture to permit prospecting, development, and utilization of mineral resources of certain national forest regions.

Linseed Oil. This is a Senate Report on hearings before the Sub-committee having in charge the Senate Bill 1289 which is intended to prevent manufacture, sale, or transportation of adulterated, mislabeled, or misbranded linseed oil, turpentine, or paint. 101 pages. Available only through the members of the United States Senate.

INTERNAL REVENUE COMMISSIONER

Denatured Alcohol. Treasury Decision 2326. This decision (Vol. 30, No. 18) gives formula No. 22 for denatured alcohol which is a special preparation for use in formaldehyde solutions. Available as a 1-page separate.

BUREAU OF FOREIGN AND DOMESTIC COMMERCE

Saving of Waste Paper Material. ANONYMOUS. This is an unnumbered circular issued May 10th, describing methods for selecting, sorting, and disposal of waste paper, rags, and other paper-making material. It is of chemical interest only as indicating the sources and methods of handling waste materials which are useful in the paper trades.

Statistical Abstract of the United States, 1915. This is No. 38 in this series of annual publications. Available through the Superintendent of Documents, bound in paper, 50 cents. 740 pp. This publication is entirely tabular in nature consisting of summaries of statistics, among others including natural resources, agriculture, forestry, fisheries, manufacturing, mining, foreign and domestic commerce, consumption estimates, prices, financial statistics and other data collected by various Governmental agencies.

DEPARTMENT OF AGRICULTURE

The following articles in the Journal of Agricultural Research for May and June are of chemical interest:

Test of Three Large-Sized Reinforced-Concrete Slabs under Concentrated Loading. A. T. GOLDBECK AND E. B. SMITH. Journal of Agricultural Research, 6 (May 8), 205–34.

Effect of Climatic Factors on the Hydrocyanic-Acid Content of Sorghum. J. J. WILLAMAN AND R. M. WEST. Journal of Agricultural Research, 6 (May 15), 261–272.

Production of Clear and Sterilized Anti-Hog-Cholera Serum. M. DORSET AND R. R. HENLEY. Journal of Agricultural Research, 6 (May 29), 333–338.

Effect of Röntgen on the Tobacco, or Cigarette, Beetle and the Results of Experiments with a New Form of Röntgen Tube. G. A. RUNNER. Journal of Agricultural Research, 6 (June 12), 383–389.

Stimulating Influence of Arsenic upon the Nitrogen-Fixing Organisms of the Soil. J. E. GREAVES. Journal of Agricultural Research, 6 (June 12), 389–416.

Availability of Mineral Phosphates for Plant Nutrition. W. L. BURLISON. Journal of Agricultural Research, 6 (June 26), 485–514.

Legal Standards for Dairy Products. ANONYMOUS. This is a revised summary issued May 1st, giving the legal standards for dairy products in force on July 1, 1915. The following products are listed for each state or major political division of the United States including insular possessions: Milk, skimmed milk, cream, butter, whole cream cheese, skimmed milk cheese, condensed milk, evaporated milk and ice cream.

Farm Manures and Fertilizers. Unnumbered leaflet of the States Relations Service which discusses the value, methods of handling, and proper application of farm manures and fertilizers. Summaries of the characteristic analyses, costs, and value of each typical fertilizer are of particular interest to the soil chemist.

COMMERCE REPORTS—JUNE, 1916

A number of plants yielding dyes are found in abundance in Ecuador. (P. 817.)

The manufacture of aniline dyes has been initiated at Amsterdam, Holland. (P. 833.)

The beet sugar industry of Canada shows a marked increase. (P. 865.)

A new superphosphate factory is to be erected in Holland. (P. 866.)

Exportation of logwood from Tabasco, Mexico, has greatly increased. (P. 890.)

A large assortment of artificial dyes is now being made in Japan. (P. 890.)

Considerable tanning extract, derived from mangrove bark and leaves is now being exported from Brazil. (P. 900.)

The important vegetable oils expressed in Marseilles are peanut, cocoanut, and palm kernel. (P. 922.)

Extensive deposits of alunite, containing about 5 per cent of potash, have been discovered on Vancouver Island, British Columbia. (P. 947.)

Data on the world's production of fertilizers, collected by the International Institute of Agriculture, include the figures for 1912 and 1913 for natural phosphates, basic slag, superphosphate, potash salts, sodium nitrate, calcium nitrate, ammonium sulfate, and calcium cyanamide. (P. 986.)

Among available sources of alcohol in the Philippines, are sugar beet, sugar cane, beet and cane molasses, cassava, potato culls, sweet potatoes, maize, nipa sap and cocoa sap. Owing to the advance in the price of gasoline (to $0.50 per gal.) the use of alcohol as motor fuel is increasing. (P. 988.)

The methods of manufacturing sand-lime bricks in Europe are described. (P. 996.)

Phosphate deposits containing about 20 per cent P_2O_5 are found in Tonkin, Indo-China. (P. 1012.)

The dyestuff industry of Japan is developing rapidly, though still hampered by lack of heavy chemicals and of a market for by-products. (P. 1016.)

The annual output of zinc ores from Japan has increased from 40,000 to 70,000 tons. A new glass factory, and an aluminum plant are nearing completion. (P. 1027.)

Titanium salts (oxalate or tartrate) in connection with tannic acid are being used extensively in England as mordants for yellow, orange, khaki color, etc., on cotton. (P. 1040.)

A new hydro-electric plant for both smelting and refining of zinc is being erected in Norway. (P. 1045.)

Efforts are being made by New Zealand to increase the market for Kauri gum in the United States. (P. 1073.)

The rubber industry of the Malay Peninsula is very prosperous. Much of the rubber is being sent directly to the United States instead of *via* England, as formerly. (P. 1091.)

Since Japan acquired Formosa in 1895, the annual sugar output of Formosa has increased from 75,000 to 350,000 tons. (P. 1121.)

High-speed steel is being made by a new process in England, without using any tungsten, molybdenum, cobalt, or vanadium. (P. 1127.)

The output of copper from the Rio Tinto mines shows a marked increase, much of the product, of copper, copper ore, and sulfur ore being shipped to the United States. (P. 1133.)

Considerable amounts of zinc dust are being exported from Japan to the United States. (P. 1149.)

The bauxite and aluminum industry of this country are very active, much of the metal being used in an explosive "ammonal" consisting of aluminum powder and ammonium nitrate. (P. 1151.)

A new paper mill is to be erected near Pernambuco, Brazil. (P. 1169.)

Considerable amounts of natural dyes, principally from India, South and Central America, are being used in Bradford, England. (P. 1176.)

The annual export of chicle from Venezuela to the United States is nearly $1,000,000. (P. 1182.)

The kelp industry of British Columbia is being developed. (P. 1188.)

A Mond by-product plant, principally for the production of ammonium sulfate is being erected in South Africa. (P. 1191.)

Only one plant in France is now making aniline dyes, of which there is a great shortage. (Sup. 5d.)

SPECIAL SUPPLEMENTS ISSUED IN JULY, 1916

FRANCE, Havre, Grenoble, La Rochelle, Nice, St. Etienne—5c	UNITED KINGDOM, Liverpool—19e
FRANCE—5d	UNITED KINGDOM, Manchester—19f
NORWAY, Bergen, Stavanger—10a	UNITED KINGDOM, Bradford—19g
SPAIN, Canary Islands—15a	CHILE—41a
	PERSIA—57a

STATISTICS OF EXPORTS TO THE UNITED STATES

GRENOBLE, FRANCE— Sup. 5c	LIVERPOOL, ENGLAND —Sup. 19e	MANCHESTER, ENGLAND—Sup. 19f
Emery	Blood	Asbestos
Ferrochrome	Bones	Dyestuffs
Ferrosilicon	Coal	Aniline
Ferromanganese	Copper	Carbolic acid
Ferrotungsten	Bone dust	Sodium cyanide
Monomethyl sulfate	Fibers	Naphthylamine
	Grease	Hides
LA ROCHELLE, FRANCE —Sup. 5c	Hides	Rubber
Casein	Rubber	Indigo
Glue stock	Ferromanganese	Animal oils
Hides	Cocoanut oil	Vegetable oils
Sodium silicofluoride	Palm oil	Linoleum
	Soya oil	Paper
NICE, FRANCE—Sup. 5c	Palm kernel oil	Paper stock
Filter paper	Whale oil	Zinc dust
Glassware	Paper stock	BRADFORD, ENGLAND —Sup. 19g
		Aniline
	CHILE—Sup. 41a	Antimony compounds
BERGEN, NORWAY— Sup. 10a	Argols	Dyes
Aluminum	Beeswax	Potassium carbonate
Guano	Copper	Fibers
Hides	Glue	Grease and oils
Cod liver oil	Hides	Hides
Lubricating oil	Potassium chloride	Paper
Sugar	Sodium nitrate	CANARY ISLANDS— Sup. 15a
	Soap bark	Cochineal
	Rubber	Copper matte
PERSIA—Sup. 57a	Silver	Hides
Gum tragacanth	Zinc	Pumice
Opium	Gold	

BOOK REVIEWS

A Student's Book on Soils and Manures. By E. J. RUSSELL, D.Sc., Director of the Rothamsted Experiment Station, Harpenden. Cambridge Farm Institute Series, Cambridge: At the University Press, 1915. 190 pages of text; appendix on Soil Analysis; index; 34 illustrations. G. P. Putnam's Sons, New York City. Price, $0.90.

The text of this book is written in an easy style, readily comprehended by a layman of quite scanty technical equipment. Experiments easily performed are suggested. The text is divided into three parts: An account of the soil, the control of the soil, and fertilizers. Anyone reading the book will undoubtedly acquire in easy fashion quite a comprehensive knowledge of the art of producing crops. Technical investigators will not always agree with Dr. Russell's statements of theory but cannot cavil at his manner of making them. Practical farmers and greenhouse men in America will probably dissent pretty strongly with some of the statements which are obviously made for English conditions and from English experience.

Quotations from American sources are fairly frequent and generous. Every one pretending to keep informed on scientific matters generally, as well as students of agriculture, should read the book. Dr. Russell's very unusual knowledge of the literature of agriculture is often in evidence and every chemist and agricultural investigator will probably find something new to himself in his reading of the book. The reviewer has done so times enough to make him feel he must read it again.

FRANK K. CAMERON

Elementary Practical Metallurgy for Technical Students and Others. By J. H. STANSBIE, B.Sc., F.I.C., Associate of Birmingham University, and Lecturer on Metallurgy in the Birmingham Municipal Technical School. 151 pages. J. and A. Churchill, London; P. Blakiston's Son & Company, Philadelphia. Price, $1.40 net.

In the preface the author states that the book was prepared for the use of those who wish to obtain a practical knowledge of the common metals by means of a laboratory course. He first discusses furnaces and other apparatus, then takes up fuel, refractory materials, slags and fluxes, formation and reduction of oxides and of sulfides. This is followed by chapters on mechanical testing; non-ferrous metals and alloys; iron and steel; gold and silver.

The book is extremely elementary and "practical," and apparently written for work in a laboratory lacking modern equipment. A cut shows the wind furnace which is used for most of the work, even down to the manufacture of alloys of 50 g. To determine the calorific power of fuel the old Thompson calorimeter is used, and no other is mentioned.

For technical and night-school courses the book covers the ground in a very interesting way, and fills its purpose. The main criticism is in regard to the presentation of the subject of alloys. Their constitution ought either to have been omitted entirely, or else presented from the modern standpoint. As it is, solid solutions, eutectics, etc., are spoken of, but in such a way that the student cannot possibly obtain a correct idea of what an alloy is or the changes which take place during solidification and subsequent cooling. Such statements as "It is well known that the structure of copper-zinc alloys passes through a temperature range somewhat below a red heat. The metal becomes comparatively rotten and is readily broken up;" or, "Some metals, though well mixed in the molten state, separate either partially or completely on solidification. This is due to the fact that one metal is soluble in the other in the solid state only to a limited extent, or not at all; also to the absence of eutectics in the structure of the alloys" are misleading, to say the least.

There is still need for a book in practical metallurgy, apart from assaying, which is elementary, practical, and at the same time scientific.

WILLIAM CAMPBELL

The Metallurgists' and Chemists' Handbook. By DONALD M. LIDDELL, Consulting Metallurgical Engineer and sometime Managing Editor of *The Engineering and Mining Journal*. 603 pp. 1916. McGraw-Hill Book Co., Inc., New York and London. Price, $4.00.

The scope of this handbook is indicated by the following table of contents:

SECTION	Pages
I—Mathematics	1 – 52
II—Price and Production Statistics	53– 74
III—Physical Constants	75–232
IV—Chemical Data	233–301
V—Sampling, Assaying and Analysis	302–338
VI—Ore Dressing	339–402
VII—Cyanidation	403–412
VIII—Fuels and Refractories	413–439
IX—Mechanical Engineering and Construction	440–474
X—General Metallurgy	475–582
XI—First Aid	483–488

The author has compiled tables of factors and constants that are indispensable to the practical worker and has arranged them as systematically as it is possible to arrange such a mass of heterogeneous material. To avoid cumbersomeness, many tables of more or less special character are of necessity omitted. For the most part the author has exercised considerable care and good judgment in the selection of his material. However, it is the reviewer's opinion that if the author had seen fit to include a table of organic compounds with their properties corresponding to the table of "The Properties of the Principal Inorganic Compounds," the usefulness of the book would have been increased, particularly at the present time when organic industrial chemistry is receiving so much attention.

Besides tables of factors and constants, much useful information of a general and varied character is given. For example, a list of seventeen flotation processes is given together with a brief description of each and the names of concerns using the same.

The volume is bound in a good flexible leather binding, the type is clear and legible, the paper of a good grade, though rather too transparent. The size is $4 \times 7 \times {}^3/_4$ in. A complete workable index is given, thus making the material contained in the volume readily accessible. Altogether it is a very usable book either in the library or the plant.

As is almost invariably the case with first editions, errors are frequent. No attempt has been made to scan the book for errata, but those observed incidental to its use in the laboratory are here noted:

	Molecular Weight	Correction
P. 210—Ammonium Chromate, $(NH_4)_2CrO_4$	136.08	152.08
P. 221—Potassium Acetate, $K_2C_2H_3O_2$	137.22	$KC_2H_3O_2$ 98.13
P. 223—Sodium Acetate, $Na_2C_2H_3O_3.3H_2O$	175.27	136.08
P. 224—Sodium Sulfate, Na_2SO_4	119.07	142.06

P. 211—Ammonium phosphomolybdate. Formula given is $(NH_4)_3PO_4.10MoO_3.3H_2O$. The ratio of P_2O_5 to MoO_3 is 1 : 24 instead of 1 : 20.

The author has met a long-felt need among metallurgists and chemists for a handy reference book that is more than a collection of figures and tables.

A. A. BACKHAUS

NEW PUBLICATIONS

By IRENE DeMATTY, Librarian, Mellon Institute of Industrial Research, Pittsburgh

Alternating Current Phenomena. C. P. STEINMETZ. 5th Ed. 8vo. Price, $4.00. McGraw-Hill Book Co., New York.

Canning of Fruits and Vegetables. J. P. ZWALLA. 8vo. 226 pp. Price, $2.50. John Wiley & Sons, Inc., New York.

Chemistry in the Service of Man. ALEXANDER FINDLAY. 8vo. 255 pp. Price, $1.60. Longmans, Green & Co., New York.

Chemists: Kelly's Directory of Chemists and Druggists, Including Manufacturing Chemists. 843 pp. Price, 25 s. Kelly's Directories, Ltd., London.

Coal Miners' Pocketbook. 11th Ed. 16mo. 1200 pp. Price, $4.00. McGraw-Hill Book Co., New York.

Diesel or Slow-Combustion Oil Engine. G. J. WELLS AND A. J. WALLIS-TAYLER. 8vo. 304 pp. Price, $3.00. D. Appleton & Co., New York.

Drainage: Textbook of Land Drainage. J. A. JEFFREY. 12mo. 256 pp. Price, $1.25. The Macmillan Co., New York.

Engineering Applications of Higher Mathematics. VLADIMIR KARAPETOFF. 8vo. Pt. 2, 3, 4, 5. Price, ea. $0.75. John Wiley & Sons, Inc., New York.

Engineering Training: The Principles of Apprentice Training with Special Reference to the Engineering Industry. A. P. M. FLEMING AND J. G. PEARCE. 12mo. 202 pp. Price, $1.20. Longmans, Green & Co., New York.

Essences: Manual for the Essence Industry. ERICH WALTER. 8vo. 427 pp. Price, $4.00. John Wiley & Sons, Inc., New York.

Explosives: Industrial Nitrogen Compounds and Explosives. GEOFFREY MARTIN AND W. BARBOUR. 4to. 125 pp. Price, $2.50. D. Appleton & Co., New York.

Fertilizers: The Source, Character and Composition of Natural, Home-made, and Manufactured Fertilizers. E. B. VOORHEES. Rev. Ed. 12mo. 365 pp. Price, $1.50. The Macmillan Co., New York.

Gas Engine Ignition. E. B. NORRIS AND OTHERS. 8vo. 174 pp. Price, $1.50. McGraw-Hill Book Co., New York.

Heating and Ventilating Ducts: Practical Sheet Metal Duct Construction. W. NEUBECKER. 8vo. 194 pp. Price, $2.00. Sheet Metal Publishing Co., New York.

Industrial Chemistry: Outlines of Industrial Chemistry. F. H. THORP. 3rd Ed. 8vo. 660 pp. Price, $3.75. The Macmillan Co., New York.

Industrial Preparedness. C. E. KNOEPPEL. 12mo. 145 pp. Price, $1.00. Engineering Magazine Co., New York.

Inorganic Chemistry: Conférences de chimie minerale. MARCEL GUICHARD. 2nd Ed. 8vo. 422 pp. Price, 15 fr. Gauthier Villars et Cie., Quai des Grands Augustins, 55, Paris.

Limes and Cements. E. A. DANCASTER. 12mo. 212 pp. Price, $1.75. D. Appleton & Co., New York.

Materials: Mechanical Handling and Storing of Materials. G. F. ZIMMER. New Ed. 4to. 766 pp. Price, 42 s. Crosby Lockwood & Son, London.

Mechanical Technology. G. F. CHARNOCK. 8vo. 635 pp. Price, $3.00, net. D. Van Nostrand Co., New York.

Mineral Resources of Minas Geraes (Brazil). 8vo. 100 pp. Price, $2.00. Spon & Chamberlain, New York.

Oil: Principles of Oil and Gas Production. R. H. JOHNSON AND L. G. HUNTLEY. 8vo. 371 pp. Price, $3.75. John Wiley & Sons, Inc., New York.

Oil and Petroleum Manual for 1916. W. R. SKINNER. 8vo. 257 pp. Price, 2 s., 6 d. W. R Skinner, London.

Organic Compounds: A Method for the Identification of Pure Organic Compounds. Vol. 2. Compounds of Order 2. S. P. MULLIKEN. 8vo. 336 pp. Price, $5.00. John Wiley & Sons, Inc., New York.

Paper and Its Uses. E. A. DAWE. 8vo. 162 pp. Price, $1.50. D. Appleton & Co., New York.

Power Transmission by Leather Belting. R. T. KENT. 8vo. 114 pp. Price, $1.25. John Wiley & Sons, Inc., New York.

Sanitation: The Elements of Physiology and Sanitation. L. J. RETTGER. 12mo. 389 pp. Price, $0.80. A. S. Barnes Co., New York.

Science: Laboratory Courses in General Science. HERBERT BROWNELL. 12mo. 212 pp. Price, $0.80. The Macmillan Co., New York.

Steam Traction Engineering. S. R. EIGHINGER AND N. S. HUTTON. 8vo. 317 pp. Price, $2.50. D. Appleton & Co., New York.

Steels: British Standard Specifications for Wrought Steels. 24 pp. Price, 7 s., 6 d. Crosby Lockwood & Son, London.

Steel: The Metallurgy of Steel. F. W. HARBORD AND J. W. HALL. 2 vols. 5th Ed. 568 pp. Price, 36 s. Chas. Griffin & Co., London.

Tars: British Standard Nomenclature of Tars, Pitches, Bitumens and Asphalts. 41 pp. Price, 5 s. Crosby Lockwood & Son, London.

Water Power: The Modern Development of Water Power. A. STEIGER. Price, 2 s. E. & F. N. Spon, London.

RECENT JOURNAL ARTICLES

Acetylene or Electricity. A. G. RAKESTRAW. *Acetylene Journal*, Vol. 18 (1916), No. 1, pp. 21–22.

Alcohol: Ethyl Alcohol from Wood. F. W. KRESSMANN. *Metallurgica and Chemical Engineering*, Vol. 15 (1916), No. 2, pp. 78–82.

Antimony: Determination of Antimony. H. R. LAYNG. *Mining and Scientific Press*, Vol. 113 (1916), No. 2, pp. 57–58.

Arsenic: Stimulating Influence of Arsenic upon Nitrogen-Fixing Organisms of the Soil. J. E. GREAVES. *Journal of Agricultural Research*, Vol. 6 (1916), No. 11, pp. 389–416.

Blasting Practice in Chuquicamata, Chile. H. W. MOORE. *Mining and Scientific Press*, Vol. 113 (1916), No. 2, pp. 60–61.

Blast Furnace: The Rate of Driving the Blast Furnace. J. E. JOHNSON, JR. *Metallurgical and Chemical Engineering*, Vol. 15 (1916), No. 1, pp. 21–26.

Carbon-Dioxide Refrigerating Machine. F. T. FLENNIKEN. *Power*, Vol. 43 (1916), No. 26, pp. 903–905.

Catalysis in the Formation of Gasoline from Kerosene. GUSTAV EGLOFF AND R. J. MOORE. *Metallurgical and Chemical Engineering*, Vol. 15 (1916), No. 2, pp. 67–69.

Coal: Powdered Coal as a Blast Furnace Fuel. *Blast Furnace and Steel Plant*, Vol. 50 (1916), No. 7, pp. 315–316.

Concrete: Tests of Reinforced Concrete Pipe. J. S. LAMBIE. *Iron Trade Review*, Vol. 59 (1916), No. 2, pp. 81–82.

Copper: Comparisons between Electrolytic and Two Varieties of Arsenical Lake Copper with Respect to Strength and Ductility in Cold-Worked and Annealed Test Strips. C. H. MATHEWSON. *Transactions of the American Institute of Mining Engineers* (1916), No. 115, pp. 1185–1215.

Copper: Sources of Metal Loss in Copper Refining. LAWRENCE ADDICKS. *Metallurgical and Chemical Engineering*, Vol. 15 (1916), No. 1, pp. 11–15.

Copper Ores: Drilling and Analysis of Copper Ores. A. J. SALE. *Engineering and Mining Journal*, Vol. 102 (1916), No. 2, pp. 87–90.

Cyaniding Copper-Bearing Ores. P. W. GAEBELEIN. *Engineering and Mining Journal*, Vol. 102 (1916), No. 1, pp. 22–23.

Engine-Room Lubrication. G. A. TOWNSEND. *Power*, Vol. 43 (1916), No. 26, pp. 905–907.

Fertilizer: Making Basic Phosphate Fertilizer. *Iron Trade Review*, Vol. 59 (1916), No. 1, pp. 23–28.

Filter Paper: The Manufacture of Chemical Filter Paper. E. F. BEVAN AND W. BACON. *The Analyst*, Vol. 41 (1916), No. 483, pp. 159–165.

Fire Brick: How to Conduct Fire Brick Tests. C. E. NESBITT AND M. L. BELL. *Iron Trade Review*, Vol. 59 (1916), No. 2, pp. 71–78.

Flotation: Apparatus Used in Flotation. H. A. MEGRAW. *Engineering and Mining Journal*, Vol. 102 (1916), No. 1, pp. 5–8.

Flotation of Minerals. R. J. ANDERSON. *Mining and Scientific Press*, Vol. 113 (1916), No. 2, pp. 47–51.

Furnace and Boiler Losses. R. CEDERBLOM. *Power*, Vol. 44 (1916), No. 2, pp. 45–47.

Furnaces: How Tuyères Indicate Furnace Conditions. W. G. IMHOFF. *Blast Furnace and Steel Plant*, Vol. 50 (1916), No. 7, pp. 326–328.

Oil: The Formation of Aromatic Compounds from the Cracking of a Gas Oil. GUSTAV EGLOFF AND T. J. TWOMEY. *Metallurgical and Chemical Engineering*, Vol. 15 (1916), No. 1, pp. 15–18.

Platinum on the Pacific Coast. T. W. GRUETTER. *Mining and Scientific Press*, Vol. 113 (1916), No. 1, pp. 20–21.

Potassium: The Estimation of Potassium in Presence of Other Substances. ALEX. H. BENNETT. *The Analyst*, Vol. 41 (1916), No. 483, pp. 165–168.

Power Factor Explained. F. A. ANNETT. *Power*, Vol. 44 (1916), No. 1, pp. 4–7.

Rock Analysis: A Recording Micrometer for Geometrical Rock Analysis. S. J. SHAND. *The Journal of Geology*, Vol. 24 (1916), No. 4, pp. 394–404.

Steel: Cutting Efficiency of Tool Steel. J. O. ARNOLD. *Iron and Steel*, Vol. 59 (1916), No. 1, p. 21.

Steel: The Effect of Sulfur in Rivet Steel. J. S. UNGER. *Blast Furnace and Steel Plant*, Vol. 50 (1916), No. 7, pp. 333–338.

Tungsten: Specific Gravity Method for Tungsten Analysis. J. J. RUNNER. *Mining and Scientific Press*, Vol. 113 (1916), No. 1, pp. 11–13.

Turbines: Efficiency Tests of a 30,000 Kw. Cross Compound Steam Turbine. H. G. STOTT AND W. S. FINLAY. *The Electric Journal*, Vol. 13 (1916), No. 7, pp. 335–339.

Valves: New Valves for Regenerative Furnaces. J. A. HERRICK. *Iron Trade Review*, Vol. 59 (1916), No. 2, p. 84.

MARKET REPORT—JULY, 1916

WHOLESALE PRICES PREVAILING IN THE NEW YORK MARKET ON JULY 21.

INORGANIC CHEMICALS

Item	Unit	Low		High
Acetate of Lime, gray	100 Lbs.	7.00	@	7.05
Alum, lump ammonia	100 Lbs.	4.00	@	—
Aluminum Sulfate, high-grade	Ton	65.00	@	70.00
Ammonium Carbonate, domestic	Lb.	9½	@	10
Ammonium Chloride, gray	Lb.	10	@	15
Ammonium Phosphate, commercial, 98–100%	Lb.	—	@	—
Aqua Ammonium, 26°, drums	Lb.	5	@	5½
Arsenic, white	Lb.	6⅛	@	6½
Barium Chloride	Ton	100.00	@	105.00
Barium Nitrate	Lb.	18	@	19¼
Barytes, prime white, foreign	Ton	—	@	—
Bleaching Powder, 35 per cent	100 Lbs.	4½	@	4¾
Blue Vitriol	Lb.	8¾	@	9
Borax, crystals, in bags	Lb.	6¾	@	7
Boric Acid, powdered crystals	Lb.	11¼	@	12½
Brimstone, crude, domestic	Long Ton	30.00	@	32.00
Bromine, technical, bulk	Lb.	2.40	@	2.50
Calcium Chloride, lump, 70 to 75% fused	Ton	26.00	@	28.00
Calcium Chloride, granulated	Ton	14.78	@	15.00
Caustic Soda, 74 per cent	Lb.	3½	@	¾
Chalk, light precipitated	Lb.	4½	@	5¼
China Clay, imported	Ton	18.00	@	30.00
Feldspar	Ton	8.00	@	12.00
Fuller's Earth, foreign, powdered	100 Lbs.	80	@	1.05
Glauber's Salt, in bbls.	100 Lbs.	65	@	70
Green Vitriol, bulk	100 Lbs.	1.25	@	1.50
Hydrochloric Acid, commercial, 18°	Lb.	2½	@	2¾
Hydrochloric Acid, C. P., conc., 22°	100 Lbs.	2¾	@	3
Iodine, resublimed	Lb.	4.25	@	4.30
Lead Acetate, white crystals	Lb.	15⅝	@	15⅞
Lead Nitrate	Lb.	15½	@	16
Litharge, American	Lb.	9¼	@	9¾
Lithium Carbonate	Lb.	97	@	98
Magnesium Carbonate	Lb.	19	@	22
Magnesite, "Calcined"	Ton	—	@	—
Nitric Acid, 36°	Lb.	7¼	@	7½
Nitric Acid 42°	Lb.	8¼	@	—
Phosphoric Acid, sp. gr. 1.750	Lb.	30	@	31
Phosphorus	Lb.	—	@	—
Plaster of Paris	Bbl.	1.50	@	1.70
Potassium Bichromate, casks	Lb.	37	@	38
Potassium Bromide (granular)	100 Lbs.	2.90	@	3.00
Potassium Carbonate, calcined, 80 @ 85%	Lb.	80	@	85
Potassium Chlorate, crystals, spot	Lb.	45	@	47
Potassium Cyanide, bulk, 98–99 per cent	Lb.	—	@	—
Potassium Hydroxide	Lb.	83	@	90
Potassium Iodide, bulk	Lb.	3.90	@	—
Potassium Nitrate	Lb.	—	@	—
Potassium Permanganate, bulk	Lb.	1.60	@	1.70
Quicksilver, flask, 75 lbs.		80.00	@	—
Red Lead, American, dry	Lb.	9¾	@	—
Salt Cake, glass makers'	Ton	9.00	@	10.00
Silver Nitrate	Oz.	39⅜	@	—
Soapstone in bags	Ton	8.00	@	12.00
Soda Ash, 48 per cent	100 Lbs.		2½	
Sodium Acetate	Lb.	14	@	15
Sodium Bicarbonate, domestic	100 Lbs.	1.75	@	2.00
Sodium Bicarbonate, English	Lb.	3½	@	3⅝
Sodium Bichromate	Lb.	35	@	37
Sodium Chlorate	Lb.	30	@	32
Sodium Fluoride, commercial	Lb.	—	@	—
Sodium Hyposulfite	100 Lbs.	2.00	@	—
Sodium Nitrate, 95 per cent, spot	100 Lbs.	3.10	@	—
Sodium Silicate, liquid	100 Lbs.	1.25	@	1.75
Sodium Sulfide, 30%, crystals, in bbls.	Lb.	2	@	2½
Sodium Bisulfite, powdered	Lb.	6	@	7
Strontium Nitrate	Lb.	42	@	45
Sulfur, flowers, sublimed	100 Lbs.	2.30	@	2.70
Sulfur, roll	100 Lbs.	1.95	@	2.25
Sulfuric Acid, chamber, 60°	Ton	$25.00	@	—
Sulfuric Acid, conc., sp. gr. 1.842	100 Lbs.	2.50	@	3.00
Sulfuric Acid, oleum (fuming)	100 Lbs.	5	@	—
Talc, American white	Ton	9.00	@	12.00
Terra Alba, American, No. 1	100 Lbs.	75	@	80
Tin Bichloride, 50°	Lb.	15¼	@	16
Tin Oxide	Lb.	49	@	51
White Lead, American, dry	Lb.	8¾	@	—
Zinc Carbonate	Lb.	24	@	28
Zinc Chloride, commercial	Lb.	13	@	15
Zinc Oxide, American process	Lb.	8⅝	@	8⅞
Zinc Sulfate	Lb.	6½	@	7¼

ORGANIC CHEMICALS

Item	Unit	Low		High
Acetanilid, C. P., in bbls.	Lb.	65	@	65
Acetic Acid, 60 per cent, in bbls.	Lb.	13	@	14
Acetic Acid, glacial, 99½%, in carboys	Lb.	37	@	38
Acetone, drums	Lb.	40	@	42
Alcohol, denatured, 180 proof	Gal.	49	@	50
Alcohol, grain, 188 proof	Gal.	2.64	@	2.70
Alcohol, wood, 95 per cent, refined	Gal.	65	@	68
Amyl Acetate	Gal.	5.25	@	—
Aniline Oil	Lb.	37	@	40
Benzoic Acid, ex-toluol	Lb.	6.75	@	7.50
Benzol, 90 per cent	Gal.	70	@	—
Camphor, refined in bulk, bbls.	Lb.	54	@	—
Carbolic Acid, U. S. P., crystals, drums	Lb.	55	@	60
Carbon Bisulfide	Lb.	8½	@	—
Carbon Tetrachloride, drums, 100 gals.	Lb.	17	@	—
Chloroform	Lb.	44	@	50
Citric Acid, domestic, crystals	Lb.	64	@	67
Cresol, U. S. P.	Gal.	75	@	80
Dextrine, corn (carloads, bags)	100 Lbs.	3.30	@	3.40
Dextrine, imported potato	Lb.	9	@	—
Ether, U. S. P., 1900	Lb.	15	@	20
Formaldehyde, 40 per cent	Lb.	12½	@	13
Glycerine, dynamite, drums included	Lb.	40	@	45
Oxalic Acid, in casks	Lb.	58	@	60
Pyrogallic Acid, resublimed bulk	Lb.	2.75	@	—
Salicylic Acid	Lb.	2.75	@	2.80
Starch, cassava	Lb.	3¾	@	4½
Starch, corn (carloads, bags)	100 Lbs.	2.25	@	—
Starch, potato	Lb.	—	@	—
Starch, rice	Lb.	8	@	10
Starch, sago	Lb.	3½	@	4
Starch, wheat	Lb.	5	@	6
Tannic Acid, commercial	Lb.	60	@	64
Tartaric Acid, crystals	Lb.	70	@	72

OILS, WAXES, ETC.

Item	Unit	Low		High
Beeswax, pure, white	Lb.	47	@	55
Black Mineral Oil, 29 gravity	Gal.	13½	@	—
Castor Oil, No. 3	Lb.	16¾	@	20
Ceresin, yellow	Lb.	10	@	14
Corn Oil, crude	100 Lbs.	8.40	@	—
Cottonseed Oil, crude, f. o. b. mill	Gal.	69	@	71
Cottonseed Oil, p. s. y.	Lb.	10¾	@	—
Creosote, beechwood	Lb.	4.00	@	4.25
Cylinder Oil, light, filtered	Gal.	21	@	—
Fusel Oil, crude	Lb.	4.50	@	—
Japan Wax	Lb.	15¾	@	16
Lard Oil, prime winter	Gal.	1.04	@	1.08
Linseed Oil, raw (car lots)	Gal.	65	@	—
Menhaden Oil, crude (southern)	Gal.	48½	@	—
Naphtha, 68 @ 72°	Gal.	33	@	—
Neat's-foot Oil, 20°	Gal.	1.10	@	1.20
Paraffine, crude, 118 & 120 m. p.	Lb.	5	@	—
Paraffine Oil, high viscosity	Gal.	29½	@	—
Rosin, "F" Grade, 280 lbs.	Bbl.		6.90	
Rosin Oil, first run	Gal.	30	@	32
Shellac, T. N.	Lb.	26	@	26½
Spermaceti, cake	Lb.	25	@	26
Sperm Oil, bleached winter, 38°	Gal.	80	@	82
Spindle Oil, No. 200	Gal.	24	@	25
Stearic Acid, double-pressed	Lb.	12½	@	13½
Tallow, acidless	Gal.	92	@	—
Tar Oil, distilled	Gal.	30	@	31
Turpentine, spirits of	Gal.	44	@	—

METALS

Item	Unit	Low		High
Aluminum, No. 1, ingots	Lb.	58	@	60
Antimony, ordinary	Lb.	13	@	14
Bismuth, N. Y.	Lb.	3.15	@	3.25
Copper, electrolytic	Lb.		26½	
Copper, lake	Lb.		26½	
Lead, N. Y.	100 Lbs.		6.30	
Nickel, electrolytic	Lb.	—	@	—
Nickel, shot and ingots	Lb.	—	@	—
Platinum, refined	Oz.	60.00	@	65.00
Silver	Oz.		62¾	
Tin	Lb.		31⅞	
Zinc, N. Y.	Lb.		10	

FERTILIZER MATERIALS

Item	Unit	Low		High
Ammonium Sulfate	100 Lbs.		3.40	
Blood, dried	Unit		2.50	
Bone, 4½ and 50, ground, raw	Ton	—	@	—
Calcium Cyanamid	Unit of Ammonia		2.55	
Calcium Nitrate, Norwegian	100 Lbs.		—	
Castor Meal	Unit		—	
Fish Scrap, domestic, dried, f. o. b. works	Unit	3.00	&	10
Phosphate, acid	Unit	80	@	85
Phosphate rock; f. o. b. mine:				
Florida land pebble, 68 per cent	Ton	2.75	@	3.00
Tennessee, 78–80 per cent	Ton	5.00	@	5.50
Potassium "muriate," basis 80 per cent	Ton	300.00	@	325.00
Pyrites, furnace size, imported	Unit	15	@	15½
Tankage, high-grade; f. o. b. Chicago	Unit	2.65	&	10

ALPHABETICAL LIST OF ADVERTISERS

See pages 30-36 for Classified List of Chemical Equipment

Absorption Towers
Duriron Castings Co., N. Y. C.
German-American Stoneware Wks., N. Y. C.
Knight, Maurice A., Akron, Ohio.
Lummus Co., The Walter E., Boston, Mass.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.
Robinson Clay Product Co., Akron, O.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.
U. S. Stoneware Co., The, Akron, O.

Acetylene and Oxy-Acetylene Generators and Equipment
International Oxygen Co., N. Y. C.

Acid and Alkali Resistant Apparatus
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
Pfaudler Co., Rochester, N.Y., Detroit, & N.Y.C.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.

Acid Eggs
Bethlehem Fd'y & Mach. Co., So. Beth., Pa.
Buffalo Fdy & Mch. Co., Buffalo, N. Y.
Devine Co., J. P., Buffalo, N. Y.
Jacoby, Henry E., N. Y. C.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.

Acid Pitchers
Bausch & Lomb Optical Co., Rochester, N. Y.
Knight, Maurice A., Akron, Ohio.
Robinson Clay Product Co., Akron, O.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.
U. S. Stoneware Co., The, Akron, O.

Acid Proof Brick
Bausch & Lomb Optical Co., Rochester, N. Y.
German-American Stoneware Works, N. Y. C.
Knight, M. A., Akron (East Side), Ohio.
Robinson Clay Product Co., Akron, O.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.
Thermal Syndicate, Ltd., New York City.
U. S. Stoneware Co., The, Akron, O.

Acids
Herman & Herman, Inc., N. Y. C.

Acid Ware (Vitrified Clay)
Robinson Clay Product Co., Akron, O.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.

Acid Valves
Chadwick-Boston Lead Co., Boston, Mass.
Cleveland Brass Mfg. Co., The, Cleveland, O
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C
York Manufacturing Co., York, Pa

Agitators, or Mixers
Devine Co., J. P., Buffalo, N. Y.
Dorr Co., The, Denver, Col. & N. Y. C.
General Filtration Co., Inc., Rochester, N. Y
Pfaudler Co., Rochester, N.Y., Detroit & N.Y.C
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.
Werner & Pfleiderer Co., Saginaw, Mich

Air Compressors
Abbé Engineering Co., N. Y. City.
Crowell Engineering Co., Brooklyn, N. Y.
General Electric Co., Schenectady, N. Y.
Hubbard's Sons, Norman, Brooklyn, N. Y.

Air Washers
Carrier Engineering Corp., N. Y. C.

Ammeters
Brown Instrument Co., Philadelphia, Pa.

Ammonia
Unger, John S., Chicago, Ill.

Ammonia Fittings and Supplies
York Manufacturing Co., York, Pa.

Analysis—Gas Apparatus
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco, Cal.
Eberbach & Son Co., Ann Arbor, Mich.
Eimer & Amend, New York City
Heil Chem. Co., Henry, St. Louis, Mo.
Lenz & Naumann, Inc., N. Y. C.
Palo Company, N. Y. C.
Sarco Co., Inc., New York City.
Scientific Materials Co., Pittsburg, Pa.

Analytical Apparatus
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco, Cal.
Buffalo Dental Mfg. Co., Buffalo, N. Y
Eberbach & Son Co., Ann Arbor, Mich.
Eimer & Amend, New York City.
Heil Chem. Co., Henry, St. Louis, Mo
Laboratory Supply Co., Columbus, O.
Lenz & Naumann, Inc., N. Y. C.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburg, Pa
Thomas Co., Arthur H., Philadelphia, Pa.

Analytical Laboratories
Columbus Laboratories, Chicago, Il.
Dearborn Chemical Co., Chicago, Ill.
Sadtler & Son, Samuel P., Philadelphia.

Asphalt Flow Plates
Howard & Morse, N. Y. C.

Bakelite
General Bakelite Co., New York City.

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Bausch & Lomb Optical Co., Rochester, N. Y.
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Eberbach & Son Co., Ann Arbor, Mich.
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Heil Chem. Co., Henry, St. Louis, Mo.
Laboratory Supply Co., Columbus, O.
Lenz & Naumann, Inc., N. Y. C.

Mine & Smelter Supply Co., Denver, Salt Lake City, El Paso, N. Y. C.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburg, Pa.
Thomas Co., Arthur H., Philadelphia, Pa.

Belt Cement
Schieren Co., Chas. A., N. Y. C.

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Schieren Co., Chas. A., N. Y. C.

Boilers
Sowers Mfg. Co., Buffalo, N. Y.

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Dearborn Chemical Co., Chicago, Ill.
Merck & Co., N. Y. C.

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Abbé Engineering Co., N. Y. C.
Crowell Mfg. Co., Brooklyn, N. Y.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.

Bolting Cloth
Abbé Engineering Co., N. Y. C.

Bone Black
Booth Apparatus Co., Syracuse, N. Y.

Books
Blakiston's Son & Co., P., Philadelphia, Pa.
Chemical Publishing Co., The, Easton, Pa.
Longmans, Green & Co., New York City.
Van Nostrand Co., D., N. Y. C.

Briquette Molds
Howard & Morse, N. Y. C.

Brimstone, Crude
Union Sulphur Co., New York City.

Brinell Machines
Scientific Materials Co., Pittsburgh, Pa.

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Herman & Herman, Inc., N. Y. C.

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Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco, Cal.
Buffalo Dental Mfg. Co., Buffalo, N. Y.
Eberbach & Son Co., Ann Arbor, Mich.
Eimer & Amend, New York City.
Heil Chem. Co., Henry, St. Louis, Mo.
Laboratory Supply Co., Columbus, O.
Lenz & Naumann, Inc., N. Y. C.
Palo Company, N. Y. C.
Sargent, E. H. & Co., Chicago, Ill.
Scientific Materials Co., Pittsburg, Pa.

Burners—Pyrites
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.

Burners—Sulphur
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.

Burners—Sulphur (Rotary)
Glens Falls Machine Works, Glen Falls, N. Y.

Calorimeters—Fuel and Gas
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco, Cal.
Eimer & Amend, New York City.
Heil Chem. Co., Henry, St. Louis, Mo.
Lenz & Naumann, Inc., N. Y. C.
Palo Company, N. Y. C.
Sarco Co., Inc., New York City.
Sargent, E. H. & Co., Chicago, Ill.
Scientific Materials Co., Pittsburg, Pa.
Thomas Co., Arthur H., Philadelphia, Pa.

Capsules
Bausch & Lomb Optical Co., Rochester, N. Y.
Norton Co., Worcester, Mass.
Thermal Syndicate, Ltd., New York City.

Carboy Stoppers
Bausch & Lomb Optical Co., Rochester, N. Y.
Knight, Maurice A., Akron Ohio.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.

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Sharples Specialty Co., The, West Chester, Pa.

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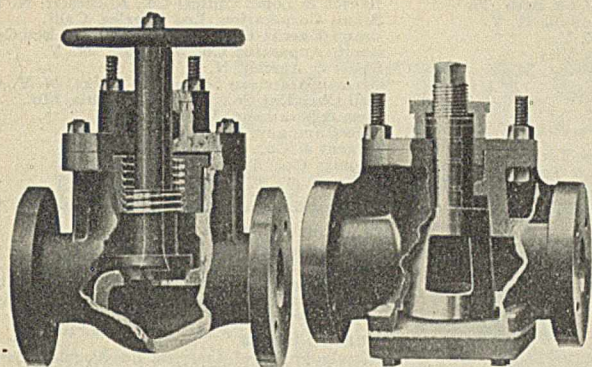
Colors—Aniline and Alizarine
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National Aniline and Chemical Co., N. Y. C.

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Spackman Eng. Co., Henry S., Philadelphia, Pa.
Unger, John S., Chicago, Ill.

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Bucyrus Copper Kettle Works, Bucyrus, O.

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Woolner & Co., Peoria, Ill.

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Brown Instrument Co., Philadelphia, Pa.
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Baker & Co., Inc., Newark, N. J. (Platinum)
Bausch & Lomb Optical Co., Rochester, N. Y.
Bishop & Co., J., Malvern, Pa. (Platinum)
Buffalo Dental Mfg Co., Buffalo, N. Y.
Dixon Crucible Co., Joseph, Jersey City, N. J.
Duriron Castings Co., N. Y. C. (Acid Proof).
Heil Chemical Co., Henry, St. Louis, Mo.
Laboratory Supply Co., Columbus, O.
Norton Co., Worcester, Mass. (Alundum).
Thermal Syndicate, Ltd., N. Y. C. (Vitreosil).

Crushers and Grinders (Industrial and Laboratory)
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Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco,Cal.
Eimer & Amend, New York City.
Heil Chemical Co., Henry, St. Louis, Mo.
Palo Company, N. Y. C.
Raymond Bros. Impact Pulv Co., Chicago, Ill.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Williams Patent Crusher & Pulv. Co., Chicago

Cupels
Bausch & Lomb Optical Co., Rochester, N. Y.
Hoskins Mfg. Co., Detroit, Michigan.
Norton Company, Worcester, Mass.

Cylinders (Oxygen and Hydrogen)
International Oxygen Co., N. Y. City.

Designing Engineers
Guarantee Construction Co., N. Y. C.

Diaphragms
General Filtration Co., Inc., Rochester, N. Y.

Diffusion Apparatus
General Filtration Co., Inc., Rochester, N. Y.
Lummus Co., The Walter E., Boston, Mass.

Dissolving Tanks—Automatic
Lummus, Co., The Walter E., Boston, Mass.
Werner & Pfleiderer Co., Saginaw, Mich.

Distilling Machinery and Apparatus
Badger & Sons Co., E. B., Boston, Mass.
Bausch & Lomb Optical Co., Rochester, N. Y.
Booth Apparatus Co., Syracuse, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co.,SanFrancisco, Cal
Devine Company, J. P., Buffalo, N. Y.
Eimer & Amend, New York City.
Electric Heating Appliance Co., N. Y. C.
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
German-American Stoneware Works, N. Y. C
Heil Chem. Co., Henry, St. Louis, Mo
Hodges Water Still Co., Woodbury, N. J.
Laboratory Supply Co., Columbus, O.
Lenz & Naumann, Inc., N. Y. C.
Lummus Co., The Walter E., Boston, Mass.
Palo Company, N. Y. C.
Pfaudler Co., Rochester, N.Y., Detroit, & N.Y.C.
Roos' Sons, August, N. Y. C.
Sanborn Evaporator Co., N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Stevens-Aylsworth Co., N. Y. C.
Stokes Machine Wks., F. J., Philadelphia.
Swenson Evaporator Co., N. Y. C.
Thomas Co., Arthur H., Philadelphia, Pa.
Unger, John S., Chicago, Ill.
Werner & Pfleiderer Co., Saginaw, Mich.
Zaremba Company, Buffalo, N. Y.

Draft Gauges
Brown Instrument Co., Philadelphia, Pa.

Dry Blast Plants
Carrier Engineering Corp., N. Y. C.

Dryers—Vacuum
Bethlehem Fd'y & Mach. Co., So. Beth., Pa.
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
Devine & Company, J. P., Buffalo, N. Y.
Jacoby, Henry E., New York City.
Stokes Machine Co., F. J., Philadelphia, Pa.
Werner & Pfleiderer Co., Saginaw, Mich.

Drying Apparatus and Machinery
Bethlehem Fd'y & Mach. Co., So. Beth., Pa.
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
Devine Co., J. P., Buffalo, N. Y.
Jacoby, Henry E., New York City.
Werner & Pfleiderer Co, Saginaw. Mich.

Ductility Machines
Howard & Morse, N. Y. C.

Dyestuffs
Farbwerke-Hoechst Co., New York City
Herman & Herman, Inc., New York City.

Dynamos and Motors
General Electric Co., Schenectady, N. Y.

Efficiency Instruments
Brown Instrument Co., Philadelphia, Pa.
Scientific Materials Co., Pittsburgh, Pa.

Electrical Instruments
Brown Instrument Co., Philadelphia, Pa.
Thwing Instrument Co., Philadelphia. Pa

Electrical Measuring Instruments
Brown Instrument Co., Philadelphia, Pa.
Leeds & Northrup Co., Phila., Pa.

Electrical Supplies
General Electric Co., Schenectady, N. Y.

Electrodes—Platinum
American Platinum Works, Newark, N. J
Baker & Co., Inc., Newark, N. J.
Bausch & Lomb Optical Co., Rochester, N. Y.
Bishop & Co., J., Platinum Works, Malvern, Pa.
Eimer & Amend, New York City.
Engelhard, Chas., New York City.
Heil Chemical Co., Henry, St. Louis. Mo.
Palo Company, N. Y. C.

Enameled Apparatus (Acid Resistant)
Elyria Enameled Prod. Co., Elyria, O.,& N.Y.C.
Pfaudler Co., Rochester, N.Y., Detroit, & N.Y.C.

Enamel Paint (Impervious to Acid Fumes)
General Bakelite Co., N. Y. C.
Toch Brothers, N. Y. C.

Evaporating Dishes
Bausch & Lomb Optical Co., Rochester, N. Y.
Duriron Castings Co., N. Y. C. (Acid Proof)
Elyria Enameled Prod. Co., Elyria, O.,& N.Y.C.
German-American Stoneware Works, N. Y.
Laboratory Supply Co., Columbus, O.
Pfaudler Co., Rochester, N.Y., Detroit, & N.Y.C.
Scientific Materials Co., Pittsburgh, Pa.
Thermal Syndicate, Ltd., New York City.

Evaporating Machinery
Badger & Sons, Co., E. B., Boston, Mass.
Bethlehem Fd'y & Mach. Co., So. Beth., Pa.
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
Devine Company, J. P., Buffalo, N. Y.
Electric Heating Apparatus Co., N. Y. C.
Jacoby, Henry E., N. Y. C.
Lummus Co., The Walter E., Boston, Mass.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.
Roos' Son, August, N. Y. C.
Sanborn Evaporator Co., New York City.
Stokes Machine Company,F.J.,Philadelphia,Pa.
Swenson Evaporator Company, Chicago, Ill
Werner & Pfleiderer Co., Saginaw, Mich.
Zaremba Company, Buffalo, N. Y.

Extracting Apparatus
Lummus Co., The Walter E., Boston, Mass.
Werner & Pfleiderer Co., Saginaw, Mich.

Extraction Cylinders
Lummus Co., The Walter E., Boston, Mass.

Faucets (Stoneware—Acid Proof)
German-American Stoneware Works,   N. V. C
Knight M. A., Akron (East Side), Ohio.
Robinson Clay Product Co., Akron, O.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.
U. S. Stoneware Co., The, Akron, O.

Fertilizer Industries Equipment
Schutte & Koerting Co., Philadelphia.

Fertilizers
German Kali Works, N. Y. C.

Filter Cloth (Metallic)
Sweetland Filter Press Co., Brooklyn, N. Y.

Filtering Media
General Filtration Co., Inc., Rochester, N. Y.
Kieselguhr Co. of America, Los Angeles. Cal.

Filter Paper
Laboratory Supply Co., Columbus, O.
Scientific Materials Co., Pittsburgh, Pa.
Whitall Tatum Co., Philadelphia, Pa.

Filter Paper (Whatman)
H. Reeve Angel & Co., N. Y. C.

Filter Presses (Laboratory & Industrial)
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco,Cal.
International Filtration Corp., N. Y. C.
Jacoby, Henry E., New York City.
Kelly Filter Press Co., Salt Lake City, Utah.
Lungwitz, E. E., New York City.
Shriver & Co., T., Harrison, N. J.
Sperry & Co., D. R., Batavia, Ill.
Sweetland Filter Press Co., Brooklyn, N. Y.
Swenson Evaporator Co., Chicago, Ill.
Werner & Pfleiderer Co., Saginaw, Mich.

Filters
General Filtration Co., Inc., Rochester, N. Y.

Filters—Water
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco,Cal.
Booth Apparatus Co., Syracuse, N. Y.
Eimer & Amend, N. Y. City.
German-American Stoneware Works, N. Y. C.
Heil Chemical Co., Henry, St. Louis, Mo.
Lenz & Naumann, Inc., N. Y. C.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Thomas Co., Arthur H., Philadelphia.

Filtros
General Filtration Co., Inc., Rochester, N. Y.

Fire Brick and Clay
Buffalo Dental Mfg. Co., Buffalo, N. Y.
Dixon Crucible Co. Joseph, Jersey City, N. J.
Kieselguhr Co. of America, Los Angeles, Calif.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.

Furnace Linings
Kieselguhr Co. of America, Los Angeles, Calif.

Furnaces—Annealing—(Hardening and Enameling)
Abbé Engineering Co., N. Y. C.
Buffalo Dental Mfg. Co., Buffalo, N. Y.
Electric Heating Apparatus Co., N. Y. C.
Hoskins Mfg. Co., Detroit, Mich.

Furnaces (Case Hardening, Rivet and Bolt)
Abbé Engineering Co., N. Y. C.

Furnaces—Electric Laboratory
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co.,San Francisco,Cal.
Brown Instrument Co., Philadelphia, Pa.
Eimer & Amend. New York City.
Electric Heating-Apparatus Co., N. Y. C.
Engelhard, Chas., N. Y. City.
Heil Chemical Co., Henry, St. Louis, Mo.
Hoskins Mfg. Co., Detroit, Mich.
Lenz & Naumann, Inc., N. Y. C.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburg, Pa.
Thomas Co., Arthur H., Philadelphia, Pa.

Furnaces—(Forge)
Abbé Engineering Co., N. Y. C.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co.,San Francisco. Cal.
Buffalo Dental Mfg. Co., Buffalo, N. Y. C.
Electric Heating Apparatus Co., N. Y. C.

Furnaces (Melting, Oil or Gas)
Abbé Engineering Co., N. Y. C.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco,Cal.
Buffalo Dental Mfg. Co., Buffalo, N. Y.
Electric Heating Apparatus Co., N. Y. C.
Heil Chemical Co., Henry, St. Louis, Mo.

Furnaces—Muffle
Abbé Engineering Co., N. Y. C.
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco,Cal.
Electric Heating Apparatus Co., N. Y. C
Heil Chemical Co., Henry, St. Louis, Mo.
Hoskins Mfg. Co., Detroit, Mich.
Lenz & Naumann, Inc., N. Y. C.
Scientific Materials Co., Pittsburgh, Pa.

Furnaces—Pyrites
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.

Furnaces—Sulphur
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.
Schutte & Koerting Co., Philadelphia.

Gas Analyzers
Bausch & Lomb Optical Co., Rochester, N. Y.

Gas Apparatus
Laboratory Supply Co., Columbus, O.
Scientific Materials Co., Pittsburgh, Pa.

Gauges
Bausch & Lomb Optical Co., Rochester, N. Y.
Brown Instrument Co., Philadelphia, Pa.

Generators (Electro-Oxy-Hydrogen)
Shriver & Co., T., Harrison, N. J.

Glass
Corning Glass Works, Corning, N. Y.
Eberbach & Son Co., Ann Arbor, Mich.
Fry Glass Co., H. C., Rochester, Pa.
Laboratory Supply Co., Columbus, O.
Whitall Tatum Co., Philadelphia, Pa.

Glass Blowing
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co.,San Francisco,Cal.
Carbondale Instrument Co., Carbondale, Pa.
Eimer & Amend, New York City.
Greiner Co., Emil, N. Y. City.
Heil Chemical Co., Henry. St. Louis, Mo.
International Glass Co., The, Millville, N. J.
Laboratory Supply Co., Columbus, O.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburg, Pa.

Glass Enameled Apparatus
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
Pfaudler Co., Rochester, N.Y., Detroit, & N.Y.C.

Graphite Products
Dixon Crucible Co., Joseph, Jersey City, N. J

Grinders
Abbé Engineering Co., N. Y. City.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco,Cal.
Raymond Bros. Impact Pulv. Co., Chicago. Ill.
Williams Patent Crusher & Pulv. Co., Chicago.

Hardness Testing Apparatus
Scientific Materials Co., Pittsburgh, Pa.

Heating Apparatus (Laboratory)
Bausch & Lomb Optical Co., Rochester, N. Y.
Electric Heating Apparatus Co., N. Y. C.
Hoskins Mfg. Co., Detroit, Mich.
Lenz & Naumann, Inc., N. Y. C.

Hot Plates (Laboratory Purposes)
Bausch & Lomb Optical Co., Rochester, N. Y
Electric Heating Apparatus Co., N. Y. C.
Hoskins Mfg. Co., Detroit, Mich.
International Glass Co., The, Millville, N. J.

Hydro Extractors
Tolhurst Machine Works, Troy, N. Y.

Hydrometers
Bausch & Lomb Optical Co., Rochester, N. Y.
Greiner Co., Emil, New York City.
Griebel Instrument Co., Carbondale, Pa.
Heil Chemical Co., Henry, St. Louis, Mo.
International Glass Co., Millville, N. J.
Laboratory Supply Co., Columbus, O.

Hydrogen Generators
International Oxygen Co., N. Y. C.

Hydrogen (Pure)
International Oxygen Co., N. Y. C.

Ice-Making Machinery
York Manufacturing Co., York, Pa.

Insulation
Kieselguhr Co. of America, Los Angeles, Calif.

Jars and Jugs (Acid Storage)
Bausch & Lomb Optical Co., Rochester, N. Y.
German-American Stoneware Works, N. Y. C.
Knight, M. A., Akron (East Side), Ohio.
Robinson Clay Product Co., Akron, O.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.

Kettles (Chemical Castings)
Bausch & Lomb Optical Co., Rochester, N. Y.
Buffalo Fdy & Mch. Co., Buffalo, N. Y.
Devine Co., J. P., Buffalo, N. Y.
Duriron Castings Co., N. Y. C.
Jacoby, Henry E., New York City.
Stevens-Aylsworth Co., N. Y. C.
Werner & Pfleiderer Co., Saginaw, Mich.

Kettles (Copper)
Bucyrus Copper Kettle Works, Bucyrus, O.

Kettles (Lined)
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
PfaudlerCo., Rochester,N.Y., Detroit, & N.Y.C.
Werner & Pfleiderer Co., Saginaw, Mich.

Kettles (Steam Jacketed)
Bucyrus Copper Kettle Works, Bucyrus, O.
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
Devine Co., J. P., Buffalo, N. Y.
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
Pfaudler Co., Rochester,N.Y., Detroit, & N.Y.C.
Sowers Mfg. Co., Buffalo, N. Y.
Sperry & Co., D. R., Batavia, Ill.
Werner & Pfleiderer Co., Saginaw, Mich.

Kettles (Stoneware)
German-American Stoneware Works, N. Y. C.
Knight, M. A., Akron (East Side), Ohio.
Robinson Clay Product Co., Akron. O.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.

Kieselguhr
Kieselguhr Co. of America, Los Angeles, Calif.

Laboratory Supplies
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco,Cal.
Corning Glass Works, Corning, N. Y.
Eimer & Amend, New York City.
Greiner Co., Emil, New York City.
Griebel Instrument Co., Carbondale, Pa.
Heil Chem. Co., Henry, St. Louis, Mo.
International Glass Co., The, Millville, N. J.
Laboratory Supply Co., The, Columbus, Ohio.
Lenz & Naumann, Inc., N. Y. C.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburg, Pa.
Thomas Co., Arthur H., Philadelphia, Pa.

Lace Leather
Schieren Co., Chas. A., N. Y. C.

Lacquer
General Bakelite Co., New York City.

Lactic Acid
Woolner & Co., Peoria, Ill.

Lamps—Arc and Incandescent
General Electric Co., Schenectady, N. Y.

Leaching Cells
Zaremba Co., Buffalo, N. Y.

Lead Burning
M. McNamara, Everett, Mass.

Lead Lined Pipe and Accessories
Schutte & Koerting Co., Philadelphia.

Leather Belting (Regular Oak Tanned and Duxbak Waterproof; Round; Link)
Schieren Co., Chas. A., N. Y. C.

Lime
Mitchell Lime Co., Mitchell, Ind.

Lubricants
Dearborn Chemical Co., Chicago, Ill.
Dixon Crucible Co., Joseph, Jersey City, N. J.

Machinery—Electrical
General Electric Co., Schenectady, N. Y.

Machinery—Mining and Metallurgical
Abbé Engineering Co., New York City.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co.,San Francisco,Cal.
General Filtration Co., Inc., Rochester, N. Y
Mine & Smelter Supply Co., Denver, Salt Lake City, El Paso, N. Y. City.
Stokes Machine Co., F. J., Philadelphia.
Werner & Pfleiderer Co., Saginaw, Mich.

Machinery—Mixing and Kneading
Abbé Engineering Co., New York City.
PfaudlerCo.,Rochester,N.Y., Detroit, & N.Y.C.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.
Werner & Pfleiderer Co., Saginaw, Mich.

Machinery—Pulverizing
Abbé Engineering Co., New York City.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co.,San Francisco,Cal.
General Filtration Co., Inc., Rochester, N. Y.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.
Raymond Bros. Impact Pulv. Co., Chicago. Ill
Werner & Pfleiderer Co., Saginaw, Mich.

Machinery (Special)
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
Devine Co., J. P., Buffalo. N. Y.
Duriron Castings Co., N. Y. C.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.
Werner & Pfleiderer Co., Saginaw, Mich.

Metallographic Apparatus
Scientific Materials Co., Pittsburgh, Pa.

Metallurgical Laboratory Equipment
Scientific Materials Co., Pittsburgh, Pa.

Meters—Flow Air, Gas, Water
General Electric Co., Schenectady, N. Y.

Microscopes
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco Cal.
Eimer & Amend, New York City.
Heil Chem. Co., Henry, St. Louis, Mo.
Laboratory Supply Co., Columbus, O.
Lenz & Naumann, Inc., N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburg, Pa.
Thomas Co., Arthur H., Philadelphia, Pa.

Microtomes
Bausch & Lomb Optical Co., Rochester. N. Y.
Heil Chemical Co., Henry, St. Louis, Mo.
Lenz & Naumann, Inc., N. Y. C.

Mills—Ball, Pebble and Tube
Abbé Engineering Co., New York City.

Mixers
PfaudlerCo.,Rochester,N.Y.,Detroit, & N.Y.C.
Sowers Mfg. Co., Buffalo, N. Y.
Werner & Pfleiderer Co., Saginaw, Mich.

Mixing Tanks
Devine Co., J. P., Buffalo, N. Y.
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C
Lummus Co., The Walter E., Boston, Mass.
PfaudlerCo.,Rochester,N.Y., Detroit, & N.Y.C.
Werner & Pfleiderer Co., Saginaw, Mich.

Montejus—Automatic
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
German-American Stoneware Works, N. Y. C.
Schutte & Koerting Co., Philadelphia.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.

Nickel-Chromium Gauze
Howard & Morse, N. Y. C.

Nitrating Pots
Devine Co., J. P., Buffalo, N. Y.
PfaudlerCo..Rochester,N.Y.,Detroit, & N.Y.C.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.

Nitre Pot Acid Pipes
Duriron Castings Co., N. Y. C. (Special Alloy)
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.
Thermal Syndicate, Ltd., N. Y. C. (Silica Ware)

Nitre Cake
du Pont de Nemours & Co., E. I., Wilmington. Del.

Nozzles (Acid Proof)
Duriron Castings Co., N. Y. C.

Oil Testing Instruments
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco,Cal.
Lenz & Naumann, Inc., N. Y. C.
Scientific Materials Co., Pittsburgh, Pa.

Optical Instruments
Bausch & Lomb Optical Co., Rochester, N. Y.

Optical Pyrometer
Scientific Materials Co., Pittsburgh, Pa.

Oxygen Generators
International Oxygen Co., N. Y. City

Oxygen (Pure)
International Oxygen Co., N. Y. City
Levin, Isaac H., Newark, N. J.

Oxygen Testing Apparatus
International Oxygen Company, N. Y City

Packings (Condensing Tower)
German-American Stoneware Wks., N. Y. C.
Knight, M. A. Akron (East Side). Ohio.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.
U. S. Stoneware Co., The, Akron, O.

Paints
Toch Brothers, N. Y. City.

Patent Attorneys
Brickenstein, John H., Washington, D. C.
Foster, A. B., Washington, D. C.
Parker, C. L., Washington D. C.
Sieck, Herbert Chicago. Ill.
Townsend, Clinton P., Washington, D. C.

Penetrometers
Howard & Morse, N. Y. City.

Percolators
Devine Co., J. P., Buffalo, N. Y.
Elyria Enameled Prod.Co., Elyria, O., & N.Y.C
Kieselguhr Co. of America. Los Angeles Cal.
Pfaudler Co., Rochester N.Y., Detroit, & N.Y.C

Photo-Micrographic Apparatus
Bausch & Lomb Optical Co., Rochester, N. Y

Pipe and Accessories
Bausch & Lomb Optical Co., Rochester. N. Y
Corning Glass Works, Corning, N. Y.

Pipe and Accessories (Acid Proof)
Duriron Castings Co., N. Y. C.
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C
German-American Stoneware Works, N. Y. C.
Knight, M. A., Akron (East Side) Ohio.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.
Robinson Clay Product Co., Akron, O.
Schutte & Koerting Co., Philadelphia.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.
Thermal Syndicate, Ltd., N. Y. C. (Fused Silica)
U. S. Stoneware Co., The, Akron, O.

Pipe and Fittings—Lead or Tin Lined
Chadwick-Boston Lead Co., Boston, Mass

Platinum (Sheet, Wire and Foil)
American Platinum Works, Newark, N. J.
Baker & Co., Inc., Newark, N. J.
Bausch & Lomb Optical Co. Rochester. N Y
Bishop & Co., J., Platinum Works. Malvern. Pa.

Platinum Ware (Laboratory)
American Platinum Works, Newark, N. J
Baker & Co., Inc., Newark, N. J.
Bausch & Lomb Optical Co., Rochester, N. Y
Bishop & Co., J., Platinum Works, Malvern Pa.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco,Cal.
Eimer & Amend, New York City.
Engelhard, Chas., New York City.
Heil Chem. Co., Henry, St. Louis, Mo.
Lenz & Naumann. Inc., N. Y. C.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburg. Pa.
Thomas Co., Arthur H., Philadelphia.

Porcelain Ware—Laboratory
Bausch & Lomb Optical Co.. Rochester. N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co.,San Francisco,Cal.
Eimer & Amend, New York City.
Heil Chem. Co., Henry, St. Louis, Mo
Laboratory Supply Co., Columbus, O.
Lenz & Naumann. Inc.. N. Y. C.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburgh, Pa.
Thomas Co., Arthur H., Philadelphia.

Potash Salts
German Kali Works. N. Y. C.

Pulverizers—Laboratory
Abbé Engineering Co., New York City.
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles. Calif.
Braun-Knecht-Heimann Co.,SanFrancisco, Cal.
Eimer & Amend, New York City.
Heil Chemical Co., Henry, St. Louis, Mo.
Palo Company, N. Y. C.
Williams Patent Crusher & Pulv. Co., Chicago.

Pumps—Acid
Bausch & Lomb Optical Co., Rochester, N. Y.
Bethlehem Fdy. & Mach. Co., So. Beth'm, Pa.
Devine Co., J. P., Buffalo, N. Y.
Duriron Castings Co., New York City.
German-American Stoneware Works, N. Y. C.
Schutte & Koerting Co., Philadelphia.

Pumps (Laboratory Vacuum)
Abbé Engineering Co., New York City.
Bausch & Lomb Optical Co., Rochester, N. Y.
Buffalo Dental Mfg. Co., Buffalo, N. Y.
Crowell Mfg. Co., Brooklyn, N. Y.
Devine Co., J. P., Buffalo, N. Y.
Hubbards' Sons, Norman, Brooklyn, N. Y.

Pumps (Vacuum)
Abbé Engineering Co., N. Y. C.
Bethlehem Fdy. & Mach. Co., So. Beth'm, Pa.
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
Crowell Mfg. Co., Brooklyn, N. Y.
Devine Co., J. P., Buffalo, N. Y.
Duriron Castings Co., N. Y. C.
Jacoby, Henry E., New York City.

Pump Parts Metal (Acid Proof)
Duriron Castings Co., N. Y. C.
Schutte & Koerting Co., Philadelphia.

Purified Cotton for Nitration, Etc.
Massasoit Mfg. Co., Fall River, Mass.

Pyrometers
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco, Cal.
Brown Instrument Co., Philadelphia, Pa.
Eimer & Amend, New York City.
Engelhard, Chas., New York City.
Greiner Co., Emil, New York City.
Heil Chem. Co., Henry E., St. Louis, Mo.
Hoskins Mfg. Co., Detroit, Mich.
Lenz & Naumann, Inc., N. Y. C.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburg, Pa.
Thomas Co., Arthur H., Philadelphia.
Thwing Instrument Co., Philadelphia, Pa.

Pyrometers (Optical)
Brown Instrument Co., Philadelphia, Pa.

Quartz
Booth Apparatus Co., Syracuse, N. Y.

Quartz Glass
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco, Cal.
Brown Instrument Co., Philadelphia, Pa.
Eimer & Amend, New York City.
Engelhard, Chas, New York City.
Greiner Co., Emil, New York City.
Hanovia Chemical & Mfg. Co., Newark. N. J
Heil Chem. Co., Henry, St. Louis, Mo.
Laboratory Supply Co., Columbus, O.
Lenz & Naumann, Inc., N. Y. C.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburg, Pa.
Sidio Co. of America, N. Y. City.
Thermal Syndicate, Ltd., New York City.
Thomas Co., Arthur H., Philadelphia.

Radium
Eimer & Amend, New York City.
Heil Chemical Co., Henry, St. Louis, Mo.
Lenz & Naumann, Inc., N. Y. C.
Palo Company, N. Y. C.

Reagents—Chemical
Baker & Adamson Chem. Co., Easton, Pa.
Baker Chem. Co., J. T., Phillipsburg, N. J.
Bausch & Lomb Optical Co., Rochester, N. Y.
Heil Chemical Co., Henry, St. Louis. Mo.
Merck & Co., N. Y. C.
Squibb & Sons, E. R., N. Y. C.

Recording Instruments
Brown Instrument Co., Philadelphia, Pa.

Refineries—(Oil—Sugar Installation)
Lummus Co., The Walter E., Boston, Mass.

Refractories
Kieselguhr Co. of America, Los Angeles, Calif.
Norton Co., Worcester, Mass.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.

Refrigerating Machinery and Apparatus
York Manufacturing Co., York, Pa.

Regulators (Automatic Humidity)
Carrier Engineering Corp., N. Y. C.

Resistance Wire
American Platinum Works, Newark, N. J.
Baker & Co., Inc., Newark, N. J.
Bishop & Co., J., Malvern, Pa.

Retorts
Dixon Crucible Co., Joseph, Jersey City, N. J.
Heil Chemical Co., Henry, St. Louis, Mo.
Laboratory Supply Co., Columbus, O.
Lummus Co., The Walter E., Boston, Mass.
Thermal Syndicate, Ltd., N. Y. C.

Retorts (Cast Iron)
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.
Devine Co., J. P., Buffalo, N. Y.

Retorts—Acid (Cast Iron)
Bethlehem Foundry & Mach. Co., So. Beth., Pa.
Buffalo Fdy. & Mch. Co., Buffalo, N. Y.

Rotary Compressors or Blowers
Abbé Engineering Co., N. Y. C.
Crowell Mfg. Co., Brooklyn, N. Y.

Rotary Lead Fans
Schutte & Koerting Co., Philadelphia.

Rubber Tubing, Laboratory
Scientific Materials Co., Pittsburgh, Pa.

Scientific Apparatus
Eberbach & Son Co., Ann Arbor, Mich.

Screens (Grinding & Pulverizing)
Raymond Bros. Impact Pulv Co., Chicago. Ill.

Separators—Air
Raymond Bros. Impact. Pulv Co., Chicago. Ill.

Separators—Centrifugal
International Equipment Co., Boston, Mass.
Schaum & Uhlinger, Inc., Philadelphia.
Tolhurst Machine Works, Troy, N. Y.

Shredders
Williams Patent Crusher & Pulv. Co., Chicago.

Sieve Agitators
Howard & Morse, N. Y. City.

Sieves (Testing)
Howard & Morse, N. Y. City.

Silica—Fused
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco, Cal.
Eimer & Amend, New York City.
Engelhard, Chas., New York City.
Greiner & Co., Emil, N. Y. C.
Heil Chem. Co., Henry, St. Louis, Mo.
Laboratory Supply Co., Columbus, O.
Lenz & Naumann Inc., N. Y. C.
Palo Company, N. Y. C.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co.. Pittsburg, Pa.
Sidio Co. of America, N. Y. City.
Thermal Syndicate, Ltd., New York City.
Thomas Co., Arthur H., Philadelphia.

Solvent Recovery Apparatus (Vacuum)
Devine Co., J. P., Buffalo, N. Y.
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
Lummus Co., The Walter E., Boston, Mass.
Pfaudler Co., Rochester, N.Y., Detroit, & N.Y.C.
Werner & Pfleiderer Co., Saginaw, Mich.

Spelter
New Jersey Zinc Co., The, N. Y. C.

Spray Nozzles
Schutte & Koerting Co., Philadelphia.

Steam Engineering Specialties
Sarco Co., Inc., New York City.

Steam Traps and Meters
Sarco Co., Inc., New York City.

Stirring Apparatus
Booth Apparatus Co., Syracuse. N. Y.
Devine Co., J. P., Buffalo, N. Y.
Duriron Castings Co., N. Y. C.
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
Pfaudler Co., Rochester, N.Y., Detroit, & N.Y.C.
Werner & Pfleiderer Co., Saginaw, Mich.

Stoneware—Chemical (Laboratory)
Bausch & Lomb Optical Co., Rochester, N. Y.
Braun Corporation, Los Angeles, Calif.
Braun-Knecht-Heimann Co., San Francisco, Cal.
Eimer & Amend, New York City.
German-American Stoneware Works, N. Y. C.
Heil Chem. Co., Henry, St. Louis, Mo.
Knight, Maurice A., Akron ,Ohio.
Lenz & Naumann, Inc., N. Y. C.
Palo Company, N. Y. C.
Robinson Clay Products Co., Akron, O.
Sargent & Co., E. H., Chicago, Ill.
Scientific Materials Co., Pittsburg, Pa.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.
Thomas Co., Arthur H., Philadelphia.
U. S. Stoneware Co., The, Akron. O.

Stoneware (Acid Proof—Industrial Purposes)
German-American Stoneware Works, N. Y. C.
Knight, M. A., Akron (East Side), Ohio.
Robinson Clay Product Co., Akron, O.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.
U. S. Stoneware Co., The, Akron. O.

Succinic Acid
Woolner & Co., Peoria, Ill.

Sulphur, Crude
Union Sulphur Co., New York City.

Syphons—Steam Jet
Duriron Castings Co., N. Y. C. (Acid Proof)
Schutte & Koerting Co., Philadelphia, Pa.
Elyria Enameled Products Co., Elyria, O., and N. Y. C.

Tachometers
Brown Instrument Co., Philadelphia, Pa.

Tanks—(For Acid or Basic Reaction)
Devine Co., J. P., Buffalo, N. Y.
Elyria Enameled Products Co., Elyria, O., and N. Y. C.
German-American Stoneware Works, N. Y. C
Knight, Maurice A., Akron, Ohio.
Lummus, Walter E., Boston, Mass.
Pfaudler Co., Rochester. N.Y., Detroit, & N.Y.C.
Robinson Clay Product Co., Akron, O.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.
U. S. Stoneware Co., The, Akron. O.

Tanks—(Measuring & Self-decanting)
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
Pfaudler Co., The, Rochester, N. Y.
Lummus, Walter E., Boston, Mass.

Tanks—Pressure
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
German-American Stoneware Works. N Y C.
Pfaudler Co., Rochester, N.Y., Detroit, & N.Y.C.
Robinson Clay Product Co., Akron, O.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.
U. S. Stoneware Co., The, Akron. O.

Tanks (Steel)
Stevens Brothers, N. Y. C.

Tanks (Storage)
Devine Co., J. P., Buffalo, N. Y.
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
German-American Stoneware Works, N. Y. C.
Knight, M. A., Akron (East Side), Ohio.
Pfaudler Co., Rochester, N.Y., Detroit, & N.Y.C.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.
U. S. Stoneware Co., The, Akron. O.

Temperature Control Instruments
Brown Instrument Co., Philadelphia, Pa.
Thwing Instrument Co.. Philadelphia, Pa.

Thermometers
Bausch & Lomb Optical Co., Rochester, N. Y.
Brown Instrument Co., Philadelphia, Pa.
Engelhard, Charles, New York City.
Greiner Co., Emil, New York City.
Griebel Instrument Co., Carbondale, Pa.
Heil Chemical Co., Henry, St. Louis, Mo.
International Glass Co., The, Millville, N. J.
Laboratory Supply Co., Columbus, O.
Lenz & Naumann, Inc., N. Y. C.
Scientific Materials Co., Pittsburgh, Pa.

Thermostats
Bausch & Lomb Optical Co., Rochester, N. Y.
Brown Instrument Co., Philadelphia, Pa.
Scientific Materials Co., Pittsburgh, Pa.
Thwing Instrument Co.. Philadelphia., Pa.

Thickeners, or Dewaterers
Dorr Co., The, Denver, Col., and N. Y. C.
International Filtration Corp., N. Y. C.

Tile (Tower)
German-American Stoneware Works, N. Y. C
Knight, M. A., Akron (East Side), Ohio.
Steiger Terra Cotta and Pottery Works, San Francisco, Cal.
U. S. Stoneware Co., The, Akron. O.

Triangles
Bausch & Lomb Optical Co., Rochester, N. Y.
Laboratory Supply Co., Columbus, O.
Thermal Syndicate, Ltd., New York City.

Vacuum Drying Apparatus
Bethlehem Fd'y & Mach. Co., So. Beth., Pa.
Buffalo Fdy & Mch. Co., Buffalo, N. Y.
Devine Co., J. P., Buffalo. N. Y.
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
Jacoby Henry E., New York City.
Pfaudler Co., Rochester, N.Y., Detroit, & N.Y.C.
Stokes Machine Co., F. J., Philadelphia.
Werner & Pfleiderer Co., Saginaw, Mich.

Vacuum Pans
Badger & Sons Co., E. B., Boston, Mass.
Devine Co., J. P., Buffalo. N. Y.
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
German-American Stoneware Works, N. Y. C.
Jacoby, Henry E., New York City.
Lummus Co., The Walter E., Boston, Mass.
Pfaudler Co., Rochester, N.Y., Detroit, & N.Y.C.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.
Roos' Sons, August, N. Y. C.
Sanborn Evaporator Co., New York Cit
Sowers Mfg. Co., Buffalo, N. Y.
Sperry & Co., D. R., Batavia, Ill.
Stokes Machine Co., F. J., Philadelphia.
Swenson Evaporator Co., Chicago, Ill.
Werner & Pfleiderer Co., Saginaw, Mich.
Zaremba Co., Buffalo, N. Y.

Vacuum Pumps
Abbé Engineering Co., N. Y. City.
Bausch & Lomb Optical Co., Rochester, N. Y.
Crowell Engineering Co., Brooklyn, N. Y.
Devine Co., J. P., Buffalo, N. Y.
Hubbard's Sons, Norman, Brooklyn, N. Y.

Vacuum Stills
Devine Co., J. P., Buffalo, N. Y.
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
Lummus Co., The Walter E., Boston, Mass.
Pfaudler Co., Rochester, N.Y., Detroit, & N.Y.C.
Pratt Eng. & Mach. Co., Atlanta, Ga. & N.Y.C.
Werner & Pfleiderer Co., Saginaw, Mich.

Valves, Ammonia
York Manufacturing Co., York, Pa.

Valves and Fittings—Lead
Schutte & Koerting Co., Philadelphia, Pa.

Varnish—Coil Impregnation
General Bakelite Co., New York City.

Vats (Rectangular)
Pfaudler Co., Rochester N.Y., Detroit, & N.Y.C.

Voltmeters
Brown Instrument Co., Philadelphia, Pa.

Vicat Needles
Howard & Morse, N. Y. City.

Viscosity Floats
Howard & Morse, N. Y. City.

Water Bath (Laboratory)
Bausch & Lomb Optical Co., Rochester, N. Y.
Electric Heating Apparatus Co., N. Y. C.
Laboratory Supply Co., Columbus, O.

Water Softening Apparatus
Booth Apparatus Co., Syracuse, N. Y.
Hodges Water Still Co., Woodbury, N. J.

Weighing Machinery—(Automatic)
Werner & Pfleiderer Co., Saginaw, Mich.

Welding and Cutting Apparatus
General Electric Co., Schenectady, N. Y.
International Oxygen Co., N. Y. C.

Wire Cloth
Howard & Morse, N. Y. City.

Wire, Resistance
Driver, Harris Wire Co., Harrison, N. J.

Wood Distillation Equipment
Badger & Sons. Co., E. B., Boston, Mass.
Devine Co., J. P., Buffalo, N. Y.
Elyria Enameled Prod. Co., Elyria, O., & N.Y.C.
Lummus Co., The Walter E., Boston, Mass.
Pfaudler Co., Rochester N.Y., Detroit, & N.Y.C.
Roos' Son, August, N. Y. C.
Swenson Evaporator Co., Chicago, Ill.
Zaremba Co., Buffalo, N. Y.