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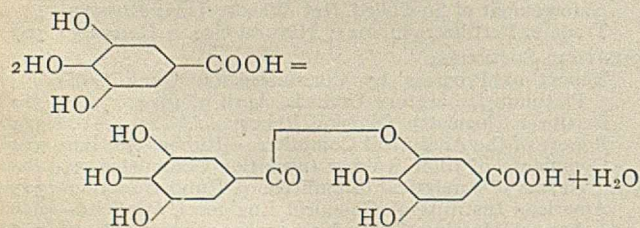
EDITORIALS

RECENT SYNTHETIC STUDIES IN THE TANNIN GROUP¹

There exist in nature a considerable number of substances whose aqueous extracts have the property of turning hide into leather. Substances which show this property have also certain other general characteristics. Tanning substances are amorphous and have an astringent taste. They usually precipitate gelatin from its solutions and form compounds with metals, those with iron, for example, being frequently colored and suitable for use as inks. Hydrolytic agents split them into a variety of products. One of these is almost always a sugar, usually *d*-glucose, while the other component is some hydroxy acid of the aromatic series. Caffetannic acid, for example, which is found in coffee and Paraguay tea, yields a sugar and caffeic acid. The tanning material of oak bark yields ellagic acid and quercite. Still others yield phloroglucinol instead of sugar.

The most thoroughly studied of these substances is ordinary *tannin*, which occurs in the nut-galls of the oak and is also found in sumach, tea and other plants. It is a colorless, amorphous substance, which is readily soluble in water and only slightly soluble in alcohol and ether. It can be prepared in a state of comparative purity, and its structure has interested organic chemists for a long time.

The most important of the earlier investigations was carried on by Strecker in 1852 [*Ann. der Chemie*, 81 (1852), 248; 90 (1854), 328]. He hydrolyzed tannin and found three molecules of gallic acid associated with one of *d*-glucose. Many chemists who studied the problem later came to the conclusion that sugar was not an essential constituent, at least in all kinds of tannin, and the observation that gallic acid, by means of phosphorus oxychloride and other dehydrating agents, could be changed to digallic acid,



led Schiff, in 1871 [*Ber.*, 4 (1871), 232, 967; 12 (1879), 33; and *Ann.*, 170 (1873), 43], to accept this synthetic digallic acid as, at least, the principal component of tannin. Such a conclusion left the optical activity of tannin unexplained, since digallic acid contains no asymmetric carbon atom. It could not be certain, however, that optically active impurities might not be present, inasmuch as tannin is an amorphous substance and the usual criteria of purity and homogeneity in organic substances are lacking.

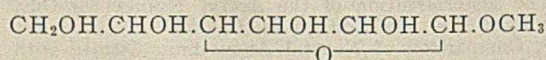
Walden, in 1897 [*Ber.*, 30 (1897), 3151; 31 (1898), 3167], made an extensive comparison of the synthetic

¹ Address before the Eastern New York Section of the American Chemical Society, Schenectady, March 27, 1914.

digallic acid with tannin, and found that their physical properties were not identical. The molecular weight of tannin is far too great for digallic acid, and neither the electrical conductivity, the absorption of light, nor the behavior with arsenic acid were found sufficiently parallel for Walden to believe in the identity of the substances concerned. Quite recently, Nierenstein made the suggestion that tannin might be a mixture of digallic acid with its optically active reduction product, leuco-tannin. Such a mixture, however, should be more acidic than tannin and have a smaller apparent molecular weight. Still more recently Feist [*Ber.*, 45 (1912), 1493] has suggested a more complex composition, but his conclusions have now been superseded by the work of Emil Fischer [*Gen. résumé, Ber.*, 46 (1913), 3253; see also 41 (1908), 2875; 45 (1912), 915, 2709, 3773; 46 (1913), 1116], which it is the principal object of this paper to describe.

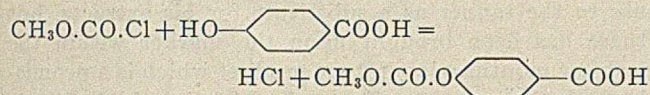
Emil Fischer's attention was first devoted to the purification of tannin with the idea of obtaining as homogeneous a material as possible. For this purpose, the purest product from Chinese nut-galls was shaken from a dilute alkaline solution by ethyl acetate. It is obvious that a substance so purified could contain no free carboxyl group, and we can assume this to be the fact concerning the material investigated by Fischer. When purified in this way, the substance is amorphous, but it is doubtless as pure a tannin as has hitherto been prepared. It is optically active, the specific rotatory power being about 70°.

The next step was the hydrolysis of tannin. This was carried out in acid solution, since alkali would act upon any sugar formed. The method was checked by blank tests made with mixtures of gallic acid and sugar, and after making any necessary corrections of this kind, it was found that hydrolysis had decomposed the tannin into a mixture of one molecule of *d*-glucose and ten of gallic acid. No other hydroxy acid was found. Since there are five hydroxyl groups in *d*-glucose, a simple interpretation of the results would suggest that we had to do with an ester of glucose in which each of the five hydroxyl groups was esterified with a molecule of digallic acid. Such a substance would not be a glucoside in the ordinary acceptance of the term. Emil Fischer is of the opinion that the latter name should be reserved for products analogous in their structure to methyl glucoside,



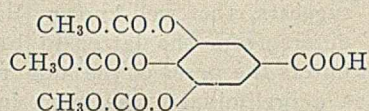
The best proof of the structure suggested for tannin would be its synthesis, but in view of the difficulty of preparing synthetic digallic acid, it seemed best to begin with the simpler problem of preparing glucose pentagallate. The method which first suggests itself for the preparation of such a compound is the treatment of the sugar by the chloride of the acid; but chlorides of substances like gallic acid are difficult to

prepare. The usual easy reaction of the chlorides of phosphorus upon the free acids is complicated in this case by the fact that the hydroxyl groups also present are attacked by the reagent. Emil Fischer was able to avoid this difficulty by a method which he had previously used in less complicated cases. This consisted of treating a hydroxy acid with methyl chlorcarbonate. This forms a carbomethoxy derivative,

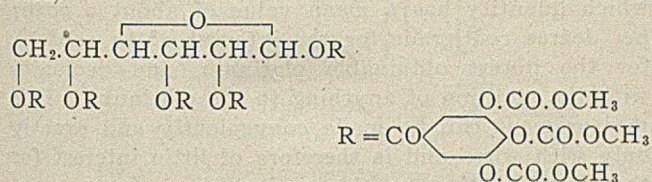


which is stable enough to tolerate reactions upon the carboxyl group, but when these reactions have been carried out, gentle hydrolytic agents exchange the carbomethoxy group for hydroxyl again.

It is obvious that under these circumstances gallic acid would yield a tricarbomethoxy derivative,

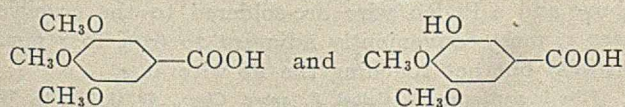


which, when treated with phosphorus chloride, would yield the corresponding acid chloride. This chloride was finally treated with *d*-glucose in dry chloroform solution, quinoline being introduced for the purpose of taking up the hydrochloric acid formed. The product was a penta-tricarbomethoxy-gallate of glucose,



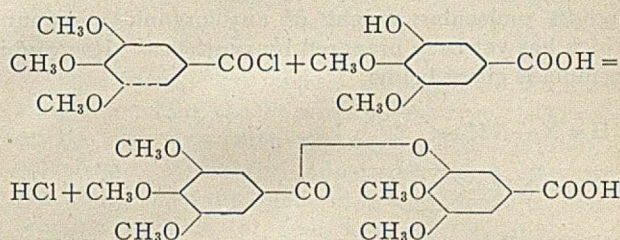
It was carefully saponified by means of a small excess of alkali dissolved in aqueous acetone. Under these circumstances the carbomethoxy groups were split off at ordinary temperature and the pentagallate of glucose was isolated. The similarity of this substance to ordinary tannin was striking. The only difference was in the rotatory power and the quantity of gallic acid formed by hydrolysis. Like tannin, the substance is amorphous, and it cannot be stated with certainty that it is perfectly homogeneous. Emil Fischer believes that there are probably at least two isomers present which stand to each other in the same relation as α - and β -glucose. This condition of things is certain in the case of glucose pentabenzoate.

The next step was the preparation of methylotannin, which proved easier to carry out than the synthesis of tannin itself. Herzig [*Ber.*, 38 (1905), 989; *Monatshfte*, 30 (1909), 543] had already prepared this substance by the action of diazomethane upon tannin, and had obtained, by hydrolysis, trimethylgallic acid and unsymmetrical *m,p*-dimethylgallic acid.



These facts suggest that tannin might be an ester

of glucose with five molecules of *m*-digallic acid. The synthesis was carried on much as before. Trimethylgalloyl chloride was first combined with *m,p*-dimethylgallic acid,



and this, in its turn, changed to its chloride and combined with *d*-glucose. The product was strikingly similar to that of Herzig, but there remains, of course, the same doubt as to its perfect homogeneity as in the other case.

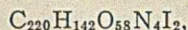
The success of these experiments seemed to warrant an attempt to prepare tannin itself: *m*-digallic acid, which had previously been synthesized, was changed to the carbomethoxy compound as in the simpler case of ordinary gallic acid, but the product was found to be amorphous, and the same is true of the chloride which is prepared from it. Finally, the combination with sugar took place only with difficulty and the synthesis cannot be regarded as successful. The essential nature of the tannins seems, however, to have been definitely settled by the investigation and we can be confident that the structure of tannin is typified by the penta-gallate of glucose. It is still possible, of course, that tannins from different sources have not exactly the same composition, and that any given sample of tannin is not homogeneous. It is also possible that the *d*-glucose is not readily formed in the molecule, but that there exists instead some polysaccharide which gives *d*-glucose upon hydrolysis.

The investigation was continued by combining gallic and other hydroxy acids with other sugars and polyatomic alcohols, and a variety of substances were thus prepared which showed the chief tannin characteristics. Doubtless the study of compounds of this class will be of considerable value to plant physiology since it is very likely that the plant builds up such substances as a means of neutralizing acids by esterification. Some practical results may also be expected, since small quantities of tannin-like substances have a marked influence upon the taste of such articles of food as tea, coffee and sweet fruits. It is, therefore, possible that synthetic materials of this type may find use as flavoring materials.

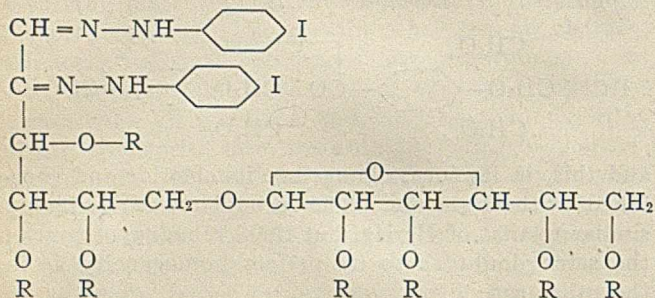
An interesting result of the investigation is to be found in the fact that syntheses of this kind lead naturally to products of extremely high molecular weight. In this property they surpass even the highest polypeptides. Emil Fischer expects to carry investigations of this sort further, believing that it is the duty of the Organic Chemist to build up as large molecules as possible, while the modern physicist is constantly dissecting the atom into smaller and smaller units.

By combining tribenzoylgalloyl chloride with the

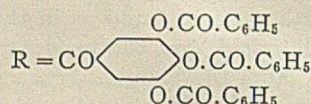
p-iodophenylosazone of maltose, Emil Fischer was able to prepare a substance of the formula



and molecular weight 4021. This substance has the highest molecular weight of any organic compound which has yet been prepared by synthesis. Its graphic formula is given below,



and the only explanation necessary is that the radical R signifies tribenzoylalloyl,



It is interesting to note that this substance follows the laws of Raoult very well. Determinations of the molecular weight by the freezing-point method, in bromoform, yielded numerical results which are surprisingly good when the magnitude of the molecule is taken into consideration.

This investigation has not led to the production of any substances which are sufficiently cheap to be of use to the tanner as a substitute for his extracts, but there has been brought upon the market within the last few months a laboratory product which is a promising substitute for tannin, and which is said by those in the trade to give good results and shorten the time required for tannage. This is the so-called "Neradol" of Stiasny [*Collegium*, 1913, p. 142]. It is prepared by treating phenol with sulfuric acid and adding formaldehyde in the proportion of one molecule of formaldehyde to two of phenol. This method of preparation suggests the well-known Bakelite, but the product differs from the latter in being soluble in water. It enters the pores of the hide readily, and when used as a preliminary treatment increases the velocity of tannage by other materials. It also produces leathers of good quality and light color.

F. J. MOORE

ORIGINAL PAPERS

A THERMOELECTRIC METHOD FOR THE DETERMINATION OF THE PURITY OF PLATINUM WARE¹

By GEORGE K. BURGESS AND P. D. SALE

At the request of Dr. W. F. Hillebrand,² Chairman of the Committee on "Quality of Platinum Utensils," of the American Chemical Society, experiments on the loss in weight due to continued and repeated heating of platinum crucibles of varying degrees of purity have been undertaken, in continuation of similar experiments carried out under the immediate supervision of members of the above-mentioned committee.

From some of these earlier experiments, and from the work of other experimenters on the evaporation of metals of the platinum group, it appears to have been hoped to be able to classify platinum ware as to purity in terms of its evaporation at a definite temperature, say 1200° C. This seemed plausible in view of the fact that the usually predominant impurity, iridium, is very much more volatile than platinum. Even if this method, however, would give an indication of the platinum purity, which appears doubtful in the light of some of our more recent experiments, it is at best a somewhat tedious and delicate operation to carry out.

These experiments showed the desirability of having an accurate and rapid method for determining platinum purity and one that could be applied to crucibles without defacing them.

The most exact method for the determination of purity of platinum appears to be by measurement of its temperature coefficient of electrical resistance,

¹ Presented at the 49th Meeting of the A. C. S., Cincinnati, April 6-10, 1914.

² See report of Platinum Committee in this issue.

which quantity has a mean value of about 0.00391 per degree centigrade for the interval 0° to 100° C. for the purest obtainable platinum, and decreases with the addition of anything to the platinum. This measurement can be made conveniently and exactly only with wires and is therefore of little interest for the determination of the purity of platinum ware such as crucibles.

The thermoelectromotive force of platinum against many of its alloys has also been determined with considerable exactness.¹

This property may evidently be made use of, therefore, in devising a method for the determination of platinum purity and one that possesses, furthermore, the advantages of accuracy, speed, convenience, and preservation intact of the objects tested.

The method as developed for use with crucibles is shown in Fig. 1. To the rim of the crucible *C* are arc-soldered, at *e* and *f*, two pure platinum wires of small diameter (0.1 or 0.2 mm.); these wires are connected to an ordinary pyrometer-galvanometer or millivoltmeter, *G*; the junction *e* is heated by a small oxy-gas or other blast flame from *d* and the junction *f* is kept cool by an air blast, *c*; a sheet of asbestos, *A*, cut as shown, serves to prevent radiation from the heated portion of the crucible reaching the cold junction *f*.

Temperatures are measured by means of a 90 Pt-10 Rh, Pt thermocouple using the cold junction as above and a Pt-Rh wire arc-soldered to the crucible near *c*, most conveniently adjacent to (0.5 to 1 mm. distant) but not touching the Pt wire at *e*.

¹ See in particular: W. Geibel, *Z. anorg. Chem.*, 69 (1910), 38; 70 (1911), 240; Burgess and LeChatelier, "The Measurement of High Temperatures," 3rd Ed., 1912, p. 171.

The wires are attached at *e* and *f* by the well known arc-soldering method, which consists in making the

crucible and at the same instant touching the wire to the crucible at this point. The end of the wire is thus fused to the crucible. With a little practice this operation may be made so that there is hardly any noticeable effect on the appearance of the crucible after the wire is removed.

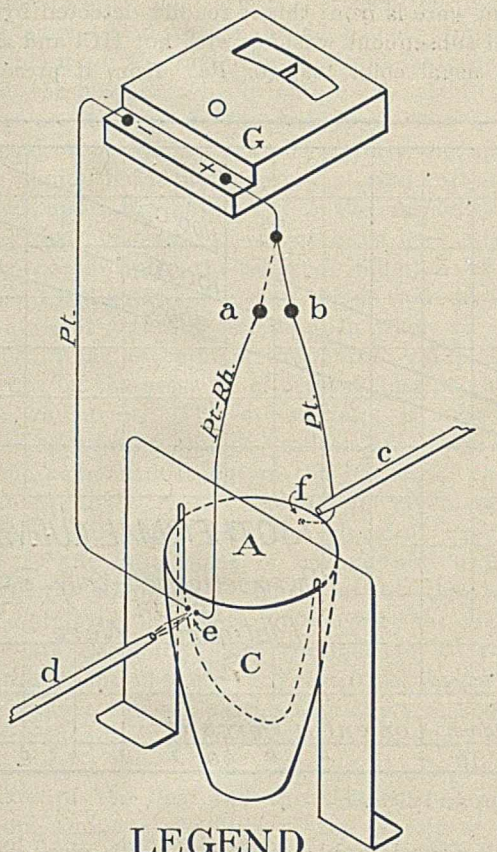
Quite satisfactory results may also be obtained by simply touching the Pt wires to the crucible at *e* and *f*, or by clamping with platinum clips, without any soldering. With the apparatus once set up, a test may then be made in a few seconds and the crucible remains, of course, absolutely intact. The homogeneity of the crucible may also be determined by this method.

By means of a commutator at *a b*, measurements may be made alternately of the temperature at *e* and of the E. M. F. developed across the crucible when *e* is at this temperature. The commutator may also be designed so as to reverse *e* and *f* if it is desired to make *f* the hot junction.

It is, of course, essential that the two platinum wires be made of strictly pure platinum. For this purpose, use is made of Heraeus normal thermoelement wire drawn down; this platinum has shown itself to be a standard, uniform product, the purity of which is easily controlled by the electric resistance method above mentioned.

In the table are given the results of a series of measurements at about 1050° C. of the E. M. F. of pure platinum against "platinum" crucibles of various makes. In Fig. II are shown the isothermal curves, at 900°, 1000° and 1100° C., for iridium content of platinum in terms of E. M. F. against pure platinum. There is also shown here the iridium equivalent of the impurities in the crucibles *c*, *h* and *k* of the table, in terms of their stated and observed iridium contents.

It will be noted that all impurities are, for conve-



LEGEND
 A- asbestos a- commutator d- gas tube
 C- crucible b- poles e- thermoelectric
 G- galvanometer c- air tube f- junctions

FIG. I—DETERMINATION OF PURITY OF PLATINUM WARE

crucible one terminal of an electric circuit of about 40 volts and a sharpened graphite pencil the other, with a

SUMMARY OF THERMOELECTRIC TESTS OF PLATINUM CRUCIBLES

SOURCE OR MAKER	1 Stated Ir content Per cent	2 Previously heated Hrs.	3 Temp. of test ° C.	4 Pt vs. crucible Mv.	5 E. M. F. Ir Pt-Ir curve Per cent	6 E. M. F. from excess Pt-Ir curve due to Ir or impurities Mv.	7 E. M. F. excess due to Ir or impurities Mv.	8 Reference letter	REMARKS	Date of purchase
Heraeus.....	0.0	17	1050	0.00	0.00	0.00	0.00	a	Normal thermoelement Pt	1911
	0.7	Heated	1050	2.00	0.70	2.00	0.00	b	Normal Tiegel platin	1911
	0.7	0	1050	2.60	0.90	2.00	0.60	c	Normal Tiegel platin	1911
American Platinum Works.....	< 0.2	24	1050	0.63	0.19	0.10	0.53	d	"Tiegel platin" Heraeus	1911
	< 0.2	16	1050	1.00	0.35	0.10	0.90	e	"Tiegel platin" Heraeus	1911
	0.5 to 1.5	11	1075	6.60	2.55	1.50 to 4.10	2.50 to 5.10	f	Commercial	1911
	0.5 to 1.5	4	1085	6.40	2.50	1.50	4.90	g	Commercial	1911
	?	0.5	1100	3.50	1.19	3.50	h	Loaned by purchaser	1913
Baker & Company.....	?	10	1050	6.75	2.72	6.75	i	Commercial	1908
	?	20	1050	6.95	2.78	6.95	j	Commercial	1908
	2.37	0	1030	6.00	2.37	6.00	0.00	k	Special analyzed make	1912
	0.702	12	1050	1.90	0.66	2.05	-0.15	l	Special analyzed make	1912
	Refined	0	1040	0.33	0.10	0.33	m	Specially refined	1911
	?	3 yrs.	1100	3.00	1.01	3.00	n	Used 3 yrs. in lab.	1909
Johnston Mathey & Company.....	?	0	1100	1.71	0.55	1.71	o	Average of seven commercial crucibles	1914
	?	67	1050	0.48	0.15	0.48	p	Best crucible ware	1911
	?	0	1070	0.68	0.23	0.68	q	Best crucible ware	1911
	?	20	1050	0.63	0.22	0.63	r	Best crucible ware	1913
J. Bishop.....	?	9 mo.	1100	2.10	0.68	2.10	s	Loaned by purchaser. Used continuously 9 mo.	1913
	?	0.5	1100	2.20	0.72	2.20	t	Loaned by purchaser. Used continuously 9 mo.	1913
	Refined	31	1050	0.60	0.21	0.60	u	Specially refined	1911
Quennessen De Belmont, Legendre et Cie.....	?	10	1050	2.79	0.98	2.79	v		1911
	?	0	1060	2.80	0.98	2.80	w		1911
	?	4	1030	1.60	0.56	1.60	x		1911

rhostat in series. The operation of soldering consists in drawing a minute arc between pencil and

nience, expressed in terms of iridium content. Of particular interest is the comparison of columns 2 and 6,

the former giving the stated iridium content (and often accompanied by the statement that there are no other impurities) and the latter giving the iridium content as determined thermoelectrically by experiment and use of Fig. II. In several instances, notably for the

certain check on the platinum purity.¹ One of the most undesirable impurities often found in commercial platinum ware is iron; this is readily detected by ignition and subsequent washing with hot HCl and applying the usual color test for Fe. Iron, if present in

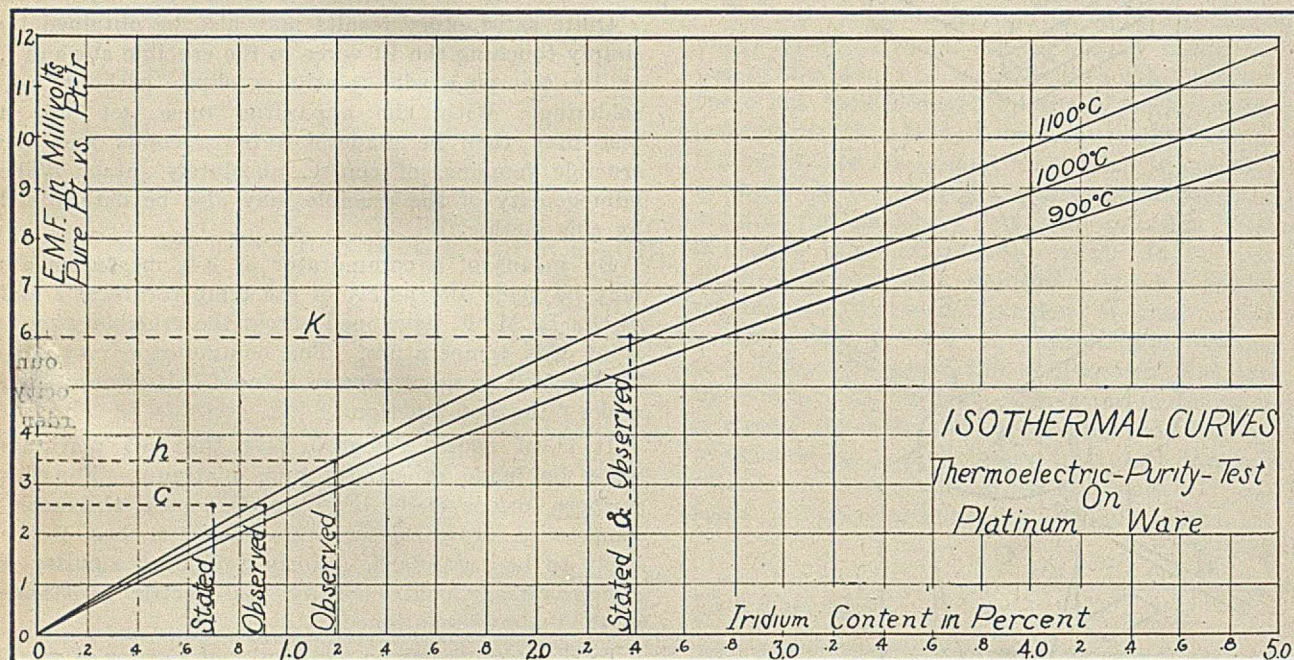


FIG. II—IRIDIUM CONTENT VS. E. M. F. AGAINST PLATINUM

crucible of normal thermoelement platinum (*a*), of Heraeus and for Baker's crucible (*k*), of 2.37 per cent iridium, the stated and found iridium contents agree exactly. On the other hand there is a wide divergence from the supposed and actual iridium content for several of the crucibles; for example, *f* of the American Platinum Works, supposed to contain not over 0.50 to 1.5 per cent iridium, actually contains an iridium equivalent of 2.50 per cent; and even in the case of refined, specially refined and best crucible ware, the equivalent iridium content is not inconsiderable. Among the 22 crucibles examined there is but one, (*i*), of Baker & Co., containing less iridium than is stated.

In comparing the stated contents, column 2 of the table, it should be borne in mind that some of these crucibles, excepting perhaps those marked refined or specially refined, may contain iridium which is put or left in the crucible material purposely, usually for the purpose of stiffening, or in the case of commercial ware, because such Pt always carries Ir. The method here described, however, provides a delicate means of determining whether or not the desired limitation of impurity, expressed in terms of iridium content, and as measured thermoelectrically, has been met by the manufacturer. For the experimental arrangement here given, the amount of impurity is readily determined to 0.01 per cent and this accuracy could be improved upon if necessary.

The method does not distinguish the various possible impurities from each other but is nevertheless a

relatively considerable quantity, will also discolor the crucible on ignition.

It would probably be advantageous to substitute rhodium for iridium in platinum crucibles when stiffness is desired and when exact weighings have to be taken before and after ignition, for the reason that rhodium is far less volatile than iridium, and somewhat less so than platinum, although it appears not yet to have been proven that the more volatile metals alloyed in small amounts with platinum retain their volatility independently of the presence of the platinum.

The above described thermoelectric method will not alone distinguish between small amounts of rhodium and iridium, but a combination of the thermoelectric and loss of weight methods might be devised that would be satisfactory. An electric discharge method operated at high temperatures might possibly be made sufficiently selective to differentiate between platinum alloys of iridium and rhodium. There is evidently room for much more work along these lines.

BUREAU OF STANDARDS
WASHINGTON

THE DETERMINATION OF THE DUST FALL IN THE NEIGHBORHOOD OF CEMENT PLANTS

By J. P. MITCHELL

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The determination of the dust fall in the neighbor-

¹ All the metals found associated with platinum, such as palladium, iridium, rhodium, etc., when alloyed with platinum (up to 90 per cent only of palladium) give, at high temperatures, an E. M. F. of the same sign against pure platinum. Therefore, there is no ambiguity in balancing one impurity against another.

hood of cement plants has recently become a matter of considerable importance, largely as a result of litigation which has arisen between certain companies engaged in the manufacture of cement, and the residents of the region in the immediate neighborhood of the plants. This litigation has taken the form of suits on the part of the residents either to secure damages, or obtain injunctions against the operation of the plants, based upon injury, alleged to have been inflicted upon the property of the residents. Two entirely distinct claims of injury have been advanced. One is based upon the alleged infliction of actual damage to vegetation, either the actual destruction of plants and actual destruction, in whole or in part, of crops, or the lessening of the market value of plants and crops by the presence of cement dust on the plant foliage and fruit. The other claim is based upon the less specific ground of "nuisance." It is alleged that the cement dust enters the houses of the complainants, causes much personal inconvenience, and in general interferes with the right of every land holder to the enjoyment of normal and unpolluted atmospheric conditions. Whatever be the basis of the complaint, it is a matter of record that such complaints are becoming more common in all parts of the country, and in every such case it becomes a matter of importance to both parties that the true conditions be determined as accurately as possible. Such a determination involves a study of the amount and character of the dust fall throughout the region in question, and a correlation of the results of such a study with the prevailing meteorological conditions. It is the purpose of this paper to deal with the methods available for the determination of the dust fall, and the proper interpretations of the results obtained.

Another aspect of the subject deserves mention. From the cement manufacturer's point of view the dust thrown from the stacks of the plant represents not only a constant source of possible legal expense and anxiety, but also a very real economic waste. The amount of dust lost from a kiln of the ordinary rotary type may amount to a number of tons in one day, and this dust is not composed of a useless waste product, but is the very substance for the production of which the whole plant is operated. The recovery of this dust, therefore, should not be regarded entirely as an expense, but as a possible measure of economy. The determination of the amount of dust lost in this way from a given plant cannot be carried out satisfactorily by the determination of the dust fall in the vicinity, but must be arrived at by a study of the conditions existing in the stacks of the plant themselves, and therefore lies outside the scope of this paper.

The methods which have been used for the determination of the amount of dust in the atmosphere may be divided into four groups.¹

The most scientifically accurate method is without doubt that of Aitkin. This depends upon condensing

¹ For a very complete discussion of the methods available, and bibliography of the subject, see U. S. Dept. of Agriculture, Bureau of Soils, *Bull. No. 68*, "The Movement of Soil Material by the Wind," by E. E. Free.

water vapor on the dust particles, and counting the resulting drops of water.

This method is too refined for the purpose under consideration. The present problem is not concerned with the ultramicroscopical particles normally present in air, or even with the increase in their number, but rather with the very much larger particles of solid matter which constitute "dust" in the ordinary meaning of the term.

Efforts have been made to catch the dust present in air in various box-like contrivances designed to catch the dust by checking the velocity of the air currents, and create conditions under which the dust will settle out and remain within the apparatus. Such methods may be useful in a study of the character of atmospheric dust, but give rise to certain difficulties when applied to the problem of determining the amount of dust fall in different localities. Since the amount of dust remaining in the box depends upon the amount of air passing into it, that is, upon the wind velocity, the results give what may be termed the "dust burden" of the air, as distinguished from the "dust fall" which would have taken place at the same point had the velocity of the air not been checked.

The dust present in the air has been determined by drawing known volumes of air through water or filters, and weighing or counting the dust particles retained. This method again gives the "dust burden" of the air, rather than the "dust fall," and while it is available for determining the amount of dust in a given volume of air at a given time, it is not available for determining the relative dust fall at different points during a somewhat extended interval of time.

The fourth group of methods depends upon the exposure, in a horizontal position, of a surface of known area coated with a substance that will retain dust falling upon it, and the subsequent removal, weighing and examination of the dust deposited upon the surface. While no method of this sort collects the small microscopic particles present in large numbers in all air, it does collect the larger particles ordinarily considered "dust," and does afford a reliable means of comparing the amount and composition of the dust actually falling upon a horizontal surface at different points during a given interval of time. It is therefore available for the purpose here in view.

The method finally adopted as most efficient and practical is the exposure of glass plates coated with vaseline, and the subsequent cleaning of the plates with gasoline, and recovery of the dust by filtration. The details follow.

The plates used were of glass and measured five by seven inches. Ordinary window glass was used, and the dimensions of the plates were carefully checked, and any plates with variations greater than one-sixteenth of an inch were discarded. Old photographic plates may be used, but are thinner and more subject to breakage than ordinary window glass. Their lighter weight also makes them more difficult to handle in the field in a high wind. These plates were laid on horizontal platforms placed at the top of high poles. The size of the platforms was such

that they just held the number of plates exposed, and projected less than half an inch beyond the outer edge of the outside plates. A wide projection, which could serve as a resting place for dust which might subsequently be blown onto the plates, was avoided. The plates were held in position by small tacks driven into the edge of the platform. The plates must be set closely together, and the tacks adjusted so as to hold the plates firmly in place, otherwise the shaking of the pole in a high wind will dislodge some of the plates. At the outset of the work some plates were lost from the platforms during a high wind storm, but with practice it was found that care in the adjustment of the tacks would prevent any loss from this cause. After the plates had been exposed for the desired length of time they were taken up, placed in tight wooden boxes provided with cleats to hold the plates apart, lowered to the ground, the box placed in a sack to exclude road dust, and taken to the laboratory.

The laboratory work involved the preparation and cleaning of the plates, and the weighing and analysis of the dust collected. Before use the plates were first thoroughly cleaned, then warmed, and while warm given a thin even coating of the purest vaseline obtainable (Kahlbaum). The vaseline while warm and liquid was best applied with a wide camel's hair brush. It was free from mineral salts, and gave no weighable residue when ignited. After exposure the plates were first carefully wiped on the edges and bottoms, and then placed in a white enamel photographic tray, and covered with filtered gasoline. The dust deposit was then loosened by rubbing with a camel's hair brush, the plates held on edge in the tray, and rinsed on both sides with a fine stream of gasoline blown from a wash bottle. All of one set of plates, which had been exposed on the same platform for the same time, were cleaned in the same tray of gasoline. Examination of the plates after the adhering gasoline had evaporated often disclosed fine streaks which looked like unremoved dust particles, but these streaks were found to be both volatile and soluble in alcohol, showing that all the mineral particles had been removed from the plates by the cleaning process.

The dust obtained in the trays was separated from the gasoline by filtration through paper filters. Some difficulty was experienced due to the hardening of the paper by the gasoline and consequent slow filtration. This difficulty can be overcome by first wetting the filter paper with alcohol, and by the use of moderately strong suction. The filtrates are never perfectly clear, but are slightly cloudy as a result, apparently, of the formation of an emulsion by the gasoline with some constituent of the vaseline. The evaporation of the filtrates in a number of cases, gave, after ignition, no weighable residue. The filter paper containing the dust was placed in a weighed platinum crucible, and heated cautiously to incipient redness until the paper was reduced to ash and the crucible and contents had attained constant weight. The result was corrected for the known weight of the filter paper ash.

The analysis of the dust samples was carried out according to standard methods. The sample was fused with sodium carbonate, digested with concentrated hydrochloric acid, and the silica determined after two evaporations and dehydrations, and was checked with hydrofluoric acid. The calcium was precipitated as calcium oxalate, after removal of iron and aluminum, from the boiling solution, and was ignited to calcium oxide until constant weight was obtained.

The choice of the locations for the exposure of the plates, the height of the platforms above the ground, the number of plates exposed in one set, and the length of time of the exposure involves the consideration of a number of factors. These factors all depend to some extent on the particular problem under investigation. Thus the topography of the country, the character of the vegetation, the climatic conditions, the season of the year, and the direction and regularity of the wind currents must all be considered, and none are identical in two specific problems. However, there are certain general principles that will apply to all cases.

Field work of this character is absolutely dependent upon fair weather. Even a light rain will interfere with the film of vaseline and adhering dust on the plates, causing "blisters" and bare spots to appear, and a heavy rain will not only wash the dust from the plates, but will also prevent the normal transportation of the dust from the plant by washing it out of the atmosphere before it has been carried to any distance. A period of settled fair weather must therefore be selected. This is vital, not only on account of the interference due to rain, but also on account of the importance of having normal wind conditions during the time of the investigation. In general, the spring and summer months are the most satisfactory; the winter months, when the wind currents are more variable, are far less desirable.

The locations selected for the exposure of the plates depend almost entirely on the objects of a particular investigation. If a lawsuit is involved, that will dictate at least some of the locations. In general, the positions selected should be at some distance from roads and buildings, and should be on exposed ground not sheltered by trees or hills. If a comprehensive study of the dust fall is under way they should be chosen in all directions from the plant, and at least one should be placed in a region clearly outside of the "dust zone" to serve as a control station.

The platforms are best placed on the top of poles from twenty-five to thirty feet above the ground. This height is important for two reasons: *First*, to place the plates above the influence of minor disturbances of the soil whether caused by man or wind. *Second*, to place the plates at an elevation where the wind currents are regular and approach, in velocity, those of the upper air currents. While nothing definite is known as to the distance to which a given dust particle will be carried by a wind of known velocity, it is known that the velocity of the wind decreases regularly downward until within about fifteen feet

of the surface of the ground. Within that distance of the ground the wind currents vary very irregularly.¹ On the other hand, the results obtained would be misleading if the platforms were placed at a great height above the surface, for then dust might be collected which would have traveled a considerable distance before falling to the ground. Platforms have been placed on the gable ends of barns during preliminary work, but this is unsatisfactory on account of the unknown effect of the building on the wind currents, and the possibility that dust formerly lodged in the roof may be blown onto the plates.

The number of plates exposed in one set, that is, on one platform at the same time, is determined by preliminary experiment, and is so selected that a sufficiently large sample of the dust will be obtained to admit of accurate weighing and analysis. It depends upon the soil and vegetation of the area in question, upon the distance from the plant, and upon the length of time of exposure. The writer has used sets of ten and twenty plates, and has found ten usually sufficient.

The choice of the time of exposure is optional within certain limits. Sets of twenty plates each are about as large as can be handled to advantage, and the time of exposure must be sufficient to allow the collection of an adequate sample under the existing conditions. On the other hand, the shorter the interval of exposure, the more accurately can the results be correlated with the corresponding wind direction and velocity records. The best plan is to use a short period of exposure, three to five days, repeatedly for a considerable length of time such as three to five weeks. In this way the results of each exposure can be compared with the corresponding wind conditions for detailed study, and the sum of the dust fall during the whole time of the investigation will show the total effect of a variety of wind conditions. The maximum time of exposure possible is set by the ability of the vaseline to retain its power of holding the dust that falls upon it. An experiment was carried out to determine this limit. Twenty plates were exposed on a platform about fifteen feet above the ground and near a dusty road. Five of these plates were left undisturbed for four weeks, five for three weeks, five were replaced by fresh plates at the end of two weeks, and five were replaced with fresh at the end of each week for four weeks. The dust collected on the fresh plates week by week was compared with that deposited upon those plates that were left untouched for two, three and four weeks. The results indicated that up to two weeks the plates held all the dust falling upon them, but that during the third week only about three-fourths of the dust falling was retained, and during the fourth week only about one-third. During the experiment the dust fall was heavy, amounting to between three- and four-tenths of a gram on five plates in one week. It is possible that with a lighter dust fall the holding power of the plates would be satisfactory through the third week, but it is probably unsafe to depend upon the accuracy of the results after an exposure of more

than two weeks, or one week in case of very heavy dust fall. It will be interesting to determine whether the decrease in the holding power of the vaseline surface is caused by simple exposure to air and sunshine, or whether it is dependent only upon the amount of dust which has lodged on the plate. The determination of this point can be carried out only by experiment, and since the experiment requires at least four weeks of continuous fair weather it will be some time before it can be completed.

The results of the field and laboratory work are obtained in terms of grams of dust deposited upon a given number of plates during a certain number of days, hours and minutes. For convenience in comparing the dust fall at different points it is necessary to express the results in terms of some selected unit. The most convenient unit is that of "pounds per acre per day." For some purposes it is interesting to calculate the results in terms of "tons per acre per year," but this latter unit is open to the objection that, since the experiments never exceed a few days in length, it presumes that the dust fall determined is constant throughout a year.

The simple determination of the total dust fall at given points does not afford sufficient information on which the distribution of the cement dust from a plant can be determined. It is necessary to separate the total dust fall into what may be termed "field dust," that is, the dust blown from the surface of the ground and normally present in the air, and "cement dust" proper. By "cement dust" is meant that solid material which issues from the stack of a cement plant. Since cement proper is prepared by the grinding of the clinker, it cannot be present in the dust, but the latter will consist of "raw mix," partially calcined but not fused, of a composition dependent upon the raw materials used and the construction of the kiln. The simplest method for separating the total dust fall into field and cement dust, is to subtract from the dust fall found near the plant that found at control stations located outside the possible path of the cement dust, and consider the difference to be cement dust. While simple, this method may be very misleading. It might be satisfactory if the topography, as well as the character of the soil and its use were uniform throughout the whole field under consideration. Such, however, is not likely to be the case. The dust fall at remote control stations is not necessarily that normal to the region near the plant, where the soil, agricultural and wind conditions may be very different, and some more accurate means of separating the dust fall into its components is required. This separation may be accomplished by the analysis of the samples, and on the basis of their silica and calcium oxide content. Cement dust contains much calcium, present either as oxide, carbonate or silicate, and relatively little silica, while field dust contains much silica and relatively little calcium. To carry out the calculation, it is first necessary to obtain a sample of the dust as it issues from the stacks of the plant, and determine the percentage of silica and calcium, usually expressed as calcium oxide, which is present. The samples col-

¹ Cf. *Bull.* 68, Bureau of Soils, pp. 41-44. Udden, *Jour. Geol.*, 2, 318.

lected in the field have been found, invariably, to contain more silica and less calcium oxide than the sample from the stacks, showing clearly the effect of the admixture of field dust. The percentage of silica increases, while that of calcium oxide decreases, with increasing distance from the plant. The silica and calcium oxide contents of the samples from the control stations are also determined. It is then possible, on the basis of the calcium oxide content, to calculate what mixture of cement dust and field dust, both of known calcium oxide content, will give the percentage of calcium oxide found in the sample obtained at any point in the field. The same calculation can be repeated on the basis of the silica content, and the results have been found to agree very closely. It is thus possible to separate the total dust fall at any point into "field dust" and "cement dust," with satisfactory accuracy. Two possible

dust. Calculations made in the same way on the basis of the silica content gave results in good agreement with those given in the table.

A number of important points are brought out very clearly by the data in Table I. It is apparent that the dust fall does not depend only on the distance from the cement plant. Thus D 34 at 2.8 miles shows a higher cement fall than D 25 at 1.2 miles. This is due, of course, to the difference in the direction from the plant. Sample D 34 was taken in the direction of the prevailing wind, while sample D 25 was taken to one side of this direction. If the wind conditions are fairly steady, the dust fall will often be restricted to a very narrow zone, but its length will depend upon the velocity of the wind. If the wind be light the dust will not travel far, but the dust fall will be heavy near the plant, while if the wind has a high velocity the dust will travel a great distance, but

TABLE I—DATA SHOWING DUST FALL IN THE NEIGHBORHOOD OF A CEMENT PLANT

Ref. No.	Distance from plant Miles	Number of plates	Time of exposure Days	Weight sample Grams	Pounds per acre per day	SiO ₂ Per cent	CaO Per cent	Calculated cement dust Per cent	Calculated pounds per acre per day	
									Cement dust	Field dust
D 26	1.2	10	3.695	1.0152	10.9	17.92	47.95	95.5	10.4	0.5
D 22	1.2	10	8.142	2.0789	10.1	17.29	47.39	94.3	9.5	0.6
D 25	1.2	10	3.691	0.5261	5.6	19.58	44.61	88.3	4.9	0.7
D 33	1.7	10	3.843	0.5639	5.8	19.59	43.19	85.2	4.9	0.9
D 30	2.2	10	3.772	0.0611	0.7	34.86	31.09	59.1	0.4	0.3
D 31	2.0	10	3.771	0.1611	1.7	26.32	33.52	64.3	1.1	0.6
D 34	2.8	10	3.885	0.6344	6.5	18.52	45.17	89.5	5.8	0.7

sources of error in this procedure are apparent. It may be that the calcium oxide content of the dust from the control stations is not identical with that of the field dust near the plant. This possible error is unavoidable, but since the amount of calcium present in soil is low, and fairly uniform, it is not serious. The presence of cement dust from the plant in the soil near at hand, prevents the use of soil analyses to control this possible error. The presence of this cement dust in the ground introduces the second possible error referred to above. It may be that some of the cement dust collected on the plates was previously deposited on the ground, and was blown up onto the plates by the wind currents. This possibility cannot be avoided, but can only be minimized as far as possible by placing the plates well above the ground, and at points removed from dusty roads or open fields with cultivated surfaces.

Some of the results actually obtained in the field, the analytical data, and the percentage of cement dust in the sample calculated as explained above, are given in Table I.

TABLE II—EFFECT OF DEPOSITION OF SAND ON COLLECTING PLATES BY A FLURRY OF WIND

Ref. No.	Conditions	No. of plates	Time of exposure Days	Weight sample Gram	Pounds per acre per day	SiO ₂ Per cent	CaO Per cent	Calculated cement dust Per cent	Calculated pounds per acre per day	
									Cement dust	Field dust
49	Normal	10	2.948	0.5125	6.8	29.9	33.7	65.0	4.4	2.4
56	Wind flurry	6	2.958	0.5236	11.7	45.0	19.2	36.8	4.3	7.4

The results given in the last three columns of Table I are calculated from the data given on the basis that cement dust contains 50 per cent calcium oxide and that field dust contains 4 per cent of calcium oxide. This basis was determined by averaging analyses of cement dust, and of field dust obtained on plates exposed at such a distance and direction from the plant that there was no chance of its containing cement

the fall near the plant will be much lighter. It is apparent that the wind conditions must be thoroughly known before the results of field work can be properly interpreted.

Examination of Table I will disclose one very important point, namely, the relatively constant value for the fall of field dust, expressed in pounds per acre per day in the last column of the table. The average value for the field dust fall was 0.6 pound per acre per day, and the highest and lowest values differed from this by only 0.3 pound. This, too, in spite of large variations in the fall of cement dust. It is apparent, therefore, that this method permits the estimation of the fall of cement dust over and above the normal fall of field dust with satisfactory accuracy. The ability to eliminate disturbing factors by this method is also illustrated by the following case: The two results given below in Table II were obtained at the same place under very nearly identical weather conditions. The location was near an open sandy region. During the time of collection of the second sample there was a short period of high wind from

such a direction that a large amount of sand from this area was blown up onto the collecting plates. In spite of this interference, however, it is clear that the cement dust fall was nearly identical throughout the two periods.

It is interesting to compare the figures for dust fall which have been given with those which have been recorded in connection with great dust

storms. Such a comparison is given in Table III.

TABLE III(a)—COMPARISON OF DATA ON EUROPEAN SIROCCO DUST FALLS WITH THAT FOUND NEAR CEMENT PLANTS

DATE	PLACE	Weight of dust in pounds per acre per day
Oct. 16, 1846	Southeastern France	5.62
March 31, 1847	Tyrol	17.80
1859	Westphalia	275.0
Feb., 1862	Salzburg, Austria	0.75
March 24, 1869	Carniola, Austria	44.7
March 19, 1901	Taormina, Sicily	24.1
March 9-12, 1901	Europe	32.4
	Maximum	3.02
	Minimum	10.9
1913 (by author)	California. Maximum in Table I	
1912 (by author)	California. Maximum found near a cement plant	22.9
1913 (by author)	California. Lowest dust fall far from a cement plant	0.42

(a) The data used for comparison in this table were obtained from *Bull.* 68, Bureau of Soils, United States Department of Agriculture, and have been recalculated into terms of pounds per acre per day. Where only the date of the dust fall was given the time was assumed to be one day.

The question of the elimination of the loss of cement dust by the installation of proper devices at the plant does not come within the scope of this paper. It is sufficient to state that it can be accomplished, and that it is a matter of record that amounts in excess of fifty tons per day of cement dust have been recovered from a five-kiln plant in such a form as to permit its use in the manufacture of cement, and that the result has been advantageous to both the cement company and its neighbors.

CONCLUSION

In this paper the problem arising from the loss of large amounts of dust through the stacks of cement plants has been outlined. The methods available for the determination of the dust fall in the field have been stated and discussed. A practical method has been given in detail, and a method of calculation by which the cement dust can be distinguished from the field dust has been explained. The importance of a study of the wind and weather conditions as a preliminary to proper interpretation of the results of field work has been emphasized. Data have been presented which show the results obtainable by the methods given, and the relation of the dust fall near cement plants to that of certain great dust storms.

The writer wishes to express his indebtedness to Mr. G. S. Bohart, of this University, for his assistance in carrying out the analytical part of the work which has been presented.

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THE COMBUSTION METHOD FOR THE DIRECT DETERMINATION OF RUBBER¹

By L. G. WESSON

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The method most in use at the present time for the determination of the caoutchouc content of rubber goods is an indirect one, in which the sample is analyzed for its moisture, mineral matter, sulfur, resin and other contents, these values being then added together and the sum subtracted from 100 per cent to arrive at the percentage of rubber present. As many of the best methods for estimating these constituents are admittedly inaccurate, the indirect method is not a satisfactory one. A number of direct methods

¹ Published by permission of the Director of the Bureau of Standards.

have been proposed and thoroughly tested, but as they depend for their accuracy on certain derivatives of rubber, the nitrosite, the nitrosate, and the tetrabromide, that have not thus far been obtained with unvarying composition, these methods have not found general acceptance either as technical or research methods for this extremely important estimation. It is hoped that the method¹ about to be described will lend itself to development not only as a dependable one for rubber research work, but also for the commercial laboratory.

The procedure, in brief, consists first in forming the nitrosite of rubber by the action of nitrogen trioxide gas upon a finely ground and acetone-extracted sample of the rubber suspended in chloroform. After the completion of the action, the insoluble nitrosite, fillers, etc., are filtered from the chloroform, and the nitrosite is dissolved in acetone. The suspension of finely divided mineral matter is then allowed to settle out, or is thrown down with the centrifuge. An aliquot portion of the solution is transferred with a pipette to a small flask, and its volume reduced by evaporation to a few cubic centimeters. This small volume of acetone solution of the nitrosite is now transferred with the help of ethyl acetate to a porcelain boat containing alundum, and, after the acetone and ethyl acetate have been expelled by warming the boat for several hours in a drying oven, the nitrosite is burned in a current of oxygen, and the carbon dioxide thus formed is absorbed in soda-lime and weighed. If all of the carbon originally in the sample as rubber, and only such carbon, reaches the soda-lime apparatus as carbon dioxide through the intermediate nitrosite, the equation



enables one to calculate the $C_{10}H_{16}$ or real caoutchouc content of the sample.

PROCEDURE AND APPARATUS IN DETAIL—The procedure and apparatus employed in obtaining the results given later are the outgrowth of many trials and experiments. Doubtless deviations are allowable in many points, but there was not opportunity to study the effect of changing various factors.

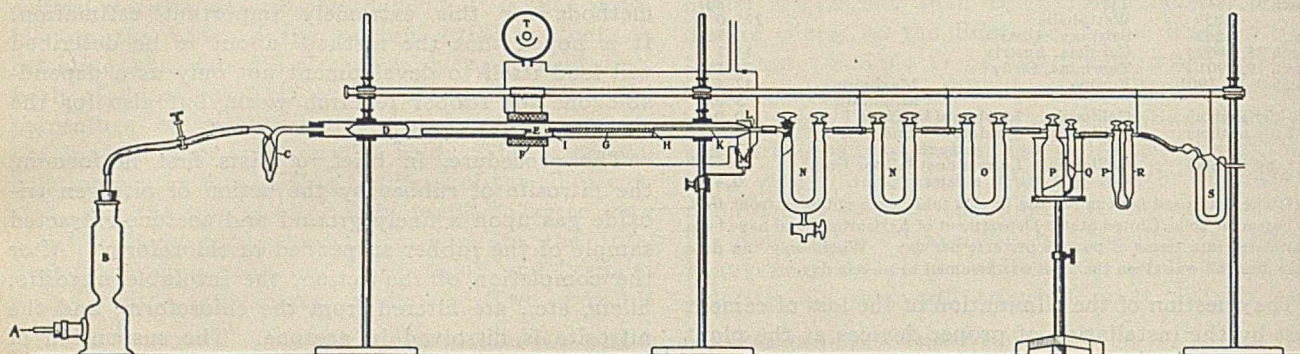
The sample should be ground to pass a 20-mesh sieve, if possible, or cut up fine with the scissors if very soft. A weighed amount of the sample, 0.5 gram for compounds containing about 50 per cent or less of rubber, and 0.25 gram for those containing a higher percentage, is wrapped into a bundle with a 9 cm. filter paper and extracted for 3 to 4 hours with acetone in an apparatus of the Wiley or Cottle type, in which the sample is extracted by the solvent at the boiling point of the latter. The residue, from which the excess of acetone has been squeezed with the fingers, is then transferred to a 50 cc. calibrated, glass-stoppered flask and allowed to dissolve or swell up in about 40 cc. of chloroform,² which action may be hastened by warming the flask.

¹ A preliminary note on this method was published in *THIS JOURNAL*, 5 (1913), 398.

² The chloroform should not be previously dried, as moisture is apparently advantageous in giving a more rapid action of the nitrogen oxides on the rubber.

The rubber is now submitted to the action of nitrogen trioxide gas, evolved by running nitric acid of sp. gr. 1.3 dropwise on arsenic trioxide contained in a flask warmed in a boiling water bath. After the gases have been passed through an empty gas-washing bottle to condense most of the moisture and nitric

stopping, for a half hour to ensure complete solution of the nitrosite in the acetone. The volume is now made exactly to the mark, the flask stoppered and shaken, and, to obtain a clear solution, the insoluble mineral matter is allowed to settle, or better, is quickly centrifuged out: 1500 revolutions per minute



APPARATUS FOR THE DIRECT DETERMINATION OF RUBBER BY THE COMBUSTION METHOD

- | | | |
|--|---|--|
| A. Oxygen enters | G. Platinum spiral about 10 cm. long (No. 20 wire) | O. U-tube containing 30-mesh granulated zinc |
| B. Moist soda lime | H. Porcelain stem | P. Soda lime |
| C. Bubble counter containing sulfuric acid (Dennstedt) | I. Platinum leg for supporting stem | Q. Calcium chloride |
| D. Closed tube of Jena combustion glass, 8 cm. long, to prevent backward diffusion | K. Platinum leads 8 cm. long (No. 10 wire) | R. Alumina |
| E. Porcelain boat | L. Short platinum wires sealed into tube | S. U-tube containing palladium chloride solution |
| F. Heating coil of nichrome ribbon | M. Mercury contacts | T. Rheostat |
| | N, N. U-tubes containing glass beads and H_2SO_4 - $K_2Cr_2O_7$ | |

acid carried over from the generator, they enter the chloroform through a delivery tube joined by a rubber connection to the gas washing bottle, and fitting closely end for end to the same. The flask containing the chloroform should be immersed in a beaker of cold water during the reaction, since the solubility of the oxides of nitrogen in chloroform is increased and the danger of the gas exerting a partial oxidation of the rubber to CO_2 is probably diminished thereby. The gas should be passed into the chloroform until a deep green color is obtained which is permanent for at least 15 to 20 minutes after the delivery tube has been disconnected from the gas generator.

The next morning the chloroform is decanted off, using gentle suction, through a small Gooch crucible provided with a mat of dry asbestos. If the filtrate is colored brown from the dissolved gases, one can be certain that a sufficient excess of the nitrogen oxide has been used. After the flask has been rinsed out several times with small volumes (5 cc.) of chloroform, each time decanting carefully through the crucible, the excess of chloroform and acid vapors which remain in the flask should be expelled by the passage for a few minutes of a gentle air current through the original delivery tube. If some nitrosite from the rubber connection is on the upper end of this tube, it may be easily removed by moistening it with acetone and wiping clean with a piece of filter paper.

The nitrosite in the crucible is then dissolved by placing the crucible in a 75 cc. beaker, adding successive small portions of acetone, and pouring each into the original flask until about 40 cc. have been used. No harm is done if a portion of the asbestos gets into the flask. In the meantime, the delivery tube has been freed from the nitrosite by sucking some of the acetone up into it from the beaker, and rinsing the outside into the same. Let the flask now remain in a beaker of water with occasional shaking, without

are sufficient to give a clear solution in from 5 to 10 minutes.

To regain the original temperature, the flask is now allowed to stand again in the beaker of water until the correct volume is once more attained. An aliquot portion of the solution (25 cc.) is now pipetted to a 50 cc. Erlenmeyer flask. To reduce the bulk of this solution to a few cubic centimeters, the flask is warmed in a dish of water while a current of air is blown into the flask. The acetone should not be completely expelled. The nitrosite is transferred from the Erlenmeyer flask to a porcelain boat 14 cm. long \times 1.1 cm. wide, and about two-thirds full of alundum,¹ which is used to secure an even combustion of the nitrosite. The acetone solution of the nitrosite should not be poured into the boat, but should be drawn up into a small (2 cc.) pipette and run out evenly over the alundum. Several 2 to 3 cc. portions of ethyl acetate² are then used to rinse out the remainder of the nitrosite, using a small wash bottle and the pipette for this purpose. Ethyl acetate is used for the expulsion from the nitrosite of acetone which is otherwise retained in small amounts, perhaps mechanically by the nitrosite or by the portion of the mineral matter which passes in solution with the nitrosite into the boat, or by reaction with the nitrosite during drying. When enough wash liquid has been added to the boat to show above the alundum, the most of it should be expelled by placing the boat for a few minutes in the drying oven. The same procedure should be repeated at least once, using small portions of ethyl acetate in the transfer as described above. When one is certain that no nitrosite remains in the flask or pipette, the boat is dried for 2 hours at about 85° C.

¹ "RR" alundum, 90-mesh, specially prepared for carbon determinations. Norton Company, Worcester, Mass. A fresh portion should be used for each combustion.

² The so-called "absolute ethyl acetate" containing about 2 per cent of alcohol. It should be redistilled before use since it may contain other organic substances not easy to volatilize.

A circulation of air in the oven to carry away the acetone and ethyl acetate vapors will assist the drying greatly. The nitrosite is now ready for the combustion.

The combustion apparatus as it was finally developed, contains features already used outside of this laboratory,¹ and others original with either my colleagues or myself. The arrangement of the parts is best understood from the figure.

The tube is of Jena combustion glass, or better of quartz, 50 cm. long and 1.5 cm. bore. The nitrosite is decomposed by the heat of an external coil, made by winding two layers of nichrome ribbon,² leaving 0.1 cm. between the turns, on an asbestos-covered copper or brass tube, 5 cm. long and of such a diameter as to leave a 0.3 cm. space between the metal and the combustion tube; and then covering it with asbestos paper to form an insulating layer 1 cm. thick. During the combustion the coil as it moves forward should never be moved so fast that its forward end reaches more than 1 cm. beyond the border between the black carbon of the undecomposed nitrosite and the white alundum of the completely burned portion. The decomposition products are carried forward by the current of oxygen over a red-hot spiral of platinum wire which serves as the catalyzer for complete oxidation. The spiral is made from 1.3 meters of No. 20 wire wound into a cylinder 0.7 cm. in diameter, and is supported in the middle of the tube by an unglazed porcelain or clay stem 12 cm. long and 0.3 cm. in diameter, the return end of the spiral passing back through the stem. The leads for the spiral are 8 cm. long and are made with No. 10 platinum wire. One end of the pipe-stem is supported by these leads, and the other end by a small leg of platinum wire. The leads may connect with the outside in one of several ways. There may be a rubber stopper at the forward end of the tube through which are pushed two heavy copper, nickel, or platinum wires, the ends of which are bent into small loops into which the platinum leads are placed before the insertion of the stopper. Nickel is the better of the first two metals named. The platinum wires are good, as they may be sealed into small glass tubes which reach just through the stopper. In any case the stopper soon deteriorates, although this action has no noticeable effect on the results of the combustion. The most satisfactory method, however, consists in drawing down the forward end of the combustion tube as in the diagram, joining on a 3 cm. tube of 0.5 cm. bore, and sealing in two 3 cm. lengths of heavy platinum wire. These are bent up on the inside and the platinum leads are slid forward so as to rest upon them.

The oxides of nitrogen formed during the combustion of the nitrosite are absorbed by a saturated solution of potassium bichromate in concentrated sulfuric acid. Acid vapors and sulfur trioxide are held back by 30-mesh granulated zinc. Attention is called

¹ The most important of these are the electrically heated platinum coil used as a catalyzer [Morse and Taylor, *Am. Chem. J.*, **33** (1905), 591]; and the electrically heated external coil used for the decomposition of the substance to be burned.

² $0.11 \times \frac{1}{2}$ " R = 1.3 to 1.5 ohms per ft., Driver Harris Wire Co., Harrison, N. J.

to the form of apparatus in the figure for the absorption of carbon dioxide by soda-lime. The capillary tube makes a good substitute for a stopcock on account of its comparative lightness, and is effective in separating the moist soda-lime from the calcium chloride. Empty, the apparatus weighs 20 to 25 grams and will hold 35 grams of soda-lime,¹ and 7 grams of calcium chloride. It will absorb 10 grams or more of carbon dioxide without renewal. The second soda-lime tube contains alumina² in its second arm, which, it is thought, dries the gas to a degree comparable, for the purposes of the present work, to the drying by the concentrated sulfuric acid which precedes the soda-lime apparatus. To be certain that complete combustion is obtained, the gas finally passes through a faintly yellow solution of palladium chloride in water. While using the apparatus in its present form, it has not been necessary to renew the palladium chloride solution, no sign of reduction having appeared in a number of combustions. The sulfuric acid-bichromate solution must be renewed in the first U-tube after every two or three analyses, while the zinc will last for a larger number of determinations.

CALCULATION—If 0.5 gram sample, and 25 cc. out of 50 cc. of the acetone solution have been taken, the weight of CO₂ found multiplied by $\frac{136}{440} \times 4 \times 100 = 123.6$ gives the percentage of C₁₀H₁₆ on the basis of C₁₀H₁₆ → 10CO₂.

TIME REQUIREMENTS—For a single nitration, about 15 minutes per sample are necessary; for the combustion, 30 to 45 minutes. Analyses of samples ground up in the forenoon of one day are completed the next. With one combustion tube, two analyses may be run per day along with other work, while by using two tubes it is thought that, after some proficiency has been acquired, as many as four determinations can be made per day.

FORMATION OF CO₂ FROM THE RUBBER DURING NITRATION—It is claimed by Alexander,³ that during the formation of the nitrosite and nitrosate of rubber, large quantities of carbon dioxide are evolved from the oxidation of the rubber by the nitrogen oxides. Gottlob,⁴ on the other hand, found only very small amounts of carbon dioxide. The writer made several tests of this important point by passing the gases from the rubber solution into a large volume of clear, saturated baryta water. Fine Para rubber, previously extracted with acetone and dried, was used for the experiments. After the chloroform, cooled as in the analytical procedure, had attained the deep green color, the apparatus was allowed to stand three to four hours, after which a current of carbon dioxide-free air was used to sweep into the barium hydroxide solution any carbon dioxide which might have formed during the interval. Only a trace of barium carbonate, a slight ring in the delivery tube, was formed, although the barium hydroxide solution remained alkaline throughout the experiment.

¹ The J. T. Baker Chemical Co. furnishes a 12-mesh soda-lime containing 15 per cent water, prepared for carbon dioxide absorption.

² F. M. C. Johnson, *J. Am. Chem. Soc.*, **34** (1912), 911.

³ *Z. angew. Chem.*, **20** (1907), 1358; **24** (1911), 684.

⁴ *Ibid.*, **20** (1907), 2213.

DETERMINATION OF THE SULFUR OF VULCANIZATION—If the statement of Alexander¹ proves to be true, that the sulfur of vulcanization of the rubber remains quantitatively in the nitrosite, this method could possibly admit of the simultaneous determination of the sulfur of vulcanization. An aliquot portion of the clear acetone solution of the nitrosite would be evaporated to dryness, and the sulfur determined in the usual way.

RESULTS—The following results were obtained by the method above described. All are given that have been obtained on good quality, soft-vulcanized compounds since the date after which no great changes in the procedure were made. Whether the method is applicable to compounds of poor quality has not been determined, and as the author is no longer in a position to work on this point the field must be left to others. This Bureau may be able, however, to work in this field at some later date.

- A. A washed and dried Up-river Fine gave 94.0, 95.1, 95.8 and 95.9 per cent $C_{10}H_{16}$.
Average = 95.2 per cent plus 3.3 per cent acetone extract = 98.5 per cent.
- B. A commercial compound containing 45 per cent Fine Para gave 42.0, 42.2, 42.7, 43.1, 43.3 and 43.4 per cent $C_{10}H_{16}$.
Average = 42.8 per cent plus 1.3 per cent acetone extract = 44.1 per cent.
- C. A commercial compound containing 48 per cent Fine Para gave 44.8, 45.1, 45.1 and 45.3 per cent $C_{10}H_{16}$.
Average = 45.1 per cent plus 2.4 per cent acetone extract = 47.5 per cent.
- D. The same compound after standing finely ground for a month gave 43.4, 43.5, 43.5 and 45.0 per cent $C_{10}H_{16}$.
Average = 43.7 per cent.
- E. A commercial compound containing 25 per cent Fine Para and 20 per cent Caucho or 45 per cent gum gave 40.3, 40.4, 40.6 and 41.1 per cent $C_{10}H_{16}$.
Average = 40.6 per cent plus 3.2 per cent acetone extract = 43.8 per cent.
- F. A commercial compound containing 41.5 per cent Coarse Para gave 39.5, 39.6, 39.8 and 40.1 per cent $C_{10}H_{16}$.
Average = 39.8 per cent plus 2.1 per cent acetone extract = 41.9 per cent.

SUMMARY

A new method for the direct determination of rubber is described, which is based upon the combustion of the nitrosite of rubber in a current of oxygen, and weighing of the carbon dioxide thus formed. The results indicate a fair degree of reliability for both raw rubber and high-grade vulcanized compounds. The use of this method for the analysis of low-grade compounds and for the simultaneous determination of sulfur of vulcanization may be possible if its application to these fields is further studied.

During this work many valuable suggestions were made by Dr. W. F. Hillebrand, Mr. J. B. Tuttle, and a number of others at this Bureau, and I take this opportunity to express to them my appreciation of the same.

BUREAU OF STANDARDS
WASHINGTON

OSAGE ORANGE—ITS VALUE AS A COMMERCIAL DYESTUFF²

By F. W. KRESSMANN

INTRODUCTION

This study is the result of an investigation on the utilization of osage orange mill waste.

The trunk of the osage orange tree is rather small in size, misshapen, and generally defective as a saw

¹ *Z. angew. Chem.*, **20** (1907), 1364; **24** (1911), 687; *Ber.*, **40** (1907), 1077.

² Presented at the 49th meeting of the A. C. S., Cincinnati, April 6–10, 1914.

log; and, although because of the valuable properties of the wood (for wagon felloes especially) closer utilization will scarcely be found in the use of any other wood, comparatively large amounts of waste are produced annually.

Osage orange has long been used in Texas in a small way as a dyewood. The roots, bark, and wood are chipped and boiled with water and a more or less permanent yellow is obtained from the extract. Sargent¹ mentions the root bark as a source of a yellow dye and it has even been suggested by some² that osage orange is superior to fustic in its dyeing qualities, although no actual comparative experiments between fustic and osage orange seem to have been recorded.

In view of these facts, it seemed advisable not only to determine the chemical nature of the dyestuff but also its dyeing value as compared with the commercial dyewood it resembles most, namely, fustic.

A qualitative study of the aqueous extract obtained from the wood showed that the dyeing principles present were, as in fustic, morin or moric acid, and morintannic acid or maclurin. From a preliminary series of dyeing experiments made at this laboratory, it was found that osage orange, like fustic, is a poly-genetic mordant dyestuff.

Since the wood seemed to contain a sufficient amount of dye to give it commercial value, a series of comparative dyeing experiments on fustic and osage orange were arranged so as to determine as accurately as possible the value of osage orange in terms of a well-known standard such as fustic. In order to have these experiments performed at institutions best equipped for the purposes and also to obtain the results of a number of different workers in this field, the coöperation of a number of the leading textile schools³ of the country was sought and the writer wishes to take this opportunity to thank them for their coöperation and assistance.

COMPARISON OF DEPTH OF COLORS PRODUCED

REPORT E⁴—"It was noticed that dyeings produced with osage orange were weaker than those obtained with fustic under the same conditions. Since the osage orange produced a shade of color slightly different from the fustic in most cases, it is difficult to determine the exact relative strength of the two products. It seems, however, that the osage orange contains something like 20–25 per cent less coloring matter than the fustic."

REPORT B—"Dyeings were made under identical conditions on chrome mordanted worsted yarn with the same amounts of both the wood and solid extracts of both substances (*i. e.*, fustic and osage orange).

"The osage orange wood gave the heaviest shades

¹ Sargent, Chas. S., "Manual of the Trees of North America."

² U. S. Dept. of Agr., *For. Serv. Cir.* **184**, "Fustic Wood, Its Substitutes and Adulterants," by G. B. Sudworth and C. D. Mell.

³ Philadelphia Textile School. New Bedford Textile School. Lowell Textile School. The North Carolina College of Agriculture and Mechanical Arts. Georgia School of Technology. Osage orange sawdust furnished by Mr. L. C. Bumpus, Farmersville, Texas.

⁴ Reports on the experiments, giving the results of their test, were submitted by the coöperators, and the information given in this article is in the form of extracts taken from these reports. The reports are designated by letters, this designation being used to separate the extracts.

but, like fustic wood, the yellow obtained had a reddish cast.

“With the extracts, the yellow obtained in each case was of a greenish tone; but that of the osage extract, while not so heavy as that given by the fustic extract, was of a much purer tone, the fustic being rather muddy.”

REPORT A—“The dyeing properties of Old Fustic and osage orange are very similar, as will be seen by comparing the shades given on various mordants by the two dye-woods.”

COMPARISON OF AMOUNT OF EXTRACT

REPORT C—“Upon extraction with water, osage orange sawdust was found to give a water-soluble extract in the proportion of 7.5 grams of extract for every 100 grams of sawdust.”

REPORT B—“A sample each of the osage orange and of chip fustic was dried to constant weight and the moisture was found to be:

	Per cent
Osage orange sawdust.....	9.70
Fustic.....	7.92

“The dried samples were then extracted with methyl alcohol and gave the following amounts of matter extractable with methyl alcohol:

	Per cent of the dry sample
Osage orange sawdust.....	26.63
Fustic.....	12.71

“The residue in each case was then extracted with

shade of yellow, with chromium mordant a tan color, and with iron mordant a dark brown.”

REPORT E—“The following table shows the results obtained by dyeing osage orange and fustic wood under the same conditions on wool mordanted with the indicated metallic salts:

PREPARATION OF WOOL SAMPLE	TABLE I		REMARKS
	DYED WITH FUSTIC	DYED WITH OSAGE ORANGE	
Unmordanted	Light yellow	Light yellow	Color produced of no value
Tin mordant	Light yellow	Light yellow	Color more pronounced than above. More reddish with fustic
Alum mordant	Bright yellow	Bright yellow	Yellower and brighter with the osage orange
Iron mordant	Dark olive	Dark olive	Not so deep and slightly greener with the osage orange
Chrome mordant	Orange-yellow to old gold	Orange-yellow to old gold	Fustic sample more reddish and deeper in tone than orange

FASTNESS TO LIGHT, WEATHER, WASHING, ETC.

REPORT E—“The colors produced by dyeing on alum and tin mordanted material were fairly good in appearance but were inferior in fastness to light, especially as compared with colors obtained with the first two salts (iron and chromium). Table II shows the result of test made on dyed samples of osage orange and fustic wood with respect to fastness to light and weather.

“It was noticed that the chrome mordanted samples showed a fastness in this respect superior to that of the other samples. The length of exposure necessary

TABLE II

	FUSTIC WOOD Time exposed			OSAGE ORANGE WOOD Time exposed		
	5 days	10 days	15 days	5 days	10 days	15 days
Direct dyeing	Faded	Color gone	Faded	Color gone
Alum mordant	Slight fading	Faded	Slight fading	Faded
Tin mordant	Slight fading	Faded	Slight fading	Faded
Chrome mordant	No change	Very little if any change	Slight	No change	Very little if any change	Slight

water. The osage orange gave so little matter extractable by water that the amount was not determined. Fustic, on the other hand, gave 4.71 per cent of the dry weight. Therefore, the total extractable matter was:

	Per cent
Osage orange sawdust.....	26.63
Fustic, 12.71 + 4.71.....	17.42

REPORT E—“The results obtained from experiments made clearly show that the osage orange product does contain a dyeing principle which is very similar in most respects to that found in fustic wood, and also this principle is found in a quantity sufficient to give the orange wood some commercial value as a dye-stuff.”

CHARACTER OF DYEINGS

REPORT D—“It would appear that this substance (osage orange) could be advantageously used for self shades also in conjunction with logwood, other mordant dyes (*i. e.*, other natural mordant dyes), as well as with alizarine. Its dyeings are mono-chroic. If made in the form of a water extract it could be used for printing for yellows, browns, greens, and chocolates.”

REPORT C—“It was found that this coloring matter was of a polygenetic character, giving, with a tin mordant, a comparatively bright shade of yellow, with aluminum mordant a somewhat duller and greener

to completely destroy the color in each case on the chrome mordant was not determined, it being simply noted that samples from each product showed very nearly the same resistance to fading for time exposed. During this time the fustic was possibly more affected than the osage orange in that there was a tendency for the color to lose some of its reddish cast.

“Samples dyed with fustic and osage orange were examined as to their fastness to washing tests, the samples in each case showing a fairly good fastness in this respect.”

REPORT D—“This substance (osage orange) was found to dye level shades of good purity and brightness on wool mordanted with chromium in the usual manner. These dyeings show good fastness to light, water, and washing.”

REPORT C—“These dyeings (with osage orange) were exposed to the light for a period of six weeks, and to combined light and weather for a period of 8 weeks. With both chromium and iron mordants the dyeings show very little change at the end of 5 weeks' exposure to sunlight. With aluminum and tin mordants the dyeings show the effect of sunlight in from one to two weeks, becoming much duller. The exposure of the sample dyed with the iron mordant to light and weather for 8 weeks shows very little change. That with chromium mordant for 8 weeks

loses its brilliancy to a considerable extent, although it does not become much lighter in color. The dyeings by aluminum and tin mordants are decidedly changed by this exposure to light and weather. The dyeings by chromium and iron mordants are sufficiently fast to prove of practical value, assuming that the coloring matter could be prepared cheap enough."

SUMMARY AND CONCLUSIONS

I—The quantity and quality of the dyestuff present in osage orange is almost identical with that of fustic.

II—Osage orange may be employed as a dyewood in all cases where fustic wood is used at present. The yellows produced by direct dyeing or by aluminum and tin mordants are too fugitive to be of commercial value. But the orange-yellows, old gold, deep tan, olive and chocolate shades obtained with chromium and iron mordants are equal to, if not better than, those obtained with fustic and are of sufficient fastness to be of commercial value.

III—A domestic source of a yellow dyewood has been found that can advantageously replace a foreign material used at present. The mill waste alone from the present manufacture of osage orange amounts to over 25,000 tons annually; and if this waste could be set down in the East for \$10 to \$12 per ton it is believed that it could compete successfully with fustic, both from cost of production and quality of color produced on dyeing.

FOREST PRODUCTS LABORATORY

FOREST SERVICE, U. S. DEPARTMENT OF AGRICULTURE
(In Coöperation with the University of Wisconsin)
MADISON

THE RESERVE SUPPLY OF PHOSPHATE ROCK IN THE UNITED STATES

By W. H. WAGGAMAN¹

Received March 20, 1914

Numerous estimates have been made from time to time on the tonnage of phosphate rock available for future use in this country. These estimates vary so widely that little importance has been attached to them, and in many cases they can hardly be considered as anything more than wild guesses.

Owing to the pockety nature of many of our deposits of phosphorites or amorphous phosphates, careful surveys and thorough and systematic prospecting are necessary to gain anything like an accurate knowledge of their value and extent. The expenses of such examinations are frequently not warranted and are seldom made except immediately before actual mining operations are begun. Again, much of the phosphate property has been acquired so cheaply that only a superficial examination was necessary to justify its purchase. Under such circumstances the owners of the land may have but a hazy idea of the amount of phosphate contained therein. On the other hand, some of the phosphate deposits, such as those in our western states and in certain parts of Arkansas and Tennessee, are of such a character that fair approximations of their tonnage might be made by careful surveys.

¹ Scientist in Investigations of Fertilizer Resources.

For some time it has been obviously desirable that both producer and consumer should have some idea (even though a rough one) of the amount of phosphate rock still unmined in the United States. Again and again disquieting reports have been spread that our supplies of this mineral, so important to the American farmer, were being rapidly depleted, and that thereby the agricultural interests of this country would soon be seriously impaired. Alarm has also been caused by the fact that several phosphate companies are owned by European capitalists, and fears have been expressed that foreign capital is seeking to acquire control of the American deposits. It has been urged even that some legislation be enacted preventing the exportation of phosphate rock.

In order to show the actual basis for such reports and whether or not there is any real need for curtailing foreign shipments of phosphates, the data herein contained were collected and compiled with considerable care. The figures were obtained by correspondence and consultation with mine owners and operators, and by direct observation and field investigations of the various deposits throughout the country. So little development work has been done in Kentucky and Arkansas that the tonnages given for these states are thought not to be close; they are regarded, however, as ultra-conservative.

The estimates given below are for rock grading from 58 to 78 per cent "bone phosphate of lime." In the case of the low-grade phosphate in the West and the wash heaps of Florida, the material is figured to its equivalent in high-grade rock.

	Tons
Utah, Idaho, Wyoming and Montana:	
High-grade.....	2,500,000,000
High-grade equivalent of all grades.....	7,500,000,000
Florida:	
High-grade equivalent of all grades.....	354,300,000
High-grade equivalent of wash heaps.....	20,000,000
Tennessee:	
High-grade equivalent of all grades.....	115,075,000
South Carolina:	
High-grade equivalent of all grades.....	10,000,000
Arkansas:	
High-grade equivalent of all grades.....	20,000,000
Kentucky:	
High-grade equivalent of all grades.....	500,000
Total.....	10,519,875,000

The production of phosphate in 1912, according to the figures of the United States Geological Survey, was as follows:

	Tons
Florida.....	2,406,899
Tennessee.....	423,331(a)
South Carolina.....	131,490
Western states.....	11,612(b)
Total.....	2,973,332

(a) Includes production of Arkansas.

(b) Includes Utah, Idaho and Wyoming.

Even assuming that there are to be no new discoveries in this country and that the average consumption during the life of the phosphate fields will be three times its present consumption, there is sufficient to last for over 1100 years provided proper mining methods are employed and means for utilizing the lower grade material are devised.

Another feature of the phosphate industry which in recent years has become of considerable interest to the operator, the fertilizer manufacturer, and the farmer, is the growing use of raw ground rock phosphate for direct application to the field. While the

value of this material as a fertilizer still seems to be an open question, many agronomists and agricultural chemists strongly recommend its use, supporting their recommendations by considerable data obtained by actual plot tests. In 1912 the annual consumption of raw ground phosphate rock, based on the amount marketed, was 48,365 tons. While this is less than 1.29 per cent of the total phosphate produced and only 4.01 per cent of that marketed in the United States, the sale of ground rock is becoming quite a factor in the phosphate industry.

BUREAU OF SOILS, WASHINGTON

DETERMINATION OF CARBON IN STEEL AND IRON BY THE BARIUM CARBONATE TITRATION METHOD¹

By J. R. CAIN

The disadvantages attending the use of weighed absorption tubes as means for accurately determining carbon dioxide obtained during the combustion of steels and irons are in part as follows:

1—The elaborate precautions required to prevent change of weight of the tube due to gain or loss of moisture, necessitating complications in the purifying train before and after the furnace and the use throughout the apparatus of drying agents of the same hygroscopic power.

2—Difficulties in weighing large glass vessels caused by electrical effects in wiping, by buoyancy and by changes in temperature between balance room and laboratory.

3—The necessity for maintaining constant conditions with respect to the atmosphere within the tube, requiring sometimes a long period of aspiration after the combustion is completed.

4—The liability to error from access of gases containing sulfur and chlorine, which may be formed during combustion of the metal or of the carbonaceous residue therefrom.

5—The difficulty of determining whether the increase in weight of the tube is due solely to carbon dioxide.

6—The time lost in waiting for absorption tubes to reach a condition of equilibrium before weighing.

Those who have used absorption tubes for work requiring a high degree of accuracy know that neglect of one or more of the precautions indicated above may easily occasion errors ranging from several tenths of a milligram to one or more milligrams. It is evident, too, that if the complicated purifying train used with an absorption tube gets out of order, or if the tube itself introduces error in some of the ways enumerated, it may often be a difficult matter to locate and correct the trouble. It is not surprising, therefore, that methods dispensing with the use of weighed potash bulbs, soda-lime tubes and the like are beginning to be used extensively by steel analysis. Of such methods the weighing of the carbon dioxide in the form of barium carbonate precipitate directly or after conversion into sulfate have been much used, the sulfate

method abroad,¹ and the carbonate method in this country. Thus, of 62 prominent American laboratories (representing manufacturers, consumers and testing laboratories) 32 used the potash bulb, 24 weighed the carbon as barium carbonate, 3 titrated the excess of barium hydroxide, 2 used soda-lime tubes and one weighed an absorption tube filled with barium hydroxide.² It is evident, in estimating the carbon dioxide by weighing the barium carbonate precipitate, or the sulfate obtained from it, that the difficulties mentioned above as peculiar to weighed absorption tubes, except number four, are eliminated or minimized; access of sulfur trioxide would still tend to cause high results.³ If, however, the barium carbonate is measured by filtering it off and titrating it against standard acid, due regard being had to proper conditions for filtration and washing, there is no likelihood of error from any of the causes enumerated. The principle of this method is described in most standard text-books on quantitative and volumetric analysis, but there seems to have been but little application in steel analysis. The purpose of this paper is to show the special suitability of this procedure for accurate and fairly rapid steel analysis, taking up in order the sources of error or difficulty and the means of avoiding or minimizing these, and finally giving the results obtained by a series of analyses of pure sugar and of Bureau of Standards analyzed irons and steels. Without further consideration it can be seen that the adoption of this method at once simplifies the purifying train required after the furnace; nothing at all likely to be present in the escaping gases can affect the results if we except finely divided oxides carried over mechanically as the result of a very violent combustion. No such oxides were noticed during this work or during the analyses of many other samples by different methods; if there is any reason to suspect their presence, a simple filter made by filling a U-tube with 20-mesh quartz, previously carefully washed with hydrochloric acid and water, will remove them.

The points involving sources of error which were investigated were: (1) completeness of absorption of the carbon dioxide, (2) amount of washing necessary to remove the excess of barium hydroxide, (3) solubility of barium carbonate in the wash water, (4) exclusion of extraneous alkaline substances. In addition there were devised (5) means for the rapid filtration and washing of the barium carbonate with exclusion of carbon dioxide from the air.

1—COMPLETENESS OF ABSORPTION—This was established by burning sugar in amounts giving approximately the weights of carbon dioxide obtained during steel analysis, comparing the percentages of carbon obtained with the theoretical. As a further check two 8-bulb Meyer tubes (shown in 1/10 size in Fig. 1)

¹ Bauer and Deiss, *Probenahme und Analyse von Eisen und Stahl*, 1912, p. 121.

² Data communicated to the author as member of a technical committee on steel analysis.

³ There is but little likelihood of SO₂ being produced in burning steel and iron. Any small amounts of BaSO₃ resulting thereby would be removed by washing (see Sec. 5 on Filtration and Washing), the solubility of BaSO₃ being approximately 0.02 gram per 100 cc. of water at 20° (Seidell, "Solubilities of Inorganic and Organic Substances," 1907).

were worked in series. So long as a moderate rate of bubbling was maintained no cloudiness was ever observed in the second tube. In this connection, McCoy and Tashiro¹ have shown that 1.0×10^{-7} grams of CO_2 can be recognized as a turbidity. If, however, the oxygen is passed too rapidly, particularly when burning steels containing more than 1 per cent carbon, some CO_2 may reach the second tube. The proper rate of gas current to retain all the CO_2 in the first tube is easily established by the operator after a few trials.

2—AMOUNT OF WASHING NECESSARY—Barium carbonate itself reacts alkaline to phenolphthalein, consequently this indicator cannot be used to show when all of the barium hydroxide has been removed. The practical question as to how much washing was necessary was decided by washing the barium carbonate obtained from the combustion of accurately weighed amounts of sugar until results agreeing with the theoretical, within a small experimental limit, were obtained. The amount of wash water thus determined was increased by 25 per cent. About 150 cc. wash water were usually found sufficient. The results are summarized in the recommendations later, under the head "Filtration and Washing of the Barium Carbonate."

3—SOLUBILITY OF BARIUM CARBONATE IN WASH WATER—The possible effect of the slight hydrolysis of the barium carbonate in causing low results by solvent action of the wash water was studied by comparing the results obtained when determining the barium carbonate from the combustion of the same weights of sugar, first by washing as above described, and then with twice and three times the amount of water recommended. The results showed that the error from hydrolysis is negligible for present purposes. (See table and footnotes *c* and *d*, p. 467.)

Holleman² found the solubility of freshly precipitated BaCO_3 in CO_2 -free water to be 1 part BaCO_3 in 64,070 parts water at 8.8° and 1 part in 45,566 parts water at 24.2° , or approximately 1 part in 50,000 at the temperature of the laboratory during this work, *i. e.*, 20° to 22° . On this basis 150 cc. wash water (see preceding section) would dissolve about 0.0003 gram $\text{BaCO}_3 = 0.000018$ gram carbon. This would be negligible for present purposes, and the amount dissolved would be really less than this because of the repression of solubility during the first washings by the barium hydroxide still present; also it is quite possible that in the rapid passage through the filter there has not been sufficient time for the wash water to become saturated with barium carbonate. Holleman's results are in good agreement with those of Bineau.³ Holleman called attention to the fact, also recorded by Bineau, that the presence of carbon dioxide in water appreciably raises the solubility of barium carbonate and Holleman explains in this way the solubility of 1 part in 14,137 parts of water observed by Fresenius, who left the carbonate in contact with water exposed to the air for several days.

¹ Orig. Communication, 8th Int. Congr. Appl. Chem., 1 (1912), 361.

² Z. physik. Chem., 12 (1893), 135.

³ Ann. chim. phys., [3] 51, 290.

4—POSSIBLE SOURCES OF EXTRANEEOUS ALKALINE SUBSTANCES—These are: (a) substances derived by action of water on the bottles containing the CO_2 -free water mentioned later; such action should be avoided by choosing a good quality of glass bottle; Jena glass containers were found satisfactory; (b) alkali carried over mechanically from soda-lime guard-tubes by the air used to force out CO_2 -free water; thick plugs of glass wool will remove this source of danger; (c) action of barium hydroxide on the walls of the Meyer tubes; these tubes should not give up alkaline substances to the standard acid after barium hydroxide has stood in them for $1/2$ hour and the tubes have then been thoroughly washed with alkali-free water; (d) action of barium hydroxide on filtering material; glass wool as a filling material is absolutely excluded on this ground; quartz has been found very satisfactory (see description of filtering apparatus); amphibole asbestos for the felt was found suitable from this standpoint as well as because of its resistance to attack by $N/10$ hydrochloric acid.

5—FILTRATION AND WASHING OF THE BARIUM CARBONATE—This is carried out with the apparatus shown in the figure. The cut is approximately one-tenth size

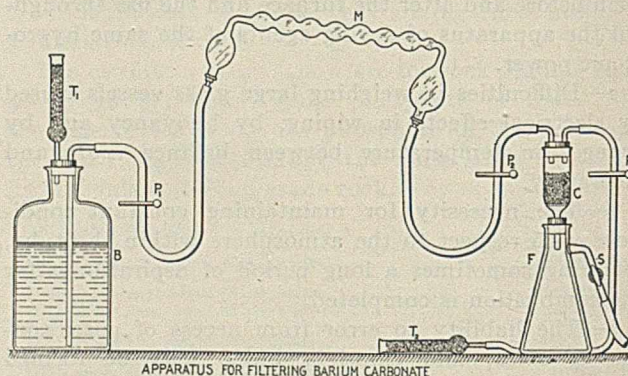


FIG. I

and is self-explanatory. *S* is a two-way stopcock connected to the suction pipe. The rubber tubing connected to the Meyer tube should be of best grade black rubber, and the lengths used should be chosen so as to permit of easy manipulation of the tube. The Meyer tube is connected or disconnected by the rubber stoppers which are left always attached to the rubber tubes. The carbon filter *C* is fitted with a perforated porcelain plate, sliding easily. The funnel is prepared for filtrations by making a felt of asbestos on the porcelain disc, using asbestos which has been digested for several hours with strong hydrochloric acid and then washed free of acid. On top of the asbestos is placed a layer of similarly washed quartz of the height shown in the figure. A mixture of grains of various sizes (approximately 50 per cent passing a 20-mesh and the remainder passing a 10-mesh and remaining on a 20-mesh sieve) is suitable. A mixture of quartz and asbestos works well and may be simply obtained by filling the funnel with a suspension of asbestos and delivering the quartz to the funnel from a beaker by means of a strong jet of water from the wash bottle, while maintaining a gentle suction. In this way the asbestos is properly mixed with the

quartz. Proper attention to these details will be found to greatly expedite filtration. The stopper is now inserted in the funnel, the Meyer tube connected as shown, and the liquid and precipitate sucked into the funnel. Only very gentle suction should be used. When necessary, P_3 is opened to admit air back of the column of liquid in the Meyer tube. When the contents of the tube have all been transferred, the large bulb nearest B is half-filled with water by opening P_1 ; the stopcock S is operated during this and subsequent operations so as to maintain a gentle suction. M is now manipulated so as to bring the wash water in contact with all parts of the interior, after which the water is sucked out through C ; P_2 is left open during this and subsequent washings. After eight washings, as directed, allowing the wash water to drain off thoroughly each time before adding more, M may be detached, the stopper removed from the funnel and the washing completed by filling C to the top with CO_2 -free water, sucking off completely and repeating the operation once. Air is now admitted through the side opening of S , C is removed and the porcelain disc carrying the quartz asbestos and barium carbonate is shoved, by means of a long glass rod, into the flask used for titrations, removing from the sides of C any adhering particles by a jet of water from the wash bottle.

APPARATUS FOR COMBUSTION

APPARATUS—The method requires no modification of any of the accepted appliances for the combustion of iron and steel. In the present work both gas and electrically heated furnaces were used. As already stated, there was no purifying train after the furnace, the Meyer tube being directly attached. Before the furnace was an electrically heated porcelain tube filled with copper oxide; then a calcium chloride tower filled with stick potassium hydroxide. Steels and irons were burned on a bed of alkali- and carbon-free alundum contained in a platinum boat. The blanks obtained by carrying through a complete determination, including filtration, washing, etc., but with omission of any carbon-containing substance, were usually 0.0 cc. and never more than 0.05 cc. of $N/10$ hydrochloric acid, showing not only that the oxygen was sufficiently purified and the apparatus in good condition, but that the operations of filtration, washing, etc., introduced no appreciable positive error.

SOLUTIONS USED

TENTH-NORMAL HYDROCHLORIC ACID—Standardized by any of the accepted methods, or as follows: 20 cc. of the approximately $N/10$ acid are measured out with a pipette, 5 cc. of nitric acid (1 to 1 by volume) added, and the silver chloride precipitated by an excess of silver nitrate solution in a volume of 50 to 60 cc. After digesting at 70° to 80° until the supernatant liquid is clear, the chloride is filtered off on a tared Gooch filter and washed with water containing 2 cc. of nitric acid per 100 cc. of water until freed from silver. After drying to constant weight at 130° , the increase of weight over the original tare is noted and the strength of the hydrochloric acid calculated on the basis of the weight of silver chloride thus ob-

tained, afterwards adjusting to the strength prescribed. Several concordant determinations with varying amounts of acid should be made. 1 cc. $N/10$ $\text{HCl} = 0.0006$ gram carbon.

TENTH-NORMAL SODIUM HYDROXIDE SOLUTION—Standardized against the hydrochloric acid solution, with methyl orange as indicator. This solution is conveniently stored in a large glass bottle fitted with a soda-lime guard tube and arranged for delivering the solution by air pressure.

METHYL ORANGE—0.02 gram dissolved in 100 cc. of hot water and filtered.

BARIUM HYDROXIDE SOLUTION—A saturated solution filtered and stored in a large reservoir from which it is delivered by air pressure, protecting from carbon dioxide by a soda-lime tube. Three or four small bulbs of the Meyer tube are filled, and CO_2 -free water is added until the remaining small bulbs are filled. When burning products high in carbon the stock solution may be used undiluted.

CARBON DIOXIDE-FREE WATER—This is conveniently made by passing air for a sufficient length of time through a soda-lime tube and into a 6- or 8-liter bottle filled with pure distilled water. The water is delivered by CO_2 -free air under pressure.

THE METHOD

The combustion is carried out in the usual manner, care being taken not to pass the oxygen too rapidly. After filtering, washing and transferring the contents of the filter to a flask, as described under "Filtration and Washing of the Barium Carbonate," a slight excess of the standard acid is added from a burette, using a portion to rinse out the Meyer tube, and the excess of acid is then titrated against the sodium hydroxide, using methyl orange as indicator.

RESULTS OBTAINED BY BARIUM CARBONATE TITRATION METHOD

Material	Weight Gram	Carbon present Gram	Carbon found Gram	Difference Gram
Sugar(a)	0.0100	0.00421	0.00427(c)	+0.00006
	0.0100	0.00421	0.00420	-0.00001
	0.0100	0.00421	0.00430	+0.00010
	0.0200	0.00842	0.00840	-0.00002
	0.0200	0.00842	0.00860	+0.00018
	0.0200	0.00842	0.00860	+0.00018
	0.0300	0.01263	0.01280	+0.00017
	0.0300	0.01263	0.01280	+0.00017
Bessemer Steel B. S., No. 23...	1.000	0.00805(b)	0.00805	0.0000
	1.000	0.00805	0.00805	0.0000
	1.000	0.00895	0.00805	0.0000
Bessemer Steel, B. S., No. 10b..	1.000	0.00373(b)	0.00372	-0.00001
	1.000	0.00373	0.00372	-0.00001
	1.000	0.02726(b)	0.02710	-0.00016
Pig Iron C, B. S., No. 5b.....	1.000	0.02726	0.02710	-0.00016
	1.000	0.02726	0.02710	-0.00016
Mean error, sugar.....				+0.000094
Mean error, steels and iron.....				-0.000056

(a) B. S. Standard Sample No. 17. A 1% aqueous solution was made and the required amounts were delivered into a porcelain boat from a burette. After careful evaporation of the water the sample was burned.

(b) Certificate values.

(c) Washed with double the usual amount of wash water. The second portion of wash water was titrated against the $N/10$ hydrochloric acid; the amounts used after deducting the blank when titrating the same quantity of the water used for washing were 0.10 cc. and 0.15 cc.

(d) Washed with three times the usual amount of wash water.

The results given in the table show that the method is as accurate as the weighing methods. It is not so subject to disturbing influences and requires less elaborate apparatus than those methods do.

NOTES AND PRECAUTIONS

1—After a little practice a precipitate can be filtered and prepared for titration in five minutes.

2—When working with steels high in carbon (above 1%) it is advisable not to use more than one gram, in order that filtration may be sufficiently rapid.

3—For very accurate work the Meyer tubes should be washed with dilute acid before beginning work each day. After a determination is finished, the Meyer tube should be completely filled two or three times with tap water, then rinsed with distilled water, in order to remove the carbon dioxide liberated when dissolving the carbonate from the previous determination.

4—The flask containing the carbonate should be very thoroughly agitated after adding the acid, since the carbonate sometimes dissolves rather slowly if this is not done; this is particularly the case if it has packed much during filtration.

5—The rubber tube connecting *B* (see figure) to the Meyer tube should be washed with a little water from *B*, before beginning determinations each day.

I am indebted to Mr. H. L. Cleaves of this Bureau who prepared the drawing of the filtering apparatus and made many determinations on steels which will appear in a later publication.

BUREAU OF STANDARDS
WASHINGTON

THE DETERMINATION OF AMMONIA IN ILLUMINATING GAS¹

By J. D. EDWARDS

Received March 21, 1914

This paper is a summary of the results of a brief investigation of the apparatus and methods employed for the commercial determination of ammonia in illuminating gas. A fuller report will be found in Bureau of Standards' *Technologic Paper No. 34*, and the application of this work in the form of operating directions for carrying out the determination will be included in Bureau of Standards' *Circular No. 48*, "Standard Methods of Gas Testing."

The method generally used for the determination of ammonia in purified illuminating gas depends upon the absorption of the ammonia in a standard acid solution, the amount of ammonia absorbed from a measured volume of gas being determined either by titration of the acid remaining unneutralized, or less frequently by allowing the gas to pass until the change in the indicator used shows that all the acid has been neutralized.

The choice of the proper indicator to use for this determination is of greater importance than the choice of apparatus, since the use of an unsuitable indicator may introduce large errors, amounting in extreme cases to more than one hundred per cent. Many indicators have been and still are commonly used which are not at all suited to the purpose, failing to meet one or more of the following essential requirements:

1—It should show a sharp end point in dilute solutions.

2—It should be sensitive to ammonia and not be seriously affected by ammonium salts.

3—The end point should be affected as little as possible by carbon dioxide.

¹ Published by permission of Director, Bureau of Standards. Abstract of paper presented at the Cincinnati Meeting of the American Chemical Society, April 6-10, 1914.

The indicators which were found to be most suitable for the determination of ammonia in gas were sodium alizarinsulfonate, cochineal and paranitrophenol. Sodium alizarinsulfonate is less sensitive to carbon dioxide than either cochineal or paranitrophenol and gives a color change from greenish yellow to brown which is quite sharp even with very dilute solutions. Sodium alizarinsulfonate was the indicator used in the remaining experimental work. Methyl orange, though less sensitive to carbon dioxide than the above indicators, does not give sufficiently sharp color changes with weak solutions. Phenolphthalein and litmus, of course, are too sensitive to carbon dioxide to be of use here.

The presence of glass beads which are used in some of the absorption apparatus may lead to erroneous results for two reasons: (1) The beads may yield alkali on contact with the absorbing liquid; (2) washing of the beads may be incomplete. Although the absolute amount of alkali which might be dissolved from the beads is small, it may be equivalent to a considerable percentage of the total amount of ammonia to be absorbed. For this reason, it is recommended that the operator test the solubility of any beads he may use. The method of washing out the apparatus should also be tested to insure complete washing with a minimum of wash water.

Five different forms of apparatus were tested: The Referees apparatus, the Emmerling tower, the Lacey apparatus, the common form of gas wash bottle and a modified form of the Cumming wash bottle. This latter form (Fig. 1) was designed at the Bureau

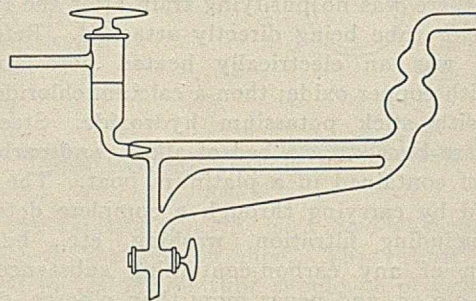


FIG. 1—MODIFIED CUMMING WASH BOTTLE (ONE-FOURTH SIZE)

of Standards for this work. In this form, the gas passing through the small nozzle acts like an injector and circulates the liquid rapidly and continuously, thus bringing fresh acid into contact with the gas.

The relative efficiency and from this the probable accuracy of the different forms of apparatus was determined by running the different forms in parallel, using gas from a common supply. A *N*/50 solution of sulfuric acid was used as the absorbing agent. It was not considered desirable to use a stronger solution than this because of the small amount of ammonia to be absorbed and the fact that small errors in the measurement of stronger solutions make a large error in the ammonia apparently absorbed.

As a result of this comparison it was found that the Emmerling tower gave results which were somewhat higher than those obtained with the other forms and that the wash bottle gave results consistently lower.

In a series of 10 tests, in each of which four different apparatus were run in parallel, the maximum difference between the high and low results of each test (expressed as a percentage of the average) was 8.3 per cent and the average difference 4.5 per cent. The ammonia content of the gas was varied from 1 to 20 grains per 100 cubic feet. (For full experimental details, see *Technologic Paper* No. 34, above referred to.)

A series of tests in which the gas after passing through the first absorption apparatus passed through an Emmerling tower, showed, in general, that the amounts of ammonia escaping the first apparatus but absorbed in the Emmerling tower were not detectable within the limits of error of the titration.

With careful operation, any one of the five forms of apparatus tested would ordinarily give results that are well within the limits of accuracy required for this determination, either for commercial control work or for the purpose of gas inspection. The wash bottle appears to be slightly less efficient as an absorber than the other forms, but still it gives satisfactory results. It is difficult to make any comprehensive statement defining satisfactory results. In general, results on gas containing up to about 5 grains of ammonia per 100 cubic feet should be within 10 per cent of the ammonia content of the gas, although the percentage error may be greater when the ammonia is 1 grain or lower. With gas containing more than 5 grains ammonia, the percentage error should decrease somewhat.

BUREAU OF STANDARDS, WASHINGTON

THE STUDY OF THE CRUDE PETROLEUM FROM BIORITSU, FORMOSA

By TETSU. KATAYAMA

Received April 1, 1914

Favorable indications of petroleum deposits have been found in Formosa Island in many districts along the western slopes of the middle mountain range. Of these oil deposits that at Shukkokosio, about seven miles south of Bioritsu station on the main railway line in the island, is the most important, and is now worked by the Howden Petroleum Company. It is reported that about 3,000 gallons of crude petroleum are obtained daily from a single well there.

Several years ago the author studied the physical properties of this crude petroleum and found that the kerosene oil obtained from the crude petroleum by the ordinary method can not be used for lamp oil, the flame being very smoky. Further study had to be postponed, but in May, 1913, it was again resumed. By means of acetic anhydride, the smoky constituent was isolated and its nature studied, and it was at last found that the kerosene oil may be refined by changing the temperature and duration of the reaction on ordinary sulfuric acid treatment. The results thus obtained are submitted below.

I—GENERAL PROPERTIES OF THE CRUDE PETROLEUM

The crude petroleum is a clear, mobile, brownish oil of specific gravity 0.831 at 15° C.; its smell is rather agreeable; it solidifies at 0° C.

FRACTIONAL DISTILLATION—300 cc. of the crude petroleum were introduced into a side tube distilling flask of about 500 cc. capacity, and distilled, the heat being so regulated that about 2.5 cc. of distillate passed over per minute. The average results of duplicate distillations are the following:

Fractions °C.	Per cent by volume	Sp. gr. at 15° C.
below 100.....	1.5
100-125.....	15.8	0.7770
125-150.....	21.8	0.7980
150-175.....	12.2	0.8155
175-200.....	8.8	0.8205
200-225.....	7.8	0.8360
225-250.....	7.6	0.8585
250-275.....	7.5	0.8790
275-300.....	4.3	
Residue.....	13.2	

Solidifies at ordinary temperature

It is remarkable that the specific gravity of each fraction of this crude petroleum was very high—much higher than that of the corresponding fraction of other crude petroleum.

BURNING TEST—The kerosene fraction, distilling between 150° C. and 275° C., was refined with concentrated sulfuric acid by the ordinary method, washed with water several times, then with a dilute alkali solution, and again with water until the oil became free from acid and alkali. The kerosene oil thus refined was burned in a lamp of ordinary construction, but the flame was very smoky.

TEST FOR NITROGEN AND SULFUR—The crude petroleum was heated to boiling for a long time with some small pieces of metallic sodium, in a flask equipped with a reflux condenser. After cooling, the sodium was tested for cyanocompounds and sodium sulfide, but no test for either of these compounds was obtained.

II—SOLUBILITY OF THE CONSTITUENTS OF THE CRUDE PETROLEUM

At first an attempt was made to isolate the smoky constituent in the petroleum by distillation, but it was impossible. Every fraction separated by cautious fractional distillation gave a smoky flame. Then an attempt was made to separate the smoky constituent by a solvent. Edeleanu suggested that aromatic compounds and other hydrocarbons, rich in carbon atoms, may be dissolved away from the oil by liquefied sulfur dioxide, and Prof. Engler recommended this method as effective.¹ It was considered desirable to try this method, but the temperature of the laboratory at that time, always above 30° C., did not permit of this procedure. Therefore another solvent, which would extract the smoky constituent from the petroleum at ordinary temperature, was sought, and at last it was found that acetic anhydride was most suitable for this purpose.

EXPERIMENT 1—250 cc. of the fraction distilling between 150° C. and 275° C. were introduced into a separating funnel and acetic anhydride was added, little by little, shaking well between each addition. At the beginning the added acid dissolved in the petroleum, but when 60 cc. of acid had been added the fluid separated into two layers, the lower layer being 9 cc. After the lower layer was drawn off, 10 cc. more of the acid were added to the upper layer, when 27 cc. separated as a lower layer. The lower layer was drawn

¹ *Ztschr. angew. Chem.*, 1913, p. 177.

off and added to the formerly separated fluid. These operations were repeated until 200 cc. of acid had been used. Then the total fluid drawn off was 295 cc., while the residual upper layer was 155 cc. Both the fluids were washed well with water until they became free from acid, when the oil in the upper layer was 140 cc. and the oil in the tapped fluid was 110 cc.; that is to say, the upper layer contained 140 cc. of oil and 15 cc. of the acid, while the lower layer contained 110 cc. of oil and 185 cc. of the acid. These results showed that the petroleum contains two different constituents, one immiscible with acetic anhydride and the other easily soluble in acetic anhydride. Their specific gravities at 30° C. are:

The fraction before the treatment.....	0.830
The immiscible oil.....	0.804
The oil easily soluble in acetic anhydride.....	0.864

It might be thought that these two constituents would have different boiling points, but the distillations of these constituents showed that their boiling points were the same.

The burning quality of these two constituents was next tested; the oil with the lower specific gravity burned brilliantly while the other gave a very smoky flame.

From the above experiment it was evident that the constituent which gives a smoky flame on burning may be separated from the petroleum by this difference of solubility in acetic anhydride. This separation depends, of course, upon the difference of solubility in acetic anhydride; it varies according to the quantity of the added acetic anhydride, and the method of adding. Though it was, of course, impossible to separate these constituents from each other absolutely, the division was sufficiently complete for practical purposes.

EXPERIMENT 2—The above experiment was repeated for the crude petroleum and every fraction of it. To 100 cc. of each fraction of the oil, acetic anhydride was added little by little, and the quantity of the acid used when the mixture just separated into two layers was noted. When the added acetic anhydride reached 100 cc. the quantities of the two layers, and the quantities and specific gravities of the oils contained in these two layers were determined.

SEPARATION OF THE OIL AND ITS FRACTIONS BY ADDING 100 CC. OF ACETIC ANHYDRIDE

Fractions Temp. in ° C.	Sp. gr. original oil at 30° C.	Acid added when layers formed Cc.	UPPER LAYER			LOWER LAYER		
			Total vol. Cc.	Oil in cc.	Sp. gr. oil at 30° C.	Total vol. Cc.	Oil in cc.	Sp. gr. oil at 30° C.
Crude petro- leum.....	0.8215	..	67.5	63.0	0.804	132.5	37.0	0.8515
Residue								
above 250.....	0.877	..	86.0	80.0	114.0	20.0
225-250.....	0.848	13.3	83.0	75.5	0.823	117.0	24.5	0.920
200-225.....	0.824	15.0	75.0	68.0	0.805	125.0	32.0	0.880
175-200.....	0.809	30.0	70.0	60.0	0.796	130.0	40.0	0.832
150-175.....	0.795	66.0	46.0	36.0	0.788	154.0	64.0	0.798

Lower fractions were more soluble in acetic anhydride than higher fractions, and fractions boiling below 150° C. gave no separations into layers when large amounts of acetic anhydride were added. The constituents in these fractions could be separated by dissolving in acetic anhydride and adding water, drop by drop. The fluid separated at first into two layers, then

into three layers, of which the lowest did not contain oil; the middle layer contained the smoky constituent and most of the acid, while the upper contained the non-smoky constituent and a little acid.

III—PROPERTIES OF THE SMOKY CONSTITUENT

The fraction boiling between 150° and 250° C. was mixed with half its volume of acetic anhydride and separated into its two constituents by the above described method; the oil obtained from the upper layer burned brilliantly, while the oil from the lower layer burned with a very smoky flame.

	Original fraction	Smoky constituent	Non-smoky constituent
Sp. gr. at 15° C.....	0.830	0.855	0.822
Refractive index at 20° C. (Abbé).....	1.4932	1.4437
Iodine number (Hübl).....	0.76	0.37

The low iodine numbers showed the comparative absence of open chain unsaturated compounds.

NITRATION TEST.—Concentrated nitric acid acts upon all the unsaturated compounds at a low temperature. A mixture of 20 grams of sulfuric acid (sp. gr. 1.84) and 16 grams of nitric acid (sp. gr. 1.375) was introduced into a small graduated cylinder equipped with a glass stopper: 10 cc. of the oil were added and mixed slowly, avoiding rise of temperature by cooling with water. After standing, the fluid separated into three layers: the upper layer contained non-reacted oil and a small quantity of nitrated substance; the middle layer consisted of nitrated oil and acid mixture; the lower layer consisted of the acid mixture and a little nitrated substance. The upper layer was separated, washed well with water and dilute alkali solution, and measured; this oil contained a small quantity (2-3 per cent?) of nitrated substance which was considered practically negligible. This volume was subtracted from 10 cc. and the difference was recorded as the volume of the nitrated portion in the fraction.

Percentages	Fraction before separation	Smoky constituent	Non-smoky constituent
Nitrated portion.....	42	73	36
Non-nitrated portion.....	58	27	64

The smoky constituent contained much nitrifiable portion. The specific gravities of both the non-nitrated portions obtained from the smoky constituent and the non-smoky constituent were 0.804 at 30° C. and both the non-nitrated portions burned brilliantly in an ordinary lamp. From these results, it was concluded that the nitrifiable oil was the cause of the smoky burning.

The nitrated portion obtained from the fraction boiling below 115° C. was reduced with hydrochloric acid, and metallic tin and excess of an alkali solution were added to it and distilled. A colorless oily substance with anilin-like odor was obtained. This substance was slightly soluble in water; the solution turned violet gradually on being exposed to air; the same change of color occurred also when bleaching powder solution was added; this substance formed a difficultly soluble crystalline salt with dilute sulfuric acid. These facts indicated that the substance contained aniline and its higher homologues.

REACTION WITH PICRIC ACID—Picric acid forms crystalline compounds with many of aromatic com-

pounds: 12 g. of picric acid were dissolved in 100 cc. of the fraction, warmed. After one day the fraction became filled with yellow crystals.

CHEMICAL COMPOSITION—The smoky constituent was treated with acetic anhydride and a comparatively insoluble portion separated from it. After repeating this operation several times a pure smoky constituent was obtained which yielded no non-nitrated portion on nitration. Similarly a concentrated non-smoky constituent was obtained which yielded 10 per cent nitrated portion on nitration. Combustions of the two constituents gave the following results:

Constituent	Per cent C	Per cent H	Ratio of C to H
Pure smoky.....	88.98	10.75	1:1.44
Concentrated non-smoky.....	85.43	14.34	1:2.00

The former is rich in carbon atoms. Being a mixture of compounds boiling between 150° C. and 250° C., its molecular formula can not be determined by the above composition, but it is supposed from the boiling point that the number of carbon atoms is between 9 and 15. If these numbers are applied to $C_1H_{1.44}$, it becomes $C_9H_{12.96}$ — $C_{15}H_{21.6}$, *i. e.*, $C_nH_{2n-5.04}$ — $C_nH_{2n-8.4}$. These formulas are richer in carbon atoms than paraffins, naphthenes, or olefins: they correspond to aromatic compounds. The above formulas indicate that the non-smoky constituent is composed of paraffins or naphthenes.

From the above described experiments it was concluded that the nitrated portion consisted of aromatic compounds, and about 42 per cent by volume of the kerosene fraction was nitrifiable. On that account the kerosene fraction was richer in carbon atoms than an ordinary kerosene oil from another origin, and burned incompletely in an ordinary lamp.

IV—REFINING THE KEROSENE OIL

In the previous paragraph it was concluded that the smoky burning of this oil was due to the presence of large amounts of aromatic compounds. Though this smoky constituent could be separated by acetic anhydride as described in Section II, this method could not be used economically for the commercial refining of burning oil, on account of the high price of acetic anhydride. Accordingly, another refining method for this oil was sought. On ordinary sulfuric acid treatment, the quantity of the acid and temperature and duration of the treatment were changed variously.

EXPERIMENT 3—100 cc. of the kerosene fraction boiling between 150° C. and 250° C. were mixed with 100 cc. of concentrated sulfuric acid (sp. gr. 1.84), stirred with an agitator connected to a water turbine. In the course of time the acid layer absorbed some portion of the oil, and increased gradually in quantity. After agitating for twenty hours, the oil layer was separated, washed well with water and a dilute alkali solution. The specific gravity of the oil decreased from 0.825 to 0.791, and the oil burned brilliantly. Next the kerosene fraction was treated with smaller quantities of the acid, and the results obtained follow.

The nitrifiable portion in the following table was determined by the method described above.

Treatment of kerosene agitated		Sp. gr. of treated oil at 30° C.	Vol. of acid layer formed	Nitrifiable portion in treated oil Per cent	Refractive index of treated oil at 20° C.	CHARACTER OF FLAME
with H_2SO_4 Cc.	for hrs.					
100	20	0.825	..	42.0	1.4705	Very smoky
75	20	0.797	125.4	12.0	1.4468	Very good
		0.797	98.0	15.0		
50	20	..	67.0	19.0	..	Very good
25	20	0.803	44.0	23.0	..	Good
10	20	0.821	20.0	Not smoky but easily undulated
25	5	0.808	43.0	23.0	1.4577	Good
10	5	0.821	17.0	35.0	..	Not smoky but easily undulated
Repeated last treatment		28.0	1.4682	Good

From the above table it was evident that there was no difference between the reactions for 20 hours and 5 hours, and that on the reciprocal proportion of the acid used, specific gravities, refractive indices and nitrifiable portions of the treated oil gradually decrease.

It was also seen that when less sulfuric acid than 25 cc. was used, the nitrifiable portion in the treated oil decreased in the ratio of about 7 cc. for 10 cc. of sulfuric acid used, and in the same ratio the acid layer absorbed the oil. When more sulfuric acid was used, the ratio decreased.

EXPERIMENT 4—100 cc. of the kerosene fraction were introduced into a flask provided with a reflux condenser, and mixed with various quantities of concentrated sulfuric acid (sp. gr. 1.84). The mixture, warmed on a water bath, was agitated for five hours by a dry air current. After treatment, the oil was separated from the acid layer, and washed. The oils thus obtained were yellow in color; their quantities are tabulated below:

Sulfuric acid used Cc.	Acid layer separated Cc.	Nitrifiable portion in oil Per cent	CHARACTER OF FLAME
Original oil	..	42	Very smoky
20	44	18	Very good
15	33	24	Very good
10	22	30	Very good
5	11	36	Good
3	7	38	Not smoky but easily undulated

From the above it was clear that sulfuric acid acts more effectively at 100° C. than at ordinary temperature. The increase of acid layer by the treatment was proportional to the quantity of sulfuric acid used. For 10 cc. of sulfuric acid used, the increase of acid layer was 12 cc., and the decrease of nitrifiable portion in the treated oil was also 12 cc. From this result, it was concluded that the nitrifiable portion by which the kerosene fraction was contaminated, was absorbed by the acid in almost definite proportions. The sulfuric acid acted upon nitrifiable oil in the kerosene fraction 1.7 (12/7) times more effectively at 100° C. than at ordinary temperature.

If the kerosene fraction was treated by sulfuric acid at 100° C. for five hours, a usable oil was obtained by 5 per cent of sulfuric acid, and the more sulfuric acid used, the better the oil obtained.

Afterwards two samples of new crude petroleum were obtained and tested as to solubility in acetic anhydride: the nitrifiable portions were determined in them by the method described in this paper and it was found that the above methods were applicable for the testing of oil for the presence of compounds rich in carbon.

THERMAL REACTIONS IN CARBURETING WATER GAS PART II—EXPERIMENTAL

By M. C. WHITAKER AND W. F. RITTMAN

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In the design of an experimental apparatus for cracking oil in accordance with the theory set forth in THIS JOURNAL, 6, 383, it is necessary to provide accurate control over the three variables: temperature, pressure, and concentration. The plan of research has been: (1) To keep pressure and concentration constant until the effect of changing temperature is understood; (2) to keep the temperature constant and change the total pressure on the system; (3) to hold both temperature and pressure constant and crack the oil in the presence of other gases in order to vary the concentrations.

Considerable time was spent in designing and building an apparatus which would be stable, durable, easy of access and replaceable in all its parts, as well

brand wrought iron pipe, 32 in. long. For a length of 18 in. the pipe is wrapped with No. 15 Nichrome resistance wire, seven turns to the inch. Between the wrought iron pipe and resistance wire five layers of asbestos paper serve as insulation. In series with the nichrome wire of the furnace is a large rheostat, with graduated steps between $2\frac{1}{2}$ and 9 amperes. An incandescent tell-tale lamp is connected across the binding posts of the furnace, to indicate when the current is on as well as to give a rough idea of the wattage in use. Both voltmeter and ammeter are connected in the circuit. (See Fig. I.)

The nichrome wire windings are enclosed in a five-inch insulation of magnesia-asbestos pipe covering to minimize radiation. A $\frac{3}{4}$ in. wrought iron pipe is welded at right angles into the $1\frac{1}{2}$ in. wrought iron furnace body to serve as a container for the pyrometer point. This side tube is likewise insulated with asbestos and is fitted with a stuffing box surrounding the

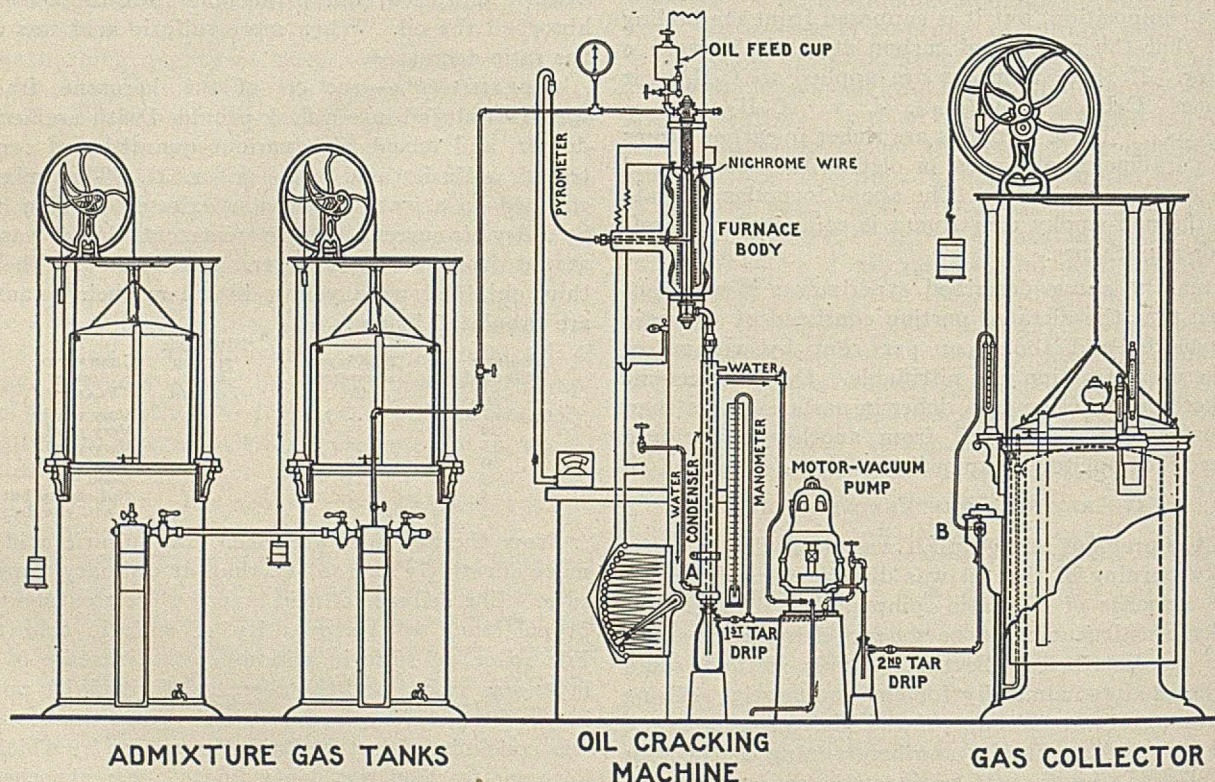


FIG. I—APPARATUS FOR OIL CRACKING AND GAS MAKING

as under complete control with respect to temperature, pressure and concentration. The machine was further designed to be of such dimensions and capacity as would indicate results which might be expected in the commercial application of the principles involved. In its completed form the apparatus covers a floor space sixteen feet by four feet and the oil feed cup at the top of the machine is nine feet from the floor. With this equipment it is possible to maintain any temperature up to 1000° C. within five degrees, and any pressure ranging from one-thirtieth of one atmosphere absolute to three atmospheres absolute.

FURNACE BODY

The furnace body is made from $1\frac{1}{2}$ in. "Reading"

pyrometer rod. With this side tube it is possible to keep the pyrometer point directly in the furnace body at all times. The pyrometer couple is of the iron-nickel type connected with a millivoltmeter calibrated in degrees Centigrade.

After continued use at the higher temperatures the furnace body warps or the nichrome wire burns out. Duplicates for all parts are kept on hand, and the apparatus is so designed that any part may be replaced within a few minutes.

In order to vaporize the oil before it reaches the cracking zone, the upper part of the furnace tube is filled with $\frac{5}{8}$ in. steel balls. These are held in place by a thin post which runs vertically through the furnace supporting a perforated plate. The vertical rod

is bent to permit the centering required for the pyrometer. The object of the steel balls is to spread the oil in thin films and facilitate vaporization, but not to serve as cracking surface; in order to accomplish this they are kept at a safe height above the cracking zone. Lowering them into the cracking zone has a marked influence on the products obtained from the cracking process. The furnace, together with condenser, oil feed, pressure gauge and admixture gas-inlet pipe are vertically supported by iron clamps attached to an upright 3 in. \times 6 in. yellow pine timber. The assembled apparatus was tested at 100 lbs. hydraulic pressure.

OIL FEED

A Powell sight feed oil cup of one quart capacity is joined by a $\frac{3}{4}$ in. elbow and nipple to a $1\frac{1}{2} \times 1\frac{1}{2} \times \frac{3}{4} \times \frac{3}{4}$ in. cross which forms the upper end of the furnace body. The pressure in the oil cup is equalized through a small internal pipe which communicates with the furnace body below the point of oil discharge. As a result of this equalizing tube, regardless of whether the apparatus is under increased or reduced pressure, the oil supply is always under a supply pressure equal to its own head. As this head decreases the rate of flow may be regulated by the needle valve controlling the feed inlet. The rate of supply may be determined by counting the drops for a given time.

PRESSURE GAUGES

For all vacuum work the apparatus is connected with a mercury manometer calibrated in inches. A mechanical vacuum gauge is also placed at the top of the apparatus to indicate a free path in the cracking tube. In the course of the experiments under certain conditions, sufficient carbon was deposited to clog up the apparatus and show a considerable difference in the pressure between the two gauges. Such a condition, however, is limited to experiments involving high temperatures and pressures (atmospheric or greater), where the deposition of carbon is at its maximum. There is never any clogging under reduced pressures. For pressure work, the mercury column is disconnected and a mechanical pressure gauge is substituted.

CONDENSER

At the lower end, the generating tube or furnace body discharges through a Liebig type condenser into a tar drip for the collection of liquid condensates. The cooling water enters at the bottom of the condenser and on leaving continues through the jacket of the vacuum pump. The condenser pipe is offset from the furnace body rather than placed directly under it so that the furnace may be cleaned by simply removing the lower plug and withdrawing the contents. It is thus possible to remove and weigh the deposited carbon from the furnace body after each run.

TAR DRIPS

For vacuum work the tar drips are of glass, as this facilitates observation of the gas, as well as the nature and the rate of tar formation. In vacuum work it was soon found that the lighter condensates would con-

tinue through the vacuum pump because of the low pressure in the first tar drip. To collect the liquids drawn through, a second tar drip was placed beyond the vacuum pump. Upon reaching the second drip these lighter hydrocarbons condense, as the pressure is then approximately atmospheric. When working under one-thirtieth of an atmosphere it was found that only a small percentage of the hydrocarbons would collect in the first tar drip. For pressure work a steel tar collector was substituted.

VACUUM PUMP

Vacuum in the system is maintained by a May-Nelson two-ring vacuum pump. By means of a by-pass connection joining the outlet and inlet of the pump, it is possible to regulate the valve so as to maintain any desired vacuum down to one-thirtieth of an atmosphere.

CONNECTION FOR PRESSURE WORK

The vacuum pump is mounted on a movable concrete foundation and by disconnecting a few couplings the vacuum attachments may be removed. For pressure work there is substituted an all metal tar collector, connecting pipe, and release pressure valve, as shown in Fig. II. The apparatus may be changed from vacuum to pressure, or *vice versa*, in twenty minutes. When working under pressure, the gas

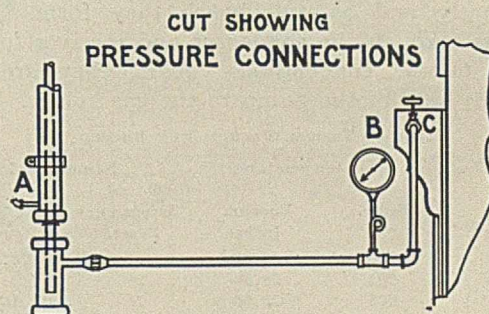


FIG. II

generated in the furnace body creates its own pressure. This pressure is controlled by an ordinary release valve placed at the inlet to the gas collector. By regulating the release of this valve, the apparatus may be set to work under any pressure from atmospheric to 30 lbs. per square inch above atmospheric, which seemed to be the upper safe working limit of the furnace under the conditions of operation. A pressure gauge is placed in the discharge line and used as a check on the pressure gauge near the oil feed.

GAS COLLECTOR AND ADMIXTURE GAS TANKS

The gas generated is collected in a 12 cu. ft. capacity gas holder, made by the American Meter Company. The tank is graduated in tenths of a cubic foot. By multiplying the number of cubic feet by 28.32 the volume is reported in liters. To avoid relying upon natural diffusion for mixing the gases, the bell of the holder is fitted with an internal mechanical stirrer directly connected through a stuffing box to an electric motor located on top of the bell. Perfect mixing of the gases may be attained in two minutes with this

equipment whereas natural diffusion would require from one to two hours.

The equipment also contains two 6 cu. ft. capacity admixture tanks of the same design as the large holder. These serve as gas supply tanks when cracking oil in the presence of other gases such as H₂, CO, or mixtures of the two. A 1/4 in. steel pipe, fitted with needle control valves, connects these two tanks with the inlet end of the furnace body. The piping is so arranged as to connect any two of the three tanks.

METHOD OF MAKING A RUN

When the apparatus is operated under vacuum the furnace body is first heated by the resistance coils to the desired temperature for cracking the oil into fixed gases. The oil is permitted to enter the upper part of the generating tube where it spreads over the steel balls and is vaporized. In the meantime, the vacuum pump has been set in operation and draws the oil vapors downward into the cracking zone of the furnace body, whereupon these vapors are immediately cracked into fixed gases and other products. These products, before opportunity is offered for polymerization or decomposition of the hydrocarbon gases, are withdrawn by the vacuum pump from the cracking zone and their place is taken by a quantity of oil vapor from the vaporizing zone above. In this manner the hydrocarbon gases are withdrawn continuously and as quickly as they form. After they pass through the condenser and receiver for the removal of the condensable vapors, they are forwarded by way of the pump to the gas holder.

TABLE I—TYPICAL VACUUM RUN RECORD

Time	Temp. ° C.	Vacuum Inches	Oil feed Drops in 10 sec.	Cu. ft. gas observed
11.43	900	29.00
12.00	903	28.50	24
12.15	905	28.00	26	2.16
12.30	895	28.75	23	3.01
12.45	895	28.75	21	3.76
1.00	895	28.50	24	4.86
1.30	895	28.50	26	6.86
1.45	895	28.50	25	7.76
2.00	895	28.75	17	8.36
2.07	Run ended			8.75

Corrected to standard conditions as follows:

$$8.75 \times \frac{273}{288} \times \frac{756}{760} \times 28.32 = 234 \text{ liters}$$

ANALYSIS OF PRODUCTS FROM TABLE I—GAS ANALYSIS

	Per cent by vol.	Liters at std. cond.
CO ₂	0.1
Ill.....	52.1	122.0
O ₂	0.9
CO.....	0.4
CH ₄	24.0	56.0
C ₂ H ₆	1.3	3.0
H ₂	17.3	40.0
N ₂ (difference).....	3.9
	100.0

Carbon, 3 grams.

"Tar" 60 cc. Sp. gr. 20° Bé.

GAS SAMPLING AND ANALYSIS

After the gas in the holder is thoroughly mixed by the mechanical stirrer, three or four sampling

tubes are filled for analysis. As a guide for all methods of gas analysis, Dennis' 1913 edition of "Gas Analysis," using the Hempel equipment, is followed. To determine the hydrogen in a mixture of hydrogen, methane and ethane, Hempel's fractional combustion method¹ is used. All analyses are made in duplicate.

DATA OBTAINED FROM A RUN

In this work all conditions outside of temperature, pressure, and concentration were maintained as uniform as possible. Four hundred cc. of oil were used per run, fed at the rate of about 3 cc. per minute. The oil used is technically known as "150° (F.) water white oil;" its specific gravity at 15° C. is 0.7984; boiling point between 150° and 290° C. Oil from the same tank was used throughout the experiments. All runs were made in duplicate.

EFFECT OF TEMPERATURE CHANGES

The experimental data on changes in temperature in the cracking of the oil, with pressure and concentration maintained constant, agree with the data recorded in the literature from the experiments by Haber,² Hempel,³ Ross and Leather,⁴ Lewes,⁵ Fulweiler,⁶ and others. It is difficult to make accurate comparisons of two experiments conducted in different apparatus on account of the great variety of conditions involved. Subjecting a gas to a temperature of 900° C. for five seconds is quite different from subjecting it to the same temperature for five minutes.⁷ Leading the gases through a 1/8 in. pipe heated to 900° C. would give different results from those obtained by leading the same gases through a 2 in. pipe heated to 900° C. While the difference due to experimental apparatus would not be so great as the examples cited, there is sufficient difference to affect the value of direct comparison.

TABLE II

Oil used, 400 cc.

Temp. ° C.	Press.	Gas liters Standard conditions	Carbon Grams	Tar Cc.
650	atmos.	135	3	163
750	atmos.	206	18	80
900	atmos.	382	115	11

Analysis of Gas from Table II

Temp. ° C.	C ₂ H ₆ Per cent	CH ₄ Per cent	H ₂ Per cent	Ill Per cent
650	10.2	33.7	9.1	43.6
750	4.9	41.1	19.2	30.6
900	None	46.7	38.6	13.1

However, the general results obtained at different temperatures by different experimenters are comparable. As the temperature increases, the quantity of valuable gas from a given amount of oil increases to a maximum, after which the gaseous hydrocarbons rapidly decrease and the deposited carbon increases.

¹ *Ztschr. angew. Chem.*, 1912, 1841.

² *Jour. f. Gasb.*, 1896, 799, 813, 830.

³ *Ibid.*, 1910, 53.

⁴ *Journal of Gas Lighting*, 1906, 825.

⁵ *Jour. Soc. Chem. Ind.*, 1892, 584.

⁶ Rogers' "Industrial Chemistry."

⁷ Lewes, *Proceedings of Royal Soc.*, 1894, 90; W. A. Bone, *Jour. f. Gasb.*, 1908, 803; Bone and Coward, *Jour. Chem. Soc.*, 1908, 1197.

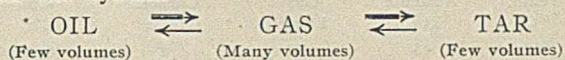
The quantity of tar decreases, but this is due to a dissociation of the hydrocarbons and cannot be construed favorably. Upon subjecting the oil to temperatures of 650°, 750° and 900° C., under atmospheric pressure, the results are as shown in Table II.

EFFECT OF INCREASED PRESSURE ON GASEOUS REACTIONS

It seems reasonable to expect that a high pressure

expand to many volumes of gas than under reduced pressure.

In the system



one would expect that the application of high pressures would increase the difficulty of generating gas, and after the gas is generated it would make easier

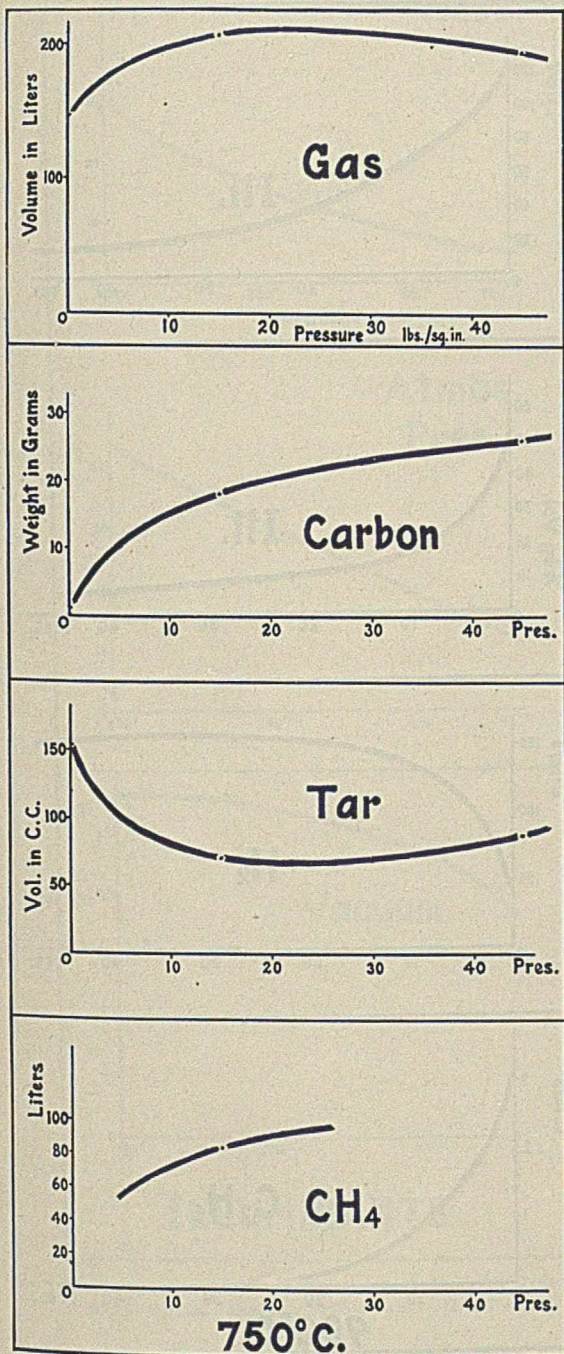


FIG. III—VARIATIONS IN YIELDS OF PRODUCTS AT 750° C., UNDER VARYING PRESSURES. (TABLES II, III, IV AND VI)

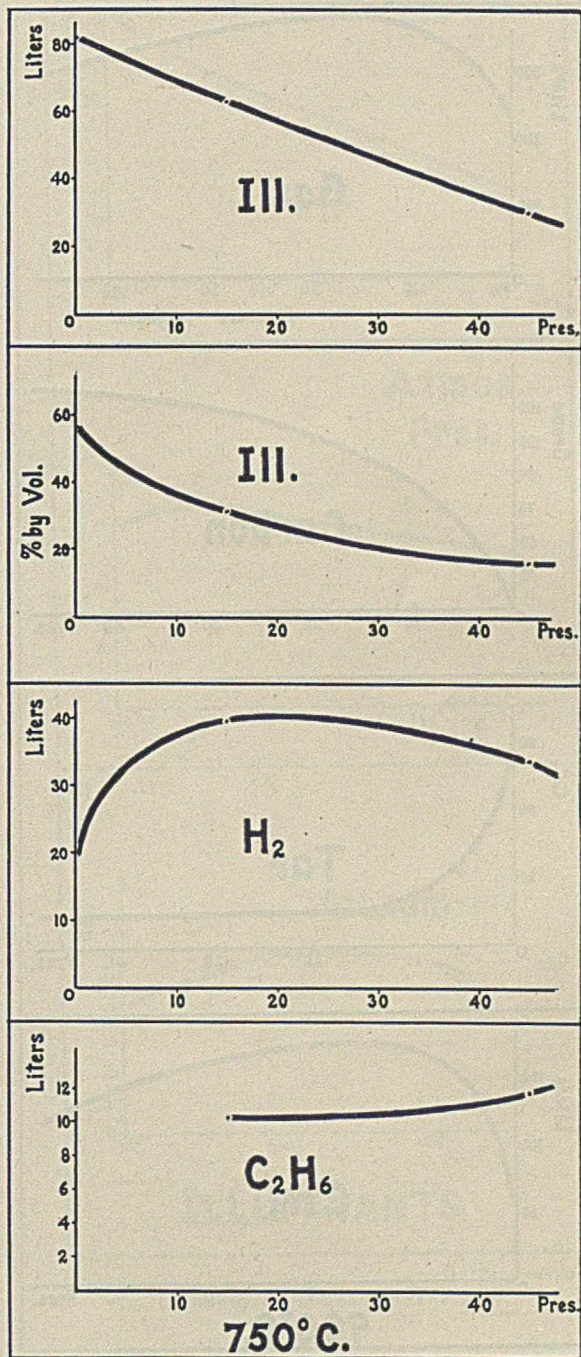


FIG. IV—VARIATIONS IN YIELDS OF PRODUCTS AT 750° C., UNDER VARYING PRESSURES. (TABLES II, III, IV AND VI)

will assist materially in condensing three volumes of acetylene into one volume of benzene: $3\text{C}_2\text{H}_2 \rightleftharpoons \text{C}_6\text{H}_6$. On the other hand, it seems reasonable to expect that, under a high pressure, it will be considerably more difficult for one volume of oil vapor to break up or

the condensation reactions which proceed to the tar stage. Since the unsaturated hydrocarbons, ethylene and acetylene, polymerize most readily, increased pressure should preferably condense them with the formation of tar compounds. In addition to the direct

TABLE III
Oil used, 400 cc.

Temp. ° C.	Pressure Absolute Lbs.	Gas Liters	Carbon Grams	Tar Cc.
650	45	145	8	133
750	45	194	26	87
900	45	310	165	9

influence of pressure, it may be assumed that when working under increased pressure the gaseous hydro-

Analysis of Gas from Table III

Temp. ° C.	C ₂ H ₆ Per cent	CH ₄ Per cent	H ₂ Per cent	Ill Per cent
650	11.5	45.1	9.3	30.5
750	6.1	56.6	17.5	15.5
900	None	41.6	50.0	5.0

The yields of gaseous hydrocarbons are lower than those shown in Table II, which were obtained at the same temperatures, and likewise the maximum yield

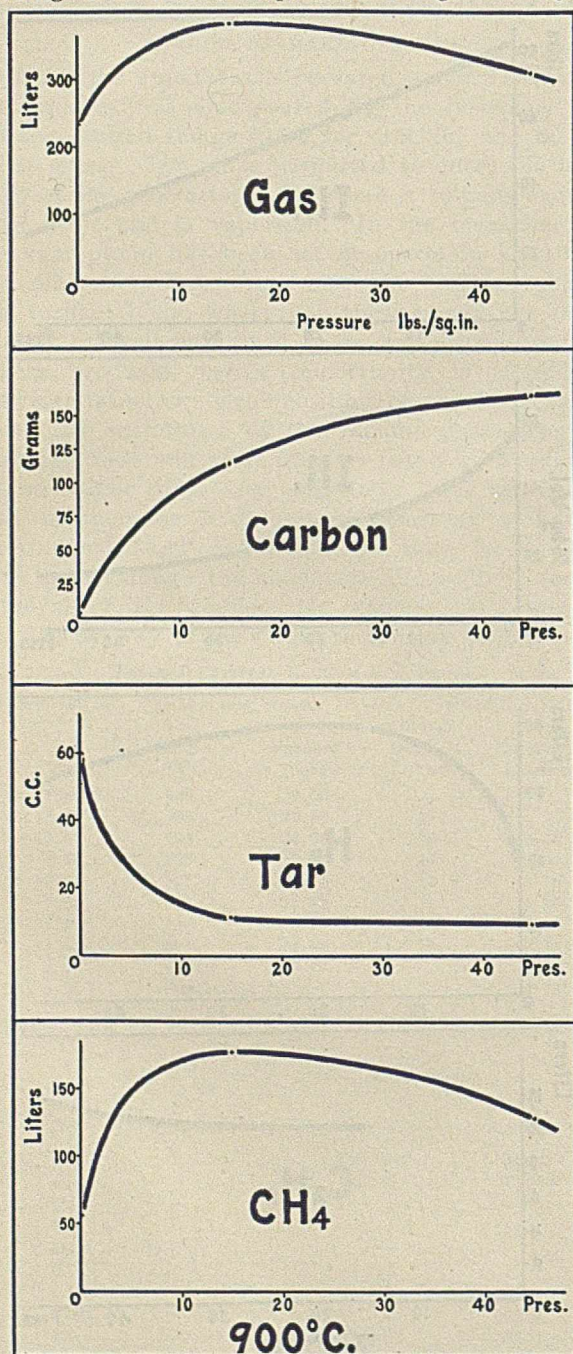


FIG. V—VARIATIONS IN YIELDS OF PRODUCTS AT 900° C., UNDER VARYING PRESSURES. (TABLES II, III, IV AND VI)

carbons are subjected to the influence of heat for a longer time, which further tends towards the formation of heavy condensation products at the expense of the illuminants. The series of experimental results in Table III appears to justify these conclusions.

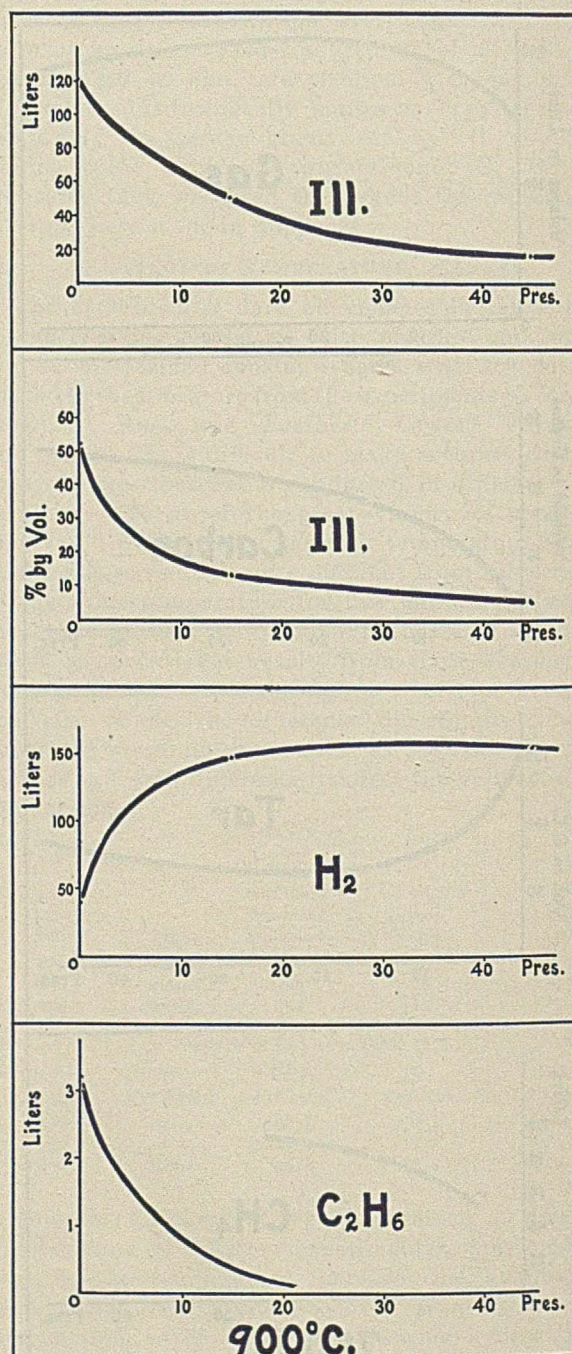


FIG. VI—VARIATIONS IN YIELDS OF PRODUCTS AT 900° C., UNDER VARYING PRESSURES. (TABLES II, III, IV AND VI)

is lower than the maximum obtained under atmospheric pressure.

EFFECT OF DIMINISHED PRESSURE ON GASEOUS REACTIONS

By referring to the OIL-GAS-TAR system cited

TABLE IV—Oil used, 400 cc.

Temp. ° C.	Pressure Absolute	Gas Liters	Carbon Grams	"Tar" Cc.
750	1/20 to 1/30 atmos.	146	1	153
850	1/20 to 1/30 atmos.	211	3	100
900	1/20 to 1/30 atmos.	234	3	60
950	1/20 to 1/30 atmos.	235	12	58

Analysis of Gas from Table IV

Temp. ° C.	C ₂ H ₆ Per cent	CH ₄ Per cent	H ₂ Per cent	III Per cent
750	12.5	56.1
850	3.4	20.5	15.6	52.9
900	1.3	24.0	17.3	52.1
950	Trace	27.0	20.8	46.9

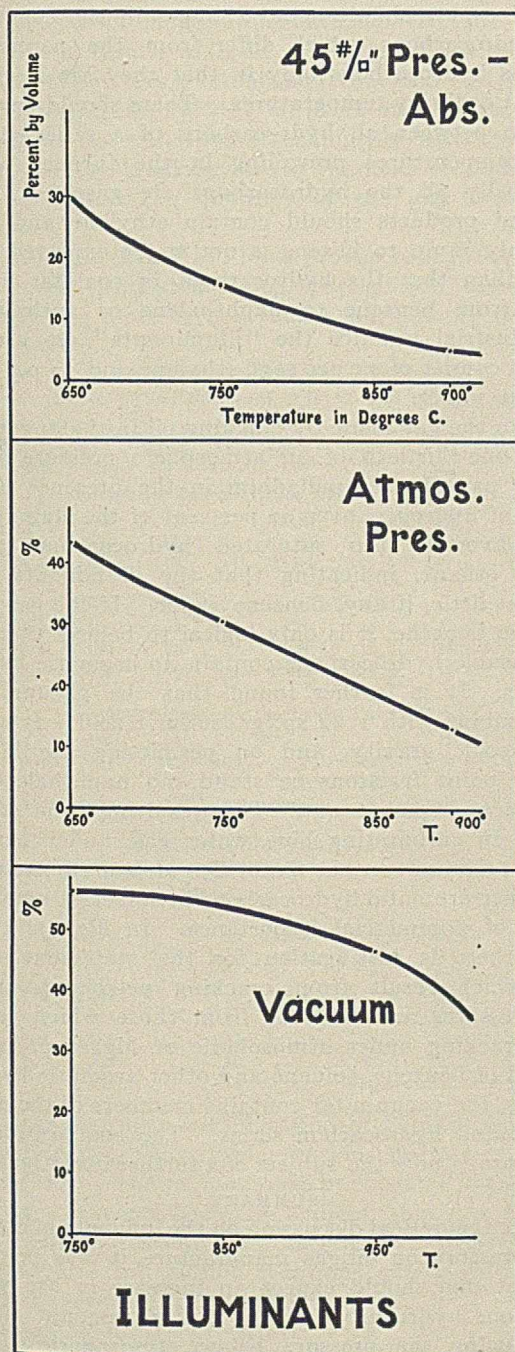


FIG. VII—PERCENTAGES OF ILLUMINANTS UNDER VARYING TEMPERATURES AND PRESSURES. COMPILED FROM TABLES II, III AND IV

above, it becomes evident that a high vacuum would favor the increase in volume due to cracking the oil into gas and at the same time withdraw the gas from the heat zone before it could form tar. The effects of this reduced pressure can best be observed from the results of the experiments in Table IV.

This striking difference in end products due to di-

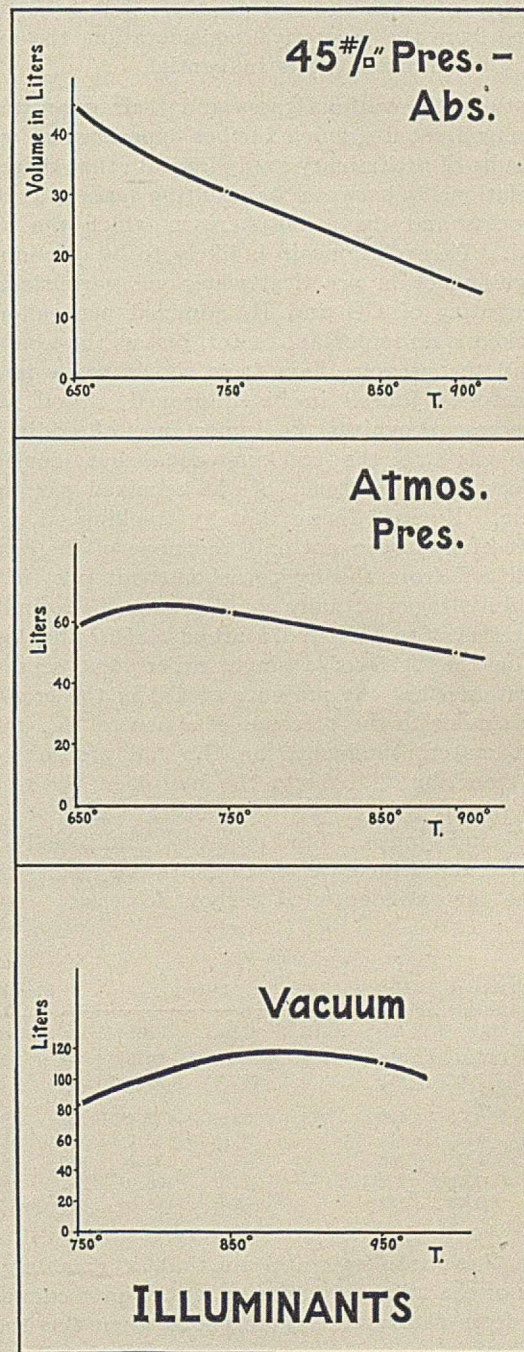


FIG. VIII—LITERS OF ILLUMINANTS UNDER VARYING TEMPERATURES AND PRESSURES. COMPILED FROM TABLE VI

minished pressure seems to have been overlooked, perhaps because for the first few pounds per square inch vacuum the increase is not marked.

INFLUENCE OF CONCENTRATION CHANGES ON GASEOUS REACTIONS

The present investigation has merely opened this

field. It has been established that oil cracked in an atmosphere of a gas, such as hydrogen, which reacts chemically with the end products of the cracking process, will yield products which are not analogous to those resulting from a physical mixture of the two gases. Not only does the mere presence of the admixed gas influence the end products, but as is to be expected from the theoretical consideration, the quantity of the admixed gas is influential.

To study the various gases and their quantitative relation will require much further experimental work. The results of preliminary study indicate that there is a vital relationship between the resulting gases in a cracking process and the atmosphere in which the oil is cracked. This relationship is likely to be of commercial significance in practical water gas carburization. The quantity of CO and H₂ admixed per gallon of oil cracked is an important factor, just as the temperature and the pressure have been shown to be important factors. Jones,¹ in his improved all oil water gas process, recognizes the importance of adding an "active gas" to the cracking zone, but considers the effect of the presence of the admixed gas to be catalytic. Hempel² found that by cracking oil in the presence of hydrogen not only did none of the hydrogen split off from the hydrocarbons, but part of the admixed hydrogen actually combined for the formation and preservation of hydrocarbons. On the other hand, on the basis of a single experiment reported, he maintains that the presence of CO in the cracking zone is similar to the presence of a neutral gas and is without material influence on the end products obtained from the oil. As to the hydrogen, the results of this research agree with the observations of both Hempel and Jones. The quantity and quality of gas per cc. of oil increase, and qualitative results show that the tar and deposited carbon decrease.

TABLE V

Temp. ° C.	Pressure Absolute Lbs.	H ₂ admixed. L.	Liters			Shrinkage in H ₂ Liters	
			C ₂ H ₄	CH ₄	Ill	H ₂	
750	15.0	358	15.4	125.0	70.6	308	50
800	15.0	412	18.0	116.0	83.2	335	77
750	0.75	400	9.5	52.0	112.0	381	19
810	0.75	413		86.5	140.0	378	35
860	0.75	388		99.5	133.0	350	38
900	0.75	292		92.0	120.0	272	20
960	0.75	382		95.0	113.0	348	34

From these results it appears that a greater percentage of the admixed hydrogen enters into combination to form saturated hydrocarbons when the cracking process is carried out under atmospheric pressure, than is the case under greatly reduced pressure. The percentage increase in yield of the illuminants when the cracking process is carried out under reduced pressure in the presence of hydrogen is about as great, however, as is the percentage increase in illuminants when the reaction is carried out under atmospheric pressure.

¹ *The Gas Age*, 1913, p. 369; *American Gas Light Journal*, 1913, p. 272; *Gas World*, 1913, 916.

² *Jour. f. Gasb.*, 1910, p. 53, et al.

INFLUENCE OF TEMPERATURE, PRESSURE AND CONCENTRATION CHANGES ON COMPOSITION OF RESULTANT TARS

If changing temperature and pressure have a marked influence on the quantity and quality of gaseous hydrocarbons obtained from cracking petroleum oil, one should expect simultaneous changes in the condensable hydrocarbons, which differ from the permanent gaseous hydrocarbons only in that they are liquid or solid at ordinary temperatures. There should be equilibrium between all hydrocarbons of a series at the high temperatures prevailing in the furnace where practically all the hydrocarbons are gaseous. That the end products should contain ethylene and then suddenly jump to hexene is not to be expected, any more than that the hydrocarbons in coal tar would jump from benzene to naphthalene or anthracene. In industrial practice the "illuminants" are usually said to consist of 75 per cent ethylene and 25 per cent benzene vapor.

When the gas made by cracking oil in the apparatus under one-thirtieth of an atmosphere pressure absolute is passed over palladium in the presence of an excess of hydrogen, over 90 per cent of the illuminants are converted into saturated hydrocarbons, principally ethane, indicating that the illuminants contain but little, if any, benzene vapor. If the gas contains no benzene, it is only logical to believe that the condensable hydrocarbons contain no aromatic hydrocarbons. It is further found that the vacuum tar will combine with 1.82 sp. gr. sulfuric acid. It has a low specific gravity, and on permitting the higher boiling point fractions to stand, no naphthalene or anthracene separate out. Tars resulting from cracking oil in carbureting blue water gas under atmospheric pressure contain quantities of benzene, toluene and other aromatic hydrocarbons in sufficient amounts to be of commercial importance. In view of these facts, there is justification for the statement that tars which result from cracking petroleum under low pressures are different from those which result from cracking under atmospheric or higher pressure. Instead of benzene, toluene and other aromatic hydrocarbons, the vacuum tar contains members of the more unsaturated hydrocarbon series. The composition of these tars is now the subject of a further investigation.

SUMMARY

In the theoretical discussion on the influence of diminished pressure on oil gas manufacture, it was pointed out that one should expect an increase in the yield of gaseous hydrocarbons from a given amount of oil by reducing the pressure below atmospheric. This increase should reach a maximum as the absolute zero of pressure is approached. The correctness of this is shown by results recorded in Tables IV and VI. Not only are the gaseous hydrocarbon yields greatly increased, but the deposited carbon is practically eliminated, and there is much less gaseous hydrogen produced than in the product obtained at the same temperatures under higher pressure.

It was pointed out that increasing the total pressure under which the oil is cracked to several atmospheres

will decrease the gaseous hydrocarbon yields from a given amount of oil. Experimental results, shown in Table III, have proven this correct.

It was pointed out that varying the pressure on the system would enable one to better control the quantity and quality of "tar" obtained than at present where all tar is made under atmospheric pressure. Experimental results indicate considerable flexibility.

It has further been established that the end products resulting from cracking oil in an atmosphere of a gas, such as H_2 , which reacts chemically with the end products of the cracking, are a function of both the composition and the quantity of the gas admixed, per Table V.

Experiments, Table IV, have proven that it is possible to "crack" oil at a temperature of $900^\circ C.$ without depositing more carbon than 1 per cent by weight of the oil used.

TABLE VI—SUMMARY OF GAS TABLES

(All based on 400 cc. oil and calculated to $0^\circ C.$, and 760 mm. pressure)

Temp. $^\circ C.$	Pressure Lbs. per sq. in.	Gas L.	Carbon G.	Tar Cc.	C_2H_6 L.	CH_4 L.	H_2 L.	Ill L.
Atmospheric Pressure Group (See Table II)								
650	15.0	135	3	163	13.8	45.5	12.1	58.8
750	15.0	206	18	80	10.15	84.5	39.6	63.0
900	15.0	382	115	11	Trace	178.1	148.2	50.0
High Pressure Group (See Table III)								
650	45.0	145	8	133	16.7	65.2	13.1	44.3
750	45.0	194	26	87	11.8	110.0	33.9	30.1
900	45.0	310	165	9	None	128.9	155.0	15.5
Low Pressure Group (See Tables I and IV)								
750	0.75	146	1	153	18.3	82.0
850	0.75	211	3	100	7.16	43.2	32.9	111.5
900	0.75	234	3	60	3.0	56.0	40.0	122.0
950	0.75	235	12	58	Trace	63.4	48.8	110.0
Admixed Gas Group (See Table V)								
		Hydrogen admixed L.	Hydrogen shrinkage L.					
750	15.0	358	50	15.4	125.0	308.0	70.6	
800	15.0	412	77	18.0	116.0	335.0	83.2	
750	1.0	400	19	9.5	52.0	381.0	112.0	
810	1.0	413	35	..	86.5	378.0	140.0	
860	1.0	388	38	..	99.5	350.0	133.0	
900	1.0	292	20	..	92.0	272.0	120.0	
950	1.0	382	34	..	95.0	348.0	113.0	

Through a proper consideration of equilibrium and mass action conditions under various degrees of temperature and pressure, much can be expected in gaseous reactions. It soon becomes evident that the single stage method wherein endothermic and exothermic, expansion and contraction reactions are combined in a single apparatus, is open to question.

CHEMICAL ENGINEERING LABORATORY
COLUMBIA UNIVERSITY, NEW YORK

NOTE ON A BLACK BAND IRON ORE FROM THE STEARNS COMPANY'S COAL MINES AND ON THE CALCIUM-MAGNESIUM RATIO IN KENTUCKY CARBONATE ORES¹

By ALFRED M. PETER

In September, 1913, the Stearns Coal Company sent to this Experiment Station, for analysis, a sample of

¹ Presented at the regular meeting of the Lexington Section of the American Chemical Society, February 11, 1914.

iron ore found immediately over the coal in their mines at Stearns, McCreary County, Kentucky. The ore occurs in a layer of about 18 inches maximum thickness, resting on the coal, and is capable of being mined with the coal. It is seal-brown in color, has a horizontal lamination or banding with carbonaceous matter between the laminae, and breaks transversely to the laminae, giving a rather smooth surface. On account of its structure and the presence of carbonaceous matter, it is here classed as "Black Band" ore, though distinctly brown, not black, in color and containing much less carbonaceous matter than is usual in black band.

An analysis by Mr. J. S. McHargue follows:

ANALYSIS OF THE AIR-DRIED SAMPLE No. 43255	
Specific gravity ($18^\circ/18^\circ$) 3.268	
Ferrous oxid.....	51.77 per cent
Ferric oxid.....	0.85
Alumina.....	0.60
Manganous oxid.....	0.51
Calcium oxid.....	1.94
Magnesium oxid.....	3.57
Phosphorus pentoxid.....	0.08
Sulfur.....	Trace
Silica.....	1.16
Organic matter.....	2.77
Carbon dioxide, etc., by difference.....	36.69
Total.....	100.00
Total iron.....	40.87
Total phosphorus.....	0.035

Treatment with carbon disulfide extracted an appreciable amount of brown, bituminous matter, the quantity of which was not determined. The figure for organic matter was obtained by digesting a portion of the ore in HCl , filtering on a Gooch filter, washing, drying at 100° , weighing and burning, the loss in weight being taken to represent organic matter.

PROBABLE COMBINATIONS, CALCULATED FROM ABOVE ANALYSIS

Ferrous carbonate.....	83.50 per cent
Ferric oxid.....	0.85
Alumina.....	0.60
Manganous carbonate.....	1.29
Calcium carbonate.....	3.30
Magnesium carbonate.....	7.49
Tricalcium phosphate.....	0.17
Sulfate.....	Trace
Silica.....	1.16
Organic matter.....	2.77
Total.....	101.13

The analysis shows that this is quite a pure iron ore, containing, however, considerable quantities of manganese, calcium and magnesium. A notable feature is that there is more than twice as much magnesium carbonate present as calcium carbonate. Calculating to molecular proportions and including the manganese in the comparison, gives 1 $MnCO_3$: 2.94 $CaCO_3$: - 7.81 $MgCO_3$, or, in round numbers 1 : 3 : 8. The ratio of calcium carbonate to magnesium carbonate is 1 $CaCO_3$ to 2.66 $MgCO_3$. Of course this may be only a chance relation, but there is room to speculate why there is more magnesium carbonate than calcium carbonate. Has it a relation to the greater solubility of magnesium carbonate in carbonated water? Or is it because there was more magnesium than calcium in the original material from which this ore was formed? No answer is suggested, but a fact like this, in the hands of a well informed chemical geologist, might help to explain the process by which this kind of iron ore is formed.

To show that magnesium usually predominates over calcium in the ferrous carbonate ores of Kentucky, all the analyses of such ores made for the first Kentucky

Geological Survey, 1856 to 1860, by Dr. Robert Peter, Chemist of the Survey, and published in the first four chemical reports, have been tabulated and compared.

The total number of samples analyzed was 71 from 20 counties, as follows: Bath 3, Bullitt 2, Butler 2, Carter 3, Crittenden 2, Edmondson 1, Estill 4, Greenup 26, Hancock 1, Hopkins 2, Laurel 4, Lincoln 1, Montgomery 1, Morgan 1, Muhlenberg 7, Nelson 2, Owsley 2, Powell 1, Pulaski 2, Whitley 3.

The geological formations represented were mainly the Coal Measures and the Devonian, with some from the "Sub-Carboniferous" of Owen, or Mississippian, and the Silurian.

The kinds of ore were various, including kidney ore, oolitic ore, massive kinds, black band, block ore and two samples which would better have been classed as ferruginous limestones. Probably most of the block ores should not have been included because, in these, the proportion of ferric oxid often exceeds that of ferrous carbonate.

The whole tabulation would occupy too much space here. Inspection of it brings out the following facts:

	No.
Analyses tabulated.....	71
Per cent MgCO ₃ exceeds per cent CaCO ₃	57
Per cent MgCO ₃ equals per cent CaCO ₃	1
Per cent MgCO ₃ is less than per cent CaCO ₃	13
Per cent Insoluble matter exceeds 10 per cent.....	45
Per cent CaCO ₃ exceeds 10 per cent.....	2

These last two, of course, are included in the thirteen which contain more CaCO₃ than MgCO₃. The large number containing more than 10 per cent insoluble matter shows that the collection includes many impure ores.

To gain a general idea of the composition of the samples in which the MgCO₃ predominated over the CaCO₃, the 57 analyses of this class have been averaged, giving the figures in the following table, which includes also the extreme range of each constituent, all stated as per cent of the ore, dried at 100° C.:

COMPOSITION OF 57 SAMPLES	Average	Highest	Lowest
Ferrous carbonate, FeCO ₃	60.78	87.53	20.19
Ferric oxid, Fe ₂ O ₃	8.09	51.31	Trace
Alumina, Al ₂ O ₃	1.40	5.15	Trace
Calcium carbonate, CaCO ₃	2.71	7.25	Trace
Magnesium carbonate, MgCO ₃	5.92	13.99	0.83
Manganous carbonate, MnCO ₃	1.57	5.03	0.12
Insoluble residue.....	16.36	40.88	4.87
Combined H ₂ O, bituminous matter and undetermined.....	3.17
Total.....	100.00		

It is seen that the proportion of magnesium carbonate is more than twice that of the calcium carbonate and that the proportion of manganous carbonate is the smaller of the three. Calculated to molecular proportions, the relation is 1 MnCO₃ : 2 CaCO₃ : 5.1 MgCO₃. It is presumably only an accident, however, in such a calculation as this, that the molecular ratios are almost exactly whole numbers. The ratio of calcium carbonate to magnesium carbonate is 1 CaCO₃ to 2.55 MgCO₃, which does not differ very far from the ratio found in the Stearns sample, *viz.*, 1 CaCO₃ to 2.66 MgCO₃.

KENTUCKY AGRICULTURAL EXPERIMENT STATION
LEXINGTON

A NOTE ON THE UTILIZATION OF MUDS AND SCUMS FROM SUGAR REFINERIES

By R. F. GARDINER¹

Received March 3, 1914

A description of the muds and scums in the sugar refining processes can be found in most works on industrial chemistry. Thorp says that "these muds have been thrown away." The possibility of their utilization is now being seriously considered by some sugar producers, as is shown by recent correspondence between this laboratory and a prominent American sugar producer, who says: "The mud was thrown away for quite a time and now we are trying some experiments in drying the same for the phosphoric acid content."

Sample I was analyzed by the author; the nitrogen determination was kindly made by Mr. T. C. Prescott, of the Bureau of Chemistry. The analysis of Sample II, by Plicque, is cited because it also shows a considerable amount of water.²

TABLE I—SHOWING COMPOSITION OF MUD FROM A SUGAR MILL

Percentages	I Gardiner	II Plicque
H ₂ O.....	67.32	52.70
Sugar.....	...	3.50
Nitrogenous matter.....	...	3.72
N.....	1.14	...
K ₂ O.....	Trace	...
P ₂ O ₅	8.33	4.77
Lime, silica, iron, etc.....	...	26.07

In order to economically recover the valuable fertilizer elements, such as phosphoric acid and nitrogen, it will be necessary to find some satisfactory drying process, for which modern improvements in drying processes may offer ultimate success in the utilization of this potentially valuable fertilizer, now entirely lost.

BUREAU OF SOILS, DEPARTMENT OF AGRICULTURE
WASHINGTON

THE EFFECT OF HIGH TEMPERATURE ON YEAST

By C. B. COCHRAN³ AND J. H. PERKINS⁴

Received October 17, 1913

In the experiments recorded below, the yeast was added to 30 cc. of simple syrup in sterilized test tubes plugged with cotton, raised to the temperatures indicated and held there for the stated periods of time. The tubes were then placed in an incubator and kept at a temperature of about 31° C.

SP. GR. OF SYRUP, AND TIME	TUBE No.	TEMP. ° C.	RESULTING FERMENTATION			
			Fleischmann's (b)	Magic (b)		
SP. GR., 1.125..... TIME, 5 min.....	{ 1 & 2	35	Prompt and vigorous			
	{ 1 & 2	40				
	{ 1 & 2	45				
SP. GR., 1.125..... TIME, 5 min.....	{ 1 & 2	55	None } Active after 2 days	Slight } None		
	{ 1	68				
	{ 2	68				
SP. GR., 1.120..... TIME, 30 min.....	{ 1 & 2	85	Prompt and vigorous			
	{ 1, 2 & 3	48				
	{ 1, 2 & 3	58				
SP. GR., 1.125..... TIME, 30 min.....	{ 1, 2 & 3	65	Slow	Slow		
	{ 1 & 2	70			None	None
	{ 3	70				
SP. GR., 1.126(a).... TIME, 15 min.....	{ 1, 2 & 3	68	None	None		
	{ 1, 2 & 3	72				
	{ 1, 2 & 3	72				

(a) In these tests the heat was applied after leaving the yeast in the syrup for 24 hours at a temperature of about 12° C.

(b) To each 30 cc. of syrup, 1/32 of a Fleischmann's or 1/16 of a Magic yeast cake was added.

PHILADELPHIA

¹ Scientist in Soil Laboratory Investigations.

² "Handbook for Planters and Refiners," Lock and Newlands Bros 1888, p. 489.

³ Chemist for The Dairy and Food Department of Pennsylvania.

⁴ Chief Chemist for The Charles E. Hires Company.

PHYSICAL FACTORS WHICH INFLUENCE THE PERCENTAGE OF WET AND DRY GLUTEN IN WHEATEN FLOUR

By B. H. KEPNER

Received January 28, 1914.

The determination of crude wet and dry gluten is affected by so many physical conditions and is so unreliable that it has been almost wholly given up and the proteins are now determined in most laboratories by the more accurate Kjeldahl method. In exceptional cases when it is necessary to know the quality of the gluten, a qualitative determination is made. Arpin¹ recognizes the fact that there are numerous sources of error in the separation of the gluten. Jago² states that it is difficult and in many cases impossible to wash away the whole of the starch from flour without also washing away some of the more soluble parts of the gluten itself. In consequence, gluten determinations vary according to the thoroughness of washing and this differs in different hands. In spite of this assertion Jago still continues to attach importance to properly conducted gluten determinations, holding that the estimation of the quantity and quality of the wet gluten determines the quality of the resultant dough and bread. It has been my experience that, within the same grade, Straight, Patent, Bakers, etc., the higher the percentage and the better the quality of the wet gluten, the better baking results are obtained.

Because there is no standard method, each laboratory at present uses a method which is the most suitable for its use. Results are affected by the conditions and methods of manipulation by which they are obtained. They are not absolutely comparable from different laboratories because of the difference in physical conditions.

The following method we have adopted as being especially suitable for routine work: Weigh out thirty grams of flour in a large size coffee cup. Mix in enough water, about 17 cc., with a stiff bladed knife to make a dough of standard stiffness and continue the mixing until an absolutely uniform dough is obtained. Cover the dough with cold water, let stand one hour and then wash in a small stream of water at exactly 65° C. (over a sixty-mesh sieve) until nearly all the starch is washed away. Return gluten to cup, cover with cold water, let stand one-half hour longer and then wash until free from starch. The wash water should be clear and not milky. Cover the gluten with cold water until it is ready to weigh, then dry between the hands, wiping the excess of water on a clean, lint-free towel, place on white tared paper 2" × 2" and weigh. Place in oven at 170° C. to expand the gluten to a porous mass and bake it, then dry for twenty-four hours at 105° and again weigh.

It was found very difficult to check the determinations from day to day, hence, the effect of the different physical conditions were determined.

It is necessary to make an absolutely uniform dough of the water and flour, otherwise portions of flours are washed away like starch. Poorly mixed samples

give results which are 1 per cent lower in wet gluten and 0.2 to 0.5 per cent lower in dry gluten than a well mixed sample. The poorer the mixing, as a rule, the greater the loss.

If an excess of water is used for making the dough, the yield of wet gluten is increased from 1 to 3 per cent for the different grades, while insufficient water causes the percentage of gluten obtained to be lower. The percentage of dry gluten is not affected by this condition, showing that the higher or lower percentage of wet gluten is due wholly to water held by it.

If the doughs are kept covered with cold water before washing the percentage of wet gluten obtained in most cases increases with the length of time they are allowed to stand up to eight hours. They are kept cold in order to offset the influence of temperature and any fermentation. Arpin¹ shows that the wet gluten is increased 1.66 per cent by standing four hours, while the percentage of dry gluten remains unaltered. I find that very short patents and flours which have aged at least nine months are exceptions to this because there is only a very slight increase for even eight hours. Bakers and straight grades, when freshly ground, showed an increase of 1.50 to 3.50 per cent on standing eight hours. Low grades show an increase of only 0.3 per cent on standing one hour and a decrease of 2.5 per cent on standing eight hours. The low grades being high in natural ferments or bacteria suffer a loss by their action. The aged flours and short patents are unaffected because they contain less of these natural ferments than the freshly ground bakers and straight grades. All grades begin to decrease at the end of sixteen hours which would strengthen this supposition. The percentage of dry gluten remains the same up to sixteen hours on all flours with the exception of the low grade, which suffers a loss. After glutes are washed and allowed to stand for even twenty-four hours there is no change in weight. This shows that the increase or decrease in weight on standing eight hours before washing is due to some agency in the flour which is not included in the crude wet gluten.

The thoroughness of washing affects the results to a great degree. When glutes are washed in the usual manner, weighed, and then vigorously washed for five minutes more there is a loss of 5 per cent on the low grades, 3 to 4 per cent on the straights and bakers and 2.5 per cent on the high patents and old flours. There is also a loss of dry gluten. This loss varies with the manipulation and the vigorousness of the operation.

Arpin² states that the yield of gluten is increased 1.16 per cent with an increase of 10° in temperature of the water used for washing. We find that wet gluten increases 1.50 per cent for the same increase of temperature for all grades except the low grade, which shows a slight decrease. This can perhaps also be explained by the more active action of natural ferments caused by higher temperature. Arpin also found an increase of 1.16 per cent for the same range of temperature in the dry gluten. This point we have

¹ *Jour. Soc. Chem. Ind.*, 1902, pp. 1417 and 1560.

² "Technology of Breadmaking," p. 296.

¹ *Jour. Soc. Chem. Ind.*, 1902, 1417 and 1560.

² *Chem. Centr.*, 2 (1902), 1019 and 1347.

carefully investigated and at no time did we find an increase in the percentage of the dry gluten due to the increase in temperature.

In order to test the effect of temperature on the washed glutes they were allowed to stand twelve hours at 25° C., having been washed at 15° C. The glutes had become very sticky and hard to dry. The low grades all showed that fermentation had started. The low grades showed a marked decrease while the high grades showed only a slight decrease. This was also true of the dry glutes. Whatever agency causes the increase in weight, when the temperature of the wash water is higher than ordinary, is within the flour itself and is not contained in the wet gluten.

A higher percentage of gluten is obtained by washing in hard water than in soft. Hardy¹ is of the opinion that electrolytes or salts, which may be organic or inorganic, impress the property of tenacity and ductility on the gluten. Wood's² researches also prove that inorganic salts have a binding effect on gluten. All authorities agree that gluten cannot be washed from flour with pure distilled water because the gluten will not hold together. At first it forms a coherent mass but as soon as the salts, natural to the flour, are dissolved, the gluten scatters and cannot be collected again. If it be submerged in hard water when it first starts to scatter it forms again a coherent mass. There was an increase of 1 to 2 per cent in wet gluten for all grades when the glutes were washed in very hard water. There was only a slight change of about 0.25 per cent for the dry gluten at the most, showing that the hard water usually increased the water-absorbing capacity of the gluten.

CONCLUSIONS

It can readily be seen that conditions may be so different in various laboratories that the determinations of wet gluten are not comparable. Even for ordinary routine work, where comparative results alone are required, unless the utmost care is taken, the determination is unreliable. Since the percentage of dry gluten is very slightly affected by these same conditions it is more reliable.

1—Thoroughness of mixing affects both the per cent of wet and dry gluten.

2—An excess of water used in making the doughs increases, and insufficient water decreases the per cent of wet gluten. The dry gluten is the same.

3—The length of time the dough is allowed to stand increases the percentage of wet gluten up to eight hours. High patents, old flours and low grades are exceptions. The dry gluten remains unaltered except in the low grade where some fermentation has taken place.

4—Overwashing decreases the percentage of both the wet and dry gluten.

5—A larger per cent of wet gluten is obtained with warm wash water than with cold. The dry gluten is unaffected.

6—More wet gluten is obtained with "hard" wash

water than with soft. The dry gluten is slightly increased by the hard water.

WHEAT AND FLOUR TESTING LABORATORIES
MAPLE LEAF MILLING CO., LTD.
PORT COLBORNE, ONT.

THE DETERMINATION OF THE ACETYL NUMBER OF OILS, FATS, ETC.¹

By EDWARD B. HOLLAND

Received January 13, 1914

INTRODUCTION

The various hydroxy compounds that occur in oils, fats and waxes form derivatives on heating with acetic anhydride, the acetyl radical displacing the hydrogen of the alcoholic hydroxyl groups. This property serves as the basis of analytical methods for the quantitative determination of these compounds. The proposed acetyl number indicates the milligrams of potassium hydroxide required for the saponification of the acetyl assimilated by one gram of an oil, fat or wax on acetylation.² On saponifying with alcoholic potash the acetyl is hydrolyzed to acetic acid and combines with the alkali to form potassium acetate. The results are expressed in terms of milligrams of potassium hydroxide to conform with the general practice in fat analysis. The compounds involved are mono- and dihydroxy acids and their glycerides, mono- and diglycerides and free alcohols.

USE OF THE TEST

In the examination of oils and fats a determination of acetyl number is necessary, in most instances, for a thorough understanding of the nature and quality of the product. Some of the hydroxy compounds are natural and others are the result of hydrolysis or of oxidation. Glycerides of hydroxy acids are a natural constituent of certain oils and fats although they do not appear to be very widely distributed in any considerable amount. Castor oil, composed largely of ricinolein, is a notable illustration. Hydroxy acids probably occur more frequently as the result of oxidation of unsaturated acids. Oleic acid has been shown repeatedly to be comparatively unstable. By the assimilation of oxygen and water it may be converted into dihydroxystearic acid, a saturated compound.

$C_{17}H_{33}COOH + H_2O + O = C_{17}H_{33}(OH)_2COOH$.
Whether the oxidation takes place in the glycerides or in the fatty acids after hydrolysis is uncertain, although the latter appears the more probable supposition.

Mono- and diglycerides result from the hydrolysis of triglycerides and free fatty acids condition their presence. The absence of free fatty acids in a commercial product, however, does not necessarily preclude the presence of mono- and diglycerides.

Solid alcohols of the cyclic series (sterols) occur in oils and fats both in combination as esters and as free

¹ The writer is pleased to acknowledge many suggestions and helpful criticisms by Dr. J. S. Chamberlain, Mr. F. W. Morse, Mr. J. C. Reed, and Mr. J. P. Buckley.

² Benedikt and Ulzer and Lewkowitsch report on the basis of the acetylated product.

¹ Supplement Jour., 4 (1910), 52; Jour. Board of Agric.

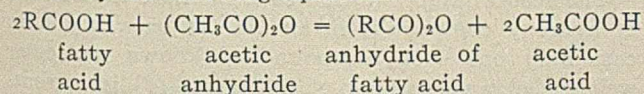
² Jour. Agric. Science, 2 (1907), 267.

alcohols.¹ The amount of cholesterol or phytosterol is generally small, often inappreciable, and is indicated approximately by the unsaponifiable matter which it characterizes. Alcohols of the ethane and other series, free and in combination, compose a considerable proportion of waxes.

Oils and fats, therefore, may contain glycerides of mono- and dihydroxy acids, possibly free hydroxy acids, mono- and diglycerides and free alcohols; and the insoluble acids, separated from the oils and fats, may contain mono- and dihydroxy acids and free alcohols. A portion, at least, of the free alcohols found in the insoluble acids probably occurred in the fat as esters. With the exclusion of the natural glycerides of hydroxy acids and a small amount of free alcohols, the acetyl number of many oils and fats may be deemed an index of quality and when considered in conjunction with the acid and iodine numbers, may serve to measure (more or less imperfectly to be sure) the amount of hydrolysis and of oxidation the product has undergone. To differentiate between products of hydrolysis and of oxidation, the acetyl number of the insoluble acids should also be determined.

EARLIER METHODS

The several analytical processes that have been offered are based on the same chemical reactions but differ in application and in details of procedure. The original method was devised by Benedikt and Ulzer² and applied to the insoluble acids. The acetyl number indicated the milligrams of potassium hydroxide required to neutralize the acetic acid obtained on saponifying one gram of acetylated insoluble fatty acids and was determined by the difference between the acid and saponification numbers of the acetylated acids (acetyl ether number). The actual procedure consisted in saponifying the acetylated acids after neutralizing in alcohol. Lewkowitsch³ has shown, however, that the results so obtained were generally in excess of the true values due to the conversion of a part of the fatty acids, on heating with a large excess of acetic anhydride, into their anhydrides as illustrated by the following equation:



These fatty anhydrides are fairly stable compounds but may become hydrolyzed to some extent on washing with boiling water. Subsequent treatment with cold alcohol in the determination of the acetyl acid number will continue the hydrolysis although a portion is likely to remain unchanged, thereby yielding too low an acid number due to the inability of the anhydrides to combine with alkali. As complete hydrolysis occurs on saponification the acetyl (ether) number would be too high and even appear when none exists.

Lewkowitsch⁴ proposed the acetylation of the nat-

¹ See numerous references: Abderhalden, "Physiological Chemistry," (1908); Hammarsten, *Ibid.* (1911); Leathes, "The Fats" (1910).

² *Monatsh. Chem.*, **8** (1887), 41-48.

³ "Analysis of Oils, Fats and Waxes," **1** (1909), 344-5.

⁴ *Loc. cit.*, **1** (1909), 337-8.

ural product. In conformity thereto the acetyl number indicates the milligrams of potassium hydroxide required for the neutralization of the acetic acid obtained on saponifying one gram of an acetylated oil, fat or wax. This method requires the saponification of the acetylated fat and the determination of the resulting acetic acid by either a filtration or distillation process. The former process is an adaptation of the regular method for the direct determination of soluble acids, and the latter process is a modified Reichert-Meissl test with repeated distillation of the aqueous solution until the distillate is free from acids. The presence of natural soluble or volatile acids necessitates a similar treatment of the unacetylated fat in order to determine the amount of alkali assimilated by those acids for which proper corrections must be made to obtain the true acetyl number. The occurrence of the lower acids makes the determination a long and tedious operation.

PROPOSED METHOD

Analytical methods for the examination of oils and fats is a subject that has been given considerable study by the writer in connection with feeding experiments and other investigations made at the Massachusetts Agricultural Experiment Station. During the past few years the determination of acetyl number has received particular attention with a view of evolving a process that might be free from the objections cited for the Benedikt and Ulzer and Lewkowitsch methods. Believing that this object has been obtained in some measure, a report of progress is now offered in the hope that it may lead to further improvement.

The custom of reporting acetyl number on the basis of the acetylated product appears unwarranted. It is contrary to general practice in analytical work and is the exception in fat analysis. The definition¹ adopted places the acetyl number on a par with other tests and is as follows: The acetyl number indicates the milligrams of potassium hydroxide required for the saponification of the acetyl assimilated by one gram of an oil, fat or wax on acetylation.

METHOD IN DETAIL

The development of the method extended over a period of several years and finally resolved into an adaptation of several well known processes. For instance, ceresine is used to solidify the acetylated fat so that it may be washed by decantation as in the determination of insoluble acids. The saponification number of the acetylated fat is determined by the same process as that of the original fat and the difference measures the amount of acetyl that has been assimilated. The process may be appropriately described as a method of analogy.

The reagents employed in the determination are summarized so that their application may be clearly understood:

Acetic anhydride, Kahlbaum's.

Ceresine, pure white, filtered.

Alcohol, redistilled, free from acids and aldehydes.

Alcoholic potash, 50 cc. of a saturated solution of potassium

¹ The hydroxyl value of Twitchell is reported in a similar manner. *Jour. Amer. Chem. Soc.*, **29** (1907), 566-71.

hydroxide, free from carbonate, to 1000 cc. of alcohol. The solution should be allowed to stand at least 24 hours and filtered immediately before use.

N/2 hydrochloric acid.

Alkali blue (6B), 1 gram to 100 cc. of alcohol. The indicator should be digested in a stoppered bottle for several days at room temperature, with occasional shaking, and then filtered.

Phenolphthalein, 1 gram to 100 cc. of alcohol, neutralized.

After what has been said, the details of the method should be sufficiently evident as to require no further explanation.

Into a 300 cc. Erlenmeyer flask are brought 5 grams of fat together with 10 cc. of acetic anhydride. The flask is connected with a spiral or other form of reflux condenser and heated in a boiling water bath (immersed in the water) for from 1 to 1.5 hours. Longer heating yields higher results but is accompanied by partial decomposition of the fat with formation of aldehydes or other bodies that give a reddish color with caustic alkali. After acetylating, the flask is removed from the bath and sufficient ceresine added to form, with the fat, a solid disc when chilled in cold water. The amount of ceresine required will vary with the consistency of the product under examination. For butter fat 0.4 to 0.5 gram is ample, for softer fats and oils rather more, and for harder fats less. The flask is heated on the water bath and the contents rotated until the ceresine and acetylated fat form a homogeneous mixture. 150 cc. of boiling water are then poured carefully into the flask with as little disturbance of the fat layer as possible and the solution heated on the bath with occasional agitation to remove occluded acetic acid. The flask is immersed in cold water to solidify the ceresine fat, after which the solution is decanted through a dense, ether-extracted filter, care being taken not to break the insoluble cake. Another 150 cc. of boiling water are added, thoroughly agitated, heated as above, cooled and decanted, the process being repeated until the final filtrate gives a decided color with 2 or 3 drops of *N*/10 alkali, using phenolphthalein as indicator (about 6 times). Prolonged washing is likely to cause slight dissociation of the acetylated product.

The filter and inverted flask containing the cake of ceresine-fat are allowed to drain in a cool place until practically dry. The small particles adhering to the filter are then scraped into the flask, and 50 cc. of alcoholic potash, accurately measured with a burette, 50 cc. of alcohol and several glass beads added. The flask is connected with a spiral or other form of reflux condenser and the solution boiled on a water bath until saponification is complete—about 60 minutes. The flask is placed in a water bath at 60° C. and the solution, after cooling to that temperature, titrated with *N*/2 hydrochloric acid, using 1 cc. of alkali blue as indicator. Phenolphthalein may be employed, though less satisfactory for colored solutions. The alcoholic mixture is again brought to boil to free any alkali occluded in the ceresine and retitred, if necessary. Several blank determinations should be run with every series of tests under precisely similar conditions as to time and treatment except that the ceresine may be omitted. However, every lot of ceresine must be tested, should be free from soluble matter and not assimilate any alkali on saponification. The difference between the titration of the blank and that of the excess alkali in the test is the acid equivalent of the fat after acetylation, which should be calculated to milligrams of potassium hydroxide for 1 gram of fat.

1 cc. of *N*/2 acid is equivalent to 28.054 milligrams of potassium hydroxide.

The difference between the saponification number of the fat before and after acetylation is the acetyl number. In case the original fat contains *free soluble* acids, their titer should be determined and proper correction made for the same.

Limit of error 0.50 acetyl number.

SYNOPSIS OF REACTION

A better conception of the method may be obtained by a summary of the reactions.

Acetylation of glycerides of mono- and dihydroxy acids, mono- and diglycerides and free alcohols (see formulas).

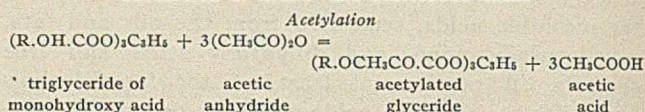
Saponification of the acetylated product (see formulas).

Saponification of the original or unacetylated product.

Titration of excess alkali.

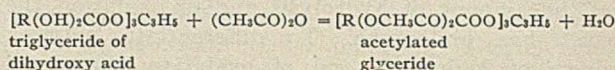
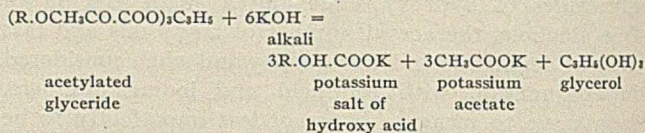
Acetyl number by difference.

GLYCERIDES OF MONO- AND DIHYDROXY ACIDS

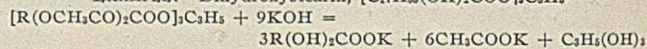


EXAMPLE: Ricinolein, $(\text{C}_{17}\text{H}_{32}\text{OH.CO})_2\text{C}_2\text{H}_5$

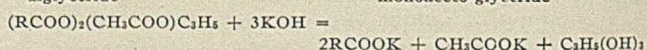
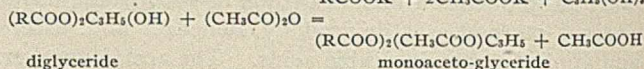
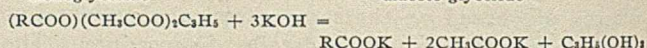
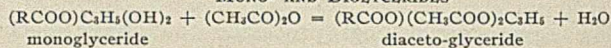
Saponification



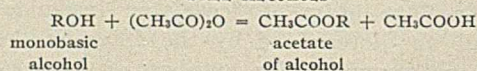
EXAMPLE: Dihydroxystearin, $[\text{C}_{17}\text{H}_{32}(\text{OH})_2\text{CO}]_2\text{C}_2\text{H}_5$



MONO- AND DIGLYCERIDES



FREE ALCOHOLS



EXAMPLES: Cholesterol phytosterol, $\text{C}_{27}\text{H}_{46}\text{OH}$

Considerable variation is possible in writing the above formulas which, at best, poorly express the structure. In some instances the reaction is indicated at some sacrifice of form.

CALCULATED DATA FROM THE ACETYL NUMBER

The acetyl number (*c*) serves to measure the amount of hydroxy compounds in an oil, fat or wax and in case only one such compound of known molecular weight (*m*) and number of hydroxyls (*d*) is present, its amount (H) can be readily calculated by the following formula:

$$H = \frac{cm}{56108d}$$

The derivation of the formula is comparatively simple. The theoretical acetyl number of a compound containing (*d*) hydroxyl groups is:

$$\frac{56108d}{m}$$

The amount of such a compound in an oil, fat or wax is, therefore:

$$\frac{c}{56108d} = \frac{cm}{56108d}$$

The same results may be calculated more easily from the following table, dividing the determined acetyl number by the theoretical acetyl number or multiplying by its reciprocal:

The formation of anhydrides during the acetylating process will affect the accuracy of these calculations.

The computation of the amount of hydroxy com-

ACETYL NUMBER ON ORIGINAL PRODUCT, MASSACHUSETTS METHOD

Name	Formula	Molecular weight	Saponification number	Theoretical acetyl number	Reciprocal
GLYCERIDES					
Ricinolein, (C ₁₇ H ₃₃ .OH.COO) ₂ C ₂ H ₅		932.832	180.444	180.444	0.0055419
Dihydroxystearin, [C ₁₇ H ₃₃ (OH) ₂ COO] ₂ C ₂ H ₅		986.880	170.562	341.124	0.0029315
MONOGLYCERIDES					
Monopalmitin, (C ₁₅ H ₃₁ COO)C ₂ H ₅ (OH) ₂		330.304	169.868	339.736	0.0029435
Monostearin, (C ₁₇ H ₃₃ COO)C ₂ H ₅ (OH) ₂		358.336	156.579	313.159	0.0031933
Monolein, (C ₁₇ H ₃₃ COO)C ₂ H ₅ (OH) ₂		356.320	157.465	314.930	0.0031753
DIGLYCERIDES					
Dipalmitin, (C ₁₅ H ₃₁ COO) ₂ C ₂ H ₅ (OH)		568.544	197.374	98.687	0.0101330
Distearin, (C ₁₇ H ₃₃ COO) ₂ C ₂ H ₅ (OH)		624.608	179.658	89.829	0.0111323
Dirolein, (C ₁₇ H ₃₃ COO) ₂ C ₂ H ₅ (OH)		620.576	180.826	90.413	0.0110604
HYDROXY ACIDS					
Ricinoleic, C ₁₇ H ₃₃ .OH.COOH		298.272	188.110	188.110	0.0053160
Dihydroxystearic, C ₁₇ H ₃₃ (OH) ₂ .COOH		316.288	177.395	354.791	0.0028186
FREE ALCOHOLS					
Cholesterol, C ₂₇ H ₄₆ .OH		386.368	145.219	0.0068862
Phytosterol, C ₂₇ H ₄₆ .OH		386.368	145.219	0.0068862

GRAVIMETRIC PROCESS¹

After acetylating, a gravimetric process for acetyl number may be conducted in a manner similar to that for the quantitative determination of insoluble fatty acids, observing all the precautions therein noted as to ceresine, washing, drying, weighing, etc.

This modification is apparently rather more difficult, tedious and subject to error than the saponification or volumetric process (Massachusetts method). A certain amount of loss arises from the dehydration of free fatty acids by acetic anhydride during acetylation, and is difficult to prevent, although of little consequence where the amount of free acids is relatively small.

The acetyl number (*a*) is calculated from the increase in weight (*i*) by the following formula:

$$a = \frac{56108i}{42.016} \text{ or } 1335.39604i$$

In case only one hydroxy compound of known molecular weight (*m*) and number of hydroxyls (*d*) is present, its amount can be calculated from the increase in weight (*i*) of the oil, fat or wax on acetylating. The theoretical increase for a hydroxy compound is:

$$\frac{42.016d}{m}$$

The amount (*H*) of such a compound in an oil, fat or wax is therefore:

$$H = \frac{i}{\frac{42.016d}{m}} \text{ or } \frac{im}{42.016d}$$

MOLECULAR WEIGHT OF HYDROXY COMPOUNDS

The molecular weight of the hydroxy compounds can be calculated from the weight (*w*) of fat taken and the increase (*i*) on acetylating, provided the number (*d*) of hydroxyls in the molecule is known:

$$w : w + i = m : m + 42.016d$$

$$m = \frac{42.016dw}{i}$$

¹ Has not received sufficient study in this laboratory to warrant positive statements, but is similar to the methods described by Lewkowitsch (*Loc. cit.*, 1, 358-63, 466-67).

pounds by the gravimetric process is greatly facilitated by use of the following table:

ACETYL GRAVIMETRIC PROCESS ON ORIGINAL PRODUCT

Name	Molecular weight	Molecular weight after acetylating	Theoretical increase in weight per gram on acetylating ^(a)	Reciprocal
GLYCERIDES				
Ricinolein.....	932.832	1058.880	0.135124	7.40061
Dihydroxystearin...	986.880	1238.976	0.255447	3.91471
MONOGLYCERIDES				
Monopalmitin.....	330.304	414.336	0.254408	3.93069
Monostearin.....	358.336	442.368	0.234506	4.26428
Monolein.....	356.320	440.352	0.235833	4.24029
DIGLYCERIDES				
Dipalmitin.....	568.544	610.560	0.073901	13.53162
Distearin.....	624.608	666.624	0.067268	14.86591
Dirolein.....	620.576	662.592	0.067705	14.76996
HYDROXY ACIDS				
Ricinoleic.....	298.272	340.288	0.140865	7.09900
Dihydroxystearic...	316.288	400.320	0.265682	3.76390
FREE ALCOHOLS				
Cholesterol.....	386.368	428.384	0.108746	9.19574
Phytosterol.....	386.368	428.384	0.108746	9.19574

(a) Acetyl number = 1335.39604i.

ACETYL NUMBER OF INSOLUBLE FATTY ACIDS

The acetyl number of the insoluble fatty acids is determined by the Massachusetts method in precisely the same way as that of the original fat. The gravimetric process is not applicable on account of the formation of anhydrides of the fatty acids. The method for preparing the stock of insoluble acids for analysis is the same as that for the determination of "Insoluble Acids," with the elimination of such features as are necessary only for quantitative work.

In order to interpret the results satisfactorily it is necessary to know the percentage of insoluble acids so that the acetyl number of the acids may be considered in conjunction with the acetyl number of the fat.

RESULTS BY DIFFERENT METHODS

For convenience, the theoretical acetyl numbers of some hydroxy compounds by the Benedikt and Ulzer and Lewkowitsch methods are tabulated to permit comparison with the acetyl numbers by the Massachusetts and gravimetric processes previously stated. When only one hydroxy compound of known com-

ACETYL NUMBER ON ACETYLATED PRODUCT, BENEDIKT AND ULZER AND LEWKOWITSCH METHODS

Name (Acetylated)	Formula	Molecular weight	Saponification number	Theoretical acetyl number	Reciprocal
GLYCERIDES					
Ricinolein, (C ₁₇ H ₃₂ .OCH ₂ CO.CO) ₂ C ₃ H ₅		1058.880	317.928	158.964	0.0062907
Dihydroxystearin, [C ₁₇ H ₃₃ (OCH ₂ CO) ₂ COO] ₂ C ₃ H ₅		1238.976	407.572	271.715	0.0036803
MONOGLYCERIDES					
Monopalmitin, (C ₁₅ H ₃₁ COO)(CH ₂ COO) ₂ C ₃ H ₅		414.336	406.250	270.833	0.0036923
Monostearin, (C ₁₇ H ₃₃ COO)(CH ₂ COO) ₂ C ₃ H ₅		442.368	380.507	253.671	0.0039421
Monolein, (C ₁₇ H ₃₃ COO)(CH ₂ COO) ₂ C ₃ H ₅		440.352	382.249	254.832	0.0039242
DIGLYCERIDES					
Dipalmitin, (C ₁₅ H ₃₁ COO) ₂ (CH ₂ COO)C ₃ H ₅		610.560	275.688	91.896	0.0108819
Distearin, (C ₁₇ H ₃₃ COO) ₂ (CH ₂ COO)C ₃ H ₅		666.624	252.502	84.167	0.0118811
Diolein, (C ₁₇ H ₃₃ COO) ₂ (CH ₂ COO)C ₃ H ₅		662.592	254.039	84.680	0.0118092
HYDROXY ACIDS					
Ricinoleic, C ₁₇ H ₃₂ .OCH ₂ CO.COOH.....		340.288	329.768	164.884	0.0060649
Dihydroxystearic, C ₁₇ H ₃₃ (OCH ₂ CO) ₂ .COOH.....		400.320	420.474	280.316	0.0035674
FREE ALCOHOLS					
Cholesterol, CH ₃ COOC ₂₇ H ₄₅		428.384	130.976	0.0076350
Phytosterol, CH ₃ COOC ₂₇ H ₄₅		428.384	130.976	0.0076350

position is present in an oil or fat, the results can be readily converted from the basis of the original to that of the acetylated product and *vice versa*. In other cases conversion is generally impracticable on account of the marked differences in assimilation of acetyl by the several classes of hydroxy compounds. Formulas may show the relation, however, that the results by different methods bear to each other, (*m*) indicating the molecular weight of the hydroxy compound, (*d*) the number of hydroxyls, and (*i*) the increase in weight on acetylating:

Massachusetts Method Gravimetric Method

$$\frac{cm}{56108d} \qquad \frac{im}{42.016d}$$

Benedikt and Ulzer and Lewkowitsch Methods

$$\frac{c(m + 42.016d)}{56108d}$$

SUMMARY

The acetyl numbers of a fat and of the insoluble acids afford valuable information relative to the nature and the quality of a product. Apparently many analysts have been deterred from making the determinations on account of the time required, tedious manipulation involved or inability to interpret the results. The proposed method is comparatively short and simple and readily understood because of its similarity to other fat methods in common use. It is practically free from the objections cited for the earlier methods and the results are directly comparable with other fat determinations, being on the same basis.

MASSACHUSETTS AGRICULTURAL EXPERIMENT STATION
AMHERST

A COMPARISON OF NEUTRAL AMMONIUM CITRATE WITH SODIUM CITRATE AND *N*/10 CITRATE ACID¹

By PAUL RUDNICK, W. B. DERBY AND W. L. LATSHAW

Since the appearance of a previous paper² on the subject of neutral ammonium citrate, the use of sodium citrate as a substitute for ammonium citrate has been proposed by Bosworth.³ The values obtained by him with a solution of sodium citrate of the same molar concentration as the official ammonium citrate solution are, however, not in good concordance with those

¹ Presented at the 49th Meeting of the American Chemical Society, Cincinnati, April 6-10, 1914.

² THIS JOURNAL, 5 (1913), 1013.

³ *Ibid.*, 6 (1914), 227.

obtained by neutral ammonium citrate. This fact was further confirmed by us in the determinations on a sample of acid phosphate shown in Table I.

These determinations were made substantially according to the official methods of the A. O. A. C.,¹ except that the neutral ammonium citrate solution was prepared as described in our previous paper.² The phosphoric acid was in all cases determined by the volumetric molybdate method. The proportions of reagent, whether neutral ammonium citrate, sodium citrate or *N*/10 citric acid, to the weight of sample taken, were the same as in the official method.

TABLE I—DETERMINATION OF INSOLUBLE PHOSPHORIC ACID WITH SODIUM CITRATE AND NEUTRAL AMMONIUM CITRATE

Sodium citrate.....	1.46%	Ammonium citrate.....	0.30%
Av.....	1.42		0.34
	1.44		0.32

The specific gravity of this sodium citrate solution was 1.163 as against 1.09 for neutral ammonium citrate solution. Various trials showed that more concentrated solutions of sodium citrate give results approaching more nearly those obtained by the use of neutral ammonium citrate, as shown in Table II.

TABLE II—DETERMINATION OF INSOLUBLE PHOSPHORIC ACID IN ACID-PHOSPHATE WITH VARYING CONCENTRATIONS OF SODIUM CITRATE AS COMPARED WITH AMMONIUM CITRATE

	G. per l.	Specific gravity	Percentages			
			1	2	3	4
Ammonium citrate.....	...	1.09	0.53	2.58	0.77	0.62
				2.62	..	0.65
Sodium citrate.....	80.0	1.043	1.83
	159.5	1.084	..	3.14
	178.5	1.093	1.53
	247.7	1.123	1.59
	293.8	1.147	..	3.06	..	1.28
			1.37
	312.8	1.153	..	2.99
	326.6	1.163
	443.0	1.220	..	2.92
	496.8	1.231	0.92
	568.8	1.282	0.49	..
	0.46	..

NOTE—The concentration of sodium citrate is expressed in grams of crystallized sodium citrate [(C₆H₅O₇Na₃)·11H₂O] per liter.

The disadvantages of working with such heavy solutions suggested the possibility of using a citric acid solution of suitable concentration as a substitute. After trying various strengths ranging from two per cent, as used for basic slag analyses, downward, it was found that a *N*/10 solution of citric acid gave results approximating quite closely to those obtained with neutral ammonium citrate. The results are shown in Table III.

¹ Rev. Bur. of Chem., *Bull.* 107, pp. 1-5.

² THIS JOURNAL, 5 (1913), 1013.

TABLE III—DETERMINATION OF INSOLUBLE PHOSPHORIC ACID WITH N/10 CITRIC ACID AND NEUTRAL AMMONIUM CITRATE (PERCENTAGES)

	Neutral ammonium citrate	N/10 citric acid		Neutral ammonium citrate	N/10 citric acid
Acid phosphate.....	2.60	2.50	Raw bone meal....	18.75	20.50
Av.....	2.58	2.45		18.75	20.35
	2.59	2.48	Av.....	18.75	20.42
Acid phosphate.....	2.07	1.94	Complete fertilizer.	1.64	1.57
Av.....	2.01	1.95	Av.....	1.60	1.55
	2.04	1.95	Av.....	1.62	1.56
Tankage.....	6.53	6.90	Complete fertilizer.	0.14	0.23
Av.....	6.49	6.88	Av.....	0.18	0.25
	6.51	6.89	Av.....	0.16	0.24

N/10 citric acid, therefore, is entitled to careful consideration as a promising substitute for neutral ammonium citrate solution, as it not only gives results substantially identical with those obtained with the present official reagent for determining insoluble phosphoric acid, but has, moreover, at least two important advantages over ammonium citrate:

1—N/10 citric acid is much more easily prepared and standardized than neutral ammonium citrate solution.

2—Extraction with N/10 citric acid is as easy and rapid as with hot water, the time of filtration, particularly in bone, tankage and complete fertilizers, being cut down to a very few minutes.

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THE EFFECT OF ENSILAGE FERMENTATION AND ANIMAL DIGESTION ON THE SOLUBILITY OF PHOSPHORIC ACID IN PHOSPHATE ROCK

By C. A. MOOERS
Received March 20, 1914

A recent article¹ by Forbes and Fritz reports some laboratory results which indicate that the ensilage process may be used to render floats soluble and hence available both to animals and plants. With the object of showing the practical extent to which the solubility of the rock was increased, the writer has prepared Table I from their data. The table should be considered, however, as at best only a close approximation, for the article referred to did not furnish either the composition of the phosphate rock or other data which would have been of value in this connection. In making the calculations of Table I, the writer assumed the floats to contain 14 per cent of P, and that there were used 83 parts, by weight, of green silage corn to 1 part of floats. According to this table, 0.2

TABLE I—CALCULATIONS BASED ON FORBES AND FRITZ TABLE

Material	Amount of dry matter taken for basis of calculation Grams	Total P Gram	Inorganic P soluble in HCl per gram of phosphate rock	
			0.2 per cent HCl Gram	0.2 per cent HCl per gram of phosphate rock Gram
Green corn (whole plant)	83.0	0.1718	0.0689
Green corn.....	83.0	0.3118	0.1445	0.0756
Phosphate rock.....	1.0			
Ensilage.....	76.7	0.1718	0.0851
Ensilage.....	81.2	0.3118	0.1924	0.1073
Phosphate rock.....	1.0			

per cent HCl dissolved 54.0 per cent of the P of the floats when mixed with the green silage corn, but before passing through the ensilage process, and 76.65 per cent after going through the ensilage process. This amounts, therefore, to an increased solubility of 3.17 pounds of P, or 7.26 pounds of P₂O₅ per 100 pounds of phosphate rock (floats).

¹ THIS JOURNAL, 6 (1914), 222, 223.

In the proportions used by Forbes and Fritz, a 100-ton silo would require 800 pounds of floats, a quantity which, even with full allowance for the increased solubility, would amount to a moderate application for only two acres. Not to mention the very narrow margin of profit which this indicates, two questions arise at this point: (1) Would there be any detrimental effect on the feeding value of ensilage treated with floats? (2) What would be the effect of the process of animal digestion on the solubility of the floats?

To answer the first question satisfactorily would require many more data than the author has at hand, but Table II gives the results of some feeding experiments made at the Tennessee Agricultural Experiment Station in 1909. In these experiments about 2 pounds of finely ground phosphate rock were mixed with

TABLE II—FEEDING TEST OF ENSILAGE TREATED WITH FLOATS AS COMPARED WITH UNTREATED ENSILAGE

The "treated" ensilage was prepared by mixing about 2 lbs. of finely ground phosphate rock with 100 lbs. of chopped corn (whole plant) at time silo was filled.

Date (1909)	Animal	Weight of animal Lbs.	Feed per day		Ensilage uneaten Lbs.
			Ensilage Lbs.	Cottonseed meal Lbs.	
Ensilage containing floats					
Feb. 2	Cow No. 30	875	38	2	5
Feb. 3	Cow No. 30	...	28	2	18
Feb. 4	Cow No. 30	...	22 1/2	2	16
Feb. 5	Cow No. 30	...	20	2	14
Feb. 6	Cow No. 30	830	10(a)	2	10
Feb. 2	Blue heifer	480	22 1/2	1	2 1/2
Feb. 3	Blue heifer	...	22 1/2	1	4
Feb. 4	Blue heifer	...	20	1	1
Feb. 5	Blue heifer	...	20	1	7
Feb. 6	Blue heifer	463	10(a)	1	4 1/2
Ensilage without floats					
Feb. 2	Cow No. 10	1000	44 1/2	2	0
Feb. 3	Cow No. 10	...	45	2	0
Feb. 4	Cow No. 10	...	45	2	0
Feb. 5	Cow No. 10	...	45	2	0
Feb. 6	Cow No. 10	990	22 1/2(a)	2	0
Feb. 2	Fawn heifer	460	25	1	0
Feb. 3	Fawn heifer	...	28	1	0
Feb. 4	Fawn heifer	...	28	1	0
Feb. 5	Fawn heifer	...	25	1	1
Feb. 6	Fawn heifer	480	12 1/2(a)	1	2 1/2

(a) Morning feed only.

NOTE—Both groups were fed plain ensilage and meal for two days previous to beginning of record, each animal eating the amounts given for Feb. 2 without waste for that period.

every 100 pounds of the corn at the time that the silo was filled. The results show plainly that in this case too much rock was present, for the animals soon refused to eat more than a small part of the treated ensilage, although its odor and appearance indicated excellent material.

In connection with the feeding experiment, some data were obtained bearing on the second question, in regard to the availability of the P₂O₅ of the phosphate rock when voided by the animals. Samples of dung were saved from each animal, and Table III gives the per cent of total P₂O₅ in each sample when calculated to a moisture-free basis. Assuming that

TABLE III—TOTAL PHOSPHORIC ACID (P₂O₅) IN DUNG FROM ANIMALS USED IN FEEDING EXPERIMENT

Animal	Kind of feed	Results on moisture-free basis
		Per cent P ₂ O ₅ in dung
Cow No. 10	Cottonseed meal and unphosphated ensilage	1.17
Cow No. 30	Cottonseed meal and phosphated ensilage	5.95
Fawn heifer	Cottonseed meal and unphosphated ensilage	1.78
Blue heifer	Cottonseed meal and phosphated ensilage	9.34

with the same kind of feed the dung from the cows would be of similar composition, and that in like manner the dung from the heifers would be com-

parable, the amounts of phosphate rock in the dung of cow No. 30 and of the blue heifer were calculated from the analyses given in Table III. Table IV gives the quantities of the various materials taken, the amount of 0.1 per cent citric acid used as a solvent, and the percentages of P₂O₅ found. The finely ground phosphate used throughout contained 32.0 per cent of P₂O₅. The citric acid solution was shaken up in contact with the material for two days and filtered, and the P₂O₅ in the filtrate determined. From these determinations the calculation was made that the combined action of the ensilage process and the process of animal digestion resulted in an increase of 0.93 pound of P₂O₅ per 100 pounds of the rock in the case of the cow and 1.17 pounds in the case of the

TABLE IV—PHOSPHORIC ACID (P₂O₅) SOLUBLE IN 1000 Cc. OF 0.1 PER CENT CITRIC ACID

All percentages are calculated on a uniform basis of moisture-free and phosphate-free manure, *i. e.*, the plain manure from the unphosphated feed (15.0 grams) is taken as the basis of comparison.

Material	Quantity used for analysis (Water-free basis) Grams	P ₂ O ₅ found Per cent
Dung from cow No. 10 (unphosphated ensilage)	15.0	0.76
Dung from cow No. 10	15.0	0.88
Phosphate rock	2.7137	
Dung from cow No. 30 (phosphated ensilage)	17.7137	1.05
Dung from fawn heifer (unphosphated ensilage)	15.0	1.38
Dung from fawn heifer	15.0	1.47
Phosphate rock	4.9256	
Dung from blue heifer (phosphated ensilage)	19.9256	1.86

NOTES—1—A water solution of manure is normally alkaline and hence much of the "available" P₂O₅ is in a precipitated form. Only a slightly acid solution is necessary in order to get a marked increase in soluble P₂O₅. 2—If manure be treated with a 0.1 per cent citric acid solution in the proportions used, the solution is apt to become alkaline in the course of a few days, due to the production of NH₃ by bacteria; hence long standing must be avoided.

heifer. The increase in soluble P₂O₅ was, therefore, very small. On the assumption that all of the phosphoric acid was voided in the dung, these results, taken into consideration with those of Forbes and Fritz, indicate a reversion of the P₂O₅ made soluble by the ensilage.

SUMMARY

1—From calculations based on work reported by Forbes and Fritz, 22.65 per cent of the total phosphoric acid of phosphate rock (floats) was rendered soluble in 0.2 per cent HCl through the ensilage process, 1 part of rock being used to 250 parts of green silage corn.

2—Feeding experiments by the author proved that when 2 pounds of finely ground phosphate rock were mixed with 100 pounds of green silage corn at time of filling of the silo, the resultant ensilage, though excellent in odor and appearance, was not suitable to be fed in ordinary amounts, the animals soon refusing to eat it.

3—The solubility in 0.1 per cent citric acid of the P₂O₅ of the phosphate rock in the dung of the animals fed phosphated ensilage was increased only 3.28 per cent of the total P₂O₅ as an average of two trials.

4—Based on the assumption that all of the phosphoric acid was voided in the dung, the data at hand indicate that a reversion of the soluble P₂O₅ took place during the process of animal digestion.

5—The general conclusion is indicated that the silo does not offer a practical means of making the P₂O₅ of phosphate rock available for plant use.

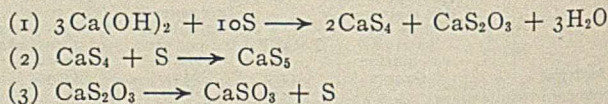
THE THEORETICAL BASIS FOR THE PROPORTIONS OF LIME AND SULFUR USED IN THE COMMERCIAL PREPARATION OF THE LIME-SULFUR SPRAY

By HERMAN V. TARTAR

Received March 11, 1914

Various formulas (*i. e.*, proportions of lime, sulfur and water) have been recommended for use in the preparation of the commercial lime-sulfur spray. In the early literature on this subject, the proportions of lime (CaO) and sulfur vary within wide limits; the more recent work¹ shows the proper ratio of lime to sulfur to be approximately 1:2. There are, however, some differences still existing among the recommendations made in this connection by the different agricultural experiment stations. It is well known, too, that various factors such as concentration and length of time of boiling, have an influence on the amounts of lime and sulfur required. Even the length of time of cooling, following the boiling period, modifies the composition of the solution and consequently the requirements of raw material. For example, in one of the experiments at our local station plant where 108 gallons of material were prepared, a sample taken and immediately cooled just at the close of the cooking period had a gravity of 33°, while one taken after the solution had cooled in the tank for 12 hours, had only a 30° strength. This decrease in gravity was due, no doubt, to the decomposition of calcium thiosulfate in the hot solution. Similar experiments, in which the entire solution was cooled immediately after boiling, showed no decrease in gravity upon standing. Because of these numerous factors which influence the composition of the spray, the formulas given by different investigators have been worked out largely by the "cut and try" method. Different amounts of lime, sulfur and water have been cooked for various lengths of time and from the analyses made of the resulting solutions and sediment (sulfite), the formulas have been derived. But little, if any, attention has been given to the exact chemical reactions which occur.

To all familiar with this subject, it is very evident that any formula used in making lime-sulfur must necessarily be based on the reactions taking place. Investigations carried out in this laboratory² have shown that these reactions are represented by the following equations:



There is also some oxidation of the polysulfides when the material is exposed to the air but this is so slight, under ordinary conditions of commercial preparation, where large, tall cooking vats are used, that it need not be considered here. The knowledge of the exact nature of these chemical reactions affords a theoretical basis for determining the proportions of lime and sulfur required in the preparation of a given sample of lime-

¹ Cordley, unpublished results of this station; Stewart, Penn. Agr. Exp. Sta., Bull. 99; Van Slyke, N. Y. Agr. Exp. Sta. (Geneva), Bull. 329.

² Jour. Amer. Chem. Soc., 27 (1914), 495.

sulfur. This will be brought out in the discussion which follows.

It will be seen from the equations given above that the compounds formed by the reaction between calcium hydroxide and sulfur, under ordinary commercial conditions of manufacture, are calcium tetrasulfide, pentasulfide, thiosulfate, and sulfite. All of these compounds are readily soluble in water with the exception of the sulfite, which is comparatively insoluble. This being true, the chemical analysis of the lime-sulfur solution shows the amounts of lime (slaked with water to form hydroxide) and sulfur that have reacted in the formation of the same except the amounts of these substances necessary to form the insoluble sulfite produced. From the knowledge of the chemical reactions that occur, however, it is not a difficult matter to estimate, from the chemical analysis of the solution, the quantity of sulfite which has been formed. Equation (1) shows that when calcium hydroxide and sulfur combine, one-third of the calcium is combined as thiosulfate and two-thirds as polysulfide. Since there is no decomposition of the polysulfide, the quantitative estimation of the calcium combined in this form gives a means for determining the amount of thiosulfate which has been formed. The difference between this total estimated amount of thiosulfate and the amount actually present in the solution, is the quantity that has decomposed; and from this data the amount of sulfite can be easily calculated. The initial ratio of lime to sulfur is also easily determined when one knows not only the amounts of calcium and sulfur present in a given lime-sulfur solution, but also the insoluble sulfite produced in the preparation of the same.

The analytical methods for determining the amounts of calcium combined as polysulfide as well as the amounts of other constituents of lime-sulfur, have been very thoroughly worked out¹ and it is unnecessary to discuss them in this paper.

The actual application of the discussion given above is brought out in Table I. The chemical compositions of several samples of commercial lime-sulfur solution are given; also the estimated amounts of insoluble calcium sulfite formed and the calculated ratios of lime

TABLE I—CHEMICAL COMPOSITION OF LIME-SULFUR SOLUTION

No.	Sp. Gr.	LIME (CaO)			SULFUR (S)			RATIO CaO : S REQUIRED
		Combined as Poly- sulfide	Thio- sulfate	Esti- mated as insol. sulfite	Combined as Poly- sulfide	Thio- sulfate	Esti- mated as insol. sulfite	
1	1.2585	9.464	1.820	4.060	26.313	2.080	2.320	1 : 2.00
2	1.3335	13.943	0.672	5.152	37.498	0.768	2.944	1 : 2.08
3	1.2825	11.357	1.120	4.558	30.780	1.280	2.604	1 : 2.03
4	1.2560	10.248	1.008	4.116	27.926	1.152	2.354	1 : 2.04
5	1.2820	11.424	0.840	4.872	31.283	0.960	2.784	1 : 2.04
6	1.3110	11.802	1.198	4.703	31.930	1.370	2.688	1 : 2.04
7	(a)	10.834	1.286	4.131	28.980	1.470	2.360	1 : 2.01

(a) Not determined

to sulfur. The results relating to chemical composition are expressed as grams per 100 cc. of solution.

The data given show that the proportion of lime (CaO) to sulfur which react in the preparation of the more concentrated commercial lime sulfur solutions is but a mere trifle greater than 1 : 2. Unfortunately the author has not had opportunity to examine more

dilute solutions prepared under commercial conditions. It is evident, however, from the work of Thatcher¹ and Van Slyke² that the ratio in this case would be somewhat greater than 1 : 2; in some cases it would be perhaps 1 : 2.25.

The theoretical basis given here will not exactly apply, of course, to the preparation of small amounts of solution, say 150 gallons or less, where the oxidation of the polysulfides occurs to a considerable extent through contact with the air.

Acknowledgment is due to Mr. R. H. Robinson who made several of the chemical analyses reported above.

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THE DETERMINATION OF CAMPHOR IN TABLETS AND PILLS

By EDWIN DOWZARD

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In so far as the writer has been able to learn, the methods used elsewhere for the determination of camphor in tablets and pills have not proved satisfactory. For this reason a description of a method is given which has been in use for over five years with satisfactory results:

Camphor may be rapidly and completely removed from tablets and pills by distillation in a current of steam. The watery distillate contains both dissolved and undissolved camphor, which can be extracted with benzol. By determining the optical rotation of the benzol solution, the amount of camphor present in the tablets or pills can be readily calculated.

A special apparatus is required for the distillation, because an ordinary condenser cannot be used as the camphor blocks up the tube.

After a number of trials the arrangement shown in the figure proved satisfactory.

The apparatus consists of a flask, *A*, for generating steam, a second flask, *B*, for the steam distillation, and a receiver, *C* (a retort with the tube bent as shown), for the distillate.

The retort is kept cool by two streams of water (*D* and *E*). *D* impinges on the wide part of the neck, and *E* on the extreme end of the tube, which is closed with a rubber stopper fitted with a glass tube open at both ends; thus every part of the retort is kept covered with a film of water, ensuring a complete condensation. The large funnel *F* conveys the water to a sink by means of a rubber tube.

The funnel should be kept about half filled with water, the flow of water from the funnel being regulated by pinchcock *G*.

PROCEDURE FOR A DETERMINATION—A number of tablets or pills containing about 2½ to 3 grams of camphor are placed in the flask *B*. The tablets are just covered with water and the apparatus connected. Sufficient water to cover the bottom of the tube *H* is placed in the retort.

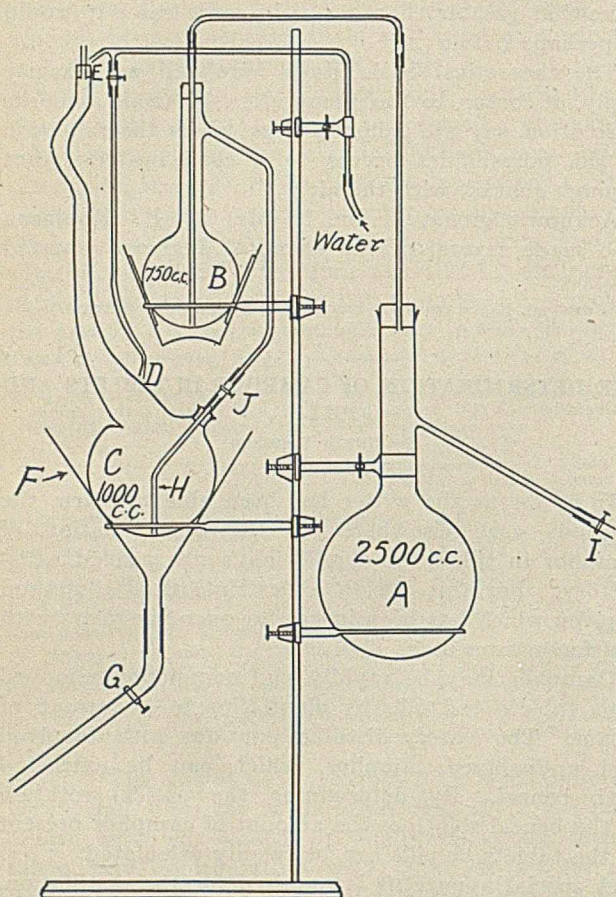
The water in *A* is now boiled, with *I* closed, the steam passing into *B* through the tube, which almost

¹ Jour. Amer. Chem. Soc., 27 (1905), 244; THIS JOURNAL, 2 (1910), 271; Mich. Agr. Exp. Sta., Tech. Bull. No. 6.

² Jour. Amer. Chem. Soc., 30 (1908), 63.

³ Loc. cit.

touches the bottom of the flask, carries over the camphor from the disintegrated tablets. The distillation is continued until the distillate measures about 500 cc.



The pinchcock *I* is now opened and the flame removed from *A*. The water is allowed to run over the retort for 5 minutes longer to thoroughly cool the distillate.

The retort is now disconnected, leaving the tube *H* with the pinchcock *J* attached and screwed tight; 25 cc. of benzol measured at 25° C. are allowed to flow into the retort through the neck, after removing the rubber stopper fitted with the glass tube opened at both ends. The retort is securely closed with a rubber stopper and the contents vigorously agitated for several minutes. The position of the retort should be frequently changed to allow the benzol to reach every part, thus ensuring that all the camphor is dissolved in the benzol. The mixture is transferred to a separator, the water drawn off, and the benzol solution of camphor filtered through a small plug of cotton wool into a 100 mm. tube, the rotation being taken at 25° C.

If the pills or tablets do not disintegrate readily, they should be coarsely ground in a mortar, before distilling.

The writer has determined the rotation of a number of samples of camphor. All examined had practically the same rotation.

The following figures give the camphor equivalent in 50 cc. of benzol solution for each 0° 1' of rotation:

Rotation taken in 100 mm. tube at 25° C.			
Grams camphor dissolved in benzol	Total volume Cc.	Rotation	Camphor
6	50	0° 1'	=0.019402
3	50	0° 1'	=0.019745
1.5	50	0° 1'	=0.019692

Average 0° 1' = 0.019613 gram of camphor in 50 cc. or 0.009806 gram camphor in 25 cc. for each 0° 1' of rotation.

To test the apparatus, 2 grams of camphor were distilled and the distillate extracted with 25 cc. of benzol, as described. The following figures were obtained:

Rotation in 100 mm. tube at 25° C. = 3° 5' or 185'.

As the above solution consists of 2 grams of camphor and 25 cc. of benzol, not 2 grams made up to 25 cc., a correction is necessary and is determined as follows:

$$185' \times 0.009806 = 1.8144$$

From the figures previously obtained, we know that the above rotation indicates the presence of 1.8144 grams of camphor in 25 cc. of the solution. It is now necessary to calculate how much benzol we have in 25 cc. of this solution.

As the specific gravity of camphor at 25° C. is about 0.99, 1.8144 grams of camphor displace 1.8324 cc.

$$\frac{1.8144}{0.99} = 1.8324$$

Therefore, we have 23.1676 cc. of benzol in 25 cc. of the solution.

$$\begin{array}{r} 25.0000 \text{ cc.} \\ 1.8324 \text{ cc.} \\ \hline 23.1676 \text{ cc.} \end{array}$$

Now if 23.1676 cc. of benzol have 1.8144 grams of camphor, 25 cc. will have 1.957 grams.

$$\frac{1.8144 \times 25}{23.1676} = 1.957$$

or, 97.85 per cent of the amount taken.

Several determinations were made with varying amounts of camphor, with very close results. It will be seen that a slight correction is necessary for loss.

As each apparatus and the manipulation of different chemists will vary somewhat, several determinations, using known weights of camphor, should be made and the factor for the apparatus calculated.

The factor for the apparatus used by the writer is 0.010001 gram of camphor for each 0° 1' of rotation in 100 mm. tube at 25° C.

The average amount of camphor in the tablet or pills is obtained by dividing the number taken into the weight of camphor found.

Using the above method, a determination may be completed within two hours.

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LABORATORY AND PLANT

THE STATUS AND TENDENCY OF THE GAS INDUSTRY¹

By WALTER R. ADDICKS

The artificial gas business was never in so advantage-

¹ Presented at the meeting of the N. Y. Section of the Society of Chemical Industry, The Chemists Club, March 27, 1914.

ous a position strategically as at this writing, yet two influences are felt adverse to progress: the first is an obsolete candle power standard; the second, which is not confined to the gas industry, a tendency to deny to proprietorship a percentage return on investments commensurate with the character of the service and

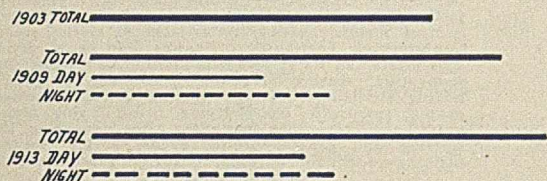
the ability of management necessary to produce results demanded by modern conditions.

Had the incandescent gas mantle, equally useful for coal or carburetted water gas, been invented and perfected before the advent of a supply of cheap oil

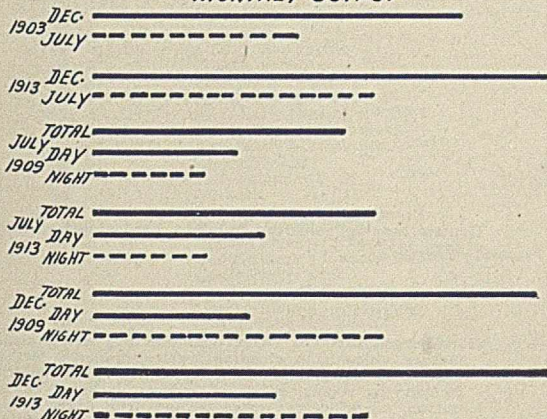
affected the supply of oil for carbureted water gas manufacture, coal gas, while still an ultimate, would not as yet be an immediate necessity to the gas busi-

DIAGRAM SHOWING APPROXIMATE TYPICAL LARGE CONSUMERS OF GAS.

ANNUAL OUTPUT.



MONTHLY OUTPUT



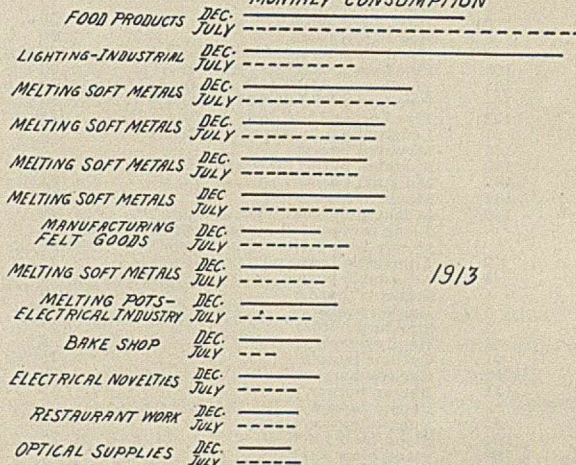
NOTE: SCALE FOR MONTHLY OUTPUT IS 10 TIMES THAT OF ANNUAL OUTPUT

for gas manufacture, the modern carbureted water gas apparatus with its former high candle power possibilities would not have displaced coal gas manufacture or have reached its present important place in the gas industry; it is also probable that electricity would not have had its encouragement to enter the

ANNUAL CONSUMPTION



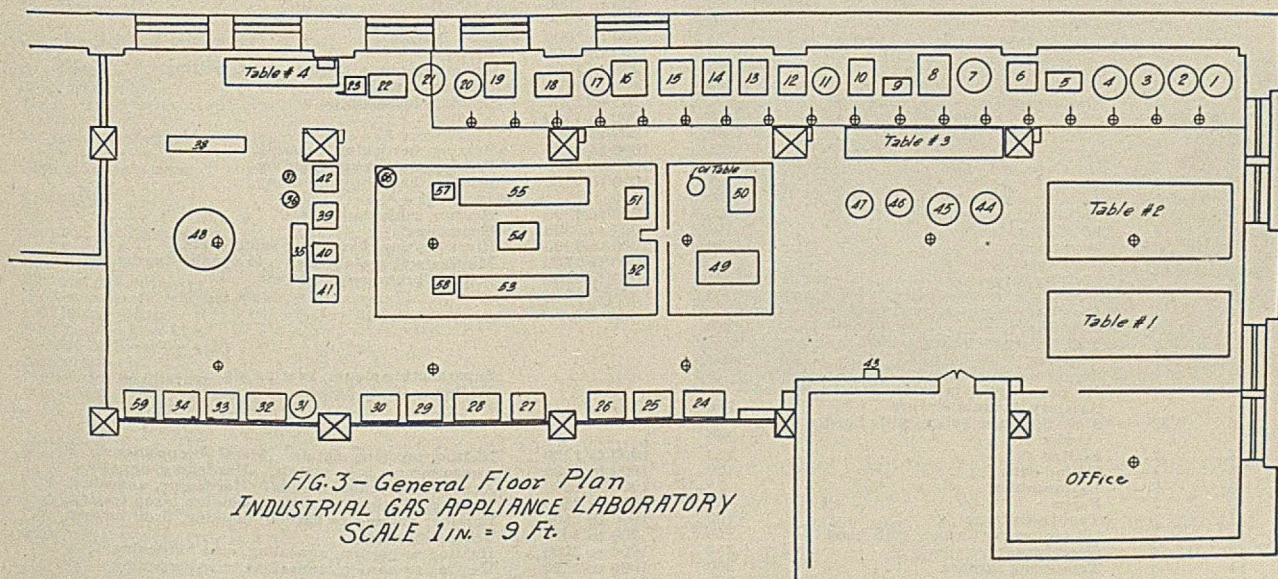
MONTHLY CONSUMPTION



1913

NOTE: SCALE FOR MONTHLY CONSUMPTION IS 10 TIMES THAT OF ANNUAL CONSUMPTION

ness. These observations are made to direct your attention to a study of cause and effect in the gas industry.



lighting field, the first important step in electrical development. Had the perfection of operation of the automobile followed by other natural mechanical developments involving large consumption of oil not

Electricity has forged ahead in the lighting field and the power field, but in the heating field gas holds a stronger position than ever before, and it is still no mean competitor of electricity in the lighting and

No. ON PLAN	TRADE No.	KIND OF APPLIANCE	Max. gas consumption per hr. Cu. ft.	Practical range of working temperature ° F.	SOME USES (NOT ALL)	
					BURNERS	
					Melting gum, heating cylinders, tinning, soldering, floor finishing, replacing coal in confectionery stores, raising steam, candy manufacture, annealing, smoking meats, heating rubber compounds, etc., hat drying, heating plating tanks, drying photos, umbrella steaming, syrup manufacture, heating pleating and singeing ovens, steam boilers, cold cream, etc.; bending glass, etc.; drying nuts, foodstuffs, etc.; soap manufacture, glue manufacture, melting metals, heating water-jacketed kettles, manufacturing ink, heating drying tanks, heating oil, drying blue-prints, hot water for soda fountains, singeing, hat renovating, leather steaming, pitch kettles, general boiling, sterilizing, heating, sweating on tables, sawdust drying, embossing, wood drying, brush drying, drying vats, kegs, etc.; china and glass kilns, Japanning, lacquering and general drying ovens; drying feathers, bending wood, paint burning, testing oil, general roasting.	
Table No. 3	103	High power burner	175			
	68	Cyclone burner	75			
	157	Mounted burner	150			
	733 B	Hot plate burner	60			
	732 B	Hot plate burner	40			
	731 B	Hot plate burner	20			
	105	High power burners	300			
	34	Solid type burner	700			
	13	Triple industrial burner	300			
	106	High power burner	200			
	113	Pipe burner 1 1/2" X 30"	100			
	119	Pipe burner 2" X 36"	200			
	000	Hand blow torch	25			
	00	Hand blow torch	35			
	0	Hand blow torch	40			
	1	Hand blow torch	50	200 to 2000		
	2	Hand blow torch	100			
	3	Hand blow torch	150			
Table No. 4	81	Hand blow torch	30			
	82	Hand blow torch	75			
	83	Hand blow torch	100			
	84	Hand blow torch	175			
	85	Hand blow torch	225			
	8 B	Hand blow torch	15			
	8 C	Hand blow torch	30			
	8 E	Hand blow torch	50			
	8 F	Hand blow torch	100			
	109	Pipe burner 1 1/4" X 24"	60			
	105	Pipe burner 1" X 18"	30			
	101	Pipe burner 3/4" X 12"	15			
	148	Pipe burner 1" X 18"	25			
	91	Longitudinal burner	75			
	146	Mounted burner	75			
	140	Mounted burner	20			
	147	Mounted burner	35			
	142	Mounted burner	18			
	149	Mounted burner	60			
		3-ring concentric burner	100			
		3" core with 8 No. 7 Bunsens	100			
	2	Adjustable burner	15			
	8	Adjustable burner	25			
	4	Students' burner	10			
	6	Bunsen burner	10			
Table No. 1	5	Standard burner	15			
	3	Handy burner	8			
	61	Cyclone burner	15			
	62	Cyclone burner	20			
	51	Bunsen burner	5			
	52	Bunsen burner	7			
	53	Bunsen burner	9			
	000	Stand blow torch	25			
	00	Stand blow torch	35	200 to 2000		
	0	Stand blow torch	40			
	1	Stand blow torch	50			
	2	Stand blow torch	100			
	3	Stand blow torch	150			
	1	Buzzer soldering iron heater	15	200 to 1500		
	2	Buzzer soldering iron heater	30			
	1	Johnson soldering iron heater	15			
	101	Johnson soldering iron heater	30			
	410 A	Soft metal furnace	40	250 to 800		
	626	Glue heater	15	150	Melting soft metals.	
	17	Candy batch warmer	35	120 to 150	Glue heatin.	
		2 ft. ribbon burner	200	200 to 1000	Warming candy	
	6	Soft metal burner	250	200 to 1600	Singeing, roasting and cylinder heating.	
	93	Longitudinal burner	150	200 to 1600		
	10	Cluster burner	75	200 to 1000		
	4	Umbrella burner	50	200 to 1000		
	3	Pattern radiator burner	100	200 to 1000		
	2	Babbitt melter	60	400 to 1700	Japanning ovens, drying ovens, melting.	
		Two motor heads	150 ca	200 to 2000	Soft metal melting.	
Table No. 2	Type S	Soldering iron heater	25	200 to 1500	Brazing, bending.	
	2	Soldering iron heater	40	200 to 1500		
		Bench forge	25	1000 to 2300	Brazing, hardening, bending.	
	2	Bench forge	100	1000 to 2300	Brazing, hardening, bending.	
	1	Bench forge	25	1000 to 2300	Brazing, hardening, bending.	
	Model R	3-quart glue heater	10	150		
	3	Soldering iron heater	60	200 to 1500	Heating soldering irons.	
	1	Muffle furnace	50	1000 to 2300	Enameling.	
	1	Rivet heater	100	1000 to 2300	Rivet heating, hardening small parts.	
	12	Soft metal furnace	90	1000 to 1700	Melting soft metals, lead and cyanide hardening.	
	1	Melting furnace	25	1000 to 2200	Melting precious metals, etc.	
	1	3 1/2 H. P. steam boiler with standard automatic water feeder and feed water heater				
	2	1 H. P. steam boiler, with Lawler water feeder	280			
	3	4	Steam superheater	75		
	4	40 gal. copper-jacketed kettle—tinned			Superheating steam, heating air.	
	5	Sanitary vacuum cleaner	150		To demonstrate boilers.	
	6	3 H. P. steam boiler, with Lawler water feeder	240		?	
	7	2	Melter	100	1000 to 2500	Melting precious metals. Metal compositions.
	8	1	Round annealer	100	1000 to 1600	Annealing precious metals. Hardening, annealing.
	9	2	Bar annealer	75	1000 to 1600	Annealing precious metals. Hardening, annealing.
	10	7	Forge	125	1000 to 2500	Brazing, heating to harden, bending, drop forging.
	11	1	Tool room forge	100	1000 to 2500	Brazing, heating to harden, bending, drop forging.
	12	4	Oil tempering furnace with hood	50	200 to 1000	Tempering steel.
	13	34	Oven furnace	250	1000 to 2000	Heating to harden, annealing, case hardening.
	14	3	Tempering furnace	200	1000 to 1500	Heating to harden, annealing, case hardening.
	15	692	Rivet heater	200	1500 to 2800	Rivet heating.
	16	722	Muffle furnace	200	1000 to 1500	Enameling, hardening, tempering.
	17	36	Oven furnace	240	1000 to 2400	Heating to harden—case hardening.
	18	43	Lead hardening	95	1000 to 1800	Lead hardening, cyanide hardening, metal melting.
	19	7	Soft metal furnace	125	1000 to 1600	Lead hardening, cyanide hardening, metal melting.
	20	3	Muffle furnace	125	1000 to 1600	Enameling, hardening, tempering.
	21	87	Portable melter	60	200 to 800	Soft metal melting.
	22	430 A	Soft metal furnace	150	200 to 800	Soft metal melting, oil tempering, sweep reducing.

No. ON PLAN	TRADE No.	KIND OF APPLIANCE	Max. gas consumption per hr. Cu. ft.	Practical range of working temperature ° F.	SOME USES (NOT ALL) BURNERS
23	2	Brazing table	100	200 to 2000	Brazing, bending, hardening.
24	Model C	10 gallon glue heater	35	150	Glue, wax, compounds.
26	Model C	Height 2 japanning oven	150	120 to 800	
25	3 A	Clothes pressing machine	50		
27	046	Japanning oven	125	120 to 800	Japanning, lacquering, drying.
28	4	Incinerator	120		Garbage, sweep reducing.
29	6	China kiln	360	1000 to 2000	China firing, silver deposit.
30		95 gallon cauldron furnace	150	200 to 800	Water heating, syrups, potash, general boiling.
31	F. 503	Cauldron furnace with pump and agitator	300	200 to 800	Water heating, syrups, potash, general boiling.
32	R. 590	Butcher's boiler	90	200 to 212	General boiling.
33		Sawdust drier	70	150 to 212	Cleaning metal parts.
34	1052	Laundry stove	80		General boiling.
35	F 7	Rect. cyanide furnace with hood	260	1000 to 1800	Tinning, cyanide hardening, lead hardening.
36	G 4	Crucible furnace	210	1000 to 2000	Metal melting
37	E 4	Lead hardening furnace with hood	135	1000 to 1800	Lead hardening, cyanide hardening.
38	649	Wagon tire heater	500	1200	Expanding tires.
39	C 9	Semi-muffle furnace	280	1000 to 2000	Hardening and case hardening, tempering.
40	D 9	Muffle furnace	280	1000 to 1700	Enameling, hardening, tempering.
41	A 4	Forge	250	1000 to 2500	Heating to harden, brazing, bending.
42	31/2	Root's acme blower			
48	Type C 304	3/4 H. P. Diehl motor automobile tire vulcanizer	250	200 to 300	Re-treading auto tires.
49	13704	6 H. P. gas engine	120		
50	325706	7 1/2 H. P. motor			
51	2	Positive pressure blower			
52	F	Positive pressure blower			
53	29	Automatic heating machine	400	1000 to 1700	Heating to harden, annealing.
54	26	Automatic heating machine	60	300 to 1000	Tempering, bluing.
55	8	Automatic heating machine	300	1000 to 1700	Heating to harden.
56		Clay annealer	75	200 to 250	Boiling out for jewelers.
57		Salt water bath			
58		Oil bath			Hardening baths.
59		Fresh water bath			
43	0	Style C water sterilizer	2	212	Sterilizing water.
44	331	Confectioner's furnace fan blower	250	150 to 400	Candy, syrups, etc.
45	635	Confectioner's furnace positive pressure			
46	330	Confectioner's air	360	150 to 400	Candy, syrups, etc.
47	695	Confectioner's furnace	300	150 to 400	Candy, syrups, etc.
Out 60	650	Confectioner's furnace	135	150 to 400	Candy, syrups, etc.
		Automobile tire heater	1300(a)	1200	Expanding tires (steel bands).

(a) Gas consumption given is maximum and not operating consumption.

APPLIANCES ACTUALLY INSTALLED

APPLIANCE	BUSINESS	1913 Consumption cu. ft.
Mould drying oven and crucible furnace	Jewelry manufacture	594,300
Indirect heated oven	Jewelry " drying lacquer	
Annealing oven	Brass goods manufacture	839,700
4 HP boiler	Feather "	275,900
Direct heated oven mould drying	Bronze goods	2,172,300
Steam pleating box	Dress plaiting, etc.	203,700
1 HP boiler	Clothing manufacture	493,900
9 HP boiler	Hat	984,500
5 HP boiler	Silversmiths	589,900
1 ton coal stereotype melting furnace converted to gas	Newspaper	875,200
Furnace, forge and vulcanizer	Artificial limbs	563,300
Soft metal furnace	Electrotypes	472,800
Tire heater	Wagon repair—stable of department store	55,200
Oven furnace—oil tempering	Hack saw blade manufacture	135,400
Water still	Carbonated waters	21,900
Glass firing kilns	Glass stainers and enamellers	33,500
Wood drying oven and glue heater	Woodworkers—marquetry	126,500
Indirect heated japanning oven	Platers and japanners	596,000
Glass annealing oven	Thermos bottle manufacture	1,700,900
Tire heater	Auto and wagon wheel manufacture	541,000
Air blast gas furnaces	Case hardening, engravers' steel plates	33,000
Oven furnaces	Lava tip manufacture	654,000
Air blast gas furnaces	Electrical novelties	9,229,600
Gas ovens for drying raw silks	Silk testing	1,509,200
100 gallon cauldron	Syrup manufacture	369,500
Steel muffle china kiln	China ware, etc.	1,118,500
Melting furnaces for gold	Assaying
Assay furnaces	See No. 46
Burners for distillation	See No. 46	4,251,100
Water still	See No. 46

Notwithstanding this disadvantage the incandescent gas light is better than electric light in many circumstances and more often so than is generally realized, in the home the factory and the office; distant control of gas is now used and will shortly be perfected for gas lighting, with all the safety desirable; the pilot light is very satisfactory with the ordinary hand control in single fixtures.

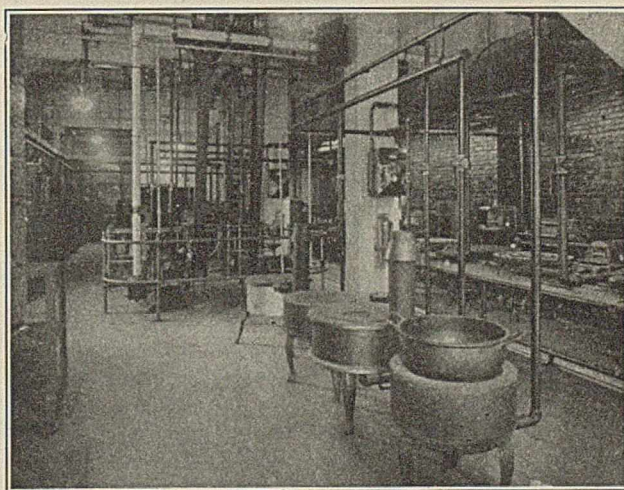


FIG. 4—GAS LABORATORY, GENERAL VIEW

power field. Where the gas engine is used as the prime mover for manufacturing electricity, all phases of light, heat, and power in all industrial industries are competitively open to the artificial gas industry.

The electric system of lighting has superseded gas lighting in many cases because electric lighting units may be conveniently located as required and be still controlled from a distant convenient point with ease and yet no great care must be exercised to provide against local overheating as in case of gas lighting.

For power purposes gas used in the gas engine is an active competitor as against the steam engine, but must be used with a belt drive when applied to a multiplicity of machines unless used to drive an electric generator.

In domestic apartment cooking, gas has already displaced coal and is likewise rapidly driving coal from use in private houses and hotel and restaurant work. The gas industry does not fear the invasion

of electricity in this field, and even in the fireless cooker field gas is developing types that will meet any probable electrical developments in sight.

In house and office heating, gas is constantly advancing and with the increase of manufacture of gas from bituminous coal large quantities of gas house

mantle lighting, and (2) surface combustion which greatly increases the efficiency of gas used in heating,

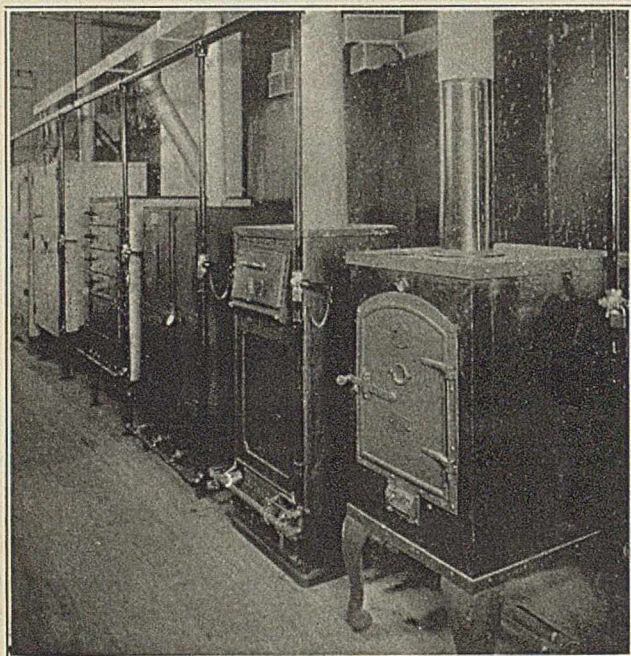


FIG. 5—JAPANING OVENS, CORE DRYING, INCINERATOR, CHINA KILN

coke will come on the market, which, with gas as an auxiliary heating agent, will reduce the use of mined coal in cities.

For general industrial uses, including domestic and industrial laundry purposes, gas is progressing very rapidly as will be shown later.

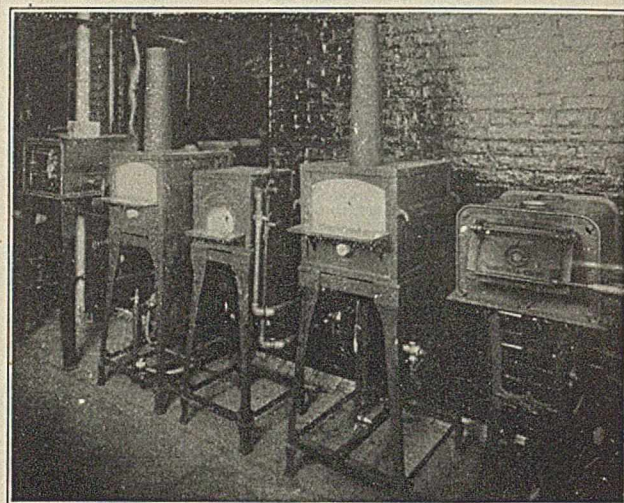


FIG. 7—OVEN FURNACES AND RIVET HEATERS



FIG. 8—FORGES, ANNEALING AND MELTING FURNACES

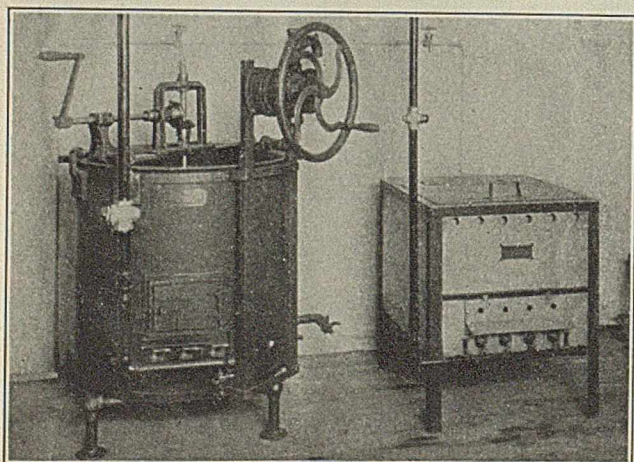


FIG. 6—CAULDRON FURNACE, BUTCHER'S FURNACE

Two interesting developments are attracting the attention of all engineers and give promise of wide application; both require combustion under greater pressures than are common in street distribution today: (1) High pressure lighting in which the efficiency is double the present ordinary incandescent

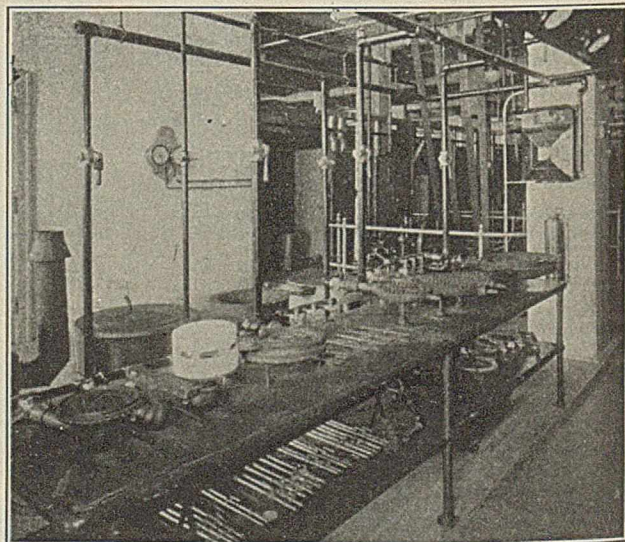


FIG. 9—ASSORTED BURNERS AND BLOW TORCHES

cooking and the industrial appliances where radiant heat will be of value.

High pressure gas lighting may be seen outside of many of the gas offices; good examples may be noted at 16th Street and 4th Avenue, and 41st and 42nd Streets, West of 6th Avenue, New York City.

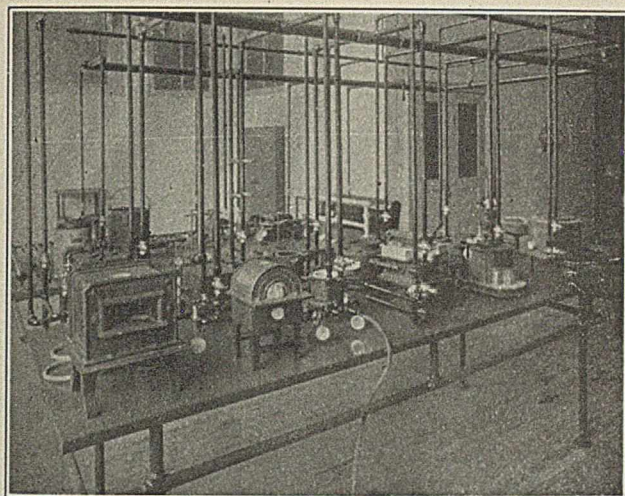


FIG. 10—SOLDERING IRON, MUFFLE, MELTING, ETC.

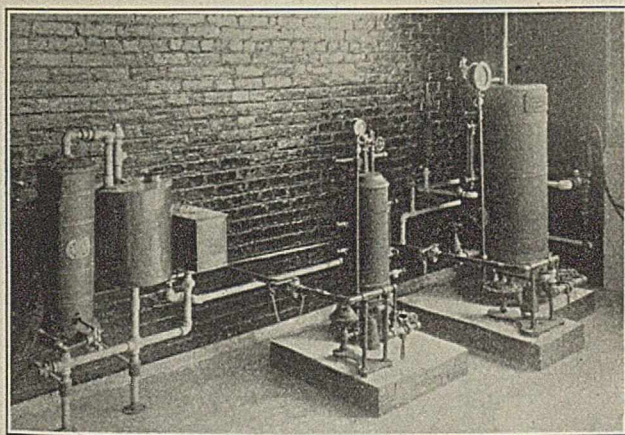


FIG. 11—FEATHER STEAMER, STEAM GENERATORS

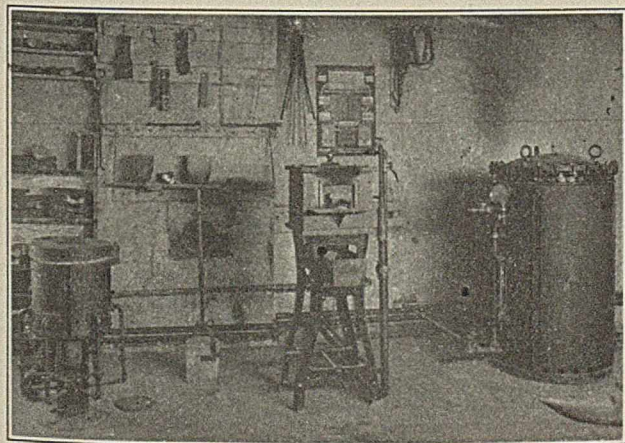


FIG. 12—ARTIFICIAL LIMBS MELTING FURNACE, FORGE, GAS STEAM VULCANIZER

Surface combustion to my mind has most fascinating possibilities to-day. The problem is one of development of design and application to present uses.

It is quite within the range of practical dreams to foresee gas supplying heat for hot water, cooking and industrial operations in summer; heating of living spaces in spring and fall and auxiliary to gas house

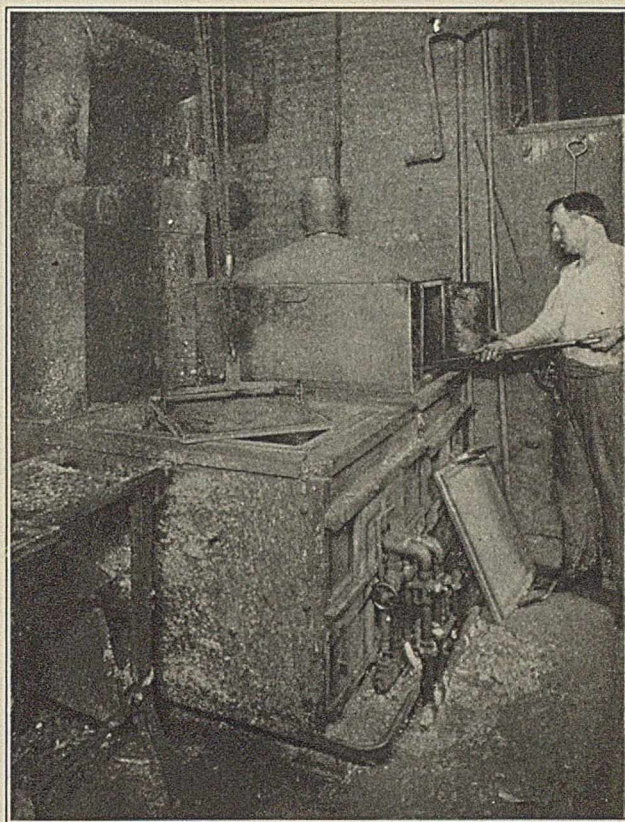


FIG. 13—ELECTROTYPES, SOFT METAL FURNACE

coke burned in low pressure boilers in winter. Electricity will supply power for elevator and power use and for lighting, where gas is not so easily applicable, though gas for lighting and power may be found most valuable under certain circumstances.

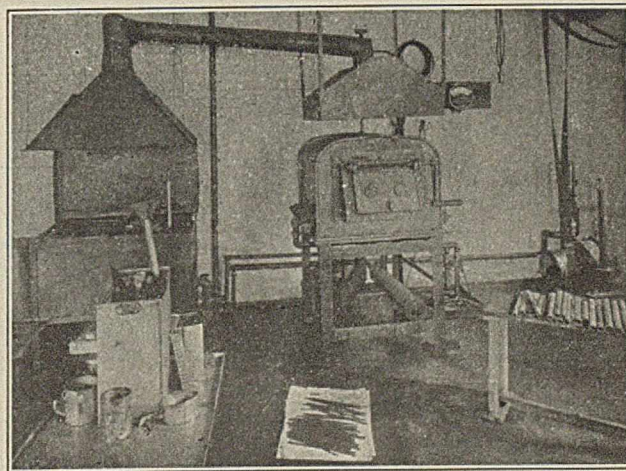


FIG. 14—HACK SAW BLADES, OIL TEMPERING, OVEN FURNACE

It is being recognized that the electrical and gas engineers should work together harmoniously to produce the best results in giving the most efficient service to the public. It follows as an essential

element that the recent pernicious practice of leaving gas pipes out of buildings must be combatted and architects convinced that their clients' interests are sacrificed by so doing.

The new office building of the Consolidated Gas Company located at 15th Street and Irving Place covers an area of 300 by 84 feet, is nineteen stories

well as the day and night outputs in July and December, 1909 and 1913.

A number of examples of sales of gas have been selected from special industries showing the annual

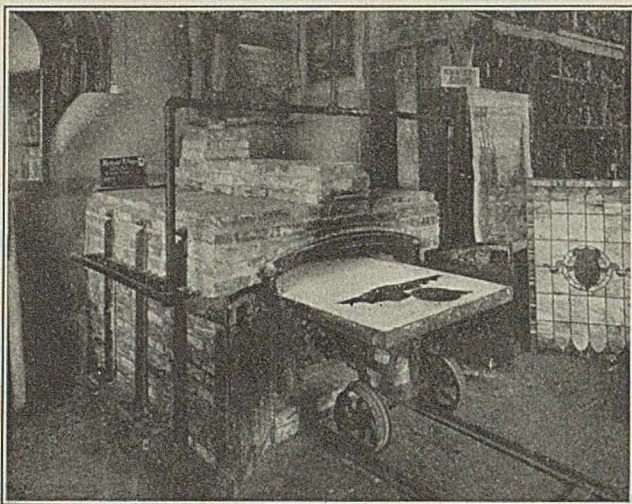


FIG. 15—GLASS FIRING KILN

in height and is completely equipped for both gas and electric lighting; the gas pipes are tested to carry many pounds per sq. in. as against the present street pressure of about $\frac{1}{9}$ lb. per sq. in. No products for light, heat and power will be used save those supplied by the central stations of either the gas or electric industry. This building, when completed in all its parts, will be well worth attention as all types of gas

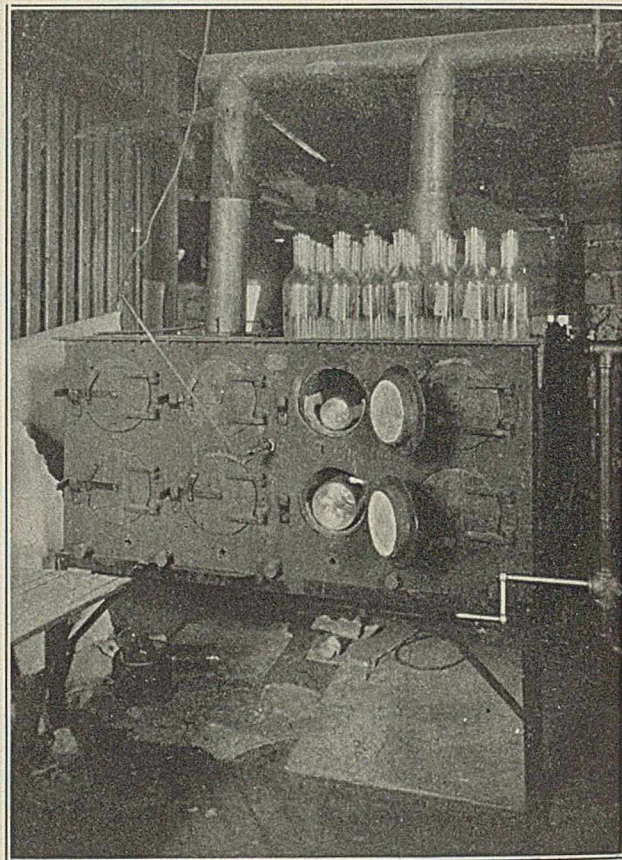


FIG. 17—GLASS ANNEALING OVEN, THERMOS BOTTLES

as well as December and July sales. One is largely for illuminating purposes, while the remainder are fuel users largely. It will be noted that the peak load is not always in December as was formerly the case when the sales were for illumination only.

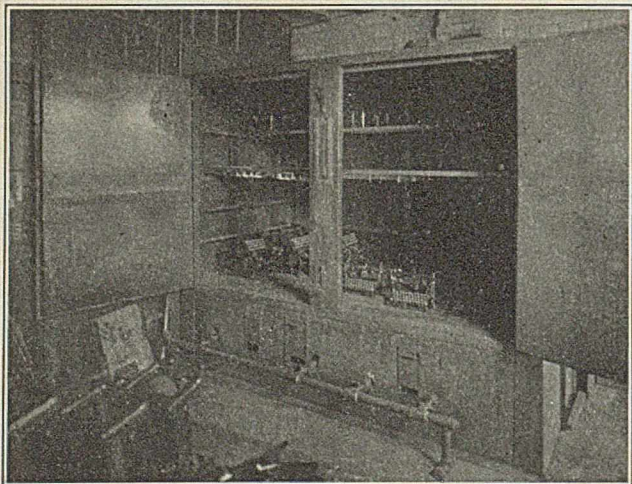


FIG. 16—INDIRECT HEATED JAPANING OVEN

appliances for every conceivable use will be displayed.

It is interesting to note the growth of the gas business over a period of years.

Fig. 1 shows the relative total 12 months output of gas in 1903, 1909 and 1913, as well as of the day and night outputs in 1909 and 1913. It also shows the total December and July output in 1903 and 1913, as

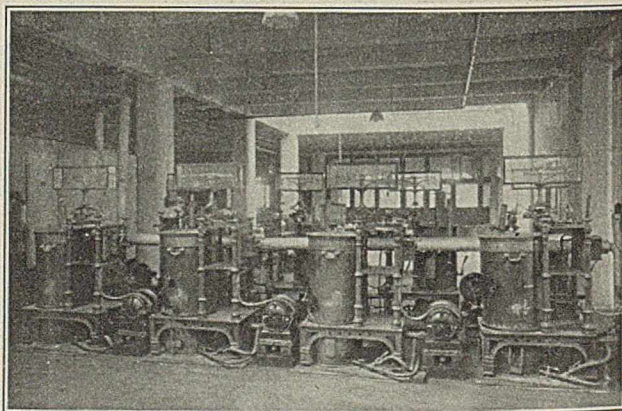


FIG. 18—RAW SILK DRYING OVENS, SILK TESTING

All branch gas offices have on display many gas burning devices. At Madison Avenue and 42nd Street (New York) will be found a model apartment as well as a large line of domestic and industrial appliances.

At 2nd Avenue and 22nd Street is shown a practical working experimental laboratory for public use. The gas company invites any consumer to bring to this laboratory materials for treatment in any appliance suitable, with electricity and gas furnished free of charge for experimental use, and instruments for careful measurements. Figs. 3-10 give a good idea of this useful aid to the chemist and practical operator and manufacturer.

A table (referred to Fig. 3) of all the appliances in the laboratory, listing the trade number, the name of appliance, the maximum, but not working, gas burning capacity, the range of working temperatures in practical use and some of the uses for which the appliance is now in service. This table is made up for practical every-day reference with the hope that

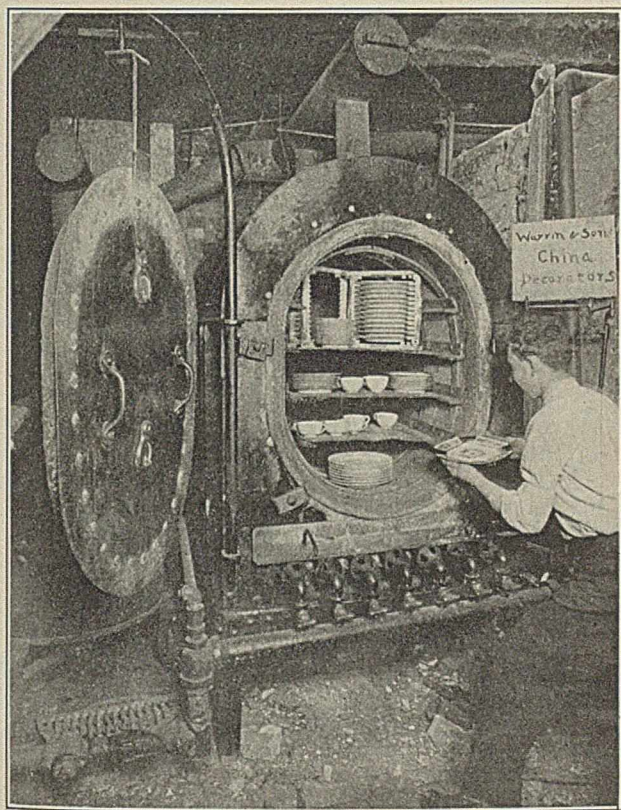


FIG. 19—STEEL MUFFLE CHINA KILN

it may be of use in selecting possible appliances for operations that may be profitably performed by gas as the heating agent.

The maximum hourly gas capacity varies from 5 cu. ft. to 1300 cu. ft. and the working temperatures from 120° to 2800° F. Higher temperatures are attainable if desired.

The gas engineer no longer assumes that gas cannot be economically used in place of coal for the reason that on a unit cost basis gas has a heavy handicap to overcome when compared with coal on a heat unit basis.

From the point of view of heat units only, no one would suppose that a gas-fired steam boiler could be economically used, yet they are being used by scores. Elimination of dirt and hard labor, preparedness for

an uncertain or irregular demand for manufactured product, immediate answer to sudden maximum demand, absolute uniformity of temperature within a wide range, in fact flexibility to a degree, make gas a

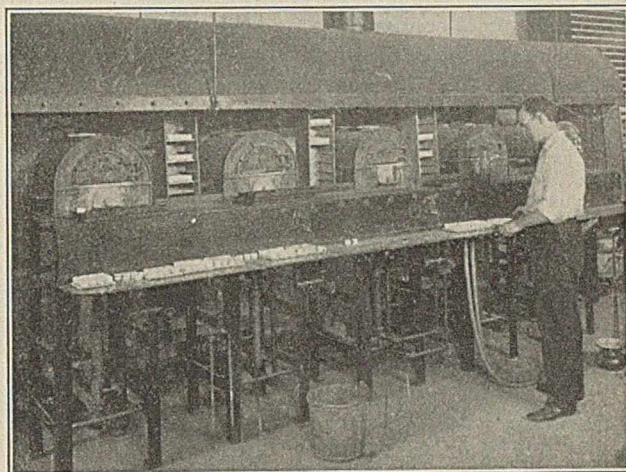


FIG. 20—U. S. ASSAY, ASSAY FURNACES

most desirable agent for the manufacturing chemist and industrial manufacturer as well as all engaged in food preparation in domestic, restaurant and hotel service.

4 IRVING PLACE, NEW YORK

A SIMPLE EXTRACTION APPARATUS

Stand and Condenser by PERCY H. WALKER¹

Flask by LORIN H. BAILEY²

Received February 20, 1914

A simple apparatus has been designed in the Bureau of Chemistry for general extraction work.

The condenser proper is of the coil metal tube type, originally designed by G. T. Cottle, but generally known as the Underwriters' Laboratories model,³ modified by lengthening the inlet tube and lengthening and bending the outlet tube, so that the condenser forms a syphon. The stand for supporting the condensers is made entirely of metal and is without clamps. Fig. 1 shows this stand. A is the feed water pipe; the water passes through the pipes B and C; a plug, D, closes the end of C; to each of the cocks E is soldered a 1/8-inch copper pipe, bent as shown, to serve as a water inlet for an individual condenser; the main drain pipe F has an outlet to the sink at H, and is plugged at I; the vertical tubes G, which act as supports and at the same time drain the individual condensers, should have an inside diameter of not less than 1/2 inch.

The condenser and stand may be used with practically any type of continuous extraction. Fig. 2 shows several home-made condensers of this type, adapted to various forms of extractors, the whole battery being heated by an electric hot plate. Beginning at the left we have: (1) an apparatus having a Soxhlet syphon with Knorr flask and mercury seal; (2) a conical flask with a Gooch crucible hung to the coil; (3) a flask with a flattened constriction at the bottom of the neck and an alundum thimble; (4) a

¹ Chief, Contracts Laboratory, Bureau of Chemistry.

² Assistant Chemist, Plant Chemistry Laboratory, Bureau of Chemistry.

³ THIS JOURNAL, 4 (1912), 535 and 856.

flask holding a 25 cc. Gooch crucible (this is much the most convenient form—see Fig. 5 with description); and (5) a condenser alone as it hangs on the drain pipe when not in use, the extreme right space showing the inlet and outlet tubes without any condenser.

The all-metal condenser is strong and inexpensive and can be used for almost all extraction work. While various sizes may be used, it is believed the following specification, which is being used by the Bureau of Chemistry, will prove satisfactory for general work.

SPECIFICATION FOR METAL EXTRACTION
APPARATUS CONDENSER

The condenser is to be made entirely of copper, nickel-plated, in accordance with the dimensions indi-

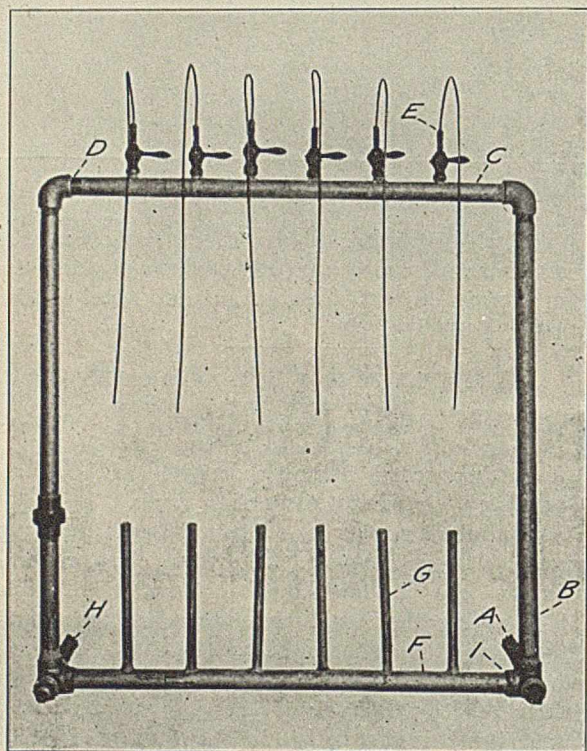


FIG. 1—STAND FOR SUPPORT OF CONDENSERS

cated on the accompanying drawing (Fig. 3). The parts indicated on the drawing as (a), (b), (c), (d) and (e) are to be one continuous piece of tubing, $\frac{1}{4}$ " inside diameter with walls $\frac{1}{32}$ " thick. The coil (b) is to be from $1\frac{3}{8}$ " to $1\frac{7}{16}$ " outside diameter, and from $2\frac{3}{4}$ " to 3" long from the top of the plate (f) to the bottom of the coil, and to have not less than four complete turns. The cover (f) is to be one piece of metal, $\frac{1}{32}$ " in thickness and $2\frac{1}{2}$ " in diameter, turned down at the margin to form a vertical flange $\frac{3}{8}$ " high. The cover is to be brazed to the inlet and outlet tubes in vapor-tight joints. The tubes (a) and (c) must meet the cover at right angles, and the coil (b) must be concentric with the cover (f).

Necessary conditions for acceptance shall be:

- (1) That water shall circulate freely through the continuous tubing and coil.
- (2) That the coil shall enter, without bending, a tube of $1\frac{7}{16}$ " inside diameter.

Where it is necessary or desirable to avoid contact of metal with the solvent, a glass condenser may be adapted to the stand by the use of one straight and one bent copper tube and a rubber stopper, which can be securely fastened with wire, the specifications for such a glass condenser being as follows:

SPECIFICATIONS FOR GLASS INSIDE CONDENSER

To be made entirely of well annealed glass as per dimensions given in the accompanying drawing (Fig. 4), the neck to be $1\frac{1}{2}$ " long and 1" inside diameter, with the opening slightly flanged to take a cork securely; the bulb (B) to be from $2\frac{1}{2}$ " to $2\frac{3}{4}$ " diameter; the barrel (C) to be from $2\frac{1}{2}$ " long to $2\frac{3}{4}$ " and not less

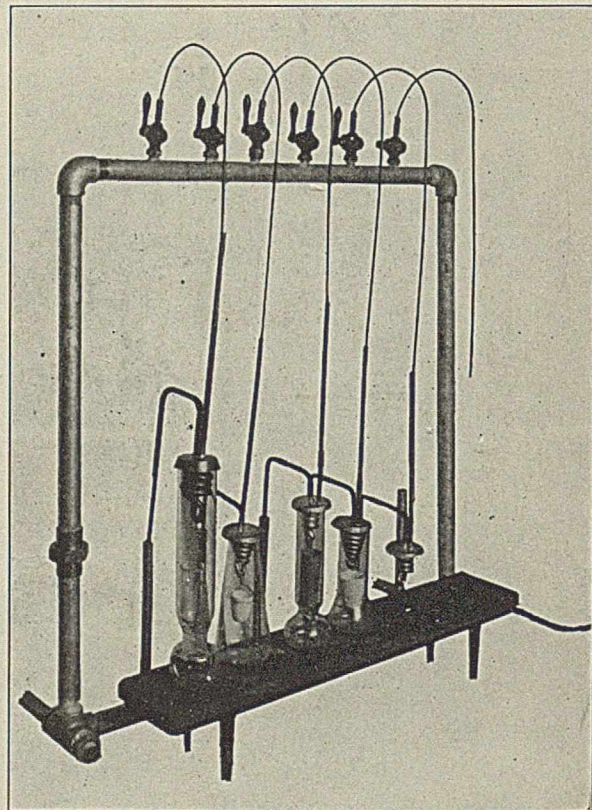


FIG. 2—HOME-MADE CONDENSERS ADAPTED TO VARIOUS FORMS OF EXTRACTION APPARATUS

than $1\frac{1}{4}$ " nor more than $1\frac{3}{8}$ " outside diameter, with a hook (D) about $\frac{1}{4}$ " long at the lower end; the flange (E) to be about $2\frac{1}{2}$ " in diameter, and $\frac{1}{4}$ " deep, sealed to bulb (B) near its outer extremity, and concentric with (A) and (C).

Fig. 5 shows the type of extraction flask, mentioned above, which accommodates either a small siphon tube or a 25 cc. porcelain Gooch crucible. The tube or crucible is held in position by the inward projections in the side of the flask. When the extraction is completed the tube or Gooch crucible may be removed, a solid crucible inserted, and the solvent thus recovered. The flask is made according to the following specifications.

SPECIFICATIONS FOR EXTRACTION FLASK

To be made of good glass, well annealed. Height of flask 130 mm., inside diameter of top from 39 to 41

mm., outside diameter at base from 50 to 56 mm., flask to have 3 equi-distant inward projections, made to touch the circumference of a circle 27 mm. (26 to 28 mm.) in diameter and concentric with the sides of the flask, points of projection to be 30 mm. (29 to 31 mm.)

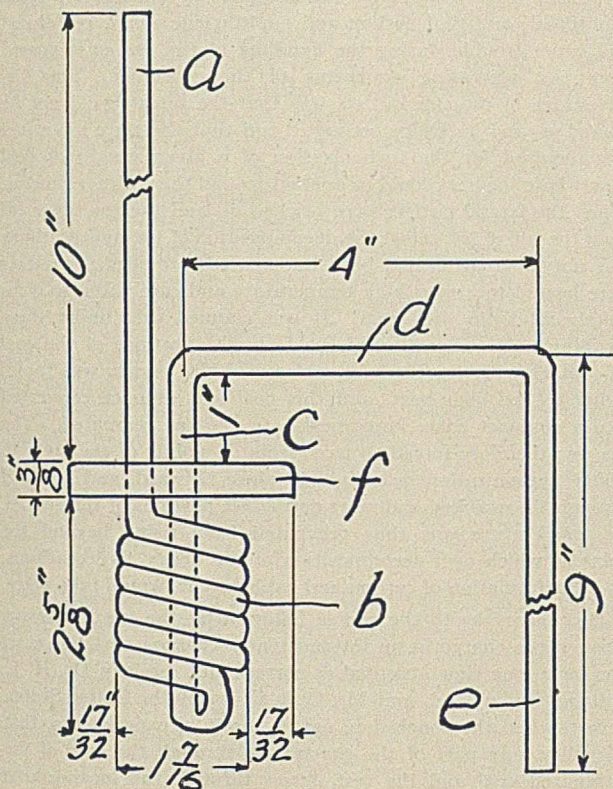


FIG. 3—METAL EXTRACTION APPARATUS CONDENSER

above base. Enlargement of cylinder to form base to begin immediately below projections; bottom to be entirely flat with rounded edge, top made smooth and parallel to base, flask to be of approximately uniform thickness throughout; weight of the flask to be from 40 to 50 grams.

The advantages of this apparatus are: (1) the stand

offers a rigid support for the condenser at all times, without the use of the clamps; (2) a cheap, durable and efficient condenser, which may be adapted to practically any form of continuous extraction apparatus; (3) the elimination of all rubber, corks, ground glass,

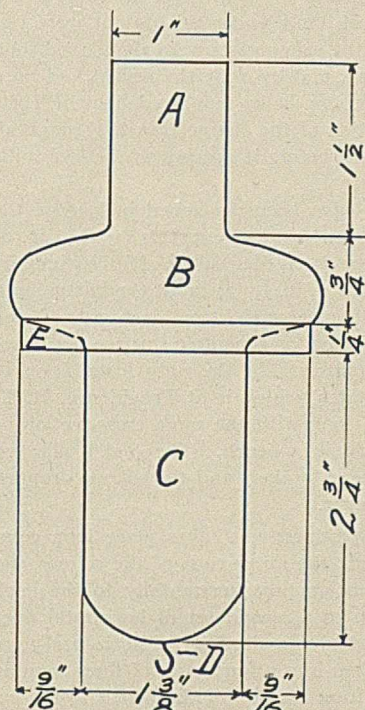


FIG. 4—GLASS INSIDE CONDENSER

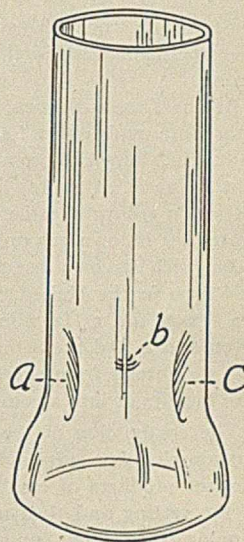


FIG. 5—EXTRACTION FLASK WHICH ACCOMMODATES A 25 CC. GOOCH CRUCIBLE

or mercury seal connections; (4) extractions may be safely run over night, since there is practically no danger of breakage due to change in water pressure; (5) the flask is light enough to be accurately weighed, can be easily cleaned, and is of such a form that all of the extract can be transferred.

BUREAU OF CHEMISTRY, DEPARTMENT OF AGRICULTURE
WASHINGTON

ADDRESSES

RECENT IMPROVEMENTS IN GAS MANUFACTURE¹

By ALFRED E. FORSTALL

Understanding it to be intended that I should cover only the manufacture of illuminating gas in central station plants I have limited myself to this phase of the general subject of gas manufacture.

The process of manufacturing illuminating gas divides itself into two stages, the generation of crude gas from the raw materials, and the purification of this crude gas in order to make it fit for general use.

GENERATION OF CRUDE GAS FROM RAW MATERIALS

Taking up first the generation of coal gas, an important recent improvement has been the development and quite general installation of retorts set vertically, which were unsuccessfully experimented with in the early years of the nineteenth century. Retorts set horizontally were, however, finally adopted and used solely, until, in 1885, M. Andre Coze developed a setting in which

¹ Read before the New York Section of the Society of Chemical Industry, The Chemists' Club, New York, March 27, 1914.

the retorts were inclined at an angle of from 29° to 33° to the horizontal. At such angles the coal would spread fairly evenly over the whole length of the retort by gravitation and the coke would run out fairly easily when the charges were thoroughly carbonized. Though adopted quite generally on the Continent of Europe and in Great Britain only three important installations of inclined retorts, and not more than five or six minor ones, were made in the United States.

VERTICAL RETORT SETTINGS

In the year 1902 two different types of vertical retort settings, the intermittent and the continuous, were brought to the attention of gas men. The intermittent type, in which an amount of coal which nearly or entirely fills the retort is dropped in at one time and allowed to remain until completely carbonized when the resulting coke is dropped out, also at one operation, was developed by Dr. Bueb of the German Continental Gas Company in the gas works at Dessau, Germany.

INTERMITTENT VERTICAL RETORTS—DESSAU

The first patent taken out for these retorts, in the early part of

1902, described them as provided with outlets, spaced from top to bottom along the whole length of the retort and opening into a vertical flue so placed in the setting as not to be exposed to a high heat, the object being to permit the gas as it was driven out of the coal to escape to the hydraulic main without being subjected to contact with either the highly heated coke or the walls of the retort. Settings built under this patent were put into operation in 1903 but the side outlets were abandoned soon after actual operation began, and by 1905 the design had been developed to that which is now being employed, in which the gas evolved from each portion of the charge travels through all the superincumbent portions, before it escapes to the hydraulic main.

As the Dessau verticals have been developed in Europe the retorts are made either four meters or five meters (13 ft. 2 in. or 16 ft. 5 in.) long with cross-sectional dimensions of approximately 9 in. by 22 in. at the top and 14 in. by 27 in. at the bottom and are set in groups of either twelve or eighteen, each group being heated by its own gas producer, or generator furnace, and provided with its own recuperators. In the only installation of this type in the United States, which is at Providence, R. I., the retorts are 13 ft. 2 in. long with the same cross-section as given above and are set ten in a bench. The coal is charged into the retorts from overhead bunkers and the coke is dropped into buggies or into a conveyor.

The combustion of the producer gas takes place in a combustion chamber surrounding the lower ends of the retorts and the hot products of combustion pass horizontally to the back of the setting and then up to a second set of horizontal flues running to the front, up again to a third set of horizontal flues running to the back and then into a fourth set of flues running to the front and pass out from the top of the bench to the recuperators and thence to the chimney.

INTERMITTENT VERTICAL RETORTS—UNITED GAS IMPROVEMENT CO.

In the United States the United Gas Improvement Company began experimenting with intermittently filled vertical retorts about 1908. As a result of these experiments the benches of this type last built contain nine retorts each 18 ft. 6 in. long with an oval section 12 in. by 22 in. at the top and 18 in. by 30 in. at the bottom. The reasons given for the use of the larger cross-section are that when it was smaller the coal coked too rapidly at the top of the retort causing excessive pressure at the bottom, that with the small retort the discharge of the coke did not take place satisfactorily except when the whole charge of coal was thoroughly carbonized, while a large retort will discharge readily even when the coal has not been thoroughly carbonized, and that the larger retorts produce larger coke, an advantage in many localities.

In this type of setting the combustion of the producer gas also takes place around the lower ends of the retorts and the products of combustion pass upward to the top of the setting.

The German practice is to fill the retorts completely with coal while the United Gas Improvement Company leave 4 ft. of the retort at the top empty, to provide a space in which the heavy hydrocarbon vapors may be converted into gas by the action of radiant heat.

It is interesting to note that in 1880 Mr. C. F. Dietrich obtained a United States patent making claims for a setting of retorts which were very similar to those of the original Buebe patent, including even the lateral openings into a channel protected from heat through which the gas could pass out from the retort. At that time, however, conditions in this country were favorable to the manufacture of carbureted water gas and the field for coal gas did not seem to warrant the trouble of developing a new method of manufacture, so that apart from building one experimental bench nothing was done by Mr. Dietrich to develop these retorts.

PASSAGE OF GAS THROUGH INTERMITTENT RETORTS

The reason for the provision of side outlets in the retorts under the original Buebe patent was the belief that, if compelled to pass up through the incandescent charge, the hydrocarbons first evolved from the coal would be over-decomposed into hydrocarbons of lesser worth from the illuminating and calorific value standpoint, and that carbon and naphthalene would be formed and cause trouble during the handling of the gas as it passed from the generating apparatus to the consumer. The disadvantage of the side outlets was that the retort could not be heated around its entire perimeter and that therefore more fuel was required for the carbonization of a given weight of coal than where the heat could be applied around the entire perimeter. When the lateral outlets were bricked up and the gas taken off from the top of the retort the decomposition of the hydrocarbons was not as great as had been anticipated, provided the retorts were heated to a very high temperature and care taken to completely fill them with coal. It was claimed that under these conditions the generation of gas from the portions of the coal in contact with the internal surfaces of the retorts was rapid, owing to the high heat, and this coal was at once converted into a compact coke, impermeable to the gas generated. The gas was therefore forced to pass from all points of the layer in which carbonization was taking place, inward and upward through the uncoked and more permeable portions of the charge. Its temperature was thus prevented from rising beyond the point at which over-decomposition of the heavy hydrocarbons, with the formation of carbon and naphthalene, would take place.

This theory as to the course followed by the gas in passing through the charge in an intermittently charged vertical retort was for a long time accepted as correct, but recently Dr. H. L. Colman, in England, and Mr. O. B. Evans, in the United States, have reached the conclusion, as the result of experiments, that in reality only part of the gas travels through the core of uncarbonized coal and the rest passes through the incandescent coke.

Dr. Colman argues that if even the largest portion of it traveled through the uncarbonized coal, the gas would show some of the characteristics of a gas produced at a low temperature, modified to a certain extent by the gas produced in the latter stages as the temperature of the coke is raised, and that the tar produced would be a low-temperature tar containing but a small amount of aromatic substances. As a matter of fact his analyses had shown that the gas possessed all the characteristics arising from exposure to high temperatures, the proportion of hydrogen to methane being even higher than that obtained from the same coal when distilled in highly heated horizontal retorts, and that the tar produced, although containing more paraffin derivatives than did tar from horizontal retorts, consisted chiefly of aromatic substances, showing that, during its formation, the vapors produced at low temperatures must have been subjected to a considerably higher temperature before passing out from the retort. This could not happen if even the larger part of these vapors passed through the uncarbonized coal, since then the travel would be in a direction which would expose them to a constantly decreasing, instead of to a higher, temperature. Moreover, the existence of a high pressure in the retort at the commencement of carbonization when the area of the core was the greatest and the diminishing of this pressure as carbonization proceeded, although the rate at which gas was produced did not decrease greatly during the first seven hours of the charge, while the area of the core did decrease quite rapidly, was strong evidence that the gas did not find its principal direction of travel through the core. He concludes that the great bulk of the gas is produced on the outer side of the pasty layer formed by the coal as it carbonizes and that this gas mainly travels through the hot coke, mixing with the poorer gas produced by the continued action of heat on the low-temperature

coke first produced. The gas produced on the inner side of the pasty layer will pass through the coal and he considers it probable that the vapors which pass in this direction form the undesirable paraffin constituents of the tar so that as far as travel through the core does take place it is disadvantageous.

Mr. Evans, reasoning from the pressure conditions existing in the interior of an intermittently charged vertical retort, came to the conclusion that during the early part of the charge, when the layer of coke formed around the perimeter of the retort was compact and offered no ready means of passage to the gas, most of the gas produced was obliged to force its way through the pasty layer of coal, in the initial stage of carbonization, and pass up through the uncarbonized coal, but that as the outer layer of coke contracted and cracked under further heating most of the gas formed in the latter portions of the charge passed up through this coke. He also concluded that of the gas made during the first six hours of the charge, which amounted to 70 per cent of the total amount of gas made, 45 per cent passed up inside of the pasty layer through the core and 55 per cent through the hot coke next to the retort walls, and that the gas evolved at low temperatures escapes through the core while that evolved at high temperatures escapes along the wall. Mr. Evans considers that this is advantageous since the low-temperature gas is more subject to injury by undue exposure to heat, and this is correct provided that after leaving the coal this gas is exposed to heat in a free space at the top of the retort.

This discussion as to the path followed by the gas in passing out from intermittently charged vertical retorts has been given somewhat at length because of the claims originally made that the freedom from naphthalene experienced with gas made in such retorts was largely due to the fact that the travel was through the cool core of uncarbonized coal.

ENGLISH DEVELOPMENT OF CONTINUOUS VERTICAL RETORTS

While the work of the Germans in connection with vertical retorts was confined entirely to those charged intermittently, in England the design of such settings was approached with the idea of adapting them to continuous carbonization. This had been experimented with in connection with the horizontal retorts and had given good results as far as the quantity and quality of the gas were concerned but had always proved a failure from the manufacturing standpoint because of mechanical troubles.

In 1902 there was built in Exeter a setting of vertical retorts into which the coal, in amounts varying from 2 lbs. to 7 lbs., was charged at regular intervals, the length of which could be varied. The retorts, about 9 ft. long, were made straight for part of the distance and then were curved so that the coke was withdrawn through an opening in the side of the setting at right angles to that in the top through which the coal was charged. The coke was drawn intermittently in comparatively large amounts at one time so that the extent to which the retort was filled with coke and coal undergoing carbonization varied quite considerably. After being tried in several places in England, further use of the settings was abandoned because of trouble experienced with the working of the coal-feeding device and in keeping the curved portion of the retort from cracking, and also with the formation of lampblack owing to the large variation in the volume of the charge because of the intermittent drawing of the coke and the consequent variation in the extent to which the gas passing off from the charge was exposed to heat in the vacant space left at the top of the retort.

WOODALL-DUCKHAM VERTICAL RETORTS

While this retort was being experimented with Messrs. Woodall and Duckham were developing at Bournemouth, England, another type of continuously charged and operated vertical

retorts. Starting with a mechanical coal feed as well as mechanical and continuous extraction of the coke, the former was soon abandoned because of operating difficulties and the introduction of coal into the retort is now brought about entirely as a result of the extraction of coke at the bottom. The construction of the coke extractors has also been very materially changed from the form shown in the original patent and the first plant erected.

As now built each retort is surmounted by a charging magazine filled through a rotary valve from an overhead coal bunker and holding enough coal for two hours' supply. There is free communication between the magazine and the top of the retort, the coal in the former being supported by that in the retort. As the coke extractor withdraws the coke from the bottom the whole column of material in the retort settles and coal runs in at the top from the magazine, the supply of coal in which is replenished at intervals of from twenty minutes to an hour.

The-coke extracting device consists of a rotating horizontal shaft provided with arms placed spirally around it and of loose arms hung on hinges at their upper end and having sufficient weight to hold back the coke except as their lower ends are pushed out by the action of the revolving shaft. The coke is discharged into a closed hopper which will hold the amount produced during two hours and is intermittently emptied either into dumping wagons running on an industrial railway track, or into a conveyor. The extractor can be run at varying rates of speed to suit the differing amounts of coal that can be carbonized under varying conditions.

As originally built the settings consisted of four oval retorts 25 ft. long and having cross-sectional dimensions of practically 9 in. by 23 in. at the top and 20 in. by 29 in. at the bottom. In recent installations the retorts have been replaced by rectangular ovens, or slots, of the same length and having cross-sectional dimensions of 8 in. by 3 ft. 10 in. at the top and 20 in. by 5 ft. 3 in. at the bottom. At the top of these slots there is a division plate, extending down an adjustable distance, which forms two separate spaces, into one of which the coal magazine opens while the other is kept free from coal so that the gas can pass out through it and in passing out be exposed to radiant heat for the purpose of decomposing the hydrocarbon vapors with low boiling points. As the settings are operated it would seem that the desired effect was obtained to only a very limited extent, if at all.

The combustion of the producer gas begins at the top of the setting and the products of combustion pass down, surrounding the retorts and are taken off into the recuperators a short distance above the bottom. From the recuperators they pass either directly to the chimney or in some cases are taken through the tubes of a waste heat boiler which furnishes practically all the steam required for the operation of the plant.

The primary air is heated before entering the furnace by passing across the setting in contact with the side walls of the retorts at their lower end, and the coke is sufficiently cooled in this way to require no quenching.

GLOVER-WEST VERTICAL RETORTS

While Messrs. Woodall and Duckham were developing their design, Messrs. Young and Glover were also doing, at St. Helens, England, work which has resulted in what is now known as the Glover-West system of continuous vertical retorts. The chief differences between the Woodall-Duckham and the Glover-West systems are in the form of the coke extractor and in the methods of heating the retorts. In the Glover-West, as in the Woodall-Duckham, coal runs into the retorts, from a charging magazine in free communication with the top, as the coke is extracted at the bottom. The coke extractor is in the form of a worm set with its axis vertical and slowly revolved, and delivers coke into a receiving chamber, large enough to hold that produced during a period of two hours and regularly discharged

at intervals of any length less than two hours. The retorts are oval in section and have a total length of 20 ft. with cross-sectional dimensions of 10 in. by 30 in. at the top and 22 in. by 36 in. at the bottom. To the bottom of each one is added a cast iron chamber 3 ft. deep, at the bottom of which is placed the coke extractor. The retorts are set in groups of eight and the setting is divided into either two or four chambers so that the retorts can be worked in units of four or two. The combustion of the producer gas takes place at six points evenly spaced along the portion of each chamber extending from the bottom to within about 5 ft. of the top, and the products of combustion, after passing horizontally around each set of retorts, ascend through vertical flues to chambers surrounding the upper 5 ft. of the retorts, from which they pass to the chimney. The secondary air is heated by passing the cast iron chambers at the lower end of the retorts and this cools the hot coke on its way to the coke extractors, and does away with the necessity of quenching it when taken from the coke chambers.

OPERATION OF HORIZONTAL RETORTS MODIFIED

Shortly after, and partly as a consequence of, the successful introduction of intermittently charged vertical retorts the method of operating horizontal retorts was substantially modified. This had been to charge such retorts with a layer of coal about 4 in. to 5 in. thick leaving a large free space above the coal, through which the gas passed to the mouthpiece and standpipe. The maximum charge for each of the 9 ft. retorts with one end permanently closed, in general use in the United States, was from 340 lbs. to 350 lbs. of coal. The front end of the retort being enclosed in the front wall of the bench for a depth of 13 in., almost a foot of the length was not effective for carbonizing purposes and the weight of charge was not more than 45 lbs. and in many cases not over 40 lbs. per lineal foot of the effective portion. There had been some theoretical discussion of the advisability of more completely filling the retorts, but this had not produced any result in practice until the completely filled vertical retorts afforded an object lesson of the freedom from carbon and naphthalene troubles secured by reducing the extent to which gas was exposed to contact with the highly heated walls of a retort. In England, where the use of through retorts, or those open at both ends, was common, it was easy to change from the lighter charges to the heavier ones, but in the United States it was necessary to change to the use of through retorts instead of single end ones before the increase in weight of charge could be made, since where the coke must be drawn from the retort by means of a rake it is necessary to leave sufficient space above the charge to permit the free passage of the rake, while with the through retorts, from which the coke can be pushed, provision for the passage of the rake does not have to be made. The present practice, where through retorts are in use, is to charge about 70 lbs. of coal per lineal foot of the effective portion of the retort. When so charged from 66 per cent to 70 per cent of the area of the retort is occupied by coal leaving only 30 per cent to 34 per cent of the area for the passage of the gas, while when weighing 40 lbs. to 45 lbs. per lineal foot of retort the charge only occupies from 30 per cent to 36 per cent of the area. The expansion of the charge during the coking process makes the actual free space left in the retort still smaller in proportion for the heavy charges.

The use of heavier charges has resulted in a large increase in the amount of gas made per pound of coal with a decrease in the operating difficulties caused by the presence of free carbon in the gas and in the tar. In England the yield of gas, which formerly averaged only about 10,500 cu. ft. per ton of 2240 lbs. of coal now averages nearly 12,000 cu. ft., while in one plant in the United States, that at Worcester, Mass., the former average yield of 5 cu. ft. per pound has been increased to 5.9 cu. ft. In other works in the United States which have adopted the heavy charges the yields of gas have been increased about 10

per cent as compared with those formerly obtained. This increase in yield has not been accompanied by a decrease either in the illuminating value or in the calorific value per cubic foot and has therefore resulted in obtaining a greater total illuminating value and calorific value in the gas from a given quantity of coal.

The yield from the continuous vertical retorts is also greater than that formerly obtained from horizontal retorts operated with light charges, but in this country has not been as large as that obtained from the horizontal retorts at Worcester, Mass.

COMPARISON OF GASES FROM HORIZONTAL AND VERTICAL RETORTS

Some analyses have been obtained of the gas made in each of two plants, one having horizontal retorts and the other vertical retorts of the Woodall-Duckham type. The horizontal retorts are operated with moderately heavy charges which, however, do not come up to 70 lbs. per lineal foot of retort. These plants are under the same management and the coal used is purchased from the same company and is presumably practically the same, so that the analyses show, to some extent, the difference in the gas due to the difference in method of carbonization.

	Horizontal retorts Per cent	Vertical retorts Per cent
Carbon dioxide.....	1.44	1.49
Benzol.....	0.67	0.55
Illuminants.....	3.15	3.98
Oxygen.....	0.48	0.28
Carbon monoxide.....	4.75	6.90
Hydrogen.....	51.36	45.02
Methane.....	33.60	37.33
Nitrogen.....	4.55	4.44
Candle power (Sugg D burner).....	100.00	99.99
Calorific value (B. t. u. per cu. ft. by calculation).....	13.60	15.57
Calorific value (B. t. u. per cu. ft. observed).....	610.00	615.00
Yield, cu. ft. per pound of coal.....	4.85	5.39

The total illuminants run slightly higher, and there is much less hydrogen in proportion to the methane, in the gas from the vertical retorts than in that from the horizontal retorts. The illuminating value of the gas from the vertical retorts is also higher while the yield per pound of coal was 10 per cent higher. During the period over which the analyses of the gas from the horizontal retorts were taken, very wet coal was being carbonized and this may have affected the illuminating value and the yield of gas which were lower during this period than the average for the preceding month, but even the averages for that month were lower than the results obtained from the vertical retorts which, however, were those made during a test lasting eight days and in regular working the results have not been quite as good.

Comparable analyses of the gas made in the working scale testing plant at the gas works in Birmingham, England, both in intermittent vertical retorts, of the Dessau type but apparently charged so as to leave a free space above the coal at the top of the retort, and in horizontal retorts have been given by Dr. W. B. Davidson as follows:

	Dessau Verticals Per cent	Horizontals Per cent
CO ₂	2.4	2.2
C _n H _m	2.8	3.4
O ₂	0.5	0.5
CO.....	10.3	9.7
CH ₄	28.0	31.5
H ₂	51.0	47.3
N ₂	5.0	5.4
Illuminating value, candles.....	100.0	100.0
Net calorific B. t. u. per cu. ft.....	15.0	18.0
	500.0	525.0

NOTE—The illuminating values given by Dr. Davidson cannot be compared directly with those given previously since they were obtained from the Metropolitan No. 2 Argand burner and are probably about three candles higher than would have been obtained had the Sugg D Argand been used as was done in the other case.

Dr. Davidson observes that the gas made in the vertical retorts is deficient in unsaturated hydrocarbons and methane and high in hydrogen, and that a cursory examination of the analyses

leads to the conclusion that the hydrocarbon gases are subject to more drastic degradation before leaving the retort in the vertical system than they are in the horizontal system, but that it is not unlikely that the gas suffers both in quality and volume by the escape uncracked of a larger proportion than usual of tar oil vapors.

Experience with the United Gas Improvement Company verticals operated with a large free space above the coal seems to show that the provision of this free space leads to an improvement in the illuminating and calorific value of the gas, which is obtained at the expense of the extra quantity of tar produced when the retorts are completely filled according to the strict use of the Dessau system.

As far as the quality and quantity of the gas are concerned intermittent vertical retorts of the Dessau system give poorer results than are obtained in horizontal retorts, while the intermittent verticals of the United Gas Improvement Company type and the continuous verticals do not give any better results along these lines than can be obtained from properly operated horizontal retorts. The improvements effected by the use of vertical retorts consist in a saving of labor in medium-sized plants in which it is impossible to work charging and discharging machinery for horizontal retorts to advantage, and in greater freedom from trouble caused by free carbon and naphthalene, while the sulfur compounds other than sulfureted hydrogen are also produced in smaller amount. In addition there is a saving in the ground space required although it is necessary to go higher into the air with verticals. To these advantages the continuous vertical retort system adds that of practically complete avoidance of the smoke and steam emitted during the charging and discharging of either horizontal retorts or intermittent vertical ones.

SUGGESTED METHOD OF DISTILLATION OF COAL

The work recently done in determining the character of the products given off by coal subjected to distillation at different temperatures indicates that the greatest efficiency, from the gas-making standpoint, in the carbonization of coal could be obtained by so adjusting the heating of continuous vertical retorts that the temperature of the upper 2 ft. or 3 ft. of the charge should never exceed 1000° to 1100° F., while that of the lower portion of the charge would be carried as high as 1800° F. By this method of heating, the rich hydrocarbons would be driven off from the coal without having to come into contact with very highly heated surfaces while the gas remaining after these hydrocarbons were driven off, which is of such a character as not to suffer to any great extent from such contact, would be entirely expelled from the coal in the lower portion of the retort. In order to decompose the heavy hydrocarbon vapors, which would otherwise condense into tar, in such a manner as to convert them into the maximum amount of permanent gas mixed with hydrocarbon vapors that could be carried by the gas, with the setting free of the minimum amount of carbon, all of the gas leaving the top of the charge should then be passed through a free space exposed to heat radiated from walls carried at a temperature adjusted to the rate of travel of the gas, but probably about 1400° to 1500° F. This free space could be maintained either in the upper part of the retort or, if this leads to difficulty in feeding the coal, it might be entirely separate from the retort and possibly common to several retorts.

This method of manufacture would increase the quantity and quality of the gas at the expense of the tar and would not be advantageous unless the value of the gas gained was greater than that of the tar lost. It has never been actually worked but in my opinion is entirely feasible.

IMPROVEMENTS IN MANUFACTURE OF CARBURETED WATER GAS

The recent improvements in connection with the manufacture of carbureted water gas consist in the devising, and putting into

general use, of appliances for measuring the amount of air blown through the fuel bed during the "blow," or heating-up period, and the amount of steam passed through the fire during the "run," or gas-making period, together with the use of electric pyrometers for indicating the temperatures existing at selected points in the checker brick of the carbureter and superheater of the type of apparatus most commonly employed in the manufacture of this gas. The use of these appliances makes it possible to determine and operate the apparatus constantly, under the conditions of blast, amount of steam used and temperature to which the oil vapors are subjected which give the best results and by so doing to decrease the amount of fuel used and increase the efficiency of the conversion of the oil into oil gas, as compared with the former more or less hit or miss operation.

PREPARATION OF CRUDE GAS FOR DELIVERY TO CONSUMER

In the second division of the process of gas manufacture, that of the preparation of the crude gas for delivery to the consumer, the recent improvements have been chiefly in connection with the removal of hydrogen sulfide and the other sulfur compounds present in the crude gas. For many years attempts have been made to use, for the removal of hydrogen sulfide from coal gas, the ammonia obtained from the gas itself. About 1886 a process for doing this was devised by Claus in Belfast, Ireland. Although chemically correct it proved too complicated mechanically and was finally abandoned largely because it was impossible to keep in working order the numerous pumps required for its operation. It is possible that if modern centrifugal pumps had been available the process might have been successfully operated.

AMMONIA FOR REMOVAL OF HYDROGEN SULFIDE

In the United States an extremely simple method of purification of gas from sulfureted hydrogen by means of ammonia has been recently devised by Mr. Jas. G. O'Neill and used on gas produced in coke ovens and sold for illuminating purposes.

The ammonia in ordinary ammoniacal liquor is already largely saturated with sulfur and carbon dioxide and one of the chief problems in connection with the use of this liquor for more complete removal of sulfureted hydrogen is to accomplish its conversion into a condition suitable for combination with sulfureted hydrogen without introducing too much complication of apparatus. Mr. O'Neill has solved this problem in a very simple manner by using liquor withdrawn from the still of the Coffey type, which is in general use in gas works for the concentration of ammoniacal liquor. He finds that when the liquor fed to the still has reached the point at which it has a temperature of 214° to 215° F. it has lost 80 per cent to 90 per cent of the hydrogen sulfide and 70 per cent to 80 per cent of the carbon dioxide, but still retains practically all the ammonia, which it contained when it entered the still. When brought in contact with crude coal gas in scrubbers of the ordinary type, this liquor can take up on an average 500 grains of hydrogen sulfide per gallon and if used in sufficient quantities will reduce the hydrogen sulfide from as much as 900 grains, down to from 20 to 30 grains, per 100 cu. ft. of gas. If the complete removal of the hydrogen sulfide with liquor is attempted the average efficiency of the liquor is much less than this and it is more economical to use the liquor only to the extent named and then finish the removal of the sulfureted hydrogen by the ordinary process of purification by means of hydrated sesquioxide of iron.

The process devised and used by Mr. O'Neill adds to the apparatus customarily found in gas works only a heat exchanger, in which heat is transferred from the hot liquor coming from the concentrating still to the cool liquor on its way to the still and a cooler for further cooling the treated liquor, both of which are simple and inexpensive. Apparently this process could be adopted in many coal gas works with a saving in the labor re-

quired to operate the oxide of iron purifiers and also in the investment in the purifiers required for a given quantity of gas.

REMOVAL OF OTHER SULFUR COMPOUNDS

In Europe, where the gas coals contain as a rule more sulfur than do those in common use in the United States, and on the Pacific Coast, where gas is largely made from crude petroleum containing a somewhat high percentage of sulfur, the problem of reducing the amount of sulfur compounds, other than hydrogen sulfide, contained in crude illuminating gas has had some importance. In those parts of the world, therefore, attention has recently been paid to new methods of removing the principal one of these sulfur compounds, carbon bisulfide.

Of the more recent processes having this object, the one first brought to the attention of gas engineers was that devised by Messrs. Hall and Papst and used since 1908 for the treatment of all the gas made, about 3,000,000 cu. ft. per day, in the works at Portland, Oregon. In this process the gas is merely heated to a temperature of from 1300° to 1600° F. by being passed through tall cylindrical vessels, formed of steel plates lined with fire clay blocks and filled with a checker work of fire brick, which are heated by the combustion in them of fuel oil. The vessels are in pairs, one being in process of heating while gas is being passed through the other. The operations are reversed as soon as the vessel through which the gas is passing becomes cooled below the effective temperature. Under the effect of heat the carbon bisulfide reacts with the water vapor present in the gas and is largely converted into hydrogen sulfide, which is removed by passing the gas through an additional set of purifiers containing oxide of iron.

NICKEL CATALYZER FOR DECOMPOSITION OF CARBON BISULFIDE

Another process which has been employed on a large working scale is that devised by Mr. E. V. Evans and used since the beginning of 1913 to treat all the gas made, about 10,000,000 cu. ft. per day, at the works of the South Metropolitan Gas Company, of London. This process utilizes the catalytic effect of nickel in accelerating the reaction between carbon bisulfide and steam, by which hydrogen sulfide is produced and carbon set free. The catalyzer is in the form of balls, presumably of fire clay, 1 in. in diameter impregnated with nickel obtained by the reduction of the chloride in a current of hydrogen. These balls are contained in tubes having a length of 11.5 ft. and a diameter of 6 in., through which the gas to be treated is passed. Before reaching the catalyzing tubes the gas passes through heat exchangers, or recuperators, in which it absorbs heat from the gas passing out from the apparatus, and then through preheating tubes in which its temperature is raised to 750° F. With this preheating of the gas it is possible to carry on the process while maintaining a temperature of 800° F. in the catalyzing tubes and the principal direction in which the process has been gradually improved has been this preheating of the gas before it enters the catalyzers. A single combustion chamber, supplied with producer gas from an outside producer, furnishes the heat required by both the preheating and the catalyzing tubes. After about thirty days' use it is necessary to stop the flow of gas through a set of catalyzers and blow air through them in order to burn off the deposited carbon which is found to be, at times, 50 per cent in excess of the quantity calculated from the amount of carbon bisulfide reduced to hydrogen sulfide. This carbon may be obtained from the decomposition of hydrocarbons in the gas, but analyses of the gas made before and after treatment, which are given below, show that the quantity so decomposed is negligible.

No information has been given as to the cost of operating this process and although it does not require the maintenance of as high a temperature as is employed in the Hall and Papst process it would seem to be somewhat more complicated and expensive, even though no loss of nickel be suffered.

	Gas before treatment Per cent	Gas after treatment Per cent
CO ₂	1.82	1.80
C ₂ H ₆	3.61	3.79
O ₂	0.27	0.07
CO.....	8.85	8.62
CH ₄	26.62	27.45
H ₂	52.45	52.19
N ₂ (by difference).....	6.38	6.08
	100.00	100.00
Illuminating power in English candles.....	14.05	14.05
Caloric value, B. t. u. per cu. ft.....	590.00	594.00

SODA-CELLULOSE FOR REMOVAL OF CARBON BISULFIDE

Another process which has been tried only at an experimental plant of the Heidelberg, Germany, Gas Works consists in treating the gas, entirely freed from tar, ammonia, sulfureted hydrogen and carbon dioxide, with a compound of soda and cellulose obtained by treating cellulose sulfite with soda lye. The resulting material, after having been rolled and crumbled to a powder, is placed on trays in purifying vessels in the same way as oxide of iron. When brought into contact with carbon bisulfide the soda cellulose is changed into cellulose xanthogenate or viscose, the raw material from which are obtained cellulose hydrate and the formyl-cellulose used in the manufacture of non-inflammable celluloid. In the experimental plant ten tons of the soda cellulose material, known as "Athion," absorbed 1.25 tons of carbon bisulfide so that with gas containing 45 grains per 100 cu. ft., ten tons would purify over 35,000,000 cu. ft. of gas, but the cost of operation is not given.

Since it is not the custom in the United States to remove carbon dioxide from illuminating gas, the employment of this process would involve the installation of additional apparatus for that purpose.

OXIDE OF IRON CATALYZER FOR DECOMPOSITION OF CARBON BISULFIDE

A very promising process, which, however, has not yet been tried out on a working scale, is based upon the fact that at temperatures above 100° F. metallic iron acts as a catalyzer that brings about and accelerates the reaction between bisulfide of carbon and moisture in illuminating gas. The fact that gas, free from sulfureted hydrogen originally, contained this substance after having passed through a wrought iron service pipe which, because of running near a steam pipe, was heated to temperatures varying between 90° and 158° F. attracted the attention of Mr. J. G. Taplay and a number of experiments made by him showed that at a temperature of 158° F. the reaction between the bisulfide of carbon and moisture with the formation of sulfureted hydrogen took place quite rapidly, in gas traveling through a wrought iron pipe, and also that as the interior of the pipe became rusted the sulfureted hydrogen produced was absorbed by the oxide of iron and did not show at the outlet of the pipe.

A French gas engineer, M. Guillet, observed the same action taking place inside a gas holder and by experiments determined that when gas containing bisulfide of carbon was passed through ordinary oxide of iron purifying material at temperatures above 25° C. (72° F.) 25 per cent of the original content of carbon bisulfide was removed. He found 100° C. (212° F.) to be the temperature at which the change became interestingly rapid, while at a temperature of 130° C. (266° F.) more than 67 per cent of the bisulfide of carbon originally present was converted into hydrogen sulfide and removed as the gas passed through the material. The percentage removed increased with the amount originally present, the treated gas containing only from 3.01 to 5.82 grains, while the original gas contained from 9.43 to 24.42 grains of bisulfide of carbon per 100 cu. ft.

Since the efficiency of oxide of iron for the removal of sulfureted hydrogen is very much increased by heating and it should not be difficult to maintain the temperature of the purifying material at the comparatively low temperature of, say,

250° F. this process would seem to offer the simplest and most inexpensive means of removing from illuminating gas the larger portion of the bisulfide of carbon which it still contains after the treatment ordinarily given to it in gas works, whenever the amount of this impurity present is sufficiently large to make it important that it should be reduced.

84 WILLIAM STREET, NEW YORK

CHEMISTRY AN IMPORTANT FACTOR IN THE FERTILIZER INDUSTRY¹

By J. E. BRACKENRIDGE

Not so many years ago the fertilizer manufacturer looked upon the chemist as a non-producing something, which was a necessary evil. Friction continually existed between superintendent and chemist, the superintendent being sure that materials were batched on correct weights and that the chemist was wrong if the analyses did not come up to the guarantee. In one factory where a man inspected the cars of tankage as they were received and classed them by looks, as 8, 9 or 10 per cent goods, the superintendent said that this man could guess nearer than the chemist could test. Such conditions were not unusual in factory management. Rock, acid, potash salts and ammoniates were all combined wet, and it was not unusual to have to allow 10 per cent excess for potash, because it would not show as water-soluble in the final product. No account was taken of the actual chemical action when phosphate rock containing iron, alumina, silica and fluorine was mixed with sulfuric acid and potash salts added.

Conditions at the present time are entirely changed. Successful manufacturers insist that superintendent and chemist work together, and that all chemical action influencing the analysis of mixed fertilizers be carefully watched so that there shall be the greatest efficiency of the materials used. The superintendent should be held responsible for the pounds received of phosphoric acid, potash in terms of K_2O , and nitrogen, and unless conditions affecting the loss of available phosphoric acid, water-soluble potash and nitrogen are understood, the greatest efficiency will not be realized.

The manufacture of acid phosphate is one of the oldest processes now in use in the industry. Not long ago, we were satisfied with 16 per cent available phosphoric acid from 66 per cent calcium phosphate Florida rock. Now we are not satisfied unless we get 17 to 17.5 per cent available from the same grade of rock. Then it was unheard of to obtain 16 per cent available phosphoric acid from 62 to 63 per cent calcium phosphate Charleston rock; now this is possible. Such results are entirely due to taking advantage of every possible condition which affects chemical reaction, such as fineness of rock, strength of acid, time of mixing and manipulation of acid phosphate from dens to storage.

The possibilities of phosphoric acid compounds from phosphate rock and sulfuric acid have hardly been considered, due largely to the presence of arsenic. This condition no longer exists, as it is possible to reduce the arsenic content in phos-

phoric acid made from phosphate rock and sulfuric acid to a percentage that will pass the pure food standard. Hence, chemistry aids the fertilizer industry by producing the phosphoric acid not only for the soil, but for general phosphoric acid compounds. Again, electrochemistry offers, to the fertilizer industry, possibilities of securing very high temperatures and thus rendering available the phosphoric acid in refractory minerals. Fluorine compounds are now being made from the flue gases from acid phosphate manufacture, which has been made possible only by taking advantage of chemical reaction.

Chemistry has made rapid strides for the benefit of the industry from the nitrogen standpoint. We no longer have to rely on animal, mineral and vegetable ammoniates, since nitrogen recovered from the air helps to increase the supply and thus regulate prices. Chemistry has increased the nitrogen supply available for soil by giving us the conditions necessary for rendering inert nitrogen available, thus allowing us to use many waste materials.

Nature has accomplished most for the industry in regard to potash, for the natural deposits seem to outclass anything that has been done in giving us supply of this element. Possibilities of feldspar and alunite potash are still in the distance, as well as the recovery of beet sugar molasses potash by passing the molasses over zeolites, when the potash is said to be held so that it can be recovered from the zeolite.

Chemistry again gives promise of potash from kelp, where the actual cost of the potash may be reduced by the income from by-products. Cement furnaces also are being investigated, where it may be possible to volatilize and recover the potash contained in the raw materials.

Chemistry has an important place in the fertilizer industry in relation to the conditions which affect drilling of fertilizers. If the materials at hand were always the same as to chemical composition, little trouble would be experienced in mixing them. But there are so many varying materials, and so many varying compositions, that unless foresight is used as to the chemical reactions possible where materials are mixed, serious trouble will result.

Chemistry again aids the fertilizer industry in chemical engineering problems that are common to power plants.

Chemistry is the basis of all commercial values in the fertilizer industry. Where we consider that profits may be easily turned to losses by incorrect chemical work, it must be realized that the fertilizer manufacturers must have able and competent chemists to do their work.

There is much work ahead in the fertilizer industry. Unless every effort is used to develop reliable methods for chemical analysis, and research work is carried on to increase the supply of fertilizer materials, and unless chemical conditions affecting fertilizer manufacture are carefully studied to the end that greatest efficiency be gained, the chemist will not have done his part in the development of this industry.

CARTERET, NEW JERSEY

OBITUARIES

HERMAN FRASCH

With deep regret we record the death of Mr. Herman Frasch, the distinguished chemical engineer, which occurred in Paris, on May 1, 1914.

Mr. Frasch was born in Gaildorf, in Wurtemberg, in 1852, and received his early education as an apothecary in Germany. In 1868, he came to America and was, for a time, in charge of the laboratory of Professor Maisch of the Philadelphia College of Pharmacy. Being particularly interested in Industrial Chem-

¹ Chairman's address, Fertilizer Chemistry Division, 49th Meeting A. C. S., Cincinnati, April 6-10, 1914.

istry, he established a laboratory in Philadelphia, in 1874, in which he began a series of investigations which led to some of the most brilliant achievements in the field of chemical engineering.

His first invention was a process for refining paraffin wax in 1876. This process was a great success and was followed by his invention of a process for the refining of the sulfur oils of the Canadian, Ohio, and Illinois fields. Previous to this invention, these inferior oils had a very low market value and were limited to use as fuel oils. In 1885, he organized the Empire Oil Company and established a small refinery at London, On-

tario, for the purpose of developing his desulfurizing process on the Canadian oils. As a result of this work, the methods of refining sulfur oils were revolutionized, and plants for working these oils were established at Cleveland, Whiting and other centers.

Another wonderful invention was his process for extracting sulfur from the great Louisiana deposit. That this deposit existed, covered by a layer of quicksand over five hundred feet thick, had been known since 1865. Company after company was organized to develop these sulfur mines but without success until Mr. Frasch purchased the property in 1891, and attacked the problem from an entirely new standpoint. Instead of attempting to sink a shaft and mine after the customary practice, he drove wells through the sand and inserted a series of iron tubes so arranged that he was able to fuse the sulfur in place by forcing down superheated water under high pressure. The molten sulfur was permitted to flow to the surface through return pipes where it was run into large bins and solidified in commercial form. This enterprise has been in successful operation ever since and supplies all the sulfur required for the United States market with some excess for exportation.

I do not, on this occasion, go into details with regard to the other remarkable inventions of Mr. Frasch, for the reason that in December, 1911, the associated chemical societies of America conferred upon him the Perkin Gold Medal which is awarded annually for distinguished service in the field of applied chemistry, and the proceedings on that occasion were printed in full in THIS JOURNAL, in the issue of February, 1912, page 131, and included the address of acceptance by Mr. Frasch, in which he gives some details of his most interesting career.

Mr. Frasch was a member of The American Chemical Society, The Society of Chemical Industry, The American Institute of Chemical Engineers, The American Institute of Mining Engineers, The American Electrochemical Society, The Verein Deutscher Chemiker, and The Chemists' Club, New York City.

C. F. CHANDLER

RESOLUTIONS PASSED BY CHEMICAL SOCIETIES

At a meeting of the officers of the undersigned chemical organizations, held on the 11th day of May, 1914, the following resolutions were adopted:

The members of the chemical profession as represented by the officers of the chemical and allied societies, have received, with profound sorrow, the sad tidings of the demise of their distinguished colleague and fellow member, Mr. Herman Frasch.

His life, his energies and his wide knowledge were given freely and unselfishly to the development of the chemical industry. As a creator of new branches of applied chemistry his name is among the most illustrious, and in the history of technical accomplishment his work stands out as an inspiration to all coming generations.

Much as we honor his memory as a chemist, he has no less endeared himself to us as a man, as a wise counsellor and as a friend.

Notwithstanding the stress of an extraordinarily active life, he preserved within his heart a great kindness and consideration towards others and an ever ready sympathy in their efforts and struggles.

Resolved, that these resolutions be spread upon the minutes of the undersigned societies and that they be engrossed and copies presented to his widow and his daughter, to whom we extend our deepest sympathy in their bereavement.

THE CHEMISTS' CLUB
NEW YORK SECTION
AMERICAN CHEMICAL SOCIETY

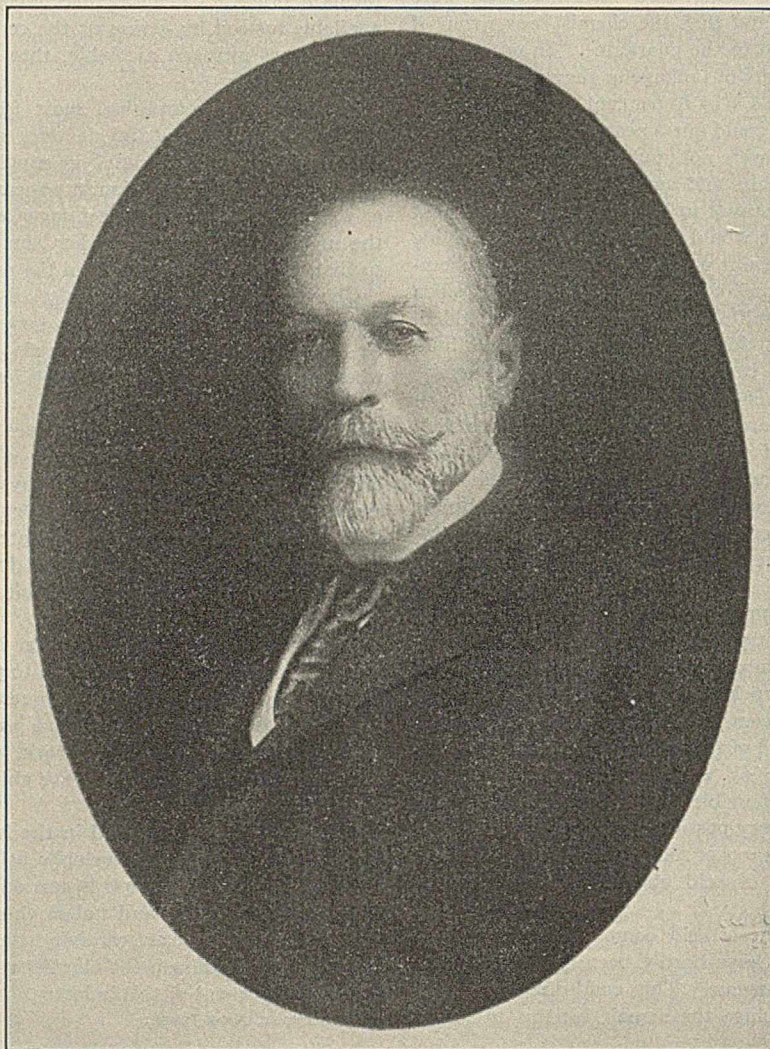
NEW YORK SECTION
SOCIETY OF CHEMICAL INDUSTRY

AMERICAN INSTITUTE OF
CHEMICAL ENGINEERS

NEW YORK SECTION
VEREIN DEUTSCHER CHEMIKER

NEW YORK SECTION
AMERICAN ELECTROCHEMICAL SOCIETY

PERKIN MEDAL COMMITTEE



HERMAN FRASCH

PAUL L. V. HÉROULT

Paul L. V. Héroult, the inventor, died at his home in Paris, on May 13th. He was born April 10, 1863, at Thury-Har-

court, Calvados, France, the son of Patrice and Elise Lepetit-Desaunay Héroult. His father was a tanner in Paris and his grandfather a leather broker in London. Both branches of the family had come originally from Normandy. As a boy he was educated in London, afterwards entering the Lycée de Carn, Calvados, France. His education was completed at the College Sainte Barbe and the École des Mines in Paris.

Mr. Héroult invented and patented, in Europe, in 1886, a process for the manufacture of aluminum. The United States Patent Office records disclosed the fact that the same process had been invented in America at about the same time, by Charles

M. Hall. As a result of the controversy, the European patents were granted to Hèroult and the American patents to Hall.

In 1887, Mr. Hèroult became the technical manager of the aluminum works at Neubrausen, Switzerland, and in 1890, was made director of the French Aluminum Company.

He spent much of his time in chemical engineering research, and in 1899, developed an electric furnace for the production of steel. He was connected, in the development of this electric steel

furnace, with The United States Steel Corporation, The Crucible Steel Company of America, Halcomb Steel Company, Electro-Metals Company, the Aktiebolaget Hèroults, Elektriska Stal, Sweden, and Edgar Allen, Thomas Firth and Vickers, all of Sheffield, England.

Mr. Hèroult was a member of The Chemists' Club, New York, The American Electrochemical Society, The American Society of Mining Engineers, The Société des Ingénieurs, Paris and The Faraday Society, London.

CURRENT INDUSTRIAL NEWS

By M. L. HAMLIN

A NEW TYPE OF ARTIFICIAL FERTILIZER

In a paper read before the Society of Arts of London by Professor W. B. Bottomly, a new type of fertilizer was described, which, according to *Engineering* (London), 97 (1914), 359, threatens serious competition with the products of the electric furnace. After giving an account of various previous attempts to utilize for fertilizing purposes the power of certain bacteria found on the roots of some plants to fix atmospheric nitrogen, and showing how these attempts had been unsuccessful, Professor Bottomly described experimental work carried on at the botanical laboratory of King's College, claiming that it had been attended with complete success. It was found that suitably treated peat formed a most excellent medium for the growth of the bacterium, and soils manured with this peat have shown a marked enhancement of their fertility. Before inoculating the peat with the bacterium in question the raw peat has to undergo a preliminary treatment by another bacterium, which was found to have the power of converting natural peat into a humated neutral medium. Attempts to achieve the same end by neutralizing the humic acid of the peat by alkalies resulted in complete failure. The peat after inoculation with the special bacterium is kept at constant temperature for a week or ten days, after which period it is sterilized by the action of live steam. It is then inoculated afresh with a mixture of *azotobacter chroococcum* and *bacillus radiicola*, and after a few days' incubation at 26° C., is ready for use. The following table shows the effect of the treatment as proved by analysis. Analyses of a garden soil and other manures are also given for comparison:

	Soluble humate Per cent	Soluble nitrogen Per cent	Total nitrogen Per cent
Raw peat.....	0.028	0.214	1.267
Bacterized peat.....	15.194	2.694	4.310
Garden soil.....	0.012	0.026	0.427
Fresh soluble manure.....	0.433	0.291	2.533
Well-rotted stable manure.....	1.46	0.439	2.848
One-year-old peat-moss litter manure.....	1.05	0.826	2.587

An important point is that the *azotobacter* continue to flourish after the peat has been added as manure to the soil to be fertilized, thus fixing further nitrogen. In a series of comparative experiments made on an exhausted soil the new manure showed the following percentage of advantage over its competitors:

CROP	Inoculated peat Per cent	Artificial Per cent	Farm dung Per cent
	Potatoes.....	123	75
Turnips.....	100	47	26
Beet.....	281	54	43
Onions.....	110	110	46
Carrots.....	260	20	28

With fertile soils the addition of a very little of the treated peat is stated to give a very large increase in the rate of growth. This is believed to be due to the presence in the peat of accessory food bodies, for which a special search is now being made.

A NEW COAL CALORIMETER

An interesting new coal calorimeter is that recently introduced by Macklow-Smith and described in *Engineering* (London), 97 (1914), 385.

The calorimeter consists essentially of an outer jar of glass provided with a reference line indicating the height to which it is to be filled with water in making an experiment. From the edges of this jar a crucible support is slung by the spring clips shown in Fig. 1. A piece of lampwick, impregnated with sodium or potassium nitrate, is embedded in a briquette of the coal under test, and is ignited by a match as indicated.

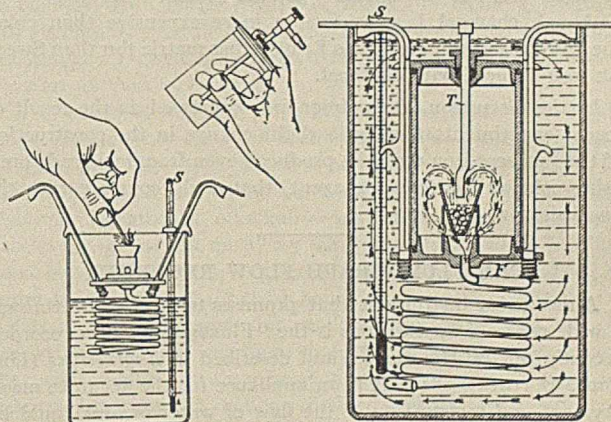


FIG. 1

FIG. 2

The glass bell, shown to the right in Fig. 1, is next lowered over the crucible and clamped, against a rubber ring, by springing back the supporting clips into their working position, as indicated in Fig. 2. A supply of oxygen is led from the oxygen bottle through an automatic reducing-valve and the flexible pipe to the inlet pipe T, which, as will be seen, is fitted with an additional regulating-valve, D. This oxygen causes the coal to ignite, and the products of combustion flow through the coil F, and escape through small holes near the end of it, and finally pass up through the water. In their journey these gases part with their heat, and at the same time thoroughly stir the water up. A series of readings of the thermomometer S are taken at equal intervals of time, and plotted against a time base, for determining the radiation correction. Simultaneous observations are, of course, made of the room temperature. The maximum temperature recorded on the plot is noted, and the temperature rise deduced from this is corrected, for the loss of heat externally, by means of a table of coefficients supplied with the instrument. The water equivalent of the coil and bell and other components of the calorimeter is also supplied by the makers, and as a consequence the user has merely to fill the apparatus to the level engraved, and may then treat the whole as having a constant specific heat, and obtain his results by multiplying a constant (provided by the makers) by the corrected temperature rise, and then dividing this product by the weight, in grams, of the coal taken.

While the briquette form is the most convenient in which to burn the coal, powdered coal may be and often is used. In actual operation the circulation of the water in the calorimeter is very perfect and regular at all rates of combustion, so that not only is the whole of the apparatus raised to the same temperature with certainty, but the radiation is also very regular in character. The amount of radiation while the water is being circulated is very different from that under quiescent conditions.

ELECTRIC SMELTING OF IRON AT HARDANGER, NORWAY

The hitherto unsatisfactory results of electric smelting at Hardanger were discussed at a recent meeting of the Polyteknisk Forening of Kristiania by Gustaf Ödquist, *Chemiker-Zeitung*, 38 (1914), 294. The reason for the failure lies in a series of unfortunate coincident circumstances, and not, according to Ödquist, in the type of furnace used. These furnaces are, however, not adapted to the use of coke for reduction, the electrodes being probably too large in diameter, and an economic use of power is rendered impossible by the large variations in load which make it necessary to have at command more than 800 k.w. in excess of the average amount of electricity used. The experience of the Hardanger works has shown that iron smelting with coke is possible, but not economical with the system used at present. Although charcoal is considerably more expensive than coke, the Hardanger iron cost \$3 to \$4 more per metric ton than Swedish iron reduced with charcoal.

In the discussion, the opinion was expressed as the result of recent experiments that by a slight change in the construction of the furnaces iron could be produced even from low-grade ores, using coke as the reducing agent, that could compete with the Swedish iron.

THE FLUXOGRAPH FLOW RECORDER

A measuring instrument that promises to be of value through a wide range of applications is the "Fluxograph" flow recorder, recently put on the market and described in *Engineering* (London), 97 (1914), 284. It is an appliance for the accurate measurement and registration of the flow of water or any liquid by means of the V-shaped notch discharge method, based on the formula

$$Q = cH^{5/2}$$

proposed by Mr. James Thomson, M.A., D.Sc., LL.D., F.R.S., Professor of Civil Engineering at Queen's College, Belfast, and at Glasgow University. The results of his investigations are given in the subjoined table, where:

H = the vertical height in inches of the still water level from the vertex of the notch.

Q = the corresponding discharge over the notch in cubic feet per minute, as found by experiment.

c = the value of the coefficient calculated from the formula $Q = cH^{5/2}$.

H	Q	c
7	39.69	0.3061
6	26.87	0.3048
5	17.07	0.3053
4	9.819	0.3068
3	4.780	0.3067
2	1.748	0.3088

The figures contained in the above table give the average results for the series of experiments made in the years 1860 and 1861. The mean of the six values of c is 0.3064, but from a comparison of his experiments Professor Thomson finally adopted 0.305 as the coefficient, and gives as his formula for the right-angle notch:

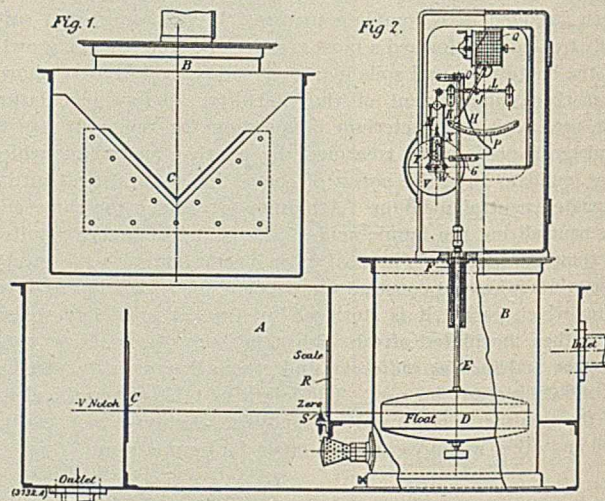
$$Q = 0.305 H^{5/2}$$

The total results of later observations proved how remarkably

accurate Dr. Thomson was when he gave 0.305 for the average value of the coefficient c for heads ranging from 2 in. to 7 in.

In the Fluxograph recorder is presented a practical and commercial application of the results of this valuable research work. Devised with the object of meeting the demand for a reliable integrating or weir meter, a number of these instruments have already been installed and successfully employed in various capacities. The machine, in addition to registering permanently upon a ruled chart the rate of flow at any moment in gallons per hour, also provides, by means of a pointer on a figured open-scale dial, a simple method of reading off directly, without calculation, the amount of water passing at any instant. Figs. 1 and 2 will serve to illustrate the machine and make its action clear.

On referring to the engravings, it will be seen that after entering the right-hand compartment of the measuring-tank A, Fig. 2, the water passes through baffling and diaphragm plates, and becomes tranquil before passing over the V-notch C, shown in Fig. 1, which is a transverse section through Fig. 2. A large float, D, is situated in a cylinder, B, to which the water gains access through a grid, and in which the level is the same as in compartment A. According to the variation of the water-level, so the float rises or falls, and it is this movement of rise and fall that actuates the mechanism of the recorder. At the point where the float-rod passes through the bottom of the instrument-



case a vapor-seal and dust-excluder arrangement, F, is fixed to prevent the working parts from getting clogged or becoming affected by rust. Sliding through a frictionless roller-bearing, G, the float-rod carries a flat compensating-curve attachment, H. The pen-carriage J, on which the pointer K is also fixed, moves along silver-steel bearing bars L, and is kept in roller contact with the face of the compensating-curve H by means of a counterweight, M (see Fig. 2).

It will be seen that the rise and fall of the water causes the float D to make a corresponding movement, and the float accordingly operates the compensating curve H, which ensures that the movement of the pen O and the pointer K is directly proportional to the amount of water flowing over the weir. While the pointer K momentarily indicates upon an open-scale figured dial, P, the rate of flow in gallons per hour, the pen O makes a permanent record of that quantity upon a chart, Q, surrounding the rotating drum of a clock device. With the aid of a planimeter the total amount passed can be obtained from the area of the chart record. The actual depth of water passing over the weir can be observed at any moment, and the accuracy of the recorder can be independently checked by means of a vertical scale, R, marked in inches, which is fixed on the measuring-tank A, in correct relation to the zero pointer S and bottom of the V-notch C, as shown in Fig. 2. Oftentimes it is imperative to be able to

read off directly and quickly the total quantity of water that has flowed for a given period. A simple form of friction integrator, T, is described herewith.

A clock, not shown, affixed to the recorder case, drives a flat aluminium disc, V. Kept in light contact against the disc is a small fiber wheel, W, connected to the counter or integrator. The latter is attached to the pen-carriage J, which is actuated by the movement of the float-rod compensating curve H according to the rise and fall of the float, so the driving-wheel W and the counter X moves away from or towards the center of the disc V.

When no water is passing over the V-notch C, the counter-wheel W remains stationary at the center of the disc, where, of course, no motion is transmitted. When the water is flowing through the notch, the float D rises, and, through the compensating curve P, raises the apparatus and lowers the counter proportionately, not to the rise of the float, but to the volume flowing through, so that the integrator is driven at a speed proportional to the amount passing over the weir. Figures showing the total quantity of flow can be read off directly from the counter-dials Y. The fact that the driving of the counter is continuous and positive ensures that all liquid must be registered.

The Fluxograph flow-recorder is well suited for the measuring and recording of water supplies, boiler-feed, air-pump discharge, sewage or trade effluents, compensating water from reservoirs, and for oils, acids, and alkalis used in manufacturing processes.

INDUSTRIAL ACCIDENTS IN MASSACHUSETTS

The Industrial Accident Board of Massachusetts, at the end of its first year reports the total number of non-fatal accidents in the state as 89,694, or about one for every ten wage earners. The board believes that at least half of these can be prevented by improved inspection methods, by safeguarding machines before they leave the factory, by the maintenance of safety museums, through lectures, moving pictures, school talks and the elimination of danger spots in plants. There were 474 fatal accidents, classified as follows:

Railroad equipment.....	119	Belting.....	6
Falls.....	66	Infection from trivial cuts, burns, etc.....	5
Vehicles.....	43	Saws.....	4
Hand labor.....	37	Explosions (not boiler).....	4
Elevators.....	33	Hoists.....	4
Electricity.....	25	Illness.....	3
Street railways.....	20	Presses.....	2
Boiler explosions and burns.....	15	Gears.....	2
Excavating.....	14	Emery wheels.....	2
Cranes.....	11	Occupational diseases.....	2
Miscellaneous.....	11	Glass.....	1
Asphyxiation, drowning, etc.....	10	Wood molders.....	1
Animals, insects, etc.....	9	Assault and fighting.....	1
Shafting, set screws, etc.....	9		
Falling material.....	8		
Machinery peculiar to special industries.....	7	TOTAL.....	474

VACUUM TAR

Scientific American, Apr. 25, 1914, reports that by distilling coal under a vacuum of 15 mm. of mercury, Pictet and Bouvier obtain a so-called "vacuum tar," this appearing to be quite different from usual coal tar. Such tar contains no phenol or aromatic carbides, and when oxidized by permanganate, the products are acids of the fatty series. The properties of this tar resemble those of Caucasus petroleum which consists of hydro-aromatic carbides. These experiments tend to confirm the hypotheses advanced by Berthelot and others as to the formation of the constituents of tar.

REQUIREMENTS OF GLASS FOR BOTTLING MEDICINE

W. A. Hamor in the *American Druggist and Pharmaceutical Record*, 62 (1914), 29, states that owing to the want of or negligence in chemical control at the factories, the glasses from which bottles are made often present wide divergences in composition, the principal variations being in content of silica, calcium oxide

and magnesium oxide.¹ The limestone in use by many manufacturers varies considerably in magnesium oxide and carbon dioxide content, and it has been ascertained² that variations of calcium oxide in the "batch" cause lack of homogeneity. It follows, therefore, from these facts alone, that not only should the materials entering into the batch mixture comply with standard specifications and accordingly be under analytical control, but also the composition of the "batch" itself should be based entirely thereon.

A large number of cases of deterioration of chemical products, due to the alleged solubility of bottle glass, have been noted by various consumers. In the case of non-actinic glass bottles, such alteration in composition may often be ascribed to the fact that the glass yields sufficient alkali to cause contamination or decomposition; but in the case of colorless glass bottles the cause of any deterioration is more dubitable, providing the product contained therein is subject to photolytic action. Certain it is, however, that medicinal preparations, like reagents, should be kept only in bottles made of high-grade glass, and that more consideration should be given to the fact that not only is amber glass non-actinic, thereby screening out light rays occasioning decomposition, but it is also usually much less soluble than colorless glass.

A number of preparations affected by light (for instance, compound glycerophosphates) are supplied by some manufacturers in colorless glass bottles; whereas others (for example, codliver oil) affected by even slight amounts of alkaline matter from glass, occasionally present indications that the glass bottles in which they are supplied are soluble.

A question of importance in the trade is: With what conditions should the glass of medicine bottles comply? Lenz³ has proposed that if the bottle is half filled with distilled water and then heated in boiling water for six hours, with frequent shaking, the amount of alkaline matter extracted should not exceed 1.5, or, at the most, 2.5 mg. per sq. dm.; while Moeller⁴ considers that medicine bottles should not yield any alkali to water heated in them for three hours.

Glass sufficiently pure for medicine bottles should not, in the writer's opinion, lose more than six mg. of alkali per square dm. and should not show pronounced cracks when kept for twenty-four hours in boiling distilled water. It is sufficient to specify, however, that all bottles intended for medicinal purposes should show no alkaline reaction when filled with distilled water containing several drops of phenolphthalein solution and heated at 100° C. for six hours. This requirement is simple, yet experience has demonstrated its efficacy in practice.

In this connection reference may be made to the shape of bottles intended as containers for poisons. It has been proposed that these should be hexagonal. A most striking form is triangular. A bottle of this shape, made of dark red glass, would fill an obvious need.

METHOD OF SCURFING GAS RETORTS

The Journal of Gas Lighting and Water Supply, 125 (1914), 295, describes a method of scurfing retorts presented in a communication by M. Schmidt to the Belgian Gas Association. The appliance used is a "Centrator" blower, actuated by a 2 H. P. gas-engine. It makes from 3600 to 3900 revolutions per minute, and in this period delivers 700 cubic feet of air at a pressure of about 6 inches of water. The blower is worked by means of encased steel rollers, which have to be kept well lubricated; these rollers are replaceable. The engine and blower combined occupy but little space. The carriage measures only 4 ft. by 3 ft., and it can be easily moved. The engine cylinder is cooled by water conveyed by a flexible tube. When a retort

¹ For a general discussion of the Glass Bottle Industry in the United States, see Hamor, *THIS JOURNAL*, 5 (1913), 951.

² See *Trans. Am. Ceram. Soc.*, 15, 706.

³ *Pharm. J.*, 91, p. 531.

⁴ *Ibid.*

has to be scurfed, the machine is drawn up near the setting, and a pipe of galvanized or ordinary iron is fixed from the outlet of the blower and run into the retort to about half its length. The engine is then started, and the removal of the scurf at once begins. About two hours only are required to clear a retort.

Taking the case of gas-works having ten settings of nine 10-foot retorts, M. Schmidt gave the total annual cost of scurfing reckoning twelve operations in a year, as \$266. Notwithstanding that the greater part of the graphite is burnt, an appreciable quantity is saved, the disposal of which helps to cover the cost of maintenance and the sinking-fund charges. Compared with similar works in which the old method is used, M. Schmidt gave the total annual expense for dealing with ten settings as \$608. From this there would have to be deducted the return from the sale of the graphite. From 10,000 to 12,000 kilos of this would be produced, worth \$120, which would bring down the cost to \$488. The other method, therefore, shows a saving of about \$222. The great advantage of forced injection of air is that the works are able to produce more gas, owing to the fact that the retorts can be charged more rapidly, thereby ensuring economy of labor and heating. Indeed, the productive capacity of the works is increased by about 3.5 per cent.

INDUSTRIAL CONDITIONS IN FRANCE

Engineering (London), 97 (1914), 486, quotes the *Moniteur Industriel* to the effect that metallurgical and engineering works in the North of France, taken generally, did less business in 1913 than in 1912. In regard to mechanical industry, attention is called to the most active competition experienced on the part of Germany, Belgium, and the United States, which countries, notwithstanding high customs duties, succeed in selling their material at lower prices than the French products. Attention is also called to the insufficiency of the customs duty on certain classes of French manufactured articles, in the price of which the cost of labor forms the heaviest constituent. Complaints are raised in the article by the *Moniteur* concerning the orders placed abroad by the French railway companies; no organization, it says, which owns any concession in France should be allowed to give orders to neighboring countries unless it be ascertained beforehand as an absolute fact that French works cannot deliver them in the time stipulated. Loans made by foreign countries in France should carry with them stipulations to the effect that the material and machinery required by the said foreign countries should be ordered from French works.

ELECTRIC TESTING BUREAUS IN GERMANY

Besides the Physikalisch-Technische Reichsanstalt in Charlottenburg which undertakes investigations and tests of the most various kinds, there are seven testing stations which are officially recognized as independent and impartial public laboratories for electrotechnological investigations (*Zeitschr. d. Ver. deutsch. Ing.*, 58 (1914), 559). These stations, at Ilmenau, Hamburg, Munich, Nuremberg, Chemnitz, Frankfort-on-Main, and Bremen, have been organized as a corporation with a main office at the laboratory in Nuremberg.

STEAM RAISING BY GAS COKE

The Journal of Gas Lighting and Water Supply has recently been devoting a good deal of space to the question of the advantages of gas coke for steam raising. E. W. L. Nicol discussing this subject in 125 (1914), 567, gives some comparative data, and reviews the present situation.

Raw bituminous coal is being used almost exclusively in England for steam raising, notwithstanding the enormous cost and inefficiency of distillation in an ordinary boiler furnace, and comparatively few engineers in charge of boiler plants have any first-hand knowledge or experience of coke fuel

for steam raising. An average of many analyses of ordinary bituminous steam coal (said to have been washed) shows the following:

Moisture Per cent	Fixed carbon Per cent	Volatile matter Per cent	Ash Per cent	Calorific value (dry) B. t. u. per lb.
10	45.6	32	12.4	12,500

When consumed in a boiler furnace for steam-raising, if any degree of efficiency is to be attained, the coal must be heated until the volatile gases are distilled off. This process absorbs about 10 per cent of the total heat in the coal. The distilled gases must then be liberally diluted with air, to burn them without excessive smoke production. This excess air must, of course, be heated to the furnace temperature, and is mostly rejected to the chimney as waste heat. As the diluted gas is ignited, it should be allowed to burn in a large brick-lined combustion chamber before coming in contact with the comparatively cool heating surfaces of the boiler, and the supply of air should be gradually diminished as the distillation process is completed. Meanwhile the solid residue or coke (which is smokeless, and requires about half the amount of air necessary for the complete combustion of the gas, to burn it under the best conditions) remaining on the fire-bars must be consumed in the same furnace at the same time. So it is evident when too much or too little air is being admitted to the furnace, whether the stoking be intermittent or continuous.

Gas coke is practically a simple mixture of carbon and ash, with little or no smoke-producing volatile matter. Uniform in quality and composition within fairly narrow limits, it requires no expert attention to obtain the most economical results during combustion, and the air supply to the furnace may be predetermined and fixed at the maximum desirable—the only adjustment left to the varying skill of the stoker being the fire thickness or density—one which cannot easily be neglected or abused.

Eminent authorities on the subject are agreed that the higher the volatile contents of a fuel, the more it is liable to smoke. The percentage of volatile matter in steam-raising fuel is in reality a measure of the difficulty of burning it under boilers with perfect combustion. Conversely, the lower the percentage of volatile matter, the greater the simplicity and efficiency of combustion. Low-volatile natural fuels are high-priced. They are known as anthracites and semi-anthracites; but the volatile contents of any solid fuel, as fired, may be adjusted to the most advantageous proportions by adding carbon in the form of coke.

The volatile contents of the coal detailed above might, with advantage, be adjusted by mixing with equal parts of coke as follows:

	Carbon Per cent	Volatiles Per cent	Calorific value B. t. u. per lb.
Coal.....	45.6	32.0	12,500
Coke.....	88.0	2.5	13,000
Mixture.....	133.6	34.5	25,500
	66.8	17.2	12,750

Typical South Wales anthracite analyses:

Carbon Per cent	Volatiles Per cent	Approximate B. t. u. per 1 d. (\$0.02) of cost, at, say, 25 s. (\$6.25) per ton
93	3	108,750

Good quality gas coke analyses:

Carbon Per cent	Volatiles Per cent	Approximate B. t. u. per 1 d. (\$0.02) of cost, at, say, 19 s. (\$4.75) per ton
88	2.5	130,000

In using so-called smokeless coal at comparatively high prices when gas coke is available at reasonable cost, steam users may not, therefore, be operating their plant to the best advantage.

All bituminous fuels, when fired under boilers, require very careful management, and large brick-lined combustion chambers must be provided if they are to be consumed without excessive

smoke or loss due to the escape of unburnt hydrocarbon gases.

Gas coke is smokeless, contains no hydrocarbons, and does not require any combustion chamber. It may, with advantage, be consumed in contact with the boiler-tubes and heating surfaces. Where gas coke is used, the combustion process is much easier to control, and the risk of loss in the form of unburned carbon (black smoke) and hydrocarbon gas is avoided.

All bituminous coals contain sulfur. On heating a boiler-furnace, the volatile sulfur is liberated and burnt to form SO_2 , which may cause serious damage by attacking the metal and furnace brickwork. In coke-fired water-tube boilers, tube failures, due to this cause, are comparatively infrequent, for the carbonizing process necessary to produce coke eliminates, to a great extent, this objectionable element.

Besides these data of Mr. Nicol, the journal presents (same volume, p. 363) the following figures, published by S. Tagg in the *Bulletin of the British Commercial Gas Association*:

POUNDS OF WATER EVAPORATED AT 100 LBS. PRESSURE PER POUND OF FUEL	
Coke screenings through $\frac{3}{4}$ -inch diameter perforations.....	5.0
Large screened coke.....	6.7
Best Arley rough slack.....	7.5

A MERCURY ENGINE

A paper read before the American Institute of Electrical Engineers by Mr. W. L. R. Emmet is reported in *Engineering* (London), 97 (1914), 498. In it is described some highly interesting experimental work undertaken to ascertain the feasibility of employing mercury as the working agent of a heat-engine. Mercury boils at 677°F . at atmospheric temperature, and condenses under a 28-in. vacuum at 455°F . The proposal is to use the mercury vapor in a turbine, using the exhaust to generate steam in a contrivance which is at once a condenser for the mercury and a boiler for the steam; the steam thus produced is to be utilized in a steam turbine. The physical properties of mercury render it, in some regards, very well adapted for use as the working agent in a turbine. Its high temperature is associated with a very moderate pressure. It is perfectly clean in use, so that its boiler will never require cleaning, and it appears from Mr. Emmet's experiments that, as it does not wet the blades through which it passes in the turbine, it has no tendency to erode them. Its high density makes possible a very moderate blade speed and a simple design of turbine. Some data as to the physical constants of mercury are quoted by Mr. Emmet as follow:

Specific heat of liquid mercury.....	= 0.0373	
Specific heat of gaseous mercury.....	= 0.0248	
Latent heat at 25 lbs. absolute.....	= 117	B. t. u.
Latent heat at 15 lbs. absolute.....	= 118	B. t. u.
Latent heat at 28 in. vacuum.....	= 121	B. t. u.
Latent heat at 29 in. vacuum.....	= 121.5	B. t. u.

The disadvantages of the mercury are its cost, which is about \$0.67 a pound, and its poisonous character if it escapes. There are, moreover, Mr. Emmet states, certain difficulties in confining both the vapor and the liquid, but these are not serious. As for the question of cost, the experiments made have led Mr. Emmet to think that not more than \$10 worth of mercury would be needed per kilowatt of output, and this is a higher and not a lower limit. A tandem mercury-steam plant would, he claims, show, a gain of 44 per cent on the output per pound of fuel burnt. A boiler of considerable size is now under construction in order to investigate the matter further.

A EUROPEAN VIEW OF THE MONROE DOCTRINE

The part played by the United States in the industrial situation in South America is the subject of an article by L. W. Schmidt in *Technik und Wirtschaft*, 7 (1914), 174. The author shows how the Monroe doctrine, as originally applied to the question of territorial holdings, although surrounded by a nebula of uncertainty and never officially recognized by European na-

tions, was nevertheless a powerful influence in international relations. Now it seems to be the intention of the United States to apply the doctrine to the field of commerce and industry, an intention proved, says the author, by Lord Murray's recent failure to obtain concessions in Ecuador, Colombia and Costa Rica. This application of the doctrine was backed by popular opinion which had been worked up to a fever heat by the canal tolls question and so was in a condition to swallow whole the rumor of a second isthmian canal projected by the Pearson interests which was so diligently exploited by the yellow press.

Naturally, the expansion of the Monroe Doctrine in this direction aroused the mistrust of Europe, particularly since Europe, being the largest investor in South American development, has had the biggest share in her trade. While, however, the exports of the United States were comparatively small at the end of the last century, they have since then increased enormously; exports to Central America and the West Indies have risen from \$100,000,000 to \$180,000,000 and those to South America from \$38,000,000 to \$132,000,000. The following table gives data for the years 1902, 1906, 1909 and 1912:

	UNITED STATES EXPORTS TO CENTRAL AND SOUTH AMERICA			
	1902	1906	1909	1912
British Honduras...	\$ 8,125,000	\$ 1,160,000	\$ 1,135,000	\$ 1,542,500
Costa Rica.....	14,762,500	2,455,000	2,422,500	3,830,000
Guatemala.....	17,650,000	3,072,500	1,792,500	2,645,000
Honduras.....	10,332,500	1,712,500	1,575,000	2,585,000
Nicaragua.....	14,267,500	1,967,500	1,422,500	2,612,500
Panama.....	13,082,500	17,637,500	24,725,000
Salvador.....	9,375,000	1,470,000	1,535,000	2,542,500
Mexico.....	41,867,500	61,090,000	52,282,500	55,490,000
Cuba.....	27,955,000	50,152,500	46,107,500	65,312,500
Argentine Republic	10,292,500	34,307,500	35,397,500	55,815,000
Bolivia.....	92,500	155,000	887,500	1,040,000
Brazil.....	10,910,000	15,257,500	18,405,000	36,412,500
Chile.....	3,900,000	9,100,000	5,765,000	16,267,500
Colombia.....	10,447,500	3,665,000	3,862,500	6,037,500
Ecuador.....	1,535,000	2,110,000	1,942,500	2,250,000
Paraguay.....	177,500	55,000	55,000	170,000
Peru.....	2,687,500	5,075,000	4,785,000	5,797,500
Uruguay.....	1,665,000	3,052,500	3,527,500	7,225,000
Venezuela.....	2,932,500	3,420,000	2,695,000	4,940,000

The reason for this increase is largely the fact that the development of commercial influence has gone hand in hand with the strengthening of intellectual influence. Where the rich young South American formerly went to Paris to round out his education, he now goes to New York or some other North American metropolis, and this indirect introduction of American ideas and ideals has found a more or less fertile soil in the unstable social and political conditions largely due to the mixed character of the population—whites, mestizos and Indians. While Europe, by investing large sums to develop natural resources, is expending her energy in preparing a sound economic foundation for the new race that will develop in South America, North America has followed other and more idealistic aims which may have a more lasting effect than the work of Europe.

Though the Monroe Doctrine has never seriously been put to the test, it has, nevertheless, had a real existence all this time, and has prevented any extended exercise of European influence in the southern continent. The United States has gone a step further, for in founding the Panamerican Union she has prepared a common ground for the interaction of common ideas and ideals which may, in time, aid in the formation of a possible confederacy, and at present aids in counteracting the great growth of European commercial influence. The evolution of this hundred-year old policy explains why the United States has left no stone unturned to prevent an influential European firm from obtaining land rights in Central America, where the possibility of a second interoceanic canal existed.

The history of South American commerce shows that those countries which have been in a position to invest the largest sums in commercial development have hitherto exercised the greatest influence, but that the United States is seeking her goal by other, perhaps more enduring, means and that she is looking ahead into a more distant future. But it is certain she cannot maintain her present rate of progress unless she is willin g

to play her part in the economic and industrial development of the continent as well as in the intellectual movement which is doing so much to draw the two Americas together.

REGULATIONS FOR ENFORCEMENT OF SO-CALLED NET WEIGHT LAW

The regulations for the carrying out of the so-called Net Weight Law, which compels manufacturers to make a clear statement of the weight, volume, or contents of their packages of food, were signed May 11th by the Secretaries of the Treasury, Agriculture, and Commerce. The regulations as signed become effective at once, although the law, passed March 3, 1913, as an amendment to the Food and Drugs Act, defers the exacting of penalties for violations until September 3, 1914.

These regulations apply to foods shipped in interstate commerce or sold in the District of Columbia or the territories and, in general, require that the manufacturer of foods shall plainly mark all packages, bottles or other containers holding more than 2 ounces avoirdupois, or more than 1 fluid ounce, to show the net weight or volume of the contents. The volume of liquids must be computed at 68° F. The quantity stated on the container must represent the actual quantity of food exclusive of wrappings and container.

In general, solids must be stated in terms of weight and liquids in terms of volume, except that where there is a definite trade custom otherwise any marking of the package in terms that are generally understood to express definite quantities will be permitted.

The regulations also permit the statement of minimum volume or weight as "Minimum weight, 12 ounces," "Minimum volume, 1 gallon," "Not less than 4 ounces." In such cases the amount stated must approximate the actual quantity. No variations below the stated minimum quantity will be permitted.

TOLERANCES

In the packing and bottling of many foods, it would be impossible, or else add unnecessarily to cost, for the manufacturer to place an absolutely accurate statement of the amount of the food in every package, and for this reason the regulations permit tolerances or variations in packages where the discrepancies are due exclusively to unavoidable errors in weighing, measuring, or counting which occur in packing conducted in compliance with good commercial practice. This tolerance is allowed in order to permit the use of weighing and measuring machines which, like human operators, cannot weigh or measure every package with absolute accuracy. The regulations, however, provide that a run of such packages must show as many cases of overweight and as much excess as it does cases of underweight or undervolume.

Because goods shipped from one part of the country to another lose in weight by natural evaporation due to differences of atmospheric condition or temperature, tolerances will be allowed for such changes. The proper tolerances to be allowed will be determined on the facts in each case, and it is probable that the Department will establish tolerances for evaporation for various foods.

Packages containing 2 ounces avoirdupois or 1 fluid ounce of food, or less, are considered small, and are exempted from marking in terms of weight since providing accurate small bottles

and accurately measuring their contents in the case of a number of articles sold in small packages for 5 to 10 cents would be prohibitive in cost to the manufacturers and would force them to raise the price for the package or to put less food in it for the same price.

RUSSIAN TRADE IN FERTILIZERS IN 1913

International unrest caused great uncertainty in the fertilizer market in Russia during 1913, and the decreased demand embarrassed both wholesalers and retailers. Instead of the previous yearly increase of 20 per cent in the use of fertilizer, the consumption remained the same as in the previous year, and indeed, for some kinds of fertilizer (*e. g.*, saltpeter) it decreased.

Chemie in its issue for March 28, 1914, gives the following data:

	RUSSIAN FERTILIZER IMPORTS (TONS)	
	1912	1913
Natural phosphates.....	52,128	58,878
Basic slag (Thomas meal).....	202,518	202,968
Superphosphates.....	206,838	202,968
Stassfurt salts.....	91,296	84,582
Potassium chloride and sulfate.....	6,642	3,744
Chile saltpeter.....	56,718	47,646
Potassium nitrate and nitrite.....	2,466	2,556

NATURAL GAS IN HUNGARY

Until recently little capital had been invested in natural gas in Hungary, but the beginning of large scale operations came when an enormous gas supply was accidentally discovered at a depth of 990 ft. in a search for potash at Kissármás near Sármas in April, 1909. The gas which is nearly pure methane has kept up its pressure since then at 28-32 atmospheres, and the amount delivered daily in this region is estimated at about 6,000,000 cubic feet, *Chem. Ztg.*, 38 (1914), 469.

The nearest large cities which offer a market for this gas for power and illumination are Klausenburg and Maros Vásárhely about 62 miles away; a much larger undertaking than a line to these cities is however being considered, for a line to Budapest, 250 miles distant is projected. Even with this expensive overland line, which is to be modeled on the American pattern, it is believed that natural gas can offer keen competition in the capital. The fact that the gas wells are located in a country with little water makes their local commercial utilization difficult, although a small beginning in this direction has been made in lighting the trains of the state railway.

In March, a 45-mile pipe line, the longest in Europe, was opened from Kissármás to Torda and thence to Marosujvár. The pipes, of drawn steel, are from 20 to 45 feet long and 5 to 10 inches in diameter. They are joined with a special rubber packing, are covered with pitch and cased in an asphalt-jute coating. The country through which the line is laid offered great difficulties—among others frequent land slides; the cost of the line was in the neighborhood of \$700,000.

The "Methana" Erdgas-Studiengesellschaft has been organized in Budapest to study the chemical possibilities of the situation, for in the gas country there are rich supplies of mineral products; the problem of manufacturing nitric acid from the air by the Bender process is also being studied.

In the northwestern part of Hungary too, a supply of gas, accompanied here with oil, was discovered as recently as the end of 1913. The first well is yielding 1½ carloads of oil a day and about 17,000 cubic feet of gas (95 per cent methane) are given off hourly.

SCIENTIFIC SOCIETIES

INDUSTRIAL CHEMISTS AND CHEMICAL ENGINEERS DIVISION—REPORT OF PROGRESS BY COMMITTEE ON QUALITY OF PLATINUM LABORATORY UTENSILS, APRIL 9, 1914

The Committee on Quality of Platinum Laboratory Utensils submits the following report of progress, the first of a formal

character since the preliminary report published in *This Journal*, 3 (1911), 686:

The committee itself has been unable to do more than to continue for a time the line of experimental work described in the report above referred to. The results are confirmatory in general of those already reported and need not be set forth in de-

tail here. The main reason for the long delay in securing data of sufficient importance to communicate formally has been due to the difficulties encountered in devising suitable means for accurately determining the volatility losses suffered by commercial ware when subjected to the temperatures ordinarily employed in laboratory operations. These difficulties have been, it is hoped, overcome through the assistance of the Heat and Metallurgical Divisions of the Bureau of Standards, and it is expected, as opportunity permits, to accumulate data of value.

There is, however, another more serious difficulty arising from the fact that the committee has been unable to obtain reliable information on the composition of much of the ware which is at its disposition, either through loan or purchase. Until exact data of this kind are available it will be difficult, if not impossible, to determine all the causes of poor quality in ware or to explain certain differences observed in the behavior of ware from different sources. This information, it is realized, is not to be had from the makers, but can be gained only by carrying out an elaborate investigation involving the preparation of pure metals and of some of their alloys and also by the careful analysis of commercial ware. It is hoped that in time the Bureau of Standards may be able to take up such an investigation, which it is quite impossible for the committee to assume. This Division, or better still the Society, could lend most valuable support to a project of this kind by urging upon the proper authorities the great need for a thorough study of the platinum metals and their alloys, a study which will involve the expenditure of a considerable sum of money. The investigation should not be restricted to a study of the subject from the point of view of the chemist alone, but should be made comprehensive as to the physical constants and physical behavior of the metals and alloys, so that all users of the platinum metals might benefit.

In the meantime, however, Dr. G. K. Burgess, of the Bureau of Standards, assisted by Mr. P. D. Sale, has developed a method for determining with exactness and rapidity the total impurity in any platinum alloy in terms of iridium, which is the most common associate of platinum in commercial ware. This method depends upon the measurement of the electromotive force of the alloy against pure platinum at a given temperature. So far the method has shown itself very reliable for platinum containing known amounts of iridium, and its application will be extended, if suitable materials can be obtained, to other binary alloys of platinum and its common associates. At present it is not possible to determine what other metal or metals may be alloyed with platinum, but, as said above, their joint effect can be given in terms of iridium. The determination takes but a few minutes. The method is printed in full in this issue (p. 452) in the form of a paper by Messrs. Burgess and Sale, with the results obtained on the various samples loaned to our committee by American manufacturers of platinum ware, as well as upon articles of English, German and French make and some purchased specimens of American make.

Since this work is a direct outcome of the activity of our committee, we feel that substantial progress has been made, notwithstanding that we have no experimental data of importance to present on our own immediate behalf, and we take this opportunity to express to Messrs. Burgess and Sale and to the Director of the Bureau of Standards our appreciation of their efforts to forward our work.

It was hoped that, after the publication of our first report, American manufacturers would furnish to purchasers commercial ware of decidedly better quality, as to iron and perhaps other base metal content, than they had been offering in recent years. This hope has proved illusory as to some at least of the ware now sold, so far as we can judge from specimens recently submitted for test at the Bureau of Standards, on request of one of us.

In order that more information may be acquired as to the quality of ware sold from now on, the committee asks purchasers to submit to it one or two specimens (particularly crucibles) from each lot of new and unused ware. The committee is authorized to say that these samples will be examined at the Bureau of Standards (for the present without charge) in such manner as not to injure the articles, and reports will be made to the senders. The tests will be directed, so far as can now be foreseen, toward the detection of iron or other base metal (as shown in part by the appearance of the surface of the ware after ignition) and to the determination of the sum of alloyed metals in terms of iridium by the thermoelectric method above mentioned. The determination of loss on prolonged heating at high temperatures will not, for the present, figure in the tests. The Bureau will reserve the right to make such use of the results of its examination as it may find desirable.

It is believed that the adoption of this course by a sufficiently large number of users of platinum utensils, during the next few years, will bring about speedy improvement in the quality of commercial ware. If not, the committee will probably make other recommendations for the protection of the public.

W. F. HILLEBRAND
E. T. ALLEN
PERCY H. WALKER

AMERICAN CHEMICAL SOCIETY—49th MEETING, CINCINNATI, APRIL 6-10, 1914 FERTILIZER CHEMISTRY DIVISION REPORT

The meeting was called to order by Mr. J. E. Breckenridge, Chairman. After the transaction of the usual routine business, the papers were presented as published in the official program.

An amendment to the by-laws was adopted, making a general Committee on Research and Methods of Analysis in place of the Committees on Nitrogen, Phosphoric Acid, Phosphate Rock and Potash.

The meeting was very well attended considering the conditions which would enforce the absence of a good many of the members at this season of the year.

F. B. CARPENTER, *Secretary*

REPORT OF THE COMMITTEE ON FERTILIZER LEGISLATION

The question of uniform fertilizer legislation having occupied the attention of this division for some time, the following resolution adopted by the Association of Southern Agricultural Workers at the annual meeting in Montgomery, Ala., Feb. 26th, will be of interest:

"WHEREAS, the wide variation in the requirements now existing in our Southern States for the branding of fertilizer packages are a source of confusion to purchasers, a waste of time and liability to errors in inspection, and an unnecessary expense to manufacturers,

"THEREFORE, BE IT RESOLVED, that this Association endorses uniformity and simplicity in branding, and urges the officials in charge of fertilizer control in our Southern States to use their influence in securing such legislation as will bring about simplicity and uniformity in branding of commercial fertilizers."

Your committee was represented at this meeting and participated in the discussion.

In the matter of new legislation there have been a few minor changes since the last report, but not of such a nature as to materially affect either the manufacturer or consumer.

F. B. CARPENTER, *Chairman*

REPORT OF COMMITTEE ON PHOSPHORIC ACID

This Committee has been asked to prepare a résumé of the work done since its organization, for the guidance of the Committee on Methods of Analysis.

This Committee first turned its attention to the determination

of insoluble phosphoric acid, since it thought a method which would give more concordant results was needed.

Since there are several factors which influence the determination of insoluble, such as the neutrality of the ammonium citrate, time required for filtering insoluble, etc., the best way of attacking the problem seemed to be to vary one of the factors at a time, keeping the others constant.

The first question taken up was the preparation of neutral ammonium citrate. A solution of citrate was prepared by one of the members of the Committee and sent out with the samples. Those to whom samples were sent were asked to prepare a citrate solution and run samples with the solution prepared by them and also with that sent out by the Committee.

The results¹ showed that it was possible to prepare a solution which would give results which were the same as those obtained by the Committee citrate, but results from different analysts showed great variation between them. One of the chief causes of this great variation was due to the great difference in time required for filtration.

Another set of samples was prepared and together with these samples was sent a standard sample on which the Committee had determined the total phosphoric acid.

The total in standard sample ran about the same as the highest insoluble. The instructions which accompanied were the same as those sent out with the previous lot of samples, except that the method of filtration was shortened by transferring the precipitate to another filter in cases where filtration was difficult. The results² showed some improvement in the case of goods difficult to filter, but in the case of acid phosphate the variation between analysts was the same.

The results obtained on the last set of samples are somewhat disappointing. The differences do not follow any rule, hence it is difficult to assign any reason for the variation. The most probable cause of the variations is due to the failure, on the part of the analyst, to observe the details of the method closely. Analysts who have had considerable experience with the method think that much better results should be obtained where the details of the method are closely observed.

COMMITTEE { J. Q. BURTON
W. J. JONES
J. R. POWELL
G. FARNHAM, *Chairman*

REPORT OF THE ANALYTICAL COMMITTEE—RUBBER SECTION AMERICAN CHEMICAL SOCIETY

[The following report was presented to the Rubber Section of the A. C. S. at Cincinnati on April 8, 1914, and the Section authorized its publication for the purpose of bringing the results obtained to the attention of those interested in the work and in answer to the Joint Rubber Insulation Committee's request for criticisms. DORRIS WHIPPLE, *Secretary*.]

Your Committee begs to present herewith the third report of the investigations made by this Committee, together with the results obtained.

The major part of the work of this Committee has been confined to certain methods of procedure as outlined by the *Joint Rubber Insulation Committee* for the analysis of materials used for insulating purposes. The results obtained on previous work were presented to the members of the Rubber Section at the Milwaukee meeting and at the Rochester meeting. The Committee has not included the previous work in the present report (although the same has a direct bearing on the present report) as some of the methods have been changed or revised. In the time intervening between the Rochester meeting and the present day, this Committee has investigated the completed preliminary procedure of the *Joint Rubber Insulation Committee* and these final results are the ones tabulated below.

¹ THIS JOURNAL, 3 (1911), 118.

² *Ibid.*, 5 (1913), 956.

The determinations as reported herewith were made on three samples which were prepared and averaged with the greatest care by Mr. G. H. Savage and the formulas used by him are given below in detail in as much as it has a direct bearing on the present report.

The various ingredients which went to make up these three compounds were weighed with extreme accuracy and compounded with the greatest care in order that uniform average samples might be obtained. The vulcanized compounds were then ground according to the requirements of the procedure under investigation, carefully mixed and placed in small vials which held the approximate amount required for the various determinations. These were sent by express to the various members and the analysis on the first determination started by each member within approximately twenty-four hours of the time of grinding the sample. These samples were re-analyzed at intervals as indicated in the report.

This Committee respectfully refers the members of the Rubber Section to THIS JOURNAL, 6, 75, for the complete method of procedure of the *Joint Rubber Insulation Committee* as followed by this Committee in the analysis of the three samples prepared by Mr. Savage.

The formulas of the three compounds, together with the analytical results obtained, are tabulated below.

FORMULAS USED IN PREPARING COMPOUNDS ANALYZED

	A	B	C
Fine Para.....	297	309	324
Hard Paraffin.....	25
Ceresine.....	..	25	..
Sulfur.....	19	19	19
Litharge.....	70	70	70
Zinc Oxide.....	350	350	350
Whiting.....	239	227	237
	1000	1000	1000

No corrections, additions or omissions have been made in reporting these analytical results. The analysts' numbers refer to the following: 1—E. W. Boughton; 2—D. W. Whipple; 3—W. A. Ducca; 4—G. H. Savage; 5—J. B. Tuttle.

SAMPLE A—FIRST ANALYSIS

Analyst's No.....	1	1	2	2	3	4	4	5
Date of Extraction with Acetone.....	11/13	11/15	11/14	11/20	11/20	11/13	11/13	11/13
Total Acetone Extract (%).....	4.16	4.12	3.97	4.03	4.18	3.84	3.78	4.08
Free Sulfur (%).....	0.87	0.86	0.80	0.82	0.90	0.70	0.66	0.82
Unsaponifiable Material (%).....	2.97	2.70	2.76	..	3.29	2.71	2.69	2.69
Waxy Hydrocarbon A (%).....	2.29	2.25	2.18	..	2.27	2.39	2.31	2.40
Waxy Hydrocarbon B (%).....	0.34	0.20	0.32	..	0.56	0.14	0.20	0.11
Total Waxy Hydrocarbon (%).....	2.63	2.45	2.50	..	2.83	2.53	2.51	2.54
By Difference								
Organic Extract (%)	3.29	3.26	3.17	3.21	3.28	3.14	3.12	3.26
Saponifiable Acetone Extract (%).....	0.32	0.56	0.41	0.43	0.43	0.57
Unsaponifiable Resins (%).....	0.34	0.25	0.26	0.18	0.18	..

SAMPLE A—SECOND ANALYSIS

Analyst's No.....	1	1	2	2	3	4	4	5
Date of Extraction with Acetone.....	11/20	11/21	11/25	11/23	12/4	11/20	11/20	11/20
Total Acetone Extract (%).....	4.13	4.16	4.14	4.02	3.72	3.90	4.00	4.20
Free Sulfur (%).....	0.88	0.79	0.88	0.80	0.89	0.65	0.65	0.91
Unsaponifiable Material (%).....	2.32	2.75	2.82	2.81	2.54	2.41	2.51	2.60
Waxy Hydrocarbon A (%).....	1.97	2.31	2.31	2.37	2.32	2.11	2.32	2.51
Waxy Hydrocarbon B (%).....	0.17	0.24	0.25	0.19	0.49	0.10	0.10	0.11
Total Waxy Hydrocarbon (%).....	2.55	2.64	2.56	2.56	2.81	2.21	2.42	2.62
By Difference								
Organic Extract (%)	3.25	3.37	3.26	3.22	2.83	3.25	3.35	3.29
Saponifiable Acetone Extract (%).....	0.93	0.62	0.44	0.41	0.29	0.84	0.84	0.69
Unsaponifiable Resins (%).....	0.18	0.20	0.26	0.25	0.09	..
Fillers (%) Sulfur-free.....	..	70.7	..	71.6(a)	..	72.68	..	71.1

(a) This determination was not made by the same analyst.

lished, one of which concerns the determination of fillers and the other, the division of resins into saponifiable and unsaponifiable.

There has been but one compound analyzed, of which there are three variations: one with paraffin, one with ceresin and one without waxy hydrocarbons. We understand that these were made from the same lot of raw rubber. They contain practically the same percentages of the various mineral ingredients, so that the determinations of fillers and of resins are practically a repetition of the determinations of one compound. The Analytical Committee draws general conclusions as to the value of these determinations from the results on this one compound.

No data are given on the raw rubber and we cannot judge whether it contained the normal amount of insoluble matter. From the tables of the Analytical Committee it appears that all the results on fillers are too high. It is possible that the compound used may have certain characteristics which magnify the errors of the determination. It is known to the Committee that errors may occur and the subject is being investigated. It has been the experience of the Committee that the magnitude of these errors is less after practice with the procedure.

We have assumed above that all members of the Analytical Committee followed the prescribed procedure absolutely. An examination of the results on the different constituents of the acetone extract indicates that the procedure has not been followed by all the members of the Analytical Committee.

In this connection we would like to call attention to the following note in the tentative report of this Committee (THIS JOURNAL, 6, 81): "With a procedure of this length, it is impossible to explain every detail without undue elaboration and the Committee wishes to point out that while to experienced chemists the procedure may seem overburdened by detail, yet every specified detail was found necessary in order that the conditions essential to accurate and consistent work might be reproduced by all chemists using the procedure. For this reason it is extremely important that all instructions be observed even if their significance is not perceived by the individual chemist. It will probably be found that even with the instructions properly observed, some experience will be needed to apply the method successfully."

The results given for Compound A (11/20 and 11/21) under No. 1 on Total Waxy Hydrocarbons, contain what may be assumed to be a clerical error. However, in all the results given under No. 3 we can find no clerical error to account for the absurdity of the results. For instance, in every case but one there is a greater value given for total waxy hydrocarbons than for unsaponifiable material, which, according to the procedure, is an absolute impossibility. We do not attempt to explain the results, but we do point out that the results are necessarily in error and cannot be considered in judging the procedure.

The results under No. 4 for total acetone extract and free sulfur are consistently low. If the results under Nos. 1, 2 and 5 are averaged and compared with the average results obtained by No. 4, it will be seen that the lower results on free sulfur probably account for the lower acetone extract of No. 4.

Sample	Act. Ext.	Free S	Act. Ext.	Free S	Act. Ext.	Free S
Av. Nos. 1, 2 and 5.....	4.07	0.83	4.13	0.85	4.07	0.86
Av. No. 4.....	3.81	0.68	3.95	0.65	4.05	0.74
Difference.....	0.26	0.15	0.18	0.20	0.02	0.12
Sample B						
Av. Nos. 1, 2 and 5.....	4.00	0.76	4.05	0.79	3.98	0.79
Av. No. 4.....	3.72	0.55	3.98	0.71	3.99	0.82(a)
Difference.....	0.28	0.21	0.07	0.08	-0.01	-0.03
Sample C						
Av. Nos. 1, 2 and 5.....	1.49	0.77	1.56	0.78	1.47	0.77
Av. No. 4.....	1.12	0.54	1.40	0.68	1.43	0.67
Difference.....	0.37	0.23	0.16	0.10	0.04	0.10

(a) Very poor checks

These results indicate that the acetone extraction was not properly made. The Committee believes that the prescribed form of extraction apparatus was not used, and that used would

account for the loss of free sulfur and the consequent lower acetone extract.

Another apparent discrepancy in the results can be explained on close examination. If we average all the results on saponifiable acetone extract, obtained by Nos. 1, 2, 4 and 5, on each variation of the compound analyzed, we obtain the following:

Analyst's No.	1	2	4	5
Sample A.....	0.55	0.40	0.60	0.63
Sample B.....	0.60	0.48	0.66	0.79
Sample C.....	0.47	0.50	0.52(a)	0.65

(a) Eliminating results of 0.10 and 0.12, 11/13, obviously in error.

The averages given under No. 2, on Samples A and B are lower than those obtained by Nos. 1, 4 and 5, and on examination of the acetone extracts obtained by No. 2, it will be seen that he obtained consistently lower results than Nos. 1, 2 and 5. The results indicate that the acetone extraction was not properly made and that this has probably caused lower saponifiable resins. If we, therefore, eliminate the results of No. 2, those obtained by Nos. 1, 4 and 5 for "A" and "B" are about what we would expect from this compound. When we come to results for "C," we do not see how any conclusions can be drawn.

The object of the separation of the resins into saponifiable and unsaponifiable was to differentiate between different rubbers, and we are sorry that the Analytical Committee did not investigate other rubbers in order to show the differences between them.

Considering the results as a whole we are of the opinion that the report of the Analytical Committee would have had more bearing on the procedure under consideration if that procedure had been carefully followed.

We hope that the work of the Analytical Committee will lead to other criticisms in order that we may avail ourselves of the experience gained this year, and incorporate whatever improvements we may decide upon in a later report.

JOINT RUBBER INSULATION COMMITTEE

WM. A. DEL MAR, Secretary

April 29, 1914

AMERICAN INSTITUTE OF CHEMICAL ENGINEERS 6th SEMI-ANNUAL MEETING, TROY, JUNE 17-20, 1914 PROGRAM OF PAPERS

1. Address of Welcome. HONORABLE C. F. BURNS, Mayor of Troy.
2. Presidential Address. M. C. WHITAKER.
3. The Saratoga Septic Tanks (Lantern). WM. P. MASON.
4. The Application of Physical Chemistry to Industrial Processes. W. F. RITTMAN.
5. Studies on Filtration. J. W. BAIN and A. E. WIGLE.
6. Scrubber for Vacuum Apparatus for Laboratories. CHARLES BASKERVILLE.
7. Shoddy and Carbonized Waste. L. J. MATOS.
8. A Combination Water Softener and Storage Tank (Lantern). L. M. BOOTH.
9. The Present Patent Situation. M. TOCH.
10. Ozone and Ventilation. J. C. OLSEN and WM. H. ULRICH.
11. Bleaching Cotton Fiber. J. C. HEBDEN.
12. Development of Rotary Furnaces (Lantern). R. K. MEADE.
13. An Oil Shale from Nevada (Illustrated). CHARLES BASKERVILLE.

EXCURSIONS

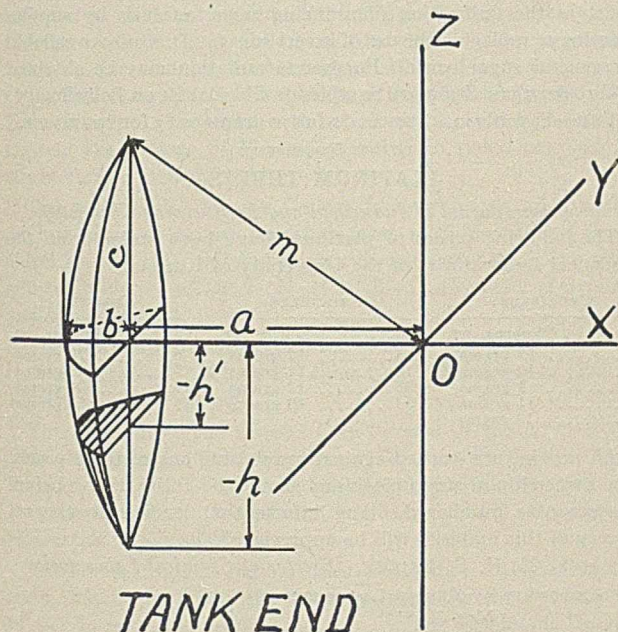
- Laboratories of Rensselaer Polytechnic Institute.
U. S. Arsenal (Heavy Guns).
Geo. P. Ide & Co. (Collars and Shirts).
General Electric Co. Plant at Schenectady.
Saratoga (The N. Y. State Reservation, the Springs and Sewage Disposal Plant).
Water Works at Albany (Slow Sand Filter Beds and Mechanical Scrubbing Filters).
West Virginia Pulp & Paper Co. Plant at Mechanicsville.
Burden Iron Works (Puddling Process for Wrought Iron).
Cohoes Filter Plant (Mechanical Filters).
Freihoffer Baking Co.

NOTES AND CORRESPONDENCE

FORMULA FOR CONTENTS OF CYLINDRICAL TANKS WITH SPHERICAL ENDS

Editor of the Journal of Industrial and Engineering Chemistry:

In most all chemical plants of any size many cylindrical tanks with spherical ends are used for liquids. To ascertain contents of these tanks by measuring the depth of the liquid inside, it is necessary to consult a curve representing the gallons per inch of depth. It is a comparatively simple matter to calculate the volume per inch of the main portion of the tank, but calculating that of the spherical ends is a more complicated problem.



The DuPont Fabrikoid Co., manufacturers of artificial leathers, use about ten of these cylindrical tanks with spherical ends. They are mostly of different sizes and I was given the task of plotting the necessary curves. I have worked out the following formula:

$$V = 2 \int_{-h}^{-h'} \int_a^{\sqrt{m^2 - z^2}} \int_b^{\sqrt{m^2 - x^2 - z^2}} dz dx dy$$

The limits are obtained from the equation of the sphere,

$$x^2 + y^2 + z^2 = m^2, \text{ where } m = \frac{c^2 + b^2}{2b}$$

By integration and expansion, $V = [Az + Bz^3 + Cz^5]_{-h}^{-h'}$

$$\text{where, } A = \frac{\pi}{2} m^2 - am - \frac{a^3}{6m} - \frac{3a^5}{40m^3} - ac$$

$$B = \frac{a}{6m} - \frac{\pi}{6} - \frac{a^3}{36m^3} - \frac{3a^5}{80m^5} + \frac{a}{6c}$$

$$C = \frac{a}{40m^3} - \frac{a^3}{80m^5} - \frac{9a^5}{320m^7} + \frac{a}{40c^3}$$

Substituting the value of $m/2$ for z in the formula, we obtain the volume in both ends of half the tank, $V_{1/2}$. By substituting h , the depth as measured for z in the formula and subtracting this volume, V_z , from $V_{1/2}$, we get the volume in the ends for the desired depth of liquid,

$V = V_{1/2} - V_z$, or the formula becomes:

$$V = [A \frac{m}{2} + B \frac{m^3}{8} + C \frac{m^5}{32}] - [Ah + Bh^3 + Ch^5]$$

L. E. CARPENTER

110 GRAND STREET, NEWBURGH, NEW YORK
February 17, 1914

NOTE ON INFLUENCE OF RATE OF STIRRING ON TITRE DETERMINATIONS IN FATS

Editor of the Journal of Industrial and Engineering Chemistry:

At the time that the official method now in use in determining the Titre of Fats was adopted, it was noted that the stirring method, *i. e.*, continuous stirring of the fatty acids until the temperature was stationary for 30 seconds, gave tests higher than if the fatty acid was cooled to the stopping point without stirring; this higher test ranged from over 1° C. in case of oils such as Cottonseed to only 0.1° or less in the case of Stearine.

Since this stirring method was adopted, the custom in this laboratory has been to stir the fatty acids by means of a stirrer run by a water-motor and it was soon noticed that the number of revolutions per minute of the fatty acids (the acids being revolved and the thermometer kept stationary) had a decided influence on the test. I give below a number of figures made in two different fats, the first being a Garbage Grease (Naphtha extracted) and the second a tallow:

	Revolutions per min.	Stopping point	Final highest point
Garbage Grease	60	36.35	37.05
	80	36.50	37.20
	180	37.20	37.55
	220	37.40	37.65
Tallow	60	43.60	44.60
	60	42.70	44.50
	180	44.60	45.00
	220	44.60	45.00

It will be noticed that when the speed is only 60 revolutions the first stopping point is much lower than when it is 180 or 220, in fact, the less the speed the lower the first stopping point and as the speed is increased, the stopping point rises with each increase.

In the case of the tallow, the rate above 180 makes no difference with either temperature but with the grease a difference of 0.1° is noted. A quite remarkable difference in the first stopping point is noted in the tallow at 60 revolutions, although the final temperature is practically the same.

In my opinion, a large number of the differences between chemists on this test is due to this point. At the present time the directions state only that the acids "shall be stirred slowly," which might mean anything. I believe that a definite rate of stirring should be specified and my judgment is that 180 revolutions should be taken as the standard, as a higher rate is rather impracticable and a lower certainly does not give correct figures. As the titre point is the highest temperature recorded by the heat of crystallization of the fatty acids, any method that will give this highest heat should be the correct one, as it is obvious that the only source of heat is from the crystallizing. In fact, a higher rate of stirring would, if anything, tend to cool the acids and so give a low test.

A. G. STILLWELL

76 1/2 PINE ST., NEW YORK
March 30, 1914

A SHAKER FOR THE MECHANICAL ANALYSIS OF SOILS

Editor of the Journal of Industrial and Engineering Chemistry:

In an article in February, 1914, THIS JOURNAL, entitled "A Shaker for the Mechanical Analysis of Soil," by Freeman Ward, the author makes a comparison between the shaking machine devised by him and the one used by the Bureau of Soils. He states: "The writer believes that his method is better than the one used by the Bureau of Soils in two respects. In the first place, the machine is simple and inexpensive. A greater advantage is the saving of time. Clean separations can be effected in three hours, and in some cases two hours; this is a saving of at least four hours over the Bureau method. If the quantity of the water used in the bottles is reduced one-half, a clean separation results in much less than three hours, but there is a tendency for the grains to suffer some abrasion."

The machine is undoubtedly simple and inexpensive. Attention must be called to the fact, however, that the Ward apparatus carries only 16 bottles while the Bureau of Soils apparatus carries 48, giving the latter three times the capacity. The Ward shaker must be run at a certain definite speed to be efficient. If the speed is too high, there is a centrifugal effect resulting, and if too slow, the action is merely pouring. The Bureau of Soils machine is even more efficient at high speeds than at moderate speeds.

As to the second conclusion, that a saving of time results, the author has taken the statement in Bureau of Soils Bulletin No. 84 that the samples of soil are "shaken by the mechanical shaking machine for at least seven hours," to mean that seven hours are essential. As a matter of fact, this time is taken because in some rare instances as long a time as this has been found necessary. A majority of soils, especially sandy soils, do not need seven hours. The length of time of shaking does not result in any loss of time to the operator, as the analyst merely keeps 48 soils shaking on the machine all the time and cannot complete the analyses rapidly enough with a force of three men to keep the machine cleared of samples ready for analysis. If more samples should be required, all that is necessary is to run the machine during the night, and thus the capacity of the machine would be doubled.

In the light of these facts, it seems to the writer that Mr. Ward's statement that his "method is better than the one used by the Bureau of Soils" is misleading. A more accurate claim would be that his machine is satisfactory where such a small amount of work is done as to preclude buying an expensive piece of apparatus. His machine can be cheaply constructed and is greatly superior to pestling.

C. C. FLETCHER

BUREAU OF SOILS

U. S. DEPARTMENT OF AGRICULTURE, WASHINGTON

February 24, 1914

A NOTE ON THE QUANTITY OF BENZOIC ACID CONTAINED IN PRUNES AND CRANBERRIES

Editor of the Journal of Industrial and Engineering Chemistry:

Although it is well recognized that certain fruits contain relatively large amounts of benzoic acid or its salts, data specifically stating the exact quantities are not numerous. In connection with work in progress in this laboratory, it became desirable to determine the quantity of benzoic acid in prunes and in cranberries.

For the estimation of benzoic acid the method of Krüger (*Zeitschr. f. Untersuch. d. Nahr. u. Genussmittel*, 1913, July) was employed, 50 grams of fruit being taken for a determination.

From several duplicate analyses in each instance it was found that prunes as obtained in the market contained approximately 0.05 per cent benzoic acid. Cranberries have a somewhat higher benzoate content, benzoic acid to the extent of 0.06 per cent being found.

These results were furnished from the weight of sublimed

benzoic acid and by titration—the data in each instance agreeing closely.

MORRIS J. RADIN

SHEFFIELD LABORATORY OF PHYSIOLOGICAL CHEMISTRY
YALE UNIVERSITY, NEW HAVEN, CONN.

March 7, 1914

BUREAU OF STANDARDS' ANALYZED SAMPLES

The Bureau of Standards is prepared to issue purified dextrose as a standard reducing sugar. The substance is free from ash and contains less than 0.1 per cent of moisture. In an atmosphere of average humidity it is slightly hygroscopic, but if dried at 70° C., it retains less than 0.01 per cent of moisture. A certificate of analysis showing the quantity of remaining impurity accompanies the sample. The purpose of the standard is to assist in the unification of reducing sugar analysis by supplementing or replacing the use of invert sugar. It is, also, available for general sugar work. Further information may be obtained in Circular 25 on Standard Samples or Circular 44 on Polarimetry.

FEE—Payable in advance: \$2 for 70 grams or \$3 for 140 grams.

PLATINUM THEFTS

Editor of the Journal of Industrial and Engineering Chemistry:

The following pieces of platinum have been stolen from the Chemical Laboratories of the University of Kansas:

CRUCIBLES		CRUCIBLES		DISHES	
No.	Approx. wt.	No.	Approx. wt.	No.	Approx. wt.
3.....	12 grams	4.....	12 grams	6.....	20 grams
7.....	12 grams	10.....	11 grams	7.....	20 grams
27.....	8 grams	25.....	12 grams	24.....	24 grams
1.....	11 grams	33.....	13 grams	25.....	25 grams
5.....	12 grams				

All persons are warned against purchasing any of these pieces. The ownership of scrap platinum should be fully traced before the scrap is purchased. Any information leading to the recovery of this material will be appreciated.

E. H. S. BAILEY, *Director of Chemical Laboratories*

UNIVERSITY OF KANSAS, LAWRENCE

May 7, 1914

Editor of the Journal of Industrial and Engineering Chemistry:

Platinum valued at some six hundred dollars disappeared from one of the Chemistry Laboratories of the University of Minnesota sometime on Monday, April 27, 1914. This platinum consisted of eighteen crucibles—Nos. 20, 21, 22, 25, 26, 27, 29, 39, 40, 41 and 45 with covers of the same numbers and crucibles Nos. 35, 36, 37, 42, 44, 47 and 52 without covers; also one platinum dish that weighed about 43 grams.

If your readers hear of any platinum being offered for sale or have had any experience in the recovery of such property that might help us, we shall appreciate their immediate cooperation.

J. C. POUCHER

UNIVERSITY OF MINNESOTA, MINNEAPOLIS

May 5, 1914

PERSONAL NOTES

Dr. J. A. Watkin, of the U. S. Public Health Service, has been sent to Pittsburgh by the Secretary of the Treasury to investigate the effect of mill environment on the health of employees. This work, which constitutes a governmental innovation, will last until the end of the year.

Dr. Francis H. Eckhardt, research chemist for the Grasselli Company, died at the age of 46 on April 3rd at his home in Elizabeth, N. J.

Thomas H. Johnson, consulting engineer to the Pennsylvania Lines West, died in Pittsburgh on April 16th.

Prof. C. F. Chandler is the guest of honor at the 7th annual Dinner of the Columbia University Alumni Club of Philadelphia, April 17th, at the Hotel Adelphia. The speakers were

Professor Chandler, C. H. Mapes, Lemuel Whitaker, Robt. Arrowsmith, G. B. Compton and Arthur H. Elliot.

Prof. Alan W. C. Menzies, now head of the Department of Chemistry at Oberlin College, has been elected Professor of Chemistry at Princeton University.

Data collected recently by the College of Engineering of the University of Illinois show that of the 2,165 graduates, 1,933, or 89 per cent, are engaged in engineering work and that only 173 or about 8 per cent have gone into other fields.

Mr. H. de B. Parsons, of the Metropolitan Sewerage Commission, gave an illustrated talk on the plans as proposed by the Commission before the American Society of Mechanical Engineers, New York, May 12, 1914.

Among the members of the National Academy of Sciences elected at the annual meeting on April 23rd were the following: Moses Gomberg, University of Michigan; Edward Curtis Franklin, Stanford University; Francis Gano Benedict, Carnegie Institution.

Dr. Chas. L. Parsons spoke on "The Radium Work of the Bureau of Mines" before the North Carolina Section of the A. C. S., May 1st.

Messrs. Carnahan, Ahlbrandt and Aupperle, of the American Rolling Mills Co., addressed the Columbus Section of the A. C. S., May 8th, on "The Manufacture and Properties of Pure Iron."

The establishment of the Isaac Plaut Fellowship, the first traveling pharmaceutical fellowship in this country, was announced at a recent meeting of the Board of Trustees of the College of Pharmacy, Columbia University. The fellowship has been founded by Vice-President Albert Plaut, in memory of his father, for the encouragement of graduate study and original research. The fellow is to be selected from the graduating class in the College of Pharmacy and is to spend one year in study at a foreign university.

The trustees of Clemson College have appropriated \$300 for an investigation of the limestone and marl deposits of South Carolina and their value for agricultural purposes. The work will be in charge of Dr. F. H. H. Calhoun, Professor of Geology and Mineralogy.

The Southern California Section of the A. C. S. was addressed on May 21st by Dr. Chas. P. Lipman on "The Effect of Smelter Wastes on Plant Growth."

The program of the Kansas City Section of the A. C. S., on May 16th, included the following: "The Rare Earths and Their Separation," Dr. D. L. Randall, Baker University; "Recent Progress in Organic Industrial Chemistry," Mr. J. B. Whelan, University of Kansas.

The Forest Products Laboratories of Canada were established in the spring of 1913 under the Forestry Branch of the Department of the Interior and located on the grounds of McGill University at Montreal. The laboratories are to be modeled somewhat after those at Madison, Wisconsin, and are designed to study the best methods of utilizing the various native woods. Mr. John S. Bates, formerly with the Arthur D. Little, Inc., of Boston, was appointed Superintendent of the Laboratories on April 1st. Mr. Bates is also in direct charge of the Division of Pulp and Paper, which is the most important in the organization. An experimental paper mill capable of handling products on a semi-commercial scale is being equipped. The Timber Testing Division is now fully organized for Mechanical Testing and the Timber Physics Division is being started. Departments of Wood Preservation, Wood Pathology, etc., will be organized later. The scientific investigations are intended to act as a stimulus to wood-using industries and the organization will also serve as a Bureau of Information on Forest Products in Canada.

The U. S. Department of Agriculture has established an office in the Bureau of Chemistry for the promotion of a closer and more cordial cooperation among the city, state and federal food and drug officials of the country in the enforcement of the food and drug laws. Mr. J. S. Abbott, for nearly seven years dairy and food commissioner of Texas, was appointed to this office and began active service on April 3, 1914.

Prof. William Campbell, of Columbia University, spoke before the Rochester Section of the A. C. S. on April 6th, the subject being, "Some Uses of Metallography as a Method of Testing Materials."

The Louisiana Section of the A. C. S. was addressed at its 77th meeting, May 15th, by Mr. Chas. S. Williamson, on "Factors Influencing Soil Fertility—Some Modern Developments."

The following poem was written and read by Dr. P. N. Evans, of Purdue University, before the Indiana Section of the American Chemical Society at their annual banquet in February:

THE SENTIMENTAL SCIENCE

The public has a notion that our science is a thing
That lodges in our brain-cells, but never makes us sing;
That sentiment originates and grows within the heart,
And never touches science, but inspires only art.
It seems to me, however, that the case is not so bad—
That science suffers only from the treatment that it's had
From dry-as-dust professors, who can't read between the lines,
But see in every rose bush but an aggregate of spines,
Who think they're scientific when they're burrowing in facts,
And find no time for thoughts they're so submerged in streams of acts;
And mercenary analysts, and office-holders too,
And engineers, and routine hacks—of course I don't mean you,
But unromantic people that refuse to see the thing
That's right beneath their very nose—it makes me mad, by Jing!

Now chemistry is crowded with the tenderness of things—
Attractions and affinities—of such it fairly sings;
Its full of symbolism, fire, and feuds of families,
And its most important element, you'll admit it surely is
What poets in their frenzy vaguely feel but scarcely know,
And express by shouting madly in their ecstasy—just O!
The perfume of the flowers, too, is scarce so pure and sweet
As our synthetic tribute is, with which it must compete;
And nature's rainbow's so outclassed it's very rarely seen,
It knows it could not stand the test of meeting aniline.

In asking of some Christian friends why steadfastly they cling
To ritual, and formula, and symbol for a thing,
They answer with sincerity, on higher things intent,
The symbols and the formulas are mainly sentiment.
If formulas and symbols sentimental are, you bet
Our chemistry's the most so of all things discovered yet!

And if in doubt you still remain, but turn your thought with me
To names that throng organic books, and then I think you'll see
That ester, yes, and ethylene, and ethyl, sit in state
With cis and anti, polymer, and even silicate.
And don't forget the sweetness that some carbohydrates show—
The sweetest thing on earth is but a chemical, you know,
The poets praise the ether, and they seem to think it's blue,
But ethyl and O-ethyl is what it is—that's true.
And then how like us humans do these little atoms act,
With their strong and weak affinities—it certainly's a fact
That the atom hates to live alone, but evidently yearns
To establish some co-partnership, but from like atom turns
To one of nature opposite, and when (I grieve to state
This kind of thing has happened very oft with us of late),
Though linked by well-established bonds, one of the pair perceives
A fairer atom hovering round, it simply ups and leaves,
And doubtless tells enquiring friends of cruelty and fights,
And how a woman-atom now must boldly claim her rights.
I hesitate to name a case, especially a pair
Of which I formed one member and the other was so fair,
But recently, I must admit, with Ethyl I had joined,
Along came Sodium Ethylate—my better half purloined;
Then Ethyl got—O Ethyl—and when I was bereft,
I said "I'll take the Sodium that faithless thing has left."
And Ethyl said I was the first who tried to break away,
Each blamed it on the other, but who can really say?
I didn't like that Sodium, and so I sought release,
And then existed all alone—a period of peace;
Then sought the aid of alcohol and phosphorus, and tried
And found again my Ethyl! Now we're Ethyl Iodide.

This frequent change of partners is less likely to occur
With solid folk who stay at home—he stays at home with her;
In liquid state the restlessness increases very fast,
And bonding grows less stable, and often does not last;
But worst of all the cases that you or I have met
Are the volatile, unstable, vap'rous things, the dancing set;
They whirl and dip, collide and bounce, and their velocity
Increases with the temperature—the results we may foresee;
From one step comes another, they hesitate, prove false,
Then pivot, use the grape-vine—O this hesitation waltz!

We well may view with deep concern those substances that act
Towards others in such different mood it's sometimes worse than tact;
Approached by some they seem to show their principles are such
That we may safely count on them with others, just as much,
To show the self-same nature, but we presently find out!
From noting their behavior we're entirely in doubt
What fundamental principles their conduct underlie—
We call them tautomeric, for of stronger terms we're shy;
We might have called them double-faced, or hypocrites, or crooks,
These tautomeric substances of scientific books.

To prove again that sentiment is not to art confined
Let me recount an incident that now I have in mind;
It shows, I think, how different a thing may seem to be,
How its effect is opposite, let's say, on A and B.
The inference is obvious, that sentiment's the cause,
So sentiment must be a part of laboratory laws.
I have some water in the lab., quite normal every way;
Its freezing point and boiling point were never known to stray;
Yet freezing point and boiling point have proved themselves at fault
And acted up quite contrary, because I added salts;
The boiling point jumped up at once, in no way was distressed,
Contrariwise, the freezing point was very much depressed.

I think I have convinced you that there's lots of sentiment
In chemistry (and H₂S is not the scent I meant);
If I've omitted anything I hope you will forgive—
You surely see the sentiment in that by which we live;
It permeates our science just as light pervades the sky,
And the chemistry of coal tar—it has taught us how to dye.

Dr. L. J. Henderson, Assistant Professor of Biological Chemistry, has been appointed the Professor from Harvard University for the second half of the year 1914-15 under the interchange agreement between Harvard University and the four Western colleges—Beloit, Grinnell, Knox and Colorado.

The Syracuse Section of the A. C. S. was addressed on April 24th by Mr. Carleton Ellis, of Montclair, N. J., on "Hydrogenation of Oils."

Mr. E. J. Sweetland, of the Sweetland Filter Press Company, New York City, sailed on May 19th for Europe, where he expects to spend some time on business.

Edward P. Hyde, Director of the Physical Laboratory of the National Electric Lamp Association, announces that hereafter the Laboratory will be known as the Nela Research Laboratory of the National Lamp Works of the General Electric Co., under which name its abstract-bulletin and other publications will appear.

Prof. George B. Frankforter, of the University of Minnesota, addressed the Detroit Chemists on "Some New Phases of Catalysis," April 24th.

Mr. Henry E. Jacoby announces the removal, on May 1st, of his offices to larger and more spacious quarters at 95 and 97 Liberty Street, New York City.

The Franklin Institute, Philadelphia, on May 20th, presented its Elliott Cresson Medals to Dr. Edgar Fahs Smith and Dr. Orville Wright. Addresses were made on "Scientists from the Keystone State," by Dr. Smith and on "Stability of Aeroplanes" by Dr. Wright.

Dr. Frank B. Kenrick, of the University of Toronto, gave an illustrated lecture on "Superheating and Supercooling" before the Rochester Section of the A. C. S., May 4th.

The Buffalo Foundry and Machine Company announce that they have withdrawn their New York representative and will handle all future inquiries in connection with their product directly through their Buffalo office.

The St. Louis Section of the A. C. S. was addressed by Prof. W. D. Harkins, of the University of Chicago, on "The Smelter Smoke Problem" at their meeting on May 11th.

Dr. Leo H. Baekeland, Charles Frederick Chandler Lecturer for 1914, has announced that the subject of his address, to be given at Columbia University on May 29th, will be "Some Aspects of Industrial Chemistry."

Smith, Emery & Co., Chemical Engineers and Chemists of San Francisco and Los Angeles, announce that Mr. Carlton R. Rose, for a number of years Superintendent of the U. S. Zinc Co., Pueblo, Col., subsidiary to The American Smelting & Refining Co., has recently resigned his position and become associated with them. Mr. Rose will be located in San Francisco.

Noel Deerr has resigned his position as director of the Experiment Station of the Hawaiian Sugar Planters Association to accept the appointment as Expert in Sugar Cane Agriculture and Sugar Manufacture to the Cuban Government.

Dr. Douglas McIntosh, Associate Professor in McGill University, Montreal, has been appointed Associate Professor of Chemistry and acting head of the Department in the newly established University of British Columbia.

GOVERNMENT PUBLICATIONS

By R. S. McBRIDE, Bureau of Standards, Washington

NOTICE—Publications for which price is indicated can be purchased from the Superintendent of Documents, Government Printing Office, Washington, D. C. Other publications can usually be supplied from the Bureau or Department from which they originate. Consular Reports are received by all large libraries and may be consulted there, or single numbers can be secured by application to the Bureau of Foreign and Domestic Commerce, Department of Commerce, Washington. The regular subscription rate for these Consular Reports mailed daily is \$2.50 per year, payable in advance, to the Superintendent of Documents.

TREASURY DEPARTMENT

Treasury Decisions. A weekly publication is issued giving the decisions of the board of general appraisers and the court of customs appeals in matters of duties on imports. Many of these decisions are of importance as showing the accepted classification of raw materials or manufactured articles of use in or in competition with American chemical industries. It is impracticable to review these decisions, and the original reports must therefore be consulted. The subscription price is \$1.75 per year, payable in advance to the Superintendent of Documents, Government Printing Office, Washington.

Value of Foreign Coins. Treasury Decisions, p. 6, No. 15, Vol. 26, dated April 9. Estimated by the Director of the Mint and adopted by the Treasury Department. Of industrial interest in foreign shipments, and especially in connection with imports.

AGRICULTURAL DEPARTMENT

The Effect of Heat on Hawaiian Soils. By W. P. KELLEY and Wm. McGEORGE. Bulletin 30, Hawaii Agricultural Experiment Station. 38 pp. 10c. The results of the various physical and chemical changes produced in soils by heat are reported (see THIS JOURNAL, 6, 223).

Rice Soils of Hawaii; their Fertilization and Management. By W. P. KELLEY. Bulletin 31, Hawaii Agricultural Experi-

ment Station. 23 pp. 5c. Of interest in soil investigations for other tropical or semitropical regions.

Service and Regulatory Announcements. These monthly circulars issued by certain Bureaus give information, instructions, and notices of a regulatory nature. They are issued by the following Bureaus: Animal Industry, Biological Survey, and Chemistry, and the Insecticide and Fungicide Board.

The Action of Manganese in Soils. By J. J. SKINNER and M. X. SULLIVAN, assisted by others. Department Bulletin 42, from Bureau of Soils. 32 pp. 5c. The results given in this bulletin throw considerable light on the effect of catalytic fertility in various soils. Manganese as a fertilizer is practically unknown and untried in this country, so that discussion of its action is necessarily theoretical; yet it is of great interest to those growers whose technical training induces them to experiment with new substances to increase or control crop production.

Laboratory and Field Assay of Arsenical Dipping Fluids. By R. M. CHAPIN. Department Bulletin 76, from Bureau of Animal Industry. 17 pp. 5c. A popular account of laboratory tests for actual arsenious oxid and for total arsenic, together with methods of field assay for dips used for tick eradication. Of special interest to officials and others concerned with the analysis and control of these preparations.

The Cost of Pasteurizing Milk and Cream. By J. T. BOWEN. Department Bulletin 85, from Bureau of Animal Industry. 12 pp. 5c. This bulletin deals with the question of costs from an engineering point of view; the bulletin is addressed to managers of creameries and designers of pasteurizing apparatus.

Alum in Foods. Department Bulletin 103. 7 pp. 5c. A contribution from the Referee Board of Consulting Scientific Experts, giving their findings as to the effects of the use of alum in food.

The Organic Nitrogen of Hawaiian Soils. By W. P. KELLEY and ALICE R. THOMPSON. Bulletin 33, Hawaii Agricultural Experiment Station. 22 pp. 5c.

The Agricultural Outlook. Farmers' Bulletin 590, from the Bureau of Statistics. 20 pp. Includes among other crop estimates, a report on the Louisiana sugar crop of 1913.

BUREAU OF MINES

The Prevention of Waste of Oil and Gas from Flowing Wells in California. By RALPH ARNOLD and V. R. GARFIAS. Technical Paper 42. 15 pp. A preliminary report on measures to prevent or remedy conditions leading to oil and gas waste, as well as measures for preventing the escape of artesian waters into oil or gas strata.

Weathering of the Pittsburg Coal Bed at the Experimental Mine near Bruceton, Pa. By H. C. PORTER and A. C. FIELDNER. Technical Paper 35. 35 pp. 5c. A large number of tests are reported on samples of weathered coal and coal from the same mine unaffected by weathering. The character of the work is indicated by the following general conclusions: "The weathering effects are manifested in a lower content of carbon and hydrogen and higher content of oxygen in the coal substance, a correspondingly lower calorific value of the coal, the presence of a larger proportion of combined water in the coal substance, and a much diminished power of consuming oxygen from the air and of liberating methane. A rusty appearance on the surface of lumps is not necessarily an indication of any depreciation in the fuel value of the coal."

The Inflammable Gases in Mine Air. By G. A. BURRELL and F. M. SEIBERT. Technical Paper 39. 24 pp. 5c. "The authors present this study as showing that normal mine-air samples obtained from many mines in various parts of this country contain only methane as the combustible gas." In addition to experimental results, there are given some historical data; and a description of the methods of analysis is also included. The apparatus used has been described previously by the Bureau of Mines.

Fires in Lake Superior Iron Mines. By EDWIN HIGGINS. Technical Paper 59. 34 pp. Principally of interest in mining work, but a considerable number of ore analyses and a discussion of the spontaneous ignition of pyritic material, such as black slate, are included.

Drilling Wells in Oklahoma by the Mud-laden Fluid Method. By A. G. HEGGEM and J. A. POLLARD. Technical Paper 68. 28 pp. Of interest in petroleum and natural-gas industries, as showing means of preventing waste during the well drilling.

Gases Found in Coal Mines. By G. A. BURRELL and F. M. SEIBERT. Miners' Circular 14. 24 pp. A popular treatment of this subject.

BUREAU OF FISHERIES

A New Method for the Determination of the Food Value of Proteins, with Application to *Cynoscion Regalis*. By G. F. WHITE and ADRIAN THOMAS. Document 784, from Bulletin 32. 5 pp. "Sørensen's method for the determination of amino acids was applied to a study of the tryptic proteolysis of *Cynoscion regalis*. The results were regular and in accord with those obtained by the nitrous acid method for the analysis for amino nitrogen. A practical method for the determination of the food value of proteins has therefore been developed."

Properties of Fish and Vegetable-Oil Mixtures. By G. F. WHITE and ADRIAN THOMAS. Document 785, from Bulletin 32. 14 pp. The viscosity, density, index of refraction, saponification number, acid number, and iodine number of the following oils were measured: China-wood, dogfish liver, soya-bean, linseed, and mixtures of these.

The Effect of Water-gas Tar on Oysters. By P. H. MITCHELL. Document 786, from Bulletin 32. 6 pp. Report of a laboratory investigation from which the following conclusions, among others, are drawn: "These experiments show no noticeable effects of water-gas tar on oysters in constantly-renewed sea water. This is true in spite of the fact that large amounts of tar mixed with stagnant sea water, or small amounts injected

into oysters which are kept in stagnant water, do cause serious or fatal effects." * * * * "In stagnant water the organism can not be effectively washed out, and effects involving a loss of sensitiveness in the mantle result. That consumption of the dissolved oxygen in the stagnant water by tar may have some effect on oysters is a possibility."

The Oxygen Requirements of Shellfish. By P. H. MITCHELL. Document 787, from Bulletin 32. 14 pp. A report on a laboratory investigation in which the "oxygen requirements" and the "resistance to lack of oxygen" were studied on oysters, clams and quahogs.

Fishery Products. Statistical Bulletins are issued, usually several each month, on the quantities and values of fishery products; for example, the following four subjects are covered in those issued during March, 1914: Quantities and values of products landed at Boston and Gloucester, Mass., in 1913, (1) classified by fishing ground, (2) classified by months, (3) for Jan., 1914, and (4) for Feb., 1914.

BUREAU OF STANDARDS

Bulletin No. 3 of Volume 10, contains the following 5 articles of chemical interest:

(1) **Critical Ranges of A₂ and A₃ of Pure Iron.** By G. K. BURGESS and J. J. CROWE. Scientific Paper 213. 56 pp. There is given a critical, historical summary of the experimental investigations of the position of A₂ and A₃, a brief mention of the theoretical aspects of the subject, and a report on tests made on several samples of pure iron which were studied by two methods over the range 500 to 1000°. A new method of testing has been devised since the earlier work was reported and tests are now reported on samples prepared recently. The methods of testing, as well as the results, are given in detail. Without exception, 130 curves show A₂ and A₃ as sharply defined and distinct critical ranges. Reduced to basis of zero rate of heating, the results are: A₂ = Ac₂ ± Ar. = 7680 ± 0.05; Ac₃ = 9090 ± 1; and Ar₃ = 898° ± 2.

(2) **Note on the Setting of a Mercury Surface to a Required Height.** By M. H. STILLMAN. Scientific Paper 214. 4 pp. The improvement is simply an artifice to enable detection of a smaller dimple in the Hg surface; it consists of a scale of alternate white and black lines each 0.5 mm. wide placed behind the pointer so that the images of the lines appear parallel when the pointer does not touch the mercury but are distorted when the smallest dimple is formed.

(3) **Micrometer Microscopes.** By A. W. GRAY. Scientific Paper 215. 16 pp. This article discusses some errors affecting micrometer microscopes and methods of determination of the corrections and their applications.

(4) **The Pentane Lamp as a Working Standard.** By E. C. CRITTENDEN and A. H. TAYLOR. Scientific Paper 216. 28 pp. A discussion of the standardization adjustment, and use of the pentane lamp as a photometric standard, including detailed operating directions and tables giving corrections to be applied to correct for the effect of atmospheric conditions. A large number of results obtained during the use of lamps at the Bureau of Standards for several years, are summarized in the article.

(5) **Comparison of the Silver and Iodine Voltmeters and the Determination of the Value of the Faraday.** By G. W. VINAL and S. J. BATES. Scientific Paper 218. 26 pp. A report on the comparison made of the silver voltameters of the form previously used by the Bureau of Standards and the iodine form used by Washburn and Bates, giving the following results:

Ratio of silver to iodine.....	0.85017
Electrochemical equivalent of iodine.....	1.31502
Value of the faraday (I = 126.92).....	96,515
Value of the faraday (Ag = 107.88).....	96,494
Value recommended for general use.....	96,500

Industrial Gas Calorimetry. By C. W. WADNER and E. F. MUELLER. Technologic Paper 36. (In press.) This paper reports an investigation of the factors which affect the accuracy

of the determination of heating value of gas, such as: Completeness of combustion, accuracy of the temperature measurements, the magnitude of the various heat losses from the calorimeter, the effect of varying the volume of the products of combustion, and the measurement of the quantities of gas and water. The summarized results are given of a critical study of 8 calorimeters of the flow type and one of the comparison type. There are also included the results of an experimental investigation of laboratory gas meters, showing the errors to which such meters are liable, the precautions to be observed, and the accuracy attainable in their use.

Variations in Results of Sieving with Standard Cement Sieves. By R. J. WIG and J. C. PEARSON. Technologic Paper 29. 16 pp. A report of tests made to determine the differences in standard sieves, the magnitude of the personal factor of the observer, the precision of results obtainable, etc.

Some Leadless Borosilicate Glazes Maturing at about 1100° C. By E. T. MONTGOMERY. Technologic Paper 31. 22 pp. Details are given of an attempt to prepare glazes equally desirable to substitute for those containing lead. Previous work, indicating the causes for the failure in this line, is confirmed.

Production of Temperature Uniformity in an Electric Furnace. By A. W. GRAY. Scientific Paper 219. 21 pp. A furnace giving the desired uniformity for work on the expansivity of metals is described.

Standard Density and Volumetric Tables. Circular 19, 4th edition, with supplement. The following tables (among numerous others of somewhat similar character) are given: Density of water; density of alcohol-water mixtures; temperature corrections for alcoholometers; density of methyl alcohol-water mixtures; densities of solutions of cane sugar and of sulfuric acid; temperature corrections for saccharometer readings; conversion tables for Baumé degrees, density, and specific gravity; density of air; apparent weight of water in air; temperature corrections for glass volumetric apparatus; and master scales for hydrometers.

Polarimetry. Circular 44. 140 pp. A very full report intended as a circular of information on the apparatus and methods of polarimetry, including a discussion of the theory involved, descriptions of the apparatus, its calibration, precautions to be observed in operation, and other methods. The circular is practically a text book of sugar methods as well as a laboratory handbook for general polarimetry. There are also included the U. S. Treasury Department "*Regulations Governing the Weighing, Taring, Sampling, Classification, and Polarization of Imported Sugars and Molasses.*"

The Testing of Materials. Circular 45. 90 pp. This circular is designed to give information on the subject of the testing done by the Bureau of Standards on each class of materials, the conditions under which such work is undertaken, and the limitation of such work due to the present status of technical knowledge. The information given is not only of interest in connection with the work done by the Bureau, since many of the topics are treated in such a way as to indicate the value and significance of tests, either chemical or physical, which can be made on certain sorts of materials.

BUREAU OF FOREIGN AND DOMESTIC COMMERCE

Imports and Exports. Several sets of publications are issued periodically by this Bureau, as follows:

- (1) Exports of domestic breadstuffs, cottonseed oil, food animals, meat, dairy products, cotton, and mineral oils. This is a monthly bulletin, the number for February being No. 8 of the series 1913-14.
- (2) Imported merchandise entered for consumption in the U. S., etc. This is a quarterly, subscription price 75c per year.
- (3) Total values of imports and exports of U. S. A monthly series of pamphlets on this subject is issued.
- (4) Monthly summary of commerce and finance of U. S.

This includes a section showing details of imports and exports, which section is sold in the form of advance sheets at 10c a single copy or \$1.00 per year.

Trade Directory of South America. Miscellaneous series publication No. 13 (1914). A report designed to promote American export trade with South America. Cloth, \$1.00.

Production and Use of Denatured Alcohol in Principal Countries. By CHAS. A. CRAMPTON. Special Agents Series 77. 32 pp. Paper 5c.

CONSULAR REPORTS, MARCH (Concluded)

The Potash shipments authorized for 1914 by the German Potash Syndicate amount to 635,300 tons of K_2O for domestic consumption and 531,300 tons of K_2O for foreign consumption. (P. 909.)

Public sales of Billiton Tin, in Batavia, in 1913, aggregated 4,938,800 lbs., at an average of \$0.4261 per lb. (P. 921.)

Russian Coal and Iron statistics show that the Donetz Basin provides 55.5 per cent of the coal consumed in Russia. The iron and steel industry shows increased activity. (P. 942.)

A large amount of Coconut Fiber (or Coir) is now wasted in Jamaica and the Philippines, but could be extracted and used for the manufacture of mats, etc., which are now made mostly from the British East Indian product. (P. 943.)

Coating of Coffees in Germany is regulated by law, especially the use of shellac, etc., which may contain arsenic. (P. 954.)

Wood Block Pavements in Berlin are made of blocks impregnated with zinc chloride or creosote, and laid in tar or asphalt, on a concrete base. (P. 956.)

The Commercial Museum of Trieste, maintained by the Chamber of Commerce, furnishes commercial and tariff information, investigates industries, and examines and analyzes imported goods. (P. 958.)

An exposition of machinery, apparatus, and supplies used in Wine Production, Brewing and Distilling, will be held in Budapest, May 23, to July 6, 1914. (P. 971.)

Spanish Garnets from Almeria Province are imported into the U. S. for abrasive purposes. (P. 974.)

A commercial review of Nova Scotia Ports includes statistics of the Coal and Iron Industries of that region. (P. 977.)

As a result of Rubber Cultivation in Choco, Columbia, 1,500,000 trees have been planted. Alluvial deposits of Gold and Platinum in Choco are being worked, mostly by hand. (P. 997.)

Petroleum production in Burma in 1912 amounted to 240,500,000 gallons. (P. 1007.)

The Sugar Crop of Austria-Hungary in 1912-13 showed a large increase in the production of beet sugar over previous years. (P. 1031.)

The Constantinople Gas Works has been purchased by a European firm, who will sell gas at \$1.43 per 1000 cu. ft. for lighting, \$1.14 for cooking, and \$1.00 for power. (P. 1051.)

Rubber shipments from the Amazon Valley in January, 1914, were lower by 2,877,799 lbs. than in January, 1913. (P. 1055.)

The United States supplied 65 per cent of the Coal imported into Ecuador in 1913. (P. 1063.)

All the Oil and Gasoline used in Prince Edward Island, Canada, is imported from the U. S. (P. 1070.)

The output of Vegetable Butter and Oils, principally from cocoanut oil, in Odessa, has increased rapidly in the last few years. (P. 1071.)

The Olive Oil production of Tuscany for 1913-14 was only one-third the normal output. (P. 1083.)

Petroleum in Persia is being exploited by three companies. (P. 1083.)

The cultivation of Sugar-cane has largely supplanted the Wine industry of Madeira. (P. 1095.)

The Governments of British Columbia and Ontario have passed regulations regarding the development of Radium deposits and have offered rewards of \$5,000 and \$25,000, respec-

tively, to persons discovering workable radium deposits in these provinces. (P. 1110.)

The statistics of the Russian sales to the U. S. for 1912 and 1913 include Chemicals, Fertilizers, Glue, Sheet Iron, Manganese Ore, Fusel Oil, Mineral Oil, Platinum, Rubber, Liquors, Tar, Turpentine, and Wood Pulp. (P. 1112.)

Petroleum is being prospected for in Palestine. (P. 1115.)

A new Glass Factory at Tobata, Japan, to cost \$1,000,000, is nearly completed and will make sheet and plate glass. (P. 1125.)

Platinum products manufactured in Switzerland may be officially stamped to indicate the platinum content (iridium being considered as platinum). (P. 1128.)

Exports of Tin from Hongkong to the U. S. in 1913 showed an increase while the exports to Europe showed a marked decrease. (P. 1148.)

The Antwerp Diamond trade flourished in 1913, Antwerp being now ahead of Amsterdam in this industry. The International Metric Carat of 200 milligrams has now been adopted by Belgium and the U. S., leaving England as the only important country in which it has not been adopted. (P. 1169.)

Cocoa Beans in Trinidad are usually coated with clay to preserve them and render them uniform in color. Efforts are now being made to prevent fraudulent excessive claying. (P. 1176.)

South African Collieries produced 4,500,000 tons of Coal in 1913, so that only 65,000 tons were imported from Great Britain. (P. 1177.)

The trade statistics for Ceylon for 1912 and 1913 include in the exports Coconut Fiber and Oil, Citronella Oil, Plumbago, and Rubber. Imports include Cement, Coal, Iron, Fertilizers, Kerosene, Sugar and Lead. (P. 1185.)

Vinegar and Pyroligneous acid are admitted free to the Netherlands when intended for use in Zinc Etching Establishments. (P. 1203.)

In connection with the reduction of the duty on Sugar, Syrups, etc., into Sweden, syrups are defined as containing not over 70 per cent sugar and more than 1.3 per cent ash. (P. 1205.)

CONSULAR REPORTS, APRIL

According to a bulletin of the Philippine Bureau of Agriculture the fiber or floss from the fruit of the kapok tree is being used extensively for filling mattresses, life-saving appliances, etc.; it can also be spun into yarn. (P. 10.)

Statistics of the zinc industry show that the world's production of zinc in 1913 was the largest ever recorded. Separate statistics are given for Australia, Austria Hungary, Germany, Italy, Russia and Spain. (P. 17.)

The beet sugar industry in Canada is conducted in 3 factories, the output of which in 1913 was 27,232,124 lbs. raw sugar. (P. 30.)

Statistics of the iron and steel production and the iron reserves, reported by the British Board of Trade, show the consumption of iron ore and the production of pig iron and steel for the principal countries, in 1910, 11 and 12. The deposits of ore now being worked will supply the world for less than two centuries, but known deposits as yet unworked, will greatly increase the supply. (P. 40.)

Pyrite deposits in Norway, containing 33 per cent sulfur, and 1 per cent copper are about to be worked on a large scale, the proposed annual output being 100,000 tons. (P. 43.)

The important mineral products of Nova Scotia are coal, gypsum, and limestone. (P. 43.)

The principal mineral products of China, are coal, iron, copper, tin and antimony. (P. 45.)

A trade review of Liverpool includes figures for the trade with U. S. in metals, drugs, oils, rubber, etc. (P. 49.)

The British Navy has just made large contracts for Scotch shale oil, for fuel. (P. 78.)

The foreign commerce of the Philippines in 1913 includes exports of copra, hemp, and sugar. (P. 92.)

The Scotch mineral oil companies produced 75,000,000 gallons of oil in 1913. Illuminating oil sold for \$0.14 per gal., and "motor spirit" for \$0.30 per gal. (P. 101.)

The principal minerals obtained in the Madrid district in Spain are coal and iron. (P. 134.)

The lime-fruit industry of Dominica in the Seeward Island includes the manufacture of calcium citrate and oil of limes. (P. 142.)

A great increase in the imports of fertilizers into Japan is noted. (P. 143.)

Both imports and exports of the Japanese foreign trade showed a marked increase in 1913. The imports included dyes, fibers, rubber, iron, fertilizers, paper pulp, and sugar. The exports included coal, copper, whale oil, menthol, and camphor. (P. 148.)

The production of maple syrup and maple sugar in the U. S. and Canada is rapidly decreasing. (P. 150.)

Methods of detinning in Germany include electrolytic methods, using both alkaline and acid electrolytes, and the chlorine method, the latter being most used. (P. 151.)

Cardiff, Wales, is the world's principal coal shipping port. Iron and steel, tin plate and galvanized steel are produced there in large amounts. Total shipments of coal from ports in Wales in 1913 were 40,000,000 tons. (P. 161.)

A new oil-bearing nut has been discovered in the Philippines belonging to the family Meliaceae. The dry nuts yield 45 per cent of oil which produces a good grade of soap. The production of coconut oil in the Philippines has increased very rapidly. (P. 172.)

The mineral products of Somerset, England, include coal, lead and zinc ores, fire clay, fuller's earth, and celestite (strontium sulfate). Imports of petroleum oils into Bristol are increasing. Large amounts of oil-seeds are imported for the manufacture of vegetable oils, including cottonseed, linseed, and soya bean. (P. 177.)

Fertilizers are to be admitted free of duty to all Belgian colonies after Jan., 1914. (P. 209.)

At a conference of commercial gas users in London, England, it was stated that municipal gas plants do not sell as large quantities of gas per consumer as do private companies. The increased use of gas stoves in London has materially improved the fog conditions. (P. 220.)

Italian olive oil is frequently adulterated with treated olive oil, *i. e.*, oil that has been deodorized or decolorized by chemical means and the presence of which is very difficult to detect. (P. 222.)

Exports from Manitoba, Canada, include flaxseed, wood pulp, and pyrite. (P. 232.)

Linseed and sunflower seed are used extensively in Russia for the production of vegetable oils. (P. 242.)

In the Spanish olive-oil industry the oil from the first two pressings is sold for edible oil, and that obtained by extraction with carbon bisulfide is used for industrial purposes. Inferior oils, when deodorized and decolorized are sometimes mixed with high-grade oil. To prevent adulteration of olive oil with other oils, the law requires the addition of wood pitch or coal tar to any cottonseed or rapeseed oil imported. (P. 246.)

In the manufacture of sawdust briquettes in British Columbia, the sawdust, chips, etc., are sometimes compressed into briquettes without the use of a binder, though usually coal dust and tar are added. (P. 249.)

Exports from Haiti to the U. S. include beeswax, cottonseed, lignum-vitae wood and gum, logwood, and fustic. (P. 266.)

Refrigeration in France is increasing in storage houses, cars and vessels. In cars, small machines, using CH₂Cl and operated by power from the car wheels, are frequently used. (P. 269.)

Exports from Belgian Kongo include copal, gum, copper, gold, palm nuts and oil, mabula panza (an oil nut) and rubber. (P. 273.)

The production of petroleum in the Dutch East Indies in 1913 was over 1,500,000 tons. (P. 285.)

Exports from Persia include drugs, gums, genna (a vegetable pigment), hides, and precious stones. (P. 298.)

The campaign for smoke abatement in England includes accurate measurement of atmospheric pollution in all the large cities, organization of classes for engineers and stokers, and the use of various forms of smoke-preventing apparatus. (P. 301.)

An increase is noted in the export from Ceylon of papain, a digestive extract from the *Carica papaya*. (P. 302.)

The export of peanuts from Hongkong to the U. S. has increased. (P. 311.)

The exports of Australia include coal, copper, chemicals, gold, lead, fertilizers, silver, soap, tallow, and tin. (P. 321.)

The exports of Trinidad include asphalt, balata gum, copra, petroleum, sugar, and molasses.

In the wine industry of Argentine, the grape residues are distilled for alcohol, and some tartaric acid and cream of tartar are recovered. (P. 335.)

Owing to adulteration of Chinese cassia (cinnamon) with dirt, sticks, etc., new standard contracts have been made. Large amounts of the spice and the oil are imported into the U. S. (P. 348.)

A company has been organized to develop nickel and iron deposits in Athabaska, Canada. (P. 349.)

Exports from Curacao (Dutch West Indies) include coal, dividivi, hides, salt, gold, phosphates, and aloes. (P. 358.)

The exports of New South Wales include coal, copper, gold, lead, leather, hides, cocoanut oil, silver, tallow, and tin. (P. 401.)

Exports from Manchuria include soya bean, cake and oil, coal, hemp, ginseng, and castor oil. (P. 417.)

The aluminum industry in Germany yielded dividends of 20 per cent in 1913. (P. 425.)

Importations of crude petroleum oil and kerosene into Hongkong from the U. S. showed a marked increase in 1913. (P. 436.)

Soya beans from Southern Manchuria contain 16-17 per cent of oil, and those from Northern Manchuria only 15 per cent of oil. (P. 439.)

Importations of chemical fertilizers into the Canary Islands are increasing. (P. 443.)

Imitation gold leaf, "blatt-metall," consisting of 100 parts copper and 15-20 parts zinc, is made and used extensively in Germany. Imitation silver leaf consists of 90 per cent tin and 10 per cent zinc. (P. 446.)

The first natural gas pipe line in Hungary was recently completed. (P. 451.)

Exports from Lombardy to U. S. in 1913 included drugs, dyeing and tanning extracts, glycerine, glue, and "oleostearin." (P. 455.)

A thorough investigation has shown that Southern India is well adapted to the cultivation of sisal hemp. (P. 461.)

The new Canadian tariff includes changes in the rates of duty on iron and steel, amyl alcohol, peanut and soya-bean oils, chloride of lime, and caustic soda. (P. 482.)

Exports from Paris to the U. S. include aluminum, glue, glycerine, marble, platinum, iridium, palladium, rubber, silver, and wines. (P. 519.)

Exports from Hongkong to the U. S. include aniseed oil, camphor, cassia, cassia oil, peanuts, peanut oil, sugar and tin. (P. 529.)

A prosperous industry in Turkestan is the cultivation of *Sevant* wormseed, from which the drug *santonin* is extracted. (P. 539.)

Exports from Almeria, Spain, include iron, zinc, lead, and copper ores and garnets. (P. 553.)

A plant has been erected in British Columbia to use the *Laurentia* process for pasteurizing milk, in which the milk, after having been heated to 155-165° F., is passed through a "homogenizer" in which the fat globules are broken up. (P. 557.)

Exports from Glasgow to the U. S. include aluminum, ammonium, and potassium salts, creosote oil, and iron. (P. 574.)

A Florentine inventor, Ulivi, claims to be able not only to explode powder, etc., by the so-called "F rays," but also to locate mineral deposits. (P. 587.)

BOOK REVIEWS

Allen's Commercial Organic Analysis. Vol. VIII. Edited by W. A. DAVIS AND SAMUEL S. SADTLER. 4th edition, 696 pages. Philadelphia: P. Blakiston's Son & Co., 1913. Price, \$5.00 net.

This is the last volume of the fourth edition of a work which has been long and favorably known, and which has become a practical necessity in laboratories where technical investigations in any field of organic chemistry are carried out. The present volume deals with the protein and related substances, or those products in which the proteins are the most characteristic or important elements.

The revision of a work of this character has long passed beyond the power of a single individual because no one person can be expected to be expert in so many specialties. In this volume, as in the others of the series, we find the labor has been divided among a number of men, in this case eleven, all of whom are well known authorities in different fields of technical chemical research. The general editorship remains with Messrs. Davis and Sadtler. The contributors to the various sections of Volume VIII are E. Frankland Armstrong, S. B. Schryver, L. L. Van Slyke, Henry Leffmann, Cecil Revis, E. Richards Bolton, W. D. Richardson, J. A. Gardner, G. A. Buckmaster, Jerome Alexander and W. P. Dreaper.

In the last edition of the work, the volume which covered the same general ground was issued about fifteen years ago. Since then there have been great advances in all lines of technical organic chemistry, and especially in our knowledge of the protein substances and related bodies which are taken up here. These changes have been so great as to call practically for the rewriting of the whole book. In general, the work has been well done and apparently the most recent literature has been considered in every field covered.

The longest single section is that by W. D. Richardson on Meat and Meat Products. This covers 205 pages, or nearly one-third of the whole book. Mr. Richardson, as head of the laboratory of one of the largest meat-producing plants in the world, has had opportunities such as are open to but few men for becoming acquainted with the whole field described and this is shown by the wealth of first-hand information evident in every part of the text, in the discussion of analytical methods as well as in the presentation of details of production.

The large field of Proteins of Milk, Milk and Milk Products has been covered by L. L. Van Slyke, H. Leffmann, and Cecil Revis and E. R. Bolton in chapters filling 147 pages. Each one of the authors has long been known as an original investigator in the subject of the chemistry of milk. There are few fields

in which methods of analysis have been so abundantly supplied as in that of milk investigation. In reading these sections one is impressed by the fact that little of importance is omitted. The discussion of the various products formed from milk is excellent. (Butter is considered in another volume.) It is interesting to note that the much advertised Sanatogen is described as a *mixture* of casein and sodium glycerophosphate, or possibly as a salt of casein so mixed.

Somewhat shorter sections have been contributed on Enzymes and on Proteins of Plants by E. F. Armstrong; on Proteins and Albuminoid Substances, and on the Digestion Products of the Proteins by S. B. Schryver; on Hemoglobin and Its Derivatives by J. A. Gardner and G. A. Buckmaster; on Albuminoids or Scleroproteins by Jerome Alexander; and on Fibroids by W. P. Dreaper. It will be noticed that there is some confusion in the use of the term albuminoid. In the discussion of the digestion products of proteins, the description of Pancreatin does not correspond very well to what is actually produced in this country. Under the methods used in testing enzymic activity the author describes the convenient Fuld method for pepsin but does not describe the corresponding method, using casein, by the same authority, for trypsin. There appears to be no mention of the value of fibrin in the estimation of marked tryptic activity. The routine method of the British Pharmacopoeia for the examination of pepsin is given, but not the somewhat similar but more rapid method of the U. S. Pharmacopoeia. As the latter work is an official standard in this country the oversight should have been caught by the editors and corrected.

Attention must be called to the excellent chapter on hemoglobin, embracing the methods of blood examination for scientific or clinical work.

J. H. LONG

The Electric Furnace, Its Construction, Operation, and Uses.

By ALFRED STANSFIELD, D.Sc. McGraw-Hill Company, New York. 415 pages. \$4.00 net. 1914.

The present volume, which is the second edition, is just double the size of the first edition of 1907. In these seven years there have been great advances in the subject of which this book treats, and Dr. Stansfield has met this change in an admirable manner, and it is only fair to say that it is by far the best book which has thus far appeared on the electric furnace.

The first chapters deal with the history, classification and efficiency of electric furnaces. Good illustrations are here given of the different types of arc and resistance furnaces, with a short description of each. Under the efficiency of electric furnaces, data are included as to cost of power, etc., and the actual method of calculating the efficiency is illustrated by taking a typical run of a Hèroult steel refining furnace.

Much valuable data are given under "Construction and Design," as to the properties of refractories, resistors, electrodes, etc., with a good summary of Hering's papers on heat losses in electric furnaces. In the operation of furnaces we find methods for the control and measurement of power, descriptions of transformers suitable for large operations, and diagrams of connection for furnaces using polyphase circuits.

Some interesting figures are given as to the power densities employed in various types of furnaces, and the extraordinary densities used by Moissan account for many of the remarkable results which he obtained.

The measurement of temperature and description of pyrometers is taken up very briefly, which is perhaps just as well as we already have the excellent book of Burgess which deals with this subject exclusively.

Under laboratory furnaces we find descriptions of Hutton's pressure, Arsem vacuum, Harker tube, and the Hansen arc furnaces among many others. The subject of iron and steel is given about 100 pages of the book, and the latest developments in

this important branch are well treated, all of the important furnaces being described.

The balance of the book is taken up with furnaces for the production of carbides, graphite, silicon, sodium, aluminium, nitric acid, zinc, alundum, etc., and well describes the improvements which have been brought about in their manufacture.

Calcium cyanamid is now produced in such enormous quantities that more should have been said about it, and it would have been well to have mentioned something as to the Serpek process or the fixation of nitrogen.

The book is unusually well illustrated and is an important and timely addition to the literature of the subject in which so many are now interested.

SAMUEL A. TUCKER

Principles and Practice of Agricultural Analysis. By HARVEY W. WILEY, A.M., Ph.D. Volume III, **Agricultural Products**, pp. i-xv and 1-846. The Chemical Publishing Co., Easton, Pa. Price, \$6.00.

The appearance of the second edition of this truly remarkable volume has been delayed, as the author states in the preface, by stress of other duties which made it impossible for the author to bring it up to date. It is divided into seven parts, the first discussing methods of sampling and drying, the second and third being devoted to sugars, starches and carbohydrates, the fourth to fats and oils, the fifth to nitrogenous bodies, the sixth to dairy products, and the seventh to miscellaneous products under some twelve subheads. It is essential to everyone concerned with those branches of agricultural chemistry with which it specifically deals, not only for the information included in the pages, but as a guide to original articles, which, in most cases, the expert will wish to consult directly. The student of Agricultural Chemistry who wishes to learn more than the mere laboratory technique must also go to the original articles, for the theoretical basis of the formulas in many cases and the processes sometimes are discussed quite insufficiently. The expert who has specialized in particular lines will occasionally be disappointed. For instance, in describing the calcium saccharates (p. 274), there is no mention of Patten's research work [*Jour. Phys. Chem.*, 15, 67 (1911)]. The volume does not, however, pretend to quote the literature exhaustively, and it is amazing that so much has been included, especially of the American literature. As is natural, those methods which have been tested in the Bureau of Chemistry of the U. S. Department of Agriculture receive special consideration. This fact is, moreover, a very valuable feature of the volume. The wealth of formulas and tables make the book of extraordinary value for quick reference. A charming, as well as valuable, feature is the occasional introduction of historical matter, as in the discussion of Fehling's solution (p. 174, *et seq.*). There is a good index, and the general makeup of the volume is satisfying.

FRANK K. CAMERON

Metallography. By CECIL H. DESCH, D.Sc. (Lond.), Ph.D. (Wurz.), Graham Young Lecturer in Metallurgical Chemistry in the University of Glasgow. \$3.00 net. Second edition. Longmans, Green & Co.

The second edition of this book incorporates some of the most important results of recent investigations and brings up to date the references to publications. There is no change in treatment of the subject.

The book is divided into eighteen chapters, which deal with the following headings: Introduction; The Diagram of Thermal Equilibrium; Solid Solutions or Mixed Crystals; Ternary and More Complex Systems; Metals which are only Partially Miscible in the Liquid State; Practical Pyrometry and Thermal Analysis; The Preparation of Micro-Sections; The Microscopical Examinations of Prepared Sections; the Crystallization of Metals and Alloys; Undercooling and the Metastable State; Diffusion in the Solid State; The Physical Properties of Alloys; Density—Thermal Expansibility—Hardness—Electrical Conductivity—

Thermo-Electric Power—Magnetic Properties; Electromotive Force and Corrosion; The Construction of the Equilibrium Diagram; The Molecular Condition of Metals in Alloys and the Nature of Inter-Metallic Compounds; The Plastic Deformation of Metals and Alloys; The Metallography of Iron and Steel; The Metallography of Industrial Alloys.

This book is one of the text books of Physical Chemistry edited by Sir William Ramsay, and naturally takes up the study of metallography from that point of view. It sets forth, very clearly, our present knowledge of this subject which has been defined as the study of the internal structure of metals and alloys and its relation to their composition and to their physical and mechanical properties. The author, in a very clear manner, works out the various diagrams of thermal equilibrium and then discusses the ternary systems. The section on practical pyrometry and thermal analysis gives us in some forty chapters a clear and concise account of the methods in use and instruments for autographic registration of curves. The chapter on the crystallization of metals and alloys is well written and well illustrated by micrographs. From the chapter on the physical properties of alloys we get a very clear idea of what has been done and what remains to be done in this branch of the subject. The section dealing with the metallography of iron and steel gives us a brief summary of our present knowledge of this subject. The system iron-carbon presents considerable difficulties: the most widely accepted hypothesis is that we have two systems, Austenite-Graphite or the stable, and Austenite-Cementite or the metastable. Goerens, however, is of the opinion that graphite is always the product of decomposition of cementite; Upton's diagram avoids many of the difficulties of the double diagram. Carpenter and Keeling's thermal observations show arrests at 800 and 600°, respectively. Under conditions of equilibrium the two solid phases separating from the liquid are Austenite and graphite; at 1095° they react to form Fe_3C , at 800° this decomposes into Fe_3C , at 615° a further decomposition occurs and Fe_3C changes into Fe_2C and alpha iron. These three carbides all resemble cementite, and means of distinguishing them have yet to be found. The book ends with an appendix consisting of tables of systems of which the equilibrium diagrams have been published. The book is to be recommended, first, because it covers the ground very thoroughly, and secondly, because the author speaks about what he is evidently thoroughly familiar with from the practical standpoint. It is not merely a compilation of the work of others. Although the field is a wide one, Dr. Desch has covered it well and the book will continue to be of great assistance to those starting out in metallography, not only for what it contains, but also as a guide to further work on the subject.

WILLIAM CAMPBELL

Sugar Analysis: For Cane-Sugar and Beet-Sugar Houses, Refineries and Experimental Stations and as a Handbook of Instruction in Schools of Chemical Technology. By FERDINAND G. WIECHMANN, Ph.D., Third Edition, 8vo. 303 pages. 7 Figures. Cloth, \$3.00 net.

In the present volume, Dr. Wiechmann—the dean of American sugar chemists—has given us the best results of his ripe and many-sided experience as teacher, refinery expert, consulting chemist and secretary of the International Commission for Uniform Methods of Sugar Analysis. Certainly no one is better qualified than he to discuss fully and adequately the many difficult phases which underlie the treatment of this subject.

As stated by the author in his preface the aim in preparing the present edition has been "to cast his material in a form in which it would prove most readily available in the several branches of the sugar industry." The purpose thus expressed has been most admirably fulfilled.

The scope of the new volume is briefly indicated by the following synopsis of chapters: I, Properties of Sucrose; II, Instruments Used in Sugar Laboratories (Refractometers, Balances,

Hydrometers, Colorimeters, etc.); III, Polariscope and Accessories; IV, Sucrose Determination by Optical Analysis; V, Sucrose Determination by Chemical Analysis; VI, Sucrose Determination by Optical and Chemical Analysis; VII, Constituents of Sugar other than Sucrose (Reducing Sugars, Water, Ash, Suspended Impurities, Organic Non-Sugar, Nitrogenous Substances, Gums, Acids, Iron Oxide, etc.); VIII, Materials Used in the Sugar Industry (Bone-black, Phosphoric Acid, Limestone, Coal, Flue Gases, Sulfur, Oils, Waters, etc.); IX, Analytical Control in Cane-Sugar Manufacture (Sugar Cane, Juices, Syrup, Bagasse, Press Cake, Sugar, Molasses, etc.); X, Analytical Control in Beet Sugar Manufacture (Beets, Diffusion Juices and Waters, Thick Juices, Fill Mass, Raw Sugars, Molasses, Cattle Food, etc.); XI, Analytical Control in Refineries; XII, Résumé of the Work of the International Commission for Uniform Methods of Sugar Analysis.

All sugar chemists who have occasion to consult the Proceedings of the International Commission will thank Dr. Wiechmann for the résumé in his 12th chapter which contains information that was formerly widely scattered and often difficultly accessible.

A well-selected list of sugar tables and an index make up the final 70 pages of the volume.

The typography and general appearance of the new book are excellent and leave nothing to be desired.

The many friends of the previous editions of Dr. Wiechmann's "Sugar Analysis" will welcome the new edition of this standard work. It is a most useful guide not only to the student, but also to the sugar factory chemist and commercial analyst.

C. A. BROWNE

Industrial Poisoning from Fumes, Gases and Poisons of Manufacturing Processes. BY DR. J. RAMBOUSEK. Translated and edited by THOMAS H. LEGGE. New York: Longmans, Green and Co., 1913. xiv + 360 pages, with illustrations. Price, \$3.50, net.

Efficiency is the dominant idea in modern industry. Justice Hughes once said that we do not wish to see productive energy sapped by excessive toil or by labor under improper conditions; for, to quote Sir John Simon, "the canker of industrial diseases gnaws at the very root of our national strength." It is, therefore, only to be expected that various governments and scientific organizations are taking a growing interest in the diseases of occupation. Several countries have, for some time, been active in this field; there is now a Permanent International Committee for the Study of Industrial Diseases; and, in this country, the New York State Department of Labor, the American Museum of Safety, the Health-Education League of Boston, the Federal Bureau of Mines and the Museum of Natural History of New York have recently shown interest of purpose. The translation of Rambousek's treatise on the industries and processes attended with risk of poisoning, the incidence of such poisoning, the pathology and treatment of industrial poisoning, and the preventive measures against industrial poisoning, supplies English readers with a book wherein the whole subject of industrial poisoning is well discussed.

In his preface Rambousek states that "the book is intended for all who are, or are obliged to be, or ought to be, interested in industrial poisoning;" the translator and editor observes that no words could better describe the scope of the book, and it seems to the reviewer that the general subject matter is presented in as comprehensive and systematic a manner as is possible within the compass of a single volume of this size. Several omissions occur in the text: no reference is made to poisoning produced by vanadium ore and oxide, and the effects of osmium oxide vapor are not noted. On the whole, however, the book is very complete.

American investigators have much to learn from European reports on industrial diseases and factory hygiene, and it will be surprising to many to find how thorough are the regulations

for dangerous trades abroad. A wealth of references to the work of foreign writers occupies sixteen pages (unindexed, but chronologically classified under subjects), while the general index of the book is well done.

W. A. HAMOR

The Nickel Industry: With Special Reference to the Sudbury Region, Ontario. By A. P. COLEMAN, PH.D. Published by the Department of Mines, Mines Branch, Ontario, Canada. *Bulletin* No. 170. Size, 6½ by 10 inches. 189 pages of text, 8 pages of index, and 9 pages of the publications of the Mines Branch of Canada Department of Mines.

The author, in his letter of transmittal to the Director of the Mines Branch, Department of Mines, Ontario, Canada, states in part: "I have the honor to transmit to you a Monograph on the Nickel Industry, with special reference to the Sudbury region, with a general map, and special maps of the more important mines, the whole representing the advance made in our knowledge of the region due to three summers' work in the field. In addition to descriptions of all the known nickel ore deposits in Ontario, there are accounts of methods of mining and smelting the ores, and of the chief nickel regions of other countries."

The methods of mining and of smelting of the ores of Ontario are described, and also descriptions of proposed methods for the treatment of copper-nickel ores and of the separation of copper from nickel and from their alloys. The divisions are not handled with the idea of the importance of any one of the ranges, but in such a way that a connected treatment of the subject as a whole is given. This bulletin supplements Report No. 873 of the Geological Survey of Canada by A. E. Barlow, M.A., D.Sc., on "The Origin, Geological Relations and Composition of the Nickel and Copper Deposits of the Sudbury Mining District," which was published in 1904.

The first 19 pages are devoted to outlining the geology of the

Sudbury district and to an historical sketch of the leading mines. The next 95 pages contain a description of the ores, their mineral constituents, and the individual deposits of the more important mines. Mention is made as to the origin of the ore bodies, and the genesis of the minerals of which the typical ores are composed.

A section (pp. 116 to 125) is devoted to the better known nickel-producing districts of the United States, Europe, New Caledonia, and the Cape Colonies, for the purpose of comparison with the Canadian ranges.

Methods of prospecting and mining (pages 125 to 132) are given, and are accompanied by illustrations. The mechanical and metallurgical treatment of the nickel-copper ores is gone into thoroughly (pp. 132 to 167), starting with the ore from the time that it is hoisted from the mine and finishing with the purified copper and nickel, and the purified Monel metal. This part of the monograph is extensively illustrated by plate photographs and drawings of the processes used.

The appendix (pp. 171 to 189) contains copies of patents of methods of separating copper and nickel from ores and from alloys.

The monograph is profusely illustrated by 62 plate photographs, 14 drawings of the principal mine sections and of metallurgical flow-sheets and apparatus, and 8 geological maps of the principal ranges of the Sudbury district.

This monograph is a valuable addition to the literature on the mining of nickel ores and the metallurgy of nickel, and will be welcomed by the mining man, the metallurgist and the metallurgical chemist, for there is so little reliable literature on the subject. This, like all of the publications of the Canadian Government, is well gotten out, and one holds the assurance that the information is reliable.

E. F. KERN

NEW PUBLICATIONS

By D. D. BEROLZHEIMER, Librarian The Chemists' Club, New York

Alloys and Their Industrial Applications. By EDWARD F. LAW. 2nd Ed. 8vo. Price, \$3.25. Chas. Griffin & Co., London.

Arsenic, Antimony and Tin, Methods of Determination of, and Their Separation from the Other Elements. By H. WOELBLING. I. 8vo. Price, \$3.75. Ferdinand Enke, Stuttgart. (German.)

Carbides and Silicides. By OTTO HOENIGSCHMID. 8vo. 265 pp. Price, \$4.75. Wilhelm Knapp, Halle. (German.)

Carbohydrates, Short Handbook of. By B. TOLLENS. 3rd Ed. 8vo. 816 pp. Price, \$5.25. J. A. Barth, Leipzig. (German.)

Chemistry in America. By EDGAR F. SMITH. 8vo. Price, \$2.50. D. Appleton & Co., New York.

Chemistry, Some Fundamental Problems in Old and New. By E. A. LETTS. 8vo. 227 pp. Price, \$2.00. Constable & Co., London.

Coal Tar Distillation. By ARTHUR R. WARNES. 8vo. 185 pp. Price, \$2.50. D. Van Nostrand Co., New York.

Conductivity, Electrical, and Ionization Constants of Organic Compounds. By HEYWARD SCUDDER. 8vo. 575 pp. Price, \$3.00. D. Van Nostrand Co., New York.

Copper Handbook, Vol. XI. By WALTER HARVEY WEED. 8vo. Price, \$5.00. Walter Harvey Weed, Houghton, Mich.

Dyestuff Tables. By G. SCHULTZ. 5th Ed. Lex. 8vo. 432 pp. Price, \$10.00. Weidmann, Berlin. (German.)

Fuel, Solid, Liquid and Gaseous. By J. S. S. BRAME. 8vo. 388 pp. Price, \$3.25. Edward Arnold, London.

Glycerin, Manufacture of. By FELD AND VORSTMANN. 2nd Ed. 12mo. Price, \$1.00. H. Desforges, Paris. (French.)

Metals, the Synthetic Use of, in Organic Chemistry. By ARTHUR J. HALE. 8vo. 169 pp. Price, \$1.25. J. & A. Churchill, London.

Metals, Precious, Progress in Leaching during the last Decades. By ROLF BORCHERS. 8vo. 400 pp. Price, \$2.00. Wilhelm Knapp, Halle. (German.)

New Journal: Internationale Zeitschrift fuer physikalisch-chemische Biologie. By J. TRAUBE. J. Wilhelm Engelmann, Leipzig.

Photomicrography, Handbook of. By H. LLOYD HIND AND W. BROUGH RANGLES. 8vo. 292 pp. Price, \$2.00. Geo. Rutledge & Sons, London.

Rubber: Its Sources, Cultivation and Preparation. By HAROLD BROWN. 8vo. Price, \$1.50. John Murray, London.

Silicates in Chemistry and Commerce. By W. ASCH AND D. ASCH. 8vo. 476 pp. Price, \$6.00. D. Van Nostrand Co., New York. (Translation in English.)

Soaps, Medicinal. By WALTER SCHRAUTH. 8vo. 170 pp. Price, \$1.75. Julius Springer, Berlin. (German.)

Steels, Steel-Making Alloys and Graphite, Rapid Methods for the Chemical Analysis of Special. By CHARLES MORRIS JOHNSON. 3rd Ed. 8vo. 438 pp. Price, \$3.00. John Wiley & Sons, New York.

Sugar Industry, Chemistry of the. By OSKAR WOHRZYK. 8vo. 676 pp. Price, \$5.00. Julius Springer, Berlin. (German.)

Tanning Chemists and Leather Manufacturers, Pocketbook for. By H. R. PROCTOR, EDMUND STIASNY AND HAROLD BRUMWELL. 8vo. 250 pp. Price, \$1.25. Th. Steinkopff, Dresden. (German.)

Textile: Waterproofing of Fabrics. By S. MIERZINSKI. 2nd Ed. Cr. 8vo. 132 pp. Price, \$1.25. Scott, Greenwood & Co., London.

Triphenylmethyl. By JULIUS SCHMIDLIN. Lex. 8vo. Price, \$2.20. Ferdinand Enke, Stuttgart. (German.)

Water, Microscopy of Drinking. By GEORGE C. WHIPPLE. 3rd Ed. 8vo. 409 pp. Price, \$4.00. John Wiley & Sons, New York.

RECENT JOURNAL ARTICLES

Acetyl-cellulose: Production, Characteristics and Utilization. By WALTER VIEWEG. *Kunststoffe*, Vol. 4, 1914, No. 8, pp. 148-152.

Acetyl-nitrocellulose and its Use for Celluloid. By H. WISHIDA. *Kunststoffe*, Vol. 4, 1914, No. 8, pp. 141-142.

Ammonia from Gas, Recovery of, by the Direct and Semi-Direct Sulfate Processes. By M. DESMARETS. *Revue générale de chimie pure et appliquée*, Vol. 17, 1914, No. 7, pp. 115-122.

Analysis, Micro-Elementary, of Organic Substances by Fritz Pregl's Method. By J. V. DUBSKY. *Chemiker Zeitung*, Vol. 38, 1914, Nos. 47 and 48, pp. 505-506, and 510-511.

Iron, Electrolytic Reduction of, for Permanganate Titration. By H. C. ALLEN. *Journal of the American Chemical Society*, Vol. 36, 1914, No. 5, pp. 937-949.

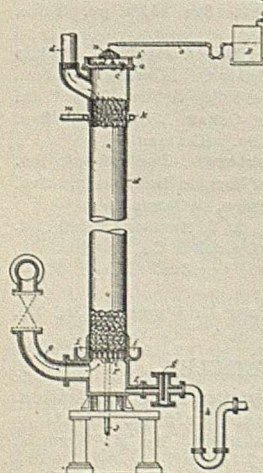
Arsenic and Antimony, Organic Derivatives of. By GILBERT T.

- MORGAN. *Pharmaceutical Journal*, Vol. 92, 1914, No. 2635, pp. 537-540.
- Arsenic, Quantitative Separation of, from Metals by Use of Hypophosphoric Acid.** By L. BRANDY. *Chemiker Zeitung*, Vol. 38, 1914, Nos. 43 and 44, pp. 461-463 and 474.
- Bronzes, Copper-Tin, Electrolytic Deposition of.** By R. KREMMANN, C. TH. SUCHY, J. LORBER AND R. MAAS. *Monatshefte fuer Chemie*, Vol. 35, 1914, No. 3, pp. 219-288.
- Coal, and the Chemistry of Its Carbonization.** By JOHN HARGER. *Journal of the Society of Chemical Industry*, Vol. 33, 1914, No. 8, pp. 389-392.
- Colloids.** By WOLFGANG OSTWALD. *Chemical Engineer*, Vol. 19, 1914, No. 4, pp. 133-137.
- Colloids in Effluents, Simple Method for the Determination of.** By R. MARC AND K. SACK. *Kolloidchemische Beihefte*, Vol. 5, 1914, No. 8-10, pp. 375-410.
- Colorimeters, Two New.** By PAUL VERBEEK. *Zeitschrift fuer angewandte Chemie*, Vol. 27/I, 1914, No. 28, pp. 203-208.
- Concentration by Refrigeration of Liquid Vegetable Extracts.** By H. PARENTY. *Comptes rendus*, Vol. 158, 1914, No. 13, pp. 921.
- Drying Bagasse, Marcs, Sawdust, Peat, Etc.** By A. HUILLARD. *Louisiana Planter*, Vol. 52, 1914, No. 16, pp. 251-252.
- Explosives: Blasting Gelatin, Some Notes and Theories.** By W. A. HARGREAVES. *Journal of the Society of Chemical Industry*, Vol. 33, 1914, No. 7, pp. 337-340.
- Fats, Unsaponifiable Constituents of Natural and Hardened.** By J. MARCUSON AND G. MEYERHEIM. *Zeitschrift fuer angewandte Chemie*, Vol. 27/I, 1914, No. 28, pp. 201-203.
- Flame Reactions.** By W. D. BANCROFT AND H. B. WEISER. *Journal of Physical Chemistry*, Vol. 18, 1914, No. 4, pp. 281-336.
- Gas, Coke-Oven and Blast-Furnace, Notes on the Utilization of, for Power Purposes.** By HEINRICH J. FREYN. *Bulletin of the American Institute of Mining Engineers*, 1914, No. 88, pp. 665-694.
- Hydrogen Peroxid, Synthesis of 100 Per Cent, by Silent Electric Discharges.** By PAUL MAX WOLF. *Zeitschrift fuer Elektrochemie*, Vol. 20, 1914, No. 7, pp. 204-219.
- Hydrogen, the Commercial Uses of.** By A. CROSSLEY. *Chemical Trade Journal*, Vol. 54, 1914, No. 1406, p. 497.
- Iron Corrosion by Dissolved Oxygen.** By J. W. COBB AND G. DOUGILL. *Journal of the Society of Chemical Industry*, Vol. 33, 1914, No. 8, pp. 403-407.
- Iron and Steel, Influence of Copper on the Corrosion of.** By E. R. HAMILTON. *American Gas Light Journal*, Vol. 100, 1914, No. 16, pp. 246-247 and 250-252.
- Nickel, Electrodeposition of.** By C. W. BENNET, H. C. KENNEY AND R. P. DUGLISS. *Journal of Physical Chemistry*, Vol. 18, 1914, No. 5, pp. 373-384.
- Nitro-bodies, Explosions of, and their Prevention.** By C. H. BORRMANN. *Chemiker Zeitung*, Vol. 38, 1914, No. 50, pp. 537-538.
- Paraffin, Liquid.** By J. WICLIFFE PECK. *Pharmaceutical Journal*, Vol. 92, 1914, No. 2634, pp. 508-509.
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- Peat, By-products from.** By F. M. PERKIN. *Chemical Trade Journal*, Vol. 54, 1914, No. 1403, pp. 417.
- Petroleum and its Derivatives.** By DAVID T. DAY. *Oil, Paint and Drug Reporter*, Vol. 85, 1914, No. 17, pp. 18-19 and 33.
- Phosphorus, Red, Formation of, by the Oxidation of Phosphorus Vapor.** By KOHLSCHUETTER AND FRUMKIN. *Berichte der deutschen chemischen Gesellschaft*, Vol. 47, 1914, No. 6, pp. 1088-1100.
- Picric Acid in Volumetric Analysis.** By A. SANDER. *Zeitschrift fuer angewandte Chemie*, Vol. 27/I, 1914, No. 26, pp. 192-194.
- Plating Baths, Use of Potassium Cyanid for.** By C. F. BURGESS AND L. F. RICHARDSON. *Zeitschrift fuer angewandte Chemie*, Vol. 27/I, 1914, No. 30, pp. 211-212.
- Refuse Destructor, the Evolution of the.** By E. KOHLMANN. *Journal of the Society of Chemical Industry*, Vol. 33, 1914, No. 8, pp. 383-389.
- Shellac, Bleached: Its Production, Examination and Utilization.** By HANS WOLFF. *Kunststoffe*, Vol. 4, 1914, No. 8, pp. 143-145.
- Sodium from Caustic Soda by Electrolysis.** By BERNHARD NEUMANN. *Zeitschrift fuer angewandte Chemie*, Vol. 27/I, 1914, No. 26, pp. 195-200.
- Steel, Determination of Cobalt in.** By P. SLAWIK. *Chemiker Zeitung*, Vol. 38, 1914, No. 48, pp. 514-515.
- Steel, Manganese, Relation of Physical Properties to Microstructure and Critical Ranges, with Especial Reference to.** By W. S. POTTER. *Bulletin of the American Institute of Mining Engineers*, 1914, No. 88, pp. 601-634.
- Tannin, Colloidchemical Studies on.** By M. NAVASSART. *Kolloidchemische Beihefte*, Vol. 5, 1914, No. 8-10, pp. 299-374.
- Water Supply, The Croton: Its Quality and Purification.** By GEORGE W. FULLER. *Journal of the American Water Works Association*, Vol. 1, 1914, No. 1, pp. 135-187.
- Water Filtration Practice, Present Day.** By GEORGE A. JOHNSON. *Journal of the American Water Works Association*, Vol. 1, 1914, No. 1, pp. 31-80.
- Water-Sampling Bottle, Improved.** By RICHARD H. ENRICH. *Engineering Record*, Vol. 69, 1914, No. 19, pp. 543-544.
- Wood Pulp, Sulfite, Manufacture of.** By G. B. STEFFANSON. *Pulp and Paper Magazine of Canada*, Vol. 12, 1914, No. 9, pp. 259-264.
- Wood Pulp in Textil Manufacturing, The Utilization of.** By H. A. CARTER. *Canadian Textile Journal*, Vol. 31, 1914, No. 4, pp. 100-103.

RECENT INVENTIONS

By C. L. PARKER, Solicitor of Chemical Patents, McGill Building, Washington, D. C.

Manufacture of Fuming Sulfuric Acid, or Oleum. Briggs and

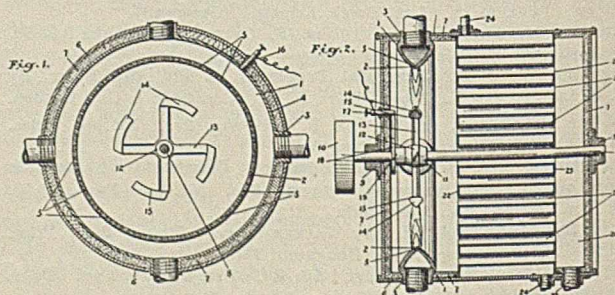


Merriman, Dec. 23, 1913. U. S. Pat. 1,082,301. In the use of this apparatus sulfuric acid having a strength of from 98 to 99.5 per cent flows from tank B over distributor *n* and through small openings *c* from which it falls in fine streamlets upon the packing of quartz in the tower A.

Air containing from 4 to 8 per cent of sulfuric anhydrid enters the tower through pipe *e* and passes upward in contact with the thin films of sulfuric acid and is absorbed thereby, the resulting fuming sulfuric acid passing out of the tower through pipe *f*. The temperature in the tower is maintained at a desired point by spraying the outside of the tower with water from the ring nozzle *k*.

Oxids of Nitrogen. J. S. Island, Dec. 30, 1913. U. S. Pat. 1,082,520. In this apparatus the tubular ring 1 forms the positive electrode and the rotor 11 the negative electrode of means to produce an electric arc flame. Compressed air is

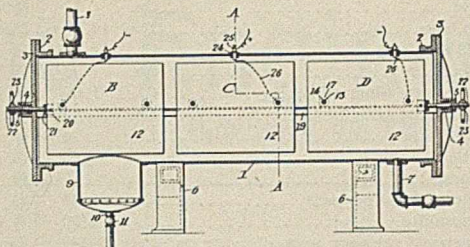
supplied to the tubular ring 1 and is forced at high velocity through minute openings 5 into the inner zone of the electric flame. The resulting gases are driven laterally by the angularly arranged blades 15 into contact with the surfaces of the cooling



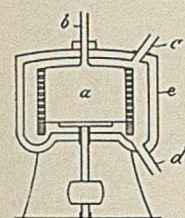
chamber 20 so that such gases are withdrawn from the middle and outer zones of the electric flame and cooled before there is any material dissociation of the nitric oxids formed in the inner zone.

Electrolytic Apparatus for the Clarification of Sugar Solutions. H. McCubbin, Jan. 13, 1914. U. S. Pat. 1,084,556. The apparatus embodies means for treating sugar solutions electrically while they are passed through a container within which are electrodes exposing large surfaces for contact with the solutions during their passage through the container. Means are pro-

vided for adjusting the relative positions of the electrodes with respect to each other and the ends of the container are made

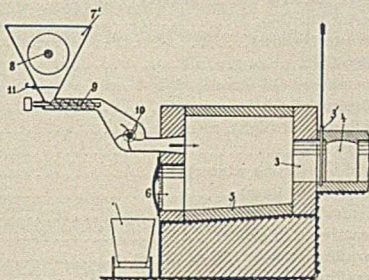


removable to permit the withdrawal of the electrodes for cleaning.

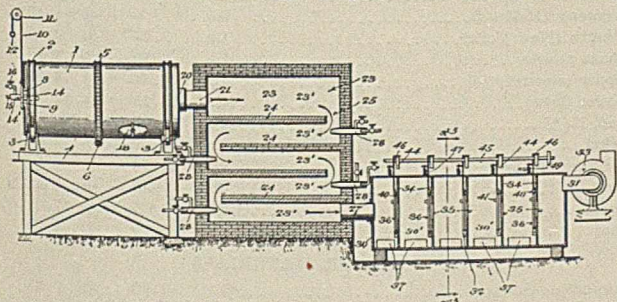


Solid Fatty Substances from Oil. J. Schlinck, Dec. 30, 1913. U. S. Pat. 1,082,707. Oils are hydrogenated by passing them over a catalyst secured within openings in the wall of the centrifugal drum *a*, in an atmosphere of hydrogen.

Making Charcoal. L. Félizat, Feb. 17, 1914. U. S. Pat. 1,087,486. Charcoal is produced from ground ligneous material, such as almond shells, peach or apricot kernels, olive husks, sawdust and the like by showering the waste material into a furnace heated to redness while at the same time introducing air to effect the complete combustion of the volatile products which are dissociated from the waste and which ignite on coming in contact with the red hot walls of the chamber. The residue remains on the bottom of the chamber in the form of incandescent charcoal. The hot gases are conducted under boilers to utilize the calorific power of the volatile products.



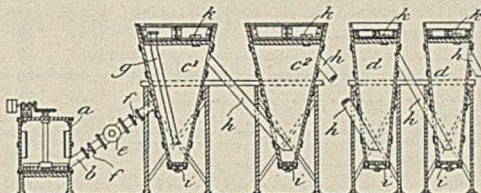
Apparatus for Making White Lead. R. A. Stewart, Feb. 24, 1914. U. S. Pat. 1,088,041. This apparatus is designed to produce a white commercial pigment from lead ores in one operation. The lead ore, such for example as sulfid ore, is first heated in the rotary drum 1. The products escaping into the supplementary roasting chamber 23 are gray or bluish by reason of



the presence of unoxidized lead particles, but in passing through chamber 23, which is heated by burners 28, it is converted into white lead so that the product passing out of the chamber 23 is a white commercial pigment which is recovered in the filter chamber 30. The products from the rotary drum 1 are drawn through the supplementary heating chamber and the filter chamber by means of a suction fan, 33.

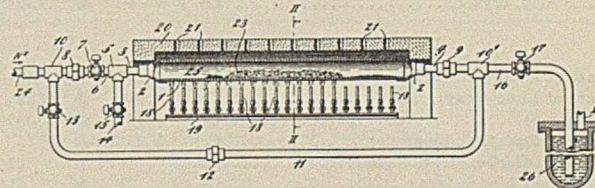
Manufacture of White Lead. F. R. Sharpe, Feb. 24, 1914. U. S. Pat. 1,088,460. The process consists in simultaneously

and continuously carbonating lead oxid, and separating with the aid of the carbonating agent the basic carbonate produced by a process resembling elutriation, *i. e.*, the particles to be separated from heavier or less bulky particles are carried to the surface of a liquid in which the particles are suspended and are removed by the overflow of the liquid. This is done by using a series of



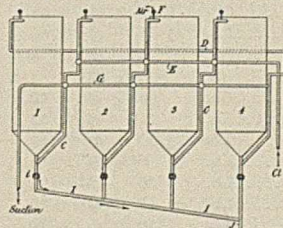
closed vessels communicating with each other in such a manner that the carbonating gas or gases pass through the liquid in each vessel, and at the same time the basic lead carbonate and the lead oxid undergoing carbonation are carried to the surface of the liquid in each vessel by the gas or gases passing through the liquid, and are carried forward from vessel to vessel as fresh liquid is admitted into the first of the series. In this manner the higher oxids of lead and the metallic lead are gradually separated.

Economic Metallurgical and Chemical Process. J. E. Bucher, Feb. 3, 1914. U. S. Pat. 1,086,019. Iron is heated with nitrogen and one or more powerful reducing elements such as sodium or potassium. A portion of the carbon in the iron combines with the purifying reagents to form a cyanogen compound



which is separated from the iron. In using the apparatus illustrated in practicing the process nitrogen is introduced into the retort through the pipe 24, the iron and reducing metal being previously heated to the boiling point of sodium or whatever reducing metal is employed.

Asphalt from Crude Mineral Oil or Residum Thereof. C. B. Forward, March 3, 1914. U. S. Pat. 1,088,692. Crude oil is subjected to a high temperature in a retort until all the volatile parts are separated from the asphalt base. The heavier portions of the volatile parts are condensed and mixed with the asphalt base, the mixture being heated in an open vessel until a desired adhesive condition is produced.



Detinning. F. von Kugelgen and G. O. Seward, Feb. 10, 1914. U. S. Pat. 1,086,921. Dry chlorine gas is circulated through tanks 1, 2, 3 and 4 containing tin scrap in various stages of detinning, the temperature being controlled to keep it below that at which the iron is attacked.

Refractory Brick. H. Wessling, March 3, 1914. U. S. Pats. 1,088,755 and 1,088,756. The brick is formed of fine particles of silica bounded together by a finely pulverized soapstone.

Hydrometallurgy of Copper. E. R. Weidlein, March 3, 1914. U. S. Pat. 1,089,096. Copper-bearing material is leached to obtain a copper solution containing not more than 3 per cent copper. The free sulfuric acid in the solution is neutralized and the copper precipitated substantially quantitatively by means of sulfur dioxide.

MARKET REPORT

AVERAGE WHOLESALE PRICES OF STANDARD CHEMICALS, ETC., FOR THE MONTH OF MAY, 1914

ORGANIC CHEMICALS

Acetanilid.....	Lb.	21 1/2	@	22
Acetic Acid (28 per cent).....	C.	1.50	@	1.65
Acetone (drums).....	Lb.	10 1/2	@	11
Alcohol, denatured (180 proof).....	Gal.	35	@	37
Alcohol, grain (188 proof).....	Gal.	2.52	@	2.54
Alcohol, wood (95 per cent).....	Gal.	45	@	47
Amyl Acetate.....	Gal.	1.55	@	1.60
Aniline Oil.....	Lb.	10	to	10 1/4
Benzoic Acid.....	Lb.	23	@	27
Benzol (90 per cent).....	Gal.	23	to	25
Camphor (refined in bulk).....	Lb.	—	@	42 1/2
Carbolic Acid (drums).....	Lb.	7 3/4	@	9
Carbon Bisulfide.....	Lb.	6 1/2	@	8
Carbon Tetrachloride (drums).....	Lb.	7 3/8	@	7 3/4
Chloroform.....	Lb.	20	@	24
Citric Acid (domestic), crystals.....	Lb.	51	@	51 1/2
Dextrine (corn).....	C.	2.77	@	2.97
Dextrine (imported potato).....	Lb.	5 1/2	@	6
Ether (U. S. P., 1900).....	Lb.	18	@	24
Formaldehyde.....	Lb.	8 1/2	@	9 1/2
Glycerine (dynamite).....	Lb.	19	@	19 1/2
Oxalic Acid.....	Lb.	7 1/2	@	7 3/4
Pyrogallic Acid (bulk).....	Lb.	1.20	@	1.40
Salicylic Acid.....	Lb.	25	@	27
Starch (cassava).....	Lb.	3 1/4	@	4
Starch (corn).....	C.	1.99	@	2.10
Starch (potato).....	Lb.	4 3/4	@	4 7/8
Starch (rice).....	Lb.	7	@	8
Starch (sago).....	Lb.	2 1/4	@	2 3/4
Starch (wheat).....	Lb.	4 3/4	@	5 1/4
Tannic Acid (commercial).....	Lb.	35	—	36
Tartaric Acid, crystals.....	Lb.	—	@	30 1/4

INORGANIC CHEMICALS

Acetate of Lead (brown, broken).....	Lb.	7 1/4	@	7 1/2
Acetate of Lime (gray).....	C.	1.50	@	1.55
Alum (lump).....	C.	1.75	@	2.00
Aluminum Sulfate (high-grade).....	C.	1.25	@	1.75
Ammonium Carbonate, domestic.....	Lb.	8	@	8 1/2
Ammonium Chloride, gray.....	Lb.	5 7/8	@	6 1/8
Aqua Ammonia (drums) 16°.....	Lb.	2 1/4	@	2 1/2
Arsenic, white.....	Lb.	3	@	3 1/8
Barium Chloride.....	Lb.	1 3/8	@	1 3/4
Barium Nitrate.....	Lb.	5	to	5 1/4
Barytes (prime white, foreign).....	Ton	19.00	@	23.50
Bleaching Powder (35 per cent).....	C.	1.22 1/2	@	1.25
Blue Vitriol.....	C.	4.80	@	5.00
Borax, crystals (bags).....	Lb.	3 3/4	@	4 1/2
Boric Acid, crystals (powd.).....	Lb.	7	@	8
Brimstone (crude, domestic).....	Ton	22.00	@	22.50
Bromine, bulk.....	Lb.	30	@	35
Calcium Chloride, fused.....	C.	60	@	65
Chalk (light precipitated).....	Lb.	4	@	4 1/2
China Clay (imported).....	Ton	14.00	@	16.00
Feldspar.....	Ton	8.00	@	12.00
Fuller's Earth, powdered, Foreign.....	Ton	16.00	@	17.00
Green Vitriol (bulk).....	C.	55	@	60
Hydrochloric Acid (18°).....	C.	1.15	@	1.65
Iodine (resublimed).....	Lb.	3.55	@	3.60
Lead Nitrate.....	Lb.	8	@	8 1/8
Litharge (American).....	Lb.	5 1/2	@	5 3/4
Lithium Carbonate.....	Lb.	65	@	70
Magnesium Carbonate.....	Lb.	21	@	35
Magnesite "Calcined".....	Ton	28.50	@	29.50
Nitric Acid, 36°.....	Lb.	3 7/8	@	4 1/4
Phosphoric Acid (sp. gr. 1.75).....	Lb.	20	@	24
Phosphorus.....	Lb.	45	@	1.00
Plaster of Paris.....	Bbl.	1.50	@	1.70
Potassium Bichromate, 50°.....	Lb.	6 3/4	@	7
Potassium Bromide.....	Lb.	39	@	40
Potassium Carbonate (calcined), 80 @ 85%.....	C.	3	@	3 1/8
Potassium Chlorate, crystals.....	Lb.	7 1/4	@	7 3/4
Potassium Cyanide (bulk), 98-99%.....	Lb.	19	@	22
Potassium Hydroxide.....	Lb.	4	@	4 1/8
Potassium Iodide (bulk).....	Lb.	2.95	@	3.00
Potassium Nitrate (crude).....	Lb.	4 3/4	@	5 1/4
Potassium Permanganate (bulk).....	Lb.	9 3/4	@	10
Quicksilver, Flask (75 lbs.).....		37.50	@	—
Red Lead (American).....	Lb.	6	@	6 1/4
Salt Cake (glass-makers').....	C.	55	@	65

Silver Nitrate.....	Oz.	37	@	39
Soapstone in bags.....	Ton	10.00	@	12.00
Soda Ash (48 per cent).....	C.	67 1/2	@	72 1/2
Sodium Acetate.....	Lb.	3 3/4	@	4 1/8
Sodium Bicarbonate (domestic).....	C.	1.00	@	1.10
Sodium Bicarbonate (English).....	Lb.	2 3/4	@	3
Sodium Bichromate.....	Lb.	4 3/4	@	5
Sodium Carbonate (dry).....	C.	60	@	80
Sodium Chlorate.....	Lb.	7 1/4	@	7 1/2
Sodium Hydroxide, 60 per cent.....	C.	1.55	@	1.57 1/2
Sodium Hyposulfite.....	C.	1.30	@	1.60
Sodium Nitrate, 95 per cent, spot.....	C.	—	@	2.20
Sodium Silicate (liquid).....	C.	65	@	1.50
Strontium Nitrate.....	Lb.	6 3/4	@	7
Sulfur, Flowers (sublimed).....	C.	2.20	@	2.60
Sulfur, Roll.....	C.	1.85	@	2.15
Sulfuric Acid, 60° B.....	C.	85	@	1.00
Talc (American).....	Ton	15.00	@	20.00
Terra Alba (American), No. 1.....	C.	75	@	80
Tin Bichloride (50°).....	Lb.	11 1/4	@	12 1/2
Tin Oxide.....	Lb.	38	@	40
White Lead (American, dry).....	Lb.	5 1/4	@	5 1/2
Zinc Carbonate.....	Lb.	8 1/2	@	9
Zinc Chloride (granulated).....	Lb.	4 1/2	@	5
Zinc Oxide (American process).....	Lb.	5 3/8	@	6 1/8
Zinc Sulfate.....	C.	2.35	@	2.70

OILS, WAXES, ETC.

Beeswax (pure white).....	Lb.	45	@	47
Black Mineral Oil, 29 gravity.....	Gal.	13 1/2	@	14
Castor Oil (No. 3).....	Lb.	8	@	8 1/8
Ceresin (yellow).....	Lb.	12	@	22
Corn Oil.....	C.	6.55	@	6.60
Cottonseed Oil (crude), f. o. b. mill.....	Gal.	45	@	46
Cottonseed Oil (p. s. y.).....	Lb.	7	@	7 1/4
Cylinder Oil (light, filtered).....	Gal.	21 1/2	@	32
Japan Wax.....	Lb.	12	@	13
Lard Oil (prime winter).....	Gal.	92	@	95
Linseed Oil (raw).....	Gal.	49 1/2	@	52
Menhaden Oil (crude).....	Gal.	nominal		
Neatsfoot Oil (20°).....	Gal.	96	@	98
Paraffine (crude, 120 & 122 m. p.).....	Lb.	3 1/8	@	3 1/4
Paraffine Oil (high viscosity).....	Gal.	27	@	28
Rosin ("F" grade) (280 lbs.).....	Bbl.	4.50	@	—
Rosin Oil (first run).....	Gal.	—	@	27
Shellac, T. N.....	Lb.	15	@	16
Spermaceti (cake).....	Lb.	30	@	31
Sperm Oil (bleached winter), 38°.....	Gal.	70	@	71
Spindle Oil, No. 200.....	Gal.	18	@	19
Stearic Acid (double-pressed).....	Lb.	9	@	12
Tallow (acidless).....	Gal.	65	@	66
Tar Oil (distilled).....	Gal.	30	@	31
Turpentine (spirits of).....	Gal.	47	@	47 1/2

METALS

Aluminum (No. 1 ingots).....	Lb.	17 1/2	@	18
Antimony (Hallet's).....	Lb.	6 3/4	@	7 1/4
Bismuth (New York).....	Lb.	2.05	@	2.10
Bronze powder.....	Lb.	50	@	3.00
Copper (electrolytic).....	C.	14.10	@	14.20
Copper (lake).....	C.	14.25	@	14.35
Lead, N. Y.....	C.	3.90	@	—
Nickel.....	Lb.	50	@	55
Platinum (refined).....	Oz.	43.50	@	44.50
Silver.....	Oz.	58	@	58 1/4
Tin.....	C.	32.95	@	37.00
Zinc.....	C.	5.15	@	5.20

FERTILIZER MATERIALS

Ammonium Sulfate.....	C.	2.80	@	2.85
Blood, dried.....	Unit	3.35	@	—
Bone, 4 1/2 and 50, ground, raw.....	Ton	30.00	@	—
Calcium Nitrate (Norwegian).....	C.	2.05	@	2.15
Castor meal.....	Unit	3.35	@	—
Fish Scrap, domestic, dried.....	Unit	nominal		
Phosphate, acid, 16 per cent bulk.....	Ton	7.00	@	—
Phosphate rock; f. o. b. mine:				
Florida land pebble, 68 per cent.....	Ton	2.25	@	2.50
Tennessee, 70-80 per cent.....	Ton	5.00	@	5.50
Potassium, "muriate," basis 80 per cent.....	Ton	39.07	@	—
Pyrites, furnace size, imported.....	Unit	0.13 1/4	@	—
Tankage, high-grade.....	Unit	3.35	&	10