The Journal of Industrial and Engineering Chemistry

Published by THE AMERICAN CHEMICAL SOCIETY

Volume V | APRIL, 1913 | No. 4

Published monthly. Subscription price to non-members of the American Chemical Society, $6.00 yearly.
Foreign postage, seventy-five cents, Canada, Cuba and Mexico excepted.
Entered as Second-class Matter December 19, 1908, at the Post-Office at Easton, Pa., under the Act of March 3, 1879.

Contributions should be addressed to M. C. Whitaker, Columbia University, New York City
Communications concerning advertisements should be sent to The American Chemical Society, 42 West 39th St., New York City
Subscriptions and claims for lost copies should be referred to Charles L. Parsons, Box 505, Washington, D. C.
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ESCHENBACH PRINTING COMPANY, EASTON, PA.

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EDITORIALS

THE NICHOLS-HESSE DINNER

The chemists of America, in whose behalf Dr. William H. Nichols, as president, and Dr. Bernhard C. Hesse, as secretary, assumed the burden of organizing and administering the affairs of the Eighth International Congress of Applied Chemistry, will celebrate the successful completion of this gigantic undertaking by a Nichols-Hesse dinner. Dr. Edward W. Morley, the faithful honorary president of the Congress, will preside at the function which will include not only a dinner but the presentation to the guests of testimonials of regard and appreciation.

The committee, made up of representatives of the American Chemical Society, American Electrochemical Society, American Institute of Chemical Engineers, Chemists' Club, American Sections of the Society of Chemical Industry and the New York Section of the Verein Deutscher Chemiker, will make this a memorable event as the closing chapter of the Eighth Congress. It is distinctly a chemists' function and will, therefore, be given at the Chemists' Club. The date selected is April 19th, and tickets may be obtained from Dr. L. H. Baekeland, of Yonkers, New York.

The Eighth Congress was the most important event in the history of Applied Chemistry in America. Our professional progress and our industries were opened to the critical inspection of picked representatives from all important foreign countries. The assumption of the responsibilities for the organization and administration of this great task took courage and foresight, and involved a most extraordinary amount of hard work and anxiety, so that its successful completion calls for the admiring gratitude of every American chemist. What was done was undertaken solely in the interest of Applied Chemistry and its followers and without regard to society or business affiliations. This celebration, therefore, becomes not a matter of this organization or that, but a sincere tribute of appreciation from American chemists in general to the men who assumed and carried our burden so successfully.

Many willing chemists will not be able to come to the dinner, but will have to rely on letters and telegrams to express their appreciation. Whatever method may be dictated by conditions, sincere and cordial admiration and applause will be general among American Chemists for the magnificent Nichols-Hesse team-work.

THE RESEARCH BUREAU OF THE CHEMISTS' CLUB

On February 22nd the Library Committee of the Chemists' Club announced the organization of a Research Bureau. The Chemists' Club Library has the largest collection of chemical literature in America. Donations by Charles F. Chandler, Morris Loeb, the American Chemical Society, the Society of Chemical Industry, J. W. Mallet, Herman Frasch and many others have made it possible to assemble complete up-to-date editions of practically all the important scientific and technical chemical publications, both American and foreign. Notable examples of unusually valuable possessions are the following:

American Chemical Journal, 1879 to date.
American Journal of Science, 1819 to date.
Analyst, 1877 to date.
Berichte der deutschen chemischen Gesellschaft, 1868 to date.
Bulletin de la Société Chimique, 1861 to date.
Bureau of Chemistry Bulletins, 1–160, 1882 to date.
Canadian Patent Office Record, 1890 to date.
Chemical News, 1867 to date.
Chemiker Zeitung, 1877 to date.
Comptes Rendus, 1835 to date.
Encyclopedie Chimique-Fremy's, complete.
Experiment Station Bulletins 1–248, 1889 to date.
Experiment Station Record, 1889 to date.
Gazzetta chimica italiana, 1871 to date.
Illustrated Official Journal (British Patents), 1900 to date.
International Sugar Journal and its predecessor, Sugar Cane, 1872 to date.
Journal für praktische Chemie, 1828 to date.
Journal of the American Chemical Society, 1879 to date.
Journal of the Association of Engineering Societies, 1881 to date.
Journal of the Chemical Society, London, Abstracts and Transactions, 1849 to date.
Journal of the Society of Chemical Industry, 1882 to date.
Liebig's Jahresberichte, 1847 to date.
Pharmaceutical Journal, 1841 to date.
Proceedings of the American Pharmaceutical Association, 1857 to date.
Repertorium der Technischen Journal Literature, 1829 to date.
School of Mines Quarterly, 1879 to date.
Transactions of the American Institute of Mining Engineers, 1871 to date.
U. S. Patent Office Gazette, 1790 to date.
Wagner's Jahresberichte, 1855 to date.
Zeitschrift für analytische Chemie, 1862 to date.
Zeitschrift für angewandte Chemie, 1887 to date.
Zeitschrift für anorganische Chemie, 1889 to date.
Zeitschrift für Untersuchung der Nahrungs und Genussmittel and its predecessor, 1881 to date.

When the Morris Loeb Library has been entirely moved in, the Chemisches Zentralblatt, 1830 to date, and Liebig's Annalen complete, 1826 to date, will be added to the above list as well as all of the important English, French and German publications on purely theoretical and physical chemistry.

The Charles Frederick Chandler Library, the most complete private technical library in the United States is only partly installed.

Information on any chemical subject that has been investigated and put into print can now be supplied at reasonable cost to busy professional men, technical publications, lawyers, patent attorneys, manufacturers and teachers. On short notice articles will be copied, translations made, bibliographies prepared and searches of chemical or patent literature carried on, both in the Chemists' Club Library and in the principal libraries of New York City. The world's work in chemistry is thus made available to all.

While the Chemists' Club Library is making every effort to obtain all the most valuable chemical textbooks and treatises and possesses a great many of them, it realizes that its greatest service to chemical progress is to be rendered by means of the scientific and technical journals that contain original articles and reports. Every chemist owns the best books on his particular branch of the subject but it is absolutely impossible for him to possess complete editions of the multitude of valuable journals now being printed or to obtain the back numbers he needs *when* he needs them. At the same time the various branches of chemistry are so intimately connected, interwoven and dependent upon each other that in research of any sort it is practically always necessary to cover the whole field in order to find out what has been done as well as to prevent duplication of work. Only such facilities as are now offered by the Research Bureau of the Chemists' Club can supply the needs of modern chemical investigation and progress.

In addition to the Research Bureau, Club members, resident and non-resident, have access to the Circulating Library, on payment of handling charges. Eight thousand duplicates of books, pamphlets, reprints, etc., are available for study and use by any member in the reach of mail or express service. Many triplicates are also on hand to be exchanged for desirable literature or sold to those who need them for the completion of personal files.

In return for the many advantages now offered by the Chemists' Club, every chemist in America owes it his most hearty support. Any books, reprints, or journals in odd numbers, volumes or broken sets can be used by it with unusual and often surprising results. Odd, old volumes and particular editions are of the utmost importance in patent and research work, as well as for completing sets that are in many cases useless if incomplete.

The Chemists' Club believes in the spread of chemical knowledge and understanding. It knows that modern chemistry can no longer depend upon accidental discoveries for progress. It offers ample material for intelligent and productive research. May the American Chemists use abundantly these new resources and justify the patience and faith of those who have labored so long to make possible this remarkable expansion of the work of the Chemists' Club!

NEGATIVE COÖPERATION

Speakers who pretend to keep up with the latest styles in matters of thought transmission sooner or later work their theme around to "Efficiency" or "Coöperation." Men trying to accomplish something worth while often find the conviction gaining on them that both of these words are "happy thoughts" but are a long way from tangible realities. People are daily becoming more reluctant to undertake public work, not because they are unwilling to sacrifice their time, ability and money for the good of others, but because they are met at the outset with a condition which for want of a better term we shall call "negative coöperation." Men of energy and ability are often elevated to positions of honor and trust and are pelted by their friends with bouquets only to find that the bouquets are sooner or later displaced by bricks. Others hesitate to accept political responsibility because they know that the blatant unreasoning criticism of the yellow press is inevitable.

Why do we not practice coöperation as well as preach it? Why do we select a most able mechanic to erect a structure of great importance to us and then give him a kit of tools containing saws, hammers and chisels made of rubber? Is it not enough to ride on the wagon we have delegated one of our fellow workers to draw for us without letting our own feet drag?

This issue of the Journal contains the final report of the Secretary of the Eighth International Congress and also an address by Dr. Hesse, drawing some striking lessons and offering suggestions which should be of inestimable value to future organizations. Some vivid examples of negative coöperation are disclosed in these records.

Early in May, 1912, definite arrangements between the officers of the Congress and the printers were completed, in which it was provided that all copy received before July 16th should be made up into 4000 sets of 24 volumes, or a total of 96,000 volumes, and delivered at the Columbia University Gymnasium before the Congress opened. The printers fully lived up to that agreement. They further agreed to give the subsequent printed matter of the Congress the right of way over everything else in their office (provided copy was completely delivered by the close of September) and to have all such matter printed and bound by November 15, 1912.

It became perfectly clear in September that the necessary coöperation on the part of lecturers, participants in discussions and contributing authors for complete delivery of copy according to agreement, would not be forthcoming. Therefore, the right-of-way arrangement with the printer had to be cancelled in order to avoid unnecessary financial loss. Not until March 11, 1913, was it possible to get from the authors all of the copy of what is now Volumes 25, 26, 27 and 28—this in spite of repeated and most urgent letters from the Secretary asking for that material. The printer was then under contract with others and the Congress matter had to take its place in line, with consequent additional delay. Even now many of those whose formal remarks or other communications should be embodied in these volumes are not represented. Had these volumes been sent to press in September with only

the material then available, they would have been very meagre and some of the most important papers would not have appeared.

The Congress had placed December 31, 1912, as its delivery date, counting with absolute confidence upon the coöperation of its own members, and for this delay the delinquent members of this Congress are alone to blame. The Congress and the printers were ready to perform their part, but they were not given any chance because the prompt coöperation they needed, and had every right to expect, had been withheld.

The fact that some of the members of the Congress will be unable to get copies of Volumes 1–24 is also due to another case of negative coöperation. In March, 1911, and in almost every one of the succeeding Congress publications, as well as in the various technical journals, attention was directed to the fact that memberships completed after July 1st could not be guaranteed the full reports. It is perfectly obvious to anyone that full information in regard to the demand was needed by the officers in order to provide the necessary number of volumes. As it was, the contracts for paper and printing had to be made when the total membership was only 1200—that after five months' active campaigning for members. Four thousand sets were then ordered with the provision that this number could be increased up to 7500 at any time prior to July 16th. Renewed efforts to obtain members were made, but at the end of ten weeks when the time limit for extension expired, the membership had risen to only 1800. Therefore, with a surplus of almost 2000 sets, it was not regarded as essential to increase the financial burden by adding to the original contract. The Congress is now, when it is too late to remedy matters, confronted with a membership far in excess of the number of sets of its proceedings available for distribution. They will be obliged to exercise the only logical course left open to them and that is to distribute all sets now in hand in order of the dates of the memberships. Will the men who were dragging their feet in the sand while the officers of the Congress pulled hard at the wagon, assume any of the responsibility for this situation?

A high and prominent place among the world's seven wonders should be given to that human quality which impels men such as Hesse to give their time and energy to a general cause when they know that by far the greater portion of what they contribute will be absorbed to offset the indifference and negative coöperation of those for whom they work.

ORIGINAL PAPERS

ACCURACY AND LIMITATIONS OF COAL ANALYSIS[1]

By A. C. Fieldner

Received January 22, 1913

INTRODUCTION

In recent years chemical and physical tests have come into use in connection with the mining and utilization of coal. Within certain limits these tests are useful not only in establishing the fuel value but in directing the coal into the proper market for which it is best adapted, and of maintaining the standard of the output to meet the requirements of this market. The growing practice of buying on specifications demands accuracy and strict uniformity in the methods of sampling and analysis. Chemists who make only occasional analyses of coal do not as a rule realize the empirical nature of the proximate analysis, and while they may obtain concordant results as far as their own laboratories are concerned, their results may not agree with those obtained in another laboratory. These discrepancies, which are more likely to occur in some determinations like that of volatile matter or of fixed carbon, tend to discredit the whole analysis in the eyes of engineers not familiar with the difficulties peculiar to each individual determination. The object of this paper is to call attention to the fact that some constituents may be determined much more accurately than others, and to present some experimental data bearing on the probable variations that may occur in good laboratory practice.

[1] Paper read before American Coal Mining Institute, Pittsburgh, Pa., Dec. 19, 1912, by permission of the Director, U. S. Bureau of Mines.

NATURE OF COAL

Coal is used principally for fuel purposes; hence its value, other things being equal, is proportional to its calorific power. The different kinds of coal, however, vary greatly in character, so much so that each has its own particular field of usefulness. For instance, a Pittsburgh steam coal cannot be burned efficiently in an anthracite furnace, nor can a coal high in sulfur or phosphorus be used for the manufacture of foundry coke. Certain other tests, then, are also required, and for this purpose we have the conventional proximate analysis and sulfur or phosphorus determinations. While these tests may be sufficient for most industrial purposes, it is desirable at times to know the elementary composition of the coal as shown in an ultimate analysis, and its clinkering properties as indicated in the relative fusibility of the ash. No doubt other tests more suitable for individual purposes will come into use as the chemist learns what coal really is.

It is generally conceded by geologists that coal has its origin in vegetable matter that was accumulated and buried in past geological ages. Several different theories have been advanced as to the exact manner of accumulation and the particular kinds of vegetation or vegetable products that were involved. At any rate, this accumulation became more or less altered and concentrated under swampy conditions and finally buried under a load of sediments, to be subjected to a further change by the action of heat and pressure. In more or less intimate mixture with

these organic remains was deposited silt, sand, clay, and other earthy materials. Sometimes layers of sediments were laid down between the coal-forming vegetation. It is not surprising, then, that the present chemical constitution of coal is not only of an extremely complex nature but that it varies from one bed to another.

For fuel purposes the constituents of coal may be grouped into the following three classes:

1. Water or moisture.
2. The mineral impurities which remain in a somewhat altered condition, as ash on burning the coal.
3. The organic or combustible matter, which like the original vegetable matter is composed mainly of the elements carbon, hydrogen, oxygen, and nitrogen.

MOISTURE

Moisture may be accurately determined in an inorganic substance like iron ore by drying the material at a temperature slightly above the boiling point of water and noting the loss in weight. The same method when applied to coal is complicated by the oxidizing tendencies of the organic compounds, and the possibility of loss of some water of composition at the temperature of drying. The present methods of analytical chemistry do not distinguish with certainty between added moisture or water that is simply absorbed by the coal particles, and water that may be in some unstable chemical combination in the coal substance. The usual method of drying a pulverized sample for one hour at 105° C. (221° F.) is, therefore, somewhat arbitrary, and to secure uniform results a strict adherence to a standard method of procedure is necessary.

In the Bureau of Mines method[1] the moisture is determined in two stages: (1) The coarse sample received by the laboratory is pulverized to $^1/_4$ inch size and then air-dried at 30°–35° C. (86°–95° F.). The loss in weight, which is called the air-drying loss, includes all the superficial moisture and a large portion of the loosely retained moisture. (2) The air-dried sample is then pulverized in a closed ball mill to avoid loss of moisture, mixed and sampled down to a small, powdered sample in which the residual moisture is determined by heating 1 gram for 1 hour at 105° C. (221° F.) in an oven through which dry air is being circulated. By this method duplicate results in the same powdered sample seldom vary more than 0.15 per cent.

ASH

Ash is the incombustible residue left on burning coal. It is derived from the mineral impurities in the coal and is largely composed of silica, alumina, lime, and iron compounds, together with smaller quantities of magnesia, titanium, and alkali compounds. The silica, alumina and titanium are derived from sand, clay, shale and slate; the iron oxide mainly from iron pyrites; and the lime and magnesia from their corresponding carbonates and sulfates.

The ash-forming constituents of coal may be classified in two groups:

1. Mineral matter present in an intimate mixture with the coal substance, derived either from the vegetable structure or from earthy matter intermixed as silt during the process of coal formation.
2. Mineral matter occurring in the form of thin bands and nodules of shale, bone, and pyrites; in this class are also the fragments of roof and floor that become mixed with the coal in mining.

Mineral matter from the first source can not be removed by washing methods; it is sometimes called the "intrinsic ash" of the coal. Mineral matter from the second source, sometimes called "extraneous ash," may be removed by methods of washing, screening, and picking. In the laboratory a similar separation can be made by the float-and-sink test. Coal has a specific gravity somewhat less than 1.35; the impurities are heavier; hence a separation may be made by placing the coal, crushed to a suitable size, in a solution of calcium or zinc chloride of 1.35 specific gravity. The coal will float, while the shale, bone, pyrites, etc., will sink. In this way the possibility of improving the quality of coal by washing can be determined.

The determination of ash, although one of the simplest operations, is also beset with certain difficulties that lead to disagreement among different laboratories, more especially with coals containing notable quantities of calcium carbonate and iron pyrites. On ignition the calcium carbonate is decomposed, and carbon dioxide driven off, either partially or completely, depending on the duration and temperature of ignition. The iron pyrites is changed to ferric oxide, while more or less of the sulfur combines with the lime to form $CaSO_4$. For example, in certain experiments with Illinois coals that contained notable quantities of calcium carbonate and sulfur, 14 per cent. ash was obtained by ignition to constant weight at a low-red heat, and 13 per cent. ash was obtained by ignition at a bright-red heat. To secure concordant results a standard temperature should be adopted. If this is done duplicate determinations on the same powdered sample will agree to within 0.2 per cent.

Coal ash as determined usually weighs less than the mineral matter from which it is produced. This is due mainly to the loss of volatile constituents during ignition: the shale and clay will lose their water of composition; the carbonates will be more or less decomposed, giving off carbon dioxide; and the iron pyrites will be changed to ferric oxide, giving off sulfur.

Several methods have been proposed to compute the weight of original mineral matter in the coal, by adding corrections to the weight of ash obtained by ignition. These methods are, however, too complicated and uncertain in their general application to all classes of coal to be used in technical work.

VOLATILE MATTER AND FIXED CARBON

The volatile matter and fixed carbon represent the relative proportions of gaseous and solid combustible matter that may be obtained from the coal by heating it in a closed vessel. This is done by heating a finely powdered sample in a small, covered platinum crucible, in the flame of a Bunsen or Méker

[1] F. M. Stanton and A. C. Fieldner, "Methods of Analyzing Coal and Coke," Bureau of Mines, *Technical Paper* 8.

burner, for exactly seven minutes. The volatile matter consists mainly of the combustible gases—hydrogen, carbon monoxide, methane, and other hydrocarbons—and some non-combustible gases such as carbon dioxide and water vapor. The volatile matter does not include water present in the coal as moisture at 105° C. (221° F.).

The residue of coke left in the crucible after deducting the ash is reported as "fixed carbon." The fixed carbon does not represent the total carbon in the coal, as a portion of this element is driven out in combination with hydrogen in the volatile matter; furthermore, fixed carbon is not pure carbon, but still contains several tenths per cent. each of carbon, hydrogen, and oxygen; from 0.4 to 1.0 per cent. nitrogen; and about half the sulfur that was in the coal. It should be clearly understood that the terms "volatile matter" or "volatile combustible matter" and "fixed carbon" do not represent any definite compounds which existed in the coal before heating. The method of determination is purely arbitrary, and variations in temperature and rate of heating will cause variations amounting to several per cent.; even with a strict adherence to the method recommended by the American Chemical Society, variations of three and four per cent. in both the volatile matter and fixed carbon may occur in different laboratories. One of the most prominent factors in causing variations is the temperature at which the crucible is heated. This is especially pronounced in anthracite and semi-bituminous coal. It is not improbable for one laboratory to report 4 per cent. and another 7 per cent. volatile matter on the same sample of anthracite, or 14 per cent. and 17 per cent. respectively on the same sample of Pocahontas coal. The different percentages of volatile matter were actually produced by different conditions of heat treatment. Caution must, therefore, be observed in making comparisons of the volatile matter and fixed carbon in proximate analyses made in different laboratories. Even determinations made at the same laboratory by the same analyst may vary to the extent of 0.5 per cent.

Recently a series of experiments was made at the Pittsburgh station of the Bureau of Mines to provide data on which to base a temperature specification at which the most uniform analytical results could be obtained.[1] A series of determinations at temperatures varying from 750° C. (1382° F.) to 1100° C. (2012° F.) were made on five different types of coal and one sample of foundry coke. These determinations were made in a 30 cc. platinum crucible, which was heated in an electric furnace under uniform conditions. Oxidation was prevented by passing nitrogen into the crucible. The results obtained are given in Table I and graphically illustrated in the curves in the accompanying illustration.

It will be seen that more volatile matter is obtained at the higher temperatures, although the ratio between temperature and volatile matter varies in the different coals tested. The curve for anthracite is remarkable in that it is practically a straight line. The curves for the three bituminous coals become horizontal at about 950° C. (1742° F.). The Pocahontas curve retains a slight upward inclination even at the higher temperatures.

From the analytical standpoint, 950°–1000° C. (1742°–1832° F.) appears to be the best temperature for the determination of volatile matter, as slight variations in temperature on the upper end of the curve produce less deviation in results.

TABLE I—VOLATILE MATTER AT DIFFERENT TEMPERATURES

C° temperatures	Per cent. of volatile matter					
	Coke	Anthracite	Pocahontas	Pittsburgh	Colorado	Illinois
740–745	0.40	..	...	32.86	32.99	...
745–750	..	2.84 2.96	14.66	...	...	32.38
750–755	0.51	..	14.80	33.13	33.00	...
755–760	0.56	..	...	...	...	32.51
765–770	..	2.93	...	...	...	...
770–775	..	..	...	33.68	...	...
785–790	..	..	...	33.69	...	...
790–795	..	..	...	...	33.70	...
795–800	..	3.25		33.89	...	...
805–810	0.55 0.54	..	15.85	34.05	34.07	...
810–815	0.60	3.33	...	34.11	...	33.60
815–820	..	3.23	...	...	...	...
825–830	..	..	16.17	...	...	33.98
835–840	0.63	..	...	...	...	...
840–845	..	3.57	...	...	...	...
850–855	..	3.59	...	...	34.12	...
855–860	0.53	..	...	34.70	...	34.54
860–865	0.44	..	...	34.75	...	34.63
865–870	..	3.70	...	..	...	...
870–875	0.53	..	...	...	...	...
885–890	..	..	16.49	...	...	...
890–895	..	..	16.45	...	...	...
895–900	..	..	...	...	...	34.99
905–910	..	..	...	34.57	...	...
910–915	0.68	3.93 4.15	...	...	34.62	...
915–920	..	..	16.70 16.92	34.75	34.80	...
920–925	..	..	...	...	...	35.18
935–940	0.71	..	...	...	...	...
955–960	..	..	...	...	34.93	...
960–965	..	..	17.03	35.00	34.94	35.60 35.70
965–970	0.80	..	...	34.92	...	...
970–975	0.85	..	17.13	...	...	...
975–980	..	4.63 4.63	...	...	...	...
1000–1005	..	..	...	34.87	...	...
1005–1010	0.95	..	17.10	34.82	34.97	...
1010–1015	0.91	4.86	...	...	35.02	35.55 35.86
1015–1020	..	5.00	...	...	...	...
1025–1030	..	..	17.30	...	...	...
1050–1055	1.02	..	...	...	...	35.63
1055–1060	1.02	..	...	...	...	35.89
1060–1065	..	..	...	...	34.89	...
1065–1070	..	5.19	17.21 17.33	34.72 34.82	34.97	...
1075–1080	..	5.36	...	...	...	...
1085–1090	..	..	17.35	...	...	...
1110–1115	..	5.60	17.49	...	...	...
1120–1125	1.22	..	...	...	...	...
1125–1130	..	5.71	...	...	...	...
1130–1135	1.25	..	...	...	...	...

SULFUR

Sulfur is usually determined in connection with the proximate analysis. It is classified with the impurities and undesirable constituents of coal, although it usually exists in a combustible form and contributes to the heating value. Commonly sulfur is present as iron pyrites, either in brassy lumps and bands or

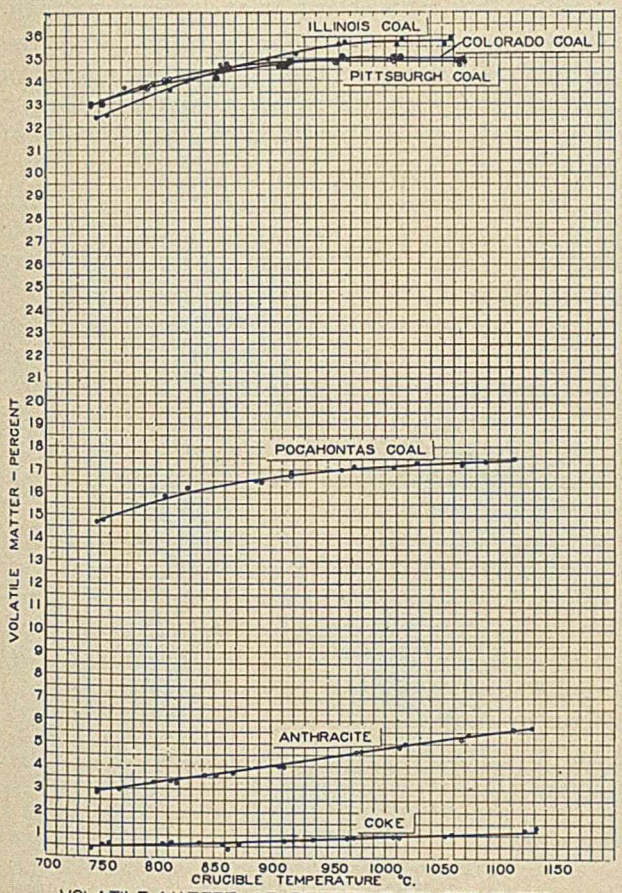
[1] A. C. Fieldner and A. E. Hall, "Influence of Temperature on the Determination of Volatile Matter in Coal," *Eighth International Congress of Applied Chemistry*, **10**, 139 (1912).

in very fine particles uniformly distributed throughout the coal. Sulfur may also occur in combination with iron and lime as sulfates and in combination with the coal substance as organic compounds.

Duplicate determinations by the "Eschka" method on the same powdered sample will usually agree to within 0.05 per cent. Where much sulfur is present in visible form, as pyrite, difficulty is experienced in obtaining a representative powdered sample from the coarse sample, and somewhat greater errors due to inaccuracies in sampling may occur.

ULTIMATE ANALYSIS

In an ultimate analysis the organic matter of the coal is resolved into its chemical elements, *viz.*, carbon, hydrogen, oxygen, sulfur and nitrogen. These elements, together with the ash, are taken as equal to 100 per cent. As there is no direct method for the determination of oxygen, it is estimated by subtracting the sum of the other five constituents from 100. This method throws the summation of all the errors incurred in the other determinations upon the oxygen.



VOLATILE MATTER AT DIFFERENT TEMPERATURES

The determination of carbon, hydrogen, and nitrogen, while requiring experience and a considerable degree of analytical skill on the part of the chemist, is not subject to the arbitrary conditions that must be maintained in the proximate analysis. As the chemist is dealing with definite chemical elements, he should be able to duplicate his determinations within the following limits: Carbon, 0.3 per cent.; hydrogen, 0.05 per cent.; nitrogen, 0.03 per cent.

It is unfortunate that no direct method has yet been devised for the determination of oxygen. The percentage of oxygen is an important factor in many of the problems connected with the classification, spontaneous combustion, weathering, and inflammability of coal. In the present method of estimation the oxygen percentage is subject to an error equal to the algebraic sum of the individual errors incurred in the determinations of carbon, hydrogen, nitrogen, sulfur, and ash. The most serious of these errors is due to the difference in weight between the ash and the mineral matter (exclusive of sulfur, which is separately determined) as it occurs in the coal. For instance, if all the sulfur is present as iron pyrites, the oxygen percentage will have a negative error equal to three-eighths of the sulfur. This correction, however, is applicable only when the percentage of pyritic sulfur is known, and a satisfactory method for determining all the different forms of sulfur found in coal is yet to be devised. Furthermore, there are errors in the opposite direction that tend to compensate the error due to the oxidation of iron pyrites, notably the loss of certain mineral constituents like carbon dioxide from carbonates and water of composition from the shaly constituents.[1] Prof. Parr has found the water of composition in Illinois coals to be approximately 8 per cent. of the ash. In a coal with 10 per cent. ash this would cause an error of +0.71 per cent. in the oxygen; if this same coal happened to contain 2 per cent. sulfur as iron pyrite, an additional error of —0.75 per cent. would be incurred in the oxygen. The two errors would thus compensate to within 0.04 per cent.; whether the final oxygen error is positive or negative would depend upon the relative proportions of shale and pyrite in the coal, and upon the quantities of carbonates of iron and lime. It is doubtful if any generally applied method of correction would materially increase the accuracy of the oxygen result.

The ultimate analysis as usually made does not distinguish between the carbon and hydrogen derived from the organic or combustible matter of the coal, and the small proportion of these elements that may be present in an incombustible form in the mineral impurities. Illinois coals, as shown by Prof. Parr[2] often contain 0.1–0.7 per cent. carbon as calcium carbonate. Most of the coal in the Appalachian field is comparatively free from carbonates. A small proportion of the hydrogen, usually from a few hundredths to about 0.1 per cent., has its origin in the water of composition of the clay or shaly material. While corrections can be made with the aid of special chemical methods for the small percentages of inorganic carbon, hydrogen, and oxygen, it is not necessary in the ordinary technical analyses.

In the consideration of an ultimate analysis it must be kept in mind that the hydrogen and oxygen of the moisture in the sample are included with the hydrogen and oxygen of the dry coal substance. Usually before making comparisons, ultimate analyses are computed to a dry-coal basis, thus giving the relative propor-

[1] S. W. Parr, Ill. State Geol. Survey, *Bull.* **8**, 154.
[2] Ill. State Geol. Survey, *Bull.* **16**, 232.

tions of hydrogen and oxygen in the dry coal.

The term "available hydrogen," often used in the computation of the calorific value of a coal from the ultimate analysis, is based on the assumption that all the oxygen in the coal is combined with hydrogen in the proper ratio to form water. The amount of hydrogen thus combined and not available for producing heat is equal to one-eighth of the oxygen; the remainder of the hydrogen, or "available hydrogen," is combined with the carbon and contributes to the heating value of the coal. This hypothesis is fairly well supported in the case of anthracite and bituminous coals by the general agreement of calorific values calculated from the ultimate analysis by Dulong's formula with the values determined by the bomb calorimeter.

CALORIFIC VALUE

The calorific value of a fuel is the total quantity of heat developed by the complete combustion of the unit weight of fuel. In the metric system of measurement which is usually used by chemists and physicists, the heat units are the gram calorie and the kilogram calorie. The gram calorie, or small calorie (cal.), is the amount of heat required to raise the temperature of one gram of water 1° C. at 15° C. and the kilogram calorie, or large calorie (Cal.), is the amount of heat required to raise the temperature of one kilogram of water 1° at 15° C.

In the English system which is generally used by engineers, the heat unit is the "British Thermal Unit," or B. t. u. The British thermal unit is the quantity of heat required to raise the temperature of one pound of water, 1° F., at 60° F. Calorific values given in calories per gram may be converted into British thermal units per pound, by multiplying by 1.8, because the temperature rise produced in one gram of water by the heat in one gram of coal will be the same as the rise produced in one pound of water by the heat in one pound of coal, hence, the relation between the calorific values of one gram and one pound of fuel is simply that of the two different thermometric scales, which is 9/5.

The most accurate method of determining the total heating value is by combustion in a bomb calorimeter. The instrument should be carefully standardized by burning substances of known calorific value such as the standard samples of cane sugar, naphthalene, and benzoic acid, that are now being furnished by the Bureau of Standards. The standardization should be conducted under exactly the same conditions, and with the same thermometer that is used in the tests. The use of calibrated thermometers is essential. In carefully conducted calorimetric work the probable error should not exceed 0.3 per cent., which corresponds to about 50 B. t. u. in a high-grade coal. This is a higher degree of accuracy than can be obtained in the usual methods of sampling, which oftentimes increase the error to 100 B. t. u.

CALCULATION OF CALORIFIC VALUE BY DULONG'S FORMULA

Before the bomb calorimeter came into general use, the heating value of a coal was frequently calculated from the ultimate analysis by Dulong's formula:

(1) Calorific value in calories per gram = $8080\,C + 34460\,(H - O/8) + 2250\,S$.

(2) Calorific value in B. t. u. per pound = $14544\,C + 62028\,(H - O/8) + 4050\,S$.

In the above formulas, C, H, O, and S are the respective proportions of carbon, hydrogen, oxygen and sulfur.

Aside from the fact that an ultimate analysis is more expensive and time-consuming than a calorimetric determination, there are the following theoretical objections to be considered:

(1) The heating value of the elements—carbon, hydrogen, and sulfur, as given in the formula—have not been established with any great degree of accuracy.

(2) The heating value of an element in the free state and as a component of a chemical compound is not necessarily the same; as a matter of fact it usually varies, due to absorption or evolution of heat in forming the compound.

(3) The usual assumption that from the standpoint of heat development all the oxygen is combined with hydrogen, may not be true; some of the oxygen may be linked with carbon.

(4) The relative accuracy of the calculated results are subject to the uncertainty of the oxygen estimation.

On the other hand, in spite of the objections just cited, there is fairly close agreement between the calorific values calculated by Dulong's formula and those determined by the calorimeter. Lord and Somermeier,[1] in a large series of analyses of the coals of Ohio, found that with very few exceptions the results obtained by Dulong's formula were within 1 — 1½ per cent. of the values obtained with the bomb calorimeter. This degree of accuracy has also been found to hold true with anthracite, semi-bituminous and bituminous coals of various parts of the United States as shown in Table II.

TABLE II—A COMPARISON OF CALORIFIC VALUES AS DETERMINED IN THE BOMB CALORIMETER AND AS CALCULATED FROM THE ULTIMATE ANALYSIS BY DULONG'S FORMULA

Calorific value in B. t. u. per pound = $14544\,C + 62028\,(H - O/8) + 4050S$

Description	Lab. no.	Proximate analysis, dry basis: Moisture Per cent.	Ash Per cent.	Vol. matter Per cent.	Calorific value, dry basis: Determined B. t. u.	Calculated B. t. u.	Difference B. t. u.	Error in calculated calorific value Per cent.
Dry peat								
Mass.....	12767	....	20.1	56.3	7745	7452	—293	—3.8
Mass.....	12766	....	11.2	64.2	9018	8477	—541	—6.0
Mass.....	10834	....	4.0	65.6	10285	9629	—656	—6.4
Mass.....	10833	....	4.0	64.4	10305	9718	—587	—5.7
Maine.....	5978	....	5.2	70.0	8471	7542	—929	—11.0
Maine.....	5983	....	3.8	67.6	9598	8600	—998	—10.4
Maine.....	5994	....	6.7	70.3	8132	7319	—813	—10.0
Maine.....	5975	....	3.8	66.6	8915	7974	—941	—10.6
Mich.....	6720	....	14.4	57.5	9139	8423	—716	—7.9
Wisc.....	6280	....	17.3	58.0	8149	7489	—660	—8.1
Wisc.....	6507	....	18.7	55.8	7835	7606	—229	—2.9
Wisc.....	6290	....	26.2	59.2	7465	7002	—463	—6.2
							Mean algebraic error in 12 samples.....	—7.4
							Maximum error.....	—11.0

[1] Geological Survey of Ohio, 4th series, *Bull.* **9**, 267 (1908).

TABLE II—(*Continued*)

Description	Lab. no	Proximate analysis as received: Moisture Per cent.	Ash Per cent.	Volatile matter Per cent.	Calorific value as received: Determined B. t. u.	Calculated B. t. u.	Difference B. t. u.	Error in calculated calorific value Per cent.
Lignite								
Valley Co.								
Mont.....	10898	42.8	4.6	25.7	6105	5785	—320	—5.2
Mont.....	10899	41.3	4.8	25.7	6260	5974	—286	—4.6
Mont.....	10900	41.6	4.1	27.1	5733	5467	—266	—4.7
Mont.....	10725	38.9	6.2	27.2	6014	5843	—171	—2.8
Mont.....	10726	35.3	7.7	31.8	5404	5227	—177	—3.3
Mont.....	10727	37.5	6.1	26.8	6580	6408	—172	—2.6
Mont.....	10724	32.6	9.1	27.4	6712	6664	—48	—0.7
Mont.....	11002	40.4	7.5	24.5	5830	5598	—232	—4.0
Mont.....	11003	39.3	5.7	24.2	6278	6059	—219	—3.5
Mont.....	11004	37.7	5.5	25.8	6700	6550	—150	—2.3
Mont.....	11005	40.7	6.2	25.2	6147	5980	—167	—2.7
Mont.....	11006	41.6	5.6	26.7	5278	4991	—287	—5.4
Morton Co.								
N. D.....	14729	36.2	8.7	29.8	6700	6559	—141	—2.1
N. D.....	1416	36.8	5.1	28.2	7204	6928	—276	—3.9
Perkins Co.								
S. D.....	12488	42.5	9.0	23.2	5954	5913	—41	—0.7
S. D.....	12453	39.2	8.4	24.7	6307	6277	—30	—0.5
Texas.....	1456	34.7	11.2	32.2	7056	6849	—207	—2.9
Texas.....	1597	33.7	7.3	24.3	7348	7155	—193	—2.6
Texas.....	2717	33.9	7.3	27.5	7497	7204	—293	—3.9
		Mean algebraic error in 19 samples.....						—3.1
		Maximum error in 19 samples.....						—5.4
Sub-bituminous coal, Rocky Mountain field								
Colorado								
El Paso Co...	10732	31.1	13.9	26.0	6199	6237	38	0.6
El Paso Co...	12099	26.2	6.5	29.7	8352	8114	—238	—2.8
El Paso Co...	10741	33.7	18.2	23.5	5506	5542	36	0.7
Wyoming								
Converse Co..	11048	28.1	4.6	31.6	8350	8057	—293	—3.5
Converse Co...	10775	27.7	10.0	26.7	7808	7763	—45	—0.6
Converse Co..	10740	27.9	8.3	27.1	7927	8001	74	0.9
Fremont Co...	10843	20.9	3.7	32.0	9781	9673	—108	—1.1
Fremont Co..	10842	21.1	5.8	31.4	9463	9293	—170	—1.8
Johnson Co...	11059	16.8	15.0	31.4	8480	8440	—40	—0.5
Johnson Co...	10827	23.5	5.2	35.6	9050	8719	—331	—3.8
Sheridan Co...	10810	22.7	3.4	32.2	9344	9221	—123	—1.3
Sheridan Co..	10811	24.7	3.7	31.8	9007	8852	—155	—1.7
Sheridan Co...	10825	22.6	4.6	32.5	9218	9131	—187	—2.0
Sheridan Co...	10826	22.1	4.4	33.4	9256	8978	—278	—3.0
Sheridan Co...	10822	23.5	4.9	31.2	9121	8935	—186	—2.0
Sheridan Co...	12005	22.3	3.7	35.0	9617	9364	—253	—2.6
Sheridan Co...	12008	23.1	3.2	31.9	9484	9302	—182	—1.9
Sheridan Co...	12006	23.4	3.6	33.6	9392	9259	—133	—1.4
Sweetwater Co.	11510	16.3	2.1	34.9	11153	11086	—67	—0.6
Sweetwater Co.	11460	13.1	3.4	35.8	11619	11576	—43	—0.4
		Mean algebraic error in 20 samples.....						—1.4
		Maximum negative error.....						3.8
		Maximum positive error.....						0.9
Bituminous coal Rocky Mt. field								
Canada								
Alberta.....	14883	10.6	16.7	36.4	9819	9828	9	0.1
Alberta.....	14882	9.8	6.8	40.2	11412	11385	—27	—0.2
Alberta.....	13177	6.5	16.2	34.5	10737	10582	—155	—1.4
Alberta.....	12737	2.0	17.6	27.4	12208	12166	—42	—0.4
Colorado								
La Plata Co...	14775	7.1	5.9	38.1	12672	12649	—23	—0.2
La Plata Co...	14772	3.0	6.9	34.8	13714	13853	139	1.0
Fremont Co..	13395	10.3	5.5	38.3	11416	11230	—186	—1.6
Las Animas Co	13364	3.3	15.6	32.0	12357	12175	—182	—1.4
Las Animas Co.	13254	5.4	8.6	33.0	13237	13176	—61	—0.5
Las Animas Co.	13253	6.3	14.1	30.2	12145	12121	—24	—0.2
New Mexico								
Colfax Co.....	13367	5.6	13.2	35.8	12019	12015	—4	0
Colfax Co.....	12235	3.3	12.3	35.7	12757	12773	16	0.1
Colfax Co.....	12338	3.6	12.0	36.1	12623	12580	—43	—0.3
Utah								
Carbon Co....	10906	7.5	5.6	39.7	12521	12523	2	0
Carbon Co....	10909	5.7	7.0	40.8	12647	12605	—42	—0.3
Oregon.....	12825	3.3	7.2	40.3	12640	12597	—43	—0.3

TABLE II—(*Continued*)

Description	Lab. no.	Proximate analysis as received: Moisture Per cent.	Ash Per cent.	Volatile matter Per cent.	Calorific value as received: Determined B. t. u.	Calculated B. t. u.	Difference B. t. u.	Error in calculated calorific value Per cent.
Oregon.....	12824	2.5	8.6	37.3	13039	13070	—31	—0.2
Washington								
Kittitas Co...	11525	3.9	12.6	37.0	12434	12546	112	0.9
Kittitas Co...	11526	3.8	11.5	37.7	12762	12840	78	0.6
Wyoming								
Uinta Co.....	11565	5.7	5.3	37.7	12580	12451	—129	—1.0
		Mean algebraic error in 20 samples.....						—0.3
		Maximum negative error.....						1.6
		Maximum positive error.....						1.0
Bituminous coal Illinois field								
Grundy Co.								
Bed No. 2....	14926	6.9	6.0	40.0	12290	12206	—84	—0.7
La Salle Co.								
Bed No. 2....	14923	5.1	9.7	41.9	12092	12139	47	0.4
Bed No. 2...	14922	6.8	9.5	41.0	11808	11732	—76	—0.6
Bureau Co.								
Bed No. 2....	14921	5.2	9.5	40.1	12078	11974	—104	—0.9
Clinton Co.								
Bed No. 6....	14712	8.4	10.3	37.5	11313	11302	—11	—0.1
Bed No. 6....	14711	7.8	10.7	38.5	11324	11363	39	0.3
Randolph Co.								
Bed No. 6....	14710	5.8	12.1	38.9	11399	11376	—23	—0.2
St. Clair Co.								
Bed No. 6....	14709	5.3	11.6	41.1	11700	11531	—169	—1.4
Bed No. 6....	14708	6.6	10.6	41.4	11637	11529	—108	—0.9
Bed No. 6....	14706	4.5	10.7	42.3	11923	11878	—45	—0.4
Montgomery Co.								
Bed No. 6....	14704	6.2	11.7	39.9	11534	11493	—41	—0.4
Sangamon Co.								
Bed No. 6....	14703	5.9	10.2	40.7	11691	11905	204	1.7
Bed No. 6....	14702	8.4	10.8	39.6	11122	11137	15	0.1
Bed No. 6....	14701	7.9	9.1	40.7	11527	11515	—12	—0.1
Madison Co.								
Bed No. 6....	14700	9.0	10.5	40.2	11164	11084	—80	—0.7
Bed No. 6....	14699	8.0	11.0	40.2	11322	11201	—121	—1.0
Williamson Co.								
Bed No. 6....	14697	5.3	8.7	37.3	12299	12164	—135	—1.1
Bed No. 6....	14696	6.8	8.1	33.9	12159	12049	—110	—0.9
Bed. No. 6....	14695	6.8	7.4	34.9	12258	12094	—164	—1.3
Bed No. 6....	14694	7.2	9.5	33.0	11902	11893	—9	0.1
Franklin Co.								
Bed No. 6....	14692	5.3	10.8	35.4	11974	12163	189	1.5
Bed No. 6....	14691	6.4	7.9	34.5	12253	12256	3	0
Bed No. 6....	14690	4.6	13.2	40.0	11749	11936	187	1.6
Fulton Co.								
Bed No. 5....	14688	4.5	12.9	39.1	11633	11821	188	1.6
Bed No. 5....	14687	5.7	12.0	38.7	11500	11657	157	1.4
		Mean algebraic error in 30 samples.....						—0.1
		Maximum negative error.....						1.4
		Maximum positive error.....						1.7
Bituminous coal Appalachian field								
Pennsylvania								
Allegheny Co.								
Pgh. bed.....	14879	3.3	6.4	36.2	13619	13799	180	1.3
Pgh. bed.....	14855	2.7	5.6	37.2	13820	13829	9	0.1
Pgh. bed.....	14849	1.9	5.1	37.0	14008	14159	151	1.1
Pgh. bed.....	13389	2.4	4.7	36.7	14058	13999	—59	—0.4
Pgh. bed.....	12579	2.7	14.9	34.9	12325	12323	—2	0
Pgh. bed.....	12578	3.4	5.3	35.9	13734	13777	43	0.3
Pgh. bed.....	12577	3.2	8.5	34.6	13279	13322	43	0.3
Pgh. bed.....	12576	2.9	5.6	37.0	13817	13921	104	0.8
Pgh. bed.....	12574	3.0	5.4	37.7	13806	13925	119	0.8
Pgh. bed.....	12573	3.1	5.8	36.8	13738	13705	—33	—0.3
Pgh. bed.....	12572	2.9	5.7	36.2	13761	13889	128	0.9
Westmoreland Co.								
Pgh. bed.....	12853	2.5	10.4	30.3	13365	13450	85	0.6
West Virginia								
Marshall Co.								
Pgh. bed.....	14488	3.4	7.0	41.6	13313	13279	—34	—0.3
Kentucky								
Bed No. 9....	13260	3.1	7.6	37.8	12208	12191	—7	—0.1

TABLE II—(*Continued*)

Description	Lab. no.	Proximate analysis as received: Moisture Per cent.	Ash Per cent.	Volatile matter Per cent.	Calorific value as received: Determined B. t. u.	Calculated B. t. u.	Difference B. t. u.	Error in calculated calorific value Per cent.
Tennessee								
Anderson Co.	13290	1.9	8.6	35.0	13777	13862	85	0.6
Anderson Co.	13265	1.5	9.2	35.9	13615	13534	—81	—0.6
Anderson Co.	13264	1.7	10.1	35.0	13462	13559	97	0.7
Anderson Co.	13238	1.9	11.5	33.0	13151	13250	99	0.7
Anderson Co.	13237	1.6	6.6	35.4	14047	14011	—36	—0.3
Alabama								
Shelby Co....	13466	3.9	7.5	35.1	13343	13370	27	0.2
Shelby Co....	13465	5.8	6.5	34.0	13298	13408	110	0.8
Shelby Co....	13464	2.6	4.9	35.4	14202	14067	—135	—0.9
Shelby Co....	13463	2.1	4.0	36.5	14447	14225	—222	—1.5
Walker Co....	13462	3.8	9.4	37.7	12895	12841	—54	—0.4
Walker Co....	13461	3.0	9.1	39.7	13118	13279	161	1.2
Walker Co....	13460	4.6	13.2	35.9	12056	12204	148	1.2
Walker Co....	13459	3.7	9.3	37.9	12992	12946	—46	—0.4
Walker Co....	13187	1.1	16.1	26.4	12793	12825	32	0.2
Walker Co....	13186	1.0	4.9	29.8	14647	14742	95	0.7
Walker Co....	13185	2.0	5.0	35.5	13997	13968	—19	—0.1
Mean algebraic error in 30 samples.....								0.2
Maximum negative error in 30 samples.....								1.5
Maximum positive error in 30 samples.....								1.3
Semi-bituminous coal, Appalachian field								
Pennsylvania								
Cambria Co.								
"B" bed.....	14243	2.7	4.6	18.1	14560	14645	85	0.6
"B" bed.....	12377	0.8	6.7	20.4	14549	14537	—12	—0.1
"B" bed.....	12376	0.8	9.4	21.4	14022	14207	185	1.3
"B" bed.....	12375	0.8	6.3	20.8	14531	14683	152	1.0
"B" bed.....	12374	0.8	6.1	20.0	14605	14688	83	0.6
Indiana Co.								
"E" bed.....	12373	1.0	9.1	26.1	13945	14038	93	0.7
Cambria Co.								
"B" bed.....	12372	0.8	7.2	22.9	14443	14575	132	0.9
"B" bed.....	12371	0.8	9.1	23.9	14040	14116	76	0.5
"B" bed.....	12370	0.9	6.7	23.1	14485	14575	90	0.6
"C" bed.....	12369	0.9	8.9	18.4	14081	14157	76	0.5
Clearfield Co.								
"B" bed.....	12368	0.8	10.9	20.4	13734	13876	42	0.3
"B" bed.....	12367	0.9	9.0	21.6	14060	14198	138	1.0
West Virginia								
McDowell Co.								
Pocahontas bed	14424	2.0	6.8	17.8	14324	14504	180	1.3
Pocahontas bed	14420	1.6	5.9	18.0	14508	14598	90	0.6
Pocahontas bed	14399	2.8	4.6	17.7	14573	14663	90	0.6
Pocahontas bed	14282	4.2	3.2	18.6	14629	14551	—78	—0.5
Pocahontas bed	14276	3.8	3.2	18.3	14690	14791	101	0.7
Mercer Co.								
Pocahontas.....	14260	3.1	3.9	18.2	14688	14823	135	0.9
Pocahontas.....	14237	3.2	3.7	17.0	14729	14848	19	0.1
McDowell Co.								
Beckley bed....	14472	2.2	8.9	17.3	13952	14058	106	0.8
Beckley bed....	14430	2.6	8.2	17.2	14006	14137	131	0.9
Beckley bed....	14408	2.2	8.4	16.4	14006	14017	11	0.1
Beckley bed....	14269	2.3	11.1	16.8	13514	13727	213	1.6
Raleigh Co.								
Beckley bed....	14291	3.3	4.3	19.0	14519	14627	108	0.7
Beckley bed....	14309	2.2	3.5	17.2	14738	14855	117	0.8
Beckley bed....	14317	3.3	3.3	17.4	14704	14839	135	0.9
Beckley bed....	14318	3.7	2.9	17.2	14668	14816	148	1.0
Fayette Co.								
Sewell bed.....	14404	3.0	3.1	25.7	14605	14717	112	0.8
Sewell bed.....	14362	3.0	3.8	28.0	14423	14512	89	0.6
Sewell bed.....	14345	2.9	2.3	25.6	14796	14854	58	0.4
Mean algebraic error in 30 samples.....								0.7
Maximum negative error in 30 samples.....								1.6
Maximum positive error in 30 samples.....								0.5
Anthracite								
Pennsylvania...	14765	4.0	12.2	8.9	12591	12555	—36	—0.3
Pennsylvania...	14764	3.9	14.7	8.5	12179	12231	52	0.4
Pennsylvania...	14763	4.9	15.4	9.5	11954	12047	93	0.8
Pennsylvania...	14762	3.2	16.4	8.0	11939	12020	81	0.7
Pennsylvania...	14761	3.2	11.5	9.3	12971	13043	72	0.6

TABLE II—(*Continued*)

Description	Lab. no.	Proximate analysis as received: Moisture Per cent.	Ash Per cent.	Volatile matter Per cent.	Calorific value as received: Determined B. t. u.	Calculated B. t. u.	Difference B. t. u.	Error in calculated calorific value Per cent.
Pennsylvania...	14760	1.6	19.7	12.2	11929	12001	72	0.6
Pennsylvania...	14759	0.9	18.8	12.1	12227	12416	189	1.5
Pennsylvania...	14758	3.8	18.4	7.1	12333	11392	59	0.5
Pennsylvania...	14757	1.7	17.6	10.7	12136	12238	102	0.8
Pennsylvania...	14720	2.0	17.9	8.1	11875	11920	47	0.4
Pennsylvania...	14719	2.7	19.7	8.4	11578	11662	84	0.7
Pennsylvania...	14718	3.1	16.2	8.9	11916	11990	74	0.6
Pennsylvania...	14520	3.0	15.1	6.4	12038	12125	87	0.7
Pennsylvania...	14209	3.8	17.1	8.2	11781	11777	—4	0
Pennsylvania...	14208	3.6	14.4	7.2	12137	12222	85	0.7
Pennsylvania								
Sullivan Co...	9664	3.6	11.9	9.1	13072	13212	140	1.1
Sullivan Co...	9665	3.4	11.5	8.5	13156	13273	117	0.9
Sullivan Co...	9652	3.4	11.7	9.3	13120	13221	101	0.8
Sullivan Co...	9653	3.7	13.1	9.2	12836	12870	34	0.3
Sullivan Co...	9654	3.4	15.6	8.4	12497	12569	72	0.6
Mean algebraic error in 20 samples.....								0.6
Maximum negative error in 20 samples.....								0.3
Maximum positive error in 20 samples.....								1.5

A total of 176 analyses were selected at random from the records of the chemical laboratory without any attempt whatever towards selecting concordant or diverging results, but simply taking them from the laboratory record as they happened to occur. Enough analyses were selected to secure about 20 to 30 samples of each representative class of coal from the most recent lignites, through the range of sub-bituminous, bituminous, semi-bituminous and anthracite coals of Pennsylvania. For comparison a number of peats are also included.

The tabulation brings out in a striking manner the variation in the relation between the heating value calculated by Dulong's formula and the true heating value as determined in the bomb calorimeter. This relation is within certain limits, characteristic of the class of coal. For instance, in coals low in volatile matter and oxygen, like anthracite and Pocahontas coal, the calculated value is usually from 0.3 to 1.5 per cent. greater than the determined value. In only very few instances is the calculated value less than the determined value; in the bituminous coals the calculated values vary both above and below the determined values usually within limits of 1.5 per cent.; in sub-

TABLE III—A SUMMARY OF THE MEAN ALGEBRAIC, MAXIMUM POSITIVE, AND MAXIMUM NEGATIVE ERRORS, IN THE CALORIFIC VALUES CALCULATED BY DULONG'S FORMULA, AS FOUND IN GROUPS OF 20 TO 30 REPRESENTATIVE ANALYSES OF EACH CLASS OF COAL

Class of coal	Error in calculated calorific value: Mean algebraic error Per cent.	Maximum plus error Per cent.	Maximum minus error Per cent.	Oxygen in coal, Moisture-and-ash-free Per cent.
Peat.....	—7.4	...	11.0	34.4
Lignite, Dakotas, Mont. & Texas..	—3.1	...	5.4	23.4
Sub-bituminous, Rocky Mt. field..	—1.4	0.9	3.8	19.1
Bituminous, Rocky Mt. field.....	—0.3	1.0	1.6	9.8
Bituminous, Illinois field.....	—0.1	1.7	1.4	10.7
Bituminous, Appalachian field....	+0.2	1.3	1.5	7.1
Semi-bituminous, Pa. and W. Va..	+0.7	1.6	0.5	3.1
Anthracite, Pennsylvania.....	+0.6	1.5	0.3	3.1

bituminous and lignitic coals the calculated value becomes less and less than the determined value as

the oxygen increases, the deviation often reaching 4 and 5 per cent.; in peat the calculated results are quite unreliable, the results being from 3 to 11 per cent. low.

A summary of Table II, giving the maximum, minimum and mean error in the calculated values, together with the average moisture-and-ash-free oxygen for each class of coal, is given in Table III.

The progressive change in the Dulong values, with the increase in oxygen, is significant and points to a combination of part of the oxygen with carbon in highly oxygenated coals. Porter and Ovitz[1] have already arrived at this conclusion from a consideration of data obtained in their studies of the volatile matter obtained by destructive distillation.

CALCULATION OF CALORIFIC VALUE FROM THE PROXIMATE ANALYSES

The calculation of calorific values by formulae depending on the proximate analysis is apt to give quite erroneous results. Such methods are subject to the considerable uncertainty of the fixed carbon and volatile matter determinations. Furthermore, the variable proportion of inert volatile matter, which does not contribute to the heating value, is not taken into consideration.

Use of "Moisture-and-Ash-Free" Values.—A fairly accurate and simple method of calculating the heating value of Appalachian, bituminous, and semi-bituminous coal from the same mine and seam is based on the constancy in heating value of the "combustible" or coal substance free of moisture and ash.

The calorific value of a coal may be referred to a moisture-and-ash-free basis by dividing the calorific value as determined by one minus the sum of the moisture and ash in the unit weight of fuel.

$$\text{Moisture-and-ash-free calorific value} = \frac{\text{Calorific value as determined}}{1 - (\text{moisture} + \text{ash})}$$

This so-called moisture-and-ash-free calorific value, which represents approximately the heating power of the combustible in the coal, has been found fairly constant for the same coal bed over limited areas, such as are covered by several adjacent mines.

TABLE IV—VARIATIONS IN "MOISTURE-AND-ASH-FREE" CALORIFIC VALUES OF MINE SAMPLES FROM THE SAME MINE

Locality and coal bed	"Moisture-and-ash-free" calorific values of individual samples B. t. u.	Average B. t. u.	Maximum difference of individual samples B.t.u.	Maximum difference of individual samples Per cent.	Maximum deviation from average B.t.u.	Maximum deviation from average Per cent.
5 samples from a mine near Figert, Pa., in Cambria Co., Middle Kittanning bed	15600 15600 15640 15610 15650	15620	50	0.3	30	0.2
5 samples from a mine near Barnesboro, Pa., in Cambria Co., Lower Kittanning or "B" bed	15710 15670 15830 15760 15660	15726	170	1.1	104	0.7
5 samples from a mine near Carrolton, Pa., in Cambria Co., Lower Kittanning or "B" bed	15670 15660 15740 15670 15820	15712	160	1.1	108	0.7
27 samples from mines near Twin Rocks, Pa., in Cambria Co., Lower Kittanning or "B" bed	15820 15730 15890 15780 15730 15710 15770 15820 15700 15800 15690 15700 15740 15770 15840 15720 15760 15740 15740 15760 15790 15770 15840 15750 75780 15830 15820	15770	200	1.3	120	0.8
10 samples from two adjacent mines near Clymer, Pa., in Indiana Co., Lower Kittanning or "B" bed	15680 15700 15750 15610 15710 15740 15740 15650 15710 15700	15700	140	0.9	90	0.6
5 samples from a mine near Beaverdale, Pa., Cambria Co., Lower Kittanning or "B" bed	15741 15768 15721 15772 15759	15752	51	0.3	31	0.2
5 samples from a mine near El Moro, Pa., in Cambria Co., Lower Kittanning or "B" bed	15763 15862 15611 15772 15887	15779	276	1.8	169	1.1
4 samples from a mine near Masontown, Preston Co., W. Va., Upper Freeport bed	15490 15470 15380 15460	15450	110	0.8	70	0.5
5 samples from a mine near Richard, Monongalia Co., W. Va., Upper Freeport bed	15530 15500 15470 15400 15510	15482	130	0.9	82	0.4
6 samples from a mine near Barnesboro, Pa., Cambria Co., Lower Freeport bed	15600 15570 15600 15660 15640 15650	15620	90	0.6	40	0.3
9 samples from a mine near Spangler, Pa., Cambria Co., Lower Freeport bed	15671 15651 15644 15656 15727 15678 15623 15705 15700	15662	204	1.3	139	0.9
10 samples from a mine near Hastings, Pa., Cambria Co., Lower Freeport bed	15698 15678 15646 15588 15604 15590 15655 15638 15608 15647	15635	110	0.8	63	0.4
4 samples from a mine near Dante, Russell Co., Va., Upper Banner bed	15426 15521 15408 15476	15458	113	0.8	63	0.4

[1] H. C. Porter and F. K. Ovitz, "The Volatile Matter of Coal," Bureau of Mines, *Bull.* 1, 1910.

TABLE IV—(*Continued*)

Locality and coal bed	"Moisture-and-ash-free" calorific values of individual samples B. t. u.	Average B. t. u.	Maximum difference of individual samples B.t.u.	Maximum difference of individual samples Per cent.	Maximum deviation from average B.t.u.	Maximum deviation from average Per cent.
23 samples from the government experimental mine near Bruceton, Pa., Allegheny Co., Pittsburgh bed	15140 15190 15170 15110 15160 14960 14930 15030 15070 15080 15040 15040 15070 15140 15070 15080 15140 15140 15140 15100 15060 15050 15080	15087	260	1.7	157	1.0
Car samples from 27 cars of coal delivered to testing station from Dec., 1910, to May, 1911, from a mine near Meadowlands, Washington Co., Pa., Pittsburg bed	14800 14910 14890 15040 14760 14950 14880 14880 14840 14900 14870 14860 14800 14830 14920 14800 14900 14810 14990 14840 14880 14880 14910 14850 14950 14840 14900	14874	280	2.0	166	1.1
Car samples from 4 cars of coal delivered to testing station from Dec. 19, 1910, to Jan., 1911, from another mine near Meadowlands, Washington Co., Pa., Pittsburgh bed	14560 14640 14640 14630	14618	80	0.6	58	0.4
Car samples from 7 cars of coal delivered to testing station from May, 1911, to July, 1911, from a mine near Burgettstown, Washington Co., Pa., Pittsburgh bed	14750 14680 14690 14790 14650 14750 14890	14743	240	1.6	147	1.0
Car samples from 44 cars of coal delivered to testing station from Aug., 1911, to April, 1912, from a mine near Arnold, Westmoreland Co., Pa., Upper Freeport bed	15180 15250 15180 15200 15190 15260 15240 15280 15190 14250 15300 15220 15170 15210 15290 15210 15220 15220 15180 15240 15100 15180 15220 15250 15190 15180 15220 15190 15220 15210 15300 15250 15290 15250 15280 15300 15100 15270 15230 15250 15230 15280 15250 15220	15226	200	1.3	126	0.8
Car samples from 5 cars of coal delivered to testing station from Nov., 1911, to June, 1912, from a mine near Monongahela City, Westmoreland Co., Pa., Pittsburgh bed	15280 15140 15150 15210 15150	15186	140	0.9	94	0.6
Car samples from 3 cars of coal delivered to testing station from Nov., 1911, to May, 1912, from a mine near Greensburg, Westmoreland Co., Pa., Pittsburgh bed	15350 15310 15400	15353	90	0.6	47	0.3

In Table IV are given some "moisture-and-ash-free" calorific values of mine samples taken by engineers of the bureau at different points in a number of mines in Pennsylvania. They are typical in showing the usual range of variation that is found in samples from different parts of a mine.

In the latter part of Table IV is given some data on the variation in "moisture-and-ash-free" calorific values that have been found in car samples of coal delivered to the power plant of the Pittsburgh Experiment Station for fuel purposes. While each set of cars grouped together is supposed to have come from the same mine, they were not loaded under the inspection of a representative of the bureau; also, the coal may have had more or less opportunity for weathering in transit to the testing station. This would explain the slightly larger variations in the car samples.

The greatest deviation in "moisture-and-ash-free" calorific value of any one individual sample from the average of the mine is usually less than one per cent. In only two cases does it reach the maximum figure of 1.1 per cent. Obviously the calorific value of samples representing various shipments of coal from the same mine and seam can be calculated with an accuracy of about 1 per cent. by multiplying the average "moisture-and-ash-free" calorific value of the coal by 1 minus the sum of the moisture and ash of the various individual samples. This method of calculating will not apply to weathered coals which on account of absorption of oxygen have lost some of their original heating value, or to coals that vary considerably in the character and amount of ash and sulfur. In the coals cited in Table IV the ash is usually less than 12 per cent. and the sulfur less than 2.0 per cent.

Where the sulfur content is larger and more variable, as in some of the coals of Ohio[1] and Illinois,[2] a further correction for the influence of sulfur should be applied.

SAMPLING

The chemist makes his analysis on a few ounces of powdered coal. If the analysis is to be of any value, this small sample must have a composition that is an average of the entire lot of coal whose value is to be established. Non-representative samples are misleading and frequently lead to much controversy as well as financial loss. Therefore, sampling should never be placed in inexperienced hands. It is just as important for the chemist to devise and direct the method of taking samples as it is for him to make the analyses. The sampling of shipments and deliveries of coal involves special difficulties on account of the usual admixture of pieces of slate, bone and other impurities, with the lumps of coal. The mechanical methods which have been found satisfactory in sampling ore can not be used for the ordinary sizes of coal on account of destroying the value of the fuel by the necessary crushing. The accuracy of sampling the commercial sizes of coal is limited to the amount

[1] N. W. Lord, "Coal," Geological Survey of Ohio, *Bull.* **9**, 4th Lines, 268 (1908).

[2] S. W. Parr, Illinois State Geological Survey, *Bull.* **16**, 212 (1909).

and size of the largest lumps of impurities and the quantity of original sample that can be taken without incurring too great a cost. The accuracy of any method of sampling should be frequently tested by taking two or more independent samples which are reduced and analyzed separately. Ordinarily the sample sent to the laboratory will weigh from three to five pounds, and will be pulverized to about $^1/_4$ inch size. The original gross sample which may amount to 500 or 1000 pounds must be pulverized, mixed and quartered in such a manner as to maintain the same relative proportions of coal and impurities until the final 3- to 5-pound sample is obtained; furthermore, coal has a marked tendency to lose moisture in crushing. This must be avoided as much as possible.

The final sample should be placed in a moisture-proof container for shipment to the laboratory. Wooden boxes or sacks should never be used for samples in which moisture is to be determined.

ACCURACY OF MINE SAMPLING

During the past nine years the Technologic Branch of the U. S. Geological Survey and its successor, the Bureau of Mines, has analyzed a large number of mine samples of coal, taken in connection with the investigation of coal as it occurs in the mine. These samples[1] are taken by cutting down a section or groove of uniform cross section across the face of the seam. The gross sample, amounting to about six pounds for each foot in thickness of the coal bed, is pulverized to pass a $^1/_2$ inch screen, mixed and quartered down to about three pounds, which is placed in a galvanized iron can, sealed moisture-tight, and mailed to the laboratory.

In taking these samples some uniform system of including and rejecting the partings or bands of impurities that occur in coal beds is essential. Usually partings more than $^3/_8$ of an inch thick and lenses or concretions of "sulfur" or other impurities more than two inches in maximum diameter and $^1/_2$ of an inch thick are excluded. By following such a system the analytical results are comparable in representing the quality of coal in different coal beds; it is quite necessary to complete the sampling and seal the sample can at the face as quickly as possible after breaking down the coal in order to avoid loss of moisture.

These mine samples usually represent a better quality of coal than is ordinarily shipped from the mine, as in the operation of a mine it is not practicable to use the same care in excluding partings and other impurities. They are of value in showing the quality of coal as it occurs in the seam, at the point of sampling; and the results are subject to the unavoidable variations due to including more or less of the partings and the errors in reducing the gross sample to three pounds. It is possible to practically eliminate the errors of reducing the gross sample by crushing the entire quantity of coal cut down from the face, to less than 4 mesh size, and then mixing and quartering to 3 pounds. However, such a procedure is of doubtful value on account of the greater errors resulting from the inclusion of more or less of the partings in cutting down the section, especially where they tend to crumble from the action of the pick.

There is also to be considered the variation in the proportion of impurities in neighboring sections; the extent of this variation is usually greater than the errors incurred in sampling the individual section.

In Table V are shown the results of a series of duplicate 3-pound can samples that were taken by the standard method of the Bureau of Mines as described in *Technical Paper* 1.[1]

The entire sample which had been cut from the face was crushed to pass a $^1/_2$ inch screen, mixed and quartered to three pounds, which was called sample No. 1; the rejected portions were then combined, mixed and again quartered to three pounds, which was called sample No. 2. Both samples were sent to the laboratory in the usual galvanized iron sample cans, which are closed with a screw cap and made moisture-tight by wrapping the joint with electricians' tape. The samples were air dried, pulverized in the usual manner by rolls and ball mill and separately analyzed. The results obtained for total moisture, ash, and sulfur are given in the table.

TABLE V—A COMPARISON OF DUPLICATE 3-LB. SAMPLES TAKEN FROM THE SAME SECTION OF COAL

Location of mine	Total moisture			Ash			Sulfur		
	Sample 1 Per cent.	Sample 2 Per cent.	Difference Per cent.	Sample 1 Per cent.	Sample 2 Per cent.	Difference Per cent.	Sample 1 Per cent.	Sample 2 Per cent.	Difference Per cent.
Danville, Vermilion Co., Ill.									
Section A	12.4	12.4	0.0	7.8	7.9	0.1	2.9	3.0	0.1
Section B	13.1	13.2	0.1	10.0	9.9	0.1	4.0	3.5	0.5
Composite	12.8	12.7	0.1	8.8	8.8	0.0	3.5	3.2	0.3
Westville, Vermilion Co., Ill.									
Section A	13.3	13.2	0.1	8.5	9.2	0.7	2.3	2.4	0.1
Section B	15.3	15.5	0.2	7.9	8.0	0.1	1.6	1.7	0.1
Section C	16.2	16.2	0.0	8.2	8.3	0.1	1.8	1.8	0.0
Section D	14.8	14.9	0.1	8.7	8.5	0.2	1.9	2.0	0.1
Section E	15.5	15.4	0.1	7.1	8.7	1.6	1.5	2.8	1.3
Section F	15.2	15.1	0.1	8.6	8.7	0.1	2.9	2.9	0.0
Composite	15.1	15.1	0.0	8.2	8.5	0.3	2.1	2.3	0.1
Georgetown, Vermilion Co., Ill.									
Section A	15.2	15.1	0.1	9.8	9.7	0.1	2.0	2.1	0.1
Section B	15.4	15.5	0.1	11.2	10.9	0.3	1.9	1.9	0.0
Section C	15.9	15.7	0.2	11.6	12.2	0.6	2.4	2.6	0.2
Composite	15.5	15.5	0.0	10.9	11.0	0.1	2.1	2.2	0.1
Danville, Vermilion Co., Ill.									
Section A	13.1	13.0	0.1	10.0	9.8	0.2	3.2	3.1	0.1
Section B	12.9	12.8	0.1	9.3	9.3	0.0	3.7	3.5	0.2
Section C	12.7	12.7	0.0	9.7	9.6	0.1	3.7	3.4	0.3
Section D	12.6	12.5	0.1	9.1	8.8	0.3	3.1	2.9	0.2
Section E	13.9	13.8	0.1	9.1	9.7	0.6	3.1	2.8	0.3
Section F	13.8	13.8	0.0	8.5	8.5	0.0	2.9	3.0	0.1
Composite	13.0	13.1	0.1	9.3	9.2	0.1	3.2	3.2	0.0
Fairmont, Vermilion Co., Ill.									
Section A	14.2	14.1	0.1	9.6	9.9	0.3	2.2	2.3	0.1
Section B	13.6	13.5	0.1	9.1	9.8	0.7	2.1	2.3	0.2
Section C	13.4	13.1	0.3	11.3	11.4	0.1	2.6	2.8	0.2
Composite	13.7	13.6	0.1	10.0	10.4	0.4	2.3	2.4	0.1
Steelton, Vermilion Co., Ill.									
Section A	15.9	15.8	0.1	10.7	10.3	0.4	2.4	2.6	0.2
Section B	14.2	14.2	0.0	9.9	10.0	0.1	2.2	2.5	0.3
Section C	14.9	14.6	0.3	8.5	8.7	0.2	2.3	2.5	0.2
Composite	15.0	14.8	0.2	9.8	9.7	0.1	2.3	2.5	0.2

[1] Method of taking mine samples is described in detail in Bureau of Mines, *Technical Paper* 1, "Sampling Coal in the Mine," by J. A. Holmes.

[1] J. A. Holmes, "Sampling Coal in the Mine," Bureau of Mines, *Technical Paper* 1.

TABLE V—(*Continued*)

Location of mine	Total moisture Sample 1 Per cent.	Total moisture Sample 2 Per cent.	Total moisture Difference Per cent.	Ash Sample 1 Per cent.	Ash Sample 2 Per cent.	Ash Difference Per cent	Sulfur Sample 1 Per cent.	Sulfur Sample 2 Per cent.	Sulfur Difference Per cent.
DuQuoin, Perry Co., Ill.									
Section A	10.4	10.7	0.3	9.6	9.0	0.4	0.8	0.8	0.0
Section B	11.5	11.6	0.1	8.7	9.0	0.3	0.8	0.9	0.1
Section C	10.7	10.9	0.2	10.6	10.8	0.2	0.9	1.0	0.1
Section D	10.5	10.6	0.1	12.2	11.7	0.5	1.0	0.9	0.1
Section E	10.1	10.3	0.2	11.6	11.8	0.2	0.9	0.8	0.1
Section F	10.8	10.7	0.1	11.0	11.5	0.5	0.8	0.8	0.0
Composite	10.7	10.7	0.0	10.7	10.6	0.1	0.9	0.9	0.0
Bruceton, Allegheny Co., Pa., Pittsburgh bed									
Section A	2.9	2.8	0.1	5.7	5.6	0.1	1.1	1.1	0.0
Section B	3.0	3.0	0.0	6.0	5.8	0.2	1.2	1.2	0.0
Section C	2.8	2.7	0.1	5.1	5.2	0.1	1.0	1.1	0.1
Section D	2.7	2.7	0.0	5.5	5.7	0.2	1.2	1.2	0.0
Composite	2.8	2.8	0.0	5.6	5.6	0.0	1.1	1.1	0.0
Colver, Cambria Co., Pa., Miller or "B" Bed									
Section A	..	..	...	5.4	5.2	0.2	1.0	1.0	0.0
Section B	..	..	...	4.9	4.7	0.2	0.9	0.9	0.0
Section C	..	..	...	5.1	5.2	0.1	1.0	1.0	0.1
Composite	..	..	...	5.1	5.0	0.1	1.0	1.0	0.0

	Moisture Per cent.	Ash Per cent.
Maximum difference in duplicate mine samples taken in Illinois	0.3	1.6(*a*)
Maximum difference in samples taken in Pennsylvania	0.1	0.2
Average difference in samples taken in Illinois	0.1	0.3
Average difference in samples taken in Pennsylvania	0.05	0.16

(*a*) Next highest difference in Illinois samples was 0.7.

On account of the high percentages of moisture, ash and sulfur and the irregular distribution of the impurities in the seam, Illinois coals are unusually difficult to sample. Yet in spite of the high moisture content, the greatest difference of moisture in duplicate samples was only 0.3 per cent., with an average difference for the 30 sections of 0.1 per cent.

In the four sections in the experimental mine the maximum difference was 0.1 per cent. and the average difference was 0.05 per cent.

The average variation in ash of the duplicate samples from seven sections in two Pennsylvania mines is 0.1 per cent.; the maximum variation being 0.2 per cent.

In the Illinois series the average ash variation is 0.3 per cent. and the maximum variation is 1.6 per cent.; the next highest difference is 0.7 per cent.

In an investigation of the quality of coal in the mine several samples should be taken at different places. These may be separately analyzed or combined at the laboratory into a composite sample. The composite analysis of several samples is subject to smaller sampling errors than the individual samples. For example, in Table V the largest ash variation in the composite analyses of samples Nos. 1 and 2 is 0.4 per cent., although several individual pairs of duplicate samples showed 0.7 per cent. difference and one pair out of the 36 had a difference of 1.6 per cent.

SAMPLING IN THE LABORATORY

Next in importance to securing a representative sample to send to the laboratory, is the further treatment in the laboratory sampling room by which a representative powdered sample is obtained for analysis.

In the method used by the Bureau of Mines, moisture loss is avoided by air drying the 3-pound sample before pulverizing. The amount of moisture expelled during the air-drying process is recorded and added to the final moisture obtained by drying the powdered sample at 105° C. (221° F.). The air-dry sample is quickly pulverized to 10 mesh by passing through a roll crusher, reduced on a riffle sampler to about 450 grams (1 pound) and ground to 60 mesh in a moisture-tight ball mill which consists of rotating porcelain jars containing well-rounded flint pebbles. The final powdered sample is preserved in a rubber-stoppered bottle to prevent change in moisture content.

This method of sampling was developed by Lord and Somermeier at the U. S. Fuel Testing Plant at St. Louis. Numerous experiments have shown that this method reduces the unaccounted for moisture changes during sampling and analysis to a lower point than any other practicable method that has yet been devised. The very uniform moisture results obtained on the duplicate mine samples of the high moisture Illinois coals cited in Table V could not have been obtained by the old methods of pulverizing on a bucking board where the exposure of finely divided coal to a dry or humid atmosphere could change its moisture content.

Usually the laboratory sampling error with respect to ash, by the method just described, is less than 0.3 per cent. and seldom greater than 0.5 per cent. Where careful work is done by the sampler the 0.5 per cent. limit should not be exceeded. In Table VI are given some results of duplicate sampling, as illustrating the usual errors.

TABLE VI—A COMPARISON OF MOISTURE AND ASH IN DUPLICATE 3 OZ. POWDERED SAMPLES MADE FROM THE 3-LB. SAMPLES RECEIVED AT THE LABORATORY

Laboratory No.	Per cent. moisture in air-dry coal Sample A	Sample B	Difference	Per cent. ash in air-dry coal Sample A	Sample B	Difference
14807	1.95	2.02	0.07	12.93	12.78	0.15
14808	2.11	2.09	0.02	12.52	12.53	0.01
14809	6.90	6.81	0.09	13.40	13.12	0.28
14812	1.89	1.91	0.02	12.55	12.70	0.15
14813	0.92	0.92	0.00	6.10	6.36	0.26
14814	0.66	0.69	0.03	8.45	8.32	0.13
14815	3.57	3.67	0.10	16.64	16.27	0.37
14817	1.14	1.22	0.08	15.36	15.49	0.13
14818	1.63	1.65	0.02	10.63	10.47	0.16
14819	1.08	1.19	0.11	9.00	8.83	0.17
Average difference			0.05			0.18
Maximum diff. in 10 samples			0.11			0.37

PREPARATION OF LABORATORY SAMPLE WITHOUT PRELIMINARY AIR DRYING

Where the highest degree of accuracy with respect to moisture is not required and where the analyses must be had in the shortest possible time, the method used in the inspection laboratory of the Bureau of

Mines will give satisfactory results.[1] This method consists in rapidly crushing the coarse sample to 20 mesh by means of a roll crusher or coffee mill type of grinder and taking a representative portion of this product for the determination of total moisture. The remainder and main portion of the sample is then mixed and divided with a riffle sampler to not less than 150 grams, which is then pulverized to 60 mesh with any suitable apparatus, such as a bucking board or disc pulverizer, without regard to moisture losses. Both samples are preserved in rubber-stoppered bottles.

The moisture determined in the 20 mesh sample represents the total moisture in the coal as received; the moisture in the final powdered sample is also determined in order to recalculate the analysis to the "as received" condition.

The results for moisture obtained by this method are somewhat low, due to unavoidable loss while crushing the coal to 20 mesh size. Wet coals must be partially air-dried and the loss of weight noted.

INCORRECT METHODS OF PREPARING COAL SAMPLES

The determination of total moisture by drying a portion of the coarse sample in a steam oven at about 105° C. (221° F.) will give low results, the error increasing with the size of the lumps.

The method of drying the coarse sample at temperatures above 100° C. (212° F.) and then pulverizing to a powdered sample for analysis, with the assumption that the powdered coal is dry coal, will give quite erroneous results. The powdered sample will never be dry but will contain from 0.3 to 1.0 per cent. moisture which it has either failed to give up while drying in the coarse condition, or has taken up from the atmosphere, while being pulverized. The heating values are directly effected by the errors in the sampling and in the moisture determinations.

ABRASION IN BALL MILLS

In using any apparatus for pulverizing samples the amount of contamination from abrasion of the pulverizing surfaces should always be investigated. Before the U. S. Fuel Testing Plant at St. Louis adopted the ball mill for the final grinding of coal samples, tests were made to determine whether there was danger of materially increasing the ash content of the samples from chipping and abrasion of the flint pebbles used in grinding. In these tests the weight of the sample ground each time on the ball mill was approximately 500 grams, and the abrasion of the pebbles (calculated as percentage of the weight of the samples ground) was determined from the loss in weight sustained by the pebbles. The results on these weighed lots of pebbles are shown in Table VII.

TABLE VII—LOSS(a) IN WEIGHT OF THREE LOTS OF FLINT PEBBLES USED FOR PULVERIZING COAL IN THE BALL MILL

Lot	Total weight of samples ground Grams	Weight of pebbles before grinding Grams	Weight of pebbles after grinding Grams	Loss by abrasion actual Grams	Loss by abrasion Ratio to coal ground Per cent.
No. 1 (250 samples ground)	125,000	4118.6	4113.1	5.5	0.004
No. 2 (230 samples ground)	115,000	3502.9	3499.4	3.5	0.003
No. 3 (245 samples ground)	122,500	4273.2	4268.3	4.9	0.004

(a) "Experimental Work of Chemical Laboratory, U. S. Fuel Testing Plant, St. Louis, Mo.," by N. W. Lord, U. S. G. S., *Bull.* **323**, 8.

The results show only 0.004 per cent. increase in ash due to the abrasion from the pebbles.

In order to determine the extent of abrasion from the porcelain jar, the following tests were made:

TABLE VIII—LOSS IN WEIGHT OF PORCELAIN JARS USED FOR PULVERIZING COAL IN THE BALL MILL

	Total weight of sample ground Grams	Weight of jar before grinding Grams	Weight of jar after grinding Grams	Loss by abrasion actual Grams	Loss by abrasion Ratio to coal ground Per cent
Jar No. 35 (100 samples of 150 grams each ground)...	15000	8095	8085	10.0	0.066
Jar No. 37 (100 samples of 150 grams each ground)...	15000	8317	8314	3.0	0.020
Jar No. 35 (100 samples of 300 grams each ground)...	30000	8085	8075	10.0	0.033
Jar No. 37 (100 samples of 300 grams each ground)...	30000	8314	8310	4.0	0.013
Jar No. 36 (30 samples of 300 grams each ground).....	9000	10789	10788	1.0	0.011

Evidently there is a little more abrasion from the porcelain jars than from the pebbles. In the experiments the same amount of material was abraded in 150-gram samples as in 300-gram samples, hence the necessity of using large samples: 500 grams is the best amount and should not cause the ash content to be raised more than 0.04 per cent. at the highest, which is less than the analytical error in the determination of ash.

A more direct test of added ash from abrasion was made by placing 400 grams of ordinary granulated sugar in the mill and rotating it continuously for four hours. Before grinding, the sugar contained 0.04 per cent. ash; after grinding, it contained 0.06 per cent. ash—an increase of 0.02 per cent.

That the ball mill is the most satisfactory appliance for pulverizing coke is shown by the following experiments: Ten samples of a hard foundry coke weighing about 25 lbs. each were crushed to 10-mesh size by first passing through a chipmunk jaw crusher and then through a pair of steel rolls. Each sample was then mixed and divided with a riffle into three portions of about 8 lbs. each. The first portion was again put through the rolls until all of the 8 pounds passed through a 20-mesh sieve, and was then mixed and reduced with the riffle to 150 grams which was rubbed down to 60 mesh on a chilled cast iron bucking board. The second 8-pound portion of each sample was also put through the rolls again until all passed the 20-mesh sieve; was then riffled down to 150 grams and pulverized to 60 mesh in a tool steel diamond mortar. The third 8-pound portion of each sample was mixed and divided on the riffle to about 300 grams, which was then placed in the ball mill and pulverized to 60 mesh. Owing to the extreme hardness of this coke, 4 hours rotation in the ball mill was required to pulverize to 60 mesh. After removing from the ball mill the entire sample was put through the 60-mesh sieve, mixed and reduced on the riffle to about 60 grams for analysis.

All the 60-mesh samples were tested for iron with

(a) "The Fuel Inspection Laboratory of the Bureau of Mines," by J. D. Davis, Bureau of Mines, *Bull.* **41**, 74 (1912).

TABLE IX—ASH IN COKE PULVERIZED BY THREE DIFFERENT METHODS

Laboratory No.	Ash in sample pulverized with bucking board Per cent.	Ash in sample pulverized with diamond mortar Per cent.	Ash in sample pulverized with ball mill Per cent.	Difference in ash found in: bucking board and ball mill Per cent.	Difference in ash found in: diamond mortar and ball mill Per cent.
11033	12.9	11.8	11.5	1.4	0.3
11034	12.9	11.4	10.8	2.1	0.6
11035	12.8	10.3	10.5	2.3	—0.2
11036	15.2	11.3	10.8	4.4	0.5
11037	..	10.0	9.7	...	0.3
11038	..	10.7	10.7	...	0.0
11039	..	11.8	10.6	...	0.2
11040	15.4	12.3	10.9	4.5	1.4
11041	..	10.3	10.1	...	0.2
11042	..	10.2	9.7	...	0.5
			Average...	2.9	0.4

a magnet. No magnetic particles were found in the ball-mill sample and only a few in the diamond-mortar sample; the bucking-board sample showed the presence of considerable metallic iron. Ash determinations were made on each of the diamond-mortar and ball-mill samples and on some of the bucking-board samples. The results are shown in Table IX.

With only one exception the samples pulverized in the ball mill contained the least ash; the diamond mortar, which crushes by impact, ranks second. Rubbing surfaces of chilled iron and even chrome steel, such as used in the bucking board and disc pulverizer, are abraded by the hard particles of coke, and, therefore, should never be used for the fine grinding of coke.

CONCLUSIONS

To state definite limits of accuracy for coal analyses is almost impossible. The factors which influence the results are many and variable. They depend on the kind and quality of coal, the method of sampling and analysis, and the skill and experience of the analyst in the special field of coal testing.

With the exception of the volatile matter, fixed carbon and oxygen determinations, the best analytical methods are more accurate than the usual methods of sampling. The method of taking the original sample is often limited by the commercial conditions. The determination of the heating value with the use of properly standardized calorimeters and thermometers is reliable and comparable. Appreciable errors, when such occur, are generally due to sampling or a change in the composition of the sample by oxidation or loss of moisture. Finally, a statement of how the sample was taken should always go with the report of analysis. This will enable one who has a knowledge of coal to properly interpret the report of analysis.

U. S. BUREAU OF MINES
PITTSBURGH

THE EFFECT OF CERTAIN PIGMENTS ON LINSEED OIL; WITH A NOTE ON THE MANGANESE CONTENT OF RAW LINSEED OIL[1]

By E. W. BOUGHTON

Received February 19, 1913

At the time the Contracts Laboratory of the Bureau of Chemistry was making some exposure tests on painted steel plates some of the mixtures of various pigments of known composition with raw linseed oil of known analytical constants were set aside in closed jars of uniform shape and size in order to learn what changes would take place in the oil upon standing. No drier was added to these samples. The paints were so prepared that after the addition of drier they had approximately the same viscosity, 47 to 50 (water = 6.2), as determined by a Stormer viscosimeter. The jars were stoppered air-tight and placed in a closet having a glass door, where the light was dim and diffused.

The following table gives the list of pigments that were used and the proportions, by weight, of pigment and oil.

TABLE I—COMPOSITION OF PAINTS

Pigment	Pigment by weight Per cent.	Oil by weight Per cent.	Pigment	Pigment by weight Per cent.	Oil by weight Per cent.
White lead (basic carbonate).....	72	28	Artificial graphite	42	58
Kaolin (contains $CaSO_4$).....	50	50	Zinc white (American)	39	61
Indian red.....	50	50	Chrome yellow (lead chromate).....	34	66
Flake graphite.....	40	60	Chromium oxid, green	51	49
Magnetic oxid, black(*a*)	46	54	Lampblack.....	16	84
Zinc yellow (zinc chromate).....	45	55			

(*a*) Analysis by H. C. McNeil: Loss at 110° C., 0.56 per cent.; Na_2CO_3, 2.82 per cent.; $FeCO_3$, 18 per cent.; Fe_3O_4, 60.4 per cent.; Fe_2O_3, 18.3 per cent.

At the end of one- and two-year periods the contents of each jar were well mixed and a portion poured out, thinned with ether, and centrifuged. After decanting the supernatant liquid from the pigment the greater part of the ether was evaporated on the steam-bath, and the oil heated for half an hour at 105° C. in an inert gas, carbon dioxid being used for the one-year and hydrogen for the two-year samples. The last traces of pigment were removed by filtering through fine filter paper. The black pigments settled with difficulty. The oil extracted from the lampblack paint was red and muddy and was not analyzed. Judging from its constants, appearance, and odor, the linseed oil purchased in the open market was undoubtedly unadulterated. The constants were as follows:[1]

Specific gravity (15.6° C.)	0.934	Iodin number.....	179.6
Saponification number....	193.6	Ash (per cent.).....	0.13
Acid number.....	1.7	Unsaponifiable matter (per cent.).....	0.68

Unfortunately all the oil was used in making the paints, so that the changes which would have occurred in keeping it without pigments could not be noted. It has been proved, however, that raw linseed oil kept in glass away from bright sunlight for two years does not change appreciably with respect to its analytical constants, except for a slight rise in acid number.[2]

As the different pigments settled with different degrees of compactness in the jars, the surface of contact was larger in some cases than in others. Since the contents of the jars were mixed before sampling, the change in analytical figures as obtained repre-

[1] Published by permission of the Secretary of Agriculture.

[1] Analysis by E. M. Dawson.

[2] *Proc. Amer. Soc. Test. Mat.*, **11**, 197 (1911).

sents an effect upon the total volume of oil. The results are shown in the following table:

TABLE II—EFFECT OF PIGMENTS ON RAW LINSEED OIL

Pigment	Time of exposure Years	Specific gravity (15.6° C.)	Iodin number	Ash Per cent.	Color
White lead (basic carbonate)	1	0.940	175.8	0.35	Bleached
	2	0.938	177.3	0.40	"
Kaolin	1	0.939	173.0	0.12	Very much bleached
	2	0.936	171.6	0.14	
Indian red	1	0.941	173.8	0.15	Bleached
	2	0.939	172.5	0.14	"
Flake graphite	1	0.934	180.9	0.21	Normal
	2	0.933	178.2	0.15	"
Magnetic black	1	0.937	174.6	0.17	"
	2	0.935	173.2	0.13	"
Zinc yellow	1	0.934	180.2	0.20	"
	2	0.934	179.5	0.18	"
Artificial graphite	1	0.935	181.0	..	"
	2	0.939	180.8	0.15	"
Zinc white	1	0.935	181.3	0.25	"
	2	0.934	179.7	0.13	"
Chrome yellow	1	0.937	176.3	0.14	Bleached
	2	0.935	175.7	0.14	"
Chromium oxid, green	1	0.937	178.0	0.01	"
	2	0.937	180.2	0.05	"
Original oil	..	0.934	179.6	0.13	

Before commenting on the figures it may be well to discuss the general action of pigments on linseed oil. Toch[1] stated that the hydrolysis of the glycerids in linseed oil by moisture is hastened by the presence of lime or lead salts. White lead, sublimed lead, and zinc oxid all showed the same bleaching power within two weeks, irrespective of the kind of raw linseed oil used. Slow chemical reactions take place between the lead and zinc pigments and the oil. Painters sometimes prefer a white-lead paste which is old. Toch also stated that the iodin number of a pure raw linseed oil may be reduced to 110 by the addition of metallic salts, as Japan drier, but this statement does not mean that such a large reduction is caused by the reaction between pigment and oil.

Sabin[2] emphasized the strong action of litharge upon linseed oil and stated that the effect of red lead (Pb_3O_4) on oil may be due to the litharge content, as, by preparing a red lead from litharge in the form of an impalpable powder, thus securing practically complete oxidation to the higher oxids of lead, the product is without the usual action when mixed with oil. A mixture of white lead and oil dries more rapidly than oil alone, but so small is this increase that it is not recognized in practice. In the drying process the action of the inert pigments, asbestine, China clay, etc., is similar to that of white lead, which may be due to physical rather than chemical causes. In fact, no pigments are really inert to oil with respect to the drying process, and this pigment action is of great value in paint.

Klein[3] stated that linseed oil extracted from paint is frequently found to have a low iodin number and quoted Boettinger's[4] experiments which showed a reduction of the iodin number from 183.3 to 131 by white lead in 17 days, and to 122.2 in two months. Whiting and ocher showed the same changes. In the original article, however, Boettinger stated that the mixtures of oil and pigments were exposed to light and air, so that it was in reality a study of the effect of pigments on the amount of atmospheric oxidation of the oil.

Gardner[1] made mixtures of pigments and linseed oil, using very small quantities, and, after allowing them to stand, determined the increase in the amount of ash. Zinc oxid, white lead, and red lead were the only pigments that gave an appreciable increase in the amount of ash.

In a later paper[2] he gave the results of allowing linseed oil to stand in contact with different pigments for two years. Although the experiments were similar to those reported in this paper, the results were very different. The pigments that Gardner used were zinc oxid, basic carbonate of lead, combinations of lead and zinc pigments, barytes, silica, basic chromate of lead, red lead, iron oxid, carbon black, and graphite. The iodin number of the original oil was 181. The samples extracted from the pigments had iodin values varying from 135 to 163. The specific gravity was greatly raised by silica and iron oxid and slightly by most of the others. The pigments containing lead and zinc caused an increase in the amount of ash, showing actual chemical combination. Where the other constants were greatly changed the acid number of the oil was much raised. Gardner called attention to the fact that the combination of pigment and oil is due to hydrolysis with the formation of free fatty acid. The determination of this increase, therefore, is of importance as showing to what degree the glycerid has been broken up, thus increasing its tendency to combine with the pigment. In the case of the mixture of oil and silica, hydrolysis took place, as shown by the high acid number, but the pigment did not go into solution. The viscosity of the mixtures, being slightly greater than that ordinarily found, is doubtless somewhat in excess of that of the paints listed in Table I.

As Gardner stated that in two of his jars a skin was formed upon the surface, it seems probable that air entered into the reaction. This may be the cause of the difference in the results. Again, if an oil containing certain metals in solution is heated in air to drive off the last traces of solvent, oxidation will occur in a short time.

TABLE III—IODIN NUMBERS OBTAINED BY HEATING OIL UNDER DIFFERENT CONDITIONS

Oil	Gas used	Time of heating Hours	Iodin number	Oil	Gas used	Time of heating Hours	Iodin number
A	Air	2	173	C	Air	2	174
	Hydrogen	2	182		Air	4	168
	Carbon dioxid	2	180		Hydrogen	2	177
B	Air	2	158		Hydrogen	4	177
	Air	4	143		Carbon dioxid	2	178
	Carbon dioxid	2	180		Carbon dioxid	4	179
	Carbon dioxid	4	174				

The need of using an inert gas when heating oils extracted from paint is illustrated by the figures in Table III. Oil A, extracted from a white-lead paste, contained some dissolved lead. The iodin number of the fatty acids was 185. Oil B was prepared by

[1] "The Chemistry and Technology of Mixed Paints," 1907.
[2] Bottler and Sabin, "German and American Varnish Making," 1912.
[3] Allen, "Commercial Organic Analysis," (4th Ed.) p. 336 (1910).
[4] *Chem. Ztg.*, **22**, 102, 558 (1898).

[1] "Paint Technology and Tests," 1911.
[2] *J. Frank. Inst.*, **174** (1912).

dissolving lead linoleate in pure raw linseed oil. The amount of lead in solution was 3.23 per cent., and the iodin number of the prepared oil was 177. Oil C was a pure raw linseed oil having an iodin number of 179. The oils were dissolved in ether, and, after distilling off the bulk of the ether, were heated at 98° to 99° C.

In order to have the iodin number furnish reliable information regarding the nature of the oil used in a paint, it is necessary to saponify and separate the fatty acids, making the determination on them. As the iodin number of the fatty acids is about 4 per cent. greater than that of the glycerid, the lower limit for oil from North American seed is about 185 and from South American seed about 178.

Gardner stated that "when paints are stored for a considerable length of time and then examined for the iodin value of their oil content, a lowering of the iodin value should not constitute cause for rejection or be sufficient evidence that the oil was adulterated with oils of lower iodin value." This is still open to question, as the results of the work in the Contracts Laboratory indicate that but little change takes place during two years. The changes that occur in a closed paint can are naturally very different from those in a drying paint film. Although not yet shown to be true, it is probable that in the latter case under the influence of light, air, and moisture there is actually more chemical combination between pigment and oil. An oil showing a high acid number has more effect upon a pigment that is basic in its nature, such as white lead, than an oil with a very low acid number as that used for this work.

The results in Table II show that the iodin number is in no case reduced sufficiently to place it below a figure frequently given by samples of pure raw linseed oil. Since duplicate determinations of the iodin number often vary as much as two units, a difference not exceeding that must be disregarded in comparing the different samples. The lower limit for oil from North American seed is placed at 178,[1] from South American seed at 171.[2] In the Contracts Laboratory many samples from unknown sources, which have shown all the characteristics of pure raw oil, have had an iodin number of 170 to 172.

It is interesting to note that while the iodin number of the oil was changed only slightly in all the mixtures, a greater decrease was caused by so-called inert pigments, kaolin, and Indian red, than by the others which include the lead and zinc pigments.

The large amount of ash from the white-lead samples undoubtedly shows that some of the pigment has been actually dissolved by the oil. The other figures for ash, however, do not show with certainty that there has been an appreciable solution of pigment.

The specific gravity of the oil was raised slightly in almost every case, but as with the iodin number the effect of two years' exposure was practically the same as that of one year. The upper limit for raw oil is placed at 0.936 at 15.6° C.[3] Many of the extracted oils are slightly above that figure.

A rise in specific gravity may be caused by the solution of some of the metallic portion of pigment in the oil, by volatilization of a portion of the oil, or by oxidation. Oxidation would be accelerated by small amounts of dissolved lead, manganese, or other catalytic agents. As the oil after two years showed no increase in specific gravity over the one-year figure (except in one case), the air enclosed in the jar probably had very little effect. The samples of extracted oil were rather small for an accurate specific-gravity determination, so that a difference of one or two units in the third decimal place must be disregarded.

Kaolin had greater bleaching effect than any of the other pigments, the black pigments and the zinc white causing practically no change in the color of the oil. The jars were filled completely, so that the influence of air must have been negligible for the first year. As a portion was removed at the end of that time, during the second year of exposure about 100 cc. of air filled the space above the paint in the jar.

The constants of an oil mixed with a pigment into a thick paste might be changed more than those of oil in the kind of paint used in these experiments, for a greater proportion, if not practically all, of the oil would then be in direct contact with the pigment. Samples of oil extracted from stiff white-lead pastes in this laboratory have yielded fatty acids with an iodin number of 185. The nature and original constants of the oils were unknown. The addition of driers and thinners would also influence these constants, and the results of these experiments are comparable only with paints made exclusively of raw oil and pigment in such proportions that the paint is ready for use without thinning.

Although the effect of white lead upon the specific gravity and iodin number is, under these circumstances, practically no greater than that of some of the other pigments, it is evident from the amount of ash that a greater weight of the pigment has gone into solution, though, considering the higher atomic weight of lead, the extent of chemical action need be no greater. The lead chromate did not combine with the oil to so great an extent as the basic carbonate.

The tendency of some of the pigments is to increase slightly the specific gravity of the oil, in many cases raising it above the higher limit for pure raw linseed oil, 0.936; hence a figure obtained on the extracted oil which is near the lower limit for pure raw linseed oil, 0.932, may indicate the presence of other oils of lower specific gravity.

NOTE ON THE MANGANESE CONTENT OF RAW LINSEED OIL

In 1911 sixteen samples of raw linseed oil of known purity and source were sent out to the members of the Subcommittee on Linseed Oil of the American Society for Testing Materials. Voorhees[1] found manganese in the ash of all the samples. In the Contracts Laboratory the same oils were ashed and the amount of manganese determined. Thirty grams of oil were

[1] *Proc. Amer. Soc. Test. Mat.*, **9**, 164 (1909).
[2] *Ibid.*, **11**, 202 (1911).
[3] *Proc. Amer. Soc. Test. Mat.*, **9**, 164 (1909).

[1] *Proc. Amer. Soc. Test. Mat.*, **11**, 209 (1911).

used and the manganese determined by the bismuthate method. Table IV shows the results. The percentage of manganese is figured on the oil.

TABLE IV—MANGANESE CONTENT OF RAW LINSEED OIL

Sample No.	Ash Per cent.	Manganese Per cent.	Sample No.	Ash Per cent.	Manganese Per cent.
1	0.16	0.0004	9	0.19	0.0008
2	0.02	0.0005	10	0.21	0.0006
3	0.03	Faint trace	11	0.05	Faint trace
4	0.16	0.0005	12	0.03	Faint trace
5	0.03	Faint trace	13	0.03	0.0005
6	0.18	0.0003	14	0.04	Faint trace
7	0.18	0.0002	15	0.16	0.0005
8	0.16	0.0003	16	0.16	0.0003

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PINE NUT OIL

By MAXWELL ADAMS AND AUGUST HOLMES

Received January 3, 1913

The nut pine tree, *Pinus Monophylla*, also known as *Pinus Fremontiana*, Pinon Pine and Grey Pine, according to Heller[1] grows along the eastern slopes of the Sierra Nevada Mountains from Steamboat Springs on the North to Lower California on the South. It varies in height from a mere shrub, on the borders of the desert, to a magnificent tree almost a hundred feet in height, in the Tehachapi Mountains. On the middle scale of the cones of this tree is borne an oblong thin-shelled seed about 15 mm. in length and weighing about one gram. The color of the nut is yellowish on the upper surface and dark reddish brown on the lower. The endocarp is resinous and oily, possessing a rich and pleasant taste.

Capt. John C. Fremont,[2] who first discovered this species of pine, in January, 1844, near the present site of Carson City, Nevada, states that the nuts from this tree constituted the principal subsistence of several Indian tribes, whom he visited during his explorations in the Great Basin. At the present time the Indians still gather the nuts in large quantities; a part is sold in the local markets, the remainder is used by the Indians for food.

The nuts used in the following experiments were gathered from trees growing on the mountains west of Walker Lake, Nevada, in the Autumn of 1911. A routine analysis of the nut kernels gave the following results:

	Air-dry Per cent.	Green Per cent.	Water-free Per cent.
Water	7.88	61.57	...
Ash	2.60	1.08	2.82
Ether extract	22.77	9.49	24.70
Crude fiber	0.65	0.27	0.70
Crude protein	8.94	3.73	9.70
Nitrogen-free extract	57.21	23.86	62.08
Nitrogen	1.43	0.59	1.55

About eight kilograms of the nuts were partially air dried, hulled, the kernels ground and the oil extracted with ether in a Jacobson extractor.[3] The nuts used in one extraction gave the following yield:

Weight of nuts used	522 grams
Weight of kernels obtained	400 grams
Weight of oil extracted	65 grams
Per cent. of oil in nuts	12.4
Per cent. of oil in kernels	16.2

[1] *Muhlenbergia*, **5**, 335.
[2] Fremont's First and Second Expeditions, p. 221.
[3] *Jour. Am. Chem. Soc.*, **23**, 2052.

The oil obtained from the evaporation of the ether extract is of the consistency of ordinary olive oil and at first is light yellow in color, but upon standing in the laboratory the color gradually fades. A sample which remained near a window for a year became entirely colorless. The oil has a pleasing aromatic odor and agreeable taste. Blasdale[1] reports that a sample, bought in the open market, and supposed to be pine nut oil, examined by him, had a disagreeable odor and rancid taste. The oil in question must have been extracted from stale nuts, or obtained from the nuts of some pine other than *Pinus Monophylla*, for the physical constants, determined by him, differ markedly from those obtained by us, when examining the freshly extracted oil.

The oil melts at —15°, turns brown and decomposes without boiling at 320°, but when heated under a pressure of 60 mm. it begins to distill without decomposition at 305°. The saponification value, determined by the method of Köttstorfer, is 189.31, and the iodine absorption value, when determined by the method of Hübl, gives the following results:

	Expt. I	Expt. II
Weight of oil used	0.2466	0.2338
Weight of iodine absorbed	0.2663	0.2525
Iodine value	107.97	108.00

The refractive index, determined with an Abbé refractometer, is as follows:

Temperature Degrees	Refractive index
10	1.4747
15	1.4733
20	1.4716
25	1.4698
30	1.4680
35	1.4662
40	1.4543

The refractive index, saponification value and iodine number of the oil, which had been bleached in the sunlight, was determined but no change was noted, except that the iodine number showed a slight diminution, which is probably due to the absorption of oxygen by the semidrying oils present.

The oil was submitted to fractional distillation, at a pressure of 60 mm., and each fraction examined with the refractometer, in order, if possible, to separate it into distinct chemical compounds, with the following results: 150 cc. of the oil were used for fractionating.

Fraction	Temperature Degrees	Vol. of distillate Cc.	Ref. index at 40°
1	305	0.5	1.4522
2	305–15	20	1.4531
3	315–20	50	1.4550
4	320–50	30	1.4562
5	350–65	20	1.4570
6	365–70	5	1.4594

At 320° the oil in the distilling flask turns brown and begins to show signs of decomposition, which increases as the temperature rises. At the end of the experiment there is in the flask a black tar-like residue. The first three fractions are liquids, the last three solids, at room temperature. The change in the refractive index and the variation in the melt-

[1] *Jour. Am. Chem. Soc.*, **17**, 935.

ing points indicate a difference in the composition of the several fractions, but when they were submitted to a second distillation they showed no tendency to produce substances with definite boiling points, and each decomposed somewhat when distilled and left a residue of tar.

The glycerine in the oil was determined by oxidizing with potassium permanganate in alkaline solution, according to the method of Benedikt and Zsigmondy,[1] and found to be 9.2 per cent.

When 200 grams of the oil, dissolved in alcohol, are saponified with potassium hydroxide, and evaporated to dryness, to free the residue from alcohol, it dissolves readily and completely in water, to a clear solution. This test indicates the absence of phytosterol.

The mixed fatty acids when precipitated from the soap solution with dilute sulfuric acid, washed and dried, are light yellow in color, have a sp. gr. of 0.904 at 15°, and the yield is about 90 per cent. of the total oil. The mixed acids, when separated by treating their slightly alcoholic solution with lead acetate, according to the method of Gusserow-Varrentrap,[2] yielded 82 per cent. of unsaturated acids and 8 per cent. of saturated acids. This method does not give exact quantitative results as Lewkowitsch[3] has already pointed out, and the following iodine value determination shows that the saturated acids thus obtained are impure:

	Expt. I	Expt. II
Weight of saturated acid used.....	0.4054	0.3844
Iodine number.....	6.6	6.5
Weight of unsaturated acid used...	0.4016	0.3782
Iodine number.....	116.8	117.1

0.6031 gram of these mixed saturated acids was dissolved by warming with 100 cc. of alcohol, sp. gr. 0.91, which had previously been saturated with pure stearic acid at 0° C., and allowed to stand in a flask surrounded by ice water for twenty-four hours. The white, laminated crystals which separated were filtered from the solution and weighed when dry 0.0657 gram. Their melting point was 66°. This yield shows the presence of 10.8 per cent. stearic acid in the mixed saturated acids, or about 1 per cent. stearic acid in the original oil.

Using the iodine number of oleic acid as 90.07, and the iodine number of the impure saturated acids, determined above, as 6.6 in the following equation $\frac{100 \times 6.6}{90.07}$ = per cent. oleic acid, we have 7.21 per cent. oleic acid present. Assuming that the impure saturated acids obtained by the lead salt method consisted wholly of stearic, palmitic and oleic acids, there would be, calculated by difference, 82 per cent. palmitic acid present in the saturated acids. There was, however, obtained by the crystallization from alcohol of the magnesium salt of the mixed saturated acids, a very small yield of an acid, which had a refractive index of 1.4236 at 72°. This is identical with that given by Partheil and Ferrie[4] for lauric acid. The quantity available was not sufficient for a more complete identification.

[1] *Jour. Soc. Chem. Ind.*, **1885**, 610.
[2] *Liebig's Ann.*, **27**, 153; **35**, 197.
[3] *Jour. Soc. Chem. Ind.*, **1890**, 845.
[4] *Archive. der Pharm.*, **1903**, 545.

The unsaturated acids obtained are light yellow in color, have a sp. gr. at 15° of 0.938 and have a refractive index as follows:

Temperature Degrees	Refractive index
15	1.4657
20	1.4635
25	1.4623
30	1.4604
35	1.4588
40	1.4570
45	1.4550

The neutralization value of the mixed unsaturated fatty acids is 211.1, and the mean molecular weight calculated from this data is 265.7.

Oleic acid, according to the investigations of Hazura,[1] is converted into hydroxystearic, and linolic acid into sativic acid, when the mixed acids are oxidized with potassium permanganate in dilute alkaline solution. After oxidizing ten grams of the mixed acids by this method, the acids were recovered by passing into the solution a stream of sulfur dioxide, until the solution had an acid reaction. The precipitated acids were then collected on a filter and separated by extracting the hydroxystearic acid in a Soxhlet apparatus with ether. The solution thus obtained was evaporated to a small volume and cooled, when the white laminated crystals of dihydroxystearic acid separated. The crystals thus obtained after a second recrystallization had a melting point of 132°. The dihydroxystearic acid obtained was 44 per cent. of the total unsaturated acids, which corresponds to 39.2 per cent. oleic acid. This method does not give quantitative results, since a considerable amount of the dihydroxystearic acid remains in the ether solution, used for extraction and purification, but it serves to fix the lower limit for the amount of oleic acid present.

The residue remaining in the Soxhlet apparatus, which was insoluble in the ether, was extracted repeatedly with hot water, and the sativic acid allowed to crystallize. The ten grams of mixed fatty acids used gave 0.8 gram of a white crystalline substance having a melting point of 171°, which is slightly lower than that given for tetrahydroxystearic acid. Were the acid pure this corresponds to 6.4 linolic acid. Besides the oleic and linolic acids the presence of small quantities of other unsaturated acids were indicated, but no confirmatory tests made were successful in proving their identity. Assuming that the mixed fatty acids consisted entirely of oleic and linolic acids, calculated from the following formula,

$$x + y = 100$$

$$\frac{90.07\,x}{100} \pm \frac{181.42\,y}{100} = 117.05$$

where x equals the per cent. of oleic, y equals the per cent. of linolic acid, and 117.05 the iodine number of the mixed acids, there would be present 70.5 per cent. of oleic and 29.5 per cent. of linolic acid. But by the method of Hazura we obtain only 6.4 per cent. of linolic acid. Experimental errors and inaccuracies of the two

[1] *Mond. f. Chemie*, **1887**, 147–260.

methods do not satisfactorily account for so great a variation in results. Careful tests by the method of Hazura failed to show the presence of linolenic acid, but from the high iodine absorption, some unidentified unsaturated acid appears to be present.

From these experiments we conclude that the oil from the nuts of *Pinus Monophylla* consists chiefly of the glyceride of oleic acid together with small amounts of the glycerides of stearic, palmitic, lauric and linolic acids.

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ANALYSES OF CERTAIN OF THE PACIFIC COAST KELPS[1]

By E. G. PARKER AND J. R. LINDEMUTH

Received February 5, 1913

While many kinds of kelp are to be found on both the Atlantic and Pacific coasts, the giant kelps, peculiar to the Pacific coast, are the most important to the United States from an economic standpoint because of their size and remarkable content of potassium salts. While the other varieties grow from two to twelve feet in length, the giant kelps are found from 30 to 200 feet in length, and in specific cases much longer.

The composition of the Pacific kelps was studied first by David M. Balch,[2] of Coronado Beach, California. He analyzed specimens of the giant kelps of the northern and southern Pacific coast of the United States, including *Nereocystis luetkeana*, *Macrocystis pyrifera*, and *Pelagophycus porra*. Later in the course of an investigation of the fertilizer resources of the United States, under the direction of Dr. Frank K. Cameron,[3] the composition was studied by Dr. J. W. Turrentine,[4] of this Bureau, who analyzed specimens of various algae collected from Alaska by Mr. E. C. Johnston, of the "Albatross;" from Puget Sound (San Juan County), Washington, by Prof. Geo. B. Rigg, of the University of Washington; from the region of Monterey Bay, California, by Prof. F. M. McFarland, of Leland Stanford Jr. University; and from the vicinity of San Diego, California, by Capt. W. C. Crandall, of the La Jolla Marine Biological Station.[5] From these analyses it has been concluded that the genuses to be considered as commercial sources of potash are, in the north—the *Nereocystis luetkeana* and *Macrocystis pyrifera*, and in the south—the *Macrocystis pyrifera* and the *Pelagophycus porra*; also, that the indications are that the northern kelps are richer in potassium chloride than the southern.

Samples.—Specimens of *Macrocystis pyrifera* and *Nereocystis luetkeana* were collected for analysis in Freshwater Bay, Washington, by Prof. George B. Rigg: from 34°–38° N, 120°–124° W, and also from 32°–33° N, 117°–118° W, by Capt. W. C. Crandall. The samples collected by Prof. Rigg were shipped dry, in glass jars, while those by Capt. Crandall were shipped dry, in canvas bags. In each case the entire sample was dried at 103° C. until it would easily powder, and was then ground, care being taken not to lose any of the adhering salts, or salts shaken off into receptacle in transit. After grinding, the whole sample was mixed well to ensure homogeneity.

Method of Analysis.—The methods of analysis used were in the main those employed by Turrentine.[1] The results of the analyses are recorded in Table I, p. 288.

The determinations of nitrogen were made by Mr. T. C. Trescott, of the Bureau of Chemistry, to whom we are greatly indebted. The determinations were made on the oven-dried samples by the Kjeldahl method. The locations from which samples were collected by Crandall are given in latitude and longitude. A detailed description of this work by Crandall and Rigg, together with a complete set of working charts of the Pacific kelp groves from Puget Sound southward, will soon be published by the Bureau of Soils.

DISCUSSION OF ANALYTICAL DATA

In the second column are given the serial numbers of the kelp samples examined. There are three series consisting of three collections of samples secured during the year 1912. They are designated by the initial letter of the name of the collector: as R, Rigg, C, Crandall, C′, Crandall (different collection).

An inspection of Table I (see p. 288) results in the following averages:

TABLE II

	KCl Per cent.	I Per cent.	N Per cent.
Series R (aver. of 11)	27.63	..	2.14
Series C (aver. of 19)	23.84	..	2.21
Series C′ (aver. of 9)	10.34	..	1.11
Macrocystis (aver. of 23)	15.82	..	..
Nereocystis (aver. of 16)	30.50	..	..
Series R (Mac) (aver. of 2)	19.47	..	..
Series R (Ner) (aver. of 9)	29.54	..	..
Series C (Mac) (aver. of 12)	19.20	..	..
Series C (Ner) (aver. of 7)	31.74	..	..
Series C′ (Mac) (aver. of 9)	10.54	..	..
Entire table (aver. of 39)	21.85	0.20	1.93

From Tables I and II the following conclusions may be drawn:

1. That the average potassium chloride content is high.

2. That apparently no definite relation exists between the different constituents of kelp.

3. That the total average potassium chloride content of the *Nereocystis luetkeana* is greater than that of the *Macrocystis pyrifera* and that for the potassium chloride content of kelp from different localities the same is true; also that in every case the potassium chloride content of the *Nereocystis luetkeana* is greater than the average potassium chloride content of the *Macrocystis pyrifera*.

4. That apparently the northern kelps are richer in potassium chloride than the southern.

5. That the kelp from the southern coast of California (Series C′, Table II) is comparatively low in potassium chloride. The fact that the average potassium chloride content of kelp from the same locality was found to be much higher both by Balch and Turrentine, and also that the nitrogen content is

[1] Published by permission of the Secretary of Agriculture.

[2] Balch, THIS JOURNAL, 1, 177 (1909).

[3] *Senate Doc.* No. 190, 62nd Congress, 2nd Session, 1912.

[4] Turrentine, THIS JOURNAL, 4, 431 (1912).

[5] Now the Scripps Marine Biological Station, of the University of California.

[1] Turrentine, *loc. cit.*

TABLE I

No.	Serial No.	Name of kelp	Location	Description	Potash (K_2O) Per cent.	Iodine (I) Per cent.	Nitrogen (N) Per cent.	Sol. salts (Na, K, Cl, SO_4) Per cent.	Organic matter Per cent.	Ash Per cent.	KCl Per cent.
1	R1	Nereocystis luetkeana	Freshwater Bay	Fronds	18.04	0.23	2.26	44.17	51.13	4.80	28.56
2	R2	Nereocystis luetkeana	Freshwater Bay	Fronds	17.61	0.24	2.21	44.17	51.11	4.72	27.87
3	R3	Nereocystis luetkeana	Freshwater Bay	Stipes	31.62	0.25	1.21	63.75	33.15	3.46	50.05
4	R4	Nereocystis luetkeana	Freshwater Bay	Fronds	16.92	0.20	2.57	43.31	51.16	5.47	26.76
5	R5	Nereocystis luetkeana	Freshwater Bay	Fronds	17.05	0.24	2.71	41.53	51.67	6.30	26.98
6	R6	Nereocystis luetkeana	Freshwater Bay	Fronds	17.32	0.28	2.53	45.40	50.12	4.48	27.40
7	R7	Nereocystis luetkeana	Freshwater Bay	Fronds	16.20	0.19	2.54	42.88	52.14	4.98	25.64
8	R8	Nereocystis luetkeana	Freshwater Bay	Stipes	16.50	0.30	2.21	59.24	37.40	3.36	26.13
9	R9	Nereocystis luetkeana	Freshwater Bay	Stipes	16.72	0.20	1.46	57.06	39.02	3.92	26.44
10	R10	Macrocystis pyrifera	Near Law Point	Fronds	11.82	0.22	2.43	28.32	67.78	3.90	18.70
11	R11	Macrocystis pyrifera	Near Law Point	Stipes and pneumatocysts	12.80	0.23	1.37	34.42	59.40	6.18	20.23
12	C1	Macrocystis pyrifera	34°-28′-20″ 120°-28′-50″		14.17	0.24	2.15	33.40	62.95	3.64	22.40
13	C2	Macrocystis pyrifera	34°-31′-10″ 120°-31′-30″		9.35	0.25	2.72	20.25	73.06	6.69	14.78
14	C3	Macrocystis pyrifera	35°- 9′-30″ 120°-42′-30″		8.62	0.14	2.35	23.14	68.26	8.60	13.63
15	C4	Nereocystis luetkeana	35°-10′-35″ 120°-48′-40″		20.83	0.24	1.93	49.06	47.86	3.08	32.99
16	C4a	Nereocystis luetkeana	35°-10′-35″ 120°-48′-40″		19.71	0.25	1.96	53.50	43.02	3.48	31.18
17	C5	Macrocystis pyrifera	35°-26′-10″ 120°-35′- 0″		4.14	0.19	2.40	13.95	82.17	3.88	6.55
18	C6	Macrocystis pyrifera	35°-37′-30″ 121°-13′-30″		6.06	0.15	2.68	14.24	79.66	6.10	9.60
19	C7	Macrocystis pyrifera	36°-56′-25″ 120°- 5′-45″		12.26	0.18	3.17	30.52	63.28	6.20	19.39
20	C8	Macrocystis pyrifera	36°-57′-30″ 122°- 8′- 0″		12.38	0.18	2.11	28.02	68.79	3.19	19.58
21	C9	Macrocystis pyrifera	37°- 6′- 0″ 122°-17′-40″		16.44	0.24	2.16	35.88	59.80	4.32	26.02
22	C10	Macrocystis pyrifera	37°-56′-30″ 122°-46′- 0″		9.52	0.18	2.38	23.90	72.37	3.73	15.07
23	C11	Nereocystis luetkeana	38°-49′- 0″ 123°-36′-30″		16.72	0.13	2.22	43.32	53.56	3.12	26.45
24	C12	Nereocystis luetkeana	38°-51′-30″ 123°-40′- 0″		20.62	0.15	2.25	45.48	51.42	3.10	32.63
25	C13	Sample decayed									
26	C14	Macrocystis pyrifera	37°-56′-40″ 122°- 7′- 0″	Young plant	17.26	0.15	2.18	38.26	58.18	3.55	27.30
27	C15	Macrocystis pyrifera	37°-56′-40″ 122°- 7′- 0″	Old plant	27.66	0.14	1.00	56.02	41.04	2.93	43.78
28	C16	Macrocystis pyrifera	37°-66′-40″ 122°- 7′- 0″	Young on bottom of old plant	8.13	0.15	2.10	21.27	75.57	3.16	12.85
29	C17	Nereocystis luetkeana	36°-37′-50″ 121°-54′-52″	Old plant	16.96	0.20	2.15	38.06	57.26	4.68	26.83
30	C18	Nereocystis luetkeana	36°-37′-50″ 121°-54′-52″	Young plant	23.82	0.17	2.41	50.51	47.26	2.22	37.72
31	C19	Nereocystis luetkeana	36°-37′-50″ 121°-54′-52″	Very old plant	21.70	0.18	1.58	46.60	50.20	3.20	34.35
32	C′1	Macrocystis pyrifera	Imperial Beach near San Diego		6.68	0.27	1.93	23.12	67.47	9.40	10.57
33	C′2	Macrocystis pyrifera	Monument Bet. Mex. & U. S., Chart 5100		3.20	0.18	1.56	16.56	76.46	6.98	5.06
34	C′3	Macrocystis pyrifera	W. Descanso Pt. (Mex. territory)		6.33	0.23	1.52	19.51	77.17	3.32	10.02
35	C′4	Macrocystis pyrifera	S. Coronado Is. So. of island		7.98	0.14	1.81	23.69	67.72	8.60	12.61
36	C′5a	Macrocystis pyrifera	32°-51′-30″ 117°-17′- 6″		7.18	0.22	0.64	20.34	76.50	3.16	11.35
37	C′5b	Macrocystis pyrifera	32°-51′-30″ 117°-17′- 6″		6.79	0.25	0.79	20.12	76.55	3.32	10.73
38	C′5c	Macrocystis pyrifera	32°-51′-30″ 117°-17′- 6″		6.39	0.24	0.66	18.62	77.78	3.60	10.10
39	C′5d	Macrocystis pyrifera	32°-51′-30″ 117°-17′- 6″		7.82	0.23	0.67	24.06	72.40	3.54	12.38
40	C′5e	Macrocystis pyrifera	32°-51′-30″ 117°-17′- 6″		6.50	0.22	0.54	22.22	74.86	2.92	10.28

also correspondingly low, leads to the belief that these particular specimens do not fairly represent the kelps of that locality.

6. That the iodine content of northern and southern kelps show no conclusive differences.

Considering, now, all the results which have been obtained on the kelps of the Pacific coast, it is to be noted first that if Balch's ratio of sodium to potassium, 1 : 4, determined for *Pelagophycus porra* held also for Macrocystis and Nereocystis, the former would have an average of 35.45 and the latter 36.93 per cent. potassium chloride. While many individual samples would undoubtedly run as high or even higher than these figures, they are almost certainly too high as averages. Turrentine's results show that the northern Macrocystis (2 samples) contain 26.5 per

cent. and the Nereocystis (4 samples) 30.9 per cent. potassium chloride, the average for all being 29.4 per cent. The southern Macrocystis (22 samples) contained 21.6 per cent. and the Nereocystis (2 samples) 35.9 per cent. or an average of 23.4 per cent. for 26 samples. Turrentine's results indicate that the northern Nereocystis contains somewhat less potassium chloride than the southern, while the reverse holds for Macrocystis and the kelps as a whole.

The results obtained by the authors thus confirm the conclusions from Turrentine's analyses. Finally, disregarding the results of Balch, the average content of all samples so far examined give:

TABLE III

	North Per cent. KCl	South Per cent. KCl	Total Per cent. KCl
Nereocystis	28.1	32.7	31.1
Macrocystis	23.0	20.8	21.0

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THE SPECIFIC HEAT OF COAL AND ITS RELATION TO THE PRESENCE OF COMBINED WATER IN THE COAL SUBSTANCE[1]

By HORACE C. PORTER AND GUY B. TAYLOR

Received March 7, 1913.

The specific heat of coal is hardly capable of precise determination. Coal is a conglomerate of varying composition, unstable and easily subject to change by heat and by exposure to the air. Nevertheless, a determination of the specific heat is possible on a given sample with some degree of accuracy, and there may be determined also the influence of the character of the coal and the percentage of moisture on the specific heat.

When coal is being heated it passes ordinarily through a range of temperature from about 20° C. to 250° or 300° C. before it ignites, or before, in the absence of air, it begins to decompose rapidly by destructive distillation. During this preliminary heating of coal, whether in a coke oven, gas retort, or gas producer or in the "green" coal layer of a furnace fuel bed, it is of interest and often of value to fuel engineers to know the relative quantity of heat communicated to the coal for a given temperature rise, *i. e.*, its specific heat.

Solids have in general a much lower heat capacity than that of water, metals in most cases less than 0.1 and sand, fire clay, porcelain or glass not far from 0.2. These solid substances thus require only $^1/_{10}$ or $^1/_5$ as much heat as is required by water, in order to raise their temperature a given number of degrees. Coal we should expect, by reason of its organic nature and its content of water, to have a higher specific heat than metals or clay. Bituminous coal, air-dry, proves to have, in fact, through the temperature interval 28° to 65° C., a value varying from 0.261 for the New River type to 0.370 for Wyoming subbituminous of 11% moisture. The specific heat becomes greater at higher temperatures, that of the New River type, for example, being 0.354 between 177° and 227°. In Table I are given the results of the determinations of specific heat made in this investigation.

Different authorities have given the specific heat of coal as varying from 0.20 to 0.45, with usually no qualifying statement as to moisture content or character of the coal. As a matter of fact, the moisture content materially affects the specific heat, and from the relationship between the specific heats of dry and undried coal may be deduced certain conclusions as to the combination of the water present.

TABLE I—SPECIFIC HEATS OF DIFFERENT COALS

$k = \frac{W}{w}\frac{dT}{dt}$, k = specific heat, W = weight of water heated + water equivalent of apparatus (14.1 grams), w = weight of substance, dT = rise of temperature in calorimeter, dt = fall of temperature in coal.

Expt. No.	Coal No. or substance	W	w	dT	dt	k	Temp. range. ° C
39	9614 (New River)	229.5	45.0	1.95	37.6	0.264	66.0–28.4
44	9614 (New River)	238.6	40.0	1.56	36.0	0.259	64.0–28.0
45	9614 (New River)	206.7	40.0	1.74	34.4	0.261	62.8–28.4
49	384 (Pittsburg)	215.6	40.0	1.85	34.8	0.286	63.4–28.6
50	384 (Pittsburg)	204.8	39.1	1.71	31.8	0.282	60.8–29.0
51	384 (Pittsburg)	209.4	40.0	1.80	32.9	0.286	61.8–28.9
52	384 (Pittsburg)	223.1	40.0	1.67	32.2	0.289	61.6–29.4
27	48 (Illinois)	236.8	40.0	2.07	35.3	0.347	64.4–29.1
28	48 (Illinois)	246.8	40.0	1.90	35.0	0.335	64.4–29.4
29	48 (Illinois)	249.5	40.0	1.98	36.4	0.339	64.4–28.0
32	48 (Illinois)	220.9	40.0	2.16	34.7	0.344	63.0–28.3
36	48 (Illinois)	218.8	39.9	2.04	33.6	0.333	62.5–28.9
37	48 (Illinois)	232.6	34.9	1.77	34.4	0.343	62.9–28.5
59	48 (in toluene)(*a*)	122.5	35.0	2.97	31.4	0.331	61.5–30.1
61	48 (in toluene)	123.3	28.8	2.56	32.0	0.342	61.0–29.0
72	575 (Wyoming)	208.6	30.0	1.97	36.2	0.378	63.9–27.7
73	575 (Wyoming)	232.2	40.0	2.40	37.2	0.374	63.4–26.2
74	575 (Wyoming)	224.2	26.5	1.71	38.5	0.375	64.2–25.7
66	575 (in toluene)	123.8	30.0	2.96	32.9	0.371	62.2–29.3
67	575 (in toluene)	123.2	25.0	2.47	33.8	0.361	63.2–29.4
76	575 (in toluene)	124.7	30.0	3.11	36.2	0.357	62.4–26.2
62	48 (dry)(*b*)	123.3	30.0	2.46	33.4	0.302	62.8–29.4
63	48 (dry)	122.3	30.0	2.44	33.1	0.301	62.6–29.5
64	48 (dry)	122.3	28.0	2.30	33.5	0.300	62.7–29.2
68	575 (dry)	122.6	30.0	2.62	34.3	0.312	63.8–29.5
69	575 (dry)	124.1	25.0	2.26	35.2	0.318	64.3–29.1
15	Copper(*c*)	218.5	103.1	1.58	35.9	0.0933	64.6–28.7
19	Copper	227.3	101.7	1.49	35.7	0.0933	64.8–29.1
17	Copper	227.4	102.2	1.53	36.5	0.0933	64.7–28.2
41	Copper	196.3	101.3	1.70	35.3	0.0933	63.5–28.2
14	Lead (0.0310 at 17°–100°)(*d*)	237.1	284.6	1.35	36.4	0.0308	64.5–28.1
3	Same	210.1	356.0	1.84	35.7	0.0305	64.3–28.6
23	Marble (0.206 at 0°–100°)(*d*)	235.5	60.0	1.88	36.8	0.201	64.8–28.0
24	Same	232.6	50.0	1.68	36.4	0.202	64.8–28.4
33	Jena glass (0.2182 at 18°–99°)(*d*)	209.8	44.5	1.35	33.1	0.192	61.5–28.4
40	Same	197.2	52.6	1.93	36.7	0.197	66.2–29.5

(*a*) Water equivalent of toluene = weight used × 0.421

(*b*) Using toluene.

(*c*) Used as standard, to determine water equivalent of calorimeter, assuming specific heat as 0.0933. In expt. 15, 204.3 grams of water were used; in No. 19, 213.3; in No. 17, 212; and in No. 41, 183.4 grams.

(*d*) Values taken from Landolt and Börnstein Tabellen.

Coal is essentially an organic material, the product of the degradation of cellulose and other plant tissues and contains, like most other materials of vegetable origin, some water in a state of physical or chemical combination. When exposed to air of average humidity, coal does not dry out, but assumes rather a state of moisture equilibrium at which there remains in the coal from one to sixteen per cent. of moisture, depending on the kind of coal and the humidity of

[1] Presented by permission of the Director U. S. Bureau of Mines, at the annual meeting of the American Chemical Society, Milwaukee, March 25–28, 1913.

TABLE Ia—SPECIFIC HEATS OF COALS AT HIGHER TEMPERATURES
(Water equivalent of calorimeter 14.1 grams)

Expt. No.	Coal No. or substance	W	w	dT	dt	k	Temp. range. ° C
99	Copper.....	207.7	45.0	2.03	100.4	0.0933	125.5–25.1
102	Copper.....	209.1	45.0	2.11	103.8	0.0945	129.5–25.7
98	384.....	216.7	10.0	1.48	106.1	0.302	131.0–24.9
100	384.....	224.1	12.0	1.73	105.1	0.307	130.0–24.9
101	384.....	226.6	11.0	1.63	104.6	0.313	130.0–25.4
105	384.....	193.2	10.0	1.68	104.6	0.310	130.0–25.4
103	9614.....	222.7	11.0	1.48	103.3	0.290	128.0–24.7
104	9614.....	205.1	10.0	1.43	102.3	0.287	127.5–25.2
106	9614.....	191.5	9.5	1.51	106.2	0.287	132.0–25.8
88	Copper.....	340.2	41.0	1.73	151.3	0.0948	176.0–24.7
90	Copper.....	324.5	37.0	1.67	155.9	0.0938	181.0–25.1
92	Copper.....	315.1	36.7	1.68	154.1	0.0936	179.0–24.9
89	384.....	313.1	10.0	1.57	153.3	0.318	178.5–24.2
96	384.....	320.9	10.0	1.48	148.0	0.321	172.7–24.7
93	9614.....	314.5	12.0	1.76	154.5	0.298	179.0–24.5
94	9614.....	308.0	10.0	1.49	151.5	0.303	176.0–24.5
108	Copper.....	296.9	20.0	1.24	195.4	0.0942	220.7–25.3
110	Copper.....	296.9	30.0	1.90	198.3	0.0948	223.7–25.4
107	384.....	330.5	6.0	1.22	200.1	0.332	225.5–25.4
109	384.....	307.1	8.0	1.73	199.8	0.332	225.5–25.7
111	9614.....	314.1	8.0	1.64	204.6	0.315	229.7–25.1
113	9614.....	278.0	9.0	2.03	201.0	0.312	226.6–25.6

TABLE Ib—SUMMARY OF AVERAGE VALUES OBTAINED FOR THE SPECIFIC HEAT OF COAL AT DIFFERENT TEMPERATURES

					Calculated Values		
Coal	63°–28°	130°–25°	177°–25°	227°–25°	130°–63°	177°–130°	227°–177°
9614	0.261	0.288	0.301	0.314	0.303	0.329	0.354
384	0.286	0.308	0.320	0.332	0.321	0.347	0.368
48	0.339	...	...	...	...	...	...
48 (dry)	0.301	...	...	...	...	...	...
575	0.370	...	...	...	...	...	...
575 (dry)	0.315	...	...	...	...	...	...

the air. In other words, the vapor pressure of the moisture in coal does not reach the normal tension of water vapor until the amount present exceeds a certain percentage; up to that percentage, therefore, the water present in coal does not exist as free superficial moisture. An experimental study of the vapor tensions of coals of different water content, bringing them to equilibrium over sulfuric acid of various dilutions, has confirmed these conclusions.[1]

By comparison of the specific heats of dry and undried coal, knowing the percentage of water present, the molecular heat of the water may be calculated. A coal from Franklin Co., Illinois, bearing 8.4 per cent. water, has a specific heat of 0.337, while in the dry condition its value is 0.301. Following Kopp's law, the specific heat of the water present in this coal is $\frac{0.337 - (0.916 \times 0.301)}{0.084} = 0.730$, and its molecular heat, $18 \times 0.730 = 13.1$. Similarly a Wyoming subbituminous of 11.0 per cent. moisture has in the undried condition a specific heat of 0.359 and in the dry state 0.315, the specific heat of its water being thus $\frac{0.359 - (0.89 \times 0.315)}{0.11} = 0.718$ and the molecular heat 12.9.

The molecular heat of the combined water in many hydrated salts has been determined by Kopp and others.[1] Some examples are given below:

TABLE II

Substances	% H_2O	Specific heat	Molecular heat of water present
$CuSO_4.5H_2O$ (Schottky).....	36.1	0.269	8.6
$CuSO_4$ (anhydrous) (Schottky)	...	0.151	..
$MgSO_4.7H_2O$ (Kopp).....	51.2	0.407	10.6
$MgSO_4$ (anhydrous) (Kopp)...	..	0.216	..
$BaCl_2.2H_2O$ (Schottky).....	14.7	0.151	9.1
$BaCl_2$ (anhydrous)	..	0.090	..
$MnSO_4.5H_2O$ (Kopp).....	37.4	0.407	14.1
$MnSO_4$ (anhydrous) (Kopp)...	..	0.182	..
Coal 48 (Ill.).....	8.4	0.337	13.1
Coal 48 (dried).....	..	0.301	..
Coal 575 (Wyo.).....	11.0	0.359	12.9
Coal 575 (dried).....	..	0.315	..

The molecular heat of the water in coal is evidently lower than that of free uncombined water and approaches that of the combined water of some crystallized salts. The conclusion therefore follows, in agreement with that drawn from the vapor-pressure relationships, that the moisture of coal is in part physically or chemically combined. Still further experimental evidence of the presence of combined water was incidentally encountered in developing the method for the determination of specific heat. In attempting to determine the value for dry coal by the method of mixtures, adding the heated coal to water in a calorimeter, so large a rise of temperature resulted that the specific heat appeared to be much greater than that of the undried coal. Since moisture should increase the specific heat of coal on account of the higher specific heat of water even when in combination, the discrepancy was at once attributed to some inherent error of the method. This error was found to lie in the development of heat by the physical or chemical action of water on the dry coal. Dry coal and water carefully brought to the same temperature in separate vessels were mixed in a calorimeter and the development of heat measured. The results given in Table III show in case of the Wyoming coal No. 43 a heat development of 20.0 calories per gram. In the specific heat determination using 30 grams of this coal dry, and mixing it with water, there were developed 600 calories therefore, as heat of combination, affecting in no small degree the result of the specific heat determination.

The values for the specific heat of dry coal were therefore determined by using toluene in place of water in the calorimeter, since toluene was found to have almost no calorific action on the dry coal. That the calorific action of water on dry coal is due to chemical or physical combination becomes highly probable in the light of the two other phenomena previously mentioned which point to the same conclusion, *viz.*, the low vapor pressure and the low molecular heat of the water in coal.

[1] Acknowledgment is due to Mr. O. C. Ralston for the greater part of the experimental work on vapor pressure of coal.

[1] Kopp, *Phil. Trans. London*, **155**, [1] 71 (1865); Schottky, *Zeit. phys. Chem.*, **64**, 415 (1908); Jackson, *J. Am. Chem. Soc.* **34**, 1470 (1912).

TABLE III—HEAT OF COMBINATION, DRY COAL AND WATER
(Water equivalent of calorimeter + water = 48 grams)

Expt. No.	Coal No.	Wt. of coal.	Wt. of dried coal	Original moisture. Per cent.	Moisture in dried coal	Temp. rise. Deg. C.	Total calories	Calories per gram original coal	Calories per gram dried coal
2a	43	15.0	11.93	20.4	1.0	4.31	207.0	13.8	17.3
2b	43	15.0	12.00	20.0	1.4	3.97	191 0	12.7	15.9
3a	43	15.0	11.80	21.6	0.0	4.92	236.0	15.7	20.0
3b	43	15.0	11.80	21.3	0.1	4.79	230.0	15.3	19.5
4a	43	15.0	14.43	3.8	17.6	0.40	19.2	1.3	1.4
b	43	15.0	13.98	6.8	14.6	0.69	33.5	2.2	2.4
c	43	15.0	13.31	11.3	10.1	1.21	58.0	3.9	4.3
d	43	14.5	12.50	14.0	7.4	1.79	86.0	5.9	6.8
e	43	15.0	12.09	19.4	2.0	3.55	170.4	11.4	14.1
f	43	15.0	11.92	20.5	0.9	4.36	209.0	13.9	17.5
5a	43	..	14.00	...	16.7	0.36	17.3	...	1.2
b	43	..	14.00	...	14.8	0.45	21.6	...	1.5
c	43	..	14.00	...	14.8	0.46	21.7	...	1.5
11	43	..	14.00	...	15.1	0.53	25.4	...	1.7
7a	48	15.0	14.70	1.98	6.7	0.16	7.7	0.51	0.52
b	48	15.0	14.43	3.79	4.9	0.20	9.6	0.65	0.66
c	48	15.0	14.23	5.18	3.5	0.36	17.3	1.15	1.22
d	48	15.0	14.04	6.38	2.3	0.58	27.8	1.85	1.99
e	48	15.0	13.86	7.60	1.1	0.66	31.7	2.11	2.30
f	48	15.0	13.75	8.32	0.4	1.08	51.8	3.45	3.80
g	48	15.0	13.70	8.62	0.1	1.21	58.1	3.90	4.20
8	48	15.0	13.77	8.25	0.5	0.98	47.0	3.13	3.33
9a	384	15.0	14.82	1.20	0.6	0.12	5.7	0.39	0.40
b	384	15.0	14.78	1.44	0.4	0.18	8.6	0.57	0.58
10a	9614	15.0	14.84	1.05	0.1	0.0	...	...	0.0
b	9614	15.0	14.85	0.98	0.2	0.08	...	...	...

EXPERIMENTAL METHODS

Experimentally, the determinations of specific heat were carried out as follows: Early in the work an attempt was made to use a method depending upon the addition by electrical means of a definite amount of heat energy to coal suspended in water, noting the rise of temperature which resulted in the coal and water mixture. Since, by this method, using a weight of coal a little more than one-tenth that of the water, the total energy change of the system was 30 times that of the coal, the experimental error was magnified 30-fold when referred to the coal. Thus, in order to obtain an accuracy of 3 per cent. in the final result, an accuracy of 0.1 per cent. was required in the experiment. This degree of precision being scarcely practicable, the simpler method of mixtures was resorted to, whereby it was possible to effect a comparatively large energy change in the coal itself. The coal was heated to a temperature about 37° above that of the calorimeter, and was subjected thus, on mixing with the water of the calorimeter, to a change of temperature of about 34°, while the water changed less than 3°. The total energy change in the coal was equal to that in the balance of the system or, in other words, one-half of that of the entire system, so that the experimental error became magnified only two-fold when referred to the coal. The determinations were easily checked with a maximum deviation of 2 per cent. from the mean (see Table I).

The calorimeter vessel was a tinned can of 400 cc. capacity resting on a cork in a large Dewar vessel more than twice the height of the can. The Dewar, tube rested in a jar of water at room temperature. A revolving propeller-shaped stirrer was used, attached to a glass rod and operated by a water motor at 150–200 revolutions per minute. The thermometer was a calibrated Beckmann of good quality. For introducing the heated coal or other substance a wide glass tube—about $^3/_4$ inch in diameter—was inserted from above so as to direct the fall of the coal properly, yet not touch the calorimeter can, stirrer, or liquid. The coal was sized between 10 and 20 mesh, so as to fall easily through the tube and mix with the water.

For the heating of the coal before mixing, a double-walled cylindrical glass vessel was used, between the walls of which a little methyl alcohol was kept boiling by a platinum coil electrically heated. The inner cylinder contained the coal, supported near the bottom by a metal plate which could be quickly pushed out, allowing the coal to drop. A calibrated thermometer graduated in tenths of a degree rested in the center of the coal in the inner tube. When the coal had reached a constant temperature, the heater was brought over the calorimeter and the coal dropped.

In order to correct for radiation, temperature readings were taken at one-minute intervals during a 5-minute period before the mixing and during a 10-minute period after the maximum temperature had been reached. The cooling correction was applied by the well-known Regnault method common in calorimetric work. The temperature rise due to the mixing reached its maximum usually in 3 minutes, and the cooling correction applied did not exceed 0.05° or about 2 per cent. of the total rise.

The amount of water used in the calorimeter was 200–230 grams; the water equivalent of the apparatus was determined by runs with pure copper shot assuming the specific heat of pure copper at 28°–67° to be 0.0933—an average of Naccari's values at 17° and 100°, and based also on Schmitz's value (0.0936) at 20°–100°.[1] On this basis the specific heats of a number of other substances were determined in the apparatus, comparing the values obtained with those given by other authorities, and the agreement was found good in most cases (see Table I).

Determinations were made upon four coals of widely varying character, two being high-grade Eastern coals of low moisture content and two from the Interior and Western fields comparatively high in moisture content. They were used both in the air-dry and the moisture-free condition, but not in the unstable moist condition as received from the mine. The moisture-free samples were prepared by drying 100 grams, 10–20 mesh size, for two hours in a 30 mm. vacuum at 100° C.

For the determinations upon dry coal, toluene was used in place of water. The water equivalent of the apparatus containing toluene was redetermined with copper. Some of the determinations made on undried coal in water were repeated in toluene and the agreement was good (see Table I).

[1] See Landolt and Bornstein's Tables (4th Ed.), and the original papers of Naccari, *Atti di Torino*, **23**, 107 (1888); Schmitz, *Proc. Roy. Soc.*, **72**, 177 (1903).

TABLE IV—DESCRIPTION OF COALS

Lab. No.	Source	Loss on air drying	Analysis of air-dried sample: Moisture	Volatile matter	Fixed carbon	Ash
9614	Sun Mine, Fayette Co., W. Va. (New River field)	2.0	1.80	20.38	72.36	5.46
384	Schoenberger Mine, Baird Station, Washington Co., Pa. (Pittsburgh bed)	1.4	1.20	34.50	58.44	5.86
48	Dering No. 11 Mine, W. Frankfort, Ill. (Franklin Co.)	*	8.40	34.95	48.23	8.42
575	Dietz No. 2 Mine, Sheridan Co., Wyo. (sub-bituminous)	14.0	11.00	38.56	40.24	10.20

* Not determined; sample allowed to dry at 20°–25° in air of room.

The specific heats of two coals (9614 and 384) were determined at higher temperature ranges, *viz.*, 130° to 25°, 177° to 25°, and 227° to 25° (see Table I*a*). For preheating the coal there was used a mercury bath heated to the desired temperature and a double-walled glass test tube for transferring the heated coal from the bath to the calorimeter. A thermometer was imbedded in the coal while it was being heated, and temperature read immediately before dropping the coal, correction being made for the exposed stem of the thermometer. About 10 grams of coal were used. The determinations were checked by copper at each temperature. On account of the difficulty of avoiding loss of moisture during the preliminary heating, no determinations were attempted on the Illinois and Wyoming coals at these higher temperatures. Having the average specific heat between 25° and 63° and the average between 25° and 130°, it is possible to calculate the average value between 63° and 130°; similarly also those between 130° and 177° and between 177° and 227° (see Table I*b*).

HEAT EVOLVED BY MIXING DRIED COAL AND WATER

For the approximate determination of the heat evolved by mixing dried coal with water, 12–15 grams of dry or partially dried coal of known moisture content were mixed with 40 cc. of water in a nickel calorimeter cup, and the mixture stirred by hand with the thermometer. The nickel cup rested on a cork in a deep Dewar vessel standing in a water bath. The thermometer was a calibrated Beckmann. The coal, before mixing with the water, was contained in a glass tube with movable bottom, and was brought to the temperature of the water by holding in an air bath of the right temperature until a thermometer imbedded in the coal became constant at the point desired. The tube of coal was then brought over the calorimeter cup and the coal dropped into the water by moving aside the supporting disc at the bottom

With the Wyoming coal the maximum temperature was reached in 2–5 minutes, while with the Illinois coal the action was slower, requiring from 8 to 12 minutes to produce a maximum temperature. The cooling correction was small.

The water equivalent of the apparatus was determined by mixing two portions of water of different temperatures, and was also calculated roughly from the known specific heats of the materials. An average value of 4.5 grams was used. To this was added, in order to obtain the total heat capacity of the system, the weight of water used and an approximate value for the water equivalent of the coal used. The latter value, however, involved an uncertainty due both to the change in specific heat of the coal by combining with water and to the conversion of part of the free water into combined water of a different specific heat. The two errors tend to balance each other and the resultant error was probably not large. Taking 12.5 grams of dry coal, its water equivalent was assumed to be 3.7 grams with a probable error of 0.3 gram, or less than 1 per cent. of the total heat capacity.

The coals used were the same as in the specific heat determinations except that in place of the air-dried sample No. 575, an undried sample from the same mine, No. 43, containing 21.42 per cent. moisture, was substituted in order to observe the full effect of saturation of the coal substance with water. The coals were ground so as to pass a 20-mesh sieve and be retained on a 40-mesh. The moisture in them, determined by the official method of the Bureau of Mines,[1] was as follows:

No. 43	Sheridan Co., Wyo	21.42%
No. 48	Franklin Co., Ill	8.70
No. 384	Pittsburg bed, Pa	1.78
No. 9614	New River district, W. Va	1.15

There is an error due to oxidation in the official method for moisture determination, rendering the results for moisture content, especially in case of the Illinois and sub-bituminous Wyoming types, appreciably lower than the true percentage. Consequently no precise comparisons between heat developed and moisture content can be made from the results in Table III, since the moisture figures are not strictly accurate. The samples used in the determinations (see Table III) were prepared as follows:

Expts. 2, 3, and 4 (Coal 43): 15 gram portions dried for varying periods of time over sulfuric acid at 2 mm. pressure, determining loss of moisture by loss of weight and the moisture remaining by difference between this loss and the total moisture (official method).

Expt. 5: 15 grams of coal saturated with water and then air-dried at room temperature.

Expt. 11: Original coal, air-dried at room temperature 72 hours.

Expt. 7: (Coal 48), dried *in vacuo* over sulfuric acid as in Expt. 4.

Expt. 8: 15 gram portions in weighing bottle, oven-dried at 105°, $1^1/_2$ hours in current of air.

Expts. 9 and 10: Coals 384 and 9614, dried *in vacuo*, as in Expt. 4.

SUMMARY

Coal has a specific heat varying according to the character of the coal substance and the amount of moisture, both free and combined, which the coal contains. The water content affects the specific heat in such manner as to indicate that up to a certain percentage the water is present in a state of physical or chemical combination. Other facts, *viz.*, the low vapor tension of the water in coal and the de-

[1] Techn. Paper, 8, U. S. Bureau of Mines, p. 5.

velopment of heat on moistening dry coal, support this theory. The heat development by the moistening of dry coal may contribute appreciably to the spontaneous heating of some coals in storage.

APPLICATION OF THE DIMETHYL SULFATE TEST FOR DETERMINING SMALL AMOUNTS OF PETROLEUM OR ASPHALT PRODUCTS IN TARS[1]

By Charles S. Reeve and Richard H. Lewis

INTRODUCTORY

The recent production of mixtures of tar with various asphalt and petroleum products for use in the road and paving industry has necessitated a satisfactory method for determining the proportions of each type of material present in such mixtures. The authors have based their research on the well-known dimethyl sulfate test proposed by Valenta[2] and later investigated and reported upon by Graefe[3] and Harrison and Perkin.[4] A. Sommer[5] modified the test in order to render it applicable for the estimation of asphalt in tar-asphalt mixtures, and Chapin[6] now uses it in a modified form for the detection of adulterants in coal tar creosote sheep dips. A brief review of the literature upon the test to date will, therefore, prove of value, before submitting our data.

THE DIMETHYL SULFATE TEST

Valenta's purpose in originating the test was to determine the amount of tar oils present as an adulterant in paraffin and rosin oils, and he claimed as a result of his investigations that while aromatic hydrocarbons are freely miscible with dimethyl sulfate, the open-chain hydrocarbons are entirely insoluble in that reagent. It was, therefore, only necessary to thoroughly agitate a measured volume of the sample under examination with a measured volume of dimethyl sulfate, and the increased volume of the latter would indicate the amount of aromatic hydrocarbons which had gone into solution. This is in general, but not absolutely, correct, and the real facts regarding the test as brought out by later investigations are briefly summed up by Chapin as follows:[7]

"1. Aromatic hydrocarbons and derivatives thereof are completely soluble in dimethyl sulfate, being miscible in all proportions.

"2. Open-chain hydrocarbons show varying degrees of solubility in dimethyl sulfate, ranging from zero to a considerable percentage, light oils as a rule being the more soluble.

"3. Treatment of a mixture of aromatic and open-chain hydrocarbons with dimethyl sulfate results in the formation of two layers; the upper contains most of the open-chain hydrocarbons plus a varying but usually small amount of aromatic hydrocarbons retained in solution; the lower layer contains practically all of the dimethyl sulfate, holding in solution most of the aromatic hydrocarbons plus a varying but usually small amount of the open-chain hydrocarbons.

"The test, therefore, is no absolutely quantitative one, as indeed is to be expected from the laws of solubility. Results obtained thereby may, however, under favorable circumstances, closely approach quantitative values, and so may prove very useful for practical work."

In order to make the test applicable to solid and semi-solid bitumens which could not be directly treated with the reagent, A. Sommer[1] proposed distilling the sample to coke.

"Four cc. of the distillate obtained are then put in a ten cc. graduated cylinder with ground glass stopper and six cc. of dimethyl sulfate are added and shaken thoroughly for one minute. If the distillate is not entirely soluble, separation takes place within a few minutes and a separating mark can be accurately read. The percentage of solubility is calculated from this reading."

His results show that not only the open-chain hydrocarbons but also the distillates from native bitumens —largely polymethylenes—are insoluble in dimethyl sulfate. A mixture of 20 per cent. tar and 80 per cent. asphalt yields a distillate which is 15 per cent. soluble in the reagent, while a mixture of 80 per cent. tar and 20 per cent. asphalt yields a distillate which is 60 per cent. soluble. These are the extreme proportions reported upon by him. The authors, upon attempting to detect 10 per cent. of asphalt in several tar asphalt mixtures, failed to get any indication of its presence by this method, and since some current specifications provide for smaller proportions than 10 per cent., the necessity for a more delicate modification of the method gave rise to the following research.

EXPERIMENTAL

A consideration of the conditions involved in Sommer's method of operation led us to the obvious conclusion that when 10 parts of a hard oil asphalt is fluxed with 90 parts of a fluid coal tar, the total distillate obtained by destructively distilling such a mixture to coke would contain but a very small percentage of anything but aromatic hydrocarbons. Since it is known, and will be again demonstrated, that such distillates are slightly soluble in dimethyl sulfate, such a small percentage in a 4 cc. sample might not be recognized. It was, therefore, thought probable that the procedure suggested by Chapin, *i. e.*, to work on separate fractions of high boiling point, might lead to our finding a fraction in which the proportion of other hydrocarbons to aromatic hydrocarbons would be sufficiently large to permit of the former being easily detected. In order to determine just which fractions might prove of most value, several varieties of products which are being used in tar-asphalt mixtures were first distilled and their higher boiling fractions tested by means of dimethyl sulfate in the method prescribed by Sommer. The distillation was made in an 8-ounce glass retort with the top of the thermometer bulb on a level with the bottom juncture of stem and body of the retort. A 100 cc.

[1] Paper presented at the Eighth International Congress of Applied Chemistry, New York, September, 1912.

[2] *Chem. Ztg.*, **30**, 266–7 (1906).

[3] *Chemische Revue ueber die Fett- und Harz-Industrie*, **14**, 112–5 (1907).

[4] *Analyst*, **33**, 2–11 (1908).

[5] This Journal, **2**, No. 5 (1910).

[6] U. S. Dept. Agric., *Circular* **167**.

[7] *Ibid.*, **167**, 2.

[1] *Loc. cit.*

sample was taken and Table I shows the results obtained. It will be understood that volume percentages are referred to throughout this paper unless otherwise noted.

TABLE I—DIMETHYL SULFATE TESTS ON DISTILLATES

Number of sample	Name of material	270–315° C. Percentage distillate by volume	270–315° C. Percentage insoluble in $(CH_3)_2SO_4$	315–350° C. Percentage distillate by volume	315–350° C. Percentage insoluble in $(CH_3)_2SO_4$	350–375° C. Percentage distillate by volume	350–375° C. Percentage insoluble in $(CH_3)_2SO_4$
1	Oil asphalt Penetration 3.6 mm.	2.2	no test	11.8	80.0	28.8	90.0
2	Oil asphalt Penetration 17.0 mm.	3.7	no test	15.5	85.0	28.0	90.0
3	Oil asphalt Penetration 15.2 mm.	3.2	no test	16.8	87.5	17.2	90.0
4	California oil asphalt Penetration 7.3 mm.	13.0	85.0	32.5	82.5	7.0	75.0
5	Fluxed Bermudez asphalt Penetration 12.2 mm.	4.0	no test	12.0	85.0	17.1	85.0
6	Residual petroleum Specific viscosity Engler 50° C.—65.1	0.6	no test	6.8	90.0	25.2	90.0

It will be noted that in evey case, except No. 4, the dimethyl sulfate insoluble tends to be greater in the higher fractions. The 270–315° C. fractions are too small for making the dimethyl tests with one exception, which in a measure indicates that it would be impossible to detect their presence in such fractions from mixtures containing small amounts of petroleum or asphaltic compounds.

These materials were then mixed with a crude coke-oven tar whose characteristics were as follows:

TABLE II—TESTS ON CRUDE COKE OVEN TAR

General character	Thin fluid
Specific gravity	1.173
Soluble in CS_2	95.23%
Insoluble in CS_2 (free carbon)	4.71%
Ash	0.06%

DISTILLATION

Fractions	Percentage by volume	Percentage by weight	Character of distillates	Insoluble in $(CH_3)_2SO_4$
Water	0.6	0.5	Cloudy	
Up to 110° C.	1.6	1.3	Clear	None
110–170° C.	0.8	0.6	Clear	None
170–270° C.	17.5	15.5	1/2 solid	None
270–315° C.	9.4	8.2	1/3 solid	None
315–350° C.	11.0	9.7	Solid	None
350–375° C.	9.7	8.4	Solid	None
Pitch (over 375° C.)	49.4	55.8	Hard, brittle, glossy	
	100.0	100.0		

The mixtures were made, unless otherwise stated, by adding the given percentage by weight of petroleum or asphaltic product to the weighed amount of tar. The materials when necessary were rendered fluid by heating before mixing and in no case was there any separation of oil apparent. Distillations were then made in the manner above referred to, and fractions were taken at 270° C., 315° C., 350° C., and 375° C. Four cc. of the distillate from each fraction were treated with 6 cc. of dimethyl sulfate in a 10 cc. cylinder graduated to 0.2 cc. The resulting supernatant layer of insoluble oil was read and calculated to its percentage of the volume of sample taken. The results are shown in the following tables.

TABLE III—TESTS ON DISTILLATES FROM 10% MIXTURE

No. of sample	Mixture	315–350° C. fraction Percentage distillate	315–350° C. fraction Percentage insoluble in $(CH_3)_2SO_4$	350–375° C. fraction Percentage distillate	350–375° C. fraction Percentage insoluble in $(CH_3)_2SO_4$
1	90% coke oven tar 10% oil asphalt No. 2	11.5	5.0	17.5	25.0
2	90% coke oven tar 10% fluxed Bermudez asphalt	12.0	5.0	13.2	12.5
3	90% coke oven tar 10% residual petroleum	11.6	10.0	13.0	22.5
4(a)	Refined tar preparation Free carbon = 19.36%	14.0	15.0	12.2	18.0

(a) Said to contain 10% oil asphalt.

In these 10 per cent. mixtures an excellent indication of the presence of oil asphalt is shown by the dimethyl sulfate test even in the 315–350° C. fraction, but there was no trace of open-chain compounds in the 270–315° C. fraction. No evidence of an oil product could be found in the total distillate obtained by distilling to coke by Sommer's method.

On account of the ease with which 10 per cent. could be detected by testing separate fractions, the test was not carried out with all the materials.

TABLE IV—TESTS ON DISTILLATES FROM 5% MIXTURES

No. of sample	Mixture	315–350° C. fraction Percentage distillate	315–350° C. fraction Percentage insoluble in $(CH_3)_2SO_4$	350–375° C. fraction Percentage distillate	350–375° C. fraction Percentage insoluble in $(CH_3)_2SO_4$
1	95% coke oven tar 5% oil asphalt No. 1	9.2	None	9.4	7.5
2	95% coke oven tar 5% oil asphalt No. 2	15.5	None	9.7	12.5
3	95% coke oven tar 5% oil asphalt No. 3	11.0	2.5	10.2	12.5
4	95% coke oven tar 5% Calif. oil asphalt	9.6	None	10.8	10.0
5	95% coke oven tar 5% fluxed Bermudez asphalt	11.0	None	10.8	8.0
6	95% coke oven tar 5% residual petroleum	8.5	7.5	12.5	12.5

In these 5 per cent. mixtures, the asphaltic base can still be detected in fairly large percentage in the last fraction (350–375° C.), but in only two cases (No. 3 and No. 6) out of six was there present a dimethyl sulfate insoluble in a lower fraction (315–350° C.).

TABLE V—TESTS ON DISTILLATES FROM 3% MIXTURES

No. of sample	Mixture	350–375° C. fraction Percentage distillate	350–375° C. fraction Percentage insoluble in $(CH_3)_2SO_4$
1	97% coke oven tar 3% oil asphalt No. 1	9.7	5.0
2	97% coke oven tar 3% oil asphalt No. 2	8.9	5.0
3	97% coke oven tar 3% oil asphalt No. 3	8.7	4.0
4	97% coke oven tar 3% Calif. oil asphalt	7.8	2.5
5	97% coke oven tar 3% fluxed Bermudez asphalt	8.1	1.25
6	97% coke oven tar 3% residual petroleum	10.4	10.0

In the 350–375° C. fraction of the distillations of the 3 per cent. mixture, the insoluble residue ranges

from 10 per cent. to 1.25 per cent. so that the detection of even three per cent. of asphalt is made possible by the method of fractionation which has been employed in this work. The 315° C. to 350° C. fraction did not give the slightest trace of insoluble products.

CONCLUSION

While the above results do not give any absolute idea of either the amount or exact nature of the material which has been fluxed with the tar, the authors feel that the improvement which they have made upon former methods may still prove of value, until a more exact method shall be forthcoming.

Specifications for tar-oil or tar-asphalt mixtures will be useless without some method of checking up the material submitted under them. We now think it possible, however, by specifying the grade and character of both the tar and petroleum or asphalt product to be used, and then obtaining data on similar laboratory mixtures, that we can determine with reasonable certainty whether our specifications are being fulfilled. In order to more closely determine the character of the material which has been fluxed with a tar, a volatilization test in conjunction with the dimethyl sulfate test will no doubt prove of great importance, and we feel that some accumulated data on these two tests will place us in a much safer position for specifying and identifying bituminous mixtures which present such difficulty to the chemist.

U. S. Office of Public Roads
Washington, D. C.

THE DETERMINATION OF OXYGEN IN METALLIC TUNGSTEN POWDER AND SOME NOTES ON THE DETERMINATION OF OXYGEN IN STEEL

By Charles Morris Johnson

Received January 22, 1913

It has been found a distinct advantage both in the manufacture and use of tungsten powders to know their oxygen content. In one of the laboratories under the author's direction, this determination is a matter of daily routine. The method involves the same principle used in the determination of oxygen in steel; *i. e.*, the ignition of the substance in a stream of hydrogen, which method is credited to Ledebur.

The electrically heated furnace introduced by the author[1] in 1908 for the direct determination of carbon in iron, steel and alloys is utilized in the process which is described in detail in this paper.

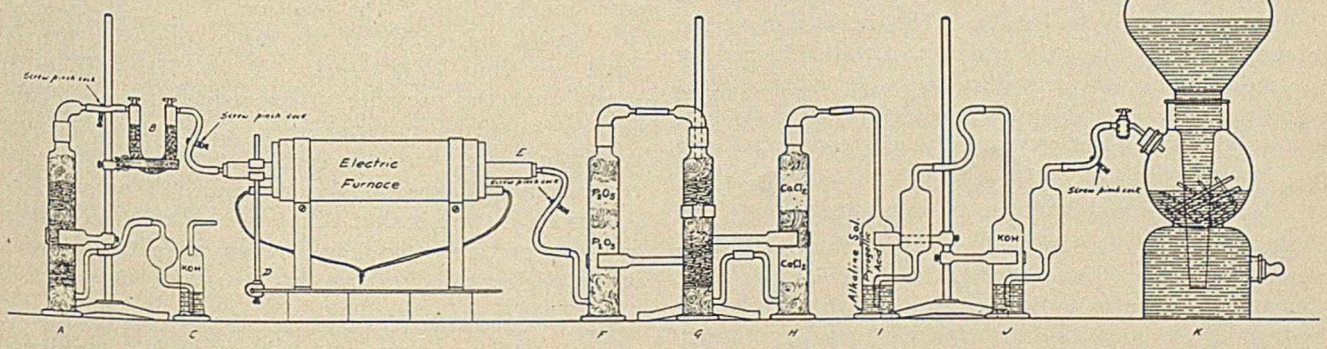
Walker and Patrick,[2] in a paper read at the Eighth International Congress of Applied Chemistry, attack the accuracy of the Ledebur method on the ground that any oxides of manganese or silicon present in the steel would not be reduced. The author regards the Ledebur method as more practical than the proposed new one[2] above noted; even if the former process does not reveal the total oxygen present, it certainly shows enough of it to furnish a basis for judgment of the quality of the steel. If the steel is sufficiently dirty and poorly melted in actual open-hearth Bessemer or crucible practice to contain oxides of manganese and silicon, then it would surely contain enough oxide of iron to condemn it.

APPARATUS

The arrangement of apparatus is indicated in the drawing and the accompanying notes. The towers (or jars) are the author's design as are also J, I and C, and were first used as part of a combustion train.[3] In this laboratory four furnaces are placed side by side. By the use of a Y tube at the outlet of jar F, one train from F to K can be made to serve two furnaces. Of course a separate set of A, B and C is necessary for each furnace. If, after making a large number of determinations, the blank begins to show a gradual increase, the contents of the various jars must be renewed.

METHOD

Blank.—Before introducing anything into the electric furnace, close all points marked "screw pinch cock." At B make a connection with a straight glass tube instead of the U tube shown. Insert



Apparatus

A—Ignited asbestos and dry stick KOH.
B—Glass wool plugs and P_2O_5 (anhydrous powder)
C—Solution of KOH.
D—A clamp to prevent the quartz tube from moving since it was found that results varied if the tube moved back and forth.
E—Quartz tube.
F—Ignited asbestos and P_2O_5 in alternate layers.
G—Ignited asbestos and dry stick KOH in alternate layers.
H—Asbestos and small lump (size of pea) $CaCl_2$ in alternate layers.
I—50 cc. alkaline solution of pyrogallic acid.
J—50 cc. KOH solution.
K—Kipp generator (2 quart)

Solutions

Potassium hydroxide: 1 gram KOH to 1 cc. water.
Pyrogallic acid: 3 grams pyrogallic acid and 2 cc. water to 3 cc. KOH solution.
Kipp Generator: Use stick zinc and dilute hydrochloric acid (1 : 1).

[1] *J. Am. Chem. Soc.*, **30**, 773.
[2] *Proc. 8th Intern. Cong. Appl. Chem.*, **21**, 139; also This Journal, **4**, 799.
[3] *J. Am. Chem. Soc.*, **28**, 862.

quickly into the quartz tube (at the point marked E) the porcelain boat that has been kept at 105° C. in an air bath. Push the boat into the center of the furnace with a heavy copper wire which is marked to show how to place the boat in the hottest part of the furnace. Stopper the tube as quickly and tightly as possible. Open all four pinch cocks and turn on the hydrogen slowly until it passes through the apparatus at the rate of about seventy (70) bubbles per minute. Allow the hydrogen to pass shrough the cold furnace for thirty minutes. Close all the pinch cocks and replace the glass tube at B by the U tube. Open all cocks and let hydrogen run for another half hour to fill the weighing apparatus with this gas. Close all pinch cocks and the glass cocks on the U tube. Remove the U tube and weigh it quickly. Insert the U tube again, open all cocks and start the hydrogen flow: turn on the electric current in the furnace and bring up the temperature to 950–1000° C. After reaching this temperature keep the heat on for two hours with the hydrogen passing continually. Close all pinch cocks, shut off the hydrogen, and close the glass cocks on the weighing apparatus B. Detach and weigh B. The difference between this weight and the first weight represents the blank to be deducted from all determinations.

Sample.—Dry the finely ground powder of the tungsten metal to constant weight at 105° C. Put two or three grams of the powder into a porcelain boat that has been dried at 105° C. Place this in the cold furnace and stopper tightly at E. Using the glass connection at B, open the pinch cocks and allow hydrogen to pass through the cold furnace for one-half hour to remove whatever air entered when the charge was inserted. Close all pinch cocks and replace the glass tube by the weighed U tube at B. Open all cocks, adjust the hydrogen flow to 70 bubbles per minute and turn on the electric current, heating the furnace to 950–1000° C. Maintain this temperature for two hours with the hydrogen passing. Close all cocks and turn off the hydrogen. Remove and weigh the U tube. The increase in weight minus the blank gives the amount of water formed by the reduction of the metallic oxides to metal. This result multiplied by 16 and divided by 18.016 is equivalent to the weight of oxygen which is converted into percentage by the usual calculations.

STANDARDIZATION OF APPARATUS

WITH C. P. TUNGSTIC OXIDE.—This material is prepared as follows: Treat five grams of 96–98 per cent. tungsten powder in a platinum dish with 10 cc. C. P. hydrofluoric acid. Pour on this mixture very slowly 30 cc. of concentrated nitric acid. This produces considerable heat, and the material is dissolved as clear as water. Now add 15 cc. of concentrated sulfuric acid, evaporate to thick fumes, cool, add 10 to 20 cc. C. P. hydrochloric acid, boil three to four minutes, add 50 cc. of water, heat, filter, and wash free from iron and sulfates by decantation in a 600 cc. beaker. Transfer to a platinum dish, ignite at a bright red heat in a muffle, and put in a glass-stoppered bottle. Before using any of this material for a test, ignite a portion of it at a blast-lamp temperature. Immediately after the blasting put one gram of the oxide in a porcelain boat dried at 105° C., and charge it at once into the furnace. It will require at least six hours treatment at 950–1000° C. to reduce this amount of oxide and carry all of the water formed over into the weighing apparatus.

WITH FERRIC OXIDE.—Dissolve 10 grams of low-carbon steel of very low phosphorus, sulfur and silicon content in 100 cc. hydrochloric acid in a liter beaker. Transfer this to a No. 7 porcelain dish and evaporate to 10 cc. Add 100 cc. nitric acid and evaporate to 20 cc. Add 50 cc. of concentrated nitric acid, and evaporate to dryness. Place the dish in a muffle and heat to redness. Cool, dissolve in 50 cc. hydrochloric acid, add 50 cc. of water; evaporate to small volume, filter out insoluble matter, such as silicic acid, and precipitate with filtered ammonia. Wash the precipitate by decantation until free from chlorides, dry in a porcelain dish, heat to redness, and place in a stoppered bottle. Blast a portion of this for three or four minutes, transfer 1 gram quickly to a porcelain boat, and place at once in the reduction furnace. Pass hydrogen for six hours after the furnace reaches 950–1000° C.

TABLE I—RESULTS OBTAINED BY APPARATUS DESCRIBED

Pure WO_3, 20.69 per cent. oxygen
1 gram gave 20.70 per cent. oxygen
0.250 gram gave 20.80 per cent. oxygen
0.500 gram gave 20.30 per cent. oxygen

Average, 20.60 per cent. plus

Pure Fe_2O_3, 30.05 per cent. oxygen
0.500 gram gave 30.16 per cent. oxygen
Blanks, 0.0030 and 0.0036

THE EFFECT OF FREE CARBON ON THE METHOD

It is an advantage to have some excess of free carbon in finished tungsten powder, and, at times, in the process of manufacture, it is necessary to know the amount of oxygen present in a powder that contains as much as three or four per cent. of charcoal. Some tests were made to see if the reaction $WO_3 + 3C = W + 3CO$ might not occur at the same time with the desired reaction $WO_3 + 6H = W + 3H_2O$. Table II shows that the presence of excessive amounts of free carbon caused no material error in the case of the pure tungsten oxide, but did cause low results when the carbon content exceeded five per cent. in the iron oxide. A curious feature is that thirty per cent. of free carbon caused practically no lower result than the addition of ten per cent.

TABLE II—RESULTS OBTAINED WITH MIXTURES OF OXIDES AND CHARCOAL

Grams of mixture		Percentage Oxygen theoretical	Percentage Oxygen found	Percentage Carbon present
WO_3	Charcoal			
0.544	0.201	20.69	20.57	26.6
0.300	0.090	20.69	19.80	23.0
0.400	0.080	20.69	20.32	16.7
0.500	0.060	20.69	20.53	10.7
Fe_2O_3				
0.5785	0.000	30.05	29.84	None
0.473	0.208	30.05	27.35	30.5
0.400	0.122	30.05	28.68	23.3
0.300	0.089	30.05	27.53	23.0
0.500	0.025	30.05	29.95	4.7
0.500	0.050	30.05	27.82	9.7

The following table shows the amounts of oxygen found in the various brands of tungsten powders made both in the U. S. and abroad. Each numeral represents a different make.

The reduction was particularly poor in the second lot received from the German manufacturer designated as II (No. 2 in his second shipment). When so much oxide is present it can be easily detected by the eye, being equivalent to 10.92 per cent. of tungstic oxide. Such so-called metal has a distinct brown color.

TABLE III

Make	Imported or domestic	Oxygen found Per cent.	Consignment
I	German	1.02	.....
II	German	1.10	No. 1
II	German	2.26	No. 2
III	German	0.18	.....
IV	German	0.53	.....
V	American	0.37	No. 1
V	American	0.47	No. 2
V	American	1.24	No. 3
VI	American	0.80	.....
VII	American	0.45	.....
VII	American	0.08	.....
VII	American	0.07	.....

THE DECARBONIZATION OF STEEL WHEN IGNITED IN A STREAM OF HYDROGEN FOR THE OXYGEN TEST

In 1909 the writer called attention to the fact that hydrogen will produce a bark, or decarbonized surface, on steels when the latter are heated in a current of this gas. Supplementing this statement[1] the following tests were made on three steels that were analyzed for oxygen:

TABLE IV

	Grams of drillings taken for the oxygen test	No. of hours ignited in hydrogen	Percentage carbon content: Before ignition	Percentage carbon content: After ignition
Sample I.....	9.6	3½	1.04	0.90
Sample II.....	16.0	3½	1.08	0.83
Sample III.....	19.5	3½	0.83	0.70

In the foregoing method, no preheating furnace or tube is used such as was recommended by Ledebur in his "Leitfaden für Eisenhütten-Laboratorien" and adopted by others who have since written on this or similar subjects, thus simplifying matters to that extent. Also, concentrated sulphuric acid is omitted, entirely, eliminating the possibility of unpleasant, not to say, dangerous accidents from this source.

The introduction of an alkaline solution of pyrogallol into the purifying train was made at the suggestion of Mr. Simon Lubousky, in July, 1912, when working under the author's direction. The latter adopted his suggestion as did Mr. McMillen,[2] of the Crescent Steel Works, who was the first to apply the electrically heated furnace, introduced by the writer, to the determination of oxygen.

LABORATORY OF THE PARK WORKS
CRUCIBLE STEEL CO. OF AMERICA
PITTSBURGH, PA.

[1] See "The Formation of White Scale on Steel and the Surface Decarbonization of Pipe-Annealed Steel," THIS JOURNAL, **1**, 459 (1909).
[2] *Met. and Chem. Eng.*, **11**, No. 2; also THIS JOURNAL, Feb., 1913.

THE DETERMINATION OF PHOSPHORUS IN FERRO-TUNGSTEN, METALLIC TUNGSTEN POWDER, TUNGSTEN OXIDE AND TUNGSTIC ACID BY DIRECT SOLUTION

By C. M. JOHNSON

Received February 7, 1913

The author found in the course of an investigation that the practice of decomposing tungsten-bearing materials by fusing them with a mixture of sodium carbonate and potassium nitrate, leaching out the fusion, acidulating with hydrochloric acid, removing the tungstic acid by several evaporations to dryness, and then using the filtrate and washings from the tungstic acid for the determination of the phosphorus, gave far less of the latter element than was actually present. He then devised the following method which he has found to give near enough to the true phosphorus for technical purposes.

FERRO-TUNGSTEN.

Add 30 cc. of concentrated nitric acid to one gram of the powdered sample, in a platinum dish; then add slowly 3 cc. of C. P. hydrofluoric acid. Keep the dish covered with a watch glass; warm the mixture. After warming and slight boiling, the material should dissolve to a clear solution. Transfer the solution to a No. 5 porcelain dish and evaporate to dryness; do not bake as there is danger of losing phosphorus at this point. Dissolve this residue with 50 cc. of concentrated hydrochloric acid. Heat with the lid on; then remove the lid and evaporate to dryness; do not bake. Dissolve again, using 20 cc. of concentrated hydrochloric acid; heat, add 50 cc. of water, stir, heat and filter out the main tungsten; wash with one part of concentrated hydrochloric acid diluted with twenty parts of water. Evaporate the filtrate and washings to 10 cc., add 20 cc. of water, stir and filter as before. Evaporate to 10 cc., add 75 cc. of concentrated nitric acid and heat with the cover on until all action is over; remove the lid and evaporate to 20 cc. Add 50 cc. of nitric acid, and evaporate to 15 cc. Add 20 cc. of water, stir, heat and filter into a 6 oz. beaker; wash with two cc. of concentrated nitric acid diluted with 100 cc. of water, washing fifteen times. Evaporate the filtrate and washings in the beaker to 40 cc. Replace the lid and add a slight excess of five per cent. solution of potassium permanganate; boil three or four minutes. Dissolve the excess of manganese oxide with a little ferrous sulfate, and precipitate the phosphorus with molybdate solution.

When dissolving ferro-tungsten in the mixture of nitric and hydrofluoric acids, a porcelain dish can be used, but a little more hydrofluoric acid may be needed to secure complete solution of the alloy on account of the tendency of the latter acid to attack the dish. Further, when a porcelain dish is used, blanks must be run, using a standard steel. The latter is dissolved in the *mixture* of the two acids and the phosphorus determined, using the porcelain dish. If the standard is found to run higher than it should, the deduction necessary to correct it constitutes the blank to be subtracted from the phosphorus found in the sample.

Any method with which the writer is acquainted, using a carbonate and niter fusion of materials contain-

ing tungsten for the purpose of obtaining the percentage of phosphorus therein, gives only a fourth, or less, of the actual content of the latter element. The following results are only a few of those obtained in this laboratory and are given in proof of the above statement.

COMPARISON OF FUSION AND EXTRACTION METHODS

Sample	Percentage author's extraction method	Phosphorus found by fusion with $Na_2CO_3 + KNO_3$
Ferro-tungsten No. 18	0.322	0.096
	0.350	0.088
	0.345	
	0.330	
A high phosphorus pig iron (0.73 per cent. P)—0.5 gram tungsten powder (98 per cent. pure) per gram of iron	0.70	0.098
		0.095
"Tungsten cake"	0.101	0.008
	0.102	
Tungsten powder oxidized to WO_3 at low red heat before extraction	0.113	0.007

TUNGSTEN ORES

Here the procedure differs only in the manner by which decomposition is effected. Grind the ore to the finest possible state of division; extract at nearly boiling temperature with 100 cc. concentrated hydrochloric acid in a No. 5 porcelain dish. About every thirty minutes, add 0.1 gram additions of $KClO_3$; on each addition of chlorate stir the sample off the bottom of the dish with a glass rod. Continue the heating, addition of chlorate and stirring until the tungsten ore has changed from a brown color to yellow in case of the dark ores; or from a light gray or brown to a very bright yellow in case of scheelite ore. Evaporate to dryness; cover, add 50 cc. concentrated HCl, heat 10 minutes to dissolve the iron and manganese, add 50 cc. of water and heat 15 minutes to allow the tungstic acid to separate well; cool and mix in some paper pulp. Filter through a double filter; wash with one part of hydrochloric acid diluted with twenty parts of water. Evaporate the filtrate and washings to 5 cc. and add 75 cc. of concentrated HNO_3; heat with the cover on the dish until all red fumes are gone and no further spraying occurs; remove the cover again and evaporate to 10 cc.; add 50 cc. of concentrated nitric and evaporate to 10 cc. again; dilute with 15 cc. of water and mix well. Filter into a 6 oz. beaker; wash with a one per cent. by volume solution of nitric acid, fifteen or twenty times. Evaporate the filtrate and washings to 40 cc. in the beaker; boil with a slight excess of permanganate solution. Add just enough ferrous sulphate to clear the excess of the hydrated oxide of manganese and boil again five minutes. Add 50 cc. of molybdate solution to the hot fluid in the beaker and finish the analysis as given for ferro-tungsten.

By careful heating and small additions of the chlorate, together with further applications of acid, if necessary, many dark ores can be so completely decomposed as to attain a clean orange color. The more complete the decomposition, the more perfect will be the extraction of the phosphorus. The hard black ferberites are the slowest to yield and take on the yellow color. The decomposition can be done to the best advantage at a low-digesting heat and will require at least 5 or 6 hours.

This somewhat lengthy method is the only one that the author has found reliable, thus far, for technical purposes in tungsten ores. The latter may contain all the way from slight traces up to 0.500% phosphorus. The fusion method with these ores gives just as low results as with the ferro-tungsten. The cause of the low results is the formation of phosphotungstic acid; this is carried from the solution with the main tungstic acid that forms when the sodium tungstate is decomposed by acidulation and evaporation with acid.

METALLIC TUNGSTEN POWDER, TUNGSTIC OXIDE AND TUNGSTIC ACID

Ignite the tungsten powder at a red heat with frequent stirring until it is all converted to the yellow oxide. Then extract exactly as in tungsten ore for at least six hours and finish according to the ore method.

The original oxidation is best accomplished by weighing the sample into the dish in which the extraction is to be made and then placing dish and all in a muffle which is at a low red heat.

Tungstic acid and oxide do not require heating to redness. Their analysis for phosphorus is exactly like that for ores beginning with the hydrochloric acid-chlorate treatment.

Reserve the tungsten residues that are filtered out after the extractions and evaporations for the tungsten determination. The purification of these residues will be made the subject of a later publication.

NOTE.—It may be well in this article to caution those who have occasion to determine the phosphorus in molybdenum compounds, that any molybdic acid separating out of acid solutions containing phosphorus will carry a considerable amount of the latter element out, forming the analogous compound, phosphomolybdic acid.

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A COLORIMETRIC METHOD FOR THE DETERMINATION OF CHROMIUM IN STEEL

By FRANK GARRATT

Received January 6, 1913

A colorimetric method for the determination of chromium in steel, based on the use of Koenig's organic chromium reagent,[1] has been in use in the writer's laboratory for some time, and has given such satisfaction that it was deemed worthy of publication. The organic reagent used, disodium 1.8-dihydroxynaphthalene 3.6-disulfonate, is extremely sensitive to chromate solutions, and the method has been very useful for detecting chromium and determining what might be considered small percentages of this element.

The usual colorimetric methods for chromium have not found application for this determination in steel.

[1] *Chem. Ztg.*, **35**, 277 (1911).

Hillebrand[1] has made use of the color imparted to alkaline solutions by hexavalent chromium for the determination of this element in rocks. This method applied to steels, however, would require too large quantities of the metal to be handled conveniently for routine determinations, especially for small percentages, as the yellow color of the chromate does not lend itself to comparison so handily as the pink color obtained in the author's method. Moulin[2] has made use of the fact recorded by Cazeneuve that acetate of diphenylcarbazide gives a purple color with soluble chromates. The writer does not know of its being used for the determination of chromium in steel, however, nor could it be so conveniently applied as the colorimetric method described in this paper. The well-known qualitative test, making use of hydrogen peroxide and ether for the detection of chromic acid and chromates, is not capable of use, of course, as the blue color of the perchromic acid is not permanent.

The writer has occasion to make numerous determinations where the chromium runs 0.20 per cent. or under, and the results obtained by the method given below have been much more reliable than those obtained by other methods. The accurate determination of these small percentages of chromium is usually more troublesome than the larger quantities, but the new method takes care of these amounts very nicely. For determining the small quantities of chromium noted above, the writer has heretofore used McKenna's method[3] and thinks it is uniformly more reliable than the other methods in vogue. The employment of concentrated nitric acid solutions and potassium chlorate is rather objectionable, however, and the method is not as convenient to handle as the colorimetric procedure described herewith. The color method is based on the fact that disodium 1.8-dihydroxynaphthalene 3.6-disulfonate gives pink to cherry-red colors when added to acid solutions of chromate salts. It is short and accurate, and as used in the writer's laboratory on routine work, is as follows:

From 0.2 to 0.4 gram of steel, depending on the amount of chromium present, is dissolved in 10 cc. of dilute sulfuric acid (1 : 3) in a 400 cc. Erlenmeyer flask. For steels containing 0.15 per cent. chromium or under the writer uses 0.4 gram of steel; for larger percentages than this 0.2 gram is sufficient to work on. When solution is complete, about $^1/_2$ cc. of concentrated nitric acid is added, and the solution boiled to near dryness to expel nitrous fumes and nitric acid. No more nitric acid is used than is necessary to oxidize the iron, as the presence of much free nitric acid interferes with the chromium color by giving it a brown tint. It is best to use a small marked tube that will deliver just about $^1/_2$ cc. for each determination. After driving off the nitric acid, 50 cc. of a 10 per cent. solution of sodium hydroxide are added, followed by the addition of about 1 gram of sodium peroxide, after which the solution is boiled for about five minutes. Since the reagent gives a color with only chromate solutions, there should never be any free peroxide left in the flask, as the subsequent acidification would cause a reduction of the chromium compound to chromic sulfate. It has always been found, however, that five minutes boiling is sufficient to decompose the excess of peroxide. The solution is cooled to room temperature and diluted to 200 cc. in a volumetric flask; 100 cc. are filtered from this solution, and acidified by adding 2 cc. of 85 per cent. phosphoric acid, and 8 cc. of concentrated sulfuric acid. The concentrated sulfuric acid is purposely used to heat the solution, as the color, on the addition of the reagent, develops more quickly in a warm solution than in a cold one. The solution should not contain more than 20 per cent., by volume, of concentrated sulfuric acid, as stronger solutions than this seem to bleach the pink color. Immediately after the acidification, 2 cc. of a 1 per cent. aqueous solution of disodium 1.8-dihydroxynaphthalene 3.6-disulfonate are added. A pink to cherry-red color develops, depending on the amount of chromium present. The solution is allowed to stand for at least 15 minutes, and is then compared with a standard steel that has been treated in the same manner as the test.

The comparisons are usually made in a set of Camp's comparison tubes, although with very small percentages (say under 0.10) it is usual to use Nessler tubes. If a 0.20% standard is used it is diluted to 200 cc., so that each cubic centimeter represents 0.001 per cent. chromium. The writer prefers to use standard potassium bichromate solution added to a chromium-free steel as a source of standard, as the standard may then be varied to suit the composition of the steels being analyzed.

There is a very slight retention of some of the chromium by the iron precipitate. Experiments made along this line by taking samples through by the method described, and then comparing against pure solutions of potassium bichromate in sulfuric acid, indicated that about 95 per cent. of the true chromium value was obtained. The chromium recovery was very uniform, however, and since the standard is given the same treatment as the test the results should be accurate within practical limits. That this is true is shown by Table III.

The reagent gives a green color with ferric solutions, but this is instantly destroyed by adding phosphoric acid or strongly acidifying with sulfuric acid. The results have been found to be more reliable, however, in the absence of iron, and for this reason the precipitated ferric hydroxide is removed by filtration. Tungsten and molybdenum do not interfere with the determination of the chromium in any way whatever. The addition of phosphoric acid prevents the precipitation of tungstic acid, and thus gives clear solutions for comparison.

Vanadium interferes with the method, however, by imparting a brownish tint to the pink chromium color. Pure vanadate solutions give light yellow to amber-colored solutions, depending on the amount of vanadium present. When determining chromium

[1] *J. Am. Chem. Soc.*, **20**, 227 (1898).
[2] *Bull. Soc. Chim.*, **31**, 295 (1904).
[3] *Chem. News*, **80**, 2, 67.

with moderate amounts of vanadium present the characteristic chromium color is but slightly changed, but the results are high as shown by Table I. The samples were compared against a 0.30 per cent. chromium standard that contained no vanadium.

TABLE I

Sample no.	Per cent. vanadium present	Per cent. chromium present	Per cent. chromium obtained	Per cent. error
1	0.10	0.20	0.23	+0.03
2	0.10	0.30	0.31	+0.01
3	0.15	0.30	0.35	+0.05
4	0.15	0.30	0.34	+0.04
5	0.20	0.30	0.37(*a*)	+0.07

(*a*) Difficulty in comparing, owing to the solutions being of different shade.

The results in Table II also show the influence of vanadium on the determination of the chromium. The samples were compared against a 0.25 per cent. chromium standard that contained 0.15 per cent. vanadium. It will be noted that the samples containing more than 0.15 per cent vanadium were high, while those containing less than this amount were low.

TABLE II

Sample no.	Per cent. vanadium present	Per cent. chromium present	Per cent. chromium obtained	Error
6	0.10	0.20	0.18	—0.02
7	0.10	0.20	0.18	—0.02
8	0.10	0.25	0.23	—0.02
9	0.10	0.30	0.24	—0.06
10	0.20	0.20	0.22	+0.02
11	0.20	0.20	0.21	+0.01
12	0.20	0.25	0.28	+0.03
13	0.20	0.30	0.34	+0.04

The color due to vanadium is only obtained in acid solutions of the pentavalent compounds, and is quite characteristic. As a matter of fact, a few experiments made with pure vanadate solutions indicate that the reaction is quantitative, and is almost as sensitive as the well-known hydrogen peroxide test for this element.

The addition of the reagent to slightly acid solutions of quadrivalent titanium gives brick-red colors that are destroyed by adding hydrofluoric acid, or strongly acidifying with mineral acids. Concentrated sulfuric acid solutions of titanium, however, give pink colored solutions. The reaction for titanium is much more sensitive than the hydrogen peroxide test, but the percentage of free acid present has a very marked influence on the color, and it is not thought that a reliable quantitative method could be developed, owing to this fact. Titanium would not interfere with the chromium determination in the method as given, of course, as any titanium present would be precipitated and removed by filtration.

The writer does not think that the method given above is more advantageous for determining large percentages of chromium in steel (say over 0.60 per cent.) than Galbraith's or some such modification, but it seems to be particularly suited for small percentages of this element in steel.

Table III shows some results obtained by the new method.

The result on sample No. 14 was obtained by making an ether separation of the ferric chloride solution, and then replacing the hydrochloric acid with sulfuric acid. The chromium was then oxidized with sodium peroxide in an alkaline solution, and the small amount of ferric hydroxide removed by filtration. The solution of sodium chromate was then acidified with sulfuric acid, the chromium reagent added, and the color matched against a pure solution of potassium bichromate in sulfuric acid.

TABLE III

Sample no.	Weight of steel taken Grams	Per cent. tungsten present	Per cent. molybdenum present	Per cent. chromium taken	Per cent. chromium obtained	Error
14	2.0	..	..	0.012	0.013	+0.001
15	0.4	..	..	0.03	0.03	none
16	0.4	..	..	0.06	0.06	none
17	0.4	..	..	0.09	0.09	none
18	0.2	..	..	0.15	0.15	none
19	0.2	..	..	0.20	0.20	none
20	0.2	..	..	0.30	0.31	+0.01
21	0.2	..	..	0.50	0.49	—0.01
22	0.2	..	..	0.70	0.70	none
23	0.2	..	..	1.10	1.10	none
24	0.2	..	..	1.40	1.38	—0.02
25	0.4	5.5	..	0.20	0.20	none
26	0.4	5.5	..	0.30	0.29	—0.01
27	0.4	20.0	..	0.20	0.20	none
28	0.4	20.0	..	0.25	0.24	—0.01
29	0.4	..	5.0	0.20	0.18	—0.02
30	0.4	..	5.0	0.30	0.29	—0.01
31	0.4	..	10.0	0.20	0.18	—0.02
32	0.4	..	10.0	0.25	0.25	none

By taking larger quantities of steel, as in the case of sample No. 14, there is no difficulty in determining 0.001 per cent. chromium. Such a minute quantity of chromium in steel has no metallurgical significance whatever, of course, but the analyst is often called upon to look for very small quantities of metals, and it is a pleasure to have a method that will detect them without difficulty.

The writer is indebted to his assistant, Mr. D. J. Giles, for performing much of the experimental work leading up to the perfection of this method.

FIRTH-STERLING STEEL CO.
MCKEESPORT, PA.

WATERS OF THE BREITENBUSH HOT SPRINGS, OREGON[1]

By WALTON VAN WINKLE[2]

Received January 16, 1913

Breitenbush Hot Springs are near Breitenbush Creek on the west slope of the Cascade Mountains in Marion County, Oregon. Their exact elevation is unknown, but it is probably somewhat more than 2000 feet above sea level. The valley of the Breitenbush is narrow and the formations exposed are lavas of the Cascade Range series. Faults at the springs were not observed by Mr. Finkbiner, though possibly the waters issue from a fault. More than 60 hot springs, in three general groups, lie along both sides of the stream for almost a third of a mile. A few cold springs, entirely different in character, are within the limits of the groups, but as they are normal for the region they are unimportant. All the hot springs have nearly the same temperature and the manner of their grouping suggests a common origin. Reputed curative properties have been assigned to the various springs, and a crude "health resort" has been con-

[1] Published by permission of the director of the U. S. Geological Survey, dated January 4, 1913.

[2] Field work by N. M. Finkbiner.

structed. Many patients take the waters each year, and if the place were more readily accessible it would undoubtedly enjoy a large patronage.

In September, 1912, Mr. N. M. Finkbiner, of the laboratory of the U. S. Geological Survey at Salem, Oregon, described 23 of the springs, and made field determinations of the temperature and alkalinity of each. He also collected from nine of these, from one cold spring, and from Breitenbush Creek above and below the springs, samples of water, which were analyzed conjointly at Salem by the writer, N. M. Finkbiner, and S. C. Dinsmore, state chemist of Nevada. The accompanying table gives results of both field and laboratory tests of these samples; the other 14 springs, field tests of which were made but are not reported, furnish water of similar character.

Analyses of the Waters of Breitenbush Hot Springs, Oregon
(Parts per million unless otherwise designated)

	A	B	C	D	E	F	G	H	I	J	K	L
SiO_2	141	134	142	138	144	141	151	147	142	142	24	24
Fe	0.85	0.10	0.17	0.20	0.10	0.10	0.80	0.10	0.05	0.27	0.70	0.01
Ca	99	93	89	90	86	95	93	97	96	93	5.2	4.4
Mg	1.7	1.5	1.2	1.5	1.5	1.5	1.0	0.8	...	1.4	2.4	1.1
Na	735	...	...	...	733	...	...	...	...	739	8.7	6.9
K	41	...	...	...	41	...	...	...	...	41		
CO_3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	...	0.0	0.0	0.0
HCO_3	154	116	128	127	128	141	128	146	...	133	27	24
SO_4	143	137	138	137	137	135	139	137	133	137	1.2	2.3
Cl	1,138	1,128	1,135	1,120	1,133	1,115	1,143	1,145	1,120	1,131	2.3	2.0
NO_3	tr.	...	...	...	tr.	...	...	...	...	tr.	0.26	tr.
Total dissolved solids at 180°	2,470	2,380	2,408	2,379	2,423	2,384	2,433	2,434	2,396	2,412	67	54
Temperature, degrees C.	67	59	73	73.5	73	69	69	83	66	70	13	...

All samples collected Sept. 27, 1912.

A. Spring near right bank of creek at NE end of group. Flows from crevice in rock. Flow about "size of wrist."

B. "Arsenic spring" (no arsenic present) near left bank of creek about 600 feet SW from A. Flow one-half that of A.

C. Spring 43 feet from B and 84° NE of A. Flow same as A.

D. Spring 58 feet from B at 87° NE. Flows same as A.

E. Spring about 150 feet from B and due west. Flow one-fourth size of wrist.

F. Spring 175 feet from E and about 95° NW.

G. Spring about 150 feet from F and 64° NW.

H. Spring at SW end of group.

I. Spring in main bath house. Difficult of access.

J. Average of analyses A to I inclusive.

K. Cold "Iron" spring on right bank of creek 250 feet above mouth of Mangfield Creek.

L. Composite sample from Breitenbush River.

The figures show that the several hot springs are merely separate outflows from a common source. The water is sodic chloride in character, but it contains small amounts of silica, calcium, bicarbonates, and sulfates. Minimum medicinal doses of both sulfates and carbonates might be obtained by drinking about 4 liters of the water,[1] but the disagreeable and even nauseating taste of the chlorides would make the drinking of that amount in one day an Herculean feat. It is certain that any curative properties attributable to the mineral content are psychologic rather than physiologic.

The water is interesting to geologists, as it is an excellent example of a type encountered in many regions of recent volcanic activity. Hot springs in volcanic regions may be placed in several general divisions, such as sulfate springs, carbon dioxide, or "soda" springs, chloride springs, and the like. The origin of the waters of many of the types can easily be explained, but no adequate explanation of the presence of the large quantities of sodium chloride in the waters of the third type has yet been advanced, so far as known to the writer.

The lavas of the Cascade Range have been erupted or through these rocks would be heated, the bicarbonates in them would thereby be decomposed, and the waters would be relieved of part of their charge of iron, aluminum, alkaline earths, and carbonates. Alkaline salts with perhaps a slight residuum of calcium, and even less magnesium, would remain in solution. The calcic carbonate and the magnesia deposited would remain behind in the rock, and the water would move on without them. The carbonic acid set free as gas would react with rock silicates, decomposing them and depositing alkaline earth carbonates, the alkalies and part of the silica entering into solution. Thus a large part of the carbonic acid would be lost from the solution. The waters, percolating still further into the heated zone, would become further heated, and would be subjected to through, and now overlie, the older sedimentaries of the continental floor. At the great depth to which the sediments have been buried, the adjoining lava is still hot. This heat has also been communicated to some extent to the sediments so that these are at high temperatures. As any waters, percolating into increasingly great pressures. They would become more strongly concentrated, because of these and other physical conditions, and would finally be forced to the surface under combined static and thermal pressure. Any carbon dioxide still held in solution would cause small amounts of calcium, encountered en route, to be dissolved, and the springs would then deliver a sodic chloride, calcic bicarbonated and sulfated water, containing appreciable amounts of dissolved silica. This explanation avoids the necessity of considering the action of magnatic, or "juvenile" waters, and, being based entirely on well-known chemical reactions, has the merit of simplicity, and freedom from speculative hypotheses.

U. S. Geological Survey
Salem, Oregon

[1] Cf. R. B. Dole, "Concentration of Mineral Water in Relation to Therapeutic Activity," in *Mineral Resources of the United States for* **1911.** U. S. Geol. Survey, 1912.

PHOSPHATES IN SURFACE WATERS

By George S. Jamieson

Received February 5, 1913

The well-known colorimetric-molybdate method

is not suitable for the estimation of phosphates in the majority of surface waters because it is only applicable to those free from color.[1] In order to apply the method to the analysis of soil extracts, Veitch[2] removed the color from the solutions by evaporating them to dryness with magnesium nitrate and igniting the residues until the organic matter was burned. In the colorimetric method of Schreiner and Brown,[3] which is based upon the precipitation of the phosphoric acid as ammonium magnesium phosphate, any color present is removed when the precipitate is filtered and washed with ammonium hydroxide.

Recently it has been found that the coloring matter of a water can be destroyed by heating the residue, obtained by evaporation, with potassium permanganate and nitric acid. In order to test the accuracy of the method, known amounts of sodium phosphate were added to 50 cc. portions of a surface water which had a light brown color. Each solution was acidified with 2 cc. of nitric acid (sp. gr. 1.07) and evaporated to dryness in porcelain dishes on the steam bath. The residue was moistened with 2 cc. of the diluted nitric acid and 0.5 cc. of a solution of potassium permanganate (1 gram per liter) was added. In the case of a highly colored water more permanganate should be added to insure complete decolorization. The solution was evaporated to dryness and the residue heated for an hour at 100° C., then the residue was heated with 3 cc. of the same nitric acid and 15 cc. of silica-free distilled water. The evaporating dish was given a circular motion to facilitate the extraction of the phosphoric acid from the insoluble matter. After standing a short time the solution was transferred to a 50 cc. Nessler tube and the dish was washed several times with small quantities of distilled water. When it is necessary to filter the solutions, the filters should be tested, as it has been pointed out by several investigators that sometimes even the best quality of washed filters may contain enough silica to contribute considerable color when the ammonium molybdate is added. It was found that thorough washing of the filter paper with hot water removed the silica. The solutions in the Nessler tubes were diluted to the mark with silica-free distilled water, and 4 cc. of ammonium molybdate solution which contained 15 grams of the neutral salt per liter were added. The color was developed and compared with freshly prepared standard tubes. The following results were obtained:

No.	Cc. of water	Mg. of P_2O_5 added	Mg. of P_2O_5 found
1	50	...	0.025
2	50	...	0.025
3	50	0.050	0.075
4	50	0.050	0.075
5	50	0.040	0.065
6	50	0.100	0.126

Two experiments were made using potassium chlorate in place of potassium permanganate with the modification that several evaporations were made with nitric acid in order to remove the larger part of the chlorides formed by the reduction of the chlorate. This method gave 0.025 milligram of P_2O_5 in duplicate, using 50 cc. portions of the water for analysis. The potassium permanganate is preferred not only on account of its rapidity as compared with the chlorate method, but also from the fact, which has been previously shown,[1] that low results are liable to be obtained with the latter method on account of the formation of *aqua regia* during the evaporations with nitric acid.

Phosphate determinations were made at various times upon several surface waters in the vicinity of New Haven, and it was observed that after a heavy or prolonged rain the amount of phosphate was considerably increased over the normal amount. It was further observed that the larger part of the phosphate was held in suspension as it could be removed by filtration. Three of the waters examined showed the normal amount of P_2O_5 to be about 0.6 milligram per liter. The average of 30 determinations (abnormally high results after rains being excluded) made upon different samples of water from a pond was 0.59 milligram of P_2O_5 per liter. In the following table, the results of several analyses are given of samples which were taken a day or two after heavy rains.

Water	Mg. of P_2O_5 per liter	Mg. of P_2O_5 per liter in filtered water
1 A	2.02	..
2 A	1.05	0.30
3 A	1.25	0.20
4 A	1.35	0.80
5 A	1.60	..
1 B	1.35	..
2 B	0.94	0.71
1 C	1.05	0.56
1 D	1.35	..
2 D	0.84	0.56

It is believed that the larger part of the phosphates introduced into these waters by the rains was due in part to the cultivated fields and in part to the inhabited areas upon the water sheds. This investigation indicates that the determination of phosphates in surface waters may be of assistance in showing pollution, and it is hoped that others more favorably situated may be able to investigate this problem thoroughly.

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[1] A. G. Woodman and L. L. Cayvan, *J. Am. Chem. Soc.*, **23**, 96 (1901).
[2] *Ibid.*, **25**, 169 (1903).
[3] *Ibid.*, **26**, 1463 (1904); also U. S. Dept. Agric., Bur. of Soils, *Bull.* **31** (1906).

THE DETERMINATION OF ZINC IN ORES

By D. J. DEMOREST

Received October 14, 1912

The following modification of Low's method has been worked out in order that it may be applicable to any ore, no matter what impurities may be present. It depends upon the fact that ammonium carbonate completely separates zinc from iron, aluminum, manganese, lead and cadmium. The zinc (and copper) stay in solution as the zinc ammonia and copper ammonia carbonates. The copper is separated by hydrogen sulfide and the zinc is titrated by the well-known ferrocyanide method.

The writer's experience coincides with that of Low, that neither lead nor aluminum are safe reagents to

[1] A. G. Woodman and L. L. Cayvan, *J. Am. Chem. Soc.*, **23**, 96 (1903).

use to remove the copper and cadmium, as both lead and aluminum interfere in the titration of the zinc unless Waring's method is used.

Zinc usually occurs in ores as sulfide, oxide, carbonate or hydrosilicate. These minerals are dissolved by boiling strong mineral acids. Ores containing zinc spinel (Gahnite) must be fused with sodium carbonate and borax glass in order to be made soluble.

PROCESS OF ANALYSIS

Place one gram of the ore in a 250 cc. beaker, add 5 cc. of HCl (sp. gr. 1.2), heat for several minutes, add 20 cc. of concentrated HNO_3 and boil with the cover on until all brown fumes are expelled. Remove the beaker from the heat and add one gram of $KClO_3$ and then boil to dryness. Do not bake the residue. Now add 50 cc. of hot water and one-half gram of KOH, but not more, and break up the cake on the bottom of the beaker with a policeman. Add 6 grams of ammonium carbonate and heat nearly to boiling for several minutes. Let the precipitate settle and filter, preferably by suction. Wash several times with a hot 5 per cent. solution of ammonium carbonate, then transfer the precipitate back to the beaker by washing it out of the funnel by a jet of water.

Dissolve the precipitate by means of 3 cc. of concentrated HCl and a little KNO_2 to reduce the MnO_2 present, add KOH solution until the acid is all neutralized, but no more, and then add 5 grams of ammonium carbonate and heat to boiling for several minutes. Filter through the same paper as was used before and wash several times with the hot 5 per cent. ammonium carbonate solution.

Make the filtrate, which should not be more than 200 cc., acid with HCl and then add 20 cc. more of strong HCl, keeping the cover on to prevent loss due to effervescence. Heat to 70° and pass a stream of hydrogen sulfide through the solution for several minutes. When the copper is practically all precipitated add, slowly and with stirring, 20 cc. of 1 : 1 ammonia and continue the stream of hydrogen sulfide for several minutes. Again heat to 70° and titrate. The volume at the beginning of the titration should be 250 cc. Fifty cc. of this should be reserved until the other $^4/_5$ is titrated roughly and then the 50 cc. added and the titration finished without consuming much time as the approximate amount of ferrocyanide required is known. The titration must be performed slowly and with constant stirring to get the best results.

STANDARDIZATION OF THE FERROCYANIDE

Dissolve 22 grams of pure $K_4Fe(CN)_6.3H_2O$ in water and dilute to one liter. One cc. of this solution will precipitate approximately 0.005 gram of zinc.

Weigh 0.2 gram of pure zinc, or better, the weight of zinc which is approximately equal to the weight of zinc in the sample to be analyzed. Dissolve in 10 cc. HCl and 20 cc. of water, add 10 grams of NH_4Cl, dilute to 250 cc., heat to 70° and titrate. Run the ferrocyanide in slowly and with constant stirring until a drop of the solution shows a brown tinge when tested on a white plate with a drop of a 5 per cent. solution of uranyl nitrate. The color should appear within a minute.

A blank must be run, using the same amount of reagents and solution as is used in the standardization. It is necessary that the standardization be made under the same conditions of temperature, acidity, volume and rate of titration as obtained when the ore is titrated.

NOTES ON THE PROCESS

Potassium hydroxide is used to combine with any NO_3 ions present in order to prevent the formation of ammonium nitrate upon the addition of ammonium carbonate, because cadmium carbonate is soluble if much ammonium salts other than the carbonate are present.

The ammonium carbonate precipitates the lead, cadmium and any manganese present in solution as carbonates, the iron and aluminum precipitate as hydroxide: of course any lime or magnesia present comes down as carbonate. If the amounts of the elements which precipitate are small, one separation is sufficient, but it is always best to make a double separation. The second precipitation requires only a few minutes.

When the copper is precipitated according to the above directions, that is, by hydrogen sulfide from a very acid solution, the copper sulfide comes down free from zinc. What little copper remains in solution is precipitated as the solution is gradually made less acid. The copper sulfide need not be filtered off, for it precipitates in a very dense form and does not interfere with the end point, even when 20 per cent. of copper is present.

Potassium chlorate used in the decomposition of the ore introduces no complications, for the presence of hydrogen sulfide during titration prevents oxidation of the ferrocyanide. The zinc ferrocyanide always comes down white. It is absolutely necessary that the titration should not be made too rapidly. If the ferrocyanide is run in rapidly and without much stirring the end point will seem to be reached long before the zinc is all precipitated. If, however, the solution be then allowed to stand for several minutes while being vigorously stirred, titration may be finished correctly. Probably when the ferrocyanide is added rapidly, zinc potassium ferrocyanide is precipitated which can react with uranyl nitrate, giving a brown color.

The following are some results obtained by this method:

	Percentages of Metals Present			Percentage zinc Found
Zinc	Cadmium	Lead	Copper	
Sample D.....	...	..	..	31.37
Sample D.....	3.6	..	..	31.42
Sample D.....	3.6	10.5	..	31.30
Sample D.....	3.6	..	10.0	31.35
Sample D.....	6.0	10.0	12.0	31.45

Sample D is the standard zinc ore of the U. S. Bureau of Standards, which the Bureau finds to contain 31.41 per cent. zinc.

Grams of Metals Present					Grams zinc
Zinc	Lead	Copper	Iron	Manganese	Found
0.4175	0.21	0.10	0.15	0.08	0.4172
0.3425	0.12	0.06	0.10	0.07	0.3405

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A POLARIMETRIC METHOD FOR THE DETERMINATION OF STARCH IN PAPER

By Christian E. G. Porst and Harry A. Crown

Received January 7, 1913

The work described in this paper is supplementary to that given in a paper under the same title, which was submitted to the Eighth International Congress of Applied Chemistry, but too late for publication, and also to the authors' paper, "Research on Lintner's Polarimetric Method for the Determination of Starch," which was published among the original communications to the Congress.

In our previous work, we found that the method could be applied to the determination of starch in paper with reliable results. But it was found necessary to determine the value of the specific rotatory power for small amounts of starch, as this value varies from that found and given in our paper on Lintner's method, depending on the amount of starch taken in work. Therefore, the object of the present work was to determine the specific rotatory power of starch for low concentrations, similar to those encountered when working with 5 grams of paper and to test the method more fully on a large number of samples of paper.

When weights of starch below one gram were taken, the value for $[\alpha]_D^{20}$ varied considerably, duplicates not checking well. This discrepancy seemed due to the precipitation of some of the starch by the phosphotungstic acid. Its use was then omitted as it is not necessary to precipitate the protein which is low in the pure starch and does not affect the reading. But when the phosphotungstic acid was not used, the starch solutions obtained were cloudy, due to the small amount of fat which is present in the starch, and the polarimetric readings could not be made with sufficient accuracy.

It was then decided to add some paper pulp to the starch solution, this producing the same conditions as when samples of paper which give clear solutions are worked upon. The result was satisfactory, the pulp absorbing the fat and giving a perfectly clear filtrate. Pure Swedish paper pulp was used and the solutions obtained were clear, even when as much as five grams of starch were dissolved. Blank tests

Weight of starch taken Grams	Reading in 200 mm. tube Degrees Ventzke	$[\alpha]_D^{20}$ Degree
0.1	0.6	207.0
0.2	1.2	207.0
0.3	1.8	207.0
0.4	2.4	207.0
0.5	3.0	207.0
0.6	3.6	207.0
0.7	4.2	207.0
0.8	4.8	207.0
0.9	5.35	205.1
1.0	5.9	203.6
1.5	8.85	203.5
2.0	11.8	203.5
3.0	17.5	201.3
4.0	23.2	200.1
5.0	28.9	199.4

were run on the paper pulp which was found to be free from starch.

Working with pure corn starch, the preceding values for the specific rotatory power were obtained.

The method by which these values were obtained is as follows:

The pure starch of known weight is mixed with 20 cc. of distilled water in a mortar, which is cooled in ice-water. To this is added 40 cc. concentrated HCl previously cooled down in a freezing mixture, stirring with the pestle until all the starch is dissolved, and then the solution is kept at 20° C. for half an hour. The contents of the mortar are then transferred to a 200 cc. flask to which had been added about one gram of pure paper pulp, disintegrated by rubbing in a mortar with HCl of 1.125 specific gravity, and the flask is filled to the mark at 20° C. with HCl of 1.125 specific gravity. The flask is kept for half an hour in a water bath at 20° C. and then the solution is filtered, first with suction through a Büchner funnel, the paper pulp acting as a filtering medium and then through a Munktell washed filter. Just fifteen minutes after the first filtration is begun, the reading is taken at 20° C. in a 200 mm. tube.

About sixty samples of paper were now tested by the above method, for starch retained, the addition of paper pulp being omitted, but the other details followed as above. The value 207.0 for $[\alpha]_D^{20}$ was used in all computations since the maximum reading obtained was 1.5° V. In all the samples tested, the percentage of starch retained varied from 0 to as high as 4.7 per cent. Two or three samples contained coloring matter which imparted a dark red color to the HCl solution, making it impossible to obtain a polarimetric reading. Some papers gave yellow solutions but this color did not affect the readings. Results in duplicate checked well and on the whole, the method worked satisfactorily.

Corn Products Refining Co.
Edgewater, N. J.

LABORATORY AND PLANT

CHEMICAL ENGINEERING AND THE NEW LABORATORIES AT COLUMBIA UNIVERSITY[1]

By M. C. Whitaker and R. K. Murphy

The development of the manufacturing industries produces an ever-increasing demand for men trained in the fundamental sciences and their applications. Manufacturing processes based upon chemical principles are advancing in importance at a most astounding rate. To supply properly trained men who can

[1] Paper presented at the New York Section of the American Chemical Society, Chemists' Club, New York, March 7, 1913.

develop, operate and direct these manufacturing processes would seem to be one of the most important present-day problems in technical education. Most institutions have not met the problem fairly; they have hesitated, procrastinated and even evaded. The problem is not one which can be solved by grafting extra instruction here and there on to some existing engineering courses nor by changing the name of the chemist to that of chemical engineer.

The new industrial development calls for a new class of men—men trained to apply engineering methods and appliances to the solution of chemical problems and the operation of chemical processes.

The training of chemical engineers has been given the most careful study by Columbia University. Prominent men have been freely consulted, and the entire problem has been attacked on its broadest and most comprehensive basis. It is felt that a distinct advance has been made, and it is the purpose of this paper to present a brief outline of present plans and progress.

It is a recognized fact that the rapid advancement in all engineering sciences has put a heavy burden on the student. The time allotted to cover the work (usually four years) has remained constant, while the ground to be covered has been greatly increased. The High Schools have endeavored to keep pace with the more severe requirements of the technical school entrance examinations, but these preparatory schools seem also to have reached their limit. They are no longer able to adequately prepare young men to complete a thorough scientific course in four years without an undue strain upon the student.

There are but two possible methods of solving the difficulty: (1) a reduction in the amount of material presented, or (2) an arbitrary increase in the time to be devoted to post-high school training. In many schools the academic subjects are being eliminated from the curriculum to the great loss of the students' general scholarship; in other institutions fundamentals are superficialized to such an extent that the graduate lacks the necessary foundation on which to develop; in still other engineering courses, cultural and fundamental subjects are retained and engineering applications neglected. In the few cases where the faculty is about evenly divided between academic and engineering representatives, the student's wail concerning the amount of time at his disposal for study and his difficulty in assimilating the various and varied subjects offered in the distended curriculum, has been drowned by the insistent appeals of the faculty members for more allotted time for each of the so-called "most important subjects" in the course. The obvious remedy to meet this deplorable situation is to give the student more time and better facilities in order to fulfill the present demands.

The Schools of Mines, Engineering and Chemistry of Columbia University will become, in 1914, regular post-graduate professional schools and will require a college degree or its equivalent for admission. The

TENTATIVE PLAN OF A THREE-YEAR COURSE OFFERED BY COLUMBIA COLLEGE TO FULFILL THE REQUIREMENTS FOR ENTRANCE TO THE POST-GRADUATE ENGINEERING SCHOOLS

FIRST YEAR

First Half SUBJECT	POINTS	*Second Half* SUBJECT	POINTS
Advanced Algebra (Math. 1)	3	Analytical Geometry (Math. 4)	3
English Composition (Eng. A)	3	Chemistry, Gen'l (2 Lect.) (Chem. 3c) (6 lab.)	5
Principles of Science (Philosophy A)	3	English Composition (Eng. A)	3
Modern Language based on Intermediate Entr. requirement.	5	Principles of Science (Philosophy A)	3
Shop Work (1 aft.)	1	Modern Language (continued)	3
Physical Education A	1	Shop Work (1 aft.)	1
		Physical Education A	1
		Total points	35

SECOND YEAR

First Half SUBJECT	POINTS	*Second Half* SUBJECT	POINTS
Calculus (Math. 15)	4	Calculus (Math. 16)	4
Gen'l (2 Lect.) (Chem. 4c) (6 lab.)	5	Chemistry (Chem 67) (2 Lect.), Qualitative (6 lab.)	5
Introduction to Eng. Lit. (English B 3)	3	Physics (3 Lect., 3 Rec.)	6
Epochs of History (Hist. A)	3	Epochs of History (Hist. A)	3
Drafting (2 afternoons)	2	Drafting (2 afternoons)	2
Geometry, Descriptive (Drafting 3)	3		
		Total points	40

THIRD YEAR

First Half SUBJECT	POINTS	*Second Half* SUBJECT	POINTS
Calculus (Math. 17)	4	Physics (same)	7½
Chemistry (Chem. 68) (2 Lect.), Qualitative (6 lab.)	5	Statics (Mechanics 2)	2
Physics (3 Lect., 3 Rec.) (1 afternoon Lab.)	7½	Political Science (Politics 4)	3
Political Science (Econ. 1)	3	Surveying (Civil Eng. 2)	2
		Mineralogy (2 Lect.) (1 afternoon)	3
		Total points	37

ELECTIVES

The 12 additional points necessary to establish the 124 points required for the degree of B.S. must be made either by extra credit for high standing by work in Summer Sessions, or by free election during the second and third years; such electives may be chosen at the student's option from among the courses in which he can satisfy the prerequisites, with the single provision that no program aggregating more than 22½ points will be approved for any half-year.

purpose of these changes is not only to provide better and broader undergraduate training for students of engineering, but also to place the engineering departments upon the same high professional plane as that occupied by the departments of law and medicine. It is a significant fact that for several years over twenty per cent. of the students in our engineering schools were college graduates, and therefore really post-graduate students.

The college training taken as a preliminary preparation for this new post-graduate course in engineering must necessarily include physics, chemistry and fundamental mathematics, in addition to the usual college courses. In a properly arranged curriculum this ground may be covered in three years. A tentative scheme, planned by the Faculty of Columbia College to meet the requirements for admission to the new post-graduate engineering schools, is given on page 305. The subjects offered in this undergraduate course are now to be found in the schedules of practically all colleges, and by judicious elections the required fundamentals may be satisfactorily completed in any good institution.

Corresponding courses have been adopted for Mining, Mechanical, Electrical and Civil Engineering. It is believed that this extension of practically two years in the time to be devoted to the combined fundamental and professional work will not only relieve the present tension on instructors and students, but will produce a class of graduates immeasurably better qualified to assume the responsibilities of their profession.

Chemical Engineering courses must eventually teach men to use engineering methods and engineering appliances in solving chemical problems and carrying on chemical processes. These engineering appliances and methods are not to be found in the test tube, beaker, or funnel of the usual chemical laboratory any more than steam, hydraulic and electric appliances and applications are to be found in a physics laboratory. Mechanical and electrical engineers long ago recognized the difference between the equipment needed for studying the application of the fundamental scientific principles to complicated engineering conditions, and the equipment necessary to study the laws and principles upon which these fundamentals are based. Laboratories, equipped with mechanical and electrical engineering appliances, have long been established to meet these needs, but the corresponding chemical engineering laboratories have been sadly lacking.

The chemist, with characteristic conservatism pro-

NEW THREE-YEAR POST-GRADUATE COURSE IN CHEMICAL ENGINEERING

FIRST YEAR

First Half SUBJECT	LECT.	AFT.	*Second Half* SUBJECT	LECT.	AFT.
Physical Laboratory	0	1	Advanced Heat	3	0
Mechanics	3	0	Mechanics	3	0
Industrial Chemistry	3	0	Industrial Chemistry	3	0
Power Machinery	2	0	Power Machinery	2	0
Physical Chemistry	3	1	Physical Chemistry	3	1
Elements of Electrical Engineering	2	0	Electrical Machinery	2	0
Hydraulics—Theory	2	0	Hydraulics—Laboratory	0	1
Quantitative Analysis	2	3	Quantitative and Engineering Chemistry	2	3
Total	17	5	Total	18	5

SUMMER WORK

Chemical Factory Inspection (2 weeks)

Factory work with detailed report on some assigned industry (6 weeks)

SECOND YEAR

First Half SUBJECT	LECT.	AFT.	*Second Half* SUBJECT	LECT.	AFT.
Organic Chemistry	3	2	Organic Chemistry	3	2
Machine Elements	2	0	Machine Elements	2	0
Food and Sanitary Chemistry	3	0	Adv. Industrial Chemistry	3	0
Engineering Thermodynamics	3	0	Engineering Thermodynamics	5	0
Direct Current Laboratory	1	1	Alternating Current Lab	1	1
Resistance of Materials	5	2	Assaying	2	2
Total	17	5	Total	16	5

SUMMER WORK

Chemical and Mechanical Engineering Laboratory (8 weeks)

THIRD YEAR

First Half SUBJECT	LECT.	AFT.	*Second Half* SUBJECT	LECT.	AFT.
Introduction to Metallurgy and Metallurgy of Copper	3	0	Metallurgy—Lead, Zinc, Gold and Silver	3	0
Metallurgy—Iron and Steel	1	0	Chemical Factory Management	3	0
Chemical Factory Machinery	3	0	Steam Power	4	2
Electrochemistry	2	1	Business Law	2	0
Gas Power	2	1	Chemical Engineering Lab. Special Problems	0	3
Commercial Organic Analysis	2	3	Seminar	6	0
Seminar	5	0			
Total	18	5	Total	18	5

duced by many disappointments, has been slow to recognize the point at which the study of principles ends and the study of application begins. He has hesitated to develop laboratories with commercial apparatus, such as pumps, siphons, tanks, filter presses, evaporators, stills, centrifugals, absorption towers, etc., but has preferred to consider his work completed with the establishment of a principle on a test-tube scale. It is at this point that the Chemical Engineer is needed to take chemical principles and engineer them in the same manner as the mechanical engineer engineers the physics of heat and the electrical engineer engineers the physics of electricity. The chemical engineer has but little chance to successfully engineer chemical operations unless he knows the fundamental methods and appliances available. The chemist cannot establish data with a beaker and test-tube on which to engineer a process any more than a mechanical engineer can arrive at a correct conclusion in regard to the performance of a steam boiler by some experiments with a tomato can.

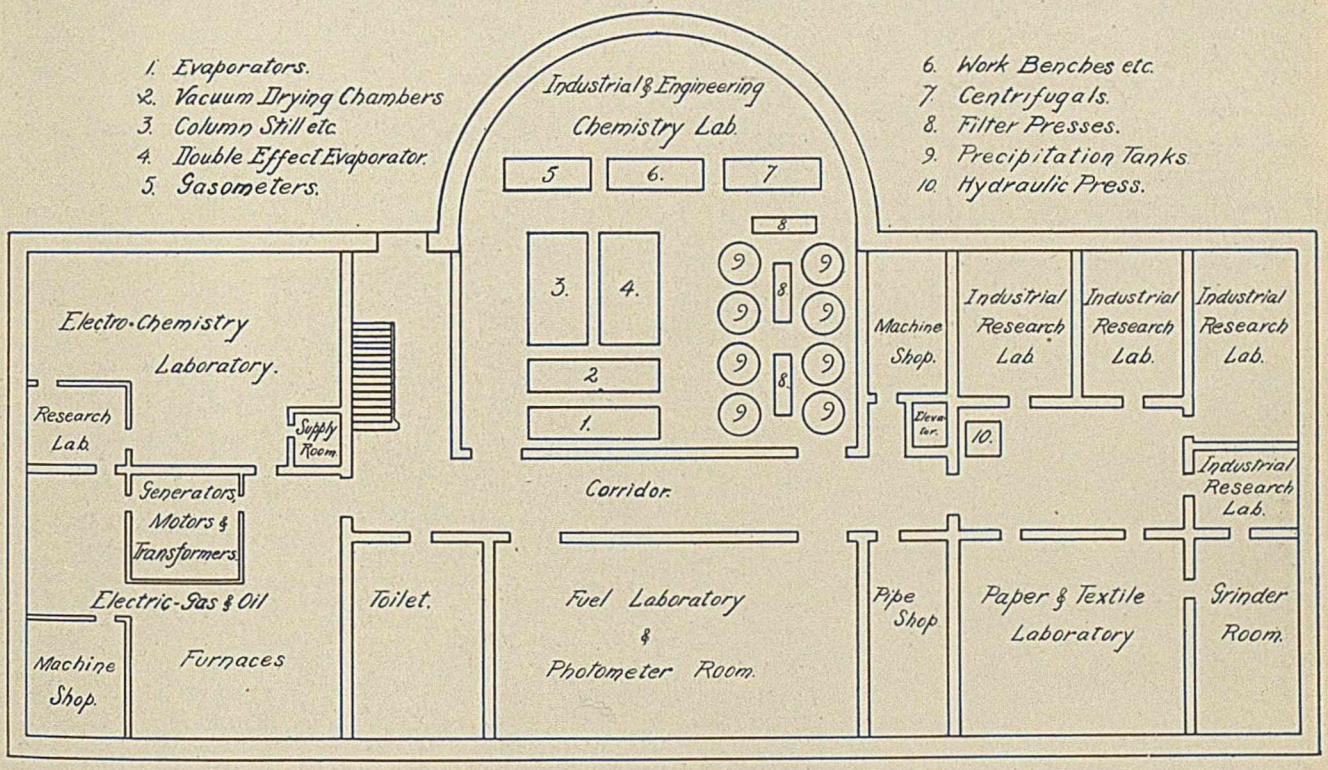
Besides the new industrial laboratories which occupy the entire lower floors of the Havemeyer building, there are the new Nichols laboratories for general chemistry and the new physical chemistry laboratories which are nearing completion. Only the former will be described.

THE NEW CHEMICAL ENGINEERING LABORATORIES

The accommodations for the industrial laboratories are by no means ideal, but while the scheme is not yet complete, we have made a bold beginning and hope to expand rapidly from year to year. Ultimately we shall have a laboratory of chemical engineering comparable with the best laboratories in mechanical and electrical engineering.

Some photographs serve to show the plan and scope of our work and the progress thus far made. The division of space is shown in Fig. I, from which it will be noted that we have, besides the large general laboratory, the electrochemical laboratories, paper and textile laboratory, four research laboratories, grinder room, pipe shop, machine shop, furnace room, etc. Some of the more typical equipment now installed and in operation is illustrated in Figs. II–VI. The solution and precipitation tanks, the filter presses, the siphons and stirrers are partially illustrated in Fig. II.

The problems assigned in all operations have been selected so as to be cyclic as far as possible with the object of reducing operating expenses to a minimum and also avoiding the accumulation of products. For example, in the work involving the use of tanks, acid siphons, pumps, filter presses, etc., a squad of four or five students, with one acting as foreman for a complete operation, is given the problem of dissolving a weighed quantity of the waste sulfates of the didymium earths in dilute sulfuric acid in a lead-lined tank, with air agitation. The solution is transferred by means of a lead siphon to another tank and precipitated as oxalates, which settle readily; the liquid is returned to the acid tank for future use, the precipitate washed by decantation, filter-pressed, and the press cake vacuum-dried and weighed. The cake is then transferred to a seamless wrought-iron tank



→ Department of Industrial Chemistry ←
First Floor Plan
Havemeyer Hall — Columbia University.

FIG. I

and converted to hydroxides, using a steam air agitator. The oxalate solution is stored for use in the evaporator and the hydroxides after filter-pressing are dissolved in acids and returned to the first tank. During this operation the student has had experience with solution, precipitation, washing by decantation, filtration of a granular crystalline precipitate, conversion from a solid insoluble in acid to one soluble, filtration of a slime and resolution in acid; air, steam and mechanical agitation; pumps, lubricators, siphons, etc. All this work is done on a quantitative basis; the losses are checked and must be accounted for at each stage.

The vacuum-drying apparatus is illustrated in Fig. III; it is used in several of the operations, such as drying press-cakes and solids obtained from evaporation, etc.

The double-effect evaporator, shown in Fig. IV, has some special features. The main connections are fitted with outside screw and yoke gate valves so that it is possible to tell at a glance which valves are open and which are closed. The connections are also arranged to permit the pans to be run as either single or double effect. The student squad on this operation, after becoming perfectly familiar with the vacuum pumps, the condenser, the sampler and the various valves and connections, makes an efficiency run by evaporating water, first as single, and then as double effect. The capacity and distribution of heat are determined by thermometer and pressure gauges, weighing the steam used, the water evaporated from each pan, the condenser water, etc. The next step is to concentrate a salt solution containing some six to eight hundred pounds of common salt. Here the solubility curves must be studied and plotted, the original solution analyzed and the maximum amount of salt obtainable calculated. The squad is then given the problem of separating two salts of different solubilities. For example, several hundred gallons of a mixed sodium oxalate and sodium chloride solution are worked; this requires careful study of the solubility curves, frequent sampling and hydrometer readings, and some skill in operation due to foaming and other difficulties which must be met and overcome. The efficiency tests with one and then two salts in solution are compared with the runs on

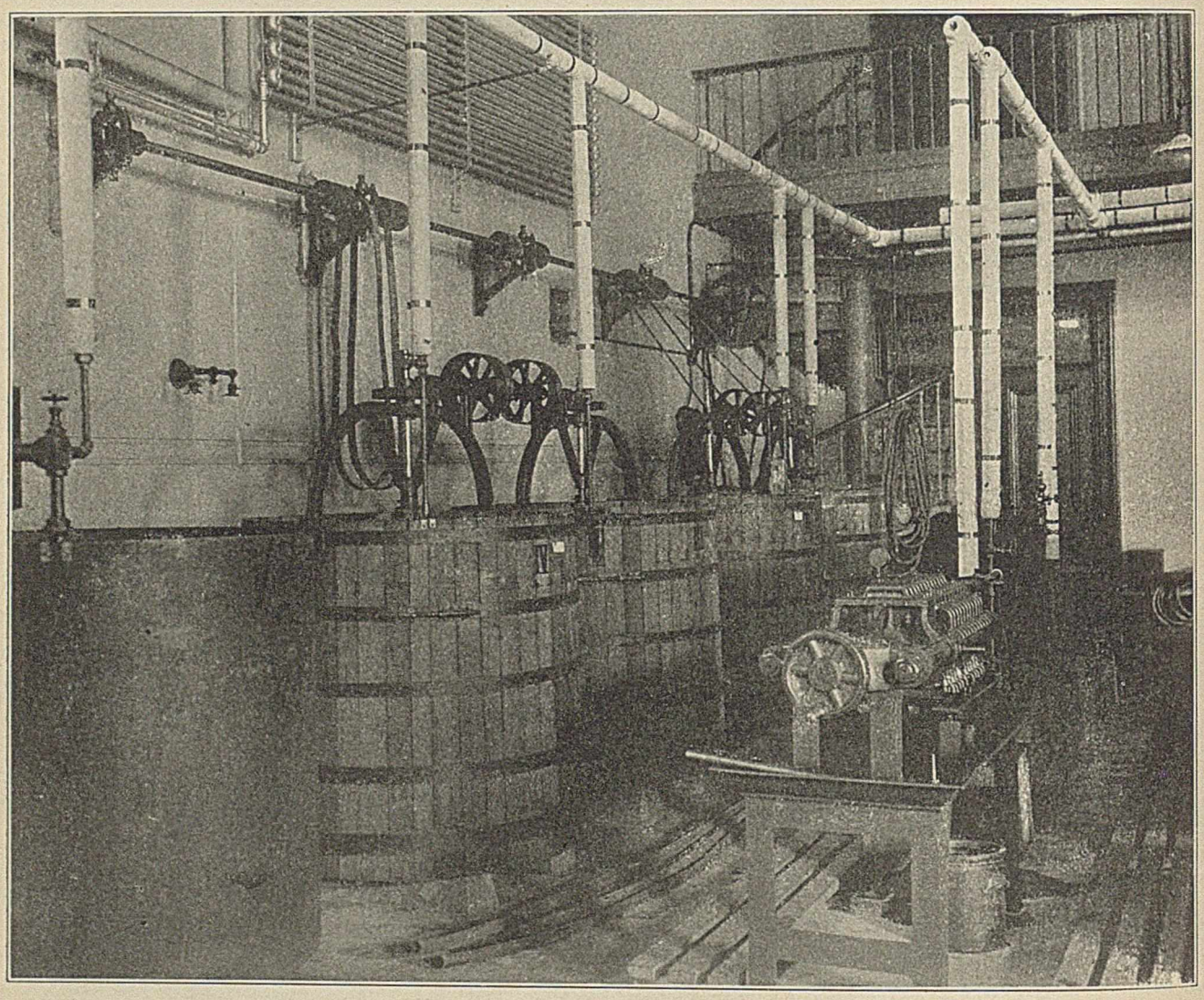


Fig. II—Filter Presses, Pumps, Siphons, Tanks, Agitators, Etc.

water alone, and the relationship of the inches of vacuum in each pan and the effect of varying either of the vacuum pumps or initial steam pressure carefully studied. The final product is weighed, then vacuum-dried to get the dry basis, and finally transferred to the proper tanks and redissolved for use by the next squad.

A part of the distillation equipment is shown in Fig. V. The bench of ten steam-jacket evaporators and the pot stills are not shown, but a fairly good idea of the copper column still with condensers and extractor may be gained from the illustration. The problems to be solved here are similar to the ones already described in some detail. After thoroughly standardizing the apparatus and operating on water alone, this squad would be given the problem of making 98 per cent. pure acetone from a 50 per cent. solution containing some mud or other solid which must first be removed in a pot still before purifying in the column still. Another problem given is to make fifty pounds of soda crystals. The squad must look up the chemistry involved and determine on the method of procedure without being told just how to perform each step.

Another view of the laboratory is illustrated in Fig. VI, showing a centrifugal dryer, centrifugal clarifier and filter, Blau-gas welding outfit and some of the gas holders.

The dyeing laboratory is equipped with the necessary apparatus including an experimental calico printing machine, to demonstrate the applications of the different classes of dyestuffs and mordants for dyeing various fabrics such as cotton, wool, silk and artificial silk. The laboratory course includes such work as is necessary to familiarize the student with the application, testing and matching of dyestuffs, determinations of fastness, etc. In connection with the dye work, instruction is given in the use of the microscope and ultra-microscope for the identification of fibers, crystals, starches, yeasts, etc., and such other testing methods as would be of value to the chemist or chemical engineer in commercial operations.

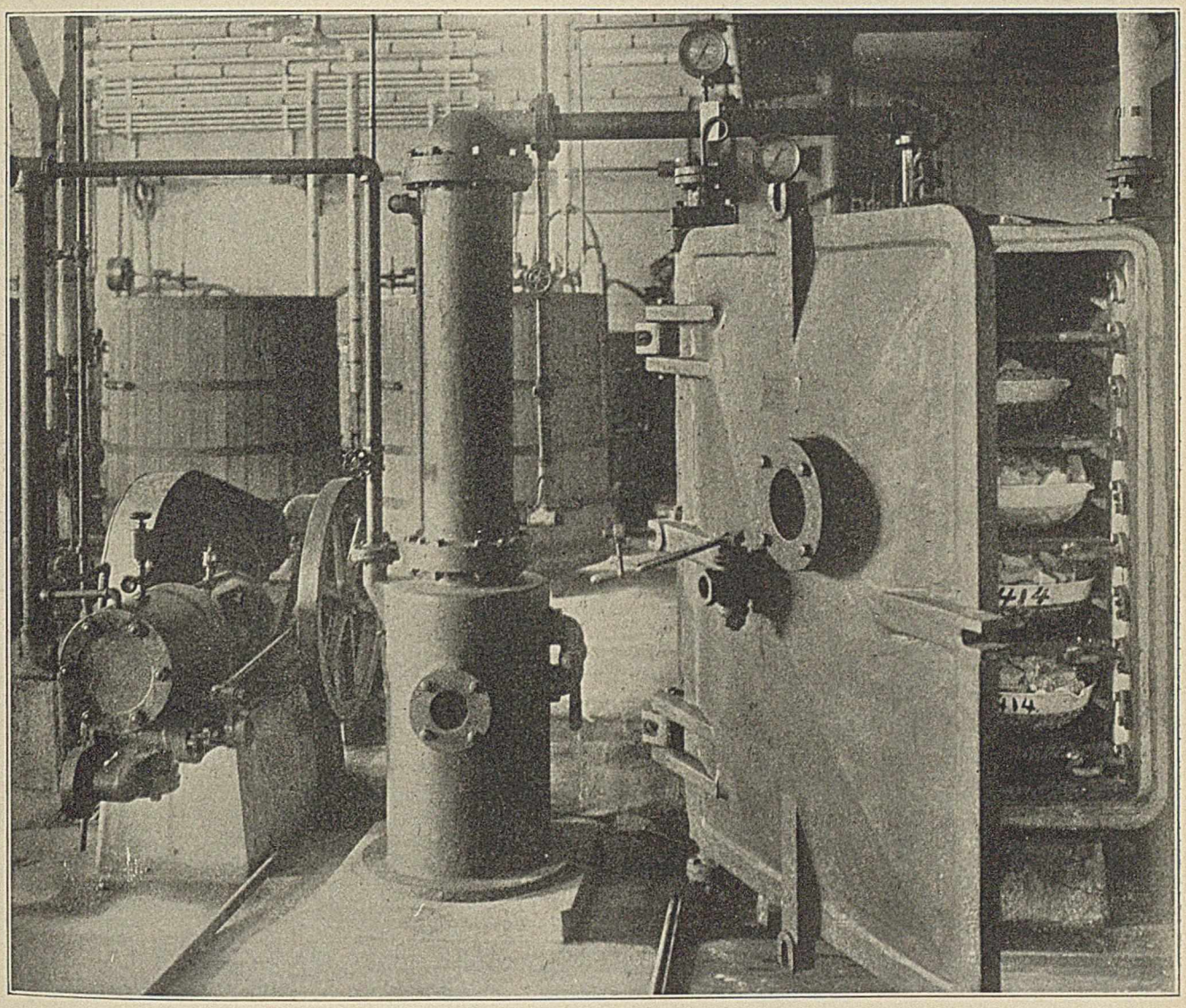


Fig. III—Vacuum Shelf Dryer, Condenser and Pump. 42 Inch Shelves

A new paper laboratory has been started and at present is equipped with an iron soda digester (2 gal.

capacity), a bronze sulfite digester (7 gal. capacity), pulp beater, rotary copper drum dryers and mechanical stirrers. Fig. VII illustrates the soda digester with stirring device. A 110 lb. steam line runs into the laboratory and this may be connected to the various digesters, etc. Several men are doing research work on the treatment of woods and on paper sizing.

Instruction in the electrochemical branch of industrial chemistry is given in the senior year of the Chemical Engineering course. The laboratory work includes not only all the usual electrolytic processes, but also considerable practice with various types of electric furnaces. Electric power from the central power plant is transformed in the dynamo room to almost any desired amperage and voltage. A part of the dynamo room is shown in Fig. VIII. This view gives an idea of the direct current dynamo set and the power available for experimental work.

Facilities for individual research work are very good, and many students avail themselves of the opportunity to do advanced work beyond the required courses. Research men find standard equipment of modern and approved types ready and available for trying out any fundamental practice on a scale and in a way which will enable them to establish data capable of being used on a practical basis and in business calculations. The problems arising as a result of transferring operations from the laboratory to the large scale factory appliance may here be met and solved under the sympathetic eye and patient hand of the developer, instead of being doomed to failure by being delivered in incomplete form to unsympathetic and busy works managers. One of the industrial research laboratories is illustrated in Fig. IX and needs no further explanation beyond that it contains all the ordinary equipment such as gas, electricity, blast, suction, water, drying ovens, etc.

Students are given the greatest possible liberty in the methods of handling their problems, and are allowed to make mistakes and get experience. They use apparatus as types of equipment already developed to carry out the devised operations. Soon they become familiar with the limitations of the appliances and learn to exercise engineering judgment in overcoming their difficulties. A preliminary report covering the chemistry involved and the general method of procedure is required of the foreman of each group, but each member of the squad must hand in a final detailed report covering all points of the work performed. It is the duty of the instructors to assign problems and supervise processes, but not to assist or tabloidize the work for the students.

In conclusion, it should be pointed out that neither

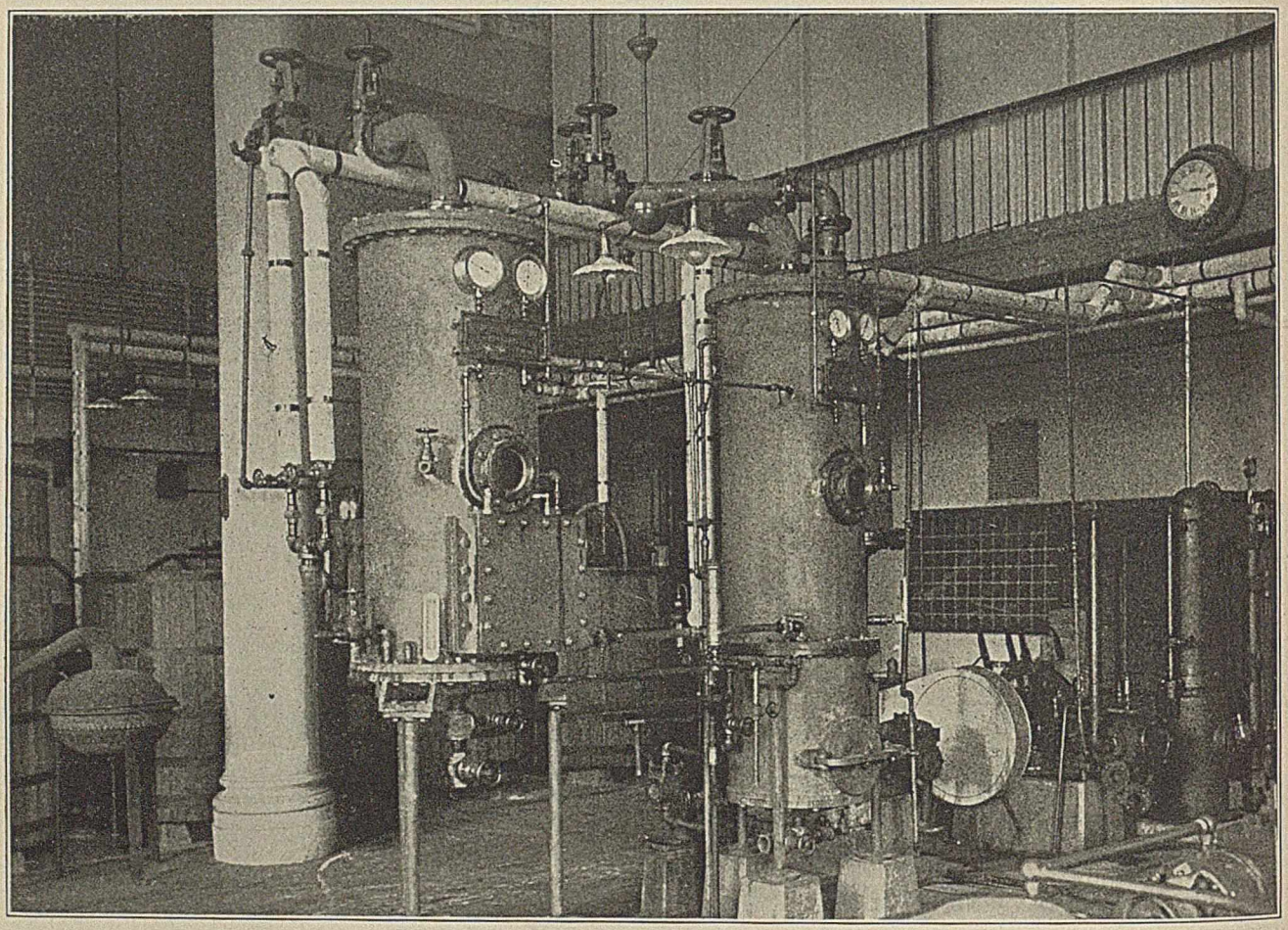


Fig. IV—Double-Effect Vacuum Pans (24 in.) with Complete Equipment. Condenser, Vacuum Pump, Circulating Pump, Salting-Out Chamber, Etc.

the course as outlined nor the laboratory as planned is in any sense intended to train men as artisans or skilled operators. The students are not taught how to dye cotton or silk or to print calico, but are instructed in the application of the fundamental chemistry and physics involved in this industry. We are not trying to teach men to run sugar pans but hope to give them a broad conception of the principles and engineering methods involved in vacuum evaporation. Familiarity with the equipment of such a laboratory will give young men that facility of application which comes now only from long experience. The student develops that engineering instinct which is so essential to the successful solution of chemical engineering problems.

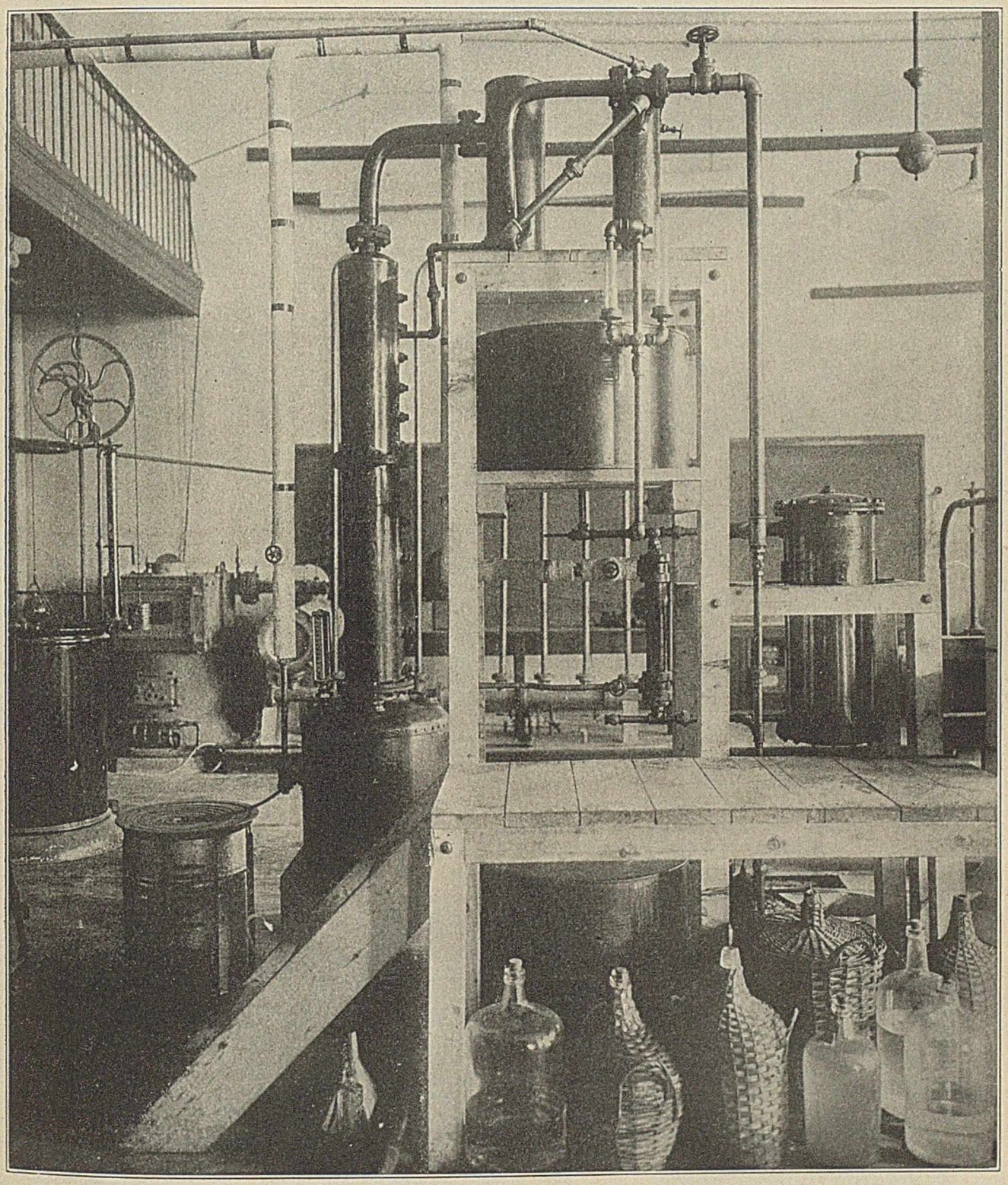


Fig. V—Column Still and Extraction Apparatus. Capacity, 10 Gals. Per Hour

It is confidently believed that the new graduate

Fig. VI—General View Showing Whizzer, Clarifier and Filter, Welding Outfit and Gas Holders

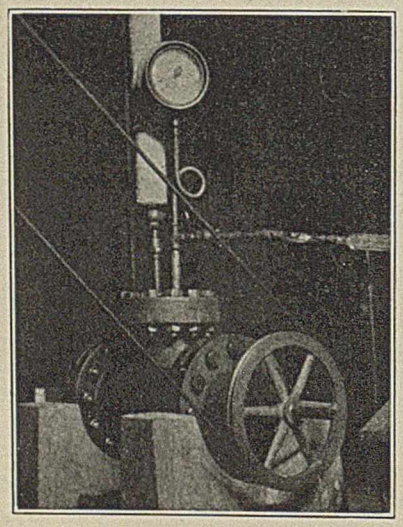


Fig. VII—Soda Digester

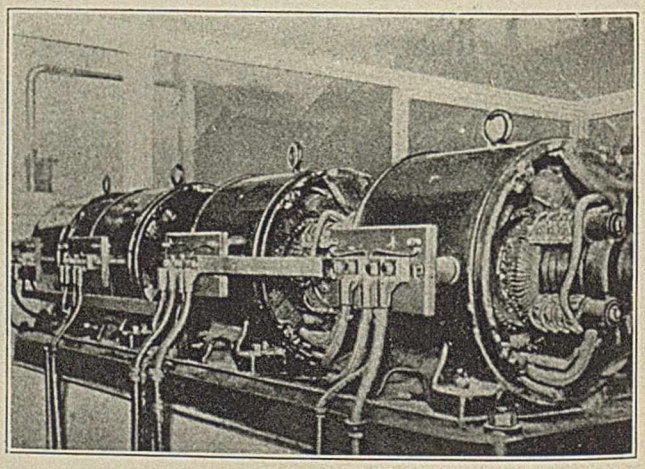


Fig. VIII—Motor-Generator Set in Electrochemical Laboratory

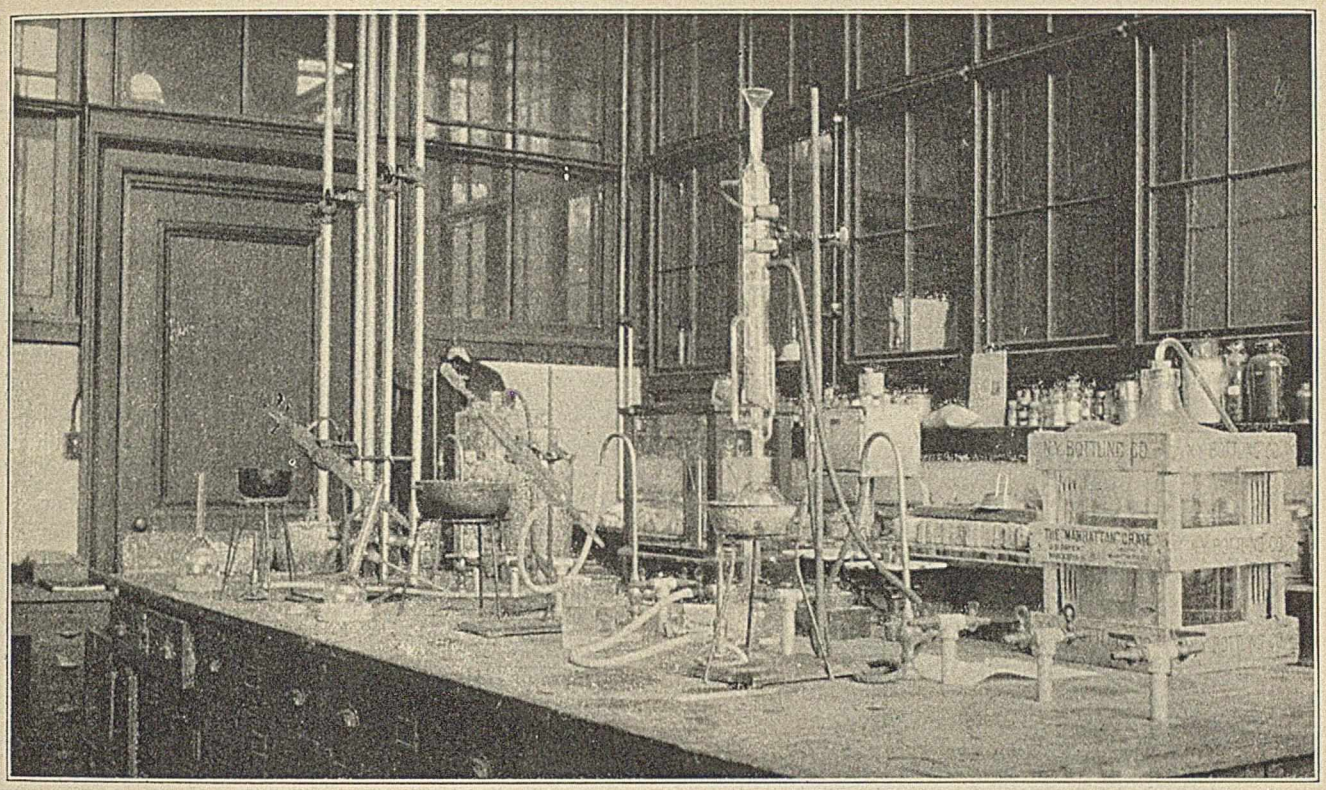


FIG. IX—TYPICAL RESEARCH LABORATORY TO ACCOMMODATE TWO OR THREE POST-GRADUATE STUDENTS

courses and instruction in Chemical Engineering at Columbia, based as they are on a good general fundamental education, will produce men capable of rapidly advancing to positions of responsibility in this most promising profession.

HAVEMEYER HALL, COLUMBIA UNIVERSITY
NEW YORK CITY

TECHNICAL ACCOUNTING AND CHEMICAL CONTROL IN SUGAR MANUFACTURE[1]

By DAVID L. DAVOLL, Jr.

(Concluded from the March issue)

THE CONTROL

SAMPLING AND ANALYSIS

THE CANE.—Determine fiber and sugar once each factory day.

Sampling.—Every hour, four representative canes are to be selected under the chemist's supervision, as they pass from cars, wagons or hopper to the first unit of the tandem. Reserve in a cool, shaded place. If the factory runs six hours or less, prepare the whole sample; if more, subsample to 24 canes as follows: Sort into three piles, one containing the pieces bearing evidence of having been cut in the fields next to the root; one, pieces cut from the middle; one, pieces bearing base of the "cogollo" or top of the cane. Take, impartially, eight pieces from each pile and cut into transverse slices $^1/_8$–$^1/_{16}$ inch thick by means of a Pellet cane cutter, feeding the tops to the machine first and rejecting no odd ends. Allow the slices to fall into a galvanized iron box large enough to hold the entire sample; throw upon a large, clean piece of enameled cloth, mix well and quickly subsample by "coning and leveling" until about a liter is obtained; weigh this carefully. Chop in a hopper resting upon a clean piece of enameled cloth, with a heavy cane knife (calaboso) when finished, brush up all pieces that may have fallen upon the cloth and again weigh.

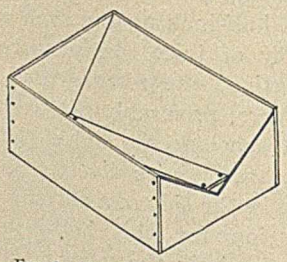


FIG. 1—HOPPER FOR CHOPPING CANE

Correct for loss by drying out during chopping. The hopper must be employed for cane exclusively.

Sugar Determination.—Lightly pack 52 grams in a continuous extractor[1] and slowly pour through the mass sufficient 40 per cent. alcohol to fill the 4-oz. Adams flask two-thirds full; maintain the flask contents faintly alkaline with basic lead acetate; extract for three-fourths hour or for such time as experience shows, under working conditions, is sufficient to extract the sugar to 0.05 per cent. limit using a perforated

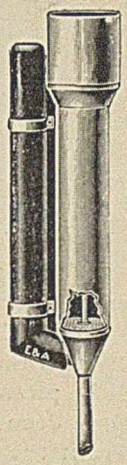


FIG. 2—CONTINUOUS EXTRACTOR

[1] Paper presented at the Fifth Annual Meeting of the American Institute of Chemical Engineers, Detroit, December, 1912.

[1] NOTE.—This extractor is made of nickeled copper and is very durable; vapor pipe insulated, thus promoting rapid exhaustion of contents besides serving as a handle; it is suitable for use with cossettes, "pulp," drug and material of many kinds calling for restricted quantity of solvent.

Especially designed to eliminate "bumping" entirely. An easily removable, screen diaphragm retains the material in place, which, after extraction, may be quantitatively removed for drying and weighing. Made by Eimer & Amend, New York. Standard size 11 cubic inches net capacity.

asbestos plate and low flame, place the flask upon the water bath and expel the alcohol; rinse into a 100 cc. flask, add sufficient basic lead acetate, fill to the mark, filter and polarize. The reading divided by 2 equals sugar.

Fiber Determination.—Lightly pack 52 grams in the continuous extractor, allow cool or tepid water to run slowly through during 1–2 hours; then, slowly, about a liter of water heated to 60° C.; connect the flask containing 40 per cent. alcohol and extract for three-fourths hour; withdraw dregs by means of the rod attached to the bottom sieve diaphragm to a drying dish; dry for 2–4 hours to constant weight at

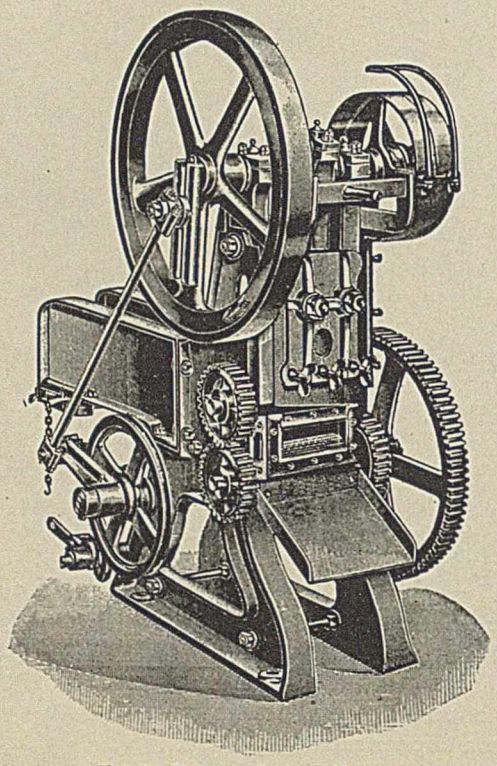


FIG. 3—BAGASSE CUTTING MACHINE

105–110° C. Calculate fiber. To dry cane or bagasse fiber, employ shallow, oblong, tin trays, 4″ by 5″ and $1^5/_8$″ deep, bottom consisting of copper cloth of 80–100 meshes per linear inch.

THE BAGASSE.—Sugar may be determined every 2 hours, fiber once a factory day. Well ground bagasse (modern milling) may be taken by the handful as it rises from the mill-boot of the discharge conveyor and tightly pressed into the sample can, which may be 2 feet deep by 1 foot diameter. Fifteen minutes intermittent sampling should fill the can and there should be the minimum delay in preparation for analysis.

Coarse bagasse (such as that of the first, second or even third unit of the tandem on old-style milling) should be taken from its particular conveyor, *clear across* the blanket and amount to at least $1^1/_2$ cu. ft. in volume.

Throw the can contents to the floor upon a large sheet of enamelled cloth, tearing coarse pieces apart by hand. If still warm (hot saturation) cover lightly with a second sheet of cloth for a few moments. Rapidly mix by stirring and rolling, exposing the minimum surface and pile in a cone; level from the apex outward to a truncated cone and withdraw a wedge-shaped sample, in size proportionate to the coarseness of the bagasse. Cut up the whole sample and mix. A fully satisfactory machine for quickly reducing large amounts of bagasse to fine "sawdust," is that made by Boot and Krantz, The Hague, Holland. See Fig. 3.

Polarization.—This is performed as under cane, with the alkalinity maintained with 5 per cent. solution Na_2CO_3.

Fiber Determination.—This necessitates a very finely divided material. The Hawaiian Sugar Chemists' Association defines fiber as "the total insoluble solids," water being the solvent.

Quantitatively remove the residue left after extraction of the sugar to a drying tray and dry for about an hour, then transfer to a large, loosely covered container; do this on every sample of the factory day. At the close of the day there should be, in the container, the practically dry residues of 12 normal weights. Mix well, weigh the accumulated samples, take one-twelfth, place in a drying tray and dry for 2–3 hours or to constant weight. Divide the weight by 26, multiply by 100, and the result is percentage fiber in bagasse.

If 10 samples only have been extracted, take one-tenth, etc.

Alternate Sugar Method.—Use that as adopted by the Hawaiian Chemists' Association, 1910, *Bull.* 32, Agricultural and Chemical Series, Experiment Station, Hawaiian Sugar Planters' Association, by W. S. Norris.

Moisture.—This serves to judge of the tax laid upon the furnaces by reason of the water to be evaporated; calculate by "difference."

MILL RAW JUICE corresponds to the diffusion juice of the beet industry. It is the main basis of sugar accounting and great pains should be taken to make it fully representative of the work.

Sampling.—The following method by means of a thin, rapidly-running stream has been found to be representative.

Modern mills, in general, discharge the mill raw juice from the free end of a pipe into a small reserve or overflow tank, which in turn serves the measuring or weighing tanks proper.

At a point a short distance below the level of the discharge (in order to secure a slight "head") the main discharge pipe is tapped by a half-inch pipe in such a manner that a small quantity of juice continuously finds the way to its destination, the reserve tank, through it. A small copper wire, preferably not over 4 inches long, may now be attached to the end of the half-inch pipe and a thin stream of juice diverted so as to discharge through a hole in the side of a covered, 2-gallon pail. Another location for this half-inch pipe, not quite so advantageous, is in the same main raw juice line but close to the pump, returning the diverted portion of juice to the pump-tank and interposing the copper wire in the stream, as described. This latter arrangement reduces the pump efficiency

about 1 per cent. in a 1000-ton factory. The half-inch pipe should have but one cock, and that next the open end, to allow for closing when the pump is not operating.

The collection of samples is apt to be a weak point, on account of placing too much reliance upon a messenger. It should be reduced to a simple system, proper conveniences supplied, and closely supervised.

In tropical work, evaporation is rapid and the air is full of spores. All samples should be removed from the factory at frequent intervals: let the chemist either analyze them at once or properly preserve them. The messenger may collect the following samples every two hours: mill raw juice, first mill juice, last mill juice, residual juice, evaporator thin juice and meladura. Assuming that these are already discharging into their containers there will be needed for substitution six other clean and dry sample pails containing sufficient formaline. Not all of the sample collected can be or should be taken to the laboratory—after mixing, the greater bulk will be at once returned to the process of manufacture. For this purpose, separate clean and dry metal paddles will be needed for stirring. A tray of light material holding six liter Mason jars, plainly labeled and with screw tops, will be needed for receiving the sub-samples.

Analysis.—Strain the sample into a clean, well-rinsed Mason jar and let stand until the foam has risen; carefully remove the same with a teaspoon. *A. The Brix:* Slowly fill a tall cylinder by pouring down the side; carefully insert the hydrometer, let stand until it has come to permanent rest, then read and note the temperature; correct for hydrometer error, if any and to standard temperature. *B. The Polarization:* Slowly fill a 100–110 cc. flask to lower mark, clearing up any uncertain meniscus with a drop of ether, run in the prescribed amount of basic lead acetate and fill to the upper mark with water; shake thoroughly, filter and polarize, employing the Schmitz table for sugar percentage. If the analyses must be made at longer intervals, composite as follows: Prepare as above for "*A*" and "*B*;" instead of proceeding with "*A*," pour 200 cc. into the compositing jar containing the proper amount of formaline, for Brix: proceed with "*B*" as far as filtration, then pour the whole contents of the flask into the compositing jar for polarization.

When making the determination, thoroughly mix the contents of each compositing jar, proceeding as under "*A*" for Brix. Filter, polarize and consult Schmitz's table for sugar percentage.

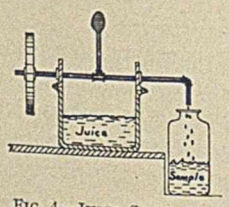


Fig. 4—Juice Sampler

C. Set aside in a suitable jar a portion of the unfiltered "leaded" solution for the daily composite determination of sucrose by the Herzfeld-Clerget method and invert sugar.

When making these last two determinations, add sufficient acetic acid to transform all basic lead acetate into neutral or slightly acid condition, correcting for increased volume in final calculation.

FIRST MILL JUICE. *Sampling.*—This must be representative of juices coming from combined crusher and first unit of the tandem. There is no agreement in either Brix or sugar content nor in the quantity of the juices falling from (1) the crusher, (2) the cane roll, (3) the bagasse roll; hence the logical place for continuously and systematically drawing this sample is in the trough conveying it to the mill raw juice pump. Fig. 4 shows a successful device for taking this sample.[1]

An objection to this will sometimes be that water used to cool the mill bearings finds its way into this sample, the effect being to raise the figure for extraction.

The trough sample then becomes useless and a second sample should be taken by placing a suitable pail beneath the cane roll of the first unit at a point where tests show that the Brix and sugar content correspond to those of the trough sample when not contaminated by water. This pail holds about 8 liters and has a cover slightly inclined; holes are made near its apex, from the inside outward, sufficient in number to about half fill the pail in 2 hours.

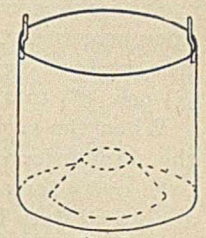


Fig. 5

Analyze as under mill raw juice, *A* and *B* only.

THIRD MILL JUICE.—The sample, where saturation is practiced, is taken from its pump discharge pipe as described under mill raw juice. Where it is not, the spoon sample may be used in the trough. For analysis, see under mill raw juice, *A* and *B* only.

RESIDUAL JUICE.—This is the drip from the bagasse roll of the last unit of the tandem and should be taken continuously, as under first mill juice and analyzed as under raw mill juice, *A* and *B*.

EVAPORATOR THICK JUICE.—The Brix of each tank filled should be taken. Where fluctuation in purity is considerable, as between 70 and 85 for instance, facilities should be at hand for rapid determination of purity. For this and similar work the author has introduced the Pellet continuous polarizing tube.

FILTER PRESS CAKE.—The loss from this source is seldom correctly determined. At reasonable intervals the laboratory messenger should go to the trucks located below the presses and as a press is dumped, break off small pieces from a dozen large cakes; if semi-fluid, a cup should be used. Analyze in the usual way but substitute 25 grams for a normal weight to allow for insoluble material. Periodically the weight of the press cakes should be determined.

THE SUGARS.—Large centrales employ fans for cooling and rendering the sugar more uniform. Where not used, the net weight per sack must be verified when cold and finally loaded. The weight is still held by custom at 325 Spanish pounds of 460 grams each.

Sampling.—A clean and dry galvanized iron box, of about 1 cu. ft. capacity and having a funnel-shaped hopper in the cover, is placed at the sugar scales, to be changed once in 6 hours.

[1] Note.—The "spoon" sampler consists of a large spoon having a hollow handle of copper communicating with a hollow shaft of small piping and discharging through an elbow into a pail charged with formaline. The shaft, driven from the mill-roll, should make about 8 revolutions per minute. The spoon is covered with fine screen to keep out "trash."

From every bag in five, and before adjusting the weight with sugar from the storage bin, the truckman will transfer a pinch of sugar from bag to sample box. As the sample is taken away, the serial number of bags filled will be recorded, in order to arrive at the number of bags represented, in proportion to which the polarization is to be adjusted in taking off averages.

Preparation.—The sugar is poured upon a plate of glass, all sticks and foreign matter removed and thoroughly mixed with a clean steel spatula. Lumps are reduced with a porcelain roller and incorporated with the rest of the sample. Polarize at once. Determine moisture once a day. Composite a small portion from each sample for the semi-monthly chemical statement upon which is to be determined: (1) polarization, (2) true sucrose, (3) dry substance, net, (4) invert sugar, (5) ash, (6) total dirt, (7) ash in total dirt.

When the sugar contains over 1 per cent. moisture, the sample for compositing is to be dried in a water-bath oven for a short time, later correcting the final analytical data back to the basis of the average of the daily polarizations upon the fresh sample.

Polarization.—A normal weight is placed in a funnel and washed into a 100 cc. flask with 50 cc. of water, completely dissolved by rotating, then clarified by basic lead acetate solution and 2 cc. of alumina cream. As a rule not over 1 cc. of lead solution is needed for high-grade centrifugal (96) sugars and from 2–6 cc. for molasses sugars (80–90). Use the minimum quantity necessary for clarification. After the lead and cream are in, allow air bubbles to rise and complete volume to 100 cc. Mix and filter in a carefully covered funnel, discarding the first runnings. Endeavor to polarize at the temperature of dilution.

THE MASSECUITE.—Applicable to either grained or blanc strikes.

Measuring.—Massecuite intended for the crystallizers should be measured after being placed therein, at the moment of entering and for every strike: the same applies to sugar wagons or tanks. The volume of material subject to crystallization in motion for long or short periods or "at rest" for many days, should be known as an important step in control.

An accurate account should be kept of the movement in and out, so that at any time, by consulting the records, a balance can be struck of the exact amount in stock.

Sampling.—Take a portion equal to 2 liters from at least three places as the mass is struck from the vacuum pans, *viz.*, after it is running well, in the middle of the flow and towards the end.

Analysis.—At the laboratory the following tests are made: (1) purity of the mother liquor, (2) Brix by double dilution, (3) polarization.

Purity of the Mother Liquor.—Grained massecuite only. Investigations upon the work of competent sugar boilers has shown that this test has a very important bearing upon economical boiling. The drop in purity may be very variable for equal purity of strike. Immediately after being drawn the sample is to be rapidly turbinated in the small laboratory centrifugal.

The author has devised special, light-weight, tin linings for the centrifugal; these can be kept on hand in any number, they slip in and out easily, collect the whole sample and do away with the necessity of cleaning the centrifugal. It is especially helpful in making a large number of such analyses.

When the lowering of purity is abnormal, either way, the several causes to which it may be due should be investigated. The purity is determined after diluting to 18–20° Brix.

Polarization.—This is made by taking either a normal or double-normal weight of the sample used for determining the Brix by double dilution, according to color, and proceeding as under the sugar polarization, excepting that after filtration the solution must be acidulated before reading, *viz.:* Fill a 50–55 cc. flask to the 50 cc. mark with the clear filtrate, add dilute acetic acid to faint acidity and complete to the 55 cc. mark with water; shake and polarize; increase the reading by one-tenth to compensate for the dilution.

When possible, clarify with neutral lead acetate and thus avoid later dilution.

EFFICIENCY OF CRYSTALLIZERS.—This refers also to work carried on "at rest" in tanks or wagons. The mother liquor is separated by the laboratory centrifugal and its purity determined at 18–20° Brix, when the product is about half discharged from the vessel.

FINAL MOLASSES.—This should invariably be weighed, being too viscid to measure and occluding much air.

Under a suitable arrangement one man can attend to the day's output. The well mixed day's composite sample is analyzed for Brix by double dilution, sugar and purity (calculated).

Daily a quantity in proportion to the amount made is set aside for the semi-monthly complete analysis, as detailed under sugar.

Control of the Boiling.—Much can be accomplished at the pan-service tanks to bring about regulated, economical boiling. If they are uniform in size, deep, rather than broad, large rather than small and with facilities for rapid and thorough cleaning, much will have been gained. The several molasses are diluted to an exact Brix, generally 60° and heated to 70° C. at a "blow-up" station over these tanks. Every tank and piece of apparatus in the factory should bear a number. These tanks should be graduated to 10 hectoliter units, vertically, upon a scale having an indicator actuated by a float.

Before the contents of a tank is started for feeding the vacuum pan the following data concerning the material is to be entered upon the sugar-boilers blackboard: Date, hour, tank No., name of material, temperature, Brix, and where possible, the purity.

Upon the basis of this data the massecuite purity is established and the finished product should agree within two points of the estimated.

WEIGHTS AND MEASURES AS AFFECTING MILL EXTRACTION.—(1) The *Sugar Cane* is bought by the Spanish ton as a matter of tradition. From the time it enters the factory the metric system should apply.

It suffers variable shrinkage in weight before it is ground and proper chemical control has necessitated the present policy of weighing it upon a beam scale at the hoist just before discharging into the mill hopper.

(2) The *Mill Raw Juice* is generally measured and when properly carried out, this will furnish a basis of calculation for the amount of sugar introduced (the real object) quite as satisfactory as weighing. Foam and occluded air have been the cause of some agitation in favor of weighing the juice, but this entails considerable expense that the author does not consider always justified. The installation of measuring tanks on the capacity basis of 3 hectoliters net per ton of cane per hour (*i. e.*, 150 hectoliters, net, for 1200 tons daily capacity) eliminates occluded air to a negligible quantity, while a float of special size and shape enables the true level of the juice to be determined with exactness and simplicity. This is shown in Fig. 6. It is made of galvanized iron, conical at both ends, weighted with $9^1/_2$ lbs. of lead and provided at the top with a tube 2 inches long for holding a very light rod of wood, which in turn bears a double arrow and which is brought to a fixed point upon a scale.

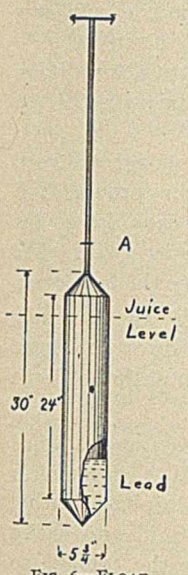


FIG. 6—FLOAT

The point A also serves as a definite spot from which to gauge the juice level for juices of different densities. The accompanying table shows the fluctuating juice level with varying Brix, the tank capacities being calculated accordingly. These levels are actually determined by floating the instrument in diluted molasses of juice density in a tall cylinder. The float rises and falls in a cage. It rests for by far the greater part well below that part of the juice containing the unliberated air and allows no air to collect below it. It measures to within one-sixteenth inch under working conditions.

Brix reading	Distance A, to juice level
21.3	17.3 cm.
15.6	15.9 cm.
9.6	14.5 cm.
4.8	13.2 cm.

The specific gravity of the juice may be adjusted for varying temperature by the use of Gerlach's table.

Measuring tanks should be calibrated by weighing water into them until duplicate weighings agree within the polariscopic error as determined by the limits of the volumetric method using the Schmidt table.

(3) *Water of Saturation.*—The average temperature of this must be known in order to arrive at its weight, as it is probably invariably measured. Duplicate tanks serve the purpose of measuring, well, but a good water meter is sufficient, provided simple means are at hand for occasionally checking it, under actual working conditions.

(4) *The Bagasse.*—The weight of this, as determined by the formula Cane + Saturation Water — Raw Mill Juice = Bagasse, is fully satisfactory, since the chemical control fails, in any case, where any one of the quantities is in error.

Stock Taking.—The short season requires frequent stock taking and the author recommends that this be taken once a week until it has been demonstrated that the factory is working normally, but after this, with cane work, once a month is sufficient for the fully detailed report. For this weekly check, advantage should be taken of a stop and all the products of the factory composited into one laboratory sample upon the basis of the several volumes, when one analysis and one calculation will give the desired information.

THE SUGAR ACCOUNT

All the sugar in the cane must be accounted for and brought up to a sum total of 100 per cent. The following form is a good general example.

General sugar account	Per cent. basis of cane	Sugar in cane 100 per cent.	Per cent. basis of juice	Tons
Sugar in first sugar	9.14	72.67	78.97	4070.5
Sugar in second sugar	1.11	8.84	9.59	494.5
Sugar in total sugar	10.25	81.51	88.56	4565.0
Sugar in press cake	0.06	0.53	0.58	29.6
Sugar in final molasses	1.16	9.22	10.02	516.5
Sugar in undetermined loss	0.10	0.77	0.84	43.3
Sugar in mill raw juice	11.57	92.03	100.00	5154.4
Sugar in bagasse	1.00	7.97	..	446.5
Sugar in cane	12.57	100.00	..	5600.9

In a full technical account, the above represents about 20 per cent. the data, the rest including averages of the analyses made of all the products, together with tons of cane ground, bags of the various grades of sugar made, time lost for different causes, data connected with the mill efficiency and the percentage yield of commercial sugars upon the basis of the cane.

The undetermined loss is due chiefly to the impossibility of accounting for all material involved in any undertaking.

There is loss from spilling, from inversion, long action of heat and errors in weights and measures with limits in accuracy of analyses. When this figure is 1 per cent. the total sugar in the juice it indicates good work, when it is 0.5 per cent. it is excellent work.

GENERAL METHODS

BRIX BY THE HYDROMETER—All solutions up to 70° Brix are to be tested by the hydrometer directly, after the removal of air bubbles.

If the reading is not made at either $17^1/_2$° C. or 20° C. corrections will be made by means of tables to be found in any standard text-book.

DOUBLE DILUTION METHOD. [For products of over 70° Brix.]—The absence of cold water in tropical work precludes the cooling of solutions that have once been heated. The following method has been found most practicable: Use nickel-plated, copper beakers of such size that the fist may readily be introduced. Select two of about equal weight and place upon opposite pans of the balance; from the heavier, file or cut off the material around the upper edge until they exactly balance. With the beakers now upon opposite pans of the balance, in one place about 400 grams or any convenient quantity of the material; into the

other pour water until exact balance is secured; remove the beakers from the balance and pour the water of the one into the other containing the material: by means of the hand, mix the two until the last grain is dissolved; do not remove the hand until the operation is complete, in fact, endeavor to keep the hand equally submerged all the time.

Allow to stand until air has risen; take the Brix by the hydrometer, correct to standard temperature and multiply the result by 2. If the beaker used for water be only lightly greased within, it will deliver the water to the other to the last drop, thus obviating pouring back.

THE DILUTION TO 18 TO 20 BRIX.—For rapid control of the process of boiling, based upon the purity, all products will be reduced to uniform density within these limits before making the test. Simple as it seems, the average chemist is longer in learning to perform this test with unfailing accuracy than any other test in sugar manufacture, this being especially true with products containing grain in suspension. The fault lies in losing some of the material by spilling before all the grain has been dissolved or before the mixture is absolutely uniform.

(1) For products positively known to contain no grain: Select a cylinder 15 inches tall by $1^1/_2$ inches diameter, having the upper edge of such a shape that it may be perfectly sealed by the palm of the hand; fill about two-thirds with water, pour in from about 110–120 cc. of the material, adding more water until within about $1^1/_2$ inch from the top; seal tightly with the palm of the hand and shake vigorously until mixture is intimate; the result should always be a solution too dense rather than too thin; pour out a portion and add water if trial test shows too dense, mixing as before; continue this until proper figure is reached.

(2) For products known or suspected to have grain: Select two-liter enamelled cups of unbroken surface—these have no corners in which the grain may lodge and thus escape solution. Pour into the cup about 500 cc. of water, add about 200 cc. of the material and by means of the hand manipulate the mass until no more grain can be felt; transfer the solution to the cylinder and proceed as under (1) until the density is reduced to 18–20° Brix.

DRY SUBSTANCE.—In the cane sugar industry this is a purely empirical process, and close conformity to certain conditions is necessary to secure even comparative results. The breaking down of the levulose molecule at temperatures above 80° C., the oxidation of non-sugars, the formation of acids that in turn produce more invert sugar which continues to decompose, make it impossible to dry to constant weight at atmospheric pressure, therefore a vacuum should be employed: when the temperature is held at 70° C., the vacuum should not be under 25 inches and a slow current of dry air allowed to pass through; weigh every 2–3 hours until constant in weight.

In the absence of any vacuum the following method may be used. It is based upon the official method of the Association of Official Agricultural Chemists, *Bull.* **107** (revised), U. S. Bureau of Chemistry, p. 64.

Place in a light crystallizing dish, provided with a watch-glass cover and stirring rod, 25 grams of broken glass in quite uniform pieces the size of coarse sand and washed free from dust. Dry quickly at 120°–140° C., cool in a desiccator and weigh. Of sugar, take 10 grams; of molasses, 5–6 grams; of mill raw juice, 35 grams. Dry for exactly 10 hours at 98°–100° C. (boiling-water jacket). The time for drying the juice should begin with the disappearance of the water.

SULFATED ASH.—About 3 grams of sugar are used and a proportionate amount of other products; if moisture exceed 25 per cent., as in cane juice, the water must be evaporated on the water bath. The true ash is calculated by multiplying by the factor 0.9.

CONDENSED WATERS.—These include all the main hot water collectors, the pan and evaporator tail-pipes. To be tested for sugar with alpha-naphthol. Some form of continuous sampler is advised.

The Test.—(Fruhling and Schultz, *Anleitung*, p. 166.) To 2 cc. of the suspected liquid add 5 drops of a 20 per cent. alcoholic, sugar-free, alpha-naphthol solution, then 10 cc. of purest sulfuric acid and shake thoroughly. With 0.1 per cent. sugar the color is so deep as not to be transparent; with 0.01 per cent. a bright red wine.

Boiler Water.—Determine the acidity of the boiler-feed water and the alkalinity of the boiler water every 12 hours.

For the test, take a small amount from each boiler in service, titrate with standard acid (1 cc. = 0.001 CaO), using methyl orange as indicator. Report as grams CaO per 100 cc. Report number of pounds soda-ash used per 24 hrs.

PREPARATION FOR CLERGET TEST AND INVERT SUGAR. *Juices.*—Determine the sp. gr. Clarify 500 cc., strained and air-free, with neutral lead acetate sol. (50° Brix); dilute to 550 cc., shake well and filter. Make the direct reading at as near 20° C. as possible. To about 150 cc., add ignited Na_2CO_3 to scant phenolphthalein alkalinity, stir and let stand 15 minutes; filter. Use exactly 75 cc. for Clerget by the Herzfeld inversion method. Use about 5 cc. for invert sugar test.

Molasses.—Wash $2^1/_2$ normals into a 500 cc. flask, clarify with solution of basic lead acetate, mix, rotate to expel air, dilute to the mark and filter; collect 250 cc., neutralize with glacial acetic acid, double the amount of acetic acid; throw the filter and precipitate into the clear liquor, mix thoroughly and again filter. Polarize and multiply by 2. Remove the lead with Na_2CO_3 and proceed as under Juices for Clerget Test and Invert Sugar, using 15–20 cc. for the latter. True sucrose should be calculated by the following formulae.

Juice	Molasses
$\dfrac{\dfrac{26 \times \text{invert pol.}}{68.18 \text{ sp. gr.}} + \text{direct pol.}}{142.66 - 0.5\,t}$	$\dfrac{\text{Direct pol.} - \text{invert pol. } (2.67)}{142.66 - 0.5\,t}$

Herzfeld's Inversion Method.—This will be found in

detail in any standard work. After inversion is complete and while still in the water bath, add 1 gram powdered zinc, heat for another 5 minutes, pass through cotton, wash the cotton until 100 cc. is obtained at 20° C.

Invert Sugar Determination.—To exactly 10 cc. of Fehling's solution, blue, and 10 cc. of Fehling's solution, white, in a 250 cc. flask, add the solution under test and enough distilled water to make 50 cc. Boil cautiously upon a square of asbestos, having a central hole, for 2 minutes, then cool quickly. Add 10 cc. of 20 per cent. solution KI, rotate, add 10 cc. of 25 per cent. H_2SO_4, then nitrate with $N/10$ thiosulfate. Work rapidly throughout, running in by 3–4 drops at the end.

Run a "blanc" upon the Fehling solution, under precise conditions of analysis to determine its value in terms of thiosulfate. Use the table of Meissl[1] and Hiller to calculate results. Careful determinations by several chemists have shown that not all of the copper present can be accounted for with cane products, and the following table of factors was been worked out:

Table of Invert Sugar Factors

For use where the copper reduced is determined by difference

Cc. decinormal thiosulfate used	Juices	Sugars		Molasses				
	± 6	± 1.2	± 3.4	± 15	± 32	± 39	± 46	± 55
	= I	= I	= I	= I	= I	= I	= I	= I
5	0.981	0.928	0.950	0.926	0.996	0.934	0.960	0.956
10	0.998	0.919	0.966	0.961	1.013	0.980	0.983	0.988
15	1.007	1.013	0.989	1.011	1.017	1.011	0.988	1.010
15.5	...	...	...	...	...	...	...	1.015
20	1.016	1.048	1.016	1.021	1.020	1.032	1.001	1.001
25	1.045	...	1.030	0.963	1.022	1.047	1.002	0.971

Example.—Used in "blanc" titration, 27.75 cc., in back titration, 6.93 cc., net utilized, 20.82 cc. 0.00636 $\times$ 20.82 = 0.1324 gram Cu, $\frac{0.1324}{2}$ = 0.0662 = Z. W = 8.275 grams material taken for determination. Polarization = 10.08. 0.0662 $\times$ 100/8.275 = Y = 0.8 per cent., 100 $\times$ 10.08/10.08 + 0.8 = 92.7 = R. 100 — 92.7 = 7.3 = I. Since 20 cc. of Fehling's solution were used instead of 50 cc., Cu/2 or 0.0662 gram must be multiplied by 2.5 to find the factor, F., which equals 0.165 gram Cu. By the Meissl and Hiller table, factor = 53.1. Hence 0.1324 $\times$ 53.1/-8.275 = 0.85 approximate invert. Referring to the above table of corrections results in that of 1.016. 0.85 per cent. $\times$ 1.016 gives 0.864 per cent. corrected.

[1] Spencer's "Handbook for Cane Sugar Manufacturers," pp. 129, 130.

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STERILIZATION OF WATER SUPPLY AT TRENTON

By H. C. Hottel[1]

Received January 6, 1913

Statistics issued by Dr. A. S. Fell, Health Officer of the City of Trenton, show that there have been 100 cases of typhoid fever in the city during the year 1912. For the previous five years the number of cases averaged over 250 annually. The total for the year 1911 was 302 cases. The highest number of cases reported in any one week in 1912 was 7 while the highest number in 1911 was 24.

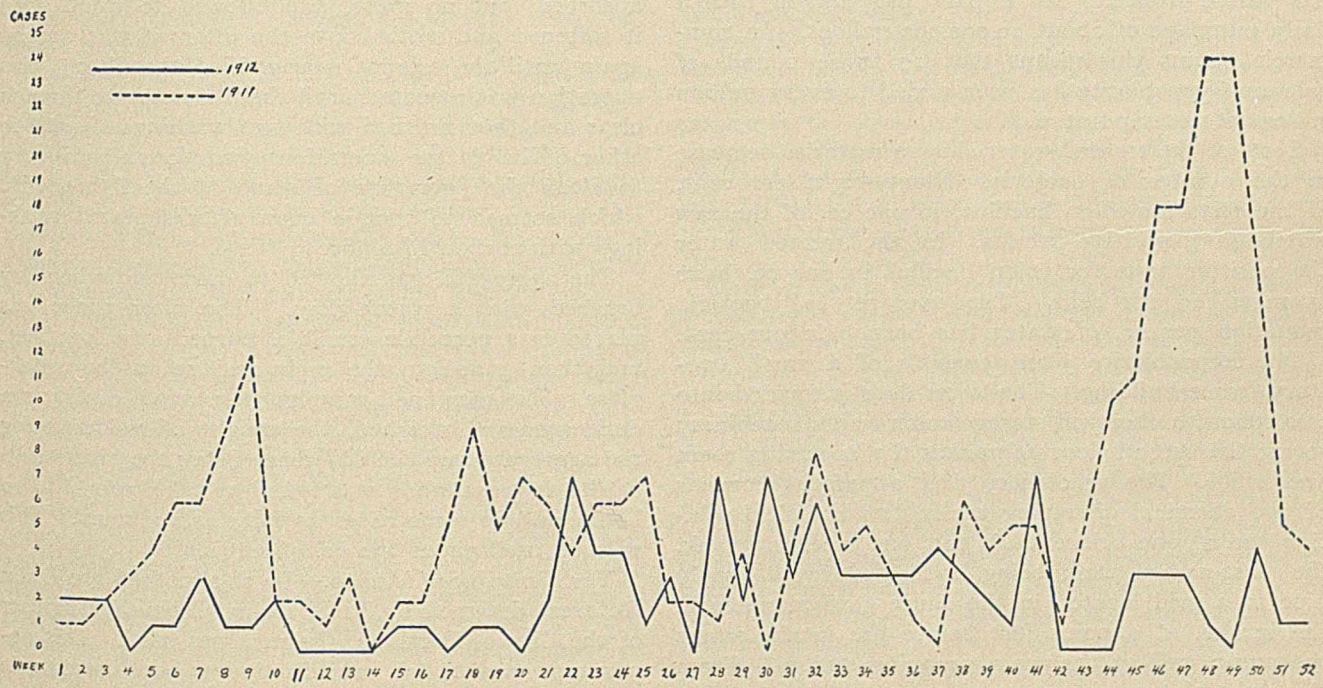

Chart showing cases of typhoid reported in Trenton, by weeks. Cases in 1911 are shown by the broken line; Cases in 1912 by the solid line

The drop in the number of typhoid cases is attributed to the use of calcium hypochlorite to sterilize the drinking-water supply, raw Delaware River water. The chart shows in a graphic manner the decrease in typhoid.

The following table shows the wide range between the two years. In 1912 there were only nine weeks in which more than three cases were reported while in 1911 there were 33 weeks in which more than this number were recorded.

[1] City Chemist, Trenton, New Jersey.

Cases each week	1911	1912
0	3 weeks	12 weeks
1	6 weeks	13 weeks
2	7 weeks	7 weeks
3	3 weeks	11 weeks
Above 3	33 weeks	9 weeks

The sterilization plant was installed in Nov., 1911, at a cost of $4,500 and has been in continuous operation since that time. It is not intended, however, to always treat the water in this manner, as plans have been completed and the contract awarded for the erection of a mechanical filtration plant, with a capacity of 30,000,000 gallons of water daily at a cost of $330,000.

When first installed the hypochlorite plant was under the supervision of the New Jersey State Board of Health but in March, 1912, the city took charge of the operation of the plant.

The hypochlorite purchased averages about 35 per cent. available chlorine, and treatment was begun with a strength of 0.4 part to the million of available chlorine. This was found to be insufficient and was later raised to 0.8 part and during the year 1912 has varied from 0.7 to 1 part per million with a daily pumpage of about 20,000,000 gallons. In commercial terms this means that 17 to 24 pounds of calcium hypochlorite has been added to every million gallons of water pumped.

Tests of the treated water show a material decrease in the number of bacteria. Ninety-eight per cent. of the tests for colon bacillus, in one cc. of the raw water gave positive results. In the treated water the positive tests for colon bacillus in one cc. have averaged 20 per cent. The average 20° bacteria reduction per cc. of water has been 94.1 per cent.

The hypochlorite plant consists of a small two-story frame building. The lower floor is divided into two rooms, a chemical storage room and a machinery room; the second floor comprises the operating room and office. The machinery and apparatus consists of two chemical dissolving tanks, two chemical solution tanks, one orifice tank and one receiving tank, the last-named tank being connected by a supply pipe to a grid located in the sluice gate manhole in the intake, at a point just before the intake enters the pump well at the main pumping station. From this point the water is pumped to a 110,000,000 gallon distributing reservoir, it requiring about 90 minutes to reach the reservoir from the pumping station. The chemical is dissolved to a 2 per cent. solution and the orifice controls the rate (in gallons per minute) at which the solution is added to the raw water. The plant is under the supervision of Chief Engineer Lenox of the pumping station and, as it is necessary to have some one in constant attendance, it requires the services of three men working on eight-hour shifts.

That the hypochlorite treatment is being used extensively in drinking-water supplies of American cities is attested by the report of the Ohio State Board of Health.[1] It states that information has been received in regard to the drinking-water supply of 99 cities of over 25,000 population in the United States, and that 40 per cent. of these cities are using hypochlorite to purify the water.

[1] Ohio State Board of Health, Mouthly Bulletin, Oct., 1912.

THE DETERMINATION OF MINERAL AGGREGATE IN BITUMEN PAVEMENTS

By C. C. O'Loughlin

Received November 21, 1912

The experiments described below were carried out as a comparison of the filtration and centrifugal methods for the determination of mineral aggregate in bituminous substances.

The following samples were used for both methods:

Refined Trinidad asphalt	51.60% bitumen
Refined Bermudez asphalt	96.22% bitumen
California "D" grade asphalt	99.94% bitumen
California "G" grade asphalt	98.86% bitumen

The Filtration Method.—Ten grams of the asphaltic mixture were weighed and placed in a $2^1/_2''$ funnel, which contained a 9 cm. Schleicher & Schull No. 597 filter paper; a 250 cc. Erlenmeyer flask was used to catch the filtrate.

Carbon bisulfide was run onto the sample until absorbed, but no more, and allowed to stand until it softened and settled into the filter; it was washed again until the solvent nearly filled the filter. This operation was repeated until the filtrate came through clear and then finished with two washings of petrolic ether. The latter operation was found necessary to clean the aggregate of some of the lighter oils, which seemed to remain even after repeated washings with carbon bisulfide.

The aggregate was allowed to drain and when dry brushed onto a watch-glass. The filter paper was placed in a porcelain crucible, burned at a low heat, and then added to the main residue on the watch-glass. The combined samples were dried, cooled in a desiccator and weighed, the weight of residue being the aggregate and the difference being the bitumen.

The above method was found to take from 4 to 10 hours, and in some cases as long as 20 hours. This method used about 250 cc. of solvent.

The Centrifugal Method.—In this method the Dulin Rotarex, designed by R. S. Dulin, Chemical Engineer of the City of Portland, Oregon, was used. This apparatus consists of an aluminum bowl receptacle for the sample, which is $3^1/_2''$ in diameter and equipped with a suitable cover; the cover is fastened on by means of a funnel-shaped screw, through which the solvent is added; this also allows the escape of gases. The above is enclosed in a copper shell, provided with a spout at the bottom side, through which the waste liquors drain; the shell is provided with a two-piece cover, the smaller one being removed to add additional solvent. The machine is run by electric motor directly underneath. The filter of proper diameter is placed between the bowl and cover, and when the machine is in operation the solvent is thrown to the sides, passing through the filter, carrying with it the soluble matter.

Fifty grams of sample were placed in the bowl, the filter put in place, the cover fastened on and 50 cc. of solvent added; the machine was rotated until

the solution ceased to run out of the spout. This operation was repeated and a final extraction with 25 cc. made; the cover was removed and the aggregate allowed to stand in the bowl for about 2 minutes; it was then brushed onto a watch-glass and finished as in the filtration method, burning the filter if necessary, which was not very often the case.

It was found that the centrifugal method on an average of several samples took from 10 to 15 minutes for the complete determination, and that the results check very closely, due to using a large amount of sample for each determination and thereby reducing the error. It was not necessary to finish with petrolic ether, as the results did not differ to an appreciable amount when using ether after the regular extraction. The average amount of solvent used for a determination was 125 cc.

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ADDRESSES

THE PROBLEM OF INTERNATIONAL CONGRESSES OF APPLIED CHEMISTRY

By Bernhard C. Hesse[1]

The object of the following is to present in concise and impersonal form some of the lessons that seem to me to be read out of the experience obtained in preparing for and in participating in the conduct of the Eighth International Congress of Applied Chemistry. I am persuaded that these notes may be useful, because of the opinions expressed by most of my friends with whom I have discussed the subject, and to whom I have presented my views.

In reading these pages the fact must not be lost sight of that they in no wise deal with the social, the factory-inspection, nor the private entertainment side of such a Congress, nor with anything that in any way deals with any "host and guest" aspect of these Congresses, all of which I regard as with perfect propriety not a matter of any concern whatever to these Congresses as an institution; they are the pleasure and the reward of the host country alone. This article is limited strictly to the outline, preparation and conduct of the scientific side—the actual hard work of the Congress about which all other functions are supposed to cluster. This work is held out as being the real justification for the existence of these Congresses and constitutes their real merit in aiding the progress of mankind.

My own conclusions, based upon what is contained in the succeeding pages, may, in part, be summed up as follows:

I. The International Congresses of Applied Chemistry of the past have been loaded down with such an overwhelming proportion of extraneous matter that their true business has been entirely submerged. The only remedy lies in curtailing such matter and limiting the activities of the meetings to matter of international and debatable character all pre-arranged, with leading discussions in print and the whole matter prepared for *viva voce* discussion.

II. Sectional meetings should be on alternate days and the intervening days be used by sectional secretaries and participants in discussion in definitely making up the record of the meetings of the preceding days; all matter not in the hands of the sectional secretaries within a reasonable time, say, three working days after the close of the Congress, should be denied admission to the printed record.

III. All manuscript *must* be typewritten and all other requirements of the printer fulfilled by all authors, otherwise the papers should be returned by the Committee to their authors.

IV. The only way that the persistent and thoroughly correct demand of members for rapid delivery of the printed proceedings can be complied with is to require all participants to be prompt in supplying their manuscript; rapidity of execution requires efficiency in organization and contributing members are part of such an organization; they must all discharge their respective duties at the same efficiency rate that they expect the officers in charge to achieve; no committee can print from nothing, nor can it proceed any faster than the slowest contributor.

V. The demands made upon the Congress for the treatment of scientific matter have grown out of all proportion to the financial ability of the Congress; prudence and caution as well as a decent regard for financial propriety all demand that that part of the undertaking of the Congress be cut down to a dimension commensurate with its income.

The reasons for these and other conclusions will be made clear as this article proceeds.

Inspection of the Reports of all preceding Congresses, inclusive of the Eighth, discloses an evergrowing tendency to increasing bulkiness, with little or no increase in the efficiency or ripeness of the actual, crystallized work of these Congresses; namely, the discussions and the resolutions offered to and considered by the Congress meeting as a whole in its last session and the Reports of Commissions or Committees created at a preceding Congress and directed to report at a subsequent Congress.

Everyone must be struck by the enormous amount of material brought together at the Eighth Congress. On suitable examination all will, no doubt, agree that more than 90 per cent. of all such material (however valuable *per se* it may be) is not fitted for nor adapted to discussion in a meeting where international interests are supposed to be primarily or almost solely involved; also that these 90 per cent. would have been written and published without the stimulus of such a Congress, in the publications now so plentifully provided all over the world. The final result is that, in an endeavor to do seeming justice to the great bulk of material offered, the real and important work of the Congress (the formulation of an expression of opinion on the part of the Congress on certain topics of international import, scope and interest) is drowned while resolutions and reports are put through hurriedly and without due consideration by the various sections, trusting to the International Commission or to the Congress itself to take care of any imperfect work.

Clearly, it is not the object of these Congresses to provide merely another vehicle of publication for papers that would be written and published without the stimulus of the Congress, nor do men go to the expenditure of time, money and effort to attend such gatherings merely to hear papers read that would reach their library desks automatically. The object of these Congresses must be to bring forth a class of communications and results which cannot be created nor accomplished by the societies, associations and publications, now so plentifully at hand. The results that these Congresses must be intended for, are those for whose accomplishment direct personal contact and direct attrition of minds of men of different and differing opinion on debatable questions are prime requisites and essentials so that out of these various opinions some order, some system, some agreement, some progress may arise. There is very little to be gained by discussing a paper which merely tells you the physical or chemical constants of a limited number of substances, cer-

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tainly not enough to make it worth the while of any considerable body of men to travel across oceans and continents to attend the reading of such papers.

In addition to these discussions of debatable questions there is the coöperative work needed for the solution of problems of international importance and the receipt and discussion of reports thereon by the Commissions appointed by preceding Congresses; like the resolutions mentioned above these too are caught in the maelstrom of papers, suffer from want of discussion and do not receive that attention which they require and merit. Yet these resolutions and reports are the *only* work that depends upon and requires such gatherings. The world does not now have ample volunteer and non-governmental machinery for them, and they have not held that prominence and that importance in past Congresses which their potential value justifies and demands.

It is easily manifest that past Congresses have not provided for the Commissions reporting to them that machinery best adapted to the attainment of worth-while results, nor have they given to the results reported that attention and publicity to which their history and origin entitles them. To serve on such a Commission or Committee is wholly a labor of love; the money grants made by the Congress are wholly insufficient to give proper publicity to the *results*, not to speak of the expenses of organization and the needed clerical work if the task is to be well done. When such a Committee does report, its reports are not accessible to the members of the Congress-at-large. It is very easy for a Congress to say to a certain Committee that such and such work shall be done by the time the next Congress meets, but it is anything but easy to accomplish that thing in the allotted time and present it in a form accessible for discussion; furthermore, when it *is* presented for discussion those receiving the report should be acquainted with its nature sufficiently in advance to enable them to prepare a useful, helpful and creditable discussion and criticism, both destructive and constructive, of the work on which so much time, labor and thought have been spent. Perfunctory acceptance or perfunctory discussion or perfunctory discontinuance of labor voluntarily assumed in the belief that it is of wide and general importance and thereupon conscientiously performed with considerable sacrifice of time, effort and labor is not conducive to causing busy men to expose themselves to such sacrifice for so unsatisfying if not distinctly discouraging reward or treatment. If such treatment continues, there can be but one result—men less qualified for the work will undertake it and reports of lesser and lessening value will be produced. If the value of such reports is to increase and these Congresses are more closely to approach the *only* field of real international usefulness open to them, then more thought and more reflection must be given to the outline of the work to be performed when a Commission or Committee is created, more consideration and discussion must be given to the report when ready, and the report itself must be more widely distributed and much more in advance of the meeting than heretofore; besides more financial assistance must be given: the same is true of resolutions expressing the conclusion or consensus of opinion of the Congress on any debatable subject or topic.

Of the 18 reports by Committees or Commissions that should have been made to the Eighth Congress only 2 were so made; further, of 25 resolutions adopted by Sections only 9 were placed before the Congress because of the failure of the sponsors of the remaining 16 to perform their respective duties.

Now, how can this be done? Merely to point out a fault without suggesting a remedy is an easy thing to do. I fully realize how difficult it is and perhaps how egotistical it may seem to propose such a remedy; nevertheless, I run the risk of error and misunderstanding and propose what seems to me to be one feasible plan, but I must not be understood to mean that I regard this as the *only* or as a *complete* solution. It is brought up for the purpose of discussion in the hope that something will result from it which may in the future prevent these Congresses from falling within themselves on account of their unwieldiness, bulkiness and their poverty in results actually accomplished.

The plan is as follows:

I. Abolish all papers on promiscuous subjects.

II. Reduce the number of sections to four, say
   1. Inorganic.
   2. Organic.
   3. Analytical
   4. Administrative.

III. Confine the work of each section to a stated program mapped out in ample time in advance of the meeting and limit its work to deliberation on reports submitted and resolutions to be proposed to the Congress as a whole.

IV. Have sectional meetings on alternate days only, *e. g.*, Monday, Wednesday and Friday with the Final General Meeting of the Congress on Saturday afternoon. Tuesday, Thursday and Saturday morning to be given up to sight-seeing, works inspection and social and other matters generally; this time must also be used by the sectional secretaries and participants in discussion in properly assembling and writing the minutes of each meeting and in preparing the proper matter for the daily paper of the Congress giving the text of the resolutions finally adopted by each section.

V. Give Monday, Wednesday and Friday afternoons, if desirable, to general lectures of the type heretofore held; these would provide ample opportunity for the broad presentation of chemical topics to the Congress and to the public in general.

VI. Hold Sectional Meetings from 10 to 12 and from 1 to 3 and general lectures at 4.

The topics on which resolutions are to be proposed to the Congress should be determined by the Committee in charge of the Congress in any way suitable to it; one way might be to present the Committee's views to each of the societies, institutions and associations represented by delegates at the last preceding Congress and to ask for criticisms of the list of topics so submitted and for suggestions as to those fitted to take part in the leading discussions; the suggestions of the Committee together with those received from these above defined organizations with respect to all topics should be sent to the technical press on a stated day as well as those left open for further criticism by the general chemical public until a certain other stated day. The Committee in charge of the Congress should select from all these topics those that it deems suitable for consideration and announce their definite selection together with their numerical designation, their assignment to sections and their places upon the program *not less than eighteen months in advance of the meeting* and send such statement to the various organizations and to the technical press. Further, this Committee should select for each debatable topic definitely put upon the program one individual to discuss the topic broadly but concisely not to exceed 4,000 words, and then assign to each of two different view points of the subject one man who shall not exceed 2,000 words (all those suggesting topics should also suggest the names of those who might be in position to discuss them to advantage, but these names should not be made public); the leader's article should be ready 120 days before the Congress meets and be sent to the other two writers, whose two articles should be with the Committee ninety days before the Congress meets. These individuals should be selected and announced one year before the Congress meets. These three contributions should each carry a proposed resolution or resolutions for submission to the relevant section. Ninety days before the Congress meets no further suggestions of topics for resolution or report should be received and no further memberships or delegateships accepted. This period of ninety days will all be needed to enable the Com-

mittees of the Congress to make proper preparation for the prompt despatch of business and the proper social and other diversions; ninety days before the Congress meets all foreign central committees and organizations should telegraph the Congress Committee the number of members in their jurisdictions and how many of them will probably attend the meeting together with the probable number of accompanying ladies and should follow this telegram up with a typewritten list, in duplicate, of all members in their respective jurisdictions together with the post-office address of each member; the same should be done with respect to those members who have declared their intention of attending and so far as possible with their addresses while attending the Congress.

Ninety days before the Congress meets all reports to be presented should be in the hands of the Committee; these reports together with all the discussion papers, should then be printed and put in the mail for distribution to members of the Congress sixty days before the Congress meets together with a copy of the program in full, giving the date and time of day when each topic or report is to come up for action. In this way every member would have foreknowledge of all the principal points at his home not less than thirty days before the Congress meets and all who are interested would have ample time to prepare creditable and really valuable criticisms and discussions of definite subjects and topics; it might also be feasible to permit absent members to send in their criticisms in writing for presentation to the section in charge, but this suggestion seems to me to be of doubtful value.

The Committee in charge of the Congress would, of course, add to this printed volume a list of all the topics presented to it as subjects for discussion and which it did not accept so that the various sections could consider them also in laying plans for the succeeding Congress; after they had placed their recommendations with the Congress, the latter, at the meeting of Saturday afternoon, could have the benefit of all these suggestions and of the various valuations placed upon them in deciding what should be, in part, the leading topics for the next Congress.

At the sectional meetings where resolutions and reports are discussed, suitable stenographic or reportographic minutes should be taken and after proper editing printed in a supplemental volume together with the final action on each topic, resolution or report. This would result in a compact, concise and clear exposition of the real work of the Congress, free from all the distraction of extraneous and promiscuous papers; of smaller dimensions than its predecessors, doubtless and necessarily, but therefore all the more valuable and meritorious.

In the program proposed there are provided six meeting periods for each section, or a total of forty-eight meeting hours. If, in each two meeting hours, one good resolution or expression of consensus of opinion can be adopted; or one good report received, thoroughly discussed and further feasible work mapped out; or a new commission established with ample and explicit working directions; the real and only field of international congressional work will have been covered in a fairly efficient manner and progress towards complete accomplishment and achievement of that real work will have been made. In this manner the attention of those in attendance at sectional meetings will be riveted on one, and only one, topic at a time; having two hours to make something out of the predigested subject-matter, with no further chance to consider it, the probabilities are that proper consideration will be bestowed upon these subjects with results bound to be more nearly worth while than any in the past; at any rate, if under these conditions worth-while results be not accomplished the responsibility for such non-accomplishment cannot be shifted to the presence of distracting papers. Each section would be known as being solely and alone responsible for the good or bad quality of the work turned out.

The responsibility of selecting worth-while topics, properly assigning them to sections and disposing of them in the program would be wholly upon the Committee in charge of the Congress; with only twenty-four items of business to arrange, it is clear that the task of selecting good and worth-while topics and placing them in the program is not so difficult as it might, at first glance, appear to be. Certainly, not so difficult and unsatisfactory as trying to arrange 789 papers to suit as many authors and several times that number of auditors.

It may be objected that this would greatly reduce the attendance at the sectional meetings; if it reduces that attendance to all those actually interested and prepared to take part in the work no possible harm can come to the Congress and its work.

From the report of the Secretary of the Eighth Congress (see p. 344, this issue) the following table, which is self-explanatory, is deduced:

	Papers presented	Papers read by authors	Per cent. of papers not read by authors	Attendance	Participants in discussion	Per cent. of attendance represented by participants in discussion
Sectional meetings	546	286	47.6	3,956	610	15.42
Joint sessions(a)	157	96	37.2	2,650	250	9.4
Grand total	703	382	45.6	6,606	860	12.9

(a) Not including three joint sessions at which discussion was not invited.

A total of 789 papers was presented to the Eighth Congress; of these 703 were presented to the meetings by the authors' representative or by title, and 86 were not presented to the meetings in any form whatever, although printed in the final report. Therefore, almost half the authors did not appear to defend or sustain their papers, 11 per cent. did not even offer their papers to the meetings and only about one-eighth of all persons in attendance made any remarks on the papers presented; surely the remaining seven-eighths might just as well have been elsewhere so far as the real work of the Congress is concerned.

At the closing general meeting of that Congress fewer than 400 were in attendance and of these only 15 or 3.75 per cent. took part in the discussions, *i. e.*, less than one per cent. of all who had registered took part in the final deliberations of that Congress. It further appears from the same report that eighty of the offered opportunities for meeting were not used and that not to exceed 217 of the 530 sectional meeting hours placed at the disposal of the Congress were utilized. Out of 217 actual meeting hours only 169 were used for reading and discussing papers, leaving 48 hours, at most, for the consideration of the total of twenty-five resolutions introduced and for the transaction of such other business as may have engaged the attention of the respective sections. It is perfectly safe to say, however, that these forty-eight hours were not nearly all used for discussions of resolutions.

It therefore seems not unreasonable to expect that a grand total of forty-eight hours of work devoted to a grand total of twenty-four different topics by four different sections, will produce a concentrated and compendious and very much worth-while piece of work—one in which the real matter is not buried under a mass of irrelevant matter, however interesting and otherwise valuable that matter may be.

This concentrated and classified program of the scientific work need in no way whatever interfere with any other of the functions incidental to such Congresses; quite the contrary, men who have undergone such concentrated and responsible work are all the more entitled to relaxation and enjoyment, and are all the more entitled to the privileges of factory and works inspection; they will have earned them many times over.

This plan of procedure would not only produce a final report of greater value, accessibility and utility than any of its predecessors, but would materially lessen the labors of the Committee in charge of the Congress and permit it to concentrate more

thoroughly on the actual and vital things needful for the accomplishment of the proper work of the Congress because these would thereby be greatly lessened in numbers and scope; necessarily with that it would prevent what cannot be, in the light of the preceding, anything but a waste of money and substance. The printing of 90 per cent. of needless matter of the Eighth Congress called for the use of upwards of fifty-four tons of perfectly good white paper (representing in value 1,080 membership fees) and this, to all practical intents and purposes, is a waste of just so much good material and membership fees, a thing which chemists, above all other professionals, should not tolerate nor encourage. I am in position to know that this unnecessary printing and the labor connected with the collection and handling of these papers, cost the Eighth Congress substantially 6,600 membership fees, whereas the paid-up membership was only 4,163 or roughly 63 per cent. of these needless expenditures. If the policy that seems heretofore to have governed Congresses continues, these needless expenditures of time, labor, money and effort will increase and sooner or later assume far greater relative proportions with respect to income from memberships and therefore must inevitably destroy the support given these Congresses by chemists-at-large; finally these Congresses must of necessity disappear—a circumstance to be regarded as nothing less than a calamity since the field for these Congresses does exist and there is need of the work they can perform.

Needless to say, no business enterprise could survive such disproportion between income and outgo and no institution operated on such inefficient lines has any real claim to support, encouragement or consideration from any source whatever. These Congresses have now passed through eight meetings and the institution as a whole is nineteen years or more old; it is not unreasonable to expect that a definite proper policy as to scope, commensurate with the income of and the actual interest in the institution, should be adopted. In this connection it may be useful to refer to a paper by Walter F. Reid (*Journal of the Society of Chemical Industry*, 1907, 75) in which he gives the total cost of some of these Congresses and their memberships.

	Total outgo, expressed in membership fees	Income, expressed in membership fees	Deficit, expressed in membership fees
1897 Vienna.....	782	....	....
1900 Paris.....	1,200	1,751	551(*a*)
1903 Berlin.....	15,000	2,533	12,467
1906 Rome.....	8,160	2,375	5,785

(*a*) Surplus.

(It might be helpful in framing a definite policy to ascertain just what things caused the surplus of 1900 to be thereafter turned into a deficit in each case greater than the total outgo of all preceding Congresses.)

Corresponding figures for the Seventh or London Congress of 1909 are not available. In the Eighth Congress the expenses for printing the proceedings were 7,000 membership fees, for propaganda 2,000, for stenographic and recording service 500, for registration, daily journals and delivery of original communications 300 or a total of 9,800 membership fees, leaving for really legitimate strict Congress-business expenses a deficit of 5,628 membership fees. It is this sort of a deficit that calls for immediate, serious scrutiny and attention; unfortunately the Berlin and Rome figures are not capable of being distributed over strict Congress-business expenses on the one hand and expenses which are chargeable to hospitality, entertainment and the like, on the other; these latter are certainly not matters of concern to the Congress but the former most certainly are and they are the only ones here considered or in any way referred to.

The only remedy seems to lie in following the old adage: "Cut your coat according to your cloth" and that is precisely what the above proposed plan is peculiarly adapted for. The amount of preparatory work would be greatly diminished and also the pre minary printing; the amount of printing needed throughout would be greatly reduced. Based upon the experience of the Eighth Congress, it is safe to say that all the needful preliminary work could be done for 600 membership fees; that all the printing necessary, all expense of reprints and placing of the volumes with the transportation lines and all strictly Congress work during the meeting could be done for 2,500 additional membership fees, *i. e.*, the entire expense of the Congress would be covered by 3,100 membership fees. Had the American Committee been at liberty to adopt some such plan as that proposed, it is safe to say that there would have been a fund left of substantially 1,000 membership fees. What effective and useful work that sum of money would have enabled the Eighth Congress to initiate for the Ninth! What an entirely different economic position these Congresses would then occupy! Instead of being dependent they would be independent; instead of justifying their existence by glittering generalities and of obtaining support and financial aid by appeals to national or civic pride they would be their own justification; the honor of being hosts to them would be spiritedly sought after; places on the Committees or Commissions to report to subsequent Congresses would be regarded as prizes to be struggled for and coveted and not, as now, accepted in a spirit of sacrifice for the *good of the cause!* The fact that all the organizations and individual chemists in the different countries would have a part in shaping and directing the program would greatly stimulate interest in the Congress by making each feel a direct interest. No doubt the Committee in charge would not be put in the embarrassing position of having 70 per cent. of its invitations to such organizations ignored and only 25 per cent. accepted; it would be spared the immense labor of sending out over 17,000 letters soliciting contributions of papers to the Congress; it would be relieved of writing up its aims and hopes from over 40 view-points and sending them out to the extent of over 300,000 pieces; it would be relieved of sending out over 120,000 application blanks for membership and receiving only one-thirtieth of them back as effective applications.

It is difficult to conceive that, among the thousands upon thousands of chemists the world over, 4,000 of them would not discern it to their advantage to subscribe the same membership fee, as heretofore, to support an enterprise of the kind outlined; the volume or volumes containing the work of the Congress would be absolutely unique in chemical literature and would perform a function not now performed, which cannot be otherwise than of use to all chemists. In so subscribing they would make it self-supporting and therefore self-perpetuating; its influence would be capable of continuous and powerful exercise and could be made to develop continuously and steadily along preconceived and clearly marked lines being controlled by a continuous and continuing policy, itself capable of adaptation to changing conditions, none of which properties are possessed by the Congresses of the past.

In view of the fact that 11% of all the papers printed were not read at the Eighth Congress even by title and that on the average only 44.5% of those in attendance at the Congress were present at the reading of those papers, that the discussion of only 22% of all the papers presented was finally accepted for printing, it must be apparent that the reading and discussing of papers form a very small part of the justification of these Congresses, nor can any great proportion of this justification be credited to the reports and resolutions, for less than 22% of the time actually spent in meetings could have been used at the Eighth Congress for the discussion of such resolutions and reports, and only 36% of all resolutions discussed in Sections ever reached the Congress itself.

At the Seventh Congress only 11% of all papers read were discussed and only 30% of the members in attendance at the Congress were present at the reading of papers.

Clearly, there must be some other justification in the minds of

those attending these Congresses than the reading of papers and discussing of reports. What can it be? The Eighth Congress planned to have a "Special Interests Registration Bureau" where attending members who desired to exchange information on special subjects could register and be placed in communication with others interested in the same specialties. Before carrying out this plan the prospective members were asked their opinion on this point at p. 12 of the Preliminary Announcement of March 6, 1911, of which 65,000 copies in 5 different languages were distributed throughout the world. They were asked to say if they cared for that kind of a bureau and if so what divisions and subdivisions they desired. The result was exactly three responses—one was indifferent and the other two thought it might be a good thing. It would seem, therefore, that personal interchange of opinion and information arranged and provided for in orderly, thorough and systematic fashion is likewise no justification for these Congresses in the minds of those attending.

Again less than 10% of the attendance participated in the excursions and factory visits, so these cannot form any great part of the attraction of these Congresses.

The Eighth Congress has therefore definitely determined that papers, reports, personal interchange of information, excursions and factory inspection are not singly nor collectively any very great determining factors in inducing attendance upon these Congresses, and the puzzle of just what does constitute this real attraction and justification to those in attendance is passed on to the Ninth Congress in the hope that a correct and positive answer to it will then be obtained.

In the event that it should be considered unwise or not feasible to alter the policy governing the Congresses, experience with the Eighth Congress has shown that certain requirements and obligations, compliance with which has heretofore been left optional with members, should be made obligatory and mandatory.

1. Members should be *obliged* to have their names and complete post-office addresses typewritten or legibly printed by hand in their applications; writing should be absolutely barred for this purpose; to decipher the slovenly and indistinctly written applications for the Eighth Congress took four peoples' time for six weeks and cost 1.5 per cent. of the total income of the Congress from membership fees. It is simply ridiculous and absurd to place such a burden so willfully and recklessly upon a working staff and a Congress Committee both of which have other and more important things to occupy their attention. All applications not so made out should be rejected until so simple a requirement is complied with. Each member's own application should be so clear that it should be used for printer's copy direct.

2. Author's manuscript *must* be typewritten and in duplicate, both as to full paper and abstract, and all figures, drawings or illustrations must also be in duplicate. Handwritten manuscript is not only archaic but most difficult for a printer to follow. All manuscripts should be provided with a protective front and a back cover and firmly fastened together, with each page numbered; the places that separate drawings are to occupy should be clearly indicated; each drawing should be clearly numbered and the same number should appear at the space in the manuscript where the drawing belongs. Original and duplicate should not be sent in the same, but in separate packages; they should not be rolled or folded but should be sent flat; the printer can then set from the original and proof-read against the duplicate; in this way danger of delay, owing to loss or defacement of copy, can be avoided. Each document should bear in typewriting on the outside—the author's name, the full address to which reprints are to be sent, the number of reprints wanted, the number of the author's or authors' Treasurer's receipt for membership in the Congress, the number of pages in the document, the number of separate drawings, if any. All drawings should be in india ink on white paper or possibly tracings on tracing cloth; blue-prints should not be sent for there is no generally adaptable method for reproducing them. The redrawing of blue-prints and slovenly or improperly made drawings cost the Eighth Congress 1.5 per cent. of its income from membership fees. Further, the author's front cover page should indicate all the different ways the title of the paper is to be indexed and cross-indexed in the final index to the Report; if the author needs facilities for an exhibition lantern and what size slides he proposes to use; if he wants experimental facilities and what; if he needs space for exhibits and how much.

It is discreditable that so many authors, as actually did in the Eighth Congress, should send manuscript slovenly written, not paged nor fastened together, without the author's post-office address, the number of reprints wanted, the number of their figures and without showing where the figures belonged in the text and with blue-prints or slovenly made drawings; that there were no more mix-ups with the manuscript is certainly no fault of such authors. This indifference of authors to their own papers caused an immense amount of extra work for the working staff and for the Committee on Papers and Publications and gave rise to a very large correspondence not only unpleasant but wholly avoidable by just a little forethought on the part of the authors, the necessity for which had repeatedly been brought to their attention. The Congress should have power and it should be thoroughly understood that the power would be rigorously exercised, to return and reject any such manuscript peremptorily. That seems to be the only way to make such useless work impossible.

3. No paper should be considered unless its author or authors be members of the Congress at the time the paper is received; all papers should be short and concise; otherwise the abstract only should be printed. It is doubtful if papers can be properly handled and printed, reprints provided and the volumes packed and addressed ready for forwarding for less than one membership fee for each printed page of text or illustration; if the expenses incident to propaganda and solicitation of papers are added, the cost per printed page of text or illustration will be very close to 1.5 membership fees or 0.67 page per member. At the Eighth Congress there were 4,163 members whose fees would have provided for the printing of a total of 2,776 pages, whereas the first twenty-four volumes alone contained 5,143 pages of printed matter; this amounts to 1.3 pages per member or twice each member's allotment; the total publication dealing with papers contains 6,500 pages or 1.5 pages per member or $2^1/_4$ times each member's allotment. There were 789 papers or an average of 8.2 pages per paper, *i. e.*, each paper used up 12.3 membership fees or 11.3 in addition to its own; the number of papers was 18.8 per cent. of the membership. It is clear that if the individual papers of the future are to be of the same average length, that for each paper submitted there will have to be no less than thirteen members of the Congress if these Congresses are to pay their own legitimate expenses. Maintaining the present membership fee, there are only two ways of avoiding this situation: increase the membership or cut down the number and length of papers. Judging from the experience of the Eighth Congress the latter is the only way feasible; 4,000 or thereabouts seems to be the limit of membership.

4. If the papers are to be printed in advance, then ninety days before the Congress meets, no further papers should be received and no further memberships nor delegateships accepted. At the Eighth Congress 560 papers were received in time to be printed before the Congress met; 229 papers were received too late for such publication; there is no reason why all but a very, very few of these papers could not have been presented at the time the other 560 were; this procrastination of authors has caused a delay and an injustice to members and authors who

have complied with the reasonable requirements entirely out of all proportion to any gain therefrom; memberships and delegateships can be determined upon and concluded ninety days before the meeting as well as at any other later time; last-minute members are rarely desirable or welcome.

With this thoroughly understood by all, the Committee in charge could have all the papers printed in advance and ready for distribution to those members in attendance; directly after the close of the Congress the volumes then ready could be forwarded to those members not in attendance; the membership list and organization list would be complete and printed and made a part of those volumes; the program could be made up and printed and mailed to every member sixty days before the Congress meets and every author would know just when and where his paper would be up for discussion. The indexing work could be brought up to date and would merely need to have added to it the matter contained in the supplemental volumes, *i. e.*, discussions, general lectures, joint-sessional addresses, minutes of the last General Meeting, errata sheet and index.

Without some such definite agreement and understanding the Committee in charge will have its time and energies absorbed in attending to the late-comers and be practically forced to neglect or slight the work necessary to prepare properly for those who have been prompt: manifestly, an improper and wholly unjustifiable state of affairs, which most properly will cause dissatisfaction; but that dissatisfaction ought not be levelled at the Committee; the procrastinating and late-coming authors and members are the ones open to censure. The only means of avoiding such dissatisfaction is to have an understanding and to stick to it. A gathering of this kind cannot be successful through the work of the Committee in charge alone; members *must* coöperate in the manner laid down by that Committee and not try to run the meeting each in his own way as so many evidently attempt to do. Members owe a duty to the Committee quite as much as the Committee owes a duty to the members, and unless members coöperate with the Committee in the way the Committee suggests, confusion and dissatisfaction must surely result and the cause thereof lies with the members themselves. It is, of course, unpleasant to write this paragraph but events at the Eighth Congress fully warrant and justify these remarks, and they are here made in the hope that the unnecessary labors which the Committee in charge of the Ninth Congress may have to perform will thereby be much less than they otherwise would be.

5. The work performed by the Committee on Papers and Publications fully justifies the introduction of this check into the organization of these Congresses and should be retained. While it is true that the final selections of that Committee are not satisfactory to all nor thoroughly consistent, yet it is equally true that this Committee prevented much discreditable matter from being printed and given the prestige of the Congress. Papers of the rankest kind of advertising nature, of the most sophomoric and puerile character, of the most verbose quality and without point or conclusion were offered to and rejected by this Committee; elaborate papers on topics of limited or no interest to chemists, papers made up largely of matters as old as chemistry itself, without any modern application were among those rejected. Direct fraud, evasion and deception were attempted by a number of would-be contributors from whom such acts would not and could not be expected or believed, but were detected and frustrated; papers published early in 1912 were offered in June, 1912, in the hope that the Committee would not know of such publication; one author had persuaded a Sectional Committee to pass a ninety-three page printed book for publication, and this author had taken extreme care to obliterate from his submitted copies every trace that would throw any light on its origin or date of publication; he overlooked, on one copy, to erase or deface the imprint of the printing house and through this the Committee located that publication and found it had been on the book-markets since 1909 and that its preface was written in October, 1908! These are merely a few of the discreditable acts on the part of would-be authors that this Committee blocked and frustrated. There were many other cases where misrepresentation, falsehood, subterfuge and equivocation were resorted to in unsuccessful attempts to have papers accepted. The conclusion seems irresistible that most of these would-be authors regarded the publications of this Congress as a convenient dumping-ground for papers not elsewhere desired—a thing which the Eighth Congress in no uncertain terms had declared that it was not. Of course, this Committee was powerless to prevent certain instances of bad faith on the part of authors of accepted manuscripts who published such accepted matter in journals in other countries between the date of acceptance and the meeting date of the Congress; there is probably no way of adequately reaching such persons. These are harsh statements, but they are true and no doubt will cause as much astonishment to others as the actual facts did to the Committee.

The constructive work of the Committee has not been as uniform nor as satisfactory as that Committee itself wished, but this is because no precedent was at hand except the vague one deducible from the nature of the papers printed by preceding Congresses and because wherever there was a doubt that doubt was resolved in favor of the author simply because of this vagueness; of course, the few guideposts to be found in the rules on papers and publications adopted by the Executive Committee of the Eighth Congress were followed but they did not cover very many cases. The problem of determining what papers should be received is a difficult one and a solution for it will probably not be readily found. Personally, I incline to the belief that no paper which does not show within itself, expressly stated, an application of its subject-matter to a going industrial operation should be accepted and then only if the subject-matter be new since the last Congress and not previously read or published elsewhere; this is offered as a first approximation toward a solution and not for any other purpose.

It must be clear, however, that a Committee which has to determine the nature of the material for which so heavy an expenditure as $1^1/_2$ membership fee per printed page is to be made, performs a necessary and useful function and is a Committee which should have for its guidance as distinct and definite a ruling as possible and power to follow that ruling regardless of consequences.

6. The difficulty with discussions is to get a printable record; many participants get up without knowing just what they want to say and make remarks which are too ephemeral for publication; others have something of more or less importance to say which may or may not be worth while perpetuating; to sift out the permanent from the ephemeral is the task of the Sectional Secretaries, and this cannot be done in the meeting itself; the safest way is to obtain from each participant a statement of his remarks and *then* the participant must be asked for help in editing or cutting down the reports. Now the hard, cold fact is that the average participant does not like to be called upon in that way; he is at the Congress for a holiday, with labor as a distinct side-issue; he wants to meet his old friends, to make new ones and to see the sights; he is altogether too busy enjoying himself to bother with writing out or checking up the transcript of his remarks. The Secretary may enclose the most pleading note with that transcript or may make the most diresome and awful threats, but your average participant serenely pursues his way and leaves the Secretary to his troubles gladly making him a present of his own into the bargain while all the Secretary can do is grin and bear it and be blamed and criticized afterwards because such participant's remarks did not appear.

The only remedy seems to be to give the average participant a little breathing time by having sectional meetings on alternate days, say Monday, Wednesday and Friday from 10 to 12 and 1 to 3; this would give each section six meeting periods of two hours each or a total of twelve hours. With the papers printed in advance and the program made up and circulated sixty days in advance of the Congress the reading of papers would be reduced to a mere formality; the discussion thereof would be the only real business left for a sectional meeting. Aside from a few comprehensive but concise introductory remarks in which the debatable points or some of them are brought prominently forward, the author, if present, should not say anything before the discussion opens; at the close of the discussion the author should be given opportunity to answer criticisms or supply additional information. At the Eighth Congress about one-half of the papers presented were discussed; the average time used in reading a paper was eight and one-half minutes, in discussion the average time was twelve minutes per paper discussed. With the above mode of reading the papers, the average time for reading need not exceed two or three minutes; in eighteen minutes two papers could on the average be presented and discussed, that is, on the average one paper every nine or ten minutes or seventy-two in all to the section. Tuesday, Thursday and Saturday morning could be available to the "average participant" in editing the transcript of his remarks or in writing them out; he would be deprived of his stock excuse that he has to rush off to another sectional meeting and must not neglect his other duties to the Congress for such clerical work as he can do later on just as well. At the Eighth Congress the participants did not get their edited remarks to the Sectional Secretaries until weeks and weeks after the Congress adjourned, and it was almost six months before the last Sectional Secretary's report came into the Secretary's hands—all because of the shilly-shallying of the participants in discussion. Perhaps, with some such arrangement of the meetings the average participant could be pinned down to his work, so that the Sectional Secretaries could all have their work cleaned up within a week after the close of the Congress for it must not be forgotten that each Sectional Secretary like Mr. Average Participant wants to meet his old friends, make new ones and see the sights; also that the former is quite as much entitled to that relaxation as is the latter.

There should be a distinct and separate authoritative provision, widely published, that all participants in discussion who do not have the corrected report of their statements in the hands of the Sectional Secretaries within three working days after the close of the Congress shall forfeit all right to have their remarks printed and that this provision is to be rigorously enforced. In the last analysis, this is the only way in which such procrastinators can be reached. As things are now, the Secretary is criticized because he goes to press too soon by those lazy and indifferent persons and criticized for going to press too slowly by those who are not in attendance—an intolerable and wholly unnecessary condition.

7. Registration by sections should be abolished. At the Eighth Congress only 228 out of 1,883 or only 12 per cent. registered by sections, and at no sectional meeting was a list of registrants for that section called for, nor was there any occasion for its use.

8. At the Eighth Congress a suggestion, made to it in the very best of good faith and which on its face gave every promise of the very best results, was followed; namely, having existing foreign societies attend to all matters of the Congress within their own territory. This has not worked out as satisfactorily as the previous method of appointing one man or one society in each country as the organizer. "What is everybody's business is nobody's business" applies to this mode of procedure quite as well as to activities in other walks of life. Concentrate the responsibility upon one man, or one organization in each country and the results will be far more satisfactory. In order to prove this for the Eighth Congress it is only necessary to compare the elaborate organizations in some countries and the results of their work in membership, papers or attendance with the less elaborate organizations and their results in other countries and to see that, in efficiency, the latter far outstripped the former; in promptness and despatch the latter also were far superior to the former.

9. Chemists should appreciate, no matter on what plan or lines future Congresses may be conducted, that attendance upon these meetings is not necessary in order that they themselves may get the worth of the membership fee. The Proceedings of these Congresses go forward automatically, when ready, to each subscribing member and these are worth to each member at least as much as, if not many times more than his fee. Chemists at large should appreciate that they are each contributing to these gatherings by subscribing, even if they do not attend and that memberships beyond those in attendance are needed and *absolutely necessary* to make these Congresses a success and independent of bounty or charity; in fact, such subscriptions are just as essential to the success of these meetings as is attendance or contribution to the papers or the discussions. But, in order that these non-attending members be not unjustly treated it is necessary that *all* members and *each* member *without exception* do his share promptly and in conformity with the suggestions of those in charge; otherwise, as in the Eighth Congress, the forwarding of the Proceedings to such non-attending members will be delayed to such an extent as to be virtually, though not intentionally, a matter of injustice to non-attending members who are each entitled to just as much consideration and courtesy as is any attending member. In the Eighth Congress, the Committee in charge was utterly powerless to prevent such a condition. If work is withheld from it, no Committee can proceed. It should be remembered that such a Committee is practically delivered into the hands of its lazy membership and such a Committee can move no faster, as a whole, than the slowest moving member of the Congress; each member must do the whole of his share and do it promptly. Had this been done by all members of the Eighth Congress there would have been literally hundreds of membership fees otherwise disbursed, now made available for constructive work for the Ninth Congress and the publications of the Congress would have been completed four months sooner than they actually were; the number of such lax members reaches an astounding and almost unbelievable total.

This brings me now to my final observation which is valid, however future Congresses may be operated and that is that a very large proportion of the attending members fail to realize that all the officers and committee members of a Congress are also members of that Congress; that the services of these officers and committee members are given gratuitously to the cause and that the relationship is not that of master and servant or employer and employee, nor of guest and host, but of colleague and colleague earnestly striving, each in his own field of responsibility, to make a success of a largely altruistic undertaking; that the very least the member can do is to ascertain the plan laid out and follow and live up to that with as little interference with the working of the organization provided as is possible. Further, the organization of such Congresses is temporary, its work is over in a few days and the greatest load is thrown upon it at the very outset; the staff is temporary, is not and cannot be familiar with all the routine (for the simple fact that no man can guess or state what that routine is surely to be), or know all the members and all the minute details of the business as can the organization of a bank, a railroad or similar large institution dealing year in and year out with a large number of the same details at once. If only a little forethought and the spirit of coöperation be exercised, matters must and will straighten

themselves out along the lines mapped out by those in charge. It is very easy to find fault and to criticize, but it is impossible to operate 2,000 or more different plans for accomplishing substantially the same thing at one and the same time, even though all these 2,000 or more plans singly, be equally good and as good as the plan decided upon.

All of the foregoing has been written in the hope and expectation that it will be of service; therefore no personal matters and no personal feeling, of both of which there is fortunately none on my part, appear therein. Some parts may be, and no doubt are, self-evident and obvious but it is the overlooking of the self-evident and the obvious that is the most common trait of men, inclusive, of course, of chemists; it is the self-evident and the obvious that so many fail to realize and consider; this oversight causes more delay and more disappointment than any other one thing and is the justification for bringing up here these self-evident and obvious facts.

The Ninth Congress received but little discretionary power at the last General Meeting of the Eighth Congress; all it has power to do is to determine the number and scope of sections and to alter their identification-numbers. In all other respects precedent would require it to proceed along lines similar to those followed by the preceding Congresses. Any change in policy must first be sanctioned by the International Commission of Congresses of Applied Chemistry whose President is Professor Paul I. Walden, President of the Ninth Congress. This Commission is not expected to take the initiative in any change of policy; the desire, if any, for such a change must come from the members of these Congresses themselves. The members of the Eighth Congress owe it to the members of the Ninth Congress to acquaint the International Commission of Congresses of Applied Chemistry with any desires they may have in respect to such changes.

90 William Street, New York

THE PRODUCTION OF SYNTHETIC AMMONIA[1]

By F. Haber and R. Le Rossignol

Ammonia in the form of commercial 25 per cent. ammonium sulfate possesses a value of 89 Pf. per kilo (9.5 cents per lb.), while the nitrogen and hydrogen of which it is composed may be valued at $2^1/_2$ Pf. and $17^1/_2$ Pf., respectively (2.14 cents for the nitrogen and hydrogen in 1 lb. of ammonia). The nitrogen is preferably obtained from air by liquefaction, as the resulting gas is relatively free from argon. It may also be made by the alternate action of air and producer gas on heated copper. Large quantities of nitrogen are generated as a by-product in the manufacture of formic acid from producer gas and caustic soda. The hydrogen may be obtained by the decomposition of distillation gases, by the alternate action of steam and reducing gases on iron, by the electrolysis of water, or by the action of water-gas on calcium hydroxide (*Ber.*, [1] 13, 719 (1880)). It is produced in large quantities also in the electrolytic manufacture of alkali and in the manufacture of oxalates from formates. The synthesis of ammonia from nitrogen and hydrogen depends upon the equilibrium represented by the expression:

$$K = \frac{p_{NH_3}}{p_{N_2}^{\frac{1}{2}} \cdot p_{H_2}^{\frac{3}{2}}}$$

where p represents the partial pressures of the respective gases and K is the reaction constant. Haber and van Oordt (*J. Soc. Chem. Ind.*, 1905, 131, 545) have studied this reaction, and found that at incipient red heat, equilibrium is attained when only traces of ammonia have been formed, and even under increased pressure, which favors the formation of ammonia, the conditions are very unfavorable at incipient red heat or higher temperatures. The yield of ammonia corresponding to the condition of equilibrium increases as the temperature falls but the reaction velocity also falls off very rapidly. Haber and van Oordt's results (*loc. cit.*) indicated that in order to be able to work at atmospheric pressure, a temperature of about 300° C. must not be exceeded, but up to the present no catalyst has been found which is active at so low a temperature. The most active catalyst found by Haber and van Oordt was manganese, but the present authors have found osmium and uranium to be much more effective. The limits of temperature for practical working are between about 500° and 700° C. Above 700° C. any advantage accruing from increased reaction velocity is more than counterbalanced by the low yield of ammonia. At a little below 500° C. it is possible, at a pressure of 125 atmospheres, to obtain 0.5 gram of ammonia per hour per cc. of space in the contact chamber, and since in high-pressure work the size of the apparatus must be limited, it is not advisable to work at a lower temperature and hence with a lower reaction velocity. The results obtained are better the higher the pressure. Curves are given showing that at (1) 100 atmospheres and (2) 200 atmospheres pressure, respectively, the theoretical yields of ammonia at different temperatures are approximately: 500° C., (1) 10.7, (2) 18.1 per cent.; 550° C., (1) 7, (2) 12.2; 600° C., (1) 4.5, (2) 8.3; 650° C., (1) 3, (2) 5.8; 700° C., (1) 2.1, (2) 4.1 per cent. It will be seen that in no case is the yield so high that it would prove remunerative to absorb the ammonia from the reaction products, and allow the residue to go to waste. A continuous process must, therefore, be

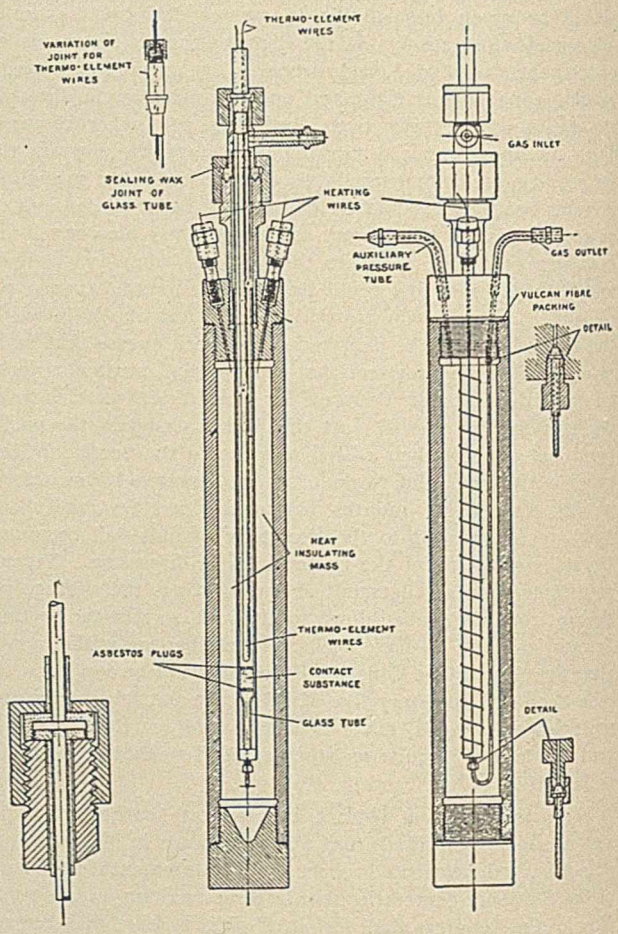


Fig. 2

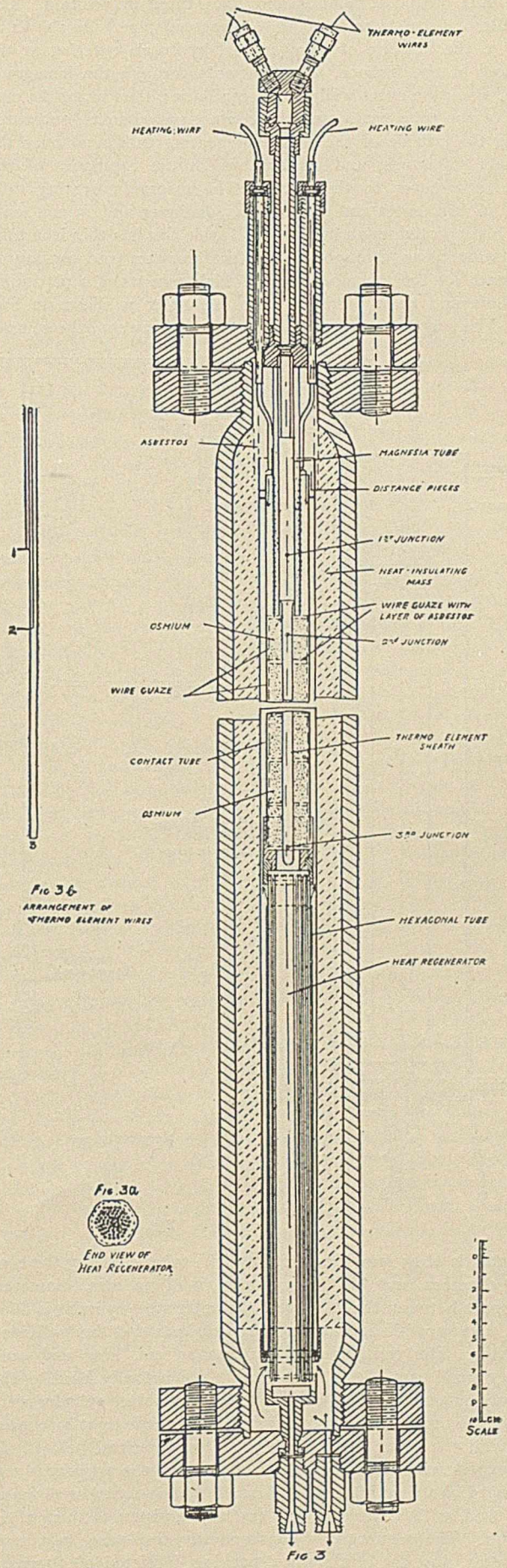
Fig. 1

[1] Abstracted by the *J. Soc. Chem. Ind.*, **32**, 134 from the *Zeitschrift für Elektrochemie*, **19**, 53 (1913).

adopted, the ammonia being removed and the residual gases, after addition of fresh nitrogen and hydrogen, being again passed over the contact substance. The simplest method of separating the ammonia is by liquefaction by cooling. Temperatures down to —75° C. may be used for this purpose, but below —75° C. there is risk of solidification of the ammonia, leading to stoppages. The regeneration of heat and cold in gas-liquefying apparatus with gases under high pressure h s proved surprisingly efficient. However low the temperature, a certain fraction of the ammonia always remains in the gaseous condition and this fraction is somewhat higher than would be expected from the vapor-pressure table.

The apparatus used for comparing the efficiency of different catalyzers consisted, in its simplest form, of a strong steel cylinder with a bore of 6 or 7 mm. diameter, opening above into a conical enlargement into which fitted the conical lower portion of a steel cover. A special joint[1] was used here, and also wherever possible in the apparatus described subsequently, the conical portion of the cover having a more acute angle (16°) than the conical portion of the bore (20°); a hollow screw-threaded plug, fitting over a flange on the cover, was screwed on to the upper portion of the cylinder, thus bringing the two conical portions into effective contact. This apparatus was heated by means of a bath of fused saltpetre. Another form of apparatus (see Fig. 1) for comparing the efficiency of catalysts consisted essentially of a thin iron tube (0.6 mm. thick, 9–13 mm. diameter) wrapped in asbestos paper and wound with nickel wire (0.4 mm. thick) so that it could be heated electrically. The contact substance was contained between asbestos plugs in an inner quartz or glass tube sealed at the upper end, in which also was a quartz capillary, enclosing a thermocouple. The lower end of the tube containing the contact substance was flush with the end of the heated iron tube, and the latter was fastened by screwing and soldering to a steel capillary through which the gases were withdrawn. The whole was enclosed in an outer strong steel cylinder as shown. In order to prevent deformation of the heated iron tube by the high pressure, an auxiliary pressure tube was provided through which the gas mixture was introduced also into the space filled with heat-insulating material (asbestos paper) surrounding the tube. All joints in this apparatus were of the type described above, except in the case of the plugs closing the outer steel cylinder, and the insulated joints for the electrical heating wires. The joints of the steel cylinder were made gas-tight by means of discs of "Vulcan fiber," and the joints for the heating wires in the manner shown in Fig. 2, the wires being soldered to a small metal plate, about 2 mm. thick, which fitted into a recess in a disc of "Vulcan fiber" and was covered by another disc of "Vulcan fiber," both discs having central openings through which the wire passed. It is stated that in this furnace, with correct adjustment of the heating coil, a field of constant maximum temperature of 4–5 cm. length can be obtained. The largest furnace, which was used in the experiments with circulation of the gas, is shown in Fig. 3. In this furnace there is a heating coil in the gas space so that the mixture of nitrogen and hydrogen is heated before it reaches the contact substance, and cools gradually in passing over the latter; the formation of ammonia thus sets in at a relatively high temperature and hence with a high velocity, and further quantities of ammonia are formed during the gradual cooling, owing to the displacement of the equilibrium in a favorable direction. This furnace is also fitted with a heat regenerator, formed by a bundle of 127 steel capillaries, wound with iron wire, and supported by two hexagonal iron plates, perforated as shown in Fig. 3a, the capillaries passing through the perforations and projecting a short distance beyond the plate, which is provided with a rim. A powdered alloy of 40 per cent. of silver and 60 of copper is melted, with exclusion of air, around

[1] D. R. G. M. 376,829 des Laboratoriums mechanikers F. Kirchenbauer.



the ends of the capillaries and fixes these in position. The bundle of tubes is enclosed in a hexagonal pipe. As shown in Fig. 3, the mixture of nitrogen and hydrogen entering at the bottom of the furnace, flows upwards through the hexagonal pipe, along the outer walls of the capillaries, then along the outside of the tube containing the contact mass, next downwards along the outside of the magnesia heating tube, and over the contact substance, and finally through the capillaries of the heat regenerator to the outlet. The magnesia heating tube is of 16 mm. outer and 9 mm. inner diameter and 15 cm. long, and is supported on an iron tube. Inside this is a thin iron tube extending down through the contact chamber and serving as a sheath for the thermo-element, which consists of a platinum-rhodium and three platinum wires arranged as shown in Fig. 3*b*. The length of the outer steel cylinder is 75 cm., the contact chamber is of 25 mm. diameter, and the tube enclosing the contact chamber and heat regenerator is of 32 mm. diameter; the space between the latter and the outer steel cylinder is filled with a stamped-in mixture of *magnesia usta* and finely-divided asbestos. The contact chamber was 25 cm. long and only a very small part of the space therein was actually filled by the contact substance. The covers of the outer steel cylinder were fastened by means of bolts, the joints being made tight by copper washers; the other joints were made as described before. In the experiments with circulation of the gas, the apparatus was arranged as shown in Fig. 4. A small double-acting steel pump was used, with a stroke of 3–6 cm. and a piston of 19 mm. diameter. The piston rods consisted of steel wire, 1–5 mm. diameter, and the stuffing-box packing consisted of alternate layers of brass and cotton impregnated with paraffin, to a total length of 1.5 cm. At 300 revolutions per minute and a pressure of 200 atmospheres, the capacity of the pump was 72 cm. per hour (at atmospheric pressure and temperature), but the actual amount of gas circulated was somewhat less, owing to the leather packing of the piston proving not quite satisfactory; the stuffing-box packing answered very well. The ammoniacal gases from the furnace pass through a copper capillary to a drier charged with soda-lime and then to a heat-exchanger (cold regenerator) consisting of three copper capillaries united at the inlet and outlet; the capillaries are wound into coils as shown, and are contained in a strong steel cylinder. From the cold regenerator, the strongly cooled gas passes into the liquefier, and the liquefied ammonia is drawn off as necessary; or by opening the outlet valve to a suitable extent, the ammonia could be blown off as a uniform current of gas. The gases leaving the liquefier, pass through the cold regenerator to the pump, and thence again to the furnace, a fresh quantity of nitrogen and hydrogen being added on the way through a valve. Through another valve samples of gas could be withdrawn. For determining the ammonia content of the gas mixture, Rayleigh's gas interferometer as supplied by Zeiss was used, the gas being examined before and after passage through sulfuric acid (compare *J. Soc. Chem. Ind.*, 1906, 802). The results of experiments with cerium and allied metals, manganese, tungsten, uranium, ruthenium, and osmium as contact substances are described.

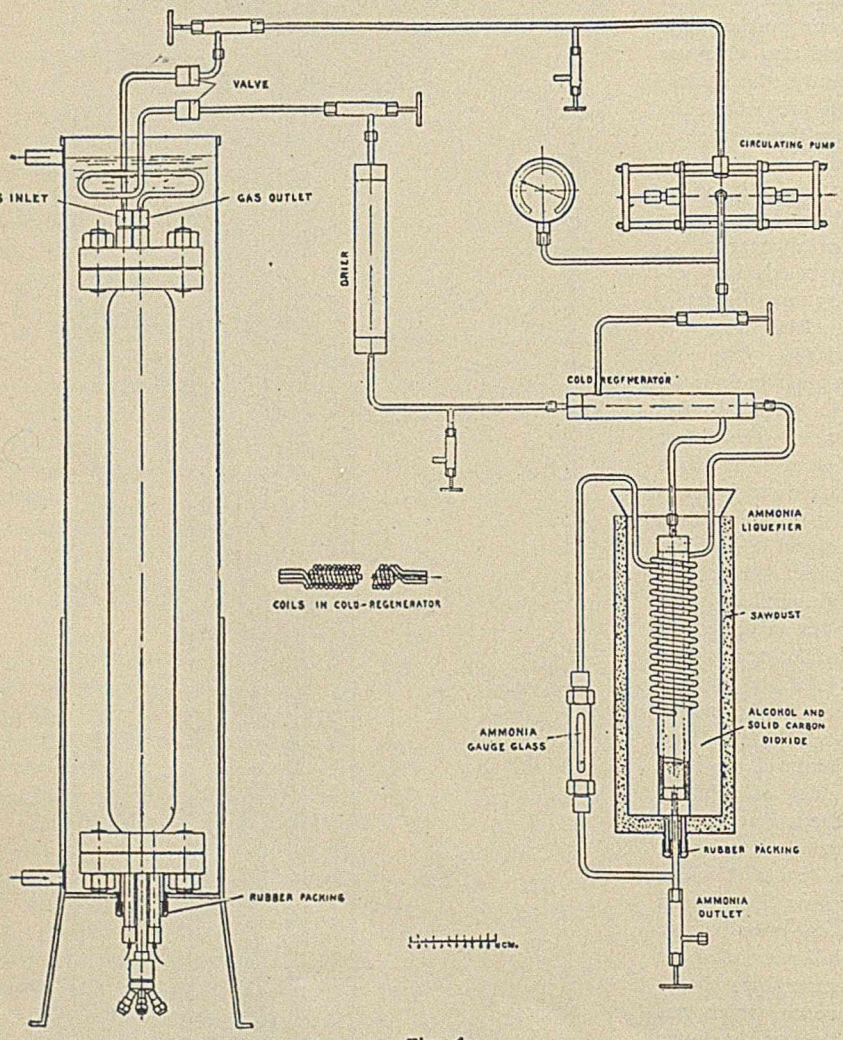


Fig. 4.

Uranium.—The commercial metal, broken up with a hammer, was used in a column of 4–5 mm. diameter and 3–3.5 cm. long. At about 600° C. vigorous formation of ammonia began. At 190 atmospheres pressure and the gas mixture flowing at a velocity of 20 liters per hour (measured at atmospheric temperature and pressure), the issuing gas contained 5.8 per cent. of ammonia constantly during $1^1/_2$ hours. With a velocity of 3 liters per hour at 580° C., the ammonia content rose to above 7 per cent. and liquefaction of ammonia occurred at the valve. At a pressure of 120 atmospheres and a velocity of 3 liters per hour at 580° C., 4.8 per cent. of ammonia was obtained, falling to 3.5 per cent. on increasing the velocity to 20 liters per hour. With a velocity of 3 liters per hour at 550° C. (120 atm.), 5.6 per cent. of ammonia was obtained, rising to 5.85 per cent. on reducing the velocity to 2 liters per hour. Good results were also obtained with uranium prepared by Moissan's method. An experiment extending over a long time with the commercial metal gave the results shown in the following table (p. 331).

The values marked with an asterisk were obtained not by determining the ammonia content at the given time with the interferometer, but by passing the gas during the interval between one test and the next through acid; they represent the yields corresponding to the mean of the values given in the same and in the succeeding horizontal series for the experimental conditions. It is important to heat up quite gradually in order to maintain the contact substance in an active condition. Experiments with uranium (pieces of the size of a pin-head) were also made in the simple steel cylinder, at a pressure of 125 kilos per sq. cm. With a velocity of about 32 liters per hour at 570° C., 5.65 per cent. of ammonia was obtained; 6.54 per

Time Minutes	Temperature ° C.	Pressure Kilos. per sq. cm.	Velocity Liters per hour	Ammonia Per cent.
0	590	121	11	2.8
70	592	121	11	3.05
133	589	119.5	11	3.1
180	591	118	11	3.13
240	591	117	11	3.15
300	591	115.5	11	3.09
360	590	114	11	3.05
420	591	112.7	11	3.02
460	591	112	11	3.02
515	590	166	20	*2.85
613	594	157	20	*2.70
714	590	145	20	*2.50
823	(quite low at times)	131	20	*1.97
1093	ca. 590	118–115	20	*2.00
1118	590	156	20	2.68
1323	593	166	20	2.74
1333	590	165	10	3.60
1340	610	165	10	3.80
1348	609	164	20	3.00
1358	630	163	20	3.15
1365	608	162	5	5.05
1398	609	160	3	5.50
1428	590	158	3	5.50

cent. at 505° C., and 25.7 liters per hour; 9.1 per cent. at 496° C., and 9.5 liters per hour; and 11–11.9 per cent. at 503°–493° C., and 2 liters per hour.

Osmium.—Finely divided osmium proved a very effective catalyst. (In both the iron and chromium groups of metals, the metals of highest atomic weight possessed catalytic properties far superior to that of the metals of lower atomic weight.) In a series of experiments with a layer of osmium 14 mm. long and 4.5 mm. diameter, 4.75 per cent. of ammonia was obtained at 592° C. and 156 atmospheres pressure and a velocity of 20 liters per hour. At the same pressure, 5.91 per cent. was obtained at 572° C., and 10 liters per hour; 7.9 per cent. at 174 atmospheres, 550° C., and 1.5 liters per hour; and over 9 per cent. at 521° C., 174 atmospheres, and 1.5 liters per hour. In a prolonged experiment with a small quantity of osmium (a layer 4 mm. diam. and 1 cm. long), 34 grams of ammonia were obtained, and the contact substance was more active after 58 hours than after 3 hours. An experiment with gas circulation was carried out with osmium as catalyst. After pumping out air from the apparatus (see Fig. 4) the mixture of nitrogen and hydrogen was introduced up to a pressure of 185 atmospheres, and the circulating pump set working. A current was now sent through the heating wire (17–18 amperes at 56 volts). The temperatures registered by the three junctions of the thermo-element varied from 580°–635° C. at that next to the heat regenerator, from 750°–825° C. at the intermediate junction and from 880°–1000° C. at the first junction. At first the liquefier was not cooled, and the ammonia content of the gas-mixture rose to 5.4 per cent. The liquefier was then cooled with a mixture of alcohol and solid carbon dioxide and kept at —25° to —39° C., mostly —30° C. The experiment was continued for 4 hours, the pressure ranging from 193 to 163 atmospheres. The ammonia content of the gas varied from 2.4 to 3.1 per cent., mostly 2.6–2.9 per cent. (Higher figures would have been obtained with a larger quantity of contact substance.) About 500 cc. of liquefied ammonia (corresponding to 336 grams or 475 liters of gas at ordinary temperature and pressure) were obtained in the 4 hours. The heat- and cold-regenerators acted very efficiently. The copper capillary through which the gases left the furnace could be held comfortably in the hand quite close to the furnace. The cold regenerator had a thick coating of ice on the side where the gases entered from the liquefier, while on the other side it was at the ordinary temperature. At the end of the experiment the residual gas mixture contained 30 per cent. of nitrogen plus argon.

CHEAPER ALUMINA AND ALUMINUM FROM MINERAL SILICATES[1]

By Alfred H. Cowles

If two briquettes be formed, *one* of kaolin (or clay) containing 19 parts of anhydrous pure clay and twenty-three and four tenths parts of salt; the *other* of 15 parts of charcoal to 100 parts of the first mixture; and these two briquettes, alike in size and form, be kept heated with water vapor in an oxidizing atmosphere at a temperature equal to or just above the vaporizing temperature of salt, it will be found that each briquette begins to be converted over its surface into a sodic-silico-aluminate which would have the formula of $(Na_2O)_2.(SiO_2)_2.Al_2O_3$ if the clay and salt were pure. The vibrating gaseous molecules of watery vapor and oxygen bring about this change over all exposed surfaces of the two briquettes. However, this difference in conversion will be found: *the briquette containing no charcoal will require nine times as long for the conversion to penetrate to its center between walls, as the briquette containing charcoal.*

This result is not self-apparent, as the reaction requires an oxidizing atmosphere to oxidize the sodium produced before it volatilizes out of the briquette. That this result would occur was based upon hypothetical reasoning on my part, and its demonstration as a truth was brought about by experiments performed in the laboratory of The Electric Smelting & Aluminum Company at Lockport, New York. By coincidence, Dr. Adolf Kayser was my assistant in performing the experiments. Dr. Kayser in the early '90's attempted to develop this reaction commercially, using briquettes containing no charcoal in large kilns of a down-draft nature. The work had proved a failure as had also the work of William Gossage in 1862, and that of Gruneberg and Vorster about 1874–76. During these experiments, Dr. Kayser was of the opinion that they could not succeed because, as he thought they would produce reducing conditions. The experiments, however, proved that the conversion penetrated the briquettes nine times faster when charcoal was in them, than was the case when the briquettes contained no charcoal. This result taught that it was necessary to expose large surfaces or porous masses to bring about a rapid conversion. This could be effected in more than one way, but the problem of cooling the gases and simplifying the elimination of dust led to the adoption of a tunnel furnace of the Grondal type, with modifications to render it suitable for the process.

The fact that the reaction could be made to proceed rapidly, opened up an entirely new vista, one of assured success to the process. It meant that the volume of fuel gases and nitrogen necessary to pass through the charged furnace would become so far reduced as to permit of efficient commercial condensation of the hydrochloric acid evolved. It meant the employment of cheap fuel, sawdust, or charcoal made from sawdust with possible by-products from the same, and far greater capacity of output with the same cost of plant. It meant that the process could be applied to other aluminous materials such as potash feldspar mixed with chloride of potash or salt, securing the chemically equivalent reaction.

Dr. Kayser had evolved a method of opening or rendering leachable alkali-silico-aluminate containing sufficient alkali, which means nearly one molecule more than that which occurs in feldspar. This he did by heating to a sintering temperature two molecular weights of lime to each molecular weight of silica in said compound, after which the product may be leached and the alkali aluminate very effectively removed. Our company acquired this patent from him.

The process is easily understood. In the manner in which we are attempting to perfect it at Sewaren, New Jersey, in our present work, briquettes similar to those in Fig. I are formed

[1] Paper presented at the Joint Meeting of the American Electrochemical Society, American Chemical Society and Society of Chemical Industry, Chemists' Club, New York, February 7, 1913.

by the same clay working machinery that produces what is known as hollow-ware or conduit bricks. When the clay, salt and sawdust are mixed together and water is added, this mixture acts almost the same as clay as to plasticity in passing through the briquetting machinery, but unlike the clay brick, it does not shrink or expand from the period of its first formation, during drying and until it is formed into a finished product containing soda, silica and alumina.

These briquettes are loaded onto flat cars, the surface of each car as now used being twelve feet long and five and a half feet wide holding fifty-five bricks. These loaded cars are passed first through a drying furnace where the free water is driven off, and then by a transfer track into a vestibule connected with the long kiln or furnace. After the door of the kiln is opened and the door of the vestibule is closed, the loaded car is gradually pushed into the charging end of the furnace by a hydraulic pusher, and a train of fourteen cars in front of it is also gradually pushed forward towards the discharging end of the furnace, while a finished car load is coming out at that end.

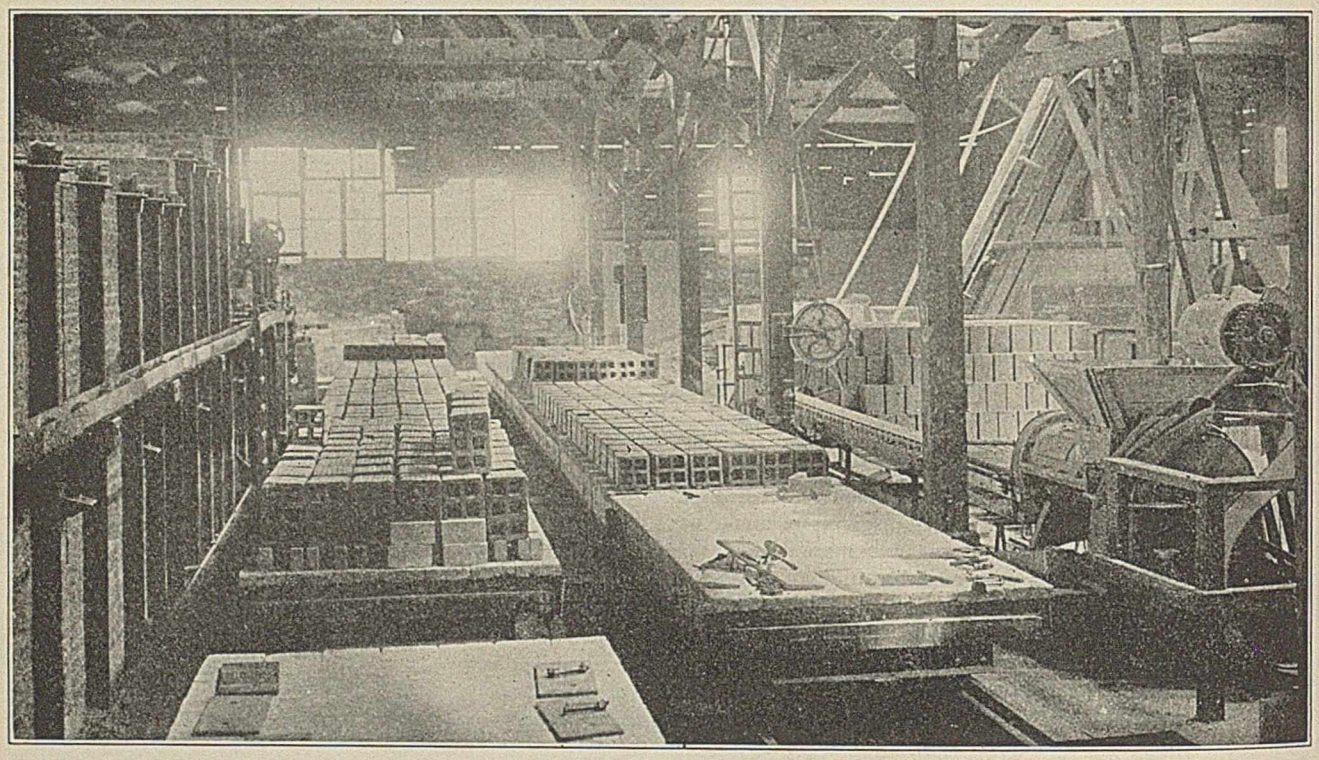


Fig. 1—Briquetting Machine and Cars with Briquettes Ready for Furnace, Sewaren, N. J., Plant

The cars are covered with fire brick, the latter of a nature to protect them from being materially attacked by salt vapors. A sand seal extends the whole length of the furnace, and pure air is blown under the train of cars in such a manner as to prevent any leakage of hydrochloric acid fumes downward. In operation, the temperatures of all exposed iron parts of the furnace are kept above the boiling point of mixed acid and water, and as is well known, at this temperature vapor of hydrochloric acid does not attack iron.

Knowing the composition of the clay and the percentage of salt in the briquettes, steam is blown into the discharge end of the furnace, and the rate by pounds weight of steam per minute is read by an indicating steam flow meter. Suppose this rate is adjusted to the rate of feed of the material on the cars to effect the decomposition of salt with the formation of say 33 per cent. hydrochloric acid in water: then there is required in the *chemical reaction only about one part of water* to 9.5 parts of water to produce the 33 per cent. acid, hence it will be noted that the quantity of steam blown in to form such acid is 9.5 times greater than that which takes place in the *reaction proper*. The amount of steam, if the heated zone in the furnace be long enough, can be so reduced as to still permit much water being admitted to the coke towers to complete the condensation of all the acid.

Above and on the end of the furnace where the cars are pushed in, is a large exhaust fan which draws all the gases from the furnace and forces them onward through a condensing system where the hydrochloric acid is condensed. The furnace is 184 feet long, or ignoring the vestibule, is 170 feet long. Beyond the zone of high temperature towards the fan, there is a distance of 79 feet where the briquette loaded cars act as scrubbers to remove any sublimed salt and other dust from the gases at the same time that the incoming briquettes are being heated by the hot gases passing over them.

In addition to this, we are constructing a dust settling and cooling chamber in the iron pipe line between the furnace and the acid condensing system.

The reaction that takes place in the furnace will vary according to the composition of the aluminous material and the amount of alkali chloride mixed therewith, but typically it may be written as follows:

$$Al_2O_3.2SiO_2 + 4NaCl + 2H_2O + yH_2O + zC + zO$$
$$= (Na_2O)_2.(SiO_2)_2.Al_2O_3 + 4HCl + yH_2O + zCO.$$

The gaseous products in the reaction are carried into the condensing system after cooling. The briquettes maintain their original form, and before the cars issue from the discharge end of the furnace they are cooled down by the inflow of the measured quantity of steam and air that is sucked in. This inflow becomes preheated by the hot bricks before it reaches the combustion zone of the furnace; the finished bricks are cool enough to be handled with gloves as they are discharged from the furnace.

In our work at Sewaren we have discovered that our acid condensing apparatus with air cooling has only about one-third the necessary capacity. It is at the present time being enlarged; also tourells submerged in water are being added into which about 18° Beaumé acid will be run against the flow

of the gases before they reach small coke towers. The escape of acid fumes has been so great that we have been able to make only four experimental runs of about thirty hours duration each with the large furnace, but the results secured have taught us the necessary changes to be made in the acid condenser system and those necessary to get a full conversion in the furnace. The whole product of the last two runs has been analyzed, but not of the first two. The furnace was operated with only one set of oil burners and a car of briquettes fed into the furnace every forty minutes.

Before starting to build our plant at Sewaren, briquettes had been made containing as high as 33 per cent. of sodic oxide and were white and free from iron.

We found by analysis that the Sewaren briquettes were but little more than half converted, hence it was rather against my desire to read this paper at this time. The briquettes still contain a large amount of salt. The furnace is provided with another set of oil burners to meet this contingency, but we have not yet used them.

After completing the enlargement of our acid condensing system we contemplate using both sets of oil burners and adding a trifle more sawdust to the bricks, in which case there is no doubt but that we shall produce the same results as in our laboratory work, with one exception. In the laboratory work, the furnace was a deep circular one filled with briquettes and an oil flame and water vapor were passed downwards through the briquettes. Under these conditions, the iron as an impurity in the clay was carried downward as chloride of iron; small portions of it passed off with the acid and other portions condensed in the bottom of the furnace and in the iron pipes through which were drawn the gaseous product from the furnace from under a grate at its bottom. As these bricks were secured white it was concluded that we would secure the same result in our large work, but this conclusion as given in a paper[1] read before the 8th International Congress of Applied Chemistry, has, by practice, proved erroneous. Since the surfaces of the cars are cooler than the briquettes, we find a portion of the iron as either ferric or magnetic oxide on the car surfaces and a part still remains on the surface of the lower portions of the briquettes: hence it does not pass out with the gases to vitiate a part of the acid. The acid produced thus far shows the presence of sulfuric acid only by the most delicate test. It is free from arsenic, and has but traces of free chlorine, iron chloride, sulfuric acid, and ammonium chloride; with our final settling dust chamber completed, it will be practically free from salt and aluminum chloride.

[1] See Vol. 25, p. 119; also *Met. Chem. Eng.*, **10**, 659.

LIME OPENING PROCESS

In order to secure from the sodic-silico-aluminate of the briquettes the sodic aluminate contained we intend to put down a kiln of the rotary type to sinter this material, which is easily friable after being mixed with lime in such proportions as to form dicalcium silicate from the silica present. This compound is insoluble, while the sodium aluminate is soluble. The material as thus treated is not fused, but sintered in such condition as to form an easily leachable mass. The reaction involved in this process is as follows:

$$(Na_2O)_2.(SiO_2)_2.Al_2O_3 + 4CaO = 2(CaO)_2.SiO_2 + (Na_2O)_2.Al_2O_3.$$

Limestone, calcite or marl in their chemical equivalents may be used instead of anhydrous lime. The product thus secured will be leached by ordinary methods.

In the foregoing reaction any amount over two molecular weights of calcium oxide to each molecular weight of silica leads to the formation of insoluble calcic aluminate, and less than two molecular weights leaves undecomposed a portion of the insoluble sodic-silico-aluminate. Under either of these contingencies, marked loss in the recovery of alumina may occur Experimentally, up to 90 per cent. of the sodic aluminate has been leached from the above clinker while there was left behind a mass of dicalcium silicate.

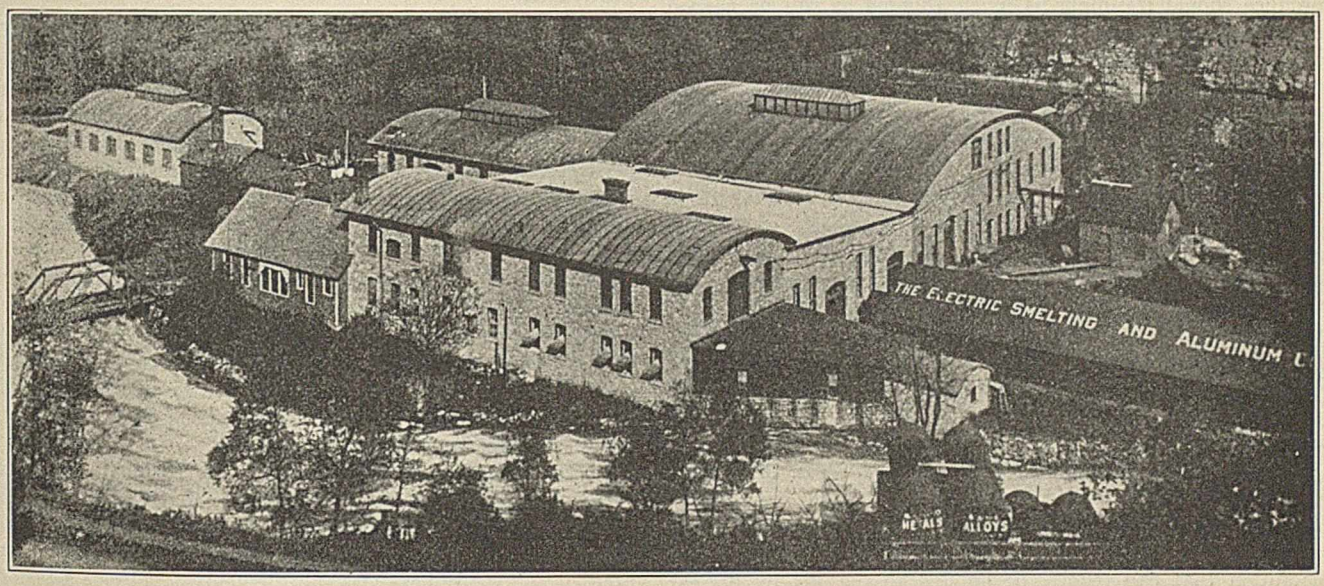


FIG. 11—LOCKPORT, N. Y., PLANT OF ELECTRIC SMELTING AND ALUMINUM COMPANY

PRODUCTION OF HYDRAULIC CEMENT

According to Newbury's formula, hydraulic cement is tricalcium silicate with up to 8 per cent. of aluminum and iron oxides in a mixed condition permissible. The above-mentioned dicalcium silicate product is hydrated. If it now be ground and then heated with one added molecular weight of lime it forms a product that is in composition the same as that of hydraulic cement, but unless the raw materials used are very pure, experience at Sewaren discloses that it will give a light gray and not a white cement as I believed when I spoke before the Eighth International Congress of Applied Chemistry. Inadvertently I also stated at that time as follows:

"When briquettes are made containing 32 per cent. of sodic oxide the output of 30 per cent. hydrochloric acid should be about one and a half tons of acid to one ton of salt and clay in the charge."

The statement should have read: "To one ton of salt in the charge, about 1.75 tons of 33 per cent. acid."

USE OF DICALCIUM SILICATE AS A MATERIAL FOR GLASS-MAKING

If, in the foregoing lime sintering process, should 15 or 20 per cent. of the alumina and alkali contents left in the insoluble dicalcium silicate after leaching, there remains a product, that is excellent for the manufacture of window and bottle glasses. Its use would call for the addition of silica and more alkali. Glass can stand a considerable percentage of ferric oxide and alumina in its manufacture.

OXALIC ACID

Excepting sawdust nearly all the ingredients needed to produce oxalic acid come from the products of this process. The carbonic oxide from the acid towers can be scrubbed to free it from the last traces of hydrochloric acid and then passed over or through sawdust mixed with a caustic alkali heated to the proper temperature producing an oxalate of the alkali, which treated with calcium chloride gives calcium oxalate and with sulfuric acid gives oxalic acid with calcium sulfate precipitated. This acid is most largely manufactured in Europe, but has a great consumption in American laundries.

USE OF HYDROCHLORIC ACID IN PRODUCING PHOSPHATE FERTILIZER

The question may well be asked what use can be made of the large amount of hydrochloric acid that this process will produce should it supply as much alumina as will be needed for the production of the world's future supply of the metal aluminum. The author has pending a number of United States and foreign patents which may place hydrochloric acid in competition with sulfuric acid as now used in the manufacture of soluble forms of calcium phosphate mixed with calcium sulfate so largely used as a fetilizer. A pure calcium phosphate rock is anhydrous tricalcium phosphate. With it, there is almost always associated considerable calcium carbonate, silica, alumina and other impurities. If this rock be ground to an even degree of fineness and then thoroughly digested with weak hydrochloric acid, the quantity of acid employed being theoretically just sufficient to dissolve all the free calcium carbonate, ferric oxide and one molecular weight of calcium oxide contained in the tricalcium phosphate, the insoluble residue that is left is dicalcium phosphate. The soluble products—calcium, magnesium and iron chlorides—will exist in solution. By filtering and passing water through this mass, the soluble chlorides formed can easily be removed, and either run into the sea or submitted to other possible uses. The insoluble dicalcium phosphate remains in a non-hygroscopic pulverulent condition that can readily be applied to the soil by present methods of distribution. This is the product that all soluble phosphates revert to when they are applied to the soil as fertilizers. It has, however, one advantage over the soluble phosphates produced with sulfuric acid; *i. e.*, it is not loaded down with a large burden of calcium sulfate upon which transportation charges must be paid. If calcium sulfate is desired for fertilizer use, it can be more cheaply produced from native gypsum rock. If the digestion is not thorough, some soluble phosphates pass into the rich solution of chlorides. These phosphates can be precipitated by the use of a proper amount of calcium carbonate.

In Idaho, Utah, Montana and Wyoming, the U. S. Geological Survey has in recent years discovered and examined the largest deposits of phosphate-bearing rock ever known to the world. In this general region, those rocks that have now been withdrawn up to September 1, 1912, covered an area of nearly two million two hundred and twelve thousand acres.

The process as thus far described ends in securing the alumina as a sodic aluminate solution which can be completely purified from silica, titanic acid and the iron oxide by a very simple means of precipitation. The final precipitation of the alumina in its purest form is brought about by Dr. K. H. Bayer's process patented about 1894. It involves a very peculiar and interesting phenomenon. When previously precipitated alumina is added to sodic aluminate solution, the precipitate begins to grow almost like a mushroom, and in a few days time nearly all of the alumina is precipitated, leaving a liquid containing nothing but caustic soda and a comparatively small amount of alumina. By an added step, which on account of patent matters I can not discuss, the last traces of alumina from the solution may be recovered, leaving a strong solution of caustic soda that needs slight concentration to prepare it for the market.

This general process opens the way to secure alumina cheaply or as a by-product in conjunction with these other products widely used in the industrial world. To produce one pound of aluminum 1.9 pounds of alumina are needed, there being no appreciable loss in the present method of manufacture of the metal. This weight of alumina costs from $6^1/_2$ to $7^1/_2$ cents according to the latest data and is the heaviest item of cost in producing the aluminum. By a general introduction of this process we may hope to see the cost of production of aluminum reduced 4 or 5 cents per pound. When it comes to a general electrification of our world's railroads and a greater use of electric transmission lines and trolley wires, we shall find aluminum a much more serious competitor of copper than at present; eventually, it will almost entirely supplant copper except in motors and generators.

POTASH ALUM FROM POTASH FELDSPAR AND ROCK PHOSPHATE

We all know the great need of potash salts as plant food and the scarcity of their supply, also the abundance of potash feldspar rocks containing great quantities of potash and the abundance of rock phosphate. Several years ago, I was experimenting with the mineral Wavellite which is a hydrated phosphate of aluminum. Knowing that calcium sulfate or burned gypsum is slightly soluble in water, and that aluminum sulfate is very soluble in water, I reasoned that if the proper proportions of aluminum phosphate and calcium sulfate were ground together and a minute amount of sulfuric acid were added to start the reaction, that the two bases should trade their acids if enough acid were present to form the insoluble calcium phosphate. On trying the experiment, I found this reaction to occur and that large amounts of aluminum sulfate could be leached away from the mixture.

Further, since dicalcium silicate is quite insoluble and calcium sulfate slightly soluble, it occurred to me that an intimate mixture of potash feldspar (*e. g.*, orthoclase, $K_2O.Al_2O_3.6SiO_2$ or other potash silicates such as leucite) with the proper proportions of rock phosphate and sulfuric acid should give a system which would result in the formation of *insoluble* dicalcium silicate and *soluble* aluminum sulfate, potassium sulfate and orthophosphoric acid (H_3PO_4). Accordingly, I ground potash feldspar with rock phosphate (enough to furnish two molecular weights of lime to each molecular weight of silica in the feldspar), heated the mixture to a sintering temperature of about 1000° C. and leached with sulfuric acid; my first experiment gave almost 100 per cent. results.

For the purpose of this lecture, I have had the work repeated. In figuring the amount of sulfuric acid to use, my assistant took a little less acid than was used in the former work, and the result of the leaching has given a product containing all the potash and all the alumina from the feldspar, but only part of the phosphoric acid. Duplicate results have been secured substituting hydrochloric acid for sulfuric acid. The liquor in these two cases will give us either potash alum or a double chloride of aluminum and potassium associated with all the phosphoric acid contained in the rock phosphate employed. By this process, the dicalcium silicate or a slightly lower form of calcium silicate would be available for use in the manufacture of either cement or glass. Feldspar and rock phosphate can be secured very free from iron. The iron, however, follows the other products into solution. From either potash alum or the

double chloride of aluminum and potassium the chances of securing pure alumina are very favorable. Upon heating to a dull red heat, aluminum sulfate breaks down into alumina with liberations of sulfuric acid (SO_3) and the potassium sulfate can then be leached away. In the case of a double chloride of aluminum and potassium at a very low heat in the presence of the vapor of water, hydrochloric acid is given off with the formation of alumina; the chloride of potassium remains and can be leached away from the alumina.

To secure pure alumina from the above solution of chloride or sulfate salts with phosphoric acid is promising. The potash alum, however, cannot be separated by crystallization. By precipitation with sodic or potassic aluminate, if too much of either of these salts be not used, pure phosphate of aluminum may be secured.

POTASH PHOSPHATE FERTILIZER

In the foregoing, I have described the sintering at a comparatively low temperature of rock phosphate and potash silicates. I wish to call attention to the fact that this mixture is in some cases of greater fertilizer value than rock phosphate when used alone: for this use sinter and treat with the proper amount of sulfuric acid or of hydrochloric acid, if hygroscopic properties are not detrimental. By this new process the silica has taken the place of much acid that would be required, and the product is not loaded down with calcium sulfate, but instead, is loaded with potassium sulfate while the lime and silica is in such form as to be sufficiently soluble to furnish silica to plant food; further, the soluble lime is in a condition to act on the potash minerals in the soil displacing potash and thus rendering more potash available for plant food.

THE SERPEK PROCESS FOR THE MANUFACTURE OF ALUMINIUM NITRIDE[1]

By J. W. Richards

The production of aluminium nitride (AlN) on a commercial scale is proposed in the process of Ottokar Serpek, followed by the decomposition of the same by water or caustic soda solution, thereby liberating the nitrogen as ammonia and producing alkaline aluminate solution from which pure alumina can be obtained. This rather daring chemical proposition has directed attention to aluminium nitride, the conditions of its formation and its properties.

The existence of aluminium nitride was suspected for some time before it was isolated. A rather indefinite number of years ago, about 1890, the writer's attention was directed by his father to the fact that when metallic aluminium in a melted condition was skimmed and the skimmings or dross laid to one side, that on sprinkling water upon them these skimmings gave off an odor of ammonia. The only explanation which came then to be figured out for this phenomenon was that the hot aluminium in the skimmings oxidized to alumina, and that the rather high temperature thus produced locally caused the aluminium also to unite with the nitrogen of the air and form the nitride. In other words, that the aluminium united first with the oxygen and immediately thereafter also with the nitrogen of the air. On sprinkling these with water, the nitride would react according to the following reaction:

$$2\,AlN + 3\,H_2O = Al_2O_3 + 2\,NH_3.$$

In the light of subsequent investigation and formation of aluminium nitride in other ways it appears that the explanation given in these early days was correct and that aluminium nitride is formed directly under such conditions from metallic aluminium and the nitrogen of the air.

Le Verrier, later, proved the presence of traces of nitrogen in commercial aluminium by dissolving it in caustic potash and passing the nitrogen evolved into Nessler solution, and obtaining from it the ammonia reaction. He thus proved the presence of nitrogen in the metal and suspected it to occur as aluminium nitride dissolved in the excess of metal. In further experiments he treated aluminium with a current of nitrogen gas until it was saturated and noted that this produced considerable diminution in the tensile strength, elastic limit and elongation of the metal.

Prof. Mallet later obtained aluminium nitride corresponding closely to the formula AlN by heating metallic aluminium in a carbon crucible to a moderate temperature for several hours in contact with dry sodium carbonate. Some alumina is formed, some sodium vaporizes and some carbon is deposited; the reaction is probably as follows:

$$3Al + Na_2CO_3 + N = AlN + Al_2O_3 + C + 2Na.$$

Aluminium nitride containing 34 per cent. nitrogen and 66 per cent. of aluminium was obtained as little, yellow crystals and amorphous drops. When calcined in the air it was found to lose nitrogen and form alumina; it is decomposed in moist air losing its transparency, becoming lighter yellow, evolving ammonia gas and finally leaving the alumina residue. Caustic potash solution and melted caustic potash both attack it actively, disengaging ammonia and forming potassium aluminate.

A later experimenter described the compound as a gray amorphous powder, but with properties otherwise similar to those described.

Coming now to the work of Serpek, we find his first proposition seemed to be the production of aluminium nitride from aluminium carbide, which is indeed a possible reaction. In his U. S. Patent, 867,615 (filed June 19, 1906, and issued October 8, 1907), he describes the passing of nitrogen or a gas containing nitrogen over aluminium carbide as an initial material heated to red heat. He describes the volume of the carbide increasing on being converted into nitride, and says that the absorption of nitrogen was increased by diluting the carbide with such materials as carbon or alumina or aluminium chloride; also that traces of hydrochloric acid or sulfur dioxide in the gas currents facilitated the conversion. The claims of this patent are essentially for the method of producing aluminium nitrides by heating aluminium carbide in an atmosphere containing nitrogen to a red heat with or without the diluting substances admixed and with or without traces of acid gas in the nitrogen used.

Attention may be called to the term "aluminium nitrides" in this patent, corresponding to the fact that in the product thus obtained by Serpek he found that part of the nitrogen in the product was driven off by contact with air, and a further part by boiling with water, and a still further part by treatment with caustic alkali solutions. These facts pointed to the non-homogeneity of the product, which thus appeared to consist of a mixture of different nitrides instead of one distinctive chemical compound.

Continuing his work we find that Serpek later concluded that it might be possible to obtain nitride directly from mixtures of alumina and carbon, which it will be recalled he used as diluent substances in the conversion of the carbide. He was apparently not able to produce nitride directly from these two substances, but found that in the presence of a small amount of a metal such as copper or iron, which is capable of forming an alloy with aluminium, the formation of nitride from the mixture took place. In his U. S. Patent, 888,044 (filed May 24, 1907, and issued May 19, 1908), he describes mixing alumina with carbon in the proportions necessary for the formation of CO gas ($Al_2O_3 + 3C$), adding about 5 per cent. of either copper or iron, or a mixture of the two, and heating the mixture to a red heat in a current of nitrogen gas, at which temperature the partial formation of an alloy of aluminium produced by reduction, with the copper or iron, would be initiated. The idea seems to have been to produce a mixture on the point of reaction from its

[1] Address presented at the Joint Meeting of the American Electrochemical Society, American Chemical Society and Society of Chemical Industry, Chemists' Club, New York, February 7, 1913.

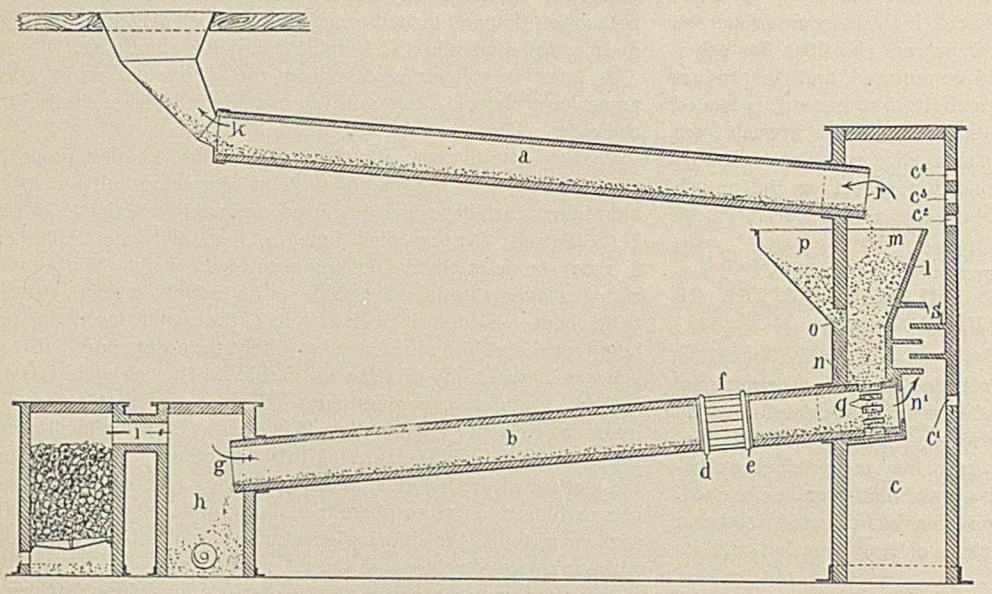
constituents and then to superadd the influence of the current of nitrogen gas. Traces of HCl or SO_2 gases are said to facilitate this reaction also, and the product is described as almost pure aluminium nitride. The reaction is said to develop a high temperature as soon as it is initiated. The claims are for producing aluminium nitrides by heating the mixture of alumina with carbon and a metal capable of forming an alloy with aluminium, to red heat in an atmosphere containing nitrogen, with or without the presence of small amounts of acid gases.

It is evident that in this method the conversion of alumina into aluminium nitride was aimed at, with the assistance of small amounts of metals as catalytic or assisting agents. Whatever may have been the results of the previously described process it led the investigator in the end to the method described in Serpek's U. S. Patent, 987,408 (filed December 15, 1909, and issued March 21, 1911), in which aluminium nitride is produced directly from Al_2O_3 and carbon in the presence of nitrogen, at a much higher temperature than previously recommended. This patent appears to be the result of considerable investigation on the conditions of this direct production and specifies very satisfactorily the temperatures at which the direct conversion takes place. The patentee speaks of the general supposition that extremely high temperatures would be required for such direct conversion, such as the highest temperatures of the electric furnace, but explains that by careful investigation he had found that if the temperature is closely watched some combination of nitrogen with aluminium can be observed even at 1100° C.; that at 1500° nitrogen is absorbed fairly rapidly, at 1700° energetically, and that at 1800° to 1850° the reaction may be almost designated as violent, producing almost chemically pure nitride. Further, Serpek found that temperatures higher than this gave a smaller yield and that somewhere above 2000° the production practically ceased, the decomposition temperature of the nitride appearing to be about 2120° C. This brilliant piece of experimental work really laid the foundation for the Serpek process as it now exists. The inventor further discovered that impure alumina, such as bauxite ore, was converted at somewhat lower temperatures than the pure material, evidently owing to the catalytic effect of the impurities present.

The claims of this patent are for producing aluminium nitride by heating aluminous compounds and carbon, or more specifically alumina and carbon, in an atmosphere containing nitrogen to temperatures not exceeding 2000° C., and preferably about 1800°.

Reference may very properly be made at this point to the experimental investigation and confirmation of these results by Prof. S. A. Tucker and Henry L. Read presented to the Eighth International Congress of Applied Chemistry and published in its transactions and in Volume 22, *Transactions of the American Electrochemical Society*. This investigation, carried on in the electrochemical laboratory of Columbia University under carefully regulated conditions, showed traces of nitrogen absorbed at temperatures of 1000 to 1100° and as much as 30 per cent. of nitrogen in selected portions of the product made between 1800 and 1900°. The product made at higher temperatures contained less nitrogen and was either sintered or fused. Impure alumina, such as bauxite, was found to be converted more easily than pure alumina, and the various statements of the Serpek patents were confirmed and verified.

Serpek's U. S. Patent, 996,032 (filed June 21, 1910, and issued June 20, 1911) contained a detailed description of the apparatus intended for the commercial manufacture of the carbide from bauxite or other aluminium ores. The plant consists of two superposed rotating cylindrical kilns, similar in construction to cement clinkering kilns. These kilns are inclined in opposite directions, so that the material being preheated in the upper kiln and passing from right to left, for instance, falls into the opening of the lower kiln for treatment at a higher temperature, where it passes from left to right. The bauxite alone is passed through the upper kiln, and there calcined, mixed with the necessary carbon in its passage from the upper to the lower kiln and the mixture treated by nitrogen at a high temperature in the lower kiln, which is provided with a detachable electric resistance furnace about midway of this length, intended to heat the charge within to the reacting temperature, about 1800° to 1900° C. The material is discharged from the lower end of this kiln into an air-tight receiver. A large gas producer of the ordinary type furnishes producer gas (approximately $^1/_3$ CO, $^2/_3$ nitrogen) to the lower end of the lower kiln. This gas entering at a temperature of about 400°, becomes highly heated as it passes through the kiln in a direction contrary to the descending charge, and at the electrically heated zone, at a temperature of 1800 to 1900°, reacts upon the mixture forming nitride. After leaving the high temperature zone the gas, enriched in CO coming from the reaction, preheats the descending charge, issues from the upper end of the kiln into a vertically closed chamber and passes through it to the opening of the upper kiln, where it meets a blast of air which burns it for the purpose of heating the contents of the upper or calcining kiln.



SERPEK APPARATUS: *a*, upper calcining kiln; *b*, lower nitriding kiln; *c*, vertical connecting chamber; c_1, c_2, c_3, c_4, air inlets; *d*, *e*, electric resistance sectional heater connections; *f*, electrical resistors; *g*, inlet for producer gas; *h*, discharge of nitride product; *k*, exit for starch gases and inlet for ground bauxite; *l*, hopper for mixing calcined bauxite with carbon; *m*, calcined bauxite; *p*, ground carbon; *o*, mixing of carbon with bauxite; *n*, mixture descending to lower kiln; *q* inlets to lower kiln; n^1, gas exit from lower kiln; *s*, baffle plates to promote partial combustion of gas; *r*, flame inlet to upper kiln.

The apparatus as thus described achieves a methodical heating of the reacting mixture, utilizes the CO gas made in the producer and also that evolved by the reaction in the furnace; aims at an intimate mixture of the charge by the rotation of the cylinders, and intimate contact of the nitrogen with the charge,

The inventor states that the silica impurity of the ore is mostly volatilized (probably as reduced silicon) from the charge at the reacting temperature and is thus carried out in the gas; and that the apparatus may be used for producing other nitrides made by analogous reactions.

The claims cover an apparatus comprising the features of a revolving inclined upper cylinder discharging into an inclined lower cylinder revolving in the opposite direction, through a vertical chamber in which the gas from the lower cylinder may be burned, combined with a detachable electric resistance heating furnace within the lower cylinder. One claim is for the single feature of a rotating calcining cylinder carrying a detachable electric resistance furnace.

Undoubtedly a large amount of excellent investigation has been put upon this process, and it deserves commercial success. The method is being tested upon a large experimental scale in France.

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CURRENT INDUSTRIAL NEWS

By W. A. HAMOR

SULFUR MINING IN TEXAS

While the sulfur mining industry near Freeport, Texas, is, according to correspondence to *The American Fertilizer*, 38, 50, as yet more or less in a preliminary stage, the preparations which have been made for carrying it on and the claims which are set forth by the promoters of the new project, indicate that it may, perhaps, reach very large proportions. The sulfur deposits are owned by a syndicate of bankers and financiers, who also purpose carrying out the development, and the construction of a terminal system and port facilities.

Attention was attracted several years ago to the sulfur possibilities of the Freeport section of Texas, when several wells that were bored in search of oil upon Bryan Heights penetrated a deposit of sulfur at a depth of 700–900 feet. Systematic boring operations were then begun and these, it is said, demonstrated that the entire mound is underlaid by the sulfur deposit, "the available supply" from which "is practically unlimited." In December, 1912, the syndicate installed equipment upon the property, bringing the sulfur to the surface by the process in use in Calcasieu Parish, near Lake Charles, La. The mining equipment so far installed consists of a 6,000 horse-power boiler plant and the necesssary pumping machinery for forcing steam into the well in operation.

NEW USES FOR NAPHTHALENE

The available annual supply of naphthalene may be thus expressed: Germany, 175,000 tons; England, 126,000 tons; France, 14.000 tons; and Belgium, 12,000 tons. Reference has already been made in THIS JOURNAL, 4, 308, to the use of naphthalene as an extraction agent; attention may be directed now to the use of an addition of naphthalene in the briquetting of bituminous coal (Grahn, *Glückauf*, October 26, 1912) and to the employment of naphthalene as a fuel for explosion engines (Ventou-Duclaux, *Mem. Compt. rend. Trav., Soc. Ing. Civ.*, 65, 514, 598). The use of naphthalene in these connections is suggested by its low price and available supply.

When used as a fuel for explosion engines, special apparatus is necessary in order to obtain the explosive mixture of air and naphthalene, since the melting point of naphthalene (79° C.) is well above atmospheric temperature. Nevertheless, a number of advantages are gained thereby, *viz.*: (1) Low cost, which may be only $0.0064 per horse-power hour; (2) security from fire; and (3) uniformity of composition. Naphthalene has been selected as the fuel for locomotives on the Trans-Siberian Railway.

NEW DEPARTURES IN ALUMINUM PLATE

An English concern which manufactures aluminum plate and vessels has had increasing success in introducing autogenously welded aluminum plate into various chemical industries. Reference has been made in THIS JOURNAL to the employment of aluminum in the brewing industry; it may be mentioned here that aluminum apparatus is now being used abroad in the manufacture of essences and sirups, in varnish-making. in the fatty acid and edible oil trades, and in handling nitric acid, Aluminum storage tanks are said to be very appropriate for containing extracts, essences and essential oils; they are highly resistant to the acids contained therein, they are cheaper than copper, and they are light, clean, and unbreakable. Over 50 firms now use aluminum pots in the varnish-making industry (*Chem. Trade J.*, 52, 103); in addition to giving improved color, the metal itself is said to have proved highly resistant to the heat and action of the fire gases. "By using a thinner pot, a well-known Midland firm runs gums two shades paler than when using a standard thickness aluminum pot." An especially strong aluminum alloy has been introduced for making stirrers, in order to avoid the excessive abrasion of copper pots caused by steel stirrers. Aluminum has also proved itself to be durable under the conditions of high temperature, pressure and vacuum encountered in the fatty acid and edible oil trades, and it is said to result in freedom from taste and color; "besides its use for the neutral oils, aluminum has been adopted by some of the largest firms in the country for dealing with the fatty acids" (*Ibid.*). Welded aluminum tanks are now being employed for blending and measuring acids, as well as for piping for conveying the acid in the works; a number of aluminum tank cars have been constructed during 1912 for the conveyance of strong nitric acid in bulk, and the success of aluminum for this purpose is reported to be favorable, although only one such tank is now in service.

THE PAPER INDUSTRY OF NORWAY AND SWEDEN

A review of the pulp and paper markets in Norway and Sweden in 1912, which appears in the December 21, 1912, issue of *Farmand*, contains the following statements regarding pulp production:

The production of sulfite cellulose in Norway and Sweden for export and for domestic consumption has been in recent years as follows, the figures being approximate estimates in tons of 2,000 lbs.:

1912, 992,079; 1911, 824,165; 1910, 809,096; 1909, 615,089; 1908, 686,739; 1907, 570,997; 1906, 440,924.

The production for 1913 is estimated at approximately 1,025,148 short tons.

The production of sulfate cellulose in the two countries was 188,495 short tons in 1911, and is estimated at about 203,927 short tons for 1912 and 212,746 short tons for 1913.

The most recent of the new sulfite mills, which were erected in the two countries as a result of the market conditions in 1908, has come into full operation during 1912 and will reach its full production in the course of 1913.

The increase in production for the year 1912 is estimated at about 149,914 short tons, but the estimates for 1913 indicate a further increase of only 33,069 short tons.

1912 STATISTICS FOR GERMANY

PIG IRON

The following table gives the total production of pig iron in

the German Empire during 1912, classified according to the purposes for which the iron was made and stated in metric tons:

	Metric tons	Per cent.
Foundry iron	3,338,839	18.7
Forge iron	525,423	3.0
Steel pig	2,201,489	12.3
Bessemer pig	388,855	2.2
Thomas (basic) pig	11,397,965	63.8
Total production	17,852,571	100.0
Increase in production over 1911	2,295,541	

Steel pig includes spiegeleisen, ferromanganese, ferrosilicon, and all similar alloys. The largest proportional increase was in steel pig, which was 26.9 per cent. The iron ore imports for the twelve months ending December 31, 1912, were 12,120,090 metric tons; exports, 2,309,628 tons.

PETROLEUM

In 1912, Germany was supplied with petroleum by the following countries:

	Metric tons (1 ton is 7.6336 bbl.)
United States	769,468
Austria-Hungary	156,567
Russia	34,939
Roumania	27,440
	988,414

POTASH SALTS

The sales of potash salts in 1912 amounted to $42,142,857, as against $38,819,047 in 1911. The exports of potash salts from the German Empire for 1912 were as follows, in metric tons:

	Total	To United States
Crude salts	1,300,559	650,297
Potassium chloride	286,528	190,775
Potassium sulfate	85,452	35,366
Potassium-magnesium sulfates	48,540	14,172
Total exportations	1,721,079	890,610

GERMAN FOREIGN TRADE DURING 1912

The total imports during 1912 amounted to 71,399,926 metric tons, as against 68,409,628 tons in 1911; the exports were 65,490,111 tons as against 59,150,188 tons in 1911. The imports and exports of materials of importance to the chemical industries were as follows:

	Metric tons	
	Imports	Exports
Earths and rocks	4,152,655	4,190,218
Ores, slags and ashes	15,493,677	2,588,810
Fossil fuels(a)	18,441,913	39,854,657
Mineral oils and other fossil crude materials	1,815,729	109,953
Coal-tar, coal-tar oils, and coal-tar products	92,493	343,670
Prepared wax, solid fatty acids, paraffin and similar candle material, candles, etc.	47,873	41,534
Chemical raw materials, acids, salts, etc.	1,226,137	2,868,892
Pigments and pigment materials	78,289	241,531
Varnishes, lacquers, putty	3,111	6,298
Ether, alcohol, various essential oils, artificial perfumes, etc.	43,545	12,735
Artificial manures	519,246	969,715
Explosives and matches	685	21,270
Chemical and pharmaceutical products not specifically mentioned	63,292	46,081
Iron and iron alloys	673,910	6,020,902
Aluminum and aluminum alloys	18,227	6,225
Lead and its alloys	93,758	54,354
Zinc and its alloys	58,112	134,935
Tin and its alloys (including britannia metal)	15,712	9,538
Nickel and its alloys	2,142	2,528
Copper and its alloys	229,189	91,174

(a) The coal production of Germany in 1912 was as follows, in metric tons:

Coal	177,094,917
Brown coal	82,339,583

The coke made amounted to 29,141,070 tons and the briquettes to 24,391,701 tons; of the briquettes reported in 1912, there were 19,058,050 tons made of lignite or brown coal.

Of the more important materials, the imports of coal, artificial manures and explosives decreased during 1912 as compared with 1911. As to the other materials, the import figures show a variable but material increase, amounting in ores and slags alone to over 2 million metric tons. Except in the case of chemical raw materials, the export figures show an increase in all classes (*Chem. Ztg.*, **37**, No. 12, 127).

POTASH FROM SEAWEED IN MEXICO

A concession has been granted to G. M. Bernstein, of Cananea, Mexico, to collect and utilize the kelp found floating along the western coast of Mexico, so far as relates to the entire coast of Baja. The principal plant is to be erected at San Diego. It is said (*Chem. Trade J.*, **52**, 165) that one company has offered to take 400 tons of the kelp per day at $5.00 per ton. This Mexican kelp has been rather a menace to shipping, for in many places it forms great islands, through which ordinary vessels find it difficult to sail.

A company has also been formed in Los Angeles, California, with a capital of $1,500,000, to manufacture potash and other products from kelp. The new company is a consolidation of the Pacific Kelp Co. and the Coronado Chemical Co. It is planned to erect a large plant near Los Angeles.

POTASH FROM FELDSPAR

It is reported from Gothenburg that a Swedish engineer, Alex. Lindblad, is conducting experiments on the production of potash from feldspar. The mineral is electrically smelted along with coal and iron, and 100 tons of potash manure salts have been produced. It is planned to erect a large plant, but the economic manufacture will be dependent upon whether such large quantities of ferro-silicon as must be produced as a main product can find a market without depressing prices.

THE PRESENT STATUS OF THE THIOGEN PROCESS

The thiogen process for the removal of sulfur dioxide from smeltery fume is based upon the reduction of the sulfur dioxide to elementary sulfur by means of fuel oil. When sulfur dioxide and a hydrocarbon of the ethylene series are heated together, the reaction which occurs is approximately as follows: $3SO_2 + 2CH_2 = 3S + 2CO_2 + 2H_2O$. Young (*Eng. Min. J.*, **95**, 369) states that this reaction is, however, slow and incomplete, and that in order to obtain satisfactory results it is necessary to use some reagent as an intermediary. Such a reagent has been found in calcium sulfide. The reaction with calcium sulfide and sulfur dioxide may be represented thus: $2CaS + 3SO_2 = 2CaSO_3 + 3S$. The reaction occurs in the dry state fairly rapidly at 100° C., while in the presence of water it takes place readily at ordinary temperatures. If the mixture of sulfide and sulfur is heated, the free sulfur distills off and the residual calcium sulfite may be reduced at a moderate red heat by means of hydrocarbon oil, whereby the calcium sulfide is regenerated. The reduction is rather difficult except in the presence of iron compounds, but with small amounts of these, reduction takes place readily at a low red heat, and, very favorably for the process, the full reducing value of the oil is realized. The reaction involved is as follows: $2CaSO_3 + 2CH_2 = 2CaS + 2CO_2 + 2H_2O$. In practice, if a mixture of sulfur dioxide and hydrocarbon vapor pass together over a mixture of calcium sulfite and calcium sulfide, the reactions given above take place side by side; that is, a portion of the sulfite is attacked by hydrocarbons and reduced to sulfide, which is in turn attacked by sulfur dioxide and reconverted into sulfite, and so on. Thus the mixture of sulfide and sulfite serves the purpose of a contact material.

Experiments conducted at the Penn Smeltery, Campo Seco, California, have eliminated many of the early difficulties encountered in the practical application of the thiogen process.

The present general scheme of operations in the process is as follows: The roaster gases pass first through a dust settler, which leaves them fairly clean, small amounts of dust seeming to produce no deleterious effect for a considerable time. From the dust chamber the gases pass to the fore-chamber, where a certain amount of oil is introduced. Thence the gases go to the contact chamber, where additional oil is introduced. The relative amounts introduced at the two places are adjusted by the temperature conditions which establish themselves in the contact chamber, the total amount being that necessary to burn out oxygen and reduce sulfur dioxide. The contact material is made up from plaster of Paris, which is mixed with water containing a small amount of iron salts, allowed to set and then broken into lumps. The issuing gases containing free sulfur pass to cooling and condensing apparatus, where the sulfur deposits. The principal chemical difficulties met have had to do with the poisoning of the contact material, owing to deposition of flue dust thereon, high temperatures, and the presence of foreign substances. The plant has been run for periods of 10 to 12 days upon the same charge of contact material, without loss of activity.

THE IODINE INDUSTRY

The ash of "tang" (sea-weed), which is now worked up for iodine in Norway, is shipped mainly to the iodine factories in England and Scotland. These factories, belonging to a trust, have depressed the price for sea-weed ash down to 2 cents per kilogram (a few years ago the price was still 3 cents); as a consequence, the calcination of sea-weed has diminished considerably in Norway. There are three small and unimportant iodine factories in Norway. The plan now is to erect a large modern iodine factory for working up 2,000 tons of sea-weed ash; this is intended to be entirely independent of the iodine trust, and is to be operated on a coöperative basis, in order to give the producers of the ash an opportunity to participate in the profits. It is also planned to manufacture norgine from sea-weed (norgine is a tungate of calcium, obtained by precipitating extracts from the sea-weed by milk of lime; it is used as an adhesive).

THE MANUFACTURE OF GAS MANTLES

Böhm, the author of "Das Gasglühlicht" (1905) and "Die Fabrikation der Glühkörper für Gasglühlicht" (1910), recently published (*J. Gas Ltg.*, 121, 33, 100, 181) a series of articles on the manufacture of mantles for incandescent gas lighting.

Cuprammonium-cellulose, Chardonnet and viscose silks are all used for the manufacture of mantles; mantles of artificial silk combine the high and well-maintained illuminating power of ramie fiber with a great elasticity of the ash skeleton, and are now coming largely into use. Cotton mantles decrease in brilliancy after use, while those of ramie fiber are fairly constant. Cotton and ramie mantles must be washed before impregnation, in order to remove the fat and mineral matter, and to accomplish this the following process is employed: the mantles are soaked over night in a 2 per cent. solution of nitric acid, and the following morning they are centrifuged, treated with a dilute solution of ammonia, and then thoroughly washed. They are next dried in a current of hot air, any imperfect ones being removed. The mantles are impregnated by being soaked in a 25 to 33 per cent. water solution of thorium and cerium nitrates in the proportion of 99 : 1, to which very small quantities of beryllium and magnesium nitrates are added to harden and impart strength to the ash skeleton. The soaking period varies from one to two minutes in the case of ramie or cotton mantles, and requires five hours for artificial silk mantles. The superfluous liquid is removed by passage through a wringing machine, and the quantity of salts left in the mantles may be varied by adjusting the rollers of the machine. The mantles are next dried at a temperature not exceeding 30° C., for which purpose the cotton and ramie mantles, which are cut into short lengths prior to impregnation, are stretched over glass cylinders, and the artificial silk mantles, which are impregnated in lengths, are run over wooden rollers. The upright mantles are next fitted with a sewn-on tulle head or with a patent head, and the latter is fixed by treating with a 50 per cent. water solution of aluminum and magnesium nitrates. The asbestos loop is next sewed on; the inverted mantles are drawn together with impregnated cotton, to form the "spider," and are then fastened on to the supporting ring. The mantle is shaped on a wooden shaper, and is then burned off by a Bunsen flame. The ash skeleton is dipped into a collodionizing solution, usually consisting of 100 parts of a 4 per cent. collodion solution, 40 parts of ethyl ether, 6.5 parts of camphor, and 3.5 parts of castor oil. After drying rapidly at 50–60° C., the mantles are trimmed to the right length, sorted out into different qualities, and the imperfect ones rejected.

ON SOME DEVELOPMENTS IN THE MINERAL OIL INDUSTRY

At a recent meeting of the Chemische Gesellschaft am Rigaschen Polytechnischen Institut, J. Hirsch presented a paper on the mineral oil industry, the following account of which appeared in *Chemiker-Zeitung*, 1913, No. 19, 198.

By means of modern rectification apparatus, benzines are now being obtained which boil within a range of less than 10° C.; the demand for benzines of low specific gravity has induced refiners in Europe to effect more satisfactory condensing arrangements; and, in Austria, the gases developed in winning petroleum are caught and condensed. Benzine products possessing a boiling point of between 130° and 180° C., and a flash-point of over 30° C. A.-P., are now finding use in varnish manufacture, replacing spirits of turpentine and rosin oil.

The burning oil industry of Europe has had much to contend with, for at the inception of the industry the lamps on the market were only suitable for burning American kerosene, and consequently there was much prejudice against the European product. However, by stronger heating and more abundant admission of air these complaints were remedied. It was also ascertained that the American oils could be duplicated by suitable methods of refining, by treatment at elevated temperatures with large amounts of sulfuric acid as monohydrate and with acids containing 20 per cent. anhydride. Treatment of the distillates with liquid sulfurous acid according to Edeleanu's patented process, was also found to be satisfactory. However, Hirsch states that it has been found that the cost of the products resulting from these methods of refining, is high. Experiments to replace caustic soda by caustic lime and to make the refining process a continuous one, have not met with success.

Hirsch states that the European petroleum industry is especially grateful to Ludwig Nobel for the introduction of methods of transportation (tank-wagons, tank-steamers, etc.), and to Victor Ragosin, who was the first to demonstrate the value of the residues for the manufacture of lubricants.

In producing lubricants from paraffin-free residues, decomposition must be carefully guarded against in the distillation; while, in the case of paraffin oils, a small amount of decomposition is desirable, for it accelerates the crystallization of the wax. The paraffin oils deposit the paraffin almost completely at 10° to 15° C. A preliminary separation of the crude paraffin is now conducted in filter presses constructed for a pressure up to 15 atmospheres. It has been found in Russian practice that the crude paraffin leaves the presses with a melting point of 44° C., as it contains 50 per cent. of oil. In freeing the wax from oil, it has been found that filter pressing according to the process of Nietsch and Winterstein, saves considerable time over the older methods of hydraulic-pressing and sweating. After refining the paraffin with acid and alkali, it is now bleached by treatment with "Hydrosilicate." Much progress has been

made in the construction of special machinery for the production and treatment of paraffin, and the author makes mention of some of the important European manufacturers.

THE WORLD'S PETROLEUM OUTPUT IN 1912

Petroleum Review notes that the world's petroleum production continues to increase, the 1912 output being as follows in barrels:

United States	219,000,000
Russia	63,000,000
Mexico	16,000,000
Dutch Indies	13,000,000
Roumania	12,500,000
Galicia	8,000,000
British India	7,500,000
Other countries	10,000,000
Total	349,000,000
Total in 1911	335,500,000

THE PROGRESS OF PAPERMAKING IN 1912

The report of Mr. A. D. Little to the American Paper and Pulp Association on the "Progress of Papermaking in 1912" appeared in *Paper*, 10, No. 11, 35.

The number of scientific papers bearing upon the paper and pulp industry which appeared during the year was only 127, and of these comparatively few had to do with important new developments or were otherwise notable.

Especially significant was the recent conference of over fifty paper manufacturers, representatives of testing laboratories, and others interested in paper specifications, which was called on January 24, by Dr. Stratton, of the Bureau of Standards, for the purpose of establishing a better understanding between the manufacturers and the Government officials who have the purchase of paper under their charge. It seems evident, Mr. Little observes, that the Government by the careful testing of deliveries furnished under its paper specifications has attained a two-fold result: First, a better and more uniform supply of paper; and, second, the protection of those manufacturers who have honestly endeavored to meet the specifications from the unfair competition of less conscientious bidders. Some progress was made at the conference toward greater uniformity of methods, and it was decided to hold similar meetings each year.

Further researches by Raitt into the papermaking qualities of bamboo amply confirm the general opinion of fiber experts to the effect that bamboo is particularly well adapted to the requirements of the industry and should become an important source of paper stock. Raitt concludes that the sulfate process is best adapted to the reduction of bamboo, and that 80 per cent. of the Indian bamboo stand is made up of species available for paper-making. He estimates that a mill producing 10,000 tons of bamboo pulp per year would require for a continuous supply of raw material a bamboo area of ten to twenty-two square miles.

Remington and others again called attention to the merits of Adansonia fiber as a stock for the manufacture of especially strong papers; the ultimate fibers resemble those of manila hemp. Von Possanner, after studying fibers from the German colonies, reports that *Ponzolzia hypoleuca* yields 58 per cent. of unbleached fiber which is long and supple, and resembles, in some respects, both linen and cotton. Beadle and Stevens obtained results on treating *Heydchium coronarium* which indicate that this tropical plant has promising possibilities; the fiber is stated to be equal in strength to the best pure manila. The utilization of the pith and fiber of waste sugar cane, after extraction of the sugar, is being developed along altogether new lines by the United Fruit Company.

Notable examples of the utilization of waste material are to be found in the new mills of the West Virginia Pulp and Paper Company at Covington, Va., for the manufacture of board of excellent quality from rossing waste; and at the mill of the Southern Wood Distillates and Fiber Company at Bogalusa, La., for the manufacture of board from waste wood by a new process involving treatment of the cooked waste under heavy crushing rollers.

The series of papers by Zacharias which appeared in sixteen numbers of *Papier-fabrikant* had, for their subject matter, the boiling and steaming processes for making brown mechanical wood pulp. These articles lead to the conclusion that boards made from boiled wood pulp show a decided advantage over steamed wood pulp board in tensile strength, resistance to folding, liability to tear, etc., and exhibit a greater stretch.

An important study into the causes of the yellow discoloration of paper has been made by Schoeller. Papers containing mechanical wood pulp discolor very readily with age, and papers containing straw and esparto are likewise subject to discoloration. Chemical wood pulp turns yellow to a less extent, while the paper made entirely from rags suffers slight discoloration. The coloring may be due to destruction of the ultramarine or other blue color used, or to actual yellowing of some component of paper. However, the most important cause of discoloration is the rosin size.

A RECORD OF INDUSTRIAL GROWTH

THE SEMI-CENTENNIAL OF THE FARBWERKE VORM. MEISTER LUCIUS & BRÜNING AT HÖCHST A/M, GERMANY

On January 4, 1913, the Farbwerke vorm. Meister Lucius & Brüning celebrated its semi-centennial. The history of this great firm is a record of the development of German chemical industry; the account which is given below is taken from *Die Chemische Industrie*, 36, 70.

In 1862, two chemists and two merchants organized a firm for the manufacture of tar colors, and the plant was started the following year with five workmen, one clerk, and one chemist. One boiler of 3 H. P. supplied the power. Fuchsin, anilin blue, alkali blue, aldehyde green, methyl violet, methyl green, and malachite green were the first products. In 1869, the manufacture of alizarine was taken up. In 1878, new buildings were erected for the manufacture of azo-dyes, and two years later the firm was formed into an Actien-Gesellschaft. In 1883, the manufacture of pharmaceutical preparations was started with antipyrine; in 1892, Koch's tuberculin and Behring's diphtheria serum were prepared and marketed; and in 1898 the manufacture of synthetic indigo was begun. The number of types of dyes and colors manufactured twenty-five years ago amounted to 1700–1800; to-day about 11,000 are manufactured. Salvarsan is another product of the concern.

In 1888, the steam engines had a total horse-power of 1840; to-day 30,000 H. P. are required.

The increase in personnel is shown in the following table:

	Year 1888	1912
Workmen	1860	7680
Foremen	50	374
Chemists	57	307
Engineers and other technical men	9	74
Office force, excluding agencies	86	611

In 1912, 8.6 million marks were paid in wages, and 5.2 million marks in salaries and bonuses. There is a pension fund, and benefit foundations have also been established.

THE LECITHIN AND ALBUMIN INDUSTRY

Lecithin became important industrially when Danilewsky discovered in 1895 that, when administered to growing animals, it was capable of stimulating their growth. It is now looked upon as the most valuable brain and nerve food yet discovered;

therefore, the discussion of its industrial aspects by Martin in *The Chemical World*, 2, 47, is of considerable interest.

The raw material for lecithin manufacture is yolk of egg, brain and nerve matter of freshly slaughtered animals, and seeds like peas and cereals generally. The bulk of the lecithin now on the market is made from yolk of eggs by extraction with solvents; by taking advantage of its different degrees of solubility in various solvents, it is possible to obtain it in a fairly pure state. Great care is, however, required, for, being an unstable ester, incautious treatment rapidly hydrolyzes it into evil-tasting products. One process of recent development consists in extracting yolk of egg in the cold with ethyl acetate, whereby fats, cholesterol, coloring matters, etc., are dissolved out, and there remains an odorless, almost colorless mass, very rich in lecithin and albumin. This residue may then be directly used by being ground up, sweetened with lactose, and mixed with casein or wheat gluten or similar protein, and marketed in the form of tablets. When, however, pure lecithin is required, the mass is next extracted with hot ethyl acetate (sometimes boiling ethyl alcohol is employed), which dissolves out the lecithin, leaving behind the protein matter. Other worked methods consist in first extracting with alcohol, and then extracting the product dissolved by the alcohol with acetone, thus leaving the lecithin behind.

It is difficult to ascertain whether vegetable lecithin has been successfully placed on the market, but serious endeavors have been made to find a suitable process for extracting it from peas, cereals, etc. The demand for yolk of egg for lecithin manufacture has reacted upon the albumin industry.

230 to 290 eggs give 1 kg. of albumin, which is sold in thin, transparent leaves. In the manufacture of albumin, the evaporation is conducted *in vacuo* at 30–50° C. It meets with extensive application for photographic paper manufacture, as a constituent of adhesives and patent foods, as a fining agent, as a mordant in dyeing and calico printing, and, mixed with potassium dichromate, for preparing blocks, the mixture being sensitive to light.

Another variety of albumin is prepared from the blood of recently slaughtered animals. The albumin imported by Germany comes mostly from China.

(The grades of lecithin now on the market run about 70 per cent., 80 per cent., 90 per cent., and 95 per cent. "Lecithalbumin" contains 30 per cent. of lecithin; letalbin contains about 20 per cent. lecithin and 75 per cent. of albumin. "Lecithan" and "Lecithin Blattmann," said to be of vegetable source, are on the German market.—W. A. H.)

HOT WATER SUPPLY SYSTEMS TO MINIMIZE CORROSION

In a paper by F. N. Speller on the "Durability of Welded Steel Pipe," published in *Engineering News*, 1911, March 23, were discussed the results of investigations on the relative corrosion of iron and steel in service, and the influence of dissolved oxygen and carbon dioxide in water, and a scheme was proposed for rendering the water practically harmless by removing the air after heating.

Speller now reports (*Eng. News*, 69, No. 7, 295) that a recent investigation has developed interesting points in regard to the present practice of laying out hot-water supply systems. He states that the influence of the arrangement of the piping on corrosion seems to be quite marked, depending upon whether the gases are liberated before the water enters the distributing system or not, although the separation of these gases is only partially accomplished under the best conditions. In his investigations, only large installations were considered, where, on account of the great quantity of hot water used, serious trouble would be most likely to occur.

The hot-water supply systems found differ in many details, but may be divided into two classes, according to whether the main vertical distributing lines are supplied from a common horizontal main in the basement, or from a similar horizontal distributing main above the level of the highest fixture near the roof. These types of installations are illustrated in Figs. 1 and 2.

The underfed system is characterized by a number of independent risers and return-risers, each supplying a separate section of the building. These risers are rarely vented at the top, and consequently the hot water is always supersaturated with air when the system is in continuous use. This is a good example of the closed type of heating so designed that it would be very difficult, if not entirely impracticable, to vent so as to remove the gases before the water is used.

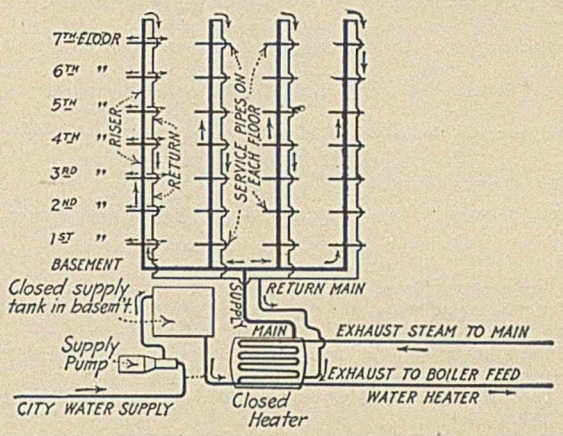


FIG. 1. UNDERFED CLOSED SYSTEM

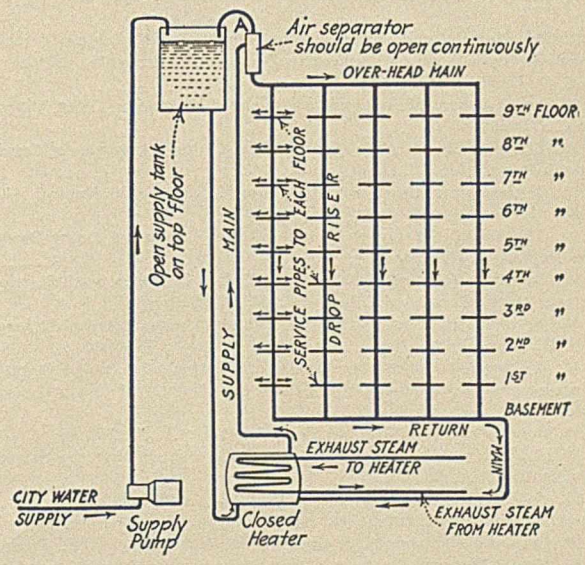


FIG. 2. OVERHEAD OPEN SYSTEM

The system illustrated by Fig. 2, on the other hand, is radically different in this respect, and to a considerable extent, although not completely, allows the escape of dissolved gases to the atmosphere at the highest point before the water is distributed throughout the system and returns to the heater. Since all the water used passes up through one riser, a vent must be provided, otherwise trouble due to the trapping of air in the upper lines would probably be experienced. It would seem to be a very simple matter to almost completely free the water from dissolved gases with such a system, by putting a simple air-separating device at the upper end of the main riser, as indicated at the point *A* in Fig. 2. To obtain the best results, the water

should be heated to about 200° F., using an inter-cooler, if desired, to reduce the temperature after leaving the air-separating chamber.

Several cases investigated, where large systems of hot-water supply lines have suffered serious damage in six or eight years, have all occurred in buildings equipped with closed heating systems. So far, Speller has not found serious trouble in systems of the open type where any attention has been given to venting, although the significance of adequate venting of such systems does not seem to be appreciated by engineers, for in some cases, where vents were provided, the practice was to keep them closed except when trouble with air-hammer was experienced.

SCIENTIFIC SOCIETIES

AMERICAN CHEMICAL SOCIETY—FORTY-SEVENTH MEETING AT MILWAUKEE. PROGRAM OF PAPERS

GENERAL MEETING

Some Applications of the Hydrogen Electrode in Instruction, Analysis and Research. (Illustrated.) *Joel H. Hildebrand.*

Copper in Steel. The Influence on Corrosion. (Illustrated.) *D. M. Buck.*

Some Projection Experiments with Spectra. (Illustrated.) *H. E. Howe.*

The Theory of Emulsions. *Wilder D. Bancroft.*

ILLUSTRATED PUBLIC LECTURE

Combustion, *Julius Stieglitz.*

DIVISION OF AGRICULTURAL AND FOOD CHEMISTRY

H. E. BARNARD, *Chairman*
GLEN F. MASON, *Secretary*

1. Chairman's Address. *H. E. Barnard.*
2. Studies upon Maize. *Carl L. Alsberg, F. Rabak, H. H. Bunzell* and *O. F. Black.*
3. The Equilibrium between Sugars and Sulfur Dioxide in Dried Fruit. *W. D. Bigelow.*
4. The Influence of Cooking on the Sulfurous Acid Content of Dried Fruit. *W. D. Bigelow.*
5. The Effect of Chicago Sewage on the Illinois River. *Edward Bartow.*
6. (*a*) The Composition of Canned Pumpkin. (*b*) The Tin Content of Canned Pumpkin. *H. E. Bishop.*
7. The Polarization of Light Colored Sugar Solutions. *A. Hugh Bryan.*
8. The Protein and Energy Requirements of Fattening Lambs as Determined by a Study of American Feeding Experiments. *Sleeter Bull* and *A. D. Emmett.*
9. Some Abnormal Factors of so-called Farmer's Cider Vinegars. *John C. Diggs.*
10. Analyses Showing the Composition of Different Grades of Commercial-pack Peas. *John C. Diggs.*
11. Methods for the Accurate Determination of Saltpetre. *J. T. Donald.*
12. The Modified Babcock Test for Fat in Sweetened Dairy Products —Ice Cream. *J. O. Halverson.*
13. Identification and Separation of Colors in Foods, with Particular Reference to the Permitted Colors. *P. W. Holtzendorff.*
14. Specific Heat Observations on Milk and Cream. *Arden R. Johnson* and *B. W. Hammar.*
15. A Thermal Method for the Determination of Ratio of Congealed to Uncongealed Moisture in Frozen Soils. *Arden R. Johnson* and *Roy E. Smith.*
16. Some Phases of National Food Control. *A. McGill.*
17. A Simple and Convenient Method for Determining the Salt Content of Butter. *A. E. Perkins.*
18. A Note Regarding an Absorption Tube and Receiver used in the Kjeldahl Nitrogen Determination. *A. E. Perkins.*
19. Organic Nitrogenous Compounds in Peat Soils. *C. S. Robinson.*
20. Some Chemical Changes taking Place during the Embryonic Development of the Chick. *R. H. Robinson.*
21. A Simple and Efficient 20° C. Bacteriological Incubator. *S. H. Ross.*
22. The Detection of Adulteration in Maple Syrup and Sugar. *J. F. Snell.*
23. A Comparative Study of the Composition of Hops Grown in Different Parts of the World. *H. V. Tartar* and *B. Pilkington.*
24. A Note on the Soluble Arsenic in Mixtures of Lead Arsenate and Soap. *H. V. Tartar* and *L. A. Bundy.*
25. Some Results of Hypochlorite Disinfection of the Baltimore City Water Supply. *J. Bosley Thomas* and *Edgar A. Sandman.*
26. Soil Acidity. *J. E. Harris.*

DIVISION OF BIOLOGICAL CHEMISTRY

CARL L. ALSBERG, *Chairman*
I. K. PHELPS, *Secretary*

1. Some Conditions Affecting the Stability and Activity of Certain Ferments. *J. H. Long.*
2. The Cholesterol and Cholesterol-esters of the Blood in Xanthoma Tuberosa Multiplex. *Jacob Rosenbloom.*
3. A Chemical Examination of the Brain of a Syphilitic Foetus. *Jacob Rosenbloom.*
4. A Problem in Metabolism. *W. D. Bancroft.*
5. The Development of Fat in the Black Walnut (Juglans Nigra). II. *F. M. McClenahan.*
6. A Study of the Renal Output of Rabbits Dying from Cottonseed Meal Poisoning. *W. A. Withers* and *J. F. Brewster.*
7. Certain Changes in the Nitrogen Ratios in Trout Eggs during Embryonic Growth. A Contribution to the Study of the Origin of the Energy of Development. *R. A. Gortner.*
8. Nephelometric Determinations of Proteins. I. Casein and Globulin in Milk. *P. A. Kober.*
9. A Laboratory Study of Well Water as a Cause of Typhoid Fever. *G. O. Higley.*
10. Influence of Cane Sugar on the Production of Tannase by Aspergillus Niger. *L. Knudson.*
11. Molds as a Factor in the Formation of Certain Nitrogenous Organic Soil Constituents. *M. X. Sullivan.*
12. Effect of Salicylic Aldehyde as a Soil Constituent. *J. J. Skinner.*
13. A Comparative Study of the Metabolism of Pneumococcus, Streptococcus, Bacillus Lactis Erythrogenes, and Bacillus Anthracoides. *M. L. Foster.*
14. A Biochemical Interpretation of the Inheritance of Acquired Characters. *L. W. Fetzer.*
15. The Chemical Effects of Ultraviolet Light on Albumin. *W. T. Bovie.*
16. Plant Growth in Relation to Acid and Alkaline Solutions. *Alfred Dachnowski.*
17. The Chemical Basis of Anesthesia and Irritability. *A. P. Mathews.*
18. Note on the Volatilization of Sulfuric Acid when Used as a Desiccating Agent in High Vacuum. (Lantern.) *H. C. Gore.*
19. A Constant Temperature Humidor. (Lantern.) *H. C. Gore.*
20. Study of the Chemical Changes in the Banana during Ripening with Special Reference to the Transfer of Water from Peel to Pulp. (Lantern.) *H. C. Gore.*
21. Study of the Effect of Uranyl Acetate and Ammonium Molybdate on the Polarization of *l*-Malic and *d*-Tartaric Acids. (Lantern.) *H. C. Gore.*
22. Cleavage of Hippuric Acid by Molds. *Arthur W. Dox* and *Ray E. Neidig.*
23. Bacterial Activities and the Rotation of Crops. *P. E. Brown.*
24. Purification of the Esterase from Pig's Liver. *George Peirce.*
25. Determination of Sulfur Dioxide in the Air. *Robert E. Swain* and *J. P. Mitchell.*
26. The Methyl and Methylene Ethers and Phenols Found in the Vegetable Kingdom and Light which They Seem to Throw on Certain Phases of Plant Metabolism. *Edward Kremers.*
27. The Analogy between the Formation of "Eyes" in Emmental Cheese and Crystal Growth. *W. M. Clark.*
28. Chemiluminescent Reactions with Physiologic Substances. *F. A. McDermott.*
29. A Note on "Oil Nut" (Pyrularia Pubera). *F. A. McDermott.*
30. On the Absorption of Water by Egg Yolk from Egg Albumen. *O. Riddle.*

DIVISION OF FERTILIZER CHEMISTRY

PAUL RUDNICK, *Chairman*
J. E. BRECKENRIDGE, *Secretary*

1. A Simple Method of Preparing Neutral Ammonium Citrate Solution. *Andrew J. Patten.*
2. A Survey of the Menhaden Industry. *J. W. Turrentine.*

Report of Committees:

Committee on Nitrogen. *Paul Rudnick.*
Committee on Phosphoric Acid. *G. A. Farnham.*
Committee on Potash. *J. E. Breckenridge.*
Committee on Phosphate Rock. *F. B. Carpenter.*
Committee on Fertilizer Legislation. *F. B. Carpenter.*

DIVISION OF INDUSTRIAL CHEMISTS AND CHEMICAL ENGINEERS

G. D. ROSENGARTEN, *Chairman*

1. The Training of the Fermentologist. *Carl A. Nowak.*
2. The Training of the Technical Chemist. *John Samuel Staudt.*
3. The Determination of Zinc in Treated Ties. *Francis C. Frary* and *M. Gordin Mastin.*
4. Preliminary Report upon a Practical Accelerated Test for Paints and Varnishes. (Lantern.) *A. M. Muckenfuss.*
5. Coal Ash. *S. W. Parr.*
6. Analyses of Glucose and Starch Sugars. *Edward Gudeman.*
7. The Specific Heat of Coal and its Relation to the Presence of Combined Water in the Coal Substance. *Horace C. Porter* and *Guy B. Taylor.*
8. Concrete Analysis. *Otto M. Smith.*
9. Fuller's Earth—Its Occurrence, Mining, Preparation, Use and Recovery. *Charles L. Parsons.*
10. The Uranium, Vanadium and Radium Situation. *Charles L. Parsons.*
11. A Simple Gasoline Gas Generator for Sulfur Determinations. *C. E. Waters.*
12. The Determination of Sulfate in Ammonium Sulfate with Special Reference to the Determination of Sulfur in Illuminating Gas. *R. S. McBride* and *E. R. Weaver.*
13. Determination of Sulfur in Illuminating Gas. *R. S. McBride* and *E. R. Weaver.*

DIVISION OF ORGANIC CHEMISTRY

TREAT B. JOHNSON, *Chairman*

WILLIAM J. HALE, *Vice-Chairman and Secretary*

1. The Action of Cyanimido Ether on Esters of Amido Acids. *Lloyd M. Burghart* and *Ralph H. McKee.*
2. Zygadenine—The Crystalline Alkaloid of Zygadenus Intermedius. *F. W. Heyl, F. E. Hepner* and *S. K. Loy.*
3. Some Constituents in the Leaves of Zygadenus Intermedius. *F. W. Heyl* and *F. E. Hepner.*
4. The Rearrangements of N-Acyl Aromatic Amines. *C. G. Derick.*
5. The Efficiency of the Preparation of Ether from Alcohol and Sulfuric Acid. *P. N. Evans* and *Lena M. Sutton.*
6. The Constitution of Surinamine. *Ben H. Nicolet* and *Treat B. Johnson.*
7. A New Method of Synthesizing β-Ketone Esters. *Treat B. Johnson.*
8. Pyrimidine Nucleosides. *Lewis H. Chernoff* and *Treat. B. Johnson.*
9. New Thiohydantoin Condensation Products. *Robert Bengis* and *Treat B. Johnson.*
10. The Constitution of "Acetylactone-Urea." *William J. Hale.*
11. Isodihydroaminocampholytic Acid and its Decomposition with Nitrous Acid. *L. F. Nickell* and *W. A. Noyes.*
12. Are Ammonium Compounds Atomic or Molecular? *R. S. Potter* and *W. A. Noyes.*
13. A Crystalline Resin Acid from the Digger's Pine. *O. A. Beath* and *Edward Kremers.*
14. The Oxidation of Indigo Blue by Phellandrene "Peroxide." *C. D. Geidel* and *Edward Kremers.*
15. Direct Halogen Substitution as a Laboratory Experiment in Organic Chemistry. *Edward Kremers.*
16. 2. Researches on Purines. On 2-Methylmercapto-6,8-Dioxypurine; 2-Methylmercapto-6-Oxy-8-Aminopurine. *Carl O. Johns* and *Emil J. Baumann.*
17. 1. Researches on Purines. On 2-Thio-6,8-Dioxypurine, 2,8-Dithio-6-Oxypurine; A New Method of Preparing Xanthine and Uric Acid. *Carl O. Johns* and *Albert G. Hogan.*

DIVISION OF PHARMACEUTICAL CHEMISTRY

B. L. MURRAY, *Chairman*

F. R. ELDRED, *Secretary*

1. Observations upon the Assay of Digestive Ferments. (Second Paper.) *H. T. Graber.*
2. Blood Fibrin in the Assay of Pepsin. *A. Zimmerman.*
3. Some New Reactions of Peptones and Enzymes. *Fred Klein.*
4. The United States Pharmacopoeia; Progress of the Ninth Revision. *Jos. P. Remington.*
5. The Analysis of Thymol Capsules. *Atherton Seidell.*
6. The Riegler Method for the Determination of Thymol. *Atherton Seidell.*
7. The Interpretation of U. S. P. Assay Processes. *Frank O. Taylor.*
8. Patent Medicine Ethics. *W. D. McAbee.*
9. Ergot. *W. A. Pearson.*
10. The Recovery of Alkaloids when Precipitated by Alkaloidal Reagents. *L. E. Sayre.*
11. Oxysulphobenzid. *F. P. Summers.*
12. An Alkaloid from Peumus Boldus. *F. P. Summers.*
13. The Alkaloidal Content of Individual Plants of Datura Stramonium D. Tatula and other Species and Varieties. *F. A. Miller* and *J. W. Meader.*
14. A Study of American Grown Belladonna. *F. A. Miller* and *R. N. Reed.*
15. The Stability of Tinctures and Fluid Extracts of Belladonna. *R. N. Reed* and *W. J. Rice.*
16. Improvements in Mercury Gas-Regulators. *A. B. Davis.*
17. Prevention of Emulsification in Extractions by Immiscible Solvents. (Second Paper.) *G. H. Meeker* and *E. L. Maines.*
18. An Automatic Apparatus for the U. S. P. Pepsin Test. *Frank R. Eldred.*
19. Crystalline Periodides of Antipyrine. *W. O. Emery* and *S. Palkin.*
20. Laboratory Studies on Malt Extract. *H. T. Graber.*
21. The Determination of Uncombined Hydrochloric Acid in Ferric Chloride Solution. *C. H. Briggs.*
22. An Oleoresin of Pseudotsuga Taxifolia (Lam.) Britton. *O. A. Beath* and *Edward Kremers.*
23. Some Aspects of Pharmacopoeial Revision. *Edward Kremers.*

DIVISION OF PHYSICAL AND INORGANIC CHEMISTRY

S. L. BIGELOW, *Chairman*

R. C. WELLS, *Secretary*

1. Some Surface Tension Phenomena. *S. L. Bigelow.*
2. The Oxygen Content of the Atmosphere. *O. F. Tower.*
3. The Role of Adsorption in the Catalysis of the Decomposition of Hydrogen Peroxide by Colloidal Platinum. *D. A. MacInnes.*
4. Detection of Cyanides in the Presence of Ferro-, Ferri-, and Sulfo-Cyanides. *O. L. Barnebey.*
5. A Method for Determining the Number of Valences in Molecules. *Albert P. Mathews.*
6. Various Reagents which Prevent the Action of Hydrochloric Acid on Permanganate in the Iron Titration. *O. L. Barnebey.*
7. Some New Ammonio Salts. *Edward C. Franklin.*
8. The Alpha-ray Activity of a Layer of Radioactive Solid as a Function of its Thickness. *Herbert N. McCoy.*
9. The Periods of Transformation of Uranium and Thorium. *Herbert N. McCoy.*
10. A Theory of the Origin and Continuance of Optical Activity in Nature without the Assumption of an Asymmetric Form of Energy or "Vital Force." *Arden R. Johnson.*
11. Atomic Weight of Yttrium. *James E. Egan* and *Clarence W. Balke.*
12. The Intergranular Cement in Metals. *W. E. Ruder.*
13. The Intermediate Ion Hypotheses and the Solubility of Salts of Higher Types. (Lantern.) *William D. Harkins, H. M. Paine* and *R. D. Mullinix.*
14. The Significance of the Exponent in Storch's Equation. *Stuart J. Bates.*
15. The Calculation of the Equivalent Conductance at Infinite Dilution. *Stuart J. Bates.*
16. Electrostenolysis. *Harry N. Holmes.*
17. Design for Specific Heat Apparatus (Electrothermal). *Arden R. Johnson* and *B. W. Hammar.*
18. The Relation of Geochemistry to a Proposed Basis for a Standard Potable Water. *Dr. J. Culver Hartzell.*
19. The Teaching of Quantitative Analysis. *E. H. Archibald.*
20. The Structure of the Trinitride Radical. *J. W. Turrentine.*
21. Demonstration of the Zeiss Gas Interferometer for the Estimation of Minute Traces of Gases. *R. H. Brownlee* and *R. H. Uhlinger.*
22. The Constitution of Certain Liquid Amalgams. *Joel H. Hildebrand.*
23. The Precipitation of Gold by Manganous Salts. *Albert D. Brokaw.*
24. Tetravalent (?) Nitrogen and Divalent (?) Chlorine. *Hans Mannhardt.*
25. On the Reaction of Sulfur with Potassium Hydroxide in Aqueous Solution. *H. V. Tartar.*
26. Binary Systems. (1) Hydrazine Ammonia. (2) Ammonium Trinitride Ammonia. (3) Ammonium Bromide Ammonia. *Fritz Friedrichs.*
27. Sulfates of Yttrium. *Charles James* and *E. H. Holden.*
28. Nitro-nitrogen Trichloride and Electromer of Ammono-Nitrogen Trichloride. Preliminary communication. *W. A. Noyes.*

SECTION OF INDIA RUBBER CHEMISTRY

D. A. CUTLER, *Chairman*

DORRIS WHIPPLE, *Secretary*

1. Wild Lettuce Rubber. *Charles P. Fox.*
2. An Adopted Wiley Extractor for Rubber Extractions. *Charles P. Fox.*
3. The Sampling of Rubber Goods. *John B. Tuttle.*
4. Preliminary Note on a New Method for the Direct Determination of Rubber. *L. G. Wesson.*

Committee Reports and General Conference.

Secretary's report. Report of the Rubber Consideration Committee and a procedure for the analysis of rubber goods as proposed to be sent to the Council of the American Chemical Society for adoption. Analytical Committee report. Specification Committee report.

STANDARD SAMPLE OF PHOSPHATE ROCK

The Committee on Phosphate Rock, Division of Fertilizer Chemists, American Chemical Society, has received a number of requests from chemists interested in the analysis of phosphate rock for a standard sample of this material. In response to these requests the Committee has decided to adopt as a tentative standard a sample of phosphate rock on which a large number of results have been obtained in coöperative work by members of this Division under the direction of the Committee. The average results obtained in this coöperative work will be furnished with this sample and while these results may not be absolutely correct, yet they are undoubtedly very close to the truth, as they represent an average of the best results obtained, most of them from analysts specializing in this line with the elimination of obviously erratic results. This standard should serve to bring about a greater uniformity in results, both by enabling those interested to check the work of analysts, as well as by enabling the individual analyst to check his own work with a sample of known composition.

Samples may be obtained from Mr. C. F. Hagedorn, Armour Fertilizer Works, Union Stock Yards, Chicago, Illinois, by enclosing 25 cents to cover cost of packing, mailing, etc.

F. B. CARPENTER,
Chairman of Committee on Phosphate Rock

AMERICAN ELECTROCHEMICAL SOCIETY—TWENTY-THIRD GENERAL MEETING

The general meeting of the American Electrochemical Society was held in Atlantic City, New Jersey, April 3rd–5th, with headquarters and meetings at the Hotel Traymore.

PAPERS READ AND DISCUSSED

Presidential Address by *President W. Lash Miller*, introducing the Symposium on the Electrodeposition of Metals.

Electrodeposition of Gold and Silver. *Francis C. Frary.*

Electrodeposition of Copper. *C. W. Bennett.*

Electrodeposition of Brass and Bronze. *C. W. Bennett.*

Electrodeposition of Cobalt and Nickel. *O. P. Watts.*

Electrodeposition of Lead. *F. C. Mathers.*

Solid, thick Lead Cathodes from Lead Acetate Solutions. *F. C. Mathers.*

Solid, thick Lead Cathodes from Lead Lactate Solutions. *F. C. Mathers and B. W. Cockrum.*

Conduction and Radiation of Heat. *I. Langmuir.*

Experiments with Furnace Electrodes. *F. A. J. FitzGerald* and *A. T. Hinckley.*

Aluminium Nitride. *J. W. Richards.*

Some Tests of the Edison Storage Battery. *C. W. Bennett* and *H. N. Gilbert.*

Concentration Cells Containing Organic Liquids Immiscible with Waters. *R. Beutner.*

Concentration Changes in Copper Sulfate Electrolysis. *C. W. Bennett* and *C. O. Brown.*

The 3-Phase-2-Phase Induction Furnace. *A. E. Greene.*

Making Electric Steel without Slag. *A. E. Greene.*

Evening Lecture—Hyperbasis. *Prof. F. B. Kenrick*, of Toronto.

Friday, April 4th, was spent in an excursion to Philadelphia where the making of crucible steel was seen and a two-ton Roechling-Rodenhauser induction steel furnace inspected at the works of the Crucible Steel Casting Co. Visits were also made to the Harrison Bros. Co. (Lead Paints, Lithophone and Contact Sulfuric Acid) and the United Gas Co. Works (Water Gas, Illuminating Gas, and Gas Testing Laboratory).

THE REPORT OF THE SECRETARY OF THE EIGHTH INTERNATIONAL CONGRESS OF APPLIED CHEMISTRY[1]

The preparatory work for this meeting began June 11, 1910. Organization for practical work was fully effected in the United States by June, 1911. The 11 American committees then formed contained upwards of 900 American chemists (not allowing for duplications, which probably did not exceed 10 per cent.). The United States Government and the Governors of 49 States, Territories, Insular Possessions of the United States and the Commissioners of the District of Columbia, as well as 30 societies, were further included in these committees by specially appointed representatives, numbering 124 in all.

In March, 1911, the Government of the United States sent formal invitations to all the governments of the world to join in this Congress. Twenty-eight foreign governments accepted and sent a total of 107 delegates. In 18 countries separate organizing committees were formed or organizers were installed, containing upwards of 573 individuals (not allowing for duplications, which probably did not exceed 10 per cent.), and 55 societies and educational institutions.

The Executive Committee of this Congress especially invited 1,483 different technical, scientific and trade societies and educational institutions the world over to join the Congress. Two hundred and thirty-seven in 28 different countries accepted this invitation and appointed 348 delegates.

The total working force in the interests of this Congress the world over amounted therefore to not less than 2,000 individuals (not allowing for duplications, which probably did not exceed 10 per cent.), 392 societies and educational institutions, and 75 governments (including State governments of the United States).

On the publicity side the American Committee placed upon its mailing list 438 trade, technical and scientific publications the world over, and all were supplied with the various publications and appropriate news items from time to time. There were distributed by the Congress, and also on its behalf by American societies, such as the American Chemical Society, the American Electrochemical Society and the Chemists' Club, upwards of 300,000 pieces of printed matter describing the expectations, aims and hopes of this Congress, as well as its accomplishments to date, from about forty different points of view.

Communication was established with central organizing committees in Austria, Hungary, Belgium, Denmark, France, Germany, Great Britain, Greece, Japan, Netherlands, Dutch East Indies, Norway, Russia and Sweden and organizers were installed in Argentina, Chile, Italy and Portugal through whom publicity was maintained, as well as memberships solicited.

The American Sectional Presidents and their Sectional Executive Committees of the 24 different sections themselves by letter solicited papers from specialists the world over. Of these 24 only 17 sections kept a record of such outgoing matters, and these 17 sections sent out 17,176 letters for that purpose.

The net result of the work of this organization the world over is a total of 4,163 members residing in 36 different countries, of which 1,883 coming from 27 different countries, attended the Congress meeting and registered, but more than 9 per cent. failed to bring their membership credentials with them although specifically requested so to do. Of these 1,883 members only 228 or 12 per cent. registered for sections; 1,284 papers were promised or submitted for the Congress; 304 of these were never supplied. Of the papers presented 20 per cent. were rejected either by the Sectional Committee or by the Committee on Papers and Publications, or by both committees. Of the 789 papers accepted by the Congress 560 or 71 per cent. were in print before the Congress assembled in New York. The remaining 229 papers appear in the Appendix. One thousand, three hundred and twenty-two members withdrew their sets of the first 24 volumes of the original communications, and of these not fewer than 514 shipped them directly out of New York City leaving not to exceed 808 sets available for use in preparing discussions; no doubt only a very few of these 808 were used for any such purpose, which was the exact purpose for which so much additional labor and effort were expended.

Two hundred and seventy-seven ladies residing in 12 different countries acquired attendance cards, and of these 246 registered and attended the social functions of the Congress.

The work of the 24 sections was accomplished in 146 meetings

[1] Report, Eighth International Congress of Applied Chemistry, Vol. 28.

TABLE I—SUMMARY OF THE WORK OF THE CONGRESS

	Meetings held	Attendance	Duration Minutes	Papers: Read	Papers: Discussed	Resolutions offered	Papers carried over	Left-over papers read later	Papers read by: Author	Papers read by: Substitute	Papers read by: Title	Exhibits and specimens	Lantern	Experiments	Participants in discussions	Minutes used: Reading	Minutes used: Discussing	Minutes used: Extra
Sectional meetings	118	3956	10127	546	252	24	147	81	286	129	131	38	8	3	610	4597	2987	2543 (25%)
Joint meetings.....	28	5747	2873	157	80	1	25	5	96	23	38	17	13	4	250	1603	929	341 (11%)
Grand total.....	146	9703	13000	703(a)	332	25(b)	172(c)	86	382	152	169	55	21	7	860	6200	3916	2884 (22%)

(a) 86 papers, or 11 per cent. of the total, were not in any way presented to the Congress and were therefore of no interest whatever to it.

(b) 172 papers, or 22 per cent. of the total, were carried over from the assigned program because of the absence of the author or his representative.

(c) Of these 25 resolutions 9 were laid before the Congress by the Commission of International Congresses of Applied Chemistry; the remaining 16 failed to be so brought up for final action because of the failure of their sponsors to attend to the necessary details required by the constitution and by-laws of the Congress.

TABLE II—SUMMARY OF EACH MEETING PERIOD

Sept.	Time	Meetings	Meetings held	Attendance	Duration Minutes	Papers: Read	Papers: Discussed	Resolutions offered	Papers carried over	Left-over papers read later	Papers read by: Author	Papers read by: Substitute	Papers read by: Title	Exhibits and specimens	Lantern	Experiments	Participants in discussions	Minutes used to: Read	Minutes used to: Discuss
6	A.M.	Sectional	21	558	1655	65	31	2	39	..	33	21	18	4	3	..	67	711	342
	P.M.	sectional	13	542	1173	60	30	..	44	8	36	10	11	7	1	..	63	507	354
		joint	1	200	150	2	..	..	..	..	2	..	..	..	2	..	...	60	...
		total	14	742	1323	62	30	..	44	8	38	10	11	7	3	..	63	567	354
7	A.M.	sectional	13	430	1209	46	29	1	33	6	22	12	7	4	1	..	79	532	436
		joint	3	385	285	16	14	..	14	..	8	7	1	1	..	..	57	131	189
		total	16	815	1494	62	43	1	47	6	30	19	8	5	1	..	136	663	625
	P.M.	joint	1	1800	60	1	..	..	..	..	1	..	..	..	1	..	...	60	...
9	A.M.	sectional	17	652	1517	94	53	5	13	11	59	26	9	3	..	1	111	764	537
		joint	2	202	215	15	8	..	1	..	6	2	7	..	..	..	27	82	116
		total	19	854	1732	109	61	5	14	11	65	28	16	3	..	1	138	846	753
	P.M.	sectional	10	376	741	41	16	..	5	1	22	11	8	..	..	1	44	295	185
		joint	3	313	270	16	8	..	1	1	7	2	7	1	1	1	8	130	45
		total	13	689	1011	57	24	..	6	2	29	13	15	1	1	2	52	425	230
10	A.M.	sectional	13	539	1185	60	29	8	6	9	35	13	12	4	1	..	80	637	373
		joint	3	423	318	18	9	..	5	1	10	3	5	2	..	..	23	222	107
		total	16	962	1503	78	38	8	11	10	45	16	17	6	1	..	103	859	480
	P.M.	sectional	8	274	659	36	12	6	4	7	17	5	14	6	..	2	38	196	198
		joint	2	319	230	11	9	..	1	..	11	..	..	3	4	1	21	120	88
		total	10	593	889	47	21	6	5	7	28	5	14	9	4	3	59	316	286
11	A.M.	sectional	5	88	399	22	14	..	7	3	13	6	3	..	..	..	36	207	99
		joint	2	1030	185	7	4	..	..	..	5	..	2	..	1	..	15	102	80
		total	7	1118	584	29	18	..	7	3	18	6	5	..	1	..	51	309	179
	P.M.	sectional	3	78	215	16	7	..	7	3	8	..	8	3	2	..	18	103	62
		joint	7	946	640	41	17	1	2	3	25	8	8	7	3	2	52	452	167
		total	10	1024	855	57	24	1	9	6	33	8	16	10	5	2	70	555	229
12	A.M.	sectional	11	366	1116	79	27	..	..	28	33	14	32	7	..	..	63	530	289
		joint	3	99	290	18	10	..	1	..	15	1	2	1	..	..	33	169	110
		total	14	465	1406	97	37	..	1	28	48	15	34	8	..	..	96	699	399
	P.M.	sectional	4	71	325	13	4	2	..	5	3	2	9	..	..	..	17	85	110
		joint	1	30	130	11	3	..	..	..	5	..	6	1	..	..	4	95	27
		total	5	101	455	24	7	2	..	5	8	2	15	1	..	..	21	180	137

(118 sectional meetings and 28 joint meetings), various details in connection with which are given in Table I.

It appears therefore that the averages of the main features per sectional meeting and per joint meeting were as follows:

	Sectional meeting	Joint meeting
Attendance per meeting.....	34	205
Duration of meeting.....	86 minutes	96 minutes
Papers read per meeting.....	4½	5½
Papers discussed per meeting.....	2	3
Participants in discussion per meeting.....	5	9
Minutes for reading a paper.....	8½	10
Minutes for discussing a paper.....	12	11

About 48 per cent. of the papers presented in Sectional meetings were discussed and about 53 per cent. of the papers presented at joint-meetings; about two-thirds as much time was spent in discussing as in reading papers at sectional meetings and about three-fifths as much in discussing as in reading papers at joint meetings.

THE ATTENDANCES

Taking the final total registration as a basis and assuming further that only members were present (which is manifestly not a thoroughly fair basis or assumption, but the best measure that can be conveniently applied) it appears that the attendance upon these 146 meetings, for each meeting period (of which there were 12 of 2 hours each), ranged from 5.4 per cent. at the last meeting period to 95.6 at the meeting of Saturday afternoon, September 7th, as shown in the following table:

Session of		Attendance	Per cent. of total registration
September 6	forenoon.....	558	29.6
	afternoon.....	742	39.4
September 7	forenoon.....	815	43.2
	afternoon.....	1800	95.6
September 9	forenoon.....	854	45.4
	afternoon.....	689	36.6
September 10	forenoon.....	962	52.1
	afternoon.....	593	31.5
September 11	forenoon.....	1118	59.4
	afternoon.....	1024	54.4
September 12	forenoon.....	465	24.7
	afternoon.....	101	5.4

Grand average attendance 44.5 per cent.

THE MEETING PERIODS

As provided for in the program there was a total of 12 meeting periods containing a total of 530 meeting hours, or 265 meetings

TABLE III—SUMMARY OF THE SECTIONAL MEETINGS

	Meetings held	Attendance	Duration Minutes	Papers Read	Papers Discussed	Resolutions offered	Papers carried over	Left-over papers read later	Papers read by Author	Papers read by Substitute	Papers read by Title	Exhibits and specimens	Lantern	Experiments	Participants in discussions	Minutes used to Read	Minutes used to Discuss	Range of attendance Max.	Range of attendance Min.	Range of attendance Av.
I. Analytical Chemistry	6	240	480	29	10	4	9	1	23	1	5	2	..	..	21	180	75	50	27	40
II. Inorganic Chemistry	5	295	396	28	11	1	1	3	12	9	7	4	..	..	24	221	80	145	8	59
IIIa. Metallurgy and Mining	5	210	385	24	10	4	15	3	11	2	11	5	4	..	18	194	97	62	10	42
IIIb. Explosives	6	254	480	23	6	1	6	7	14	4	5	..	..	..	15	285	95	50	38	42
IIIc. Silicate Industries	2	32	150	13	6	..	...	5	4	7	2	2	..	..	12	10	..	16	16	16
IV. Organic Chemistry	8	531	715	65	33	..	25	27	38	11	16	7	..	3	69	468	195	110	25	67
IVa. Coal-Tar Dyes	1	29	125	5	3	..	...	..	4	1	...	..	..	..	9	68	45	29	29	29
Va. Sugar	7	147	496	31	10	..	3	2	17	6	8	3	..	..	18	237	87	31	12	21
Vb. India Rubber	5	143	182	11	5	..	3	4	6	...	5	2	..	..	18	79	55	78	11	29
Vc. Fuels and Asphalt	3	80	260	10	4	..	7	1	6	...	4	1	..	..	12	75	115	35	15	27
Vd. Fats, Fatty Oils and Soaps	2	25	130	5	3	..	1	..	4	1	...	..	..	..	12	72	53	24	1(?)	13
Ve. Paints, Oils and Varnishes	8	367	657	18	15	..	5	3	15	3	...	2	1	..	71	312	202	69	23	46
VIa. Starch, Cellulose and Paper	8	198	530	29	17	1	2	1	13	2	14	1	..	..	48	155	308	36	15	25
VIb. Fermentation	6	131	885	35	15	1	...	..	11	22	2	1	..	..	30	612	205	38	12	22
VII. Agricultural Chemistry	8	259	770	62	24	..	20	7	25	20	17	5	..	..	62	238	181	46	19	32
VIIIa. Hygiene	4	104	503	19	14	3	7	6	16	3	...	..	..	..	29	225	148	44	7	26
VIIIb. Pharmaceutical Chemistry	9	265	855	18	13	5	1	3	3	15	...	..	..	..	25	290	450	50	20	29
VIIIc. Bromatology	7	152	550	45	5	3	2	5	10	6	29	..	..	..	11	113	50	29	14	22
VIIId. Biochemistry	4	96	210	12	(a)	..	...	..	11	...	1	..	..	..	(a)	(a)	(a)	40	6	24
IX. Photochemistry	6	122	705	33	30	..	...	..	20	12	1	..	3	..	59	336	304	36	10	20
Xa. Physical Chemistry	2	147	113	9	4	..	6	..	6	1	2	3	..	..	7	94	19	112	35	74
Xb. Electrochemistry	3	85	250	13	7	..	22	3	13	...	...	..	..	..	20	201	83	50	20	28
XIa. Law and Legislation	3	44	300	9	7	1	12	..	4	3	2	..	..	..	20	132	140	20	12	15
XIb. Pol. Econ. and Conservation	0	..	...	...	...	..	...	..	...	...	...	..	..	..	...	..	..	...	..	..
Totals	118	3956	10127	546	252	24	147	81	286	129	131	38	8	3	610	4597	2987	145	6	34

(a) No record by Sectional Secretary under this heading.

TABLE IV—SUMMARY OF THE JOINT MEETINGS

Sections and Societies	Meeting period	Attendance	Duration Minutes	Papers Read	Papers Discussed	Resolutions offered	Papers carried over	Left-over papers read later	Papers read by Author	Papers read by Substitute	Papers read by Title	Exhibits and specimens	Lantern	Experiments	Participants in discussions	Minutes used to Read	Minutes used to Discuss
4a, 5e, 9	6P	200	150	2	..	..	..	..	2	..	..	..	2	..	...	60	...
3a, 10a, 11b, A. I. M. E.	7A	200	95	6	6	..	2	..	4	2	..	..	..	..	23	46	47
3b, 11a	7A	40	70	3	2	..	12	..	3	..	..	..	..	..	9	65	60
2 and 3a	7A	145	120	7	6	..	..	..	1	5	1	1	..	..	25	20	82
2, 7 10b, 10a	7P	1800	60	1	..	..	..	..	1	..	..	..	1	..	...	60	...
2, 10a, 10b, A. E. C. S.	9A	150	120	8	6	..	1	..	5	1	2	..	..	..	18	59	51
5c and 11b	9A	52	95	7	2	..	..	..	1	1	5	..	..	..	9	23	65
8a and 8c	9P	58	105	6	2	..	1	..	4	..	2	..	..	..	5	45	25
4a and 6a	9P	55	45	6	2	..	..	..	2	2	2	..	..	..	3	35	20
2, 3a, 10a, 10b, A. E. C. S.	9P	200	120	4	2	..	..	1	1	..	3	1	1	1	...	50	...
6a, 10a, A. E. C. S.	10A	250	105	7	3	..	..	..	4	..	3	..	..	..	8	78	27
1 and 5c	10A	75	110	7	3	..	..	..	2	3	2	..	..	..	9	80	50
4 and 4a	10A	98	103	4	3	..	5	1	4	..	..	2	..	..	6	64	30
10a, 11b	10P	250	120	3	3	..	..	..	3	..	..	1	1	1	8	55	41
1 and 5c	10P	69	110	8	6	..	1	..	8	..	..	2	3	..	13	65	37
2, 7, 10a, 10b	11A	1000	60	1	..	..	..	..	1	..	..	1	1	..	...	60	...
3a and 5c	11A	30	125	7	4	..	..	..	5	..	2	..	1	..	15	42	80
2, 3a, 10b	11P	50	120	6	3	..	..	..	6	..	..	3	2	2	5	88	15
5a, 10a	11P	65	120	6	4	1	..	..	4	2	..	..	..	..	20	38	65
6a and 6b	11P	23	70	8	1	..	..	..	3	3	2	2	..	..	1	62	3
5c and 11b	11P	40	145	12	3	..	..	..	4	2	6	1	1	..	7	90	47
4 and 5b	11P	298	50	1	0	..	..	..	1	..	..	..	..	..	...	50	...
4 and 5b	11P	420	60	5	3	2	..	3	4	1	..	..	..	..	10	87	13
1, 8b, 8c	11P	50	75	3	3	..	..	..	3	..	..	1	..	..	9	37	24
5c, 11b	12A	35	140	9	4	..	..	..	7	..	2	..	..	..	6	110	30
5b, 6a	12A	14	35	4	1	..	..	..	4	..	..	..	..	..	4	6	25
1 and 3a	12A	50	115	5	5	..	1	..	4	1	..	1	..	..	23	53	55
5c, 11b	12P	30	130	11	3	..	..	..	5	..	6	1	..	..	4	95	27
Totals	28	5747	2873	157	80	1	25	5	96	23	38	17	13	4	250	1603	929

in all. The time actually consumed in meetings was 217 hours or 41 per cent. of the time provided and 146 separate meetings were held. This last figure must be augmented by the number of sections participating in joint sessions since each joint session is a meeting for each section, *i. e.*, 39 meetings more than 146 were held or the equivalent of 185 sectional meetings or 80 meetings, *i. e.*, 30 per cent. short of program capacity.

Table A (p. 347) shows the joint and sectional meetings reported by the Sectional Secretaries.

Each section could have met at most 12 times. Table B (p. 347) shows the extent to which this possibility was utilized:

A summary of the work of each meeting period is presented in Table II, showing the details of the sectional and joint meetings of each of the 12 meeting periods.

The detail of the total work of each section in its sectional meetings is given in Table III.

The detail of the total work of each Joint meeting is given in Table IV.

TABLE A

Section	Joint meetings	Sectional meetings	Total meetings	Section	Joint meetings	Sectional meetings	Total meetings
1	4	6	10	6a	4	8	12
2	6	5	11	6b	1	6	7
3a	6	5	11	7	2	8	10
3b	1	6	7	8a	1	4	5
3c	0	2	2	8b	1	9	10
4	3	8	11	8c	2	7	9
4a	3	1	4	8d	0	4	4
5a	1	7	8	9	1	6	7
5b	3	5	8	10a	8	2	10
5c	7	3	10	10b	5	3	8
5d	0	2	2	11a	1	3	4
5e	1	8	9	11b	6	0	6
				Totals	67	118	185

TABLE B

Number of meetings	Number of section
12	VIa
11	II; IIIa; IV
10	I; Vc; Ve; VII; VIIIb; Xa
9	VIIIc
8	Va; Vb; Vb
7	IIIb; VIb; IX
6	XIb
5	VIIIa
4	IVa; VIIId; XIa
2	IIIc; Vd

THE PAPERS

The total number of papers presented to the Congress was 789; of these 560 or 71 per cent. were in print at the opening of the Congress; the remaining 229 were printed in the appendix; 147 papers or 18.63 per cent. of the whole number were illustrated. The following tables contain detail of information:

TABLE V—PAPERS PRINTED BEFORE THE CONGRESS OPENED

Sec.	Pages of text	Papers: Total	Papers: Illustrated	Languages used: English	French	German	Italian
1	460	48	12	35	13	0	0
2	246	30	9	17	9	4	0
3a	150	13	5	12	0	0	1
3b	150	19	4	15	2	2	0
3c	131	14	5	14	0	0	0
4	264	36	3	31	1	4	0
4a	24	3	0	1	2	0	0
5a	104	15	1	9	3	3	0
5b	92	8	3	4	3	1	0
5c	334	33	12	27	3	3	0
5d	36	6	1	5	0	1	0
5e	212	19	5	16	2	1	0
6a	294	34	12	31	2	1	0
6b	278	28	4	21	5	2	0
7	371	53	5	39	12	2	0
8a	50	5	0	3	2	0	0
8b	95	14	0	14	0	0	0
8c	358	39	3	30	7	1	1
8d	282	39	4	19	16	2	2
9	280	33	6	33	0	0	0
10a	209	21	11	20	0	1	0
10b	252	27	11	21	3	3	0
11a	79	13	0	2	11	0	0
11b	193	10	2	10	0	0	0
Totals	4944	560	118	429	96	31	4

TABLE VI—PAPERS PRINTED AFTER THE CONGRESS CLOSED

Sec.	Pages of text	Papers: Total	Papers: Illustrated	Languages used: English	French	German	Italian
1	104	7	2	6	0	1	0
2	81	10	2	7	1	2	0
3a	23	6	1	4	2	0	0
3b	107	7	2	6	1	0	0
3c	12	2	0	1	1	0	0
4	136	31	2	21	6	4	0

TABLE VI—(*Continued*)

Sec.	Pages of text	Papers: Total	Papers: Illustrated	Languages used: English	French	German	Italian
4a	16	4	0	4	0	0	0
5a	84	15	0	4	11	0	0
5b	60	5	2	5	0	0	0
5c	154	28	2	21	1	6	0
5d	6	1	0	0	0	1	0
5e	21	3	0	3	0	0	0
6a	32	6	0	2	0	4	0
6b	54	11	1	9	0	2	0
7	80	10	0	9	1	0	0
8a	152	13	4	13	0	0	0
8b	34	5	0	4	0	1	0
8c	60	12	2	10	0	1	1
8d	8	2	0	0	2	0	0
9	32	5	1	4	1	0	0
10a	112	11	5	11	0	0	0
10b	84	14	2	12	0	2	0
11a	46	7	0	1	0	6	0
11b	130	14	1	14	0	0	0
Totals	1628	229	29	171	27	30	1

TABLE VII—TOTALS OF DETAILS OF ALL PAPERS PRINTED

Sec.	Pages of text	Papers: Total	Papers: Illustrated	Languages used: English	French	German	Italian
1	564	55	14	41	13	1	0
2	327	40	11	24	10	6	0
3a	173	19	6	16	2	0	1
3b	257	26	6	21	3	2	0
3c	143	16	5	15	1	0	0
4	400	67	5	52	7	8	0
4a	40	7	0	5	2	0	0
5a	188	30	1	13	14	3	0
5b	152	13	5	9	3	1	0
5c	488	61	14	48	4	9	0
5d	42	7	1	5	0	2	0
5e	233	22	5	19	2	1	0
6a	326	40	12	33	2	5	0
6b	332	39	5	30	5	4	0
7	451	63	5	48	13	2	0
8a	202	18	4	16	2	0	0
8b	129	19	0	18	0	1	0
8c	418	51	5	40	7	2	2
8d	290	41	4	19	18	2	2
9	312	38	7	37	1	0	0
10a	321	32	16	31	0	1	0
10b	336	41	13	33	3	5	0
11a	125	20	0	3	11	6	0
11b	323	24	3	24	0	0	0
Totals	6572	789	147	600	123	61	5
Per cent.		100	18.63	76.04	15.59	7.73	0.64

DISCUSSIONS

Table I shows that a grand total of 332 papers were discussed by a grand total of 860 participants. However, the discussions printed in volume 27 show that only 172 were discussed and by only 439 participants (*i. e.*, 247 different individuals or 13.1% of the registered attendance) and reported for publication. It appears therefore that the discussion of 160 papers or 48 per cent. and of 421 participants or 49 per cent. failed of finding a place in the final printed volume. The reasons therefor are probably correctly stated in the last paragraph on page 326 of THIS JOURNAL. Table VIII shows the detail of the recorded discussions.

PARTICIPATION IN THE SECTIONS

From Table VII it appears that papers in the English language were offered in each of the 24 sections; that papers in the French language were offered in 20 of the 24 sections, in the German language in 18 of the 24 sections, and in the Italian language in 3 of the 24 sections. It further appears that in the sections

on Fats, Fatty Oils and Soaps, Pharmaceutical Chemistry, Physical Chemistry and on Political Economy and Conservation of Natural Resources, writers in the French language made no original communications; that in the Sections on Metallurgy and Mining, Silicate Industries, Coal-tar Colors and Dyestuffs, Hygiene, Photochemistry and on Political Economy and Conservation of Natural Resources, writers in the German language made no original communications and that writers in the Italian language made original communications only in Metallurgy and Mining, in Bromatology and in Biochemistry.

TABLE VIII—DISTRIBUTION OF PAPERS, DISCUSSIONS OF WHICH WERE PRINTED IN VOLUME 27

Sec.	Vols. 1–24	Appendix	Total	No. of participants reported
1	17	3	20	56
2	8	1	9	21
3a	1	1	2	4
3b	8	1	9	14
3c	1	0	1	2
4	6	6	12	25
4a	1	2	3	7
5a	5	5	10	23
5b	2	1	3	8
5c	10	5	15	45
5d	2	1	3	7
5e	15	2	17	62
6a	8	0	8	13
6b	1	0	1	2
7	21	6	27	70
8a	0	1	1	1
8b	3	0	3	4
8c	7	1	8	16
8d	1	0	1	1
9	0	0	0	0
10a	11	7	18	56
10b	0	0	0	0
11a	1	0	1	2
11b	0	0	0	0
Totals	129	43	172	439

TABLE IX—MEMBERSHIP AND REGISTRATION

Country	Members		Ladies		Grand total	
	Total	Reg.	Total	Reg.	Total	Reg.
Africa	9	1	...	...	9	1
Argentina	81	0	...	...	81	0
Australia	6	4	1	1	7	5
Austria	113	27	8	8	121	35
Belgium	16	5	2	2	18	7
Brazil	4	2	...	...	4	2
Bulgaria	1	1	...	...	1	1
Canada	83	38	6	6	89	44
Chile	21	2	1	1	22	3
China	2	2	...	...	2	2
Cuba	6	1	1	1	7	2
Denmark	5	5	...	...	5	5
Dutch E. Indies	1	0	...	...	1	0
France	185	21	5	4	190	25
Germany	219	177	31	22	250	199
Great Britain and Ireland	99	46	13	13	112	59
Greece	23	0	...	...	23	0
Hungary	24	8	...	...	24	8
India	4	0	...	...	4	0
Italy	43	9	...	...	43	9
Japan	21	13	...	...	21	13
Malay States	1	0	...	...	1	0
Mexico	6	2	...	...	6	2
New Zealand	2	1	...	...	2	1
Netherlands	16	7	...	...	16	7
Norway	26	4	1	1	27	5
Paraguay	1	0	...	...	1	0
Peru	1	0	...	...	1	0
Portugal	15	3	1	1	16	4
Russia	56	27	...	...	56	27
Spain	23	1	...	...	23	1
Sweden	6	2	...	...	6	2
Switzerland	4	1	...	...	4	1
Turkey	1	1	...	...	1	1
West Indies	2	0	...	...	2	0
United States	3037	1472	207	186	3244	1658
Totals	4163	1883	277	246	4440	2129

REPORTS TO THE CONGRESS

Of the eighteen reports from Committees or Commissions that should have been made to the Eighth Congress only two were received and these were ordered printed, which has been done.

MEMBERSHIP AND REGISTRATION

The total membership in the Congress was 4,163 members from 36 different countries and 277 lady members from 12 different countries, the grand total membership being 4440 from 36 different countries.

The attendance was 1883 members from 27 different countries and 246 ladies from 12 different countries, the grand total attendance being 2129 from 27 countries.

FACTORY INSPECTION AND WORKS VISITS

Members of the Congress in parties of larger or smaller number and all of them in charge of the Committee on Excursions visited and inspected 146 different private chemical manufacturing plants in 24 different cities; this, in addition to a large number of private manufacturing plants of general interest as well as many public, municipal, State and Federal institutions operating along chemical lines; they further visited many public institutions and undertakings in those 24 cities and in 13 other cities, where they were officially received and entertained.

In addition to these official parties many members visited such private chemical plants both before and after the Congress but of these visits no record is here made.

This report cannot be closed without bringing to the notice of the Congress the thorough, complete and most loyal coöperation of the Rumford Press in executing the difficult and trying work of printing the Report of this Congress under exacting and harassing conditions, of Mr. Thomas A. Edison and his entire staff in originating a suitable device for recording discussions and of Miss C. E. Davidson in organizing a corps of transcribers and stenographers to reproduce these recorded discussions in typewriting. The assistance derived from all of these was of the utmost value to the Congress and far beyond any monetary consideration involved. Any shortcomings in the work of these are not due to any faults or omissions of theirs or of their aids, but are due substantially entirely to the failure of members of this Congress to do their share properly and promptly.

Respectfully submitted,

BERNHARD C. HESSE, *Secretary*

NEW YORK, MARCH 31, 1913

FINAL SHIPMENT OF EIGHTH INTERNATIONAL CONGRESS REPORTS, VOLUMES 25–29

The shipment of the last five volumes of the Proceedings of Eighth International Congress of Applied Chemistry began in March and is proceeding as rapidly as possible. This involves the sending of 4395 packages to 3036 different addresses in the United States, the total weight being 80,650 pounds. In addition to this, 37,000 pounds go abroad.

Shipping is being done by express *collect*. Each member will receive a letter of advice from the Rumford Press together with the express company's receipt. This receipt is to be used by the members and their local express agents to trace delayed or lost shipments.

NOTES AND CORRESPONDENCE

AN APPLICATION OF THE ELECTRIC RESISTANCE FURNACE TO THE DETERMINATION OF OXYGEN IN IRON AND STEEL. A CRITICISM

Editor of the Journal of Industrial and Engineering Chemistry:

In THIS JOURNAL, 5, 123, appears an article on the application of the electric resistance furnace to the determination of oxygen in iron and steel.

The writer's[1] modification of the Ledebur method is referred to, and the double electric furnace as well as certain other modifications are proposed as improvements.

The laboratories under the writer's supervision have had probably a larger and wider experience with the determination of oxygen in iron and steel than any other in this country. In our opinion the modifications proposed by McMillen present no improvements over the process used by us, and are open to criticism in respect to several important points. In a routine laboratory where a large number of oxygen determinations have to be made, the time element is of great importance. With the gas blast furnace, using a silica, tube it is possible to cool down immediately by turning on the cold air blast, so that accurate results can be obtained in one-half hour. The double electric furnace modification runs the test for a full hour, and if the analysis is started with a cold furnace, it will require an hour to reach the specified temperature of 950° C., whereas the gas blast furnace can be brought to temperature in a few minutes. In our practice we often exceed this temperature and have never found that we obtain higher results with the higher temperatures.

McMillen proposes the use of a porcelain boat instead of the nickel boat used by us. We especially avoided the use of porcelain, owing to the tendency of that material to adsorb moisture from the air.

The most important criticism of McMillen's modification that we have to offer is with respect to weighing the phosphoric acid absorption tube filled with hydrogen. We believe this to be bad practice, and numerous tests made in this manner have shown discordant results owing to the tendency of hydrogen to diffuse through "air tight" stoppers.

The McMillen method is almost identical with the one published by the writer, with the exception of the points discussed above, and for the reasons stated it does not appear that any contribution has been made to the older method. McMillen again points out the well-known fact that the Ledebur method will not determine total oxygen, owing to the fact that certain oxides and silicates are not reducible by hydrogen. Walker and Patrick[2] have proposed a method for determination of total oxygen, by making use of a vacuum electric furnace of the Arsem type. The installation for this method is very expensive and at best it could hardly become a routine determination in any chemical laboratory. On the other hand, it should not be forgotten that in most cases it is only the determination of oxygen combined with iron that yields the data desired, and it is this information which can be readily obtained by the Ledebur method. The amount of oxygen combined with silica and other oxides unreducible by hydrogen has no bearing on the question as to whether iron or steel is properly deoxidized in the process of manufacture.

ALLERTON S. CUSHMAN

INSTITUTE OF INDUSTRIAL RESEARCH
WASHINGTON, FEB. 26, 1913

[1] THIS JOURNAL, 3, 372.
[2] *Ibid.*, 4, 799.

AN ACCURATE VISCOSIMETER WITH PRACTICAL QUALITIES

Editor of the Journal of Industrial and Engineering Chemistry:

The statement of W. F. Faragher concerning "A Standard Viscosimeter" in THIS JOURNAL, 5, 167, is, unfortunately, too true to arouse the least hint of denial. Yet we can hardly expect to obtain consistent results with viscosimeters many of which, stripped of all external equipage, are fundamentally dependent on little more than a hole in a can. This statement, which perhaps may seem altogether facetious, will be verified by a glance at the Engler, Redwood and Saybolt types and others in common use.

Where then can we get an instrument of less faulty construction for our standard? Is it necessary to turn to Germany? Let us first look over what our own chemists have to offer. In 1894 the English chemists Thorpe and Rodger published a paper[1] describing a viscosimeter which introduced the foundation principle involved in a very neat and accurate instrument developed later by Bingham and White.[2] As used by them, however, it was scarcely an implement for practical industrial work until further simplified by White. In an article on the viscosities of blood and blood serum, which appeared in the *Biochemische Zeitschrift*, 37, 482 (1911), the latter gives a detailed description of his viscosimeter, the main points of which are noted below. The instrument consists of two vertical limbs of glass tubing fused into the ends of a horizontal capillary tube of about 0.1 millimeter inside diameter and of length depending upon the viscosity of the substance used; *i. e.*, varying from about 10 centimeters for liquids of small viscosity like water to 2–3 centimeters for the more viscous oils. The limbs are marked so that, by falling from the higher to the lower mark, 3 or 4 cc. of a liquid have passed through the capillary. The volume of the tube between these marks is carefully determined before fusing in the limbs. One limb is also furnished with a trap by means of which the volume used can be regulated. The fluid is forced through the capillary under a known pressure and the viscosity calculated by means of the Poisseuille formula corrected for kinetic energy as shown:

$$\eta = \frac{\pi r^4 pt}{8\,vl} - \frac{vd}{8\pi tl}$$

The volume (v) having been determined as indicated above, the constants $\frac{\pi r^4}{8vl}$ and $\frac{v}{8\pi l}$ are easily obtained by calibrating with a liquid of known viscosity, such as water. The pressure is measured with a manometer and the temperature regulated by immersing the instrument in a bath.

With regard to the accuracy of the instrument, a few data will serve better than words of praise. The following is taken from work done on fish and vegetable oils at Wood's Hole by White and Thomas:[3]

VISCOSITY OF DOGFISH OILS

Temperature Degrees	Right limb	Left limb	Average
30	0.4143	0.4140	0.4142
50	0.2113	0.2110	0.2112
70	0.1247	0.1247	0.1247
90	0.08046	0.08063	0.08055

In order to show that accuracy is attainable not by the inventor only, there are noted below a few results obtained by the writer working on butter fat with an instrument of this style.

Having demonstrated the consistency of results obtained, it remains to be seen whether there are enough points in favor of

[1] *Phil. Trans.*, **185**, 397 (1894).
[2] *Z. physik. Chem.*, **80** 670 (1912).
[3] THIS JOURNAL, 4, 378 (1912).

VISCOSITY OF BUTTER FAT

Temperature Degrees	Right limb	Left limb	Average
40	0.3200	0.3203	0.3202
60	0.1643	0.1644	0.1644
80	0.09682	0.09681	0.09682
90	0.07749	0.07759	0.07754

this type to warrant its being considered for adoption as a standard. What are its good features? In the first place it is easily made, the mediocre skill of the ordinary chemist in glass-blowing and a few hours practice being all that is required. It is convenient to handle; nor is it so fragile as to require great care in manipulating. As mentioned above, it is readily calibrated without depending upon the difficult method of actual measurement of bore and length of the capillary. The temperature is easily regulated. The apparatus required can be set up in any laboratory with the materials at hand. Only a few minutes are required for a run and as many runs as desired may be taken by simply changing the pressure from one limb to the other, taking the time as the liquid flows each way. It is by no means of little importance that only 7 to 10 cc. of a fluid are required for any number of determinations necessary. The measurements made with different instruments exhibit none of the discrepancies which, as was pointed out in the article in the January issue, are obtained with instruments in common use. The inaccuracies of the Ostwald viscosimeter and the impracticability of its use with very viscous oils, both serious faults, are not met with. Finally the viscosities obtained are in absolute units and, of course, are comparable with the results obtained by other instruments depending on the same principles.

In conclusion then, is not this instrument, possessing all these good qualities and also adaptability to practical industrial work, worthy of careful consideration before we adopt as a standard a German implement which may prove less advantageous?

R. H. TWINING

CLARK COLLEGE
WORCESTER, MASS., FEB. 24, 1913

COURT DECISION ON THE MANUFACTURE OF CARBORUNDUM

The Circuit Court of Appeals at Philadelphia has just decided in the case of the Electric Smelting & Aluminium Company *vs.* the Carborundum Company of Niagara Falls, that the Alfred H. Cowles patent No. 319,790 is substantially a basic patent for the manufacture of carborundum. This decision is said to involve the payment of more than $250,000 by the Carborundum Company to the first-named company, which also recovered a large amount some years ago from the Pittsburgh Reduction Company for infringement of a series of electrochemical patents controlled by Mr. Cowles' interests.

NEW GEOLOGICAL SURVEY BUILDING PROVIDED

After a 26-year campaign the United States Geological Survey has received generous recognition at the hands of Congress in the authorization of an expenditure of $2,596,000 for the construction of a fireproof building of "modern office-building type of architecture." With this sum it is proposed to erect a building on ground already owned by the Government which shall accommodate, besides the Geological Survey, the Reclamation Service, the Indian Office, and the Bureau of Mines, all bureaus of the Interior Department whose work is closely related to that of the Survey and among all of which there is more or less constant coöperation.

The public buildings law, which carries the Survey item, authorizes an immediate appropriation of $596,000, the balance to be appropriated as needed in construction. While this omnibus building law is only an "authorization" measure, leaving the actual appropriation of the money to a future act, $96,000 of the amount included in the measure can be expended immediately, having been appropriated by a former Congress in connection with the purchase of the site on which the Survey building is to be erected. Plans can thus go forward at once for the construction of the new building. For the needs of the Survey and the other bureaus mentioned an up-to-date, conveniently arranged, and well-lighted building is of especial importance. Too many of Uncle Sam's great army of civil employees at Washington work in part or exclusively by artificial light, in quarters that may be compared to dungeons, a condition which is suggestive of medieval times, when the first requirement of castles was walls thick enough to resist the attacks of battering rams and catapults, or of the still more ancient period when huge, ornate pillars and columns were the fashion, regardless of the arrangements with respect to light and convenience on the inside of the building. The innovation of providing a structure of the modern office type for government "workshops" in which a maximum of high-grade output is the first consideration, such as will occupy the new building, will be welcomed.

POTASH LANDS WITHDRAWN

The President has recently approved the withdrawal of three tracts of land of the desert-basin type in California and Nevada that are believed, as the result of investigations by the United States Geological Survey, to contain valuable deposits of potassium salts and brines. The survey adds:

The constructive good faith of the Government in the withdrawal of these lands in aid of legislation is indicated by the fact that concurrently with the withdrawal the Interior Department has prepared the draft of a law intended to relieve the present chaotic situation and provide a safe and sure legal basis for the development of the deposits now known and any that may be discovered in the future. This draft has been submitted to Congress for its consideration, and its enactment will be urged in order that American potash deposits may be developed under conditions that are favorable to the producer and will at the same time protect the consumer.

THE RARE EARTH MINERALS

A recent Geological Survey bulletin gives information concerning a valuable Texas deposit of these minerals. Economic interest in them centers in their incandescence on being heated, and owing to this property they have been much sought. Thoria, beryllia, yttria, and zirconia show it in the greatest degree, but thoria and beryllia, which form the bulk of the incandescent oxides used in gas mantles, are too easily volatilized to be used in an electric glower, such as that of the Nernst lamp. Yttria and zirconia, however, will stand the necessary high temperature. Before the discovery of this deposit it was practically impossible to get sufficient yttria-bearing minerals to manufacture the lamps. The needs of the Nernst Lamp Co., which owns the deposit, require only the occasional working of the mine, and after enough yttria is obtained to supply its wants for a few months ahead, the mine is closed. But a few hundred pounds a year are extracted.

BOOK REVIEWS

Chloride of Lime in Sanitation. By ALBERT H. HOOKER. Published by John Wiley & Sons. pp. 231. Price, $3.00.

The relation of sanitation to public health especially under the ever-increasing congestion of city life is of such importance that the appearance of this book is fully warranted.

The author, Mr. Albert H. Hooker, of the Hooker Electro-

chemical Company, has brought together the data collected by the Research Laboratory of the Company on the use of Chloride of Lime in Sanitation. The subject matter is extremely well arranged, the first part of the work being divided into chapters dealing with the purification of water, sewage treatment, street sprinkling and all the various problems of sanitation. The greater part of the book is devoted to over four hundred abstracts and references from the different engineering, municipal and sanitary journals dealing with the various sanitary problems.

Mr. Hooker is to be congratulated on having produced a volume of so much real value; while the book will be of greatest use to those in charge of work connected with the public health, it will be read with interest and profit by all.

OTTO KRESS

Water, Its Purification and Use in the Industries. By WILLIAM WALLACE CHRISTIE, Consulting Engineer. Published by D. Van Nostrand Company, New York, 1912. 8vo, x + pp. 219, 171 Illustrations, 3 Colored Plates. Price, $2.00.

The author is already well known to steam power engineers through his books on "Chimney Formulae and Tables," "Chimney Design and Theory," "Boiler Waters" and "Furnace Draft." In this later work he has produced a valuable reference book on water purification for the industries.

The descriptions and illustrations relating to the various water softening processes are especially clear and complete and the colored plates which relate to this subject serve greatly to assist in an understanding of the devices used in industrial water treatment.

The first chapter gives a description of the sources of water and the special properties required in numerous industries, such as concrete construction, brewing, dyeing and bleaching, ice-making, laundry work, soap and paper manufacture, steaming and sugar refining. Then follow three chapters on the various cold and hot processes of water softening and the results accomplished by such treatment.

Chapter V is devoted to a short description of pressure filters and Chapter VI to sterilization, distillation and aeration. Then follows a brief description of slow sand and open mechanical filters. The three final chapters are on the measurement of water, the effect of oil in boiler water and useful information relating to water measurement and treatment.

If the title of the book were "The Purification of Water for Use in the Industries" it would more nearly represent the character of its contents as an adequate consideration of municipal water purification is hardly the intention of the author.

Mr. Christie has given us a most valuable reference work on the treatment of water supply for industrial use and one which will meet the needs of those who desire to get the best results from water used for steaming or other manufacturing purposes.

D. D. JACKSON

The Chemical Analysis of Lead and its Compounds. By JOHN A. SCHAEFFER, A.M., Ph.D., Chief of Laboratory and Director of Research, AND BERNARD S. WHITE, Assistant Chemist. Octavo, pp. 63. No illustrations. Published by the Picher Lead Co., Joplin, Mo., for gratuitous distribution.

This little book describes methods for the analysis of lead ores, valuable merchantable furnace products, oxides of lead, basic carbonate of lead, pig lead, etc. The reading of this book indicates that it is designed more for the use of untrained chemists. The preface refers to the demand for "extreme accuracy coupled with rapidity of manipulation." It is doubtful whether the methods described in most cases comply with these requirements.

In the calculations much detail is given, but unfortunately without much regard for algebra or form of expression. Thus, on page 27 we find these two calculations:

112 : 685 :: 0.005 : X
X equals 3.058
112 : 239 :: 0.005 : X
X equals 1.067

Evidently there is a factor omitted.

Then on page 11 it is stated:

0.0025 equals $^1/_4$ of 0.01%

where similarly a factor is omitted.

These calculations, if necessary, surely should be complete in the presentation of all the factors.

The details for analysis are hardly as full as they should be to conform with the apparent purpose of the book. Thus, on page 10, in a volumetric determination of lead in lead chromate, the direction is given: "Dissolve the lead chromate in dilute hydrochloric acid (1 : 1), using as little of the acid as possible." Of course, the acid should be cold but this is not specified.

On page 10 where chromic acid is determined volumetrically by a ferrous ammonium sulfate solution, using a two per cent. solution of potassium ferrocyanide as an outside indicator, it would appear that this should preferably read: "potassium ferricyanide."

Some matters under dispute are stated as proven facts. Thus, on page 18, that sulfate dioxide found in basic lead sulfate "does not exist as a sulfite, but is instead an apparently occluded gas." It seems hardly probable that sulfur dioxide would exist in basic lead sulfate to the extent of a few hundredths of a per cent. as an occluded gas in the presence of over 15 per cent. of lead oxide and 5 per cent. of zinc oxide.

Also on page 19, referring to the determination of apparent density, the authors state that this density is dependent on two factors, the fineness and the density of the particles. On the question of apparent density, from rational consideration it would appear that fineness is less a factor than the condition of sub-division; that is, as the particles approach a uniform size, the apparent density decreases.

On page 43 the authors give typical analyses of basic carbonate of lead for impurities. We would question these analyses being typical as the acetic acid present is excessively high and out of all proportion to what exists in commercial white leads.

In the analysis of pig lead it is to be regretted that the author did not follow Fresenius' general method. Certain modifications of questionable advisability have been introduced. Thus, by Fresenius' method, after the pig lead is dissolved, if a clear solution is obtained or after filtering if a clear solution is not obtained, sulfuric acid is added in a graduated flask and an aliquot portion is taken and evaporated to fumes. The authors add the sulfuric acid and evaporate the whole mass to fumes. This is, to say the least, a hazardous operation, working on 200 grams of lead. Furthermore, following Fresenius' method, nothing is thrown out by the sulfuric acid but lead. It is probable that antimony and perhaps other constituents would be thrown out by following the authors' method of evaporation to fumes before removing the sulfate of lead.

This little book contains much valuable information which, undoubtedly on the printing of a new edition, can be put in somewhat better and more accurate form, as it fills a proper place in the literature of chemical analysis. We would suggest that in such future editions some more up-to-date and well-known methods of analysis be introduced, such as the determination of the true red lead or lead dioxide, as the case may be, in commercial red leads, following Walker's method which gives very accurate and satisfactory results.

G. W. THOMPSON

NEW PUBLICATIONS

By D. D. BEROLZHEIMER, Librarian, Chemists' Club, New York

Cerium Metals and Pyrophoric Alloys. By HEINRICH KELLERMANN. 8vo., pp. 116. Price, $1.25. Wilhelm Knapp, Halle. (German.)

Chemical Theory and Calculations. By F. J. WILSON AND I. M. HEILBRON. Sm. 8vo., pp. 138. Price, $1.00. D. Van Nostrand Co., New York.

Cyaniding of Silver Ores, The Chemical Actions during the. By E. KUEHN. 8vo. Price, $1.50. Wilhelm Knapp, Halle. (German.)

Directory of Chemical Industry, 1913, Universal. ANONYMOUS. 8vo., pp. 1053. Union Deutsche Verlagsgesellschaft, Stuttgart.

Directory of Chemical Manufacturers, etc., in Great Britain, Chemical Trade. ANONYMOUS. 5th Ed. 8vo. Davis Bros., London.

Fats, Lipoids and Waxes, Chemistry of the. By W. GLIKIN. Vol. II. 8vo. Price, $18.00. Gebrueder Borntraeger, Leipzig. (German.)

Flour Manufacture, Processes of. By PERCY A. AMOS. Cr. 8vo., pp. 280. Price, $1.50. Longmans, Green & Co., New York.

General Chemistry, A Course in. By WILLIAM MCPHERSON AND WILLIAM E. HENDERSON. 8vo., pp. 556. Price, $2.25. Ginn & Co., New York.

Iron Foundries and Allied Industries, Chemical Methods of Examination for. By ALBERT VITA AND CARL MASSENEZ. 8vo. Price, $1.00. Julius Springer, Berlin. (German.)

Iron Foundry Chemist, Practice of the. By CARL KRUG. 8vo. Price, $1.50. Julius Springer, Berlin. (German.)

Japanning, A Handbook on. By W. N. BROWN. 2nd Ed. Cr. 8vo., pp. 70. Price, $1.00. Scott, Greenwood & Co., London.

Measurements and Graphical Methods, Precision of. By H. M. GOODWIN. 8vo., pp. 104. Price, $1.25. McGraw, Hill Book Co., New York.

Mechanical Appliances of the Chemical and Metallurgical Industries. By OSKAR NAGEL. 2nd Ed. 8vo., pp. 300. Price, $2.25. Vacher & Sons, London.

Metals, the Rarer, Mineralogy of. By CAHEN AND WOOTON. 8vo. Price, $1.50. Chas. Griffin & Co., London.

Metal Statistics, 1912. ANONYMOUS. 8vo., pp. 255. American Market and Daily Iron and Steel Report, New York.

Organic Chemistry II. By V. v. RICHTER, R. ANSCHUETZ AND H. MEERWEIN. Vol. II, 11th Ed. Price, $7.00. Friedrich Cohen, Bonn. (German.)

Organic Compounds. By J. W. SHEPHERD. 8vo., pp. 348. Price, $1.75. W. B. Clive, London.

Organic Compounds, Chemical Technology of. By R. O. HERZOG. 8vo., pp. 732. Price, $5.50. Robert Kiepert, Berlin. (German.)

Organometallic Compounds of Zinc and Magnesium. By HENRY WREN. 8vo., pp. 100. Price, $0.50. Gurney & Jackson, London.

Oil Industries, Chemistry of the. By J. E. SOUTHCOMBE. 8vo., pp. 204. Price, $2.00. Constable & Co., London.

Paper Pulp, Dyeing of. By JULIUS ERFURT. 3rd Ed. 8vo. Price, $4.00. Carl Hofmann, Berlin. (German.)

Patents, Engineers' Handbook of. By WILLIAM MACOMBER. 8vo., pp. 288. Price, $2.50. Little, Brown & Co., Boston.

Qualitative Analysis. (Translation.) By F. P. TREADWELL AND WM. T. HALL. 3rd Ed. from 5th German Ed. L. 8vo., pp. 534. Price, $3.00. John Wiley & Sons, New York.

Sewage Disposal in the United Kingdom, A Textbook on. By H. L. CANNON. 8vo. Price, $2.00. St. Bride's Press, London.

Steels, The Special. By G. MARS. 8vo., pp. 517. Price, $4.75. Ferdinand Enke, Stuttgart. (German.)

Sugar; Investigations on Methods of Analysis of Cane Products. By WILLIAM E. CROSS. 8vo., pp. 83. Louisiana Agricultural Experiment Station Bulletin 135.

Wood Preservation: Some Facts about Treating Railroad Ties. By W. F. GOLTRA. Vol. II. 8vo. Price, $1.00. W. F. Goltra, Cleveland.

Woolen and Half-Woolen Goods, The Finishing of. By N. REISER. 2nd Ed. L. 8vo., pp. 695. Price, $6.75. A. Felix, Leipzig. (German.)

RECENT JOURNAL ARTICLES

Blower Sets, The Use of Turbo, in Water Gas Plants. ANONYMOUS. *American Gas Light Journal*, Vol. 98, 1913, No. 8, pp. 122–126.

Caoutchouc and Gutta Percha Resins. By G. H. HILLEN. *Archiv der Pharmazie*, Vol. 251, 1913, No. 2, pp. 94–121.

Cast Iron, Notes on. By ALBERT SAUVEUR. *Iron Age*, Vol. 91, 1913, No. 10, pp. 595–598.

Cement, Portland, The Constitution of. By P. H. BATES. *Cement and Engineering News*, Vol. 25, 1913, No. 2, pp. 46–48.

Coal, How to Sample. By E. G. BAILEY. *Cotton*, Vol. 77, 1913, No. 4, pp. 140–141.

Coal and its Wastes. By W. BOYD CAMPBELL. *Pulp and Paper Magazine of Canada*, Vol. 11, 1913, No. 4, pp. 131–132.

Coke Ovens, Progress in By-Product Recovery at. By J. E. CHRISTOPHER. *Journal of the Society of Chemical Industry*, Vol. 32, 1913, No. 3, pp. 115–125.

Dyestuffs and Pigments, Rapid Test for Fastness to Light of. By KURT GEBHARD. *Zeitschrift fuer angewandte Chemie*, Vol. 26, 1913, No. 11, pp. 79–80.

Food; Scientific Advancement of the Canning Industry. By R. T. MOHAN. *Journal of the Society of Chemical Industry*, Vol. 32, 1913, No. 4, pp. 167–171.

Gas, Water, Purification Material, Analysis of. By E. C. UHLIG. *American Gas Light Journal*, Vol. 98, 1913, No. 10, pp. 156–159.

Hydrochloric Acid Condensation, Rational. By THEODOR MEYER. *Zeitschrift fuer angewandte Chemie*, Vol. 26, 1913, No. 15, pp. 97–100.

Ink, New Principles for Official Testing of. By F. WILLY HINRICHSEN. *Chemiker Zeitung*, Vol. 37, 1913, No. 27, pp. 265–267.

Inorganic Chemistry and Electrochemistry in 1912, Experimental. By A. GUTBIER. *Zeitschrift fuer angewandte Chemie*, Vol. 26, 1913, No. 17, pp. 105–132.

Lubrication; Formation of Deposits of Oils in the Cylinders and Bearings of Steam and Power Machines. By H. SCHLUETER. *Chemiker Zeitung*, Vol. 37, 1913, No. 22, pp. 221–223.

Nitric Acid in the Presence of Nitrous Acid, New Method of Detecting. By W. N. IVANOW. *Chemiker Zeitung*, Vol. 37, 1913, No. 16, pp. 157.

Oxygen in Organic Compounds, The Direct Determination of. By MAITLAND C. BOSWELL. *Journal of the American Chemical Society*, Vol. 35, 1913, No. 3, pp. 284–290.

Paper Industry, Contributions to the Technology of the. By CLAYTON BEADLE AND HENRY P. STEVENS. *Journal of the Society of Chemical Industry*, Vol. 32, 1913, No. 4, pp. 174–179.

Paper Making, Notes on the Chemistry of. By THOMAS J. KEENAN. *Paper*, Vol. 10, 1913, No. 11, pp. 27–30.

Paper, New Sources of Supply for the Manufacture of. CLAYTON BEADLE AND HENRY P. STEVENS. *Journal of the Royal Society of Arts*, Vol. 51, 1913, No. 7.

Paper Straw and Strawboard, The Manufacture of. By LEO SCHLICK. *Pulp and Paper Magazine of Canada*, Vol. 11, 1913, No. 4, pp. 123–125.

Papers, Testing of Parchmentized. By V. FORTINI AND H. CECCHERELLI. *Chemiker Zeitung*, Vol. 37, 1913, No. 24, pp. 237–239.

Retorts, Some Notes on Charging and Discharging Machines for. W. L. HEALD. *American Gas Light Journal*, Vol. 98, 1913, No. 8, pp. 130–134.

Sewage, The Slate-Bed Treatment of. By W. J. DIBDIN. *Journal of the Society of Chemical Industry*, Vol. 32, 1913, No. 2, pp. 55–61.

Starches, Commercial, for Cotton Mill Purposes, The Value of. By G. M. MACNIDER. *Cotton*, Vol. 77, 1913, No. 4, pp. 158–161.

Steam Accumulators and Regenerative Processes, Theory of. By F. G. GASCHE. *Proceedings of the Engineers' Society of Western Pennsylvania*, Vol. 28, 1912, No. 9, pp. 723–794.

Steam, Exhaust, in the Brewery, Utilization of. By WM. CLASMANN. *Journal of the American Society of Brewing Technology*, Vol. 3, 1913, Nos. 3 and 4, pp. 83–107.

Steel Making, Titanium as Used in. By E. F. LAKE. *Metallurgical and Chemical Engineering*, Vol. 11, 1913, No. 3, pp. 144–146.

Soil Analysis. By W. HARRISON MARTINDALE. *Chemist and Druggist*, Vol. 82, 1913, No. 1726, pp. 67–69.

Sugar Cane; Recent Work on Dry Substance Determination. By WM. E. CROSS. *Louisiana Planter and Sugar Manufacturer*, Vol. 50, 1913, No. 10, pp. 158–159.

Sugar in Molasses, A Modification of the Clerget Method of Determining. By WM. E. CROSS AND W. G. TAGGART. *Louisiana Planter and Sugar Manufacturer*, Vol. 50, 1913, No. 9, pp. 142–144.

Sugar, Manufacture of Raw, in the Philippine and Hawaiian Islands By C. A. BROWNE. *School of Mines Quarterly*, Vol. 34, 1913, No. 2, pp. 119–136.

Sulfur Trioxide in Sulfur Furnace Gases, Determination of. By ERICH RICHTER. *Zeitschrift fuer angewandte Chemie*, Vol. 26, 1913, No. 17, pp. 132–134.

Water; The Determination of Moisture. By G. N. HUNTLEY AND J. H. COSTE. *Journal of the Society of Chemical Industry*, Vol. 32, 1913, No. 2, pp. 62–66.

Water; The Determination of Moisture in Organic Substances. By F. H. CAMPBELL. *Journal of the Society of Chemical Industry*, Vol. 32, 1913, No. 2, pp. 67–70.

RECENT INVENTIONS

By C. L. Parker, Solicitor of Chemical Patents, McGill Building, Washington, D. C.

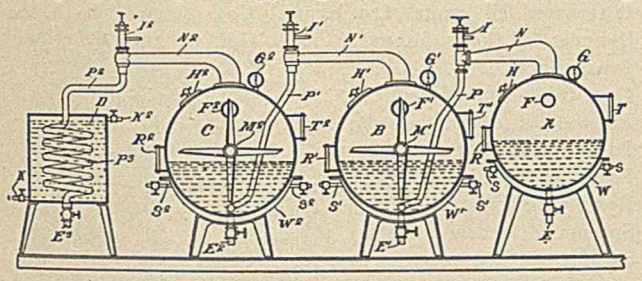
Solidified Hydrogen Peroxid and Process of Forming the Same. F. E. Stockelbach, Feb. 4, 1913. U. S. Pat. 1,051,926. A solid compound of hydrogen peroxide is obtained by dissolving in a solution of the peroxide, a small quantity of acetanilid and a large quantity of urea and then evaporating the solution.

Method of Determining the Content of Butter-Fat in Butter. R. H. Shaw, Feb. 4, 1913. U. S. Pat. 1,052,098. Hot water is added to the butter under examination and the mixture centrifuged. The aqueous solution is drawn off and while the residue is still hot a mixture of equal parts of sulfuric acid and water is added and the mixture centrifuged. The acid solution is drawn off and the residue again centrifuged and weighed.

Process of Refining Sugar. Hermann Wiese, U. S. Pat. 1,052,113. Lime is added to a hot water solution (above 70° C.) of raw sugar and the resulting mass carbonated until substantially neutral. The sugar solution is then reheated to above 70° C. in the presence of carbonate of lime and filtered.

Manufacture of Acetate of Lime and Wood Alcohol. C. W. Volney, Feb. 4, 1913. U. S. Pat. 1,052,446. By this method it is possible to produce in one operation, a comparatively pure acetate of lime and a comparatively pure wood alcohol.

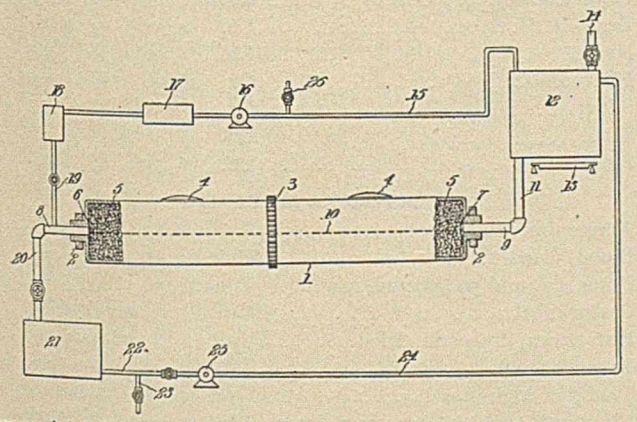
In practice, vapor of pyroligneous acid is passed through



calcium carbonate held in suspension in boiling water.

The vapors arising from the water are then passed through water holding calcium hydrate in suspension and condensed.

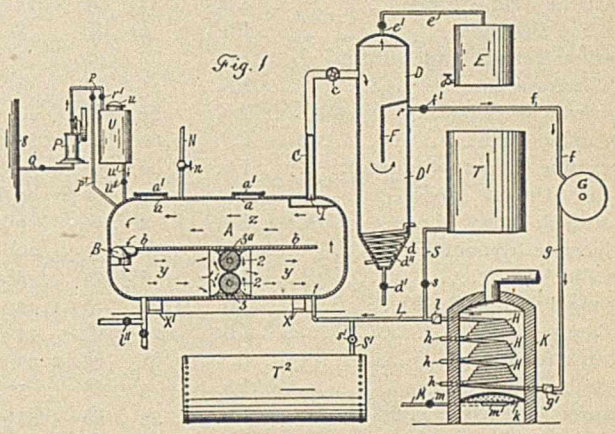
Process of Making Edible Oils. Carleton Ellis, Feb. 11, 1913. U. S. Pat. 1,052,469. Materials containing unsaturated bodies are subjected to the action of hydrogen in the presence of a porous mass of catalytic material, a traveling stream of such



material being passed through a porous mass of catalytic material, moving substantially transversely to the direction of such stream, and simultaneously contacted with such material.

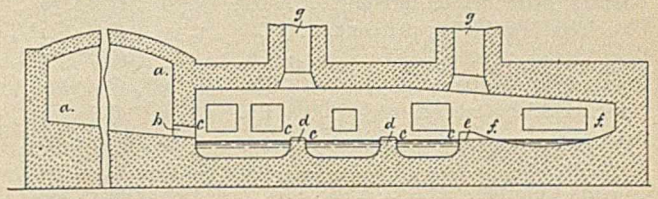
In the accompanying illustration showing apparatus in which the process is practiced, a portion of the barrel containing catalytic material is cut away to show the location of the catalyzer.

Process of Making Cellulose. Burdett Loomis, Feb. 11, 1913. U. S. Pat. 1,052,675. In this process, the woody or vegetable fibrous material in a closed chamber is subjected to the action of hot water circulated in contact with it and the softened ma-



terial subjected to the action of a beating drum in the same chamber. The circulating water which contains the gummy and resinous material is drawn off from time to time and the latter separated and collected, the circulation of water and the beating and grinding operation being continued until the material is reduced to cellulose.

Manufacture of Salt. John H. Webster, Feb. 11, 1913. U. S. Pat. 1,052,703. By this process granulated salt is produced directly from fused or melted salt without first having to allow it to solidify into blocks, and subsequently break and crush these blocks into grains of the size required.



In practice, the salt is melted, and while in its molten state it is agitated, or broken up, by mechanical means, while in a body, and it is cooled and solidified while being agitated. The grains of salt thus produced, in most cases, vary in size; and the different sizes of grains or crystals are separated according to requirements by screening.

Process of Treating Ores Preparatory to Magnetic Separation. J. B. Etherington, Feb. 18, 1913. U. S. Pat. 1,053,486. In this process ore containing non-magnetic iron sulfids is subjected to heat in a non-oxidizing atmosphere until the iron sulfids become magnetic and the mass is removed from the action of the heat before the iron sulfids are converted into oxids.

Process for the Reduction of Stannic Oxid. Zdenko Metzl, Feb. 18, 1913. U. S. Pat. 1,053,624. Tin oxid is reduced to metallic tin by reacting upon the oxid with metallic zinc at a temperature at least as high as the melting point of metallic zinc, then volatilizing the zinc oxid and finally subjecting it to the action of a reducing agent to reduce it to metallic zinc.

MARKET REPORT

AVERAGE WHOLESALE PRICES OF STANDARD CHEMICALS, ETC., FOR THE MONTH OF MARCH, 1913

ORGANIC CHEMICALS

Acetanilid	Lb.	21	@	23
Acetic Acid (28 per cent.)	C.	2.00	@	2.15
Acetone (drums)	Lb.	16 1/2	@	17 1/2
Alcohol, denatured (180 proof)	Gal.	37	@	39
Alcohol, grain (188 proof)	Gal.	2.50	@	2.55
Alcohol, wood (95 per cent.)	Gal.	49	@	52
Amyl Acetate	Gal.	2.35	@	2.55
Aniline Oil	Lb.	10 1/8	@	10 5/8
Benzoic Acid	Lb.	23	@	27
Camphor (refined in bulk)	Lb.	42 1/2	@	44
Carbolic Acid (drums)	Lb.	13	@	15
Carbon Bisulfide	Lb.	6 1/2	@	8
Carbon Tetrachloride (drums)	Lb.	8	@	8 1/2
Chloroform	Lb.	25	@	35
Citric Acid (domestic), crystals	Lb.	40	@	40 1/2
Dextrine (corn)	C.	2.60	@	2.81
Dextrine (imported potato)	Lb.	6	@	7
Ether (U. S. P., 1900)	Lb.	14	@	20
Formaldehyde	Lb.	8 1/2	@	9 1/2
Glycerine (dynamite)	Lb.	19 3/4	@	20
Oxalic Acid	Lb.	8 1/4	@	8 1/2
Pyrogallic Acid (bulk)	Lb.	1.20	@	1.40
Salicylic Acid	Lb.	29	@	31
Starch (corn)	C.	1.82	@	2.27
Starch (potato)	Lb.	5 1/4	@	5 3/4
Tannic Acid (commercial)	Lb.	35	@	35 1/2
Tartaric Acid, crystals	Lb.	30 1/4	@	31

INORGANIC CHEMICALS

Acetate of Lead (brown, broken)	Lb.	7 3/4	@	8
Acetate of Lime (gray)	C.	2.50	@	2.60
Alum (lump)	C.	1.75	@	2.00
Aluminum Sulfate	C.	90	@	1.75
Ammonium Carbonate, domestic	Lb.	8	@	8 1/2
Ammonium Chloride, gray	Lb.	6 1/4	@	6 1/2
Aqua Ammonia (drums) 16°	Lb.	2 1/4	@	2 1/2
Arsenic, white	Lb.	4 3/8	@	5
Barium Chloride	C.	1.70	@	1.80
Barium Nitrate	Lb.	5	@	5 1/4
Barytes (prime white, foreign)	Ton	18.50	@	22.50
Bleaching Powder (35 per cent.)	C.	1.35	@	1 1/2
Blue Vitriol	Lb.	5 1/4	@	5 1/2
Borax, crystals (bags)	Lb.	3 3/4	@	4 1/4
Boric Acid, crystals (powd.)	Lb.	7	@	7 1/2
Brimstone (crude, domestic)	Ton	22.00	@	22.50
Bromine, bulk	Lb.	30	@	35
Calcium Chloride	C.	65	@	90
Chalk (light precipitated)	Lb.	4	@	5
China Clay (imported)	Ton	11.50	@	18.00
Feldspar	Ton	7.00	@	9.00
Fuller's Earth, powdered	C.	80	@	85
Green Vitriol (bulk)	C.	55	@	60
Hydrochloric Acid (18°)	C.	1.15	@	1.55
Iodine (resublimed)	Lb.	3.05	@	3.10
Lead Nitrate	Lb.	8 1/8	@	8 1/4
Lithium Carbonate	Lb.	65	@	70
Magnesite (raw)	Ton	7.50	@	8.50
Nitric Acid, 36°	Lb.	3 7/8	@	4 1/4
Phosphorus	Lb.	35	@	90
Phosphoric Acid, sp. gr. 1.75	Lb.	21 1/2	@	25 1/2
Plaster of Paris	Bbl.	1.50	@	1.70
Potassium Bichromate, 50°	Lb.	6 7/8	@	7
Potassium Bromide	Lb.	39	@	40
Potassium Chlorate, crystals	Lb.	9	@	10 1/2
Potassium Cyanide (bulk) 98–99%	Lb.	19	@	24
Potassium Iodide (bulk)	Lb.	2.60	@	2.65
Potassium Nitrate (crude)	Lb.	5	@	—
Potassium Permanganate (bulk)	Lb.	9 3/4	@	11
Quicksilver, Flask		39.00	@	40.00
Salt Cake (glass-makers')	C.	55	@	65
Silver Nitrate	Oz.	36 3/4	@	38 3/4
Soapstone in bags	Ton	10.00	@	12.00
Sodium Acetate	Lb.	4	@	4 1/2
Sodium Bicarbonate (English)	Lb.	2 3/4	@	3
Sodium Bichromate	Lb.	5	@	5 1/2
Sodium Chlorate	Lb.	8 1/4	@	9 1/2
Sodium Hydroxide, 60 per cent.	C.	1.60	@	1.65
Sodium Hyposulfite	C.	1.30	@	1.60
Sodium Nitrate, 95 per cent., spot	C.	2.62 1/2	@	—
Sodium Silicate (liquid)	C.	65	@	1.50
Strontium Nitrate	Lb.	6 7/8	@	7 5/8
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.	14 1/4	@	14 1/2
Tin Oxide	Lb.	51	@	53
Zinc Chloride (granulated)	Lb.	4 1/2	@	5
Zinc Sulfate	Lb.	2 1/2	@	2 3/4

OILS, WAXES, ETC.

Beeswax (pure white)	Lb.	42	@	45
Black Mineral Oil, 29 gravity	Gal.	13 1/2	@	14
Castor Oil (No. 3)	Lb.	9 1/2	@	10 1/2
Ceresin (yellow)	Lb.	12	@	22
Corn Oil	C.	5.80	@	5.85
Cottonseed Oil (crude), f. o. b. mill	Gal.	39	@	40
Cylinder Oil (light, filtered)	Gal.	21 1/2	@	32
Japan Wax	Lb.	9 3/4	@	10
Lard Oil (prime winter)	Gal.	90	@	95
Linseed Oil (double-boiled)	Gal.	46	@	48
Paraffine (crude 120 & 122 m. p.)	Lb.	3 1/2	@	3 3/4
Paraffine Oil (high viscosity)	Gal.	26	@	28
Rosin Oil (first run)	Gal.	36	@	38
Sperm Oil (bleached winter), 38°	Gal.	72	@	74
Spindle Oil, No. 200	Gal.	18	@	19
Stearic Acid (double-pressed)	Lb.	9 1/4	@	9 3/4
Tallow (acidless)	Gal.	64	@	67
Tar Oil (distilled)	Gal.	30	@	31

METALS

Aluminum (No. 1 ingots)	Lb.	25 1/2	@	26
Antimony (Hallet's)	Lb.	8 5/8	@	9
Bismuth (New York)	Lb.	2.10	@	2.15
Copper (electrolytic)	Lb.	14 7/8	@	15
Copper (lake)	Lb.	15	@	15 1/8
Lead, N. Y.	Lb.	4.35	@	4.40
Nickel	Lb.	50	@	55
Platinum (refined)	Oz.	46.00	@	—
Silver	Oz.	56 7/8	@	57
Tin	Lb.	46 1/8	@	—
Zinc	Lb.	6.35	@	6.40

FERTILIZER MATERIALS

Ammonium Sulfate	C.	3.23	@	3.25
Blood, dried	Unit	2.85	@	—
Bone, 4 1/2 and 50, ground, raw	Ton	28.50	@	—
Castor meal	Unit		nominal	
Fish Scrap, domestic, dried	Unit		nominal	
Mowrah meal	Ton	9.00	@	—
Tankage, high-grade	Unit	2.75	&	10
Phosphate, acid, 16 per cent. bulk	Ton	7.00	@	8.00
Phosphate rock; f. o. b. mine:				
Florida land pebble, 68 per cent.	Ton	3.00	@	3.25
Tennessee, 70–80 per cent.	Ton	5.00	@	5.50
Potassium, "muriate," basis 80%	Ton	38.55	@	—
Pyrites, furnace size, imported	Unit		0.13 1/2	