

# THE JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY

VOL. I.

JUNE, 1909.

No. 6

## THE JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY

PUBLISHED BY

THE AMERICAN CHEMICAL SOCIETY.

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Published monthly. Subscription price to non-members of the American Chemical Society \$6.00 yearly.

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## EDITORIALS.

### THE DEVELOPMENT OF INTENSELY WORKING APPARATUS IN CHEMICAL ENGINEERING.

THE beginnings of the study of chemistry were various experiments which consisted in placing different substances together in the same vessel and noticing the effect produced, if any, under varying conditions, of these substances upon each other. The vessels, of course, were of different kinds and the substances were sometimes solids, sometimes liquids and later on, gases or combinations or mixtures of these. The result was in some cases found to be something of sufficient value to warrant the repeating of the experiment upon a larger scale and the development in this way of a process.

The natural form taken in this development was a simple enlargement of the apparatus with the substitution of some other material, if the material of which the original apparatus was made

would not lend itself to the construction of a larger apparatus.

Many of the first chemical industrial plants amounted to very little more than a multiplication of the dimensions of laboratory apparatus, and many are still substantially this. As more possibilities began to be discovered in the laboratory, other forms of apparatus were used and here again the industrial apparatus showed a tendency to follow very closely the form of the original laboratory apparatus.

Gradually, in many cases, it began to be seen that, by carefully studying the reactions and physical phenomena accompanying certain processes, it was possible to design very much more efficient apparatus of far greater capacity and produce more certain and better results. This was the beginning of the development of intensely working apparatus. As the apparatus becomes more intensely working, it becomes smaller, the requirements for accuracy in its construction become greater, the use of more appropriate materials in its construction becomes possible, and while sometimes at first the apparatus seems to become more complicated, generally in the end, it is discovered that the apparent complications are in reality simplifications and very great reductions in the cost and size of apparatus per unit of material passed through.

The designer of intensely working chemical apparatus, finds himself in need of knowledge of a branch of chemistry which has not been well explored. He needs to know the conditions governing the rapidity of chemical reactions. For instance, many reactions are considered to require time for their accomplishment, whereas in reality the time is but that required for thorough mixing, and for the molecules to be brought into the necessary proximity to react on one another. When this is the case, the rapidity of many reactions can often be hastened by the application of some form of energy so that the designer of intensely working apparatus must know when this can be done, and to gain this knowledge the only method, as a rule, is to himself investigate the unexplored field, and with the knowledge thus gained to design smaller



and better chemical apparatus, which are likely to be continuous-working and in which reactions which may have previously been considered unavoidably slow, take place instantly.

Is there not here a great field and are not the results to be looked for from efforts in this direction second only to results from the discovery of new substances and new forces?

FRANCIS I. DUPONT.

#### CALORIMETER STANDARDIZATION.

THE great demand for the calorific value of fuels has, within the past few years, changed its determination from a scientific to a commercial proposition. The result is that for each laboratory that had a calorimeter fifteen years ago there are probably a hundred so equipped to-day. With this extended use there are also greater discrepancies between the results obtained in different laboratories.

A prominent chemist sent portions of the same pulverized sample to ten different laboratories some time ago and received such a variety of results that his confidence in calorimetry was badly shaken.

The buying of coal on a heat unit basis has been greatly handicapped on account of the discrepancies in the results obtained in different laboratories as well as different times in the same laboratory. There is a prevalent idea that the calorific values used for practical purposes need not be as accurate as those used in scientific work, and that any type of calorimeter, kind of thermometer or method of operation will give at least comparable results. This is by no means true, and many plants that have depended upon such results as a guide in selecting the most economical coal would have been better off had they based the comparison on the proximate analysis alone.

Boiler efficiencies are often reported absurdly high or low on account of erroneous calorific determination of the fuel used.

It is in the buying of coal on a heat unit basis that the greatest accuracy is required. A man may use any kind of measuring stick he wishes so long as the results are for his own personal use, but when he buys or sells dry goods, groceries, coal or any other commodity his unit of measurement or weight must conform to fixed standards. In buying coal the weight is not of more importance than is the calorific value. The two are on the same basis and in many contracts one affects the

cost of fuel just as much as does the other. If one standard is fixed by law it is certainly unjust to allow the other to be a matter of chance. The heat unit itself is sufficiently well defined, but in a calorimeter determination what assurance has the chemist that the indicated rise in temperature is correct? Very few thermometers graduated to 1/50 or 1/100 degree can be used without corrections being applied. There is necessarily some radiation loss and the methods used in different laboratories for this correction may cause an error of 1 per cent. in the final results. The temperature of the calorimeter itself is raised as well as that of the water, and the accuracy with which its water equivalent has been determined affects every determination. In the operation of the calorimeter great skill and care are required even with a calibrated and standardized instrument. All laboratories have not taken these precautions and their results are discordant, and cause not only a great deal of controversy regarding the true value of coal, but oftentimes unjust penalties and premiums to be exacted.

There are very few contracts on a heat unit basis where 1 per cent. does not mean at least \$1,000 per year and in many of them it corresponds to as much as \$10,000. Here is where accuracy is of greater importance in practical than in scientific work, at least it is of enough importance to warrant the purchase of a good reliable calorimeter and thermometer and have the water equivalent properly determined and the thermometer carefully calibrated. The United States Bureau of Standards is prepared to do the latter at a nominal charge and they also supply pure cane sugar of known heating value that can be used in determining the water equivalent.

Accuracy is well within the reach of all chemists who care to do high-grade work, and those who continue to report inaccurate results should be considered as much criminals as the man who gives short weight.

E. G. BAILEY.

## ORIGINAL PAPERS.

### EXPERIMENTS ON THE PUTRESCIBILITY TEST FOR SEWAGE AND SEWAGE EFFLUENTS.

By D. D. JACKSON AND W. A. HORTON.

Received February 10, 1909.

A sewage or sewage effluent is putrescible if, on standing for a certain length of time, it loses



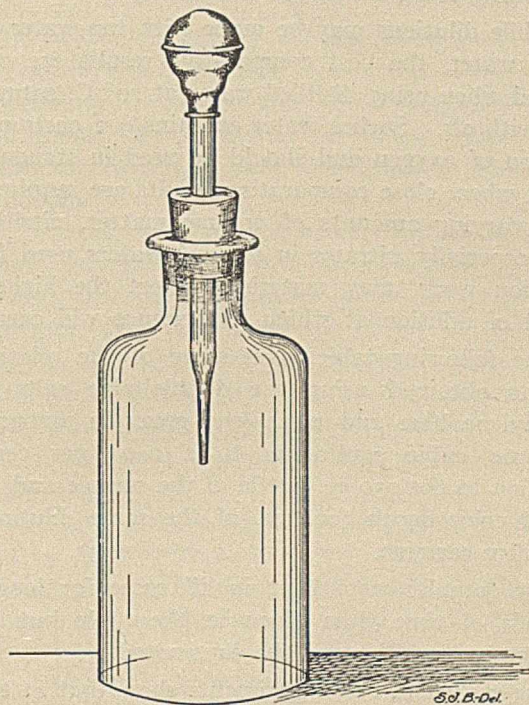
all of its available oxygen. Putrescence takes place when all the dissolved oxygen and the oxygen of the nitrates and nitrites has been used up, and the liquid still contains organic matter available for bacterial action. To determine quantitatively the amount of oxygen required to prevent putrescence, varying amounts of water, the oxygen content of which is known, can be added to the liquid to be tested till that liquid on standing is shown to contain an excess of free oxygen or oxygen in the form of nitrites or nitrates.

Many methods have been proposed and used to determine whether or not a given liquid was putrescible, and considerable variation in the results has been obtained according to the methods employed. A careful study of these methods was therefore undertaken in order to determine their relative sensitiveness, and the method which appeared to give the most concordant results. In this study a large number of sewages and sewage effluents of varying character have been tested by a series of dilutions and with different indicators.

*The Odor Method.*—In 1895 one of the authors made a long series of putrescibility tests of the sewage and sewage effluent at Worcester, Mass., under the direction of Mr. Harrison P. Eddy, at that time City Superintendent of Sewers. In these tests gallon bottles were used and varying dilutions of river water employed. By the use of these large bottles, the point of putrescibility could readily be determined by the odor. It was found that quicker and better results were obtained when the bottles were kept in a 37° incubator. A large number of bottles could not be used at one time, however, on account of the incubator space required, and the size of the bottles was reduced with a corresponding difficulty in properly determining the odor.

*Decolorization Methods.*—The methylene blue test as originally devised by Spitta<sup>1</sup> and improved by Spitta and Weldert,<sup>2</sup> removes the necessity for the use of large bottles and gives practically the same results on bottles of 250 cc. capacity, taking the decolorization as an indicator of putrescibility. By the use of this indicator the 37° incubator can be employed to advantage. The space required is small and, as has been also pointed out by Phelps and Winslow<sup>3</sup> and by Johnson, Copeland and Kimberly,<sup>4</sup> the time necessary for the test is only one-half that which would be taken if the bottles were kept at room temperature.

The only criticism of the use of the 37° incubator is in the leakage which sometimes occurs around the stoppers of the bottles, due to the expansion of the liquid when the temperature is raised, which loosens the stopper and may drive out some of the originally dissolved gas. This is occasionally followed by a backward leakage of air. The authors have experimented with various devices for preventing this leakage and have found a simple and most effective arrangement. The test is made by replacing the ground glass stopper of a 250 cc. bottle by a perforated soft rubber stopper through which is a small glass tube or medicine dropper having a heavy rubber cap of about 5 cc. capacity tightly attached to the end. The tube is drawn out like the usual medicine dropper and when in position extends into the liquid in the bottle. A measured amount of the colored indicator is placed in the bottle, which is then completely filled with the liquid to be tested. The dropper is then also completely filled with the liquid, making sure that no air bubbles are left. The tube which



DEVICE FOR  
PUTRESCIBILITY TESTS.

extends through the stopper is placed in position and the excess of water pressed out between the bulb and the top of the tube, leaving the bulb in a collapsed state.



When placed in the incubator the bulb will partially fill, due to expansion of the liquid and the giving off of some gas previously in solution. By this method the expanded water and all gas bubbles are held back and there is no loss of the original water or air in solution.

The accompanying illustration shows the rubber stoppered bottle with the tube and bulb in position. The best and most durable tube and bulb for this purpose is made by the Tyler Rubber Company, 27 Duane Street, or may be obtained from Eimer & Amend, New York City.

*A Comparison of Indicators.*—In a recent article, Clark and Adams<sup>6</sup> have compared methylene blue, methylene green, indigo carmine, and fourteen other dyes of less importance and have decided that the methylene green and indigo carmine are the most satisfactory indicators for use at a temperature of 27° C. Inasmuch as their experiments were made at that temperature instead of at blood heat, the authors decided to repeat this work, using the 37° incubator.

While dilutions may be made with tap water or river water, the best comparative results are obtained when using distilled water at 20° C. saturated with air. Such a water contains 9.2 parts per million of oxygen and should be used in standard work where close comparative results are required. The varying amounts of organic matter, nitrites, and especially nitrates in natural waters used for dilution work, may materially affect the highest point of dilution at which putrescence will occur.

The following table gives some of the relative results obtained using the double zinc salts of methylene blue and methylene green in amounts of one cubic centimeter to a 0.05 per cent. solution to the 250 cc. bottle of the sample and an equal color depth (one cc. of the same dilution) of indigo carmine.

The samples were kept in all cases for longer periods of time than given in the table but no change occurred beyond the days noted.

The results of our experiments show that at the temperature employed the indigo carmine did not always give satisfactory figures and was delayed in its action, while the methylene blue was not quite as delicate as the methylene green. There is, therefore, some reason for changing the indicator generally employed, and we would recommend the use of the methylene green as being of slightly greater delicacy.

	Days.	Methylene blue, highest dilution, putrescible.	Methylene green, highest dilution, putrescible.	Indigo carmine, highest dilution, putrescible.
Brooklyn sewage, Fulton Street	1	1-8	1-12	1-6
	2	1-13	1-13	1-11
	3	1-15	1-15	1-14
	4	1-16	1-18	1-16
	5	1-17	1-18	1-16
Bedford sewage, N. Y. Reformatory septic effluent.	1	1-5	1-5	1-2
	2	1-6	1-6	1-5
	3	1-7	1-7	1-5
	4	1-7	1-8	1-5
	5	1-8	1-8	1-5
Gowanus Canal, Brooklyn.	1	1-2	1-3	1-2
	2	1-3	1-4	1-3
	3	1-3	1-4	1-3
	4	1-4	1-5	1-4
	5	1-4	1-5	1-4
Untaminated mud from bottom of Masapequa Pond.	1	Undiluted	Undiluted	Undiluted
	2	1-1	1-1	Undiluted
	3	1-2	1-1	Undiluted
	4	1-4	1-4	1-1
	5	1-4	1-4	1-1
	6	1-4	1-4	1-1
Untaminated mud from bottom of East Meadow Pond.	1	Undiluted	Undiluted	0
	2	1-1	1-1	0
	3	1-1	1-1	Undiluted
	4	1-1	1-1	Undiluted
Jamaica, L. I. City sewage.	1	1-4	1-5	1-5
	2	1-5	1-6	1-6
	3	1-7	1-8	1-7
	4	1-7	1-8	1-7
Jamaica, L. I. Effluent from lime treatment.	1	1-1	1-2	1-1
	2	1-2	1-2	1-2
	3	1-2	1-3	1-2
	4	1-2	1-2	1-2
Bedford, N. Y. Septic tank effluent.	1	1-11	1-12	1-6
	2	1-11	1-12	1-10
	3	1-11	1-12	1-10
	4	1-11	1-12	1-10
Bedford, N. Y. Sprinkling filter effluent.	1	Undiluted	1-1	Undiluted
	2	Undiluted	1-1	Undiluted
	3	Undiluted	1-1	Undiluted
	4	Undiluted	1-1	Undiluted

Contrary to the usual belief, we found that variations in the amount of coloring matter used made noticeable differences in the point of putrescibility. The following table gives the results obtained on varying quantities of the three indicators when 0.1 per cent. solutions were employed.

It will be seen from the figures below that all three of the indicators used are slightly antiseptic so that it is best to employ the indicator in amounts as small as is possible to obtain a distinct color. These amounts are, in the case of methylene blue and methylene green, one cubic centimeter of a one-twentieth of one per cent. solution for each bottle of 250 cc. capacity, and for indigo carmine, one cubic centimeter of a one-tenth of one per cent. solution.



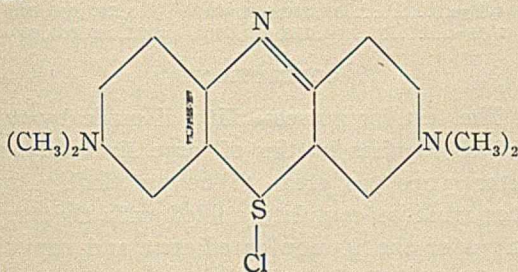
		Methylene blue, highest dilution, putrescible.	
		1 cc.	1/2 cc.
Brooklyn sewage, South Ferry	Days.		
	1	1-11	1-12
	2	1-14	1-16
	3	1-16	1-18
4	1-20	1-20	

		Methylene green, highest dilution, putrescible.	
		1 cc.	1/2 cc.
Brooklyn sewage, South Ferry	Days.		
	1	1-13	1-14
	2	1-14	1-18
	3	1-16	1-18
4	1-20	1-20	

		Indigo carmine, highest dilution, putrescible.	
		2 cc.	1 cc.
Brooklyn sewage, South Ferry	Days.		
	1	1-6	1-8
	2	1-12	1-14
	3	1-16	1-17
4	1-18	1-20	

It was thought possible that medicinal methylene blue, which is the chloride free from zinc, might be a better indicator than the common methylene blue, which is the double zinc salt. This latter salt is the coloring matter which has been generally used for the putrescibility test.

In composition the medicinal salt is the chloride of tetramethylthionine ( $C_{16}H_{18}N_3SCl$ ) with the formula:



At the same time these salts were also compared with methylene green. This latter substance is always bought as the double zinc salt, as there is at present no commercial use for the chloride. It is double zinc salt of mononitro-methylene blue.

The following are the results obtained by a comparison of these three indicators using in each case one cc. of a 0.05 per cent. solution to the 250 cc. bottle.

It will be seen from these results that the medicinal methylene blue is not as delicate an indicator as the double zinc salt or commercial variety and that the methylene green is slightly more delicate than the commercial methylene blue.

These differences are probably due to molecular construction. The zinc salts would tend to be

more easily broken down under anaerobic conditions and the methylene green would have a greater tendency to be acted upon as it is a mononitro derivative of methylene blue and for this reason may be considered to have a slightly weaker point of attack.

Days.	Methylene blue. Medicinal chloride.	Methylene blue. Commercial double zinc salt.	Methylene green. Commercial double zinc salt.	
Montefiore, home sewage, from septic tank, Bedford, N. Y.	1	1-12	1-13	1-13
	2	1-12	1-13	1-14
	3	1-13	1-14	1-14
	4	1-14	1-14	1-14
Montefiore, home sewage from effluent of sprinkling filter.	1	Undiluted	1-1	1-1
	2	1-1	1-2	1-2
	3	1-2	1-2	1-2
	4	1-2	1-2	1-4
Jamaica, L. I., city sewage.	1	1-5	1-5	1-6
	2	1-6	1-7	1-8
	3	1-8	1-9	1-9
	4	1-9	1-9	1-9
Jamaica, L. I., effluent from lime treatment.	1	Undiluted	1-1	1-2
	2	1-1	1-1	1-2
	3	1-2	1-2	1-4
	4	1-3	1-3	1-4

In treating undiluted sewages with these three indicators the methylene green was always decolorized first, then the zinc salt of methylene blue, and finally the medicinal methylene blue. This shows clearly the degrees of delicacy of the three indicators.

The indigo carmine was usually last to be acted upon, but this was not always the case. There seems to be much variation in the action of this indicator, depending upon the nature of the sewage or effluent employed. There is also great variation in the salt itself as found on the market, as it is composed of the more or less pure sodium salt of indigotine disulphonic acid or the free acid itself.

In waters containing much suspended inorganic matter, it sometimes happens that the coloring matter is precipitated or dragged down to the bottom of the bottle and the liquid above becomes colorless. Such a result should not be mistaken for putrescence, as when the bottle is shaken the indicator will become suspended and will be shown not to have been decolorized.

Some authors have criticized the indicator test as giving positive results at times when no disagreeable odor is developed. It is probable that in such cases the air is entirely used up in the bottle, but the excess of organic matter is not sufficient to develop a much further increase in bacterial ac-



tion, so that while anaerobic conditions exist, the odor does not sharply indicate the fact. In at least one case the odor of such a sample could be obtained by incubating a gallon bottle and then pouring out a portion of the liquid and shaking the bottle. In this case a strong odor became evident, which would otherwise have been undetected.

*Putrescibility and Bacterial Action.*—In this connection a series of experiments on the amount of bacterial action produced in waters and sewages of various organic contents is of interest. The following samples kept at 20° C. were examined daily for bacteria:

No. 1. Highland Spring Brewery, Boston, Mass. Well 750 feet deep.

No. 2. Highland Spring Brewery, Boston, Mass. Well 50 feet deep.

No. 3. Norfolk Brewery, Boston, Mass. Deep well.

No. 4. Burton Brewery, Boston, Mass. Deep well.

No. 5. Worcester sewage, branch sewer after heavy rain. Diluted with distilled water, one part of sewage to 100 parts of distilled water.

No. 6. Same as No. 5, only not diluted.

No. 7. Strong sewage, branch sewer, Worcester, Mass.

No. 8. Undiluted urine seeded with bacteria.

The results are given in the order of the increasing albuminoid ammonia present and the bacterial action, while not in proportion to the albuminoid ammonia, is shown to follow it in a general way, indicating that such action is in proportion to the available organic matter. If this action is sufficient to use up the oxygen in the water and reduce the nitrates and nitrites, then the water is putrescible.

The table below shows that only in the last three samples the organic matter available as food for bacteria was sufficient to produce anaerobic conditions and putrescence at the points marked by stars, while in the first five samples the organic matter present was insufficient to produce such a result.

Experiments on pure cultures showed that *B. cloacae* and *B. proteus* Zenkeri produced stronger odors than did *B. coli* and that the odor developed varied in strength, not only with the amount but with the nature of the organic matter present.

	No. 1. Albuminoid ammonia. Parts per mil. 0.002. Bacteria per cc.	No. 2. Albuminoid ammonia. Parts per mil. 0.007. Bacteria per cc.	No. 3. Albuminoid ammonia. Parts per mil. 0.009. Bacteria per cc.
Start.....	10	6	24
1 day.....	11	18	350
2 days.....	11	132	1,140
3 days.....	11	138	1,395
4 days.....	10	284	1,025
7 days.....	3	176	580

	No. 4. Albuminoid ammonia. Parts per mil. 0.014. Bacteria per cc.	No. 5. Albuminoid ammonia. Parts per mil. 0.032. Bacteria per cc.	No. 6. Albuminoid ammonia. Parts per mil. 3.200. Bacteria per cc.
Start.....	4	810	81,000
1 day.....	1,450	5,000	9,280,000
2 days.....	2,700	16,000	18,020,000
3 days.....	10,500	493,000	85,500,000*
4 days.....	3,630	5,480,000	22,100,000
7 days.....	2,820	3,613,000	1,900,000

	No. 7. Albuminoid am- monia. Parts per mil. 44.800. Bacteria per cc.	No. 8. Albuminoid am- monia. Parts per mil. 210.000. Bacteria per cc.
Start.....	250,900	35,600
1 day.....	72,540,000*	2,270,000
2 days.....	475,000,000	11,540,000
3 days.....	5,580,000,000	43,000,000*
4 days.....	19,530,000,000	3,780,000,000
7 days.....	4,278,000,000	25,110,000,000

#### CONCLUSIONS.

1. The test for putrescibility, if made by taking the odor, requires bottles of large size and an incubator space too great to be practical. As the bottles must be completely filled with the varying dilutions of the sewage or effluent and cannot be shaken except at the end of the test, a comparison of the time required to become putrescible cannot be readily made. The delicacy of the test in general is not as great as by the use of color indicators.

When the standard comparative results are required the dilutions should be made with distilled water at 20° C. saturated with air.

2. The test is best made in bottles of 250 cc. capacity, having perforated rubber stoppers, in which are inserted medicine droppers with tight rubber bulbs. This bulb compensates for any change in volume due to the expansion of the liquid and prevents loss of water or previously dissolved gas.

3. The test should be made in an incubator at 37° C., using the double zinc salt of methylene green as an indicator and the standard results

\* NOTE.—Odor developed and putrescence began.



taken at the end of four days. In the regulation of sewage filters practical comparative results may be obtained in a much shorter period. In sewage laboratories where only rough tests are required, the room temperature may be used, in which case eight days may be taken as the standard time of incubation but, as in the case of the 37° tests, practical results may be had in much shorter periods of time.

The amount of methylene green used should be one cc. of a solution containing one-half a gram per liter. Larger amounts of this, as well as of methylene blue, have a slight antiseptic action.

4. The comparison of the relative putrescibility by dilutions of sewages and effluents is a very valuable test of the various methods of sewage treatment. It not only tests the work accomplished by purification plants but gives data as to the amount of dilution necessary to prevent foul odors in the final disposal of a sewage or effluent which enters a stream or other body of water.

#### REFERENCES.

1. Spitta, 1903. Weitere Untersuchungen über Flussverreinigung. *Archiv. f. Hyg.*, XLVI, p. 64.
2. Spitta and Weldert, 1906. Indikatoren für die Beurteilung biologisch gereinigter Abwässer. Mitteilungen aus der Königlichen Prüfungsanstalt für Wasserversorgung und Abwässerbeseitigung zu Berlin, VI, p. 160.
3. Phelps and Winslow, 1906. On the Use of Methylene Blue in Testing Sewage Effluents. American Public Health Association, *Papers and Reports*, Vol. XXXII, Part II, p. 7. Also 1907 *Jour. Infect. Diseases, Supplement No. 3*, p. 1.
4. Johnson, Copeland and Kimberly, 1905. The Relative Applicability of Current Methods for the Determination of Putrescibility in Sewage. American Public Health Assoc., *Papers and Reports*, Vol. XXXI, Part II, p. 80.
5. Clark and Adams, 1908. Studies in Incubation Tests. *Journal of the American Chemical Society*, Vol. XXX, p. 1037.

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### CONTRIBUTION ON THE CHEMISTRY AND PHYSICS OF JELLY-MAKING.

By N. E. GOLDTHWAITE.  
Received February 18, 1909.

The object of these experiments has been to determine as exactly as possible the conditions necessary for the formation of fruit jellies. These conditions are doubtless well known to the manufacturers of fruit products, but diligent search of chemical literature has failed to disclose articles dealing directly with the subject in hand.

Bigelow's admirable review<sup>1</sup> of the work done on

the chemistry of pectin bodies, emphasizes the fact that as yet little has been definitely ascertained concerning the constitution and function of these compounds. The experimental results indicate in general that these pectin bodies are carbohydrates. Herzfeld<sup>1</sup> regarded them as combinations of araban and galactan, and Tollens<sup>2</sup> has suggested that they may be glucosides.

Though the constitution of these bodies is so far from being determined, yet their fundamental physical characteristic—that of forming jellies under certain conditions—has long been well known practically. Our quest in the present work is to find the conditions under which this gelatinizing power best manifests itself, *i. e.*, the conditions under which pure fruit jellies of the best texture are formed. Bigelow's arbitrary method for making jellies<sup>3</sup> by boiling together equal parts of fruit-juice (more or less diluted with water) and of cane-sugar, for 20 minutes, does not, in our experience, necessarily yield jellies of a uniformly good texture. By a jelly of good texture we mean one obtained by so treating fruit juice that the resulting mass will quiver, not flow when shaken—one which can be cut easily with a spatula, the angles so produced preserving their shape when a piece is isolated, and yet one which is not tough.

*I. Method of Procedure.*—Since the present work was begun in September, 1908, the jellies so far investigated have been made from fall fruits only. In extracting the juices for these jellies any unsound portion of the fruit was removed and the sound portion cut into small pieces if possible. The fruit so prepared was barely covered with cold distilled water (about 1 liter, or less, to 2 kgs. of prepared fruit) and the whole brought slowly to the boiling point and kept simmering until the fruit was thoroughly cooked. The mass was then removed to a filter made of a double fold of cheesecloth and allowed to drain completely. It was found that the pulp remaining on the filter could be again boiled up with water (the least amount possible) and that the resulting juice would make a good jelly. This was particularly true in the case of grapes, even the fourth and fifth extractions making an excellent quality of jelly—a quality better than that made from the first extraction because potassium acid tartrate crystals did not appear in it as is usual in the case of jelly from grapes.

<sup>1</sup> *Zts. Ver. d. Zuchr. Ind.* 41, 295, 667 (1891).

<sup>2</sup> *Ann.*, 286, 292 (1895).

<sup>3</sup> *Jour. Am. Chem. Soc.*, 23, 347. *Bureau of Chem., Bull.* 66, 50.

<sup>1</sup> *Bureau of Chem., Bull.* 94, 67-87.



Naturally, considering the nature of our problem, we followed no set method for making our jellies, but varied our methods within wide limits, *e. g.*, we varied the acidity of the fruit juice from a neutral condition to 0.669 per cent. of acid; the amount of sugar used from none to 2 volumes per 1 volume of juice; the time of boiling sugar with juice from the whole time of cooking to the last five minutes only. The total time of cooking was always limited to the time when we obtained a good jelly test, *i. e.*, when a portion of the hot liquid would jell as it was dropped from a cold metal spatula.

*II. Necessary Constituents of Jelly. (a) Pectin.*—Braconnot, who first investigated pectin, pointed out<sup>1</sup> that very palatable jellies could be produced from the alkaline pectates which he isolated from carrots by treating the latter with alkalies. He made jellies by dissolving these alkaline pectates in water, heating with sugar and then adding a small quantity of a mineral acid.

Although the jelly-maker never handles directly such alkaline pectates or even pectin itself, yet it has long been recognized that the first essential constituent of a fruit juice for making jelly is this latter body—pectin. Its presence in a given juice is readily ascertained by adding to a portion of the juice an equal volume of alcohol, shaking and cooling; a gelatinous precipitate filling a considerable portion of the total volume of the liquid indicates pectin. Good jelly-making consists in so controlling conditions as to cause this pectin to be precipitated evenly throughout the total volume of liquid allowed it. That it can be precipitated by alcohol, various metallic salts, acids and sugar was discovered by Braconnot.

*(b) Acid.*—The second essential constituent of fruit juice for making jelly is acid. This fact may be proved in a twofold manner. First, negatively, *e. g.*, when the acid of a primarily good jelly-producing juice is neutralized, no jelly can be made from the resulting juice although it contains plenty of pectin; second, positively—if a juice which contains pectin, but which in its natural condition will yield no jelly, be acidified properly, a good jelly may be produced.

Negatively, the necessity of acid in a jelly-making juice was proved as follows: A given quantity of crab-apple juice was divided into three equal portions. Of the first portion an excellent grade of jelly was produced, using an equal volume of sugar, boiling the juice 10 minutes before the addition of

the sugar and 20 minutes thereafter, *i. e.*, till the jelly-test was obtained. To the second portion of juice a sufficient quantity of N/10 NaOH was added to just neutralize the acid present, and then an attempt made to make jelly as above. Result, a dark colored thick, fruit syrup which never gave any signs of forming a jelly, but from which cane-sugar crystals were slowly deposited. To the third portion of juice sufficient N/10 NaOH was added to neutralize half the acid present, the subsequent treatment corresponding to the above. Result, a thick fruit syrup which never showed any signs of jelly formation although at the end of four months pectin could be precipitated abundantly by the addition of alcohol.

Experiments corresponding to the first and second of these cases were carried out with grape juice, with exactly analogous results. That the juices contained plenty of pectin is evident from the fact that in their natural state they easily yielded jellies.

These experimental results are summed up in Table I:

TABLE I.

Experi- ment No.	Kind of fruit.	Sp. gr. of juice.	Per cent. of acid in juice (calc. as H <sub>2</sub> SO <sub>4</sub> ).	Propor- tion of cane sugar to juice by vol.	Time of boiling.		Texture of jelly.
					Before adding sugar. Min.	After adding sugar. Min.	
5	Crab-apple	1.043	0.550	1 : 1	10	10	Excellent
6	Crab-apple	1.043	0.000	1 : 1	10	10	Syrup
7	Crab-apple	1.043	0.275	1 : 1	10	10	Syrup
126	Grape	1.025	0.313	1 : 1	30	10	Excellent
127	Grape	1.025	0.000	1 : 1	30	10	Syrup

Nos. 5 and 126 represent respectively crab-apple and grape juices in their natural condition from which excellent jellies were easily prepared. Nos. 6 and 127 represent these same juices after neutralization from neither of which could jelly be made. Though No. 7 represents only a half-neutralization of crab-apple juice yet this also refused to yield jelly. It should be noted that the low sp. gr. and acidity of the grape juice used in these experiments is due to the fact that this juice was composed of the later extractions of the pulp. On account of this low sp. gr. a considerably longer time for boiling was necessary, but for reasons shown further on, this preceded the addition of the sugar.

Positive proof concerning the necessity of acid for the formation of jelly may be cited as follows: Sweet apple, pear and peach juices contain an abundance of pectin as is evident from the alcohol test, yet under ordinary conditions it was found impossible to produce jellies from these juices.

<sup>1</sup> *Ann. de Chim. et. de phys.* [2], 28, 173 (1825); *Ibid.* [2], 30, 96.



However, addition of an organic acid to each juice so modified conditions that a very fair jelly could be made as shown in Table II:

TABLE II.

Experiment No.	Kind of fruit.	Sp. gr. of juice.	Natural % of juice (calc. as H <sub>2</sub> SO <sub>4</sub> ).	Acid-ity of juice after addition of acid.	Proportion of cane-sugar to juice by vol-ume.	Time of boiling.		Texture of jelly.
						Before add- ing sug- ar.	After add- ing sug- ar.	
80	Sweet apple	1.042	0.098	0.098	¾ : 1	10	20	Syrup
110	"	1.042	0.098	0.308	"	20	7	Fair jelly
111	"	1.042	0.098	0.395	"	20	7	Good jelly
112	"	1.042	0.098	0.552	"	20	7	Very good
117	"	1.037	0.098	0.675	"	20	10	Fair jelly
106	Pear	1.047	0.284	0.284	"	20	10	Syrup
100	"	1.047	0.284	0.405	"	20	10	Syrup
105	"	1.047	0.284	0.511	"	20	10	Good jelly
150	Peach	1.033	0.311	0.311	"	20	10	Syrup
151	"	1.033	0.311	0.575	"	20	7	Fair jelly

Examination of this table shows that the texture of these jellies was best when the acidity corresponded to about 0.5 per cent. for juices whose sp. gr. was about 1.04. It is interesting that this acidity and sp. gr. correspond in general to those of crab-apple juice, the best natural juice for jellies so far investigated in these experiments. Increasing the acidity of these juices beyond about 0.5 per cent. seemed to impair the texture of the resulting jelly, as is evident from No. 117.

III. Discussion.—It appears then that through this addition of an organic acid to a fruit juice containing naturally little acid, jellies can be made if these juices contain pectin. However, the flavor of the fruit is generally changed; the sweet apple jelly so made tasted much like that made from sour apples, the tartness of course increasing with increasing percentages of acid; the flavor of the pears, in pear jelly, was fairly well preserved, while in peach jelly the peach flavor was completely destroyed. Repeated experiments, varying the percentages of acid were made in an endeavor to preserve the peach flavor, but success was not attained in this respect, though a jelly of fair texture could be produced. From experiments testing the relative merits of tartaric and citric acids in connection with the use of one or the other to acidify a fruit juice, the balance of favor seemed to be with the former. Both texture and flavor of jellies made by its use were superior to those in which citric acid was used. Nos. 151 and 152 in Table V represent tests in which tartaric acid was the acid added, while in Nos. 153 and 154 citric acid was used. The method of making Nos. 151 and 153 corresponds, like-

wise Nos. 152 and 154. There was no question concerning the superiority of Nos. 151 and 152, *i. e.*, the jellies made by the help of tartaric acid.

That a mineral acid like hydrochloric can be substituted for an organic acid was demonstrated. While this is interesting from a chemical standpoint, it naturally would not be recommended in the preparation of fruit jellies for food.

In determining the acidity of fruit juice better end-reactions were obtained when the juice was titrated *into* the *N/10* alkali (using phenolphthalein as an indicator) rather than the reverse as is usual. In the case of grape or plum juices no indicator was necessary, but the titration was best carried out titrating the *N/10* alkali into a definite volume of the juice (well diluted) and taking as the end-reaction that point at which the pink color of the liquid just merged into green.

That no acid disappears as such in the preparation of jellies was proved by the following experiments: Crab-apple juice (100 cc. acidity = 0.532 per cent.) was made into jelly using an equal vol. of sugar. The resulting unskimmed jelly was dissolved in distilled water, diluted to 500 cc. and the acidity again determined. The acidity of this dissolved jelly was found to be 0.107 per cent., or, if calculated in terms of the original volume of juice (100 cc.) it equaled 0.535 per cent. This is identical with the original acidity (0.532 per cent.) within the limits of experimental error.

Experiments substituting lime-water and also calcium salts as a precipitant of pectin in making jellies from sweet fruit juices were carried out. However, good jellies have not so far been obtained by this method.

It was found that glycerin could be used in place of sugar in the making of crab-apple jellies. The jellies so made were of excellent texture and of fair taste and they had remarkable keeping qualities.

When glycerin and sugar were used simultaneously with peach juice, a fair jelly was produced, but from this sugar subsequently crystallized out. These crystals were remarkably large and perfect.

IV. Sugar in Jelly-making.—That so-called jellies can be produced by merely boiling down fruit-juices is well known. Naturally the flavor of such a jelly is decidedly fruity, but in texture it is very tough. The making of such jellies requires a large amount of juice, *e. g.*, we found to make one glass of such apple jelly required a volume



of juice sufficient for six glasses when made with sugar. Ordinarily, sugar is cheaper than fruit juice, so from the standpoint both of palatability and of economy (possibly also of digestibility) jellies made with sugar are preferable.

(a) *Amount of Sugar.*—The proportion of sugar to juice is a somewhat difficult one to decide. Experimental results indicate that it depends more upon the proportion of pectin present in a juice than upon any other one factor. It seems probable that in the actual practice of making jellies poor results are very likely to occur through the use of too much sugar, and this because of the relatively large quantities of water that have been used in extracting the fruit juices. Through using these relatively large quantities of water the jelly-maker is deceived regarding the actual volume of fruit juice, hence the use of an overabundance of sugar. Fruit juices which have required little water for their extraction, apparently will utilize a larger proportional amount of sugar than those that necessarily have been much diluted with water. Some of these statements will be made clearer from the following records of experiments:

TABLE III.

Experiment No.	Kind of fruit.	Sp. gr. of juice.	% of acid in juice (calc. as H <sub>2</sub> SO <sub>4</sub> ).	Volume of juice. cc.	Proportion of cane-sugar to juice by vol. time.	Time of boiling.		Amount of jelly formed. Grams.	Texture of jelly.
						Before adding sugar. Min.	After adding sugar. Min.		
1	Crab-apple <sup>1</sup>	1.0375	0.350	300	3/4 : 1	5	15	237	Tough
2	"	1.0375	0.350	300	1/2 : 1	5	12	316	Good
3	"	1.0375	0.350	300	3/4 : 1	5	10	474	Excellent
4	"	1.0375	0.350	300	1 : 1	5	9	553	Too soft
120	Grape	1.066	0.487	300	1/2 : 1	0	30	266	Tough
121	"	1.066	0.487	300	5/8 : 1	0	30	281	Fair
122	"	1.066	0.487	300	3/4 : 1	0	23	382	Good
123	"	1.066	0.487	300	1 : 1	0	20	473	Very good

Note in each of these two series of experiments that with an increasing proportional amount of sugar, the volume of jelly produced constantly increases. Note also that the texture of the grape jelly improves with increasing volume of sugar, while in the case of the crab-apple the best jelly is that in which proportion of sugar to juice is as 3/4:1; increasing this proportion of sugar impairs the texture of the jelly. An explanation of these facts concerning the texture of the jelly is suggested when we consider that we are here dealing with a grape juice of high sp. gr. and of comparatively high acidity, but with a crab-

apple juice of low sp. gr. and of low acidity, hence this grape juice is able to utilize a larger proportional amount of sugar than the crab-apple juice can.

It may also be noted here that as the proportion of sugar in grape juice increases (and consequently the volume of jelly as shown) the crystals of acid potassium tartrate naturally deposited from this jelly decreased. Possibly the increased volume of jelly from a given amount of juice accounts for this, inasmuch as this increased volume of jelly means a less concentration of the potassium acid tartrate contained in it. This behavior of grape jelly is interesting especially when taken in connection with what has been previously remarked concerning making it from other than the first juice extraction. That too great a proportion of sugar to juice (depending on the sp. gr. and acidity of the latter) impairs the texture of the jelly, is emphasized in Table IV.

TABLE IV.

Experiment No.	Kind of fruit.	Sp. gr. of juice.	% of acid in juice (calc. as H <sub>2</sub> SO <sub>4</sub> ).	Proportion of cane-sugar to juice by vol.	Time of boiling.		Texture of jelly.
					Before adding sugar. Min.	After adding sugar. Min.	
115	Sweet apple	1.037	0.675	3/4 : 1	20	15	Very good
170	"	1.037	0.675	1 : 1	20	10	Good
171	"	1.037	0.675	1 1/2 : 1	0	15	Poor
172	(1/2 of 171)	...	....	....	..	15	Poor
173	(1/2 of 171) + equal vol. of juice	...	....	....	..	20	Very good
174	Sweet apple	1.037	0.675	2 : 1	0	15	Very poor

The texture of No. 115 was very good, but increasing the proportion of sugar beyond the proportions used in this experiment resulted in a deterioration of the texture of the jelly. Nos. 171-172 and 174 are striking examples of the use of too great a proportion of sugar. Nos. 171 and 174 were a very poor quality of jelly; pectin was precipitated in lumps throughout the mass, not in a continuous, semi-firm substance as in a good jelly. Subsequent boiling of 171 (*i. e.*, 172) served only to darken the product, but not to improve its texture. However, upon the addition to 171 of an equal vol. of juice (No. 173) and upon further boiling, a jelly fully equal in texture and taste to No. 115 was produced. Evidently then, in making fruit jellies, close attention must be given to the proportion of sugar used. Too little renders the jelly tough, too much (*i. e.*, more than the proportion of pectin present in the juice will warrant) greatly impairs the texture and flavor of the jelly. This latter fact may account more than any other for jelly failures.

<sup>1</sup> Both the low sp. gr. and acidity of this crab-apple juice are explained by the fact that it was the fourth extraction of juice.



\* Hard boiling.

Experiment No.	Kind of fruit.	Sp. gr. of juice.	Natural per cent. of acid in juice (calculated as H <sub>2</sub> SO <sub>4</sub> ).	Per cent. of acid in juice as used.	Weight of juice. Grams.	Proportion of cane sugar to juice by volume.	Weight of cane-sugar. Grams.	Before adding sugar. Min.	After adding sugar. Min.	Temperature at which jelly test was apparently observed.	Sp. gr. of hot jelly.	Wt. of jelly + skimmings.	Texture of jelly.	Per cent. of cane-sugar found in fruit juice = d.	Per cent. of cane-sugar put into jelly = e.	Total per cent. of cane-sugar in jelly = d + e.	Before inversion. Saccharimeter readings.	After inversion. Saccharimeter readings.	Temp. at which saccharimeter readings were taken.	Per cent. of cane-sugar found in jelly = s.	Per cent. of cane-sugar un-inverted = s/(d+e).	Per cent. of cane-sugar inverted = 100 - s/(d+e).	Crystallization of cane-sugar at surface of jelly.
100	Pear	1.047	0.295	0.405	317	3/4:1	190.5	20	10	103.0°	1.28	340.0	Syrup	56.00	56.00	60.30	19.11	-13.39	20.0°	48.50	86.60	13.40	Slight
101	"	1.047	0.295	0.522	316	3/4:1	190.5	20	10	103.0°	1.28	338.4	Thin jelly	56.59	56.59	60.30	20.00	-13.03	21.0°	49.48	87.43	12.60	None
102	"	1.047	0.295	0.669	316	3/4:1	190.5	20	10	103.0°	1.28	344.2	Poor jelly	55.34	55.34	60.30	15.50	-12.78	20.0°	42.20	76.25	23.75	"
103	"	1.047	0.295	0.669	261	1:1	253.0	20	4	103.0°	1.30	419.5	Fair jelly	60.30	60.30	60.30	23.61	-13.47	21.0°	55.55	92.12	7.88	Much
104	"	1.047	0.295	0.669	263	1:1	253.0	0	20	103.0°	1.285	419.5	"	60.30	60.30	60.30	6.04	-12.38	22.0°	27.70	45.93	54.07	None
110	Sweet apple	1.042	0.100	0.308	312	3/4:1	190.5	20	7	103.0°	1.27	346.4	Fair jelly	7.71	55.00	62.71	24.30	-11.20	22.0°	53.30	85.00	15.00	"
111	"	1.042	0.100	0.395	314	3/4:1	190.5	20	7	103.0°	1.27	345.9	Good	7.71	55.00	62.71	21.60	-11.20	22.0°	49.30	78.45	21.55	"
112	"	1.042	0.100	0.552	315	3/4:1	190.5	20	7	102.5°	1.25	356.0	Very good	7.71	53.50	61.21	20.40	-11.16	22.0°	47.40	77.43	22.57	"
113	"	1.042	0.100	0.552	314	3/4:1	190.5	0	26	103.0°	1.26	342.0	Good	7.71	55.70	63.41	2.40	-11.00	23.0°	20.20	31.85	68.15	"
114	"	1.042	0.100	0.552	256	3/4:1	150.0	10	13	103.0°	1.27	265.0	Very good	7.71	57.00	64.71	14.00	-10.94	24.0°	37.80	58.41	41.59	"
120	Grape	1.066	0.487	0.487	316	1/2:1	95.0	0	30	104.0°	1.26	266.5	Tough	35.00	35.00	35.00	-0.30	-10.37	24.0°	15.25	43.57	56.43	Slight(?)
121	"	1.066	0.487	0.487	318	5/8:1	127.0	0	30	103.0°	1.26	281.0	Fair	45.00	45.00	45.00	+2.46	-11.15	24.0°	21.30	47.33	52.67	None
122	"	1.066	0.487	0.487	320	3/4:1	190.5	0	23	103.0°	1.26	382.0	Good	49.00	49.00	49.00	5.90	-10.64	24.0°	25.06	51.14	48.86	"
123	"	1.066	0.487	0.487	319	1:1	253.0	0	20	103.0°	1.26	473.0	Very good	53.00	53.00	53.00	9.64	-11.22	23.0°	31.56	59.54	40.46	"
124	"	1.066	0.487	0.487	298	1:1	239.0	15	4	104.0°	1.30	434.0	Very good	55.00	55.00	55.00	18.38	-12.48	23.0°	46.48	84.70	15.30	Slight.
130	Plum	1.033	0.623	0.623	254	1:1	215.0	0	27	103.0°	1.26	365.0	Good	0.0	59.00	59.00	12.60	-9.25	23.0°	33.00	56.00	44.00	None
131	"	1.033	0.623	0.623	254	1:1	215.0	10	10	103.0°	1.26	370.0	"	0.0	58.10	58.10	23.35	-9.90	23.0°	50.18	85.05	14.95	Slight
140	Medium sour apple	1.041	0.257	0.257	259	1:1	210.0	0	22	103.0°	1.27	362.0	Good	0.66	58.00	58.66	20.90	-12.10	21.0°	49.40	84.21	15.80	"
141	"	1.041	0.257	0.257	259	1:1	210.0	0	18*	103.0°	1.27	344.0	Good	0.66	61.00	61.66	20.00	-11.77	21.0°	47.50	77.03	23.00	None
142	"	1.041	0.257	0.428	259	1:1	210.0	12	13	103.0°	1.28	344.0	Very good	0.66	61.00	61.66	20.00	-12.6	21.0°	48.80	79.11	20.88	Slight
150	Peach	1.033	0.311	0.311	259	3/4:1	159.0	20	10	103.0°	1.29	253.8	Syrup	62.41	65.21	31.82	-12.18	19.0°	65.18	100.00	0.00	Some	
151	"	1.033	0.311	0.575	259	3/4:1	159.0	20	7	103.0°	1.29	263.7	Fair	60.30	61.11	26.05	-11.05	18.0°	55.63	91.03	9.00	"	
152	"	1.033	0.311	0.575	259	3/4:1	159.0	0	29	103.0°	1.28	273.1	"	57.22	60.02	12.35	-11.18	19.5°	35.05	58.41	41.59	None	
153	"	1.033	0.311	0.600	259	3/4:1	159.0	20	7	103.0°	1.28	267.7	Thin jelly	2.8	59.40	60.20	27.30	-11.71	17.5°	56.20	93.35	6.65	Some
154	"	1.033	0.311	0.600	259	3/4:1	159.0	0	32	103.0°	1.28	266.4	"	2.8	60.00	62.80	16.80	-11.60	18.0°	42.07	67.00	43.00	None

TABLE V.



(b) *Inversion of Sugar.*—It has been pointed out by Bigelow<sup>1</sup> that the amount of inversion of sugar in jellies and jams varies in general with the length of time of heating, the content of acid, and also the nature of the acid. Experimental data further substantiating these statements are tabulated in Table V.

A word of explanation concerning the data in this table should be given here.

The specific gravity of the juice in each instance was determined by means of a hydrometer. The acidity of the juice either before or after adding more acid was determined as already indicated.

The percentage of cane-sugar put into the jelly was calculated from the weight of sugar added and the total weight of jelly and skimmings obtained. Unfortunately it was necessary to weigh the jelly itself hot.

The percentage of cane-sugar found in the jelly was calculated from the direct and invert readings according to Clerget's formula  $S = 100(a-b)/(144 - t/2)$ . Polarizations were made upon normal weights of the juices and half-normal weights of the jellies, all the results being calculated to normal weights. Solutions were clarified and inverted according to the method elaborated by Sherman.<sup>2</sup>

Examination of this table shows, as we should expect, that the percentage of inversion of sugar

That the percentage of acid in the juice affects this inversion is evident from a comparison of Nos. 100-102 (note discrepancy in 101); Nos. 110-112; and No. 150 with No. 151 and No. 153. In these instances the time is generally identical.

That the nature of the acid also affects somewhat the amount of inversion is illustrated by comparison of Nos. 151-152 with Nos. 153-154, the acid added to the two former being tartaric, and to the two latter citric.

The amount of this inversion that is desirable, in order to produce jellies of good texture, is a point more difficult to decide than the proportional amount of sugar to be used. Examination of the table shows that crystallization of cane-sugar occurred more readily generally, from the jellies in which inversion was least. However, in every case, this crystallization always occurred at the surface of the jellies and in none of the cases had any care been taken to protect these jellies from the air, as is ordinarily done, by paraffining the surfaces. Hence, there was every opportunity for evaporation from the surface of these jellies and the consequent formation of crystals.<sup>1</sup> That the sugar should not be boiled sufficiently long with the juice to insure practically complete inversion, is quite probable from the following experiments, as shown in Table VI.

TABLE VI.

Experiment No.	Kind of fruit.	Sp. gr. of juice.	% of acid in juice (calc. as H <sub>2</sub> SO <sub>4</sub> ).	Wt. of juice. Grams	Sweetening constituent.	Proportion of sweetening constituent to juice by volume.	Wt. of sweetening constituent. Grams.	Time of boiling Min.		Temp at which jelly-test was apparently observed.	Sp. gr. of hot jelly.	Texture of jelly.
								Before adding sugar.	After adding sugar.			
160	Crab-apple	1.043	0.550	103	Cane sugar	¾ : 1	66.5	0	10	103°	1.27	Excellent
161	Crab-apple	1.043	0.550	103	Fructose	.....	66.5	0	10	103°	1.24	Syrup
162	Crab-apple	1.043	0.550	103	Equal wts. of { <i>d</i> -glucose and fructose	.....	{ 34.8 <i>d</i> -glucose 34.8 fructose	0	10	103°	1.26	Good (at first)
163	Crab-apple	1.043	0.550	103	Cane sugar	¾ : 1	66.5	8	2	103°	1.28	Excellent
143	Sour apple	1.041	0.257	103	Fructose	¾ : 1	62	0	20	104°	1.275	Syrup

is vastly influenced by the time the sugar and juice are boiled together, *e. g.*, compare No. 103 with 104; Nos. 112-114 with each other; No. 123 with 124; No. 130 with No. 131; No. 151 with No. 152; and No. 153 with No. 154. Note in these comparable cases that the proportional amounts of sugar and the acidity are identical.

That jellies can not be made through substituting fructose for cane-sugar is evident from Nos. 161 and 143, as only a thin syrup resulted in these cases. Corresponding experiments (not

<sup>1</sup> In other cases not cited in the table but within the writer's previous practical experience—cases in which such jellies as currant, red raspberry, and blackberry were paraffined as soon as they had set, no crystallization of cane sugar occurred even after a lapse of two years. In these cases a very small amount of inversion had probably taken place, since the sugar had been boiled with the juice not to exceed five minutes.

<sup>1</sup> Bureau of Chem., Bull. 66, p. 52.

<sup>2</sup> Organic Analysis, p. 91.



tabulated) substituting *d*-glucose for cane-sugar resulted apparently in a good firm jelly; however, *d*-glucose shortly began crystallizing from this jelly and soon could be seen throughout the mass. No. 162 in which equal weights of fructose and *d*-glucose were substituted for cane-sugar (representing a condition of complete inversion) resulted very similarly to the case in which *d*-glucose was used alone, *i. e.*, *d*-glucose crystallized out. No. 163 in which cane sugar was boiled with the juice for only two minutes finally showed evidence of cane-sugar crystals on the surface.

From all the foregoing results two things are apparently evident: First, some inversion of cane-sugar is desirable; second, an inversion approaching completeness is undesirable, see No. 162. So far as our researches now extend it seems better not to boil sugar and juice together from start to finish in jelly-making, but rather to add the sugar so that it may be boiled with the juice for a period not to exceed one-half the total time of cooking. However, we wish to investigate this point further.

Some experiments regarding excessive cooking of an over-diluted juice have been carried out. Results seem to show that the texture of the jelly may be greatly impaired in this way. This also demands further investigation.

V. *Physical Constants*.<sup>1</sup>—In connection with our work it seemed desirable to determine whether or not there is a boiling point or a definite specific gravity of the boiling jelly indicative of that condition at which a good jelly may be formed on cooling. Although it was impossible to determine these points with absolute accuracy yet reference to Tables V and VI shows a remarkable coincidence among these physical constants. Boiling points were observed by suspending a thermometer with the bulb in the boiling liquid, and specific gravities were observed by means of a hydrometer floated in the hot liquid which had been transferred with as great rapidity as possible to a glass cylinder previously heated with boiling water. That these boiling points and specific gravities coincide so nearly, even though we are dealing with different fruit-juices and very different proportions of sugar, is worthy of notice.

VI. *Artificial Jelly*.—That the essential substances for the formation of a jelly of good texture

have been indicated in the foregoing is probable from the success attending the attempts to make jelly from an artificial fruit-juice. For this purpose it was necessary to isolate pectin. Pectin was obtained from crab-apple, grape, sweet apple, and peach juices. The isolation of this pectin was a difficult matter and the methods indicated by different experimenters<sup>1</sup> were tried. The following modification of their methods was finally adopted as the one giving the best results: a given volume of the cold, filtered (previously boiled) fruit-juice was added drop by drop by means of a separatory funnel to an equal volume of alcohol, stirring constantly; the mixture was allowed to stand over night in a cold place and then the precipitated jelly-like mass of pectin was separated from the liquid by filtering off through very fine cotton cloth stretched tightly over the mouth of a 4-liter jar. The liquid was worked out of the mass of pectin by means of a spatula. Repeated re-solution and re-precipitation of the pectin improved its purity. The pectin so obtained was a very stable, nearly colorless, transparent solid, of insipid taste. Its principal reactions in solution were tried and correspond to those given by Braconnot, Fremy and others.

Jelly of excellent quality was easily made from this pectin by preparing a 1 per cent. solution of it in a 0.5 per cent. solution of tartaric acid, heating to boiling, then adding a 3/4 volume of sugar and boiling until the jelly-test was observed. The total time of boiling did not exceed 15 minutes. Jellies made as above were nearly colorless, but were excellent in texture and taste. When a few cc. of grape-juice were added to the pectin solution the color of the jellies was very pleasing. That these jellies were not made directly from fruit-juice would not be suspected from taste or texture. Jellies equally good were made in this way from the pectin from sweet apples, crab-apples, and peaches. This last fact would seem to indicate that the pectin of peaches does not differ materially, at least from a practical standpoint, from that of other jelly-making fruits.

It may be of interest to add that pectin was extracted from jelly (the jelly being previously dissolved in an equal volume of water) by the method used for extracting pectin from fruit juices. This pectin apparently corresponded in its physical and chemical characteristics with that extracted directly from fruit-juices. Good

<sup>1</sup> We understand that some work (unpublished) on these points, also on the relation between the proportion of sugar used and the amount of jelly produced, has already been done by Miss Jenny Snow, of the University of Chicago.

<sup>1</sup> Chodnew, *Ann.*, 48, 56. Fremy, *Ibid.*, 64, 383.



fruit jelly invariably gives the alcohol test for pectin. These facts would seem to indicate that pectin in the formation of jelly does not undergo any deep-seated change, but rather that the phenomenon of jelly-making is more nearly physical than chemical.

VII. Summary.—1. The essential constituents of a jelly-making fruit-juice are, first, pectin; second, acid.

2. A desirable accessory constituent is cane-sugar. Too much sugar is likely to be used in jelly-making with a consequent deterioration of the quality of the jelly. The amount of inversion preferably produced in this cane-sugar is yet undetermined.

3. Over-dilution of fruit-juice should be avoided, since this leads to the use of too much sugar; probably the extra boiling thus rendered necessary also impairs the texture of the jelly.

4. The physical constants of hot juice ready to jell on cooling are, substantially, boiling point  $103^{\circ}\text{C}$ ., and specific gravity 1.28.

5. Jelly is readily made through boiling pectin with acid, water and sugar.

6. Jelly-making seems to consist in so controlling conditions by means of acid and sugar and boiling as to cause the pectin to be precipitated in a continuous mass throughout the volume allotted to it.

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## A STUDY OF THE FATTY ACIDS OF FISH OILS.

By L. M. TOLMAN.

Received April 2, 1909.

This study was undertaken several years ago in connection with an investigation of the cod-liver oils and related fish-liver oils, part of the results of this work being published in the *Journal of the American Chemical Society*, 28, 387-395 (1906). The oils used in this study were prepared for us by the Bureau of Fisheries, Department of Commerce and Labor, so that their exact history and purity were known. The oils selected for this especial work were a sample of Norwegian cod-liver oil, American cod-liver oil, a pollock-liver oil, a hake-liver oil, a ling-liver oil, a dog-fish liver oil, a trout-liver oil and a seal blubber oil. These were chosen as they formed a series of oils representing fish closely related to the cod

fish widely separated from the cod, and a fresh water fish whose habits of food were entirely different, while the sample of seal blubber oil gave an oil from a warm-blooded animal which largely subsists upon fish. In addition, a sample of linseed oil and one of corn oil were examined in order that a comparison could be made between the highly saturated vegetable oils and the highly unsaturated fish oils. The following table (I) gives a description and analysis of the oils enumerated:

TABLE I.—DESCRIPTION AND ANALYSES OF FISH OILS.

Description of sample.	Sp. gr. $15.6^{\circ}\text{C}$ .	Index of refraction.	Iodin number.	Hexa-bromid. Per cent.	Saponification No.	Solid acids. Per cent.	Free acids. Per cent.	Unsaponifiable matter. Per cent.
2447 Cod.....	0.9265	1.4810	165.7	46.6	178.1	5.70	0.47	....
15600 Cod.....	0.9274	1.4805	168.0	34.0	188.6	15.55	0.28	0.91
14261 Pollock....	0.9245	1.4795	150.9	42.7	...	15.99	0.95	....
14304 Hake.....	0.9254	1.4812	158.2	30.3	178.1	13.58	0.56	0.76
11556 Ling.....	0.9224	1.4784	143.4	30.2	183.1	14.42	0.91	....
15604 Dog fish...	0.9220	1.4786	153.3	37.2	180.3	18.52	0.65	3.20
16780 Trout.....	0.9275	1.4801	154.8	35.1	189.4	7.01	1.69	....
15608 Seal.....	0.9246	1.4776	143.8	19.5	191.1	9.96	0.64	0.13

These oils, as will be seen from the results given in this table, do not show a very wide variation in the chemical and physical characteristics. Considering the chemical analysis alone, all of the oils would come within the limits allowed or found in our experiments for cod-liver oil, except the seal and dog fish oils, which are somewhat different in the amount of unsaponifiable matter; and and yet it would be difficult to select a more varied lot of oils as far as the source is concerned. In the study of the unsaturated acids, the halogen compounds, especially the bromin compounds, were used as the basis for the separation of the unsaturated fatty acids. After a careful consideration of the various methods, and after experimenting to a considerable extent with the oxidation with dilute permanganate, as suggested by Hazura, it was decided that the formation of the insoluble brominated acids gave more information as to the composition of the fatty acids than any other of the methods which have been applied. As the methods used in separating the saturated and unsaturated acids and the determination of the iodine numbers are in a sense empirical methods, it is necessary at least to name them, and in some cases to give the method employed in detail.

The Hanus method was used for all of the iodine absorption determinations and accounts for the fact that the results on this determination are somewhat higher than the older figures which are



based on the Hübl method, but the range of variation should not be any greater on this account, as the Hanus method gives almost uniformly slightly higher results. The iodine number of the fatty acids were all calculated on the basis that the oil would contain 95.5 per cent. of insoluble acids, which is near enough to the truth and probably more accurate than a determination would be on oils which are so easily changed by oxidation. The iodine numbers of the liquid fatty acids can be calculated, after the determination of the saturated fatty acids had been made, only a simple calculation being required. As the saturated fatty acids had, as a rule, an iodine absorption figure of from eight to ten, due to the presence of a small amount of unsaturated acids, it was always determined and corrected for in determining the true per cent. of saturated fatty acids, calculating it as oleic acid, although it may be some other one of the oleic acid series. However, the iodine numbers of all of the acids of the oleic series are so close together that it makes very little difference which is taken as the basis for the correction. This gives what has been termed the true iodine number of an oil and the results by calculation are closer to the true results than a determination would be unless the preparation of the fatty acids was so conducted that the oxidizing effects of the air were excluded in all of the operations, which is very difficult, indeed well-nigh impossible. On the other hand, the calculations depend on the determination of the iodine number of the oil itself, which can be made with great accuracy, and on the determination of the saturated fatty acids, which is satisfactory and must be employed if the liquid acids are to be prepared for direct determinations, so that the same errors will occur in either case.

The methods used for the separation and estimation of the bromine compounds were essentially those proposed by Hazura, but the mechanical details were so modified as to greatly assist in the manipulation and will be described in some detail, especially on account of the paper by Proctor and Bennet<sup>1</sup> in which they described the obstacles encountered in handling the method, and the way in which they tried to overcome these difficulties by changing the method entirely, using a different solvent. From the experience of the writer, this appears to be entirely unnecessary when using the method employed by him for the last two years on this study.

The difficulty of handling the precipitate is the chief source of trouble, due to the volatility of the solvent, and also to the peculiar gummy consistency of the precipitate, which soon clogs any kind of filter and prevents filtration. Hehner and Mitchell<sup>1</sup> tried the use of a linen filter tied over the end of a glass tube, drawing off the supernatant liquor by means of suction, but as Proctor and Bennet have shown, and any one who has tried the method finds at once, this is very unsatisfactory and inaccurate, and gives a very poor separation. In order to get around this trouble, the writer resorted to the use of the centrifuge and found that it gave quick and satisfactory results. The precipitate, being much heavier than the solvent, readily separates, and on account of its peculiar consistency, packs in the bottom of the centrifuge tube in such a manner that the supernatant liquor can be easily and accurately decanted without any loss. The determination was made in glass stoppered weighing bottles about six inches high and one inch in diameter, which had flat bottoms and weighed only about thirty grams.

The method employed in making the bromine precipitate was to dissolve the oil or fatty acid in absolute ether, acidify with acetic acid, cool in a bath of ice-water, and add an excess of bromine so as to insure complete bromination. Allow the tube to stand for half an hour in ice-water, and then place it in the centrifuge and whirl at about 1200 revolutions per minute for two to three minutes. Dry the weighing tubes, weigh the proper amount of oil or fatty acid into them, add 25 cc. of absolute ether and a drop of acetic acid, cool in ice-water to 2°-3° C., and add the bromine drop by drop until there is a considerable excess as shown by the color of the solution. The solution should be stirred constantly during the addition of the bromine, which must be added very slowly, as a large amount of heat is generated in this reaction, and the ether will boil violently if the bromine is added too rapidly. Especially is this precaution necessary when brominating the fatty acids which react very violently with the bromine. After the tubes have stood for the necessary thirty minutes, they are placed in the centrifuge in special holders, which keep them from shifting about, and whirled. Then the supernatant liquid is decanted, which can be done very completely. Before decanting, it was the practice to put the tubes back in the ice-water to cool down again so as to throw out

<sup>1</sup> *Jour. Soc. Chem. Ind.*, 25, 798 (1906).

<sup>1</sup> *Analyst*, 1898, 313.



any bromids that had been dissolved, but this was found to be practically unnecessary, as the ether solution remained clear. This shows that the precipitate does not dissolve in the time taken to centrifuge it, although the temperature rises somewhat. Ten cubic centimeters of cold ether are added and the residue in the bottom of the tube stirred with a glass rod so as to thoroughly wash out the excess of bromin. The tubes are again cooled in the bath of ice water and again whirled and decanted. Another washing like this will completely remove the excess of bromine and leave a pure white precipitate in the bottom of the tube, which is allowed to stand until the ether has evaporated, and is then dried in a water bath for thirty minutes and weighed. The bromids prepared in this way are nearly white with a slight amber color, and have a very uniform content of bromin.

The advantages of this method can easily be seen. The oil or fatty acids and the final product are weighed in the same bottle, so that there are no losses by transfer, there is no filtration with evaporation of the volatile solvent and difficulties of washing the precipitate free from the other bromids which are thrown out of the solution when the solvent evaporates; but the chief advantage is the rapidity with which the determination can be carried out which makes possible its application in technical analysis. This method can be applied to the determination of the tetrabromids as suggested by Farnsteiner, using petroleum ether as the solvent instead of sulphuric ether, but carrying out the separation in the same manner as before.

*Determination of bromin.*—The determination of bromin was made in two ways, both of which gave satisfactory results. The first was by fusion with calcium oxid and sodium carbonate by placing the material in the bottom of a small crucible, filling it with the mixture, inverting the crucible in a large crucible, and then filling the space between the walls of the two vessels with the fusion mixture. The bottom of the outside crucible is then heated. This method insures holding all of the bromin and very satisfactory results were obtained. The second method, and the one found to be most satisfactory and most rapid, was burning with sodium peroxid, as suggested by Pringsheim<sup>1</sup> in an iron crucible. This method depends on making a mixture of the material to be burned

with sodium peroxid and sugar, the proportions depending on the percentage of bromin in the material used. Instead of an iron crucible, as suggested by Pringsheim, the writer used the bomb of a Parr calorimeter, placing it in a pail of cold water and ignited the mixture with a red hot piece of iron wire. The combustion was always complete. Any bromates formed were reduced by adding a few cubic centimeters of a saturated solution of sodium sulphite to the solution before making acid with sulphuric acid. The precipitation of the silver salt of bromin is made from a solution very strongly acid with nitric acid to prevent the precipitation of silver sulphate. This method is very rapid, gives very accurate results, and was used for all of the determinations of bromin.

The amounts of fatty acids in the various bromin precipitates were calculated from the weight of the precipitate and the percentage of bromin found in them. The iodine number of these fatty acids was calculated from these results from the ratio between bromine and iodine.

The saturated fatty acids were determined by the lead salt ether method, and the iodine number was determined so that a correction could be made. This figure was generally found to be from 8 to 10, about the same as is found in olive and other vegetable oils, which throws doubt on the claim that there is present in fish oils an unsaturated fatty acid, having a lead salt insoluble in ether. The percentage of unsaturated fatty acids was calculated from the percentage of total fatty acids taken as 95, and the amount of saturated acids as determined.

The oxidation methods were practically those of Hazura, except that the oxidation was made in a more dilute solution, and this was kept at a low temperature by means of ice, as was suggested by Heyerdahl, higher yields being obtained. But even following this procedure the writer was not able to obtain any oxidation products of the higher unsaturated acids, thus confirming the results of Heyerdahl and showing that these acids do not conform to Hazura's rule of forming hydroxy acids, as do the higher unsaturated acids of the vegetable oils like linseed oil, showing that these acids are of a different series.

Table II gives the results of the study of the bromine compounds of the glycerids and of the fatty acids of the various oils studied. Results on linseed and corn oil are also included, to furnish

<sup>1</sup> *Amer. Chem. Jour.*, **31**, 386 (1904).



a comparison of the fatty acids from vegetable oils and from the fish-liver oil.

Before discussing the analytical results, an explanation of the various headings given in Table II should be made. The first column gives the iodine number by the Hanus method; the second column, the iodine number of the fatty acids calculated from the previous determination; the third column, the saturated fatty acids, or the solid fatty acids as determined by the lead salt ether method. The total liquid acids or the unsaturated acids, given in the fourth column, are obtained by taking the difference between the saturated fatty acids and 100. The next two columns, headed the "brominated fatty acids insoluble in ether," show the fatty acids which are precipitated when the total fatty acids are dissolved in ordinary ether and brominated, the column labeled "per cent."

unsaturated acids soluble in petroleum ether," were calculated from the amount of saturated fatty acids, the iodine number of the fatty acids and the per cent. of fatty acids thrown down in petroleum ether by means of bromine. It is not necessary to go into this calculation as it is a simple one; it simply shows in a general way that there is a very large amount of unsaturated fatty acids with a comparatively low iodine number.

Under the next general heading are the bromids precipitated from ether and petroleum ether solution, when the glycerids or oils themselves are brominated. The columns headed "per cent." under this general heading give the percentage of the amount of oil taken originally that the actual weight of bromine precipitate represents. The columns headed "per cent. of bromine" of course need no explanation.

TABLE II.—COMPOSITION OF THE FATTY ACIDS.

Serial No.	Description.	Iodine No.	Iodine No. of fatty acids.	Saturated acids. Per cent.	Total liquid acids. Per cent.	Brominated fatty acids insoluble in ether.		Brominated fatty acids insoluble in petroleum ether.		Brominated unsaturated acids soluble in petroleum ether.		Glycerids.				Fatty acids.			
						Per cent.	Iodine No.	Per cent.	Iodine No.	Per cent.	Iodine No.	Bromids precipitated from ether.		Bromids precipitated from petroleum ether.		Bromids precipitated from ether.		Bromids precipitated from petroleum ether.	
												Per cent.	Per cent. Bromin.	Per cent.	Per cent. Bromin.	Per cent.	Per cent. Bromin.	Per cent.	Per cent. Bromin.
2447	Cod-liver oil...	165.7	173.6	5.70	94.30	...	...	36.00	331.5	58.30	93.1	46.60	57.65	...	...	35.76	...	112.30	67.68
15600	Cod-liver oil...	168.0	175.9	15.55	84.45	11.08	358.0	28.58	356.0	55.87	132.7	34.08	59.20	95.60	58.05	36.12	69.35	93.82	68.50
15608	Seal oil.....	143.8	150.6	9.96	90.04	8.68	363.5	22.14	322.5	67.90	116.6	19.57	60.05	78.75	58.24	28.77	69.83	67.06	67.20
15604	Dog-fish liver oil.....	153.3	160.5	18.52	81.48	8.28	340.5	30.09	318.0	51.39	126.1	37.24	...	88.65	58.88	26.08	68.25	90.50	66.75
14261	Pollock oil....	150.9	158.0	15.99	84.01	8.55	362.0	23.19	344.5	60.82	128.4	42.76	58.85	92.50	57.90	27.99	69.45	73.62	68.50
14304	Hake oil.....	152.8	159.8	13.58	86.42	7.83	381.2	22.95	347.7	63.47	126.0	30.32	58.10	...	...	26.68	70.65	73.32	68.70
14278	Haddock oil...	170.5	178.5	13.11	86.89	10.83	350.4	34.96	326.1	51.93	124.2	57.54	58.30	...	...	34.78	68.95	105.30	66.80
11556	Ling oil.....	143.4	150.2	14.42	85.58	7.27	361.1	23.89	349.0	61.69	108.3	30.28	58.10	...	...	23.84	69.55	76.54	68.80
16780	Lake trout liver oil.....	154.8	162.1	7.01	92.99	8.29	351.5	24.08	321.3	68.92	123.0	35.10	59.74	...	...	26.74	69.00	73.08	67.05
	Linseed oil....	...	...	...	...	13.57	262.7	34.61	212.3	...	...	28.82	56.63	...	...	36.04	62.35	81.06	57.27
	Corn oil.....	122.7	128.5	...	...	...	...	12.86	186.0	...	...	...	...	...	...	slight	...	27.96	53.70

being the actual percentage of fatty acids precipitated by this method, while the iodine number is the percentage of iodine absorbed by these fatty acids as calculated from the percentage of bromine found in the bromine precipitate. All of the results have been calculated to the basis of iodine rather than bromine, so that these figures can be compared with the iodine number of the oil and of the fatty acids. The next column headed "brominated fatty acids insoluble in petroleum ether" shows the amount of fatty acids precipitated when petroleum ether is used as a solvent rather than ordinary ether, and the columns under this general heading, "per cent." and "iodine number," have the same significance as the two preceding columns.

The figures under the next heading, "brominated

The next general heading, "fatty acids—bromids precipitated from ether and petroleum ether," means that in these determinations the fatty acids were used instead of the glycerids, the columns headed "per cent." being the same as under glycerids, more than 100 per cent. obtained in some cases, because the bromine precipitate obtained in the determination weighed more than the amount of fatty acids taken originally.

A detailed discussion of one of the analyses will explain all, so the one that is most complete will be considered. Sample 15600, a cod-liver oil, will serve as a typical case. The iodine number of the oil is 168.0, and the iodine number of the fatty acids is 175.9. The iodine number of the fatty acids precipitated from ether by bromine is 358.0,



which compared with the same figure for the fatty acids of linseed oil, namely 262.7, shows at once a great difference between the fatty acids of cod-liver oil and the linolenic acid of linseed oil, the latter being the most unsaturated acid known to occur in any oil of the vegetable or animal kingdom, considering land animals only. This figure shows that this fatty acid must have eight unsaturated bonds to take up iodine, which confirms the work of Heyerdahl and Bull as to the presence of the highly unsaturated acids in fish-liver oils. The fact that theory would require an iodine number of about 387 for a pure acid, shows that this precipitate is a mixture of the acids requiring eight atoms of iodine with acids requiring six or less. There are 11.08 per cent. of fatty acids with this high iodine number. By using petroleum ether as a solvent a much larger amount of bromides are precipitated, representing about 28.58 per cent. of the total fatty acids having an iodine number of 356.0, which is only a little lower than the iodine number of the fatty acids precipitated from ether, and these are also undoubtedly mixtures of the higher and lower unsaturated fatty acids.

The bromides are somewhat different in consistency, the ether precipitate being amber colored and very brittle, while the precipitate from petroleum ether is dark colored and a little gummy. Figuring on the basis that the fatty acids of eight unsaturated bonds have an iodine number of 387 and those of six unsaturated bonds have an iodine number of 274, the mixed fatty acids represented by the iodine number of 358 is made up of a mixture of about 75 per cent. of the eight unsaturated bond acids, and 25 per cent. of the lower unsaturated fatty acids and the precipitate from petroleum ether maintains about the same ratio, showing that all of the octobromides are not thrown down in the ether solution. This leaves 84.45 per cent. of unsaturated fatty acids having an iodine number of 132.7, evidently made up of a mixture of acids of the oleic series and the linolic series, which shows that there is present in these fish oils a complete series of fatty acids from those with two unsaturated bonds to acids with eight unsaturated bonds. That they are quite different from the series present in linseed oil is shown by a number of different reactions—the solubility of the bromides in ether and petroleum ether, the melting point of the bromides, and the action of the fatty acids toward dilute permanganate, these acids not form-

ing the hydroxy-acids as do the fatty acids of linseed oil, but being apparently completely oxidized, while the acids with two unsaturated bonds are the only ones of the series to form hydroxy acids.

Treating the glycerids in the same manner as the fatty acids (that is, dissolving in ether and precipitating with bromine, collecting the bromides and analyzing), we find a very peculiar condition. The iodine number of the precipitated glycerids from ether is only 229, which shows at once that it cannot be the triglycerid of the fatty acid with eight unsaturated bonds, as that would have an iodine number of 369.9; in fact this figure is lower than the figure for the triglycerid of the acids with six unsaturated bonds which would be 262.15, all of which goes to show that the glycerid which is precipitated by bromine under these circumstances is a mixed glycerid and must be made up of at least one acid radical of the very unsaturated acid and one radical of the saturated acids or the acids with two unsaturated bonds; possibly the third acid radical is still different, at least there is very good evidence that here we have a combination of glycerin with at least two different fatty acid radicals, and perhaps three. This brings the iodine number of the precipitate from ether for the fish-liver oils and the linseed oil quite close together, the linseed oil having 207.0 as compared with 229.9 for the cod-liver oil, a very slight disparity considering the difference between the respective iodine numbers of the bromine precipitated fatty acids, which are 358 for fish oils and 262 for linseed oil. This only emphasizes the difference in the glycerids precipitated from the two classes of oils. This high iodine number appears to be characteristic of these fish oils and the related oils, such as those from the seal and whale and also is found for the trout liver, a fish with entirely different food and living in fresh water.

A general consideration of the figures in this table shows the remarkable similarity of the oils from the various fish including the seal oil; in all of these the same series of fatty acids seems to be present, and in about the same proportion, which makes the oils very similar. This similarity holds true also as to other properties of the oils and in the changes that take place on being exposed to air. In fact, there seems to be little doubt that these oils have about the same value, and the recent work of Bull confirms the great similarity among the oils from various fish and from animals like the seal.



In conclusion it appears that the results of this work have (1) developed a method for the determination of hexabromid, or bromin precipitate as it should be called, which is satisfactory and easy to manipulate; (2) that little, if any, information can be obtained by the oxidation of these fatty acids with permanganate, as apparently they are so readily and completely oxidized that no hydroxy acids are formed with the highly unsaturated members of the group; (3) that the presence of mixed glycerids of undetermined composition, is proven; (4) that the unsaturated fatty acids present in the fish oils belong to a series entirely different from any that are known at present in any of the vegetable or animal (land animals) oils as yet studied; (5) that the fish and blubber oils are remarkably alike in the composition of the unsaturated fatty acids and mixed glycerids present; (6) that the oil from animals living on fish is remarkably similar to fish oil in the composition of the unsaturated fatty acids and the mixed glycerids; (7) that even the oils of fresh water fish like the trout, living on entirely different foods, are remarkably similar to the oils from the cod and other similar salt water fish; (8) that these oils present a very interesting field for investigation in further separation and study of a very peculiar series of fatty acids, both along the line of preparing hydroxy acids with other oxidizing agents less active than permanganate, and in further study of the bromin compounds, as in my opinion the *special value* of the cod-liver oil in a medicinal way must be in these extremely easily oxidized series of fatty acids.

#### THE RELATION BETWEEN THE TEMPERATURE OF KEROSENE AND THE EXPLOSION PRESSURE OF THE SUPERNATANT MIXTURE OF AIR AND VAPOR.

By W. P. BRADLEY AND C. F. HALE.

Received March 24, 1909.

The testing of kerosene oil, so far as it concerns the safety of the oil as an illuminant, is based upon the relation of temperature to its rate of vaporization. The rate of vaporization is judged by the result of ignition tests. These tests are of two sorts. In one, which is called the fire-test, a determination is made of the lowest temperature at which the oil will give off vapor fast enough to support a permanent flame when ignited. In

the other, which is called the flash-test, the safety of the oil is judged by the temperature at which it first vaporizes sufficiently to form with the supernatant air a mixture rich enough to ignite at all.

The fire-point is often spoken of erroneously as the temperature at which the oil itself takes fire. But it is hardly necessary to say that the oil, as liquid, never burns; that all flame is gas or vapor flame; and that what burns on the surface of the oil, at any time and at any temperature, is only the vapor of the oil.

The danger attendant upon the use of illuminating oil is threefold: (1) danger from conflagration when the oil is in bulk; (2) danger from conflagration when the oil is spilled; (3) danger from explosion.

Those who advocate the fire-test emphasize the danger from conflagration if oil is spilled in any way. They appear to ignore the danger from explosion, as practically non-existent.

Those who advocate the flash-test, while admitting the danger from conflagration, emphasize the earlier danger arising from the presence, *in the bowl of the lamp*, of explosive mixtures of inflammable vapor and air, which, if ignited in any way, might burst the lamp, scatter the oil, and so induce conflagration, in addition to the more direct damage arising from the explosion itself.

Now it may readily be admitted that a liquid whose vapor will catch fire from a spark or match, at such temperatures as are prevalent within dwellings or without, is a dangerous liquid, whatever its name or use may be, and that a grade of kerosene which will not so ignite is safer. There can be no question that a merchantable kerosene should be required to pass a fire-test of considerably higher temperature than any which will normally prevail during its use as an illuminant. A body of oil which will stand such a test will not only not ignite at ordinary temperatures, but it will extinguish a spark or match as effectively as water does. All grades of kerosene now on the market will do as much as this.

However, danger from conflagration depends upon other factors than that of temperature, and particularly upon one, namely, the presence of some kind of absorbent material, whether itself inflammable or not, which can perform the functions of a wick. Oil which will extinguish a flame, in bulk, will take fire very easily and burn freely, if soaked into textile material. Indeed, within very wide limits it may be said that the inflam-



mability of kerosene which has fallen upon, and has soaked into, a carpet or upholstery has no relationship at all to the inflammability of the same oil in bulk. Whether a conflagration occurs in such a case depends scarcely at all on the grade of the oil, as determined by fire-tests or by flash-tests, but solely on the accidental circumstance that a flame does or does not come into contact with it. Moreover, as a grade of kerosene which would not take fire under these circumstances would not ignite at the wick of a lamp either, it is clearly impossible to avoid this particular species of danger by any test at all. A fire-test, therefore, simply guarantees against danger from conflagration while the oil is in bulk, and while it is kept at temperatures below that of the test. It does not, and from the nature of the case cannot, guarantee against danger from conflagration, after the oil is spilled.

The third danger attendant upon the use of kerosene as an illuminant will be readily appreciated in this day of gas engines and gasoline engines. Indeed, explosive mixtures of air and the vapor of kerosene itself are being used more and more to drive engines of the explosive type. Danger from explosion is always present where inflammable vapors and air are mixed together. This condition may arise in the bowl of any lamp. Above the liquid kerosene there is always kerosene vapor, and there is always air. The proportions of vapor and air depend upon the temperature of the oil. There is more vapor at a high temperature and less at a lower one.

Now it is a well-known fact that such a mixture cannot explode if it is too poor in vapor, or too rich. The proportions must lie within certain maximum and minimum limits. Even within these limits, there can be no explosion, unless a spark or flame comes in contact with the mixture. Near the maximum, and near the minimum limit, the explosion is feeble. Explosion of greatest violence occurs when the proportions are such that the amount of air present is exactly right to burn the vapor completely. An excess of either air or vapor serves as a cushion to moderate the force of whatever explosion does occur. And it is well known that the force of the explosion drops off very rapidly indeed as a result of even moderate departure from the correct proportions.

Now the minimum limit in the matter of proportions, which is the beginning of danger from explosion when the mixture is ignited in confine-

ment, is precisely the point which is approximately indicated by the flash-point of the oil, as observed in the so-called closed cup. And as the flash-point of an oil in the closed cup is from thirty to forty-five degrees<sup>1</sup> lower than the fire-point, depending on the degree of protection from drafts afforded by the various styles of cup, if the flash-point is higher than any temperature to which the oil is likely to be exposed during normal use, it is clear that danger from conflagration in bulk, as well as danger from explosion, will be eliminated. When these two dangers are avoided, all has been done which can be done by the legal establishment of standards, for danger from oil which is spilled can never be eliminated, as has been said, if the oil is to burn at all in the lamp.

There are several ways in which an explosive mixture in the bowl of a lamp might become ignited, but probably the one of most frequent occurrence arises from the use of a wick too small for the burner, whereby communication is afforded between the mixture and the flame. Experimental evidence on this point has been furnished by Kast and Rose<sup>2</sup> who found that with a wick which did not fit the burner closely an explosion always occurred when the temperature of the oil in the bowl of the lamp became sufficiently high.

Now, it should be noted that the conditions under which flash-tests are made are of great importance, if the tests are to serve as a guarantee of safety from explosion.

Two general types of apparatus, designated respectively as the *open* and the *closed* cups, are used to determine flash-points. In the closed cup an attempt is made to approximate the conditions under which vaporization takes place, to those which obtain in the bowl of a lamp. The space above the oil is closed in by a cover, except for a small aperture provided for the introduction of the ignition torch. The conditions in the case of the open cup are different. Here the oil vaporizes into the open air, which is always more or less disturbed by currents which disseminate the vapor, and which militate against the formation of the flash mixture at all. It is not surprising therefore that the flash-point of a given sample of oil is invariably several degrees higher in the

<sup>1</sup> For the sake of easier comparison with the legal standards which prevail in this country, all the temperatures given in this paper are those of the Fahrenheit scale, in the first instance. The corresponding values according to the centigrade scale are also given.

<sup>2</sup> *Journal für Gasbeleuchtung*, 39, 348-351, 364-368; and *Central-Blatt*, 1896, II, 142 and 220.



open cup than in the closed cup,<sup>1</sup> for the rate of vaporization must be more rapid in the former before a sufficiently rich mixture can be obtained.

It is evident that the fire-test, involving as it does continuous combustion of vapor, can never be made at all except in a cup which is open, for the time at least. But here again it makes a difference of about the same number of degrees whether the oil fills the cup nearly to the rim, as is customary in the usual forms of "open" cup, or whether the surface is protected somewhat from drafts by an extension of the rim upward, as in one form of the closed cup, the Elliott.

Taking the Tagliabue open cup and the Elliott closed cup as a basis for discussion, we have no less than four tests for one and the same oil. Each of these tests is spaced from its neighbor by approximately fifteen degrees. The following table illustrates this by the use of two general samples of oil, both purchased in open market, and representing respectively a rather low-grade, and a rather high-grade kerosene.<sup>2</sup> Portions of these same samples were used in the explosion tests which follow, and will be referred to later.

TABLE I.—COMPARISON OF FLASH- AND FIRE-TESTS.

Sample.	Elliott	Tagliabue	Elliott	Tagliabue
	flash. Fah.	flash. Fah.	fire. Fah.	fire. Fah.
1	112 (44.5)	127 (52.8)	141 (60.5)	158 (70)
1 A	114 (45.5)	128 (53.4)	142 (61)	159 (70.5)
1 B	114 (45.5)	129 (53.9)	142 (61)	158 (70)
2	88 (31)	103 (39.5)	114 (45.5)	126 (52.2)
2 A	89 (31.7)	104 (40)	114 (45.5)	128 (53.4)
2 B	89 (31.7)	104 (40)	114 (45.5)	128 (53.4)

The danger which ensues when a mixture of inflammable vapor and air becomes ignited is due of course to the pressure which is exerted at the moment of explosion upon the walls of the containing vessel. The quantitative relation between this pressure and the temperature of the oil at the moment of explosion seems never to have been determined experimentally, but qualitative results have been reported by several writers. Kast and Rose<sup>3</sup> withdrew samples of the air-vapor mixture from the bowl of a lamp at intervals, after certain measured portions of the oil had burned, and ignited them in a pipette. The pressures, however, were not measured. Newbury

<sup>1</sup> About fifteen degrees higher, for instance, in the Tagliabue open cup than in the Elliott closed cup.

<sup>2</sup> On Jan. 8, 1906, a lamp explosion of considerable violence occurred in this city, resulting in loss of life. Oil No. 1 was the grade which had been in use up to that time. Following that event, and pending revised legislation on the subject, oil of the grade of Sample No. 2 was used.

<sup>3</sup> *Loc. cit.*

and Cutter<sup>1</sup> separated the constituents of kerosene by fractionation and determined the temperatures at which the different fractions would give explosive mixtures with the amount of air necessary for the complete combustion of the sample. The magnitude of the pressures is not recorded beyond the statement that they were violent.

In the hope of throwing some light on this point the present investigation was undertaken.

*Method of Experimentation.*—In general, the method of experimentation consisted in the measurement of the pressures developed by the explosion of mixtures of air and vapor, obtained by passing air through oil heated to different temperatures. The apparatus employed is shown diagrammatically in Fig. 1. It is composed essentially of two parts—a stout brass cylinder, A, in which the explosion takes place, and a pressure indicator, B, of the type commonly used in the measurement of engine horse-power. The explosion cylinder was 13.5 cm. from dome to dome, 7.5 cm. in diameter, and had a capacity of 560 cc. On the left is a small copper tube I, of 3.2 mm. internal diameter, which is bent at its lower end into a small circle. The upper side of the circular portion is drilled with small holes. This tube serves for introducing the air, and it can be closed externally by the cock S. The straight tube T at the right is closed at its lower end, and contains a small amount of water in which the thermometer for measuring the temperature of the oil is immersed. At C is a stout copper wire insulated by a sheath of wood, and held in place by a brass supporting tube which is attached to the cylinder by a short coupling. The wire serves as one terminal for producing a make and break-spark, and it can be rotated within its sheath so as to bring it into contact with the inside of the neck of the cylinder, which is the other terminal of the circuit. The spark serves to ignite the mixture.

The neck of the cylinder is threaded to receive the short connection N, and is provided with a side tube, O, which permits the escape of the excess vapor and air as well as that of the products of combustion when a new charge of air is being admitted through I. When O is not in use, it is closed by the stopcock S'. To the upper part of N the indicator is attached, as shown.

Two other experimental details must be mentioned. First, provision must be made for the regulation of the air supply, and second, for the



control of the temperature. The arrangements for these will be clear from Fig. 2. The explosion cylinder is placed in a water bath, H, heated by a Bunsen burner, uniformity of temperature being secured by a wire stirrer. The air supply is de-

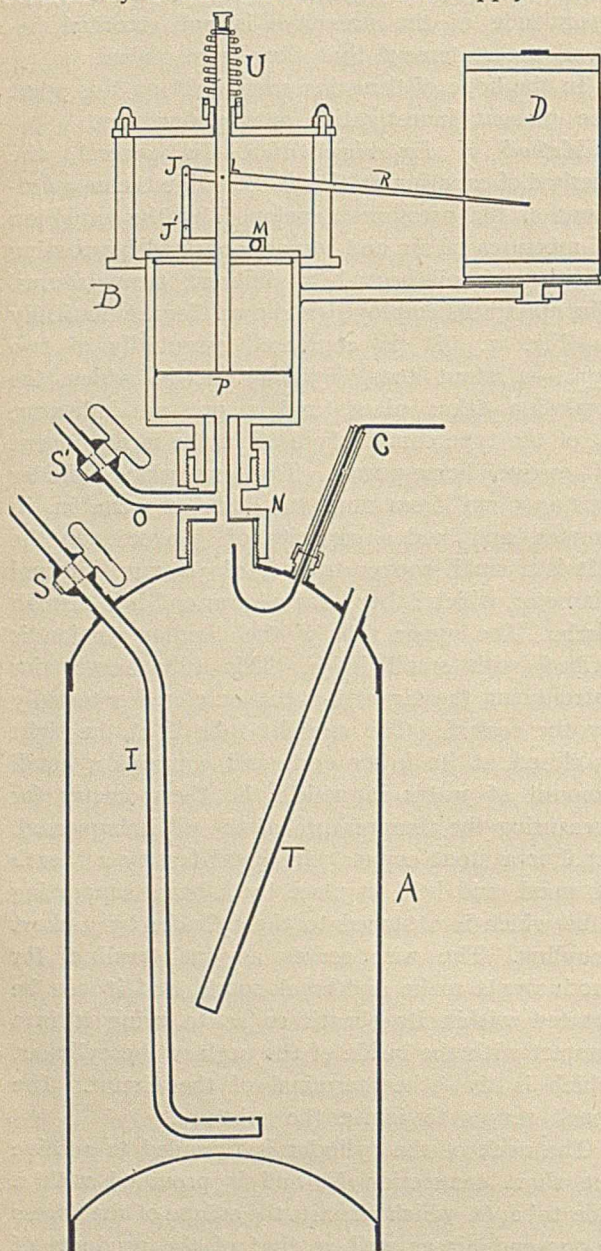


Fig. 1.

livered from the gas washing bottle A, whose air is displaced by water from the reservoir R. In all the experiments the water was allowed to flow into the wash bottle up to a certain point in its neck. Uniformity of speed in the delivery of the air supply was secured by a constant level of water in the reservoir.

Each sample of oil was first examined with reference to its two flash-points and its two fire-points, by the use of the Elliott and the Tagliabue testers. Another portion of the sample was then introduced into the cylinder. This portion, amounting to 390 cc., occupied rather more than two-thirds of the total volume of the cylinder, giving a condition comparable with that in a lamp which has been burning for some time. The indicator was then attached, and the cylinder was placed in the water bath. The temperature of the oil having been made constant at a point about one degree lower than the Elliott flash-point, the cocks S (Fig. 1) and P (Fig. 2) were opened and a charge of air allowed to bubble through the oil, escaping so far as necessary through S'. Then, with S and S' closed, the mixture was exploded. The

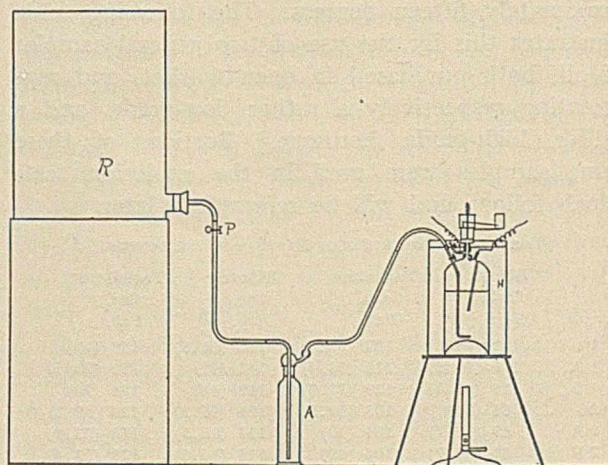


Fig. 2.

first explosion at each temperature was rejected as abnormal. The pressure obtained was higher than any subsequent explosion at the same temperature, probably owing to the excess of vapor formed during the heating process. After the first, the explosions were remarkably concordant.

Immediately after each explosion, the thermometer in T showed a rapid rise of about two degrees, after which the temperature fell in a short time to its former value. After thermal equilibrium was reached, a series of three explosions was made for each temperature, using a new charge of air in every instance. Each of these series was spaced from the next by about five degrees, and the total range of temperature covered with a given sample extended from that at which the mixture was too poor to explode, to that at which it was too rich.



Fig. 3 gives an idea of the appearance of the indicator card after a complete set of explosions.

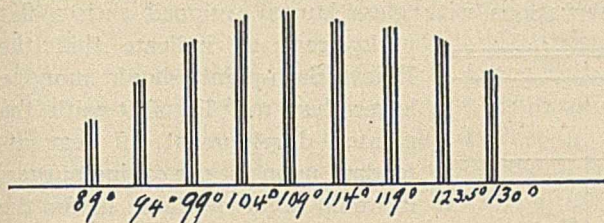


Fig. 3.

At the conclusion of an experiment, the cylinder was removed from the bath and quickly cooled to the temperature of the room, by immersion in running water. The oil was then removed, and transferred to the testers for a determination of the flash-points and fire-points as before.

Some preliminary observations had shown that the test-points of kerosene could be considerably raised by a small amount of evaporation,<sup>1</sup> and it was feared that such might be the case during these explosion tests. It was found, however, that the flash-point in the closed cup, the test which could be made with the greatest accuracy, was never raised more than two degrees, even in the case of the oil of greatest volatility. It is evident then that the explosion pressures obtained are fairly representative of the pressures which might result if the oil were to be heated at once to each of the temperatures employed, without previous explosions. Whatever change does occur in the nature of the oil by gradual evaporation during the determination of a set of explosion pressures, results in shifting the maximum pressure towards a region of higher temperature, and thus makes the indicated danger less than it really is in a fresh sample. The results follow.

Table I.—Temperatures and average explosion pressures obtained with kerosene oil, sample No. 1—Flash-point: Elliott, 112° F.; Tagliabue, 127° F. Fire-point: Elliott, 141° F.; Tagliabue, 158° F.

No. 1 A.		No. 1 B.	
Temperature. Fah.	Pressure. Lbs.	Temperature. Fah.	Pressure. Lbs.
109	3	114	33
115	38	120	49
120	48	126	62
125	58	130	68
131	68	134	69
134	69	138	68.5
140	68	142	68
145	62	147	63
150	57	154	52
160	40	164	33
		169	5

<sup>1</sup> Compare also Newbury and Cutter, *Loc. cit.*

The test-points of this sample, after the explosions, were: No. 1A—Flash-point: Elliott, 114° F.; Tagliabue, 128° F. Fire-point: Elliott, 142° F.; Tagliabue, 159° F. No. 1B—Flash-point: Elliott, 114° F.; Tagliabue, 129° F. Fire-point: Elliott, 142° F.; Tagliabue, 158° F.

Table II.—Temperatures and average explosion pressures obtained with kerosene oil, sample No. 2.—Flash-point: Elliott, 88° F.; Tagliabue, 103° F. Fire-point: Elliott, 114° F.; Tagliabue, 126° F.

No. 2 A.		No. 2 B.	
Temperature. Fah.	Pressure. Lbs.	Temperature. Fah.	Pressure. Lbs.
87.5	13	89	23
90	35.5	94	43
95	47	99	56.5
100	59	104	66
105	64	109	68.5
110	71	114	69.5
115	70	119	66
120	66	123.5	62
125	61	130	49
130	48	134.5	No. explos.
135	No explos.		

The test-points of this sample after the explosions were: No. 2A—Flash-point: Elliott, 89° F.; Tagliabue, 104° F. Fire-point: Elliott, 114° F.; Tagliabue, 128° F. No. 2B—Flash-point: Elliott, 89° F.; Tagliabue, 104° F. Fire-point: Elliott, 114° F.; Tagliabue, 128° F.

The data contained in these tables, together with the flash- and fire-points, are shown graphically in Figs. 4 and 5.

In their general form these curves are very similar. Their crests for instance lie at almost the same pressure. It is interesting to note, however, that the range of temperature covered by the high-grade oil is about sixty degrees, while that of the low-grade oil is only fifty. The latter oil gave no explosions at all beyond 130°, in spite of the fact that at that temperature the pressure was still almost fifty pounds. We are unable to offer any explanation of this curious fact. The curve was continued to the axis of temperature in such a way as to resemble that of the other oil in the corresponding region.

Of chief interest is the relation of the four test-points to the corresponding explosion pressures.

1. In the low-grade oil, the Elliott flash-point coincides almost exactly with the beginning of the explosion curve. In the case of the high-grade oil, it cuts the curve at a point where the pressure is already greater than 25 lbs. This seems to indicate that the Elliott flash-point is of greatest



diagnostic value precisely in the case of those oils which are most dangerous to use.

For oil of any grade, the Elliott flash-point is

that point should be higher than the temperature to which the oil in a lamp would be exposed. Observations with glass lamps equipped with a flat wick seem to indicate that the Elliott flash-point should not be lower than  $100^{\circ}\text{F}$ . But with the greater development of heat attendant upon the use of ring burners it would seem desirable to fix the point a few degrees higher than that. Kast and Rose,<sup>1</sup> using different styles of burners, including a "Rochester" circular burner, found that the temperature of the oil in the bowl of the lamp varied between  $90^{\circ}\text{F}$ . and  $107^{\circ}\text{F}$ . according to the burner employed, the room being at the ordinary temperature.

Again, it is not to be inferred from our results that the open cup tests are to be altogether disqualified. These tests, though indirect, are still of value if the standards are set with reference to the flash-

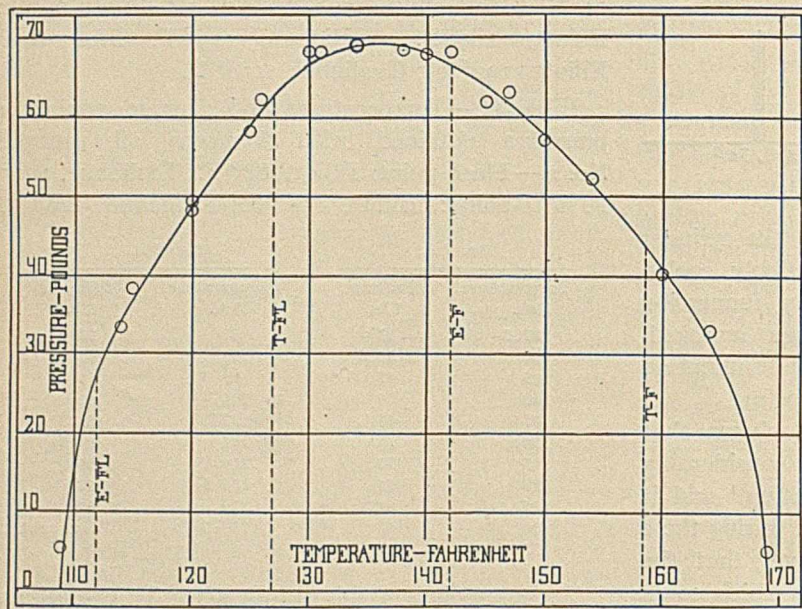


Fig. 4.—Sample No. 1.

the only one of the tests here employed, which in any sense heralds the approach of danger from explosion.

2. For both grades of oil, the Tagliabue flash-point falls upon a point in the curve just short of the crest, where the pressure is already nearly as great as it is ever going to be. This test is therefore not an indication of the approach of danger from explosion, but rather of its arrival in full. Oil in a lamp is safer at almost any other temperature than that at which it flashes in the Tagliabue cup.

3. The fire-point in the Elliott cup gives about the same indication as the Tagliabue flash-point, except that the point of greatest danger is just passed, instead of being just at hand.

4. Finally, the Tagliabue fire-point lies in the region where explosions are hard to get at all, because the mixture is already too rich in vapor. In fact, in the case of the low-grade oil, it lies almost exactly at the point beyond which explosions become altogether impossible.

While the results of these experiments do not afford direct information concerning the minimum flash-point consistent with safety, it is certain that

point in the closed cup. Thus, if  $100^{\circ}\text{F}$ . is to be chosen as the minimum flash-point in the closed cup, then the limit for the open cup should be at least  $115^{\circ}\text{F}$ . Similarly, the fire-point in the

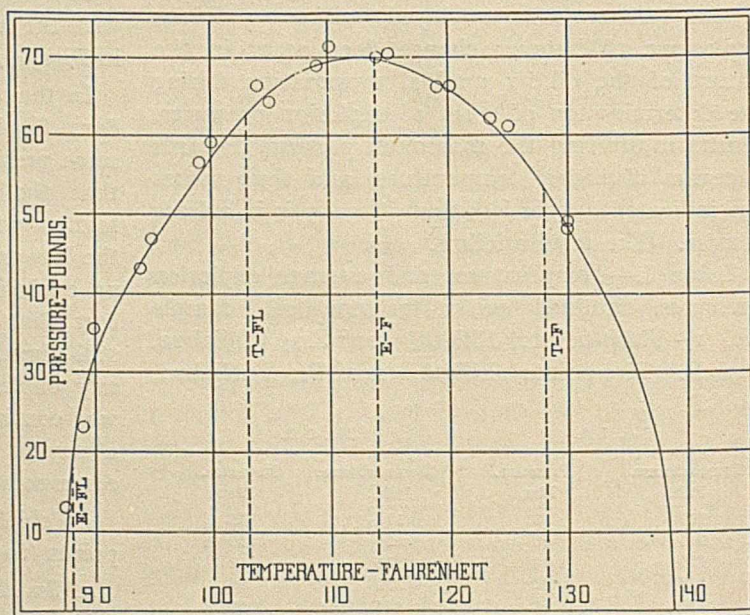


Fig. 5.—Sample No. 2.

closed cup should not be lower than  $130^{\circ}\text{F}$ ., and in the open cup,  $145^{\circ}\text{F}$ . Each of these requirements,

<sup>1</sup> *Loc. cit.*



though numerically so different, would furnish the same oil.

There are two possible sources of error in the method of measuring pressures employed in the foregoing experiments. One of these arises from the "dead space" below the piston of the indicator, which is not filled with the explosion mixture. The other is the small change in volume which is occasioned at the moment of explosion by the upward movement of the piston. Both of these render the observed pressure smaller than it otherwise would be. But it is obvious that neither of them could affect the general relation of the test-points to the pressure curve.

The only other variable factor in the experimental method lies in the possible influence which the size of the ignition spark might have. To test this, currents of different magnitudes varying from 0.5 to 2.0 amperes were used in separate experiments. Between these limits the explosions were regular, and the pressures obtained were concordant with one another. But when a current of less than 0.5 ampere was employed, the spark was so weak that occasionally no ignition followed at all, although when explosion was obtained it was concordant with those induced by the larger spark.

#### SUMMARY.

1. The explosion pressures of saturated mixtures of air and kerosene vapor have been measured over a range of temperature from a few degrees below the closed cup flash-point to several degrees above the open cup fire-point.

2. At the temperature of the closed cup flash-point the oil is already evaporating freely enough to produce a mixture which will yield an explosion pressure of several pounds, varying from about fifteen pounds in the case of a low-grade oil, to about twenty-five pounds for a high-grade oil. This test is the one which indicates most precisely the temperature at which danger from explosion begins.

3. At the temperature of the open cup flash-point the rate of evaporation is such that the mixture gives an explosion pressure almost equal to the maximum which can be obtained from the given sample.

4. At both fire-points the vaporization is so rapid that the mixture is too rich, and in consequence the explosion pressure is less than the maximum. At both temperatures the greatest

danger from explosion is passed; at the open cup fire-point, indeed, long passed.

5. The maximum pressure was practically the same for high- and for low-grade oil, amounting to about seventy pounds gauge pressure.

WESLEYAN UNIVERSITY,  
MIDDLETOWN, CONN.,  
Mar. 17, 1909.

## ANALYSES AND FRICTION TESTS OF LUBRICATING GREASES.

By HORACE W. GILLET.

Received March 19, 1909.

Since the first lubrication engineer applied a piece of fat meat to the squeaking axle of his primitive cart, practically everything that is greasy or slippery has been used in making grease. The term "lubricating grease" is one of the most comprehensive in the dictionary. In recipes given in works on lubrication we find as ingredients of greases, practically all known fats and oils, as tallow, palm oil, neatsfoot, rape, olive, sperm, castor, lard, corn, cottonseed, fish, all grades of mineral oil; soaps of sodium, potassium, calcium, magnesium, lead, zinc, aluminium and iron; rosin, rosin oil, bone tallow, wool grease, anthracene oil, paraffin, vaseline, ceresin, beeswax, Japan wax, bayberry wax, spermaceti, pitch, asphalt, naphthalene, rubber, glue, graphite, talc, infusorial earth, gypsum, magnesia, asbestos, mica, sulphur, Irish moss, kaolin, starch, wheat flour, sawdust, wood pulp, ground cork, ground glass, mercury, antimony, oxide of tin, lampblack, ivory, dust, water, alcohol, glycerine, turpentine, carbonate of iron, lead sulphide, hair, borax and salt.

Though greases are of such varied composition, the literature of lubrication shows no comparison of the lubricating power of greases of different composition.

"Grease is grease" seems to be the attitude of most makers and users of that lubricant. The following work was undertaken in an attempt to determine the relation, if any, between composition and lubricating value of the typical greases on the market to-day.

Commercial greases may be divided into the following classes:

A. The tallow type: these greases are made up of tallow and more or less of an alkali soap, commonly the sodium or potassium soaps of palm oil, mixed with a smaller amount of mineral oil. These were the principal types of lubricating grease



ten or twenty years ago, but to-day are less used than the greases of type *B*.

*B*. The soap-thickened mineral oil type: these are the most common journal greases to-day, and are composed of mineral oil of various grades made solid by the addition of calcium or sodium soaps. Calcium soap is more used than sodium.

*C*. Types *A* or *B* with the addition of a mineral lubricant—usually graphite, mica, or talc.

*D*. The rosin-oil type: These consist of rosin oil thickened by lime, or less commonly, litharge, to which is added more or less mineral oil, either paraffin or asphalt oils being used. These are sticky, usually contain 20–30 per cent. of water, and find their chief application as gear greases where true lubrication is not so essential as prevention of wearing and rattling of the gears. Some very heavy bearings are occasionally lubricated with this type of grease. Tar, pitch, graphite and such fillers as wood pulp and ground cork are often put into these gear greases.

*E*. Non-fluid oils: these are thin greases stiffened to some extent with aluminium oleate or a mixture of soaps, as sodium and calcium.

*F*. Special greases, such as a mixture of wood pulp and graphite, thin greases of any of the above types mixed with wool or cotton fibers, hot-neck greases, freak greases containing rubber, etc.

Of these *A*, *B*, and *C* are the most important as lubricants.

The analysis of a lubricating grease may have one of two objects in view: to duplicate the grease, or to determine its value as a lubricant. Without resorting to mechanical tests of the actual friction reducing power of the grease in question, the first is probably the easier problem.

In the analysis of a grease, we may cover the following points:

Consistency.

Melting point.

Flash point.

Content of free acid.

Amount and nature of soap.

Ash, not alkali, from soap, nature and amount.

Mineral filler, as graphite, talc, etc., nature and amount.

Content of unsaponified, but saponifiable animal or vegetable fats or oils, nature and amount.

Content of unsaponifiable mineral or hydrocarbon oil, nature and amount.

Content of water.

The methods used in the analysis of the greases hereinafter described, was as follows:

Note first the odor and color. These may give clues of the probable composition, and will show if the grease is perfumed with nitrobenzol or any similar substance. Note the behavior on melting. The grease should melt to a clear homogeneous fluid. The oil should not melt away from the soap, since in such a case after being in the feed cup some time, the grease may change in composition, the oil running off the bearing and the soap staying behind.

The melting point is the most important single determination, as will be seen in the discussion of the friction tests.

This has been determined in various ways by various workers. One method is to use a closed capillary tube, after the usual way of taking melting points. Here it is very difficult to tell the exact point where the grease does become transparent and fluid. Moreover, the grease has to be melted into the tube, in which case it has to be laid away for several hours in order that it may regain its normal melting point.<sup>1</sup>

Another is the drop point, suggested by Pohl, in which the grease is smeared upon the bulb of a thermometer, or the thermometer dipped into the melted grease. The thermometer is then heated in an air bath and the drop point taken either as the point where the grease begins to flow and get thinner, so that the mercury of the thermometer bulb is seen, or when the drop actually falls off, according to the directions of different writers. This method is very inaccurate, owing to the large variation caused by the difficulty of getting the same amount of grease on the bulb each time, and through differences in the size and shape of thermometer bulbs.

Ubbelohde<sup>2</sup> gives a modification of this method that overcomes most of the difficulties, but for greases melting below 100° C., we have found the most satisfactory method to be the use of an open tube of 0.4 cm. internal diameter, and about 8 cm. long. This is stuck into the grease so that a plug of grease 1 cm. long is left in the glass tube. The tube is then attached by a rubber band to a thermometer so that the plug is beside the bulb. The thermometer with the tube attached is then immersed into a beaker of water so that the bot-

<sup>1</sup> Archbutt and Deeley, "Lubrication and Lubricants," 2d. Ed., p. 202.

<sup>2</sup> Holde, "Untersuchung der Mineralöle und Fette," Berlin, 1905, p. 209.



tom of the plug is 5 cm. below the surface. The water is then heated at the rate of 3°-4° C. per minute. When the melting point is reached, the plug, which is under a pressure of 5 cm. of water, slides upward in the tube. Checks can be obtained to 0.5° C.

The following tables show the effect of varying the different conditions:

TABLE I.—EFFECT OF DIAMETER OF TUBE.

Internal diameter.	Melting point.
0.55 cm.	83.5° C.
0.50	83.5
0.40	84.0
0.30	84.5
0.15	85.5
0.10	86.0

TABLE II.—EFFECT OF LENGTH OF PLUG.

Length of plug.	Melting point.
0.5 cm.	83.5° C.
1.0	84.0
2.0	85.0

TABLE III.—EFFECT OF DEPTH OF IMMERSION.

Depth immersed.	Melting point.
7 cm.	83.0° C.
5	84.0
2	85.0

TABLE IV.—EFFECT OF RATE OF HEATING.

Rate of heating.	Melting point.
Per min.	
1° C.	83.5° C.
3°-4°	84.0
8°	84.5

The consistency at 20° C. was determined by a Legler consistometer which consists of a pointed glass rod, bearing a pan for weights, and supported by a spiral spring.<sup>1</sup> The grease was held at 20° C. for about an hour, and the total weight determined which was required to sink the rod into the grease exactly 1 cm. The spring was of such strength that it took 20 grams to depress the rod 1 cm. in air, and this 20 grams was subtracted from the total weight required, to give the consistency number. This varied in different commercial greases from 200 grams to 4 grams. Readings could be checked to within 5 per cent. of their value. The instrument gave a consistency number for the pasty mass consisting of 50 per cent. stearic and 50 per cent. oleic acids, of 75-80 grams. All the weights must be on the pan before it is released, as the penetration is less if the weights are added while the rod is falling. Attempts to take the time required for the rod to sink a certain

distance were unsuccessful, as the greases varied so much in consistency that the same weight could not be used for all, which would be essential for comparative results by the timing method. Hence recourse was had to the weight required to sink the rod a given distance. The measurement of the consistency is a well recognized part of the examination of a lubricating grease,<sup>1</sup> yet, as shown in Table X, it has no direct bearing on the lubricating power. Its value lies chiefly in showing what sort of a grease cup should be used for that particular grease, whether gravity, compression, etc.

The flash-point is taken in a 50 cc. porcelain crucible filled with the grease, into which is stuck the bulb of a thermometer. The crucible is heated at 5° C. per minute on a sand bath, and the flash determined by noting the temperature at which a 3 mm. gas flame passed over the crucible at 2° intervals causes a flash to pass over the surface. The chief value of this determination is to give some idea of the grade of mineral oil used in compounding the grease.

The content of free acid is obtained by dissolving or disintegrating, if not completely soluble, 2.82 grams of the grease in a neutral mixture of alcohol and ether containing phenolphthalein. The grease is then titrated with N/10 KOH to pinkness, each cc. of alkali used equaling 1 per cent. of free acid, figured as oleic. Most commercial greases run almost neutral, 0.5 per cent. being high, though as high as 6 per cent. has been found. Greases that contain unsaponified palm oil are especially likely to show free acid. The importance of neutrality is very great, because of the corrosion of the bearings by free acid.

The determination of the soap content may be made in several ways. The bases may be thrown down as oxalates from a turpentine-benzine-alcohol solution by oxalic acid, after the method of Conradson<sup>2</sup> and the soap content figured from the amount of base found. The soaps may be determined by Holde's method of decomposing the soaps by mineral acid, washing out the mineral acid, and titrating the fatty acids thus freed, and from the titration figuring the amount of soap.

The simplest and shortest method, however, in practically every case, is to ash the grease,

<sup>1</sup> Benedict and Lewkowitzsch, *Loc. cit.* Archbutt and Deeley, "Lubrication and Lubricants," 2d Ed., p. 162.

<sup>2</sup> *Jour. Amer. Chem. Soc.*, XXVI, 6, 705.

<sup>1</sup> *Chem. Ztg.*, 1884, VIII, 1657; *Chem. Ctrbl.*, 1884, XV, 734. Benedict and Lewkowitzsch, "Oils, Fats and Waxes," 1895 Ed., p. 74.



titrate the  $\text{Na}_2\text{CO}_3$ ,  $\text{K}_2\text{CO}_3$  or  $\text{CaO}$  in the ash with acid, and figure the soap content from the titration, one cc. of  $N/2$  acid being equivalent to

0.1612 gram potassium stearate,

0.1535 gram sodium stearate,

0.1520 gram calcium stearate.

If the Liebermann-Storch rosin test shows the soap to be partly a rosin soap and rosin oil is absent, this will give low figures, owing to the higher combining weight of abietic than that of stearic or oleic acids. In such a case, if extreme accuracy is required, the fatty acids of the soaps may be freed, and rosin determined by Twitchell's method. Ordinarily, this would not be required.

In the case of greases made with sodium or potassium soaps, the soap may be directly determined after extraction in a Soxhlet of the unsaponified and unsaponifiable fats with ether, by extraction of the residue from the ether extraction, with alcohol. With lime soaps this method fails, owing to the large solubility of calcium soap in all solvents for fats, as well as its insolubility in alcohol. It would greatly simplify the analysis of greases containing calcium soap if some solvent could be found that would extract fats and oils without extracting any of the lime soap, in the same way that ether may be used to separate the fats and oils from the soap in a soda soap grease. In attempting to make an ether extraction of a lime soap grease, we have repeatedly found from  $1/4$  to  $1/3$  the total lime in the ether extract, owing to the solubility of the calcium soap.

Gasoline extracts nearly as much. Numerous experiments have been made with other solvents and mixtures of solvents, but no satisfactory one has been found. Ethyl acetate at room temperature dissolves but little of the calcium soap, and the extract will give a fair qualitative idea of the nature of the oil used in compounding the grease.

If the grease is made up with a lead soap, the lead may be determined by decomposing the grease by boiling with a mixture of sulphuric and nitric acids, after the Neumann acid ashing method, and weighing the lead sulphate.

The nature of the base used in the soap may be determined by the usual methods, or more quickly and delicately, by microchemical methods. A small amount is ignited on a porcelain crucible cover, the ash dissolved in dilute  $\text{HCl}$  and three drops of the solution put on a microscope slide. To the first drop is added a small drop of dilute

$\text{H}_2\text{SO}_4$ , and to the second a small drop of  $\text{H}_2\text{PtCl}_6$ . The third is evaporated to dryness over a microburner, and a drop of a concentrated solution of uranium acetate slightly acidified with acetic acid, is added. The three drops are then examined under the microscope. The first will show the characteristic sheaf-like groups of gypsum crystals if lime is present, or the fine white granular plates and rhombs of  $\text{PbSO}_4$  if lead is the base used. The second will show the yellow octahedra of  $\text{K}_2\text{PtCl}_6$  if potassium is present, while the third drop will show the tetrahedra of the double acetate of uranium and sodium, if we are dealing with a soda soap.<sup>1</sup>

The amount of ash other than alkalis from the soap may be determined by the difference between the total ash and the alkali found by titration. Greases contain from zero to sometimes one per cent. of adventitious dirt, iron and aluminium oxides, silica, etc.

In the case of a grease of type C, containing graphite, talc, etc., the mineral filler may be determined directly after extraction with ether and then with alcohol, if we are dealing with an alkali soap, or in any case, by weighing the ash insoluble in acid. In the case of graphite, care should be taken to ash at a low temperature to minimize the oxidation of the graphite.

The unsaponified saponifiable matter, in the case of a grease made with an alkali soap, is best determined by saponification of the ether extract with alcoholic potash. The nature of the saponifiable may be found by evaporating off the alcohol after saponification, extraction of the unsaponifiable with ether, freeing the fatty acids from the soap left behind, and taking the melting point, iodine number and refractive index of the fatty acids, and thus identifying the fat from which the acids come. This is simple if only a single fat was used, but less so in a mixture. In most cases where a grease contains unsaponified saponifiable matter, it has been made by a partial saponification of the fat in question, and so the original grease may be saponified directly, and the total fatty acids examined.

In a calcium soap grease, the soap may be decomposed by heating with dilute  $\text{HCl}$ , washing, saponifying the freed fatty acids with alcoholic soda, evaporating the alcohol and extracting the

<sup>1</sup> For a full description of these extremely delicate and rapid microchemical methods, which are so valuable but so little known, see Behrens, *Microchemische Analyse*, or a series of articles by Chamot in *Jour. App. Microscopy*, Vols. III, IV.



mixture of soda soap and unsaponifiable, which is now in the condition of a grease originally made with soda soap, with ether. This gives us the unsaponifiable, and we may proceed with the examination of the fatty acids in the soap as above. The unsaponifiable ether extract will consist of mineral or hydrocarbon oil, and the specific gravity, flash point and refractive index will show us the nature of the mineral stock. In a rosin oil grease we may treat the mixture of rosin oil and mineral oil by any of the usual methods for quantitative separation or estimation, none of which, however, are very satisfactory.

The determination of water in lubricating greases is usually made either by the delightfully simple method of "determination by difference" or by drying down to constant weight. Archbutt and Deeley<sup>1</sup> advocate drying at 105°-110° C. until frothing ceases. Wright<sup>2</sup> dries at 105° till the weight is approximately constant at half-hour intervals of weighing. Hurst<sup>3</sup> dries at 220° F. to constant weight. Conradson<sup>4</sup> takes moisture by difference, but recognizes the fact that some oil is volatilized at 100° C. Holde<sup>5</sup> says: "The old method of mixing a weighed portion with alcohol, heating till no more bubbles appear, cooling and weighing, only gives useful results in the absence of light, volatile oils. More general is the application of the Marcusson xylol method.<sup>6</sup> By this method, six greases gave from 1 to 6.3 per cent. water, the difference between checks on the same grease being 0 to 0.2 per cent. By other methods the water content came from 0.5 to 2.2 per cent. too high."

Our attention was first drawn to this by a series of greases rather high in water content which were dried in the water oven to an apparent constancy of weight, but lacked several per cent. of adding up to 100. On investigation it was decided that the water determination was the weak point, and determinations were made by the xylol method, when the analyses added up well. Further work showed that though in this case the moisture determinations by drying were too low, in general

drying to constant weight gives high results, as claimed by Holde.

Table V shows the results obtained by drying down a lead-rosin oil grease (No. 9, Table X). Ten grams were dried in wide-mouthed drying bottles provided with a glass rod for stirring the grease. Two xylol distillations were made on 10 grams each, giving 2.48 and 2.46 cc. water, an average of 24.7 per cent. moisture.

TABLE V.—LEAD ROSIN OIL GREASE.

(A) Hours at 110° C.	Per cent. loss.	(B) Hours at 95°-100° C.	Per cent. loss.	
2	16.49	2	11.50	
6	22.66	6	14.98	
11	25.35	11	18.22	
13	25.11	13	18.94	
15	27.67	15	19.47	
17	28.27	17	19.98	
19	29.06	19	20.26	
21	29.61	Hours at 105°-110° Total		
25	29.90	2	21	23.79
30	30.53	4	23	25.63
..	...	8	27	26.36
..	...	12	33	28.54

TABLE VI.—SODA GREASE (No. 12, TABLE X).

This contained no water by anhydrous CuSO<sub>4</sub> or by xylol distillation.

Hours at 105°-110° C.	Per cent. loss.
1	0.26
7	1.13
8	1.23
12	1.69
18	2.12
23	2.44

TABLE VII.—(SEE FIG. 1) WINTER MOTOR GREASE (No. 3, TABLE X).

Moisture by xylol 0.03 per cent. Anhydrous CuSO<sub>4</sub> shows only faintest trace moisture.

A.		B.	
Hours at 105° C.	Per cent. loss.	Hours at 95°-100° C.	Per cent. loss.
2	2.3	2	1.3
5	2.9	5	1.6
7½	3.4	7½	1.8
11	3.8	11	2.1
13	3.9	13	2.25
16	4.1	16	2.4
20	4.4	20	2.6
23	4.6	23	2.8
28	5.0	28	3.1
31	5.1	31	3.25
34	5.25	34	3.35
38	5.35	38	3.45
40	5.50	40	3.55
43	5.82	43	3.67
46	5.90	46	3.75
50	6.04	50	3.85
52	6.34	52	3.96
54	6.47	54	4.06
61	6.71	61	4.36
*64	7.38	*64	4.75
67	7.43	*67	4.90
70	7.70	*70	5.10

\*61 to 64 on A at 120°.

\*54 to 61 on B at 105°

\*61 to 64 on B at 120°

\*64 to 70 on B at 105°

Another calcium soap grease showing a faint trace of moisture by anhydrous CuSO<sub>4</sub> and 0.05

<sup>1</sup> "Lubrication and Lubricants," p. 306.

<sup>2</sup> "Analysis of Oils and Allied Substances," p. 104.

<sup>3</sup> "Lubricating Oils, Fats and Greases," p. 281.

<sup>4</sup> *Jour. Amer. Chem. Soc.*, XXVI, 6, 705, June, 1904.

<sup>5</sup> "Untersuchung der Mineralöle und Fette," 2d Ed., p. 213.

<sup>6</sup> J. Marcusson, 1904. "Bestimmung des Wasser und Säures-Gehalten von Schmierfetten," *Mitt. K. Materialsprüfungsamt*, 24, 48; "von Die Bestimmung des Wassergehaltes von Ölen, Fetten, Seifern, Harzen usw.," *Mitt. K. Mater.*, 23, 58. See also Hoffmann, *Z. angew. Chem.*, 21, 2095; *Chem. Abstr.*, 3, 2, 158 (Jan. 20, 1909).



per cent. by xylol, gave a loss of 2.30 per cent. on heating 30 hours at 105°-110°.

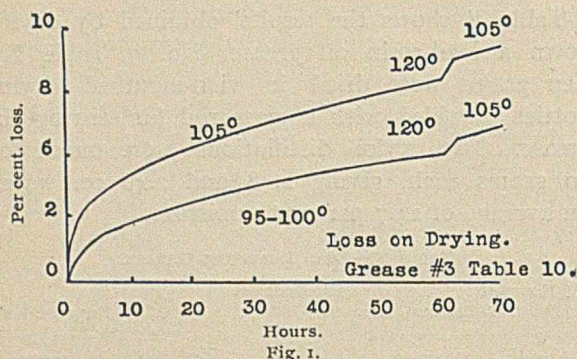


TABLE VIII.—TALLOW GREASE (No. 7, TABLE X).

{This showed moisture by CuSO<sub>4</sub> and gave 2.48% and 2.51% by xylol distillation.

Hours heated.	A at 110°. Per cent. loss.	B at 95°-100°. Per cent. loss.
2	3.70	1.71
5	4.78	2.55
7	5.11	3.09
11	5.21	2.55
14	5.30	4.50
17	5.36	4.81
19	5.39	4.96
23	5.50	5.16
25	....	5.24

Another sample of this lost 2.66 per cent. in 2 hours at 110° C., half a cc. of water having been added to the grease, and the 2.66 per cent. being corrected for this added water. This, after heating for two hours, gave no test for water by CuSO<sub>4</sub>, but further heating of this at 110° till it approached constant weight showed a loss of 5.3 per cent. in 23 hours, showing that volatile matter was being driven off.

TABLE IX.—A COMMERCIAL TALLOW, SHOWING NO TURBIDITY ON MELTING AND NO TRACE OF MOISTURE BY CuSO<sub>4</sub> OR XYLOL, HEATED AT 105°-110°, SHOWED:

Hours heated.	Per cent. loss.
2	0.59
4	0.84
6	0.85
9	1.02
12	1.10
14	1.12
17	1.13

One drop of water stirred into 15 grams of this tallow showed moisture by CuSO<sub>4</sub> and by distillation with xylol.

The curve in Fig. 1 shows that as the temperature was raised, the loss increased, as is seen from the pitch of the curves.

These tables show that in practically all types of commercial greases, drying down to constant weight gives high results. The results obtained by the xylol distillation in every case agreed with

what we would expect from CuSO<sub>4</sub> and other moisture tests. Moreover, there are no sharp breaks in the curves, by which we can tell just when the water has been driven off. In drying down ether extracts from greases, some loss is therefore also to be expected, but it is better to add this loss to the oil, than to ascribe it to moisture.

To determine the moisture in a lubricating grease, then, we first test it with anhydrous CuSO<sub>4</sub>, if the grease is sufficiently light in color. If water is present, or in the case of a dark grease, we weigh out 10 gr. on a balanced filter paper, put the grease and paper in a 300 cc. Erlenmeyer flask and cover with xylol. The xylol should be distilled from water and separated out from the water after clearing, in a separatory funnel. The flask, connected with a dry condenser, is heated in a bath of cylinder oil, and the xylol and water slowly distilled off till the xylol comes over clear. The bulk of the water comes over with the first 10 cc. of distillate. The distillate may be caught in a burette filled with water up to the lowest graduation, best with a few drops of xylol added to give the same meniscus as we will read, or in a receiver made like a funnel, with a graduated stem closed at the bottom, according to the method of Dean,<sup>1</sup> and allowed to stand over night, or till all milkiness has disappeared. A still better and more rapid method we find to be to catch the distillate in centrifuge tubes such as are used in urine analysis. The fine graduations allow of very accurate reading. Bulbs holding about 50 cc. are blown on the tops of these tubes, and the modified tubes containing the xylol-water mixture whirled in a larger centrifuge than the one they were designed for. This obviates the necessity of waiting for the xylol to clear up. A complete moisture determination may be made in 45 minutes.

Absolute accuracy in the determination of water in the technical examination of lubricating greases, is doubtless not important, though Holde has recently shown<sup>2</sup> the great influence exerted by even 1 per cent. of water on the drop point and the emulsified condition of lime soap greases. Every one who has driven off the water from a lime soap grease containing even a few hundredths of a per cent. of water, has noticed the change from a buttery opaque mass to a gelatinous trans-

<sup>1</sup> A. L. Dean, "The Estimation of Moisture in Creosoted Wood," Forest Service Circular 134, Jan., 1908.

<sup>2</sup> Holde, "The Physical Condition of Solid Fats, and of Solutions of Calcium Soaps in Oil," *Petroleum*, IV, 1, 14, Oct. 7, 1908; *Z. angew. Chem.*, 31, 2138-44 (1908); *Chem. Abstr.*, 3, 1, 123 (1909).



lucent one. Although not particularly important in the case of greases the fact that the xylol method gives more accurate results than that of drying down, is of importance in the analysis of other commercial products containing water.

Thirteen commercial greases were analyzed according to the methods given above, with the following results:

TABLE X.

No.	Name.	Flash-point, Degrees C.	Consistency, Grams, 20 C.	Melting point, Degrees C.	Per cent. calcium soap.	Per cent. other thickeners.
1	Graphite.....	195	18	93	11	16% Graphite
2	Summer motor.	160	170	87	38	..
3	Winter motor..	175	7	86	23	..
4	K 1.....	193	24	85	16	..
5	K 2.....	195	66	93	20	..
6	Auto.....	190	11	79	19	..
7	Tallow A.....	210	150 (estimated)	52	..	1.4 Potash soap.
8	Tallow XX.....	215	200	49	..	{ 2.1% Potash soap. 30% Paraffin
9	Lead rosin oil...	240	7	102	..	1.75% Lead
10	Lime rosin oil...	198	31	77	..	9.9% CaO
11	Lime rosin oil...	198	4	75	..	7.8% CaO
12	Soda grease.....	215	35	83	..	22% Sodium soap.
13	Non-fluid oil...	210	27	76	9.8	12.9% Sodium soap.
14	No. 4 Petrolatum.....	247	6	47	..	..
15	Lard oil.....	265	0	5	..	..

TABLE X—(Continued).

No.	Name.	Per cent. H <sub>2</sub> O.	Per cent. saponifiable oil.	Per cent. free acid as oleic.	Per cent. mineral oil.
1	Graphite.....	trace	17	0	56
2	Summer Motor.....	0.05	25	trace	36.5
3	Winter Motor.....	0.03	37 <sup>2</sup>	6.1	40
4	K 1.....	0.2	16	0	67
5	K 2.....	0.3	20	0.3	60
6	Auto.....	1.0	20	trace	60
7	Tallow A.....	2.5	73.5	0	22
8	Tallow XX.....	trace	48	0	20
9	Lead Rosin.....	24.7	0	0	....
10	Lime Rosin.....	trace	0	0	....
11	Lime Rosin.....	20.0	0	0	....
12	Soda Grease.....	0.0	0	0	78 <sup>3</sup>
13	Non-fluid Oil.....	0	7	0	70.3
14	No. 4 Petrolatum.....	0	0	0	100
15	Lard Oil.....	0	100	....	0

TABLE X—(Continued).

No.	Name.	Maximum temp. of bearing above room temp. Deg. C.	Coefficient of friction at end of 3-hour run.	Coefficient of friction. Average throughout 3-hour run.
1	Graphite.....	53	0.108	0.097
2	Summer motor.	39	0.075	0.075
3	Winter motor..	42	0.063	0.063
4	K 1.....	38	0.054	0.057
5	K 2.....	39	0.050	0.054
6	Auto.....	32	0.046	0.046

<sup>1</sup> Scented with nitrobenzol.

<sup>2</sup> Chiefly palm oil.

<sup>3</sup> Oil of 24.2° BÉ.

7	Tallow A.....	22	0.012	0.022
8	Tallow XX....	25	0.018	0.029
9	Lead rosin....	40	0.050	0.067
10	Lime rosin....	42	0.017	0.048
11	Lime rosin....	29	0.025	0.036
12	Soda grease....	17	0.016	0.019
13	Non-fluid oil...	25	0.016	0.026
14	No.4 petrolatum	16	0.018	0.018
15	Lard oil.....	7	0.010	0.011

Friction tests were then made on these and on a petrolatum (Standard Oil No. 4), and on a pure lard oil, as standards of reference.

The testing machine was a small Thurston, fitted with a compression grease cup, through which the grease was allowed to feed freely. The temperature was taken by a thermometer sunk in a small well filled with oil in the upper brass. The Fahrenheit thermometer supplied with the instrument was not sufficiently delicate, and was replaced by a small Centigrade one. The temperature of the bearing and the arc at which the pendulum stood, from which the coefficient of friction is figured, were noted at intervals of one minute for the first hour, and at every five or ten minutes thereafter. The temperature of the room was taken periodically, and the r. p. m. noted.

The total pressure on the bearing in all cases was 240 lbs., equivalent to 60 lbs. per square inch projected area of the bearing. This was the highest pressure that could be obtained with the machine. It is probable that at higher pressures the differences between the greases would be still more marked.

The speed was kept constant at 310-320 r. p. m. by a shunt-wound motor.

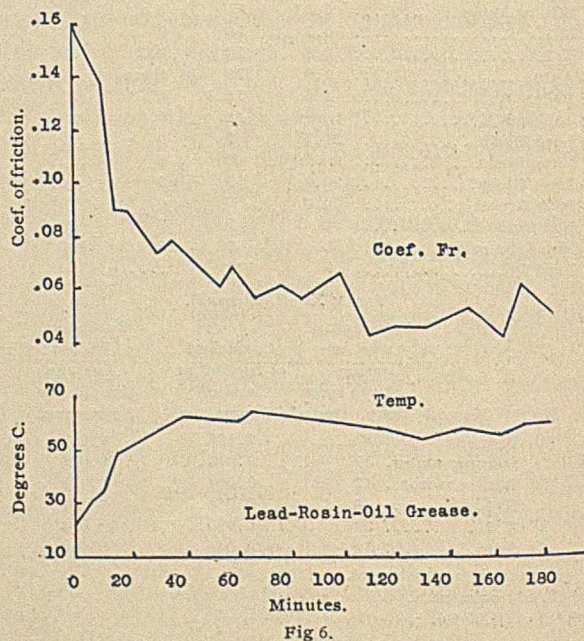
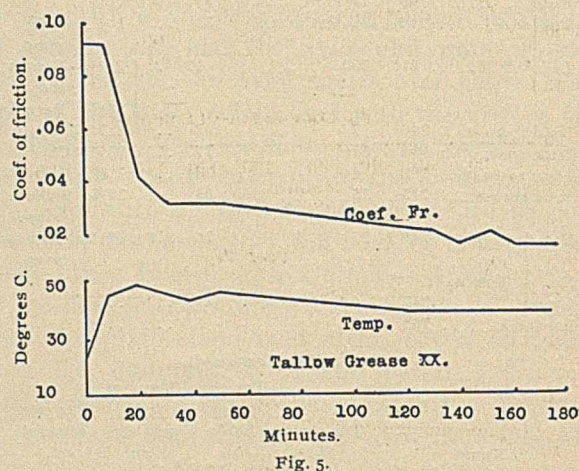
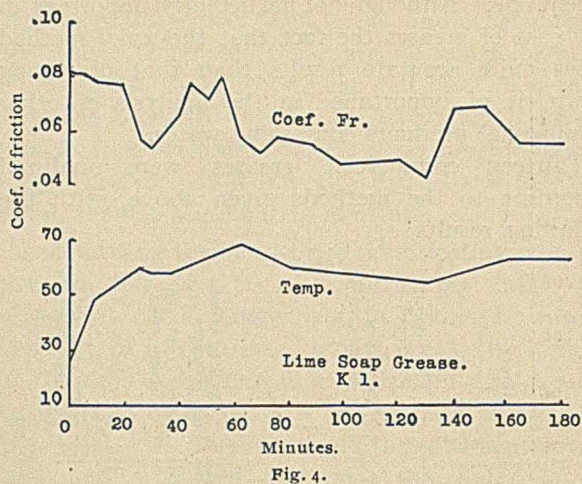
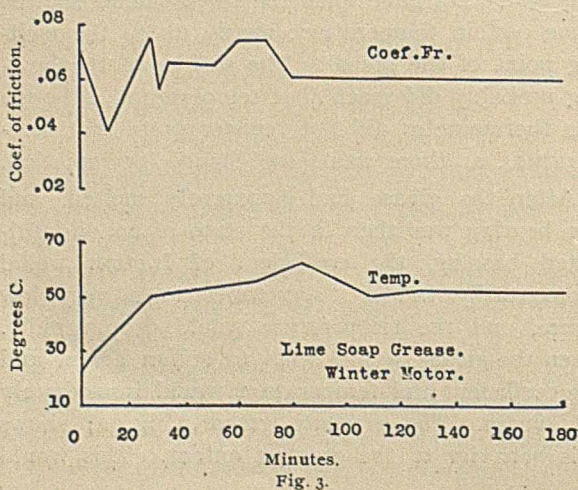
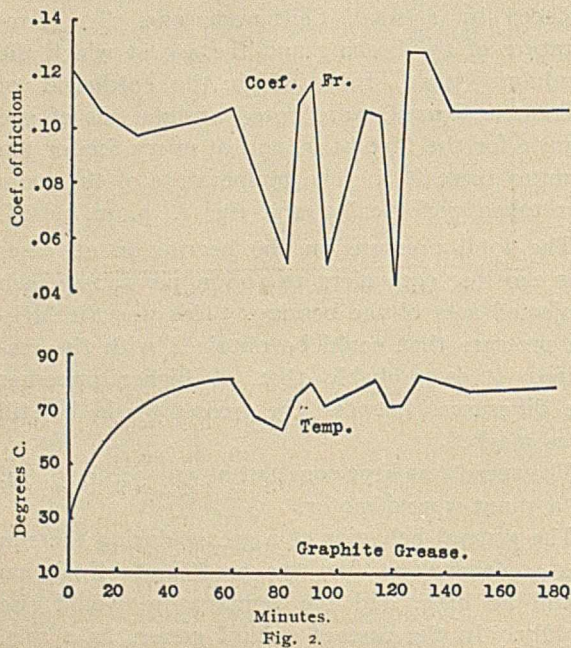
The general behavior of a grease during the run was as follows: at first the coefficient of friction would be high, and the temperature would rise rapidly. In the case of a hard grease, as a rule, this would continue until the thermometer showed some certain temperature, nearly up to the melting point of the grease. The surface of the bearing probably did reach that temperature, although the thermometer did not register quite that temperature, as there was some chance for radiation.

After the grease had apparently melted, and the bearing was then in the state of an oil-lubricated bearing, the coefficient of friction would momentarily fall off, sometimes to a very low figure, and the temperature would drop rapidly. Then the grease would seem to stiffen again, and the coefficient and temperature would immediately rise again. The graphite grease (Fig. 2) shows this behavior to the greatest extent. This would



go on for perhaps an hour, when a condition of equilibrium would be established, and a fairly constant reading would be attained. Many of these momentary fluctuations are smoothed out of the curves as here plotted, by taking averages of five- or ten-minute periods and plotting those. Figs. 2 to 9 show the curves for seven typical greases. The temperature plotted in the curves is the actual temperature registered by the thermometer; that given in column 12, Table X is the temperature of the bearing above that of the room.

Since the friction cannot be reduced till the temperature of the bearing has risen enough for the grease to melt, or at least to be softened so it can flow over the bearing, it follows that other things being equal, the grease with the highest melting





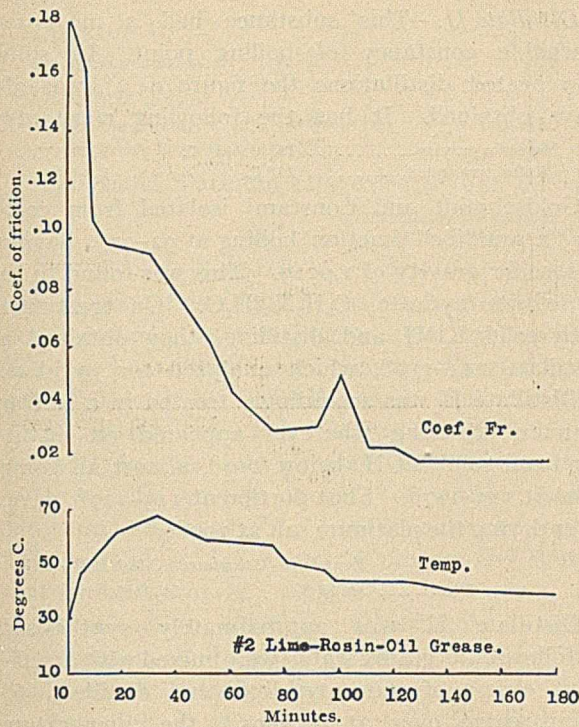


Fig. 7.

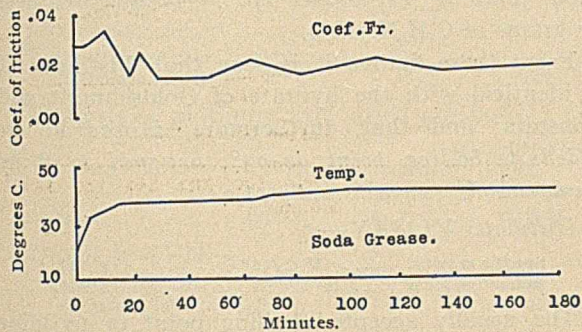


Fig. 8.

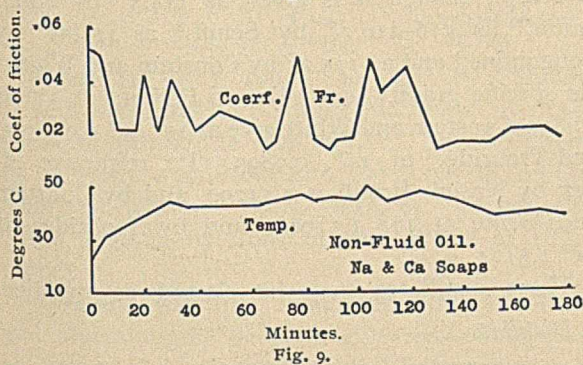


Fig. 9.

point will produce the highest coefficient of friction. Hence the lowest melting grease that will stay on the bearing will have the lowest coefficient of friction, which is only another way of saying that a grease already melted, *i. e.* an oil, will give the best results wherever it can possibly be used.

The average coefficient of friction tabulated in column 14 of Table X is found by plotting the coefficients for each reading against the time in minutes, and taking the average from the curve. Duplicate runs on the same grease gave very good checks, the fluctuations did not, of course, come at exactly the same minute of the different runs, but the general trend of the curves was the same, and the average coefficient of friction for a three-hour run would check within about 5 per cent.

There is no direct proportionality between the results of the determination of any one analytical constant, and the lubricating power though there seems to be an approximate relation between the melting point and the friction reducing power, as would be expected. This is shown in Fig. 10.

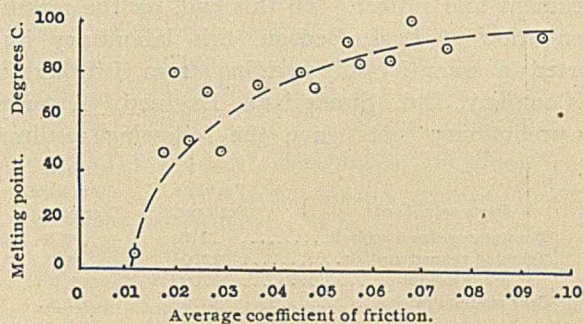


Fig. 10.

The relation, however, is not close enough to allow us to predict the lubricating value from the melting point without taking the chemical composition and the physical constants into consideration.

The graphite grease showed an unexpectedly low lubricating power, and would be best fitted for a gear grease. The rosin oil greases, which are usually considered to be very poor lubricants, showed high friction at first, but after the bearing had warmed up enough to soften them somewhat, they compared well with the more expensive greases. The high moisture content of most of these greases would seem to be no drawback, but rather an advantage in rendering them less sticky.

It will be noted that the lime soap greases, the most common type to-day, do not give as good results as the older, though more expensive type of tallow greases. It will also be seen that the greases compounded with soda soaps are better lubricants than those compounded with lime soaps.

This work was begun at the suggestion of Mr. A. D. Little, of Boston, and was carried on almost entirely in the Arthur D. Little Laboratory



of Engineering Chemistry while the writer was a member of the laboratory staff.

Sincere thanks are due Mr. R. P. Tobin, of Boston, for the loan of the Thurston testing machine.

CORNELL UNIVERSITY.

[CONTRIBUTION FROM THE NEW YORK TESTING LABORATORY.]

## COMMERCIAL PYRIDINE AND ITS VALUE AS A SOLVENT.

By KENNETH G. MACKENISIE.

Received April 3, 1909.

Bedson has shown<sup>1</sup> that pyridine may be used as a solvent to distinguish and separate the constituents of coal. His work suggested a similar use in the examination and analysis of the various bitumens and rubber. To this end, on the recommendation of Prof. Bedson, this laboratory imported a quantity of pyridine from Brotherton & Company, Ltd., Sunderland, England. A series of preliminary tests gave the following results:<sup>2</sup>

Parts soluble of	Pyridine. Per cent.	Carbon disulphide. Per cent.
Bermudez refined asphalt.....	23.0	98.6
Trinidad refined asphalt.....	22.0	56.0
"J" grade asphalt.....	27.0	99.0
Gulf residual pitch.....	20.0	97.5

Rubber was apparently insoluble in commercial pyridine.

These results led to suspicions of the purity of the pyridine, and a kilo was distilled, using an eighteen-column Young dephlegmator.

### COMPOSITION OF SAMPLE OF PYRIDINE.

Distillate.	Temperature.	Per cent.
I.....	86.0- 94.4° C.	1.0
II.....	94.4	26.1
III.....	94.4-105	0.6
IV.....	105.0-112.0	0.8
V.....	112.0-115.0	1.4
VI.....	115.0-116.0	4.5
VII.....	116.0-116.7	3.2
VIII.....	116.7-117.2	12.5
IX.....	117.2-118.0	10.5
X.....	118.0-121.0	5.0
XI.....	121.0-123.0	1.3
XII.....	125.0-131.0	4.6
XIII.....	131.0-134.0	1.7
XIV.....	134.0-136.0	8.5
XV.....	136.0-140.0	4.6
XVI.....	140.0-144.5	1.0
XVII.....	144.5	5.2
	Residue	7.5

— 100%

*Distillate I.*—Consists of two liquids of different gravity, which were miscible on agitation. The odor of benzene was plainly perceptible above the characteristic pyridine odor.

<sup>1</sup> *J. Soc. Chem. Ind.*, **27**, 147.

<sup>2</sup> By Mr. R. H. Parker.

*Distillate II.*—This substance had a most remarkable constancy of boiling point. In spite of repeated distillations the figure 94.4° was always obtained. It has the following constants:

Specific gravity.....	15° C./15° C.	1.008
Refractive index.....	25° C.	1.4418

Goldschmidt and Constam<sup>1</sup> isolated from coal-tar "a pyridine" fraction boiling at 92-93°, having a specific gravity of 1.0219. This was found to be a definite hydrate, C<sub>5</sub>H<sub>5</sub>N.3H<sub>2</sub>O. On treatment with solid KOH and distilling, they obtained a distillate 114-116°, which analyzed for pyridine.

Distillate II was accordingly treated in a similar manner. Nothing whatever appeared on distilling from solid KOH, below 110°, almost all going over at 115-117°. That portion at 115-117°, gave, after drying the platinum salt at 100°:

	Found.	Calculated (C <sub>5</sub> H <sub>5</sub> NHCl) <sub>2</sub> PtCl <sub>4</sub>
Pt.....	34.18 %	34.34%

Distillate II was approximately synthesized as follows: 40 grams water were mixed with a sufficient excess of pure pyridine, and distilled. 66 grams came over at 94.4-95°, the temperature then jumping to above 110°. Theory requires 67 grams of C<sub>5</sub>H<sub>5</sub>N.3H<sub>2</sub>O.

From these figures it is seen that Distillate II is identical with the hydrate of Goldschmidt and Constam; and that, furthermore, *instead of the indefinite boiling point 92-93°, assigned to it by them, it boils very constantly at 94.4°.*

### Distillates VI to IX.—

Specific gravity.....	15° C./15° C.	0.983
Refractive index.....	25° C.	1.5051

The usually accepted boiling point of pyridine is 116.7°, though it is given as 114.5° by Kahlbaum,<sup>2</sup> as 116-116.2° by Schiff,<sup>3</sup> as 115.51° by Louguinine,<sup>4</sup> and as 115.2° by Constam and White.<sup>5</sup> Its specific gravity, according to Perkin,<sup>6</sup> at 51° is 0.9855, Constam and White give at 15°/4° 0.989305, and Trobridge<sup>7</sup> at 14°, 0.9893. Its refractive index by Nasini, Brühl<sup>2</sup> is 1.51290, and by Constam and White at 17° 1.51068, and by Trobridge at 14° 1.5124.

These distillates may be classed as pyridine.

### Distillate X.—

Specific gravity.....	15° C./15° C.	0.9765
Refractive index.....	25° C.	1.5032

<sup>1</sup> *Ber.*, **16**, 2976.

<sup>2</sup> Siedetemp. u. Druck, 95.

<sup>3</sup> *Ber.*, **19**, 566.

<sup>4</sup> *Compt. rend.*, **128**, 367.

<sup>5</sup> *Am. Chem. J.*, **29**, 2.

<sup>6</sup> *J. Chem. Soc.*, **55**, 701.

<sup>7</sup> *J. Soc. Chem. Ind.*, **28**, 230.

<sup>8</sup> *Zeit. Ph. Ch.*, **16**, 214.



*Distillate XI.*—

Refractive index..... 25° C. 1.5020

*Distillate XII.*—

Specific gravity..... 15° C./15° C. 0.955  
Refractive index..... 25° C. 1.5009

Analysis of platinum salt, dried at 100° C:

	Found.	Calculated for picoline. (C <sub>6</sub> H <sub>7</sub> NHCl) <sub>2</sub> PtCl <sub>4</sub>
Pt.....	32.77%	32.72%

A picoline, first found in coal-tar by Goldschmidt and Constam, has a boiling point of 129°, according to Ladenburg and Lange.<sup>1</sup> Thorpe<sup>2</sup> gives 133.5° corrected. Weidel<sup>3</sup> reports that a picoline comes over 133–140°. Garrett Smythe<sup>4</sup> gives 129.5° (763 mm.) and Constam and White 128.8°.

Its specific gravity at 0°/4° is 0.9652 (Ladenburg and Lange); at 0°/4° 0.9616, and at 10° 0.95257 (Thorpe); at 15°/4° 0.94972 (Constam and White); and Trobridge at 14°, 0.9530.

Its refractive index by Gladstone<sup>5</sup> is 1.5006, by Constam and White 1.50237, and by Trobridge at 14° 1.4966.

From this it is concluded that Distillate XII must be a picoline.

*Distillate XIV.*—

Specific gravity..... 15° C./15° C. 0.946  
Refractive index..... 25° C. 1.5022

Analysis of platinum salt, dried at 100° C.:

	Found.	Calculated picoline.	Calc. (C <sub>7</sub> H <sub>9</sub> NHCl) <sub>2</sub> PtCl <sub>4</sub> lutidine.
Pt.....	32.55%	32.72%	31.26%

*Distillate XVII.*—

Specific gravity..... 15° C./15° C. 0.947  
Refractive index..... 25° C. 1.5026

Analysis of platinum salt, dried at 100° C.:

	Found.	Calculated picoline.
Pt.....	32.90%	32.72%

β-Picoline, was found in tar by Mohler.<sup>6</sup> It boils according to Schwarz and Stöhr<sup>7</sup> at 143.5°. Constam and White give 143.4°. Specific gravity by Schwarz and Stöhr at 0°/4° is 0.9726; by Constam and White at 15°/4° 0.96134. Its refractive index is given by Constam and White at 18° as 1.5720.

γ-Picoline was first found in tar by Schulze<sup>8</sup> and Ladenburg.<sup>9</sup> Beilstein gives its boiling point as 142.5–144.5°; Constam and White give 143.1°.

<sup>1</sup> A., 247, 6.  
<sup>2</sup> J. Chem. Soc., 37, 223.  
<sup>3</sup> Ber., 12, 2008.  
<sup>4</sup> J. Chem. Soc., 81, 452.  
<sup>5</sup> Ibid., 45, 246.  
<sup>6</sup> Ber., 21, 1007.  
<sup>7</sup> Ibid., 20, 413.  
<sup>8</sup> J. pr. (2), 43, 154.  
<sup>9</sup> A., 247, 10.

Its specific gravity (Beilstein) at 0°/4° is 0.9742; at 15°/4°, 0.95714 (Constam and White); at 12.5°, 0.9603 (Trobridge).

Its refractive index (Constam and White) is 1.50640 at 17°. Trobridge gives 1.5065 at 12.5°.

Distillate XVII must be either β- or γ-picoline, or a mixture of both. Its specific gravity and refractive index are nearer that of the γ-position. Trobridge, moreover, finds only the γ-isomer in his pyridine base fraction 145–150°. This distillate must be γ-picoline.

Distillate XIV, though not corresponding exactly with any of the picolines, comes nearest to the α-position, and must of necessity be placed there.

THE SOLVENT ACTION OF PYRIDINE, ETC.

In making the solubility tests, three portions were used: (a) the hydrate boiling at 94.4°; (b) distillates VI–IX boiling at 115–118°, designated "pure pyridine;" and (c) distillates XIV–XVII boiling at 134–144.5°, designated "picolines."

	CS <sub>2</sub> .	Hydrate.	Pyridine.	Picoline.
<i>Bonanza Gilsonite.</i> —				
Per cent. soluble.....	99.7	1.7	96.1	99.5
<i>Bermudez Refined Asphalt.</i> —				
Per cent. soluble.....	95.3	2.8	94.8	95.6
<i>Gulf Residual Pitch.</i> —				
Per cent. soluble.....	96.8	2.1	96.8	97.4

*Dry Sample of Rubber*, having on a dry basis the following composition, as determined by the use of acetone as a solvent:

	Per cent.
Resins.....	18.5
Rubber.....	67.9
Insoluble.....	13.7

	Pyridine hydrate.	Pyridine.	Picolines.
Per cent. soluble.....	7.0	71.8	88.0

*Wurtzelite.*—

	CS <sub>2</sub> .	Chloroform.	Picolines.
<i>Wurtzelite</i> , No. 72684—			
Per cent. soluble.....	6.53	8.73	10.1

The above figures show that the inability of commercial pyridine to dissolve bitumens to any considerable extent is due to the presence of water, which forms with the pyridine a hydrate with very little solvent action. Likewise, the further up we go in the pyridine series, the better solvents we obtain.

*Summary.*—Commercial pyridine consists of pyridine, picolines and water. The water forms with pyridine a definite hydrate which boils exactly at 94.4°. This hydrate has very little solvent action on bitumens, and by its presence in pyridine materially decreases the solvent action



of that base. The picolines are even better solvents than pyridine, in some cases being equal to and even surpassing carbon disulphide.

FLASH AND FIRE OF PYRIDINE DERIVATIVES.

	Flash.	Fire.
Hydrate, 40 per cent. water....	35° C.	55° C.
Pyridine.....	17°	17°
Picoline 125-131.....	25°	30°
" 134-136.....	33°	40°
" 1445.....	42°	48°
Crude pyridine.....	31°	38°

Apparatus = 4 oz. open tin dish 2-1/4" diameter.

NEW YORK TESTING LABORATORY,  
MARCH 31, 1909.

PURITY OF COMMERCIAL LIQUEFIED AMMONIA GAS AND APPARATI FOR TESTING IT.<sup>1</sup>

By DR. F. W. FRERICHS.

The following paper discusses the purity of commercial liquefied ammonia and describes the principal apparati, which have been proposed for testing its purity in the United States and in Europe.

The test is based upon the low boiling point of liquefied ammonia gas, which is 28° F. below zero, and upon the consideration that the impurities, which are likely to be present, have a much higher boiling point. Therefore if a sample of liquid ammonia be evaporated the impurities contained in same would probably remain in the vessel, and their quantity could be ascertained. Liquefied ammonia gas now is considered good by American ice manufacturers if no visible residue is left in a flask in which a 4 oz. sample has been evaporated.

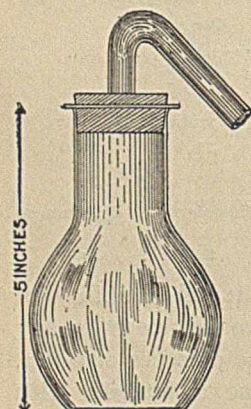


Fig. 1.

Fig. 1 shows a test bottle similar to the one in common use in the United States, and Fig. 2 shows the mode of drawing a sample from a cylinder of ammonia.

This mode of testing gives only approximate results. One source of error is the moisture contained in the surrounding atmosphere. On account of the rapid evaporation of the liquid ammonia, the iron tube by which the sample is drawn becomes very cold and so does the flask, and moisture from the air will readily condense upon the cold surfaces. By drawing the

sample quickly, and closing the flask at once with a perforated cork carrying a bent glass tube, contamination of the sample with moisture is as much as possible, but never entirely, prevented. But the fundamental source of error in this method of testing rests in the fact that small quantities of the

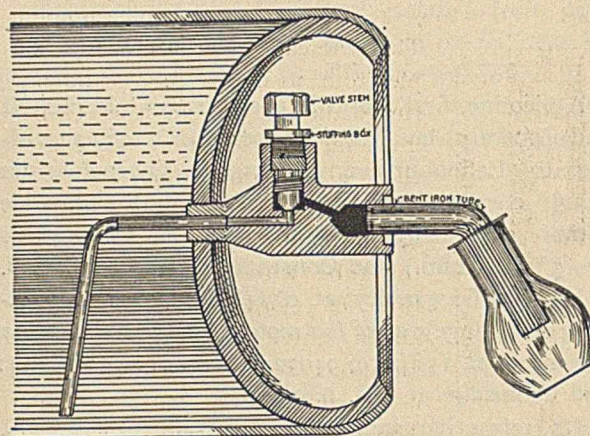


Fig. 2.

impurities often contained in the commercial ammonia will evaporate with the ammonia used for the sample, on account of which fact the evaporation test gives results, which are too low.

While this method of testing has become customary in the United States, the Linde method<sup>1</sup> was used in Europe. The apparatus shown by Fig. 3 is used for the Linde test, and consists of a wider tube with a small hole near the center, the tube being drawn out at one end into a smaller tube which is closed in the bottom. The graduation was as indicated in the picture. In making the test the tube was supposed to be filled to the opening in the center, and the sample was permitted to evaporate in the open air. In operating this apparatus it would frequently occur that the ammonia would foam and boil up and part of the sample would run out of the opening. Since the time of evaporation was directed to be about three hours, the open vessel would permit much moisture to be attracted from the air. Besides, the graduation was incorrect because it did not take into consideration the difference in the specific gravities of the liquid ammonia, and of the residue left upon evaporation. For these reasons the results could not be accurate, but the degree of purity expected by European ice makers was not very

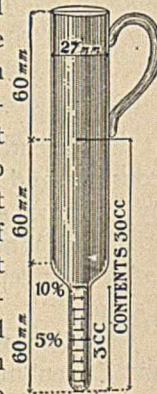


Fig. 3.

<sup>1</sup> This paper was read at the first annual meeting of the American Insitute of Chemical Engineers and will be published in full in the Transactions, Vol. 1, 1908.

<sup>1</sup> Zeitschrift f. angew. Chemie, 1897, p. 224.



great, an article leaving, upon evaporation, 1 per cent. of liquid residue being quite acceptable to them.<sup>1</sup>

Lange and Hertz<sup>2</sup> improved upon this apparatus and changed it to the form shown by Fig. 4. They omitted the overflow hole and replaced it by a mark indicating 49 cc. = 33.3 grams ammonia.

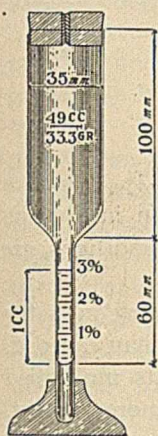


Fig. 4.

They increased the size of the sample from 30 cc. to 49 cc., and narrowed the tail end in order to permit a larger graduation. The latter they made proportionate to the specific gravities of the fluids to be measured. Finally they closed the tube with a cork during the time of evaporation, which was about three hours, letting the ammonia vapors escape through a narrow notch in the stopper. The result of tests made with this apparatus seemed to be sufficiently correct, since they worked with liquefied ammonia gas, leaving, upon evaporation, about 1 per cent. of non-volatile liquid. They also reported upon the quality of several German makes, finding that the average commercial article left, upon evaporation, about 1 per cent. of an oily substance. The nature of this residue of which they secured 185 cc. by evaporating 19.5 kilograms of commercial liquid ammonia was analyzed and ethyl alcohol, acetonitril, and pyridine were found in larger quantities, while the presence of smaller amounts of benzol, naphthalin, and carbonate of ammonium, and also lubricating oil could be established.

Since it was evident that both the Linde, and the Lange and Hertz test would show, upon evaporation, more residue than the ammonia really contained, the *Aktiengesellschaft für Chemische Industrie* in Mannheim caused Bunte and Eitner to design a new method which they published in 1897.<sup>3</sup> Their apparatus is represented in Fig. 5 and consists of a pipette having a strong glass body and a faucet on each end.

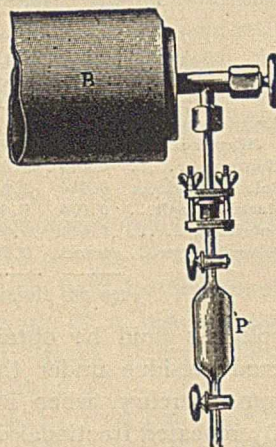


Fig. 5.

The sample was drawn under pressure, and its weight was ascertained. The pipette was then put in an upright position, the upper end connected with three caustic potash drying tubes, and by opening the upper faucet the sample was permitted to evaporate. After evaporation the pipette containing the residue was heated in an air bath to 70°–80° C. subjected to a current of air at that temperature and the weight of the non-volatile liquor was ascertained.

It is evident that the method favored the seller, since it generally would show a good test, the current of air carrying most of the residue out of the apparatus.

K. Urban<sup>1</sup> has improved upon this method, using an apparatus represented by Fig. 6. He graduated his pipette and heated the residue only to 30° C. to expel ammonia, but he did not use a current of air nor did he use caustic potash tubes. The specific gravity of the residue he determined by reading the volume on the graduation and ascertaining the weight. But he made an error by not considering the difference of specific gravities of air and ammonia gas, which was for his apparatus about thirty milligrams. Urban compared his method with other methods of testing then in use, and tabulated his results as shown in Table I.

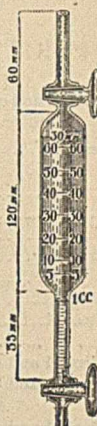


Fig. 6.

TABLE I.

## A. BY URBAN'S METHOD.

- 43 cc. = 26.5 grams NH<sub>3</sub> left 0.25 gram = 0.94 per cent. residue.  
 43 cc. = 26.5 grams NH<sub>3</sub> left 0.25 gram = 0.94 per cent. residue.  
 45 cc. = 27.7 grams NH<sub>3</sub> left 0.25 gram = 0.91 per cent. residue.

## B. BY LINDE'S METHOD.

- 30 cc. = 20.4 grams NH<sub>3</sub> left 0.534 gram = 2.61 per cent. residue.  
 30 cc. = 20.4 grams NH<sub>3</sub> left 0.530 gram = 2.59 per cent. residue.  
 30 cc. = 20.4 grams NH<sub>3</sub> left 0.535 gram = 2.62 per cent. residue.

## C. BY THE LANGE AND HERTZ METHOD.

- 50 cc. = 34 grams NH<sub>3</sub> left 0.45 gram = 1.32 per cent. residue.  
 50 cc. = 34 grams NH<sub>3</sub> left 0.46 gram = 1.35 per cent. residue.  
 50 cc. = 34 grams NH<sub>3</sub> left 0.45 gram = 1.32 per cent. residue.  
 50 cc. = 34 grams NH<sub>3</sub> left 0.51 gram = 1.50 per cent. residue.  
 50 cc. = 34 grams NH<sub>3</sub> left 0.50 gram = 1.47 per cent. residue.

The last publication upon this subject has been by Lange and Heffter 1898<sup>2</sup> who reject the Linde method as being incorrect, and prove that the Bunte Eitner method is unreliable since its results are always too low. Then they compare the Urban and the Lange-Hertz methods, and prove by a number of experiments on samples of liquid ammonia containing known quantities of benzol,

<sup>1</sup> Lunge, "Coal, Tar and Ammonia," 4th German Edition, p. 161.

<sup>2</sup> Zeits. f. angew. Chemie, 1897, p. 224.

<sup>3</sup> Journal für Gasbeleuchtung, 1897, p. 174.

<sup>1</sup> Chemiker Ztg., 1897, p. 720.

<sup>2</sup> Chemische Industrie, 1898, p. 2.



alcohol, pyridine, acetonitril, and also of mixtures of either two of these in equal parts, that Urban's method gives always too low results, while Lange-Hertz's method produces too high figures. In making the investigation they have operated with liquid ammonia leaving 0.2 per cent. residue upon evaporation, this being the best commercial ammonia which they could obtain. The results of their experiments are compiled in Table II.

TABLE II.

Kind and quantity of addition present.	Per cent.	Found.			
		Found.		Found.	
		By Urban method.	By Lange and Hertz.	By Urban.	By Lange and Hertz.
Benzol .....	1.5	0.99	1.7	66	113
" .....	1.5	0.75	1.5	50	100
" .....	1.5	1.13	1.5	75	100
" .....	1.5	0.77	1.0	51	67
" .....	1.5	0.76	1.0	51	67
Pyridine .....	1.0	0.65	1.2	65	120
" .....	1.0	0.70	1.1	70	110
Water .....	1.1	0.91	1.2	83	109
Alcohol .....	1.0	0.80	1.6	80	160
" .....	1.0	0.79	1.8	79	180
Alcohol and acetonitril...	1.0	0.79	1.5	79	150
Alcohol and benzole.....	1.3	0.79	1.4	61	108
" " " .....	1.3	0.66	1.5	51	115
Alcohol and pyridine.....	1.0	1.01	1.5	101	150
" " " .....	1.0	1.02	1.6	102	160
Benzol and pyridine.....	1.0	0.80	0.9	80	90
" " " .....	1.0	0.66	1.1	66	110

They conclude that in Urban's method part of the impurities evaporate with the ammonia, and prove this theory by leading the vapors of 20 grams ammonia through a tube filled with pumice stone and cooled to  $-30^{\circ}$  C., whereby the tube increased in weight by 26 milligrams.

They found that the amount of foreign substances evaporated with the ammonia varied with the nature of the substances. Benzol and pyridine escaped easily, alcohol and water with greater difficulty. Of the mixtures, alcohol and pyridine in equal parts did not seem to evaporate with the ammonia. However, with this statement I cannot agree. I have repeated this series of tests and the results are compiled in Table III. My figures show that a mixture of pyridine and alcohol evaporates just as well with ammonia as other substances. As a result of their investigation Lange and Heffter conclude that the results obtained by Urban's method are always too low while those obtained by the method of Lange-Hertz are too high, and they suggest to make both tests and take the average. This would seem quite arbitrary,

but Lange-Heffter do not consider it worth while to look for a better method as long as no purer ammonia is offered in the market than an article leaving upon evaporation 0.2 per cent. residue.

TABLE III.

Liquid HN <sub>3</sub> . Grams.	Addition present. Kind.	Grams.	Addition evaporated.	
			By Urban. Per cent.	By Frerichs. Per cent.
98.5	Benzol .....	1.5	41	59
99.0	Pyridine.....	1.0	33	24
99.0	Alcohol.....	1.0	20	17
98.9	Water.....	1.1	17	11
99.0	{ $\frac{1}{2}$ Alcohol <sup>1</sup> } { $\frac{1}{2}$ Pyridine } .....	1.0	00	15
Used liquid NH <sub>3</sub> leaving, upon evaporation,			0.2	0.00

This was the aspect of the situation in Europe in 1898 and it would seem that the conditions are still the same at the present day.<sup>2</sup>

In the United States, the refrigerating industry has made much quicker progress than abroad. In the same measure as the ice plants multiplied and ice-making machinery became more perfect, the importance of the purity of the ammonia used in the plants became more evident because impure ammonia produced permanent gases in ice machines, and thereby decreased the efficiency of the plants.

As early as 1892, Hans von Strombeck, then in the employ of the De la Vergne Machine Company, published the analysis of six samples so called "anhydrous liquid ammonia 100 per cent." manufactured of different materials by different processes. He evaporated the samples in a flask connected with a long upright spiral condenser cooled with a refrigerating mixture, and obtained residues corresponding to the figures given in Table IV. The

TABLE IV.

	A. Per cent.	B. Per cent.	C. Per cent.	D. Per cent.	E. Per cent.	F. Per cent.
Ammonia by difference.....	98.976	96.984	98.220	99.792	99.321	99.180
Moisture.....	0.040	0.024	0.079	0.078	0.010	0.032
Colorless fluid....	0.950	2.880	1.644	0.117	0.622	0.666
Hartshorn salt...	0.030	0.099	0.049	0.004	0.043	0.087
Lubricant oil.....	0.004	0.006	0.005	0.009	0.004	0.035
Mineral matter...	traces	0.007	0.003	traces	traces	traces
	100.000	100.000	100.000	100.000	100.000	100.000

colorless fluid he obtained by distillation of the crude residue under the reduced pressure of 200 mm. mercury, when it distilled at  $106^{\circ}$ - $122^{\circ}$  F. By repeated fractional distillation he could separate it into fractions, the boiling points of which corresponded with methyl alcohol, acetone, ethyl

<sup>1</sup> By an error in the first publication, THIS JOURNAL, 1, 224, benzol has been printed instead of alcohol. Kindly correct.

<sup>2</sup> See H. Teichman's book on "Compressed Gases," p. 101, published 1908.



alcohol, and isopropyl alcohol. Believing only alcohol and acetones present as contaminations of commercial liquid ammonia, v. Strombeck proposed to purify it by distilling over metallic sodium. This process has been patented, and it was claimed that liquid ammonia 99.995 per cent. purity could be obtained by it.

v. Strombeck evidently overlooked benzol and pyridine among the impurities, and for that reason could not have obtained as pure ammonia as he thought. I have not been able to find that this process has been carried out on a large scale, but his investigation served as an incentive for other manufacturers to improve their product, and to furnish to the ice trade an article of such purity as they do now.

In the winter of 1897-'98 the writer had to rebuild the ammonia works of the Herf & Frerichs Chemical Company, and in order to control the working of the plant the larger of these two test apparati has been constructed. It aims to test the ammonia under such conditions as are prevailing in ice machines, inasmuch as the evaporation takes place under pressure, while the sample evaporated at a low temperature produced by a freezing mixture. The test apparatus was permanently connected with the plant, and a sample was taken from every 212 pounds ammonia manufactured. Apparati were changed and purifiers were added to the plant until the resulting ammonia upon evaporation in this apparatus did not leave any visible residue. In order to be still surer that the ammonia was pure an upright Liebig condenser cooled first by liquid ammonia, and subsequently by liquid carbonic acid, was connected with the flask containing the samples, and even in this case the evaporating ammonia did not leave any visible residue.

In one instance 235 consecutive samples, one from every 212 pounds of ammonia manufactured, representing altogether 49820 pounds of ammonia, were evaporated in the same flask leaving, upon evaporation, only 0.0187 gram of a residue containing moisture, iron rust, traces of ammonium carbonate, and a little lubricating oil.

The apparatus is illustrated by Figs. 7 and 8, and Fig. 9 shows the same apparatus without the Liebig condenser attached to a shipping cylinder containing liquefied ammonia gas.

"A" represents a cylinder containing liquid anhydrous ammonia. The test apparatus is attached to the valve "B," as indicated in the cut.

Channel 4 terminates in the cross-channel 5, in the ends of which the valves 6 and 7 are located. By opening valves "B" and 6, connection with the outer air can be made through the opening 8, whereby any rust, etc., from the valve can be

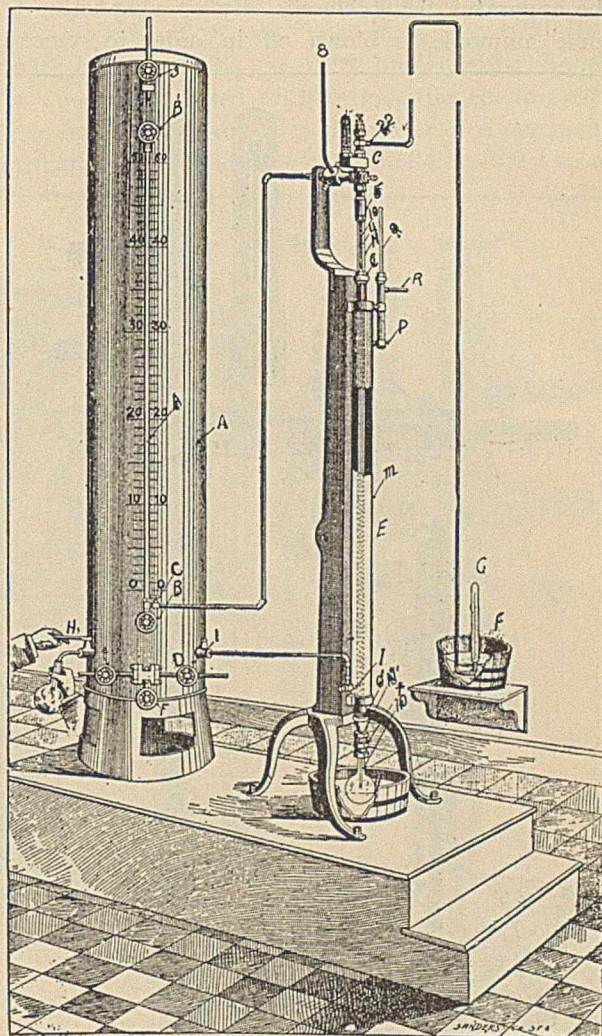


Fig. 7.

blown off. By valve 7, connection can be established between channels 4 and 9, the latter extending by means of the tube 10 to the center of the flask 11, which is strong enough to withstand a pressure of 30 pounds, to the square inch.

The interior of the flask connects by way of channels 15, 16, 17, 18 with the automatic valve 19, and the spring 20 (the pressure of which can be regulated by the screw 21) holds the valve 19 in its seat, allowing only gases to escape through channel 22 if their tension is great enough to overcome the pressure of the spring. Valve 23 controls connection with the mercury gauge 24, which



indicates the pressure under which evaporation takes place.

In operating the apparatus it is attached to the cylinder of ammonia to be tested as indicated by the cut, and valve "B" is opened, all other valves of the apparatus being closed. By opening valve 6, some ammonia is blown off in order to remove

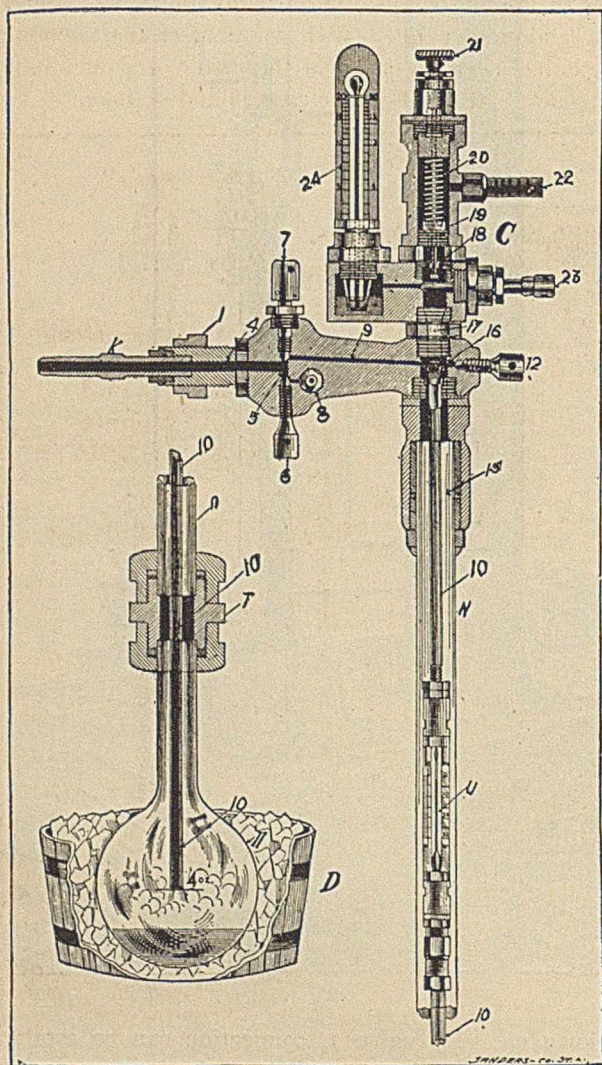


Fig. 8.

the rust or other foreign matter which may have gathered in valve B. If this has been accomplished, valve 6 is closed while valve 23 is opened and by operating valve 7, such quantities of ammonia are admitted into flask 11 as may be found convenient for the test.

The evaporation of the ammonia in flask 11 begins at once, and by means of the screw 21 the pressure under which the evaporation is to take place can be regulated to the desired degree, which

is indicated by the mercury gauge 24. The evaporated ammonia escapes at 22.

If all the ammonia which has been admitted for a sample into flask 11 has been evaporated, a new sample can be filled into the same flask by operating valve 7, the apparatus being in the same condition in which it was at the beginning of the test, except that flask 11 now contains whatever impurities may have been left from the evaporation of the first sample. In this way a great many tests may be made in succession, and the entire contents of the cylinder can be evaporated in the small flask 11 in which remain, at the end of these tests, all the non-volatile impurities which have been contained in a very large amount of ammonia.

The smaller of these apparatus has been constructed in more recent time; its particular object is to make the test independent from the moisture of the atmosphere. It is illustrated by Figs. 10 and 11, which explain themselves.

The apparatus is attached to the vessel containing the ammonia as shown in Fig. 10, and all rubber tubing is securely fastened with copper wire, wherever such joints occur. Ready for use the main valve on the ammonia cylinder is opened, and by opening the thumbscrew 1 some ammonia is blown off in order to remove rust and dirt which may have accumulated in the valve; this also will remove the small quantity of oil, which may have been used for lubricating the screw on the valve stem and which is always likely to be present in the interior of the valve.

If sufficient ammonia has been blown off to clear the valve, thumbscrew 1 is shut, and by opening thumbscrew 2 a sample of ammonia is admitted to the test bottle.

Care must be taken that ammonia is not admitted too quickly, otherwise the pressure in the bottle will become too great and either the bottle or the rubber connections are liable to burst. A Bunsen valve permits the ammonia vapor to escape, but prevents moisture of the air from entering the bottle.

The sample is permitted to evaporate while the apparatus remains attached to the cylinder. Evaporation may be hastened by dipping the graduated tail of the test bottle in cold water, whereby a gentle and steady boiling of the ammonia is kept up. Care should be taken not to dip the test bottle too deep into water, otherwise the ammonia may boil too rapidly and the bottle may explode



by excessive pressure. The slower the sample is evaporated the more reliable is the test. One hour and a half is a good time for evaporating 100 cc. ammonia in this apparatus. If evaporation is effected in much less time, the rapidly boiling ammonia will take with it some of the impurities, particularly hydrocarbons, if such are present and an inferior ammonia may show a good test.

During the evaporation, the tail of the test bottle will be heavily coated with ice, which will readily melt off after the sample of ammonia has evaporated by gently agitating the water in the surrounding vessel.

If 100 cc. are taken for the test, and if there remains some liquid in the tail of the test bottle, each subdivision in the scale indicates  $1/10$  of 1 per cent. by volume in the ammonia.

A second and third sample can be evaporated in the same bottle without removing the apparatus from the cylinder and this may be continued until the entire contents of a cylinder has been evaporated, in which case the test bottle will contain all of the impurities which have been contained in the entire cylinder.

Before taking a new test some ammonia is blown off by opening thumbscrew 1 in order to remove as much as possible the traces of oil which may have seeped into the valve from the lubricant used for the valve stem.

If more accurate results are desired the Bunsen valve on the side outlet of the flask is replaced by a bent glass tube leading the ammonia gas into water where it is absorbed. A Bunsen valve attached to the end of the glass tube, which dips into the water, prevents the water from backing up into the test flask. The amount of the ammonia used for the test is then ascertained by titrating the solution of ammonia in water, and the amount of residue left upon evaporation is determined by weighing the test flask before and after the test on a chemical balance. Care must be taken to close

the outlets of the flask, and to have constant temperatures while weighing, also to have the flask entirely filled with ammonia gas in order to exclude errors resulting from the difference in the specific gravities of air and ammonia gas.

Liquefied ammonia gas seems to be an absorbent for permanent gases. They may be determined with the aid of this apparatus by collecting them in a eudiometer filled with water after the ammonia gas has been absorbed in the water of a pneumatic trough. If this be done care should be taken to use

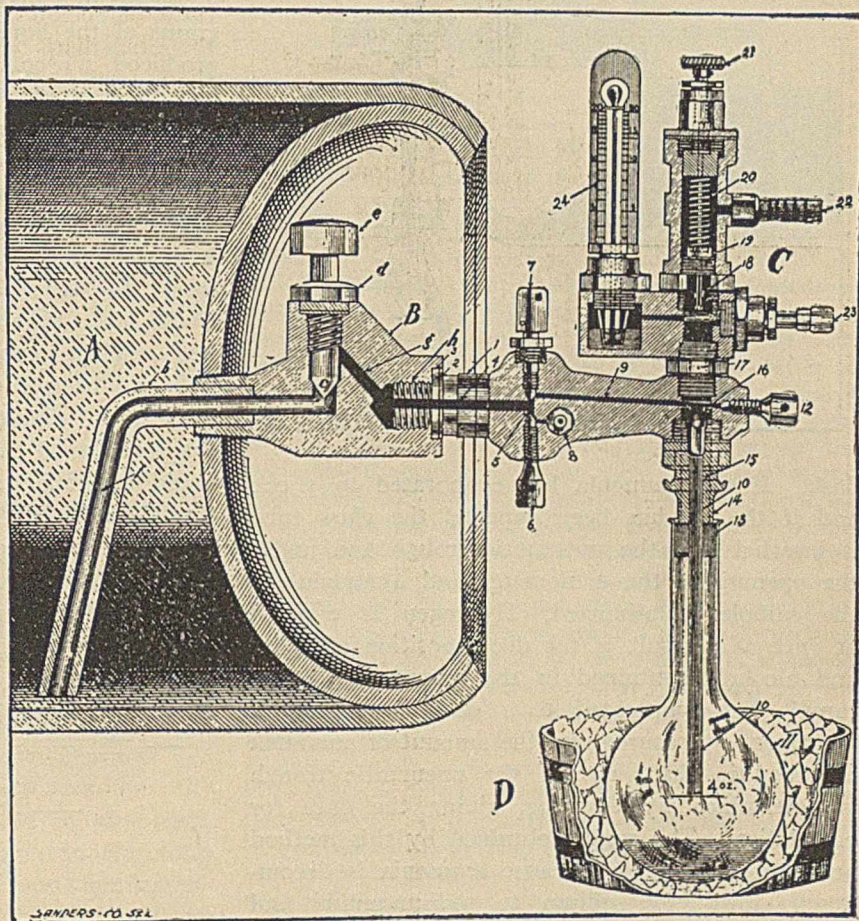


Fig. 9.

water which has been freed from air by protracted boiling since ammonia gas disengages air from water which is saturated with it.

Small quantities of alcohols and water can be determined in liquid ammonia by treating it with metallic sodium and measuring the quantity of hydrogen evolved. The mode of operation is as follows:

Sufficient sodium is inserted into the test flask, which is then connected with the test valve and provided with the extension tube and eudiometer



filled with water and standing in a pneumatic trough. Some liquid ammonia is admitted which dissolves the sodium to a dark blue liquid, the evaporating ammonia expelling the air from the

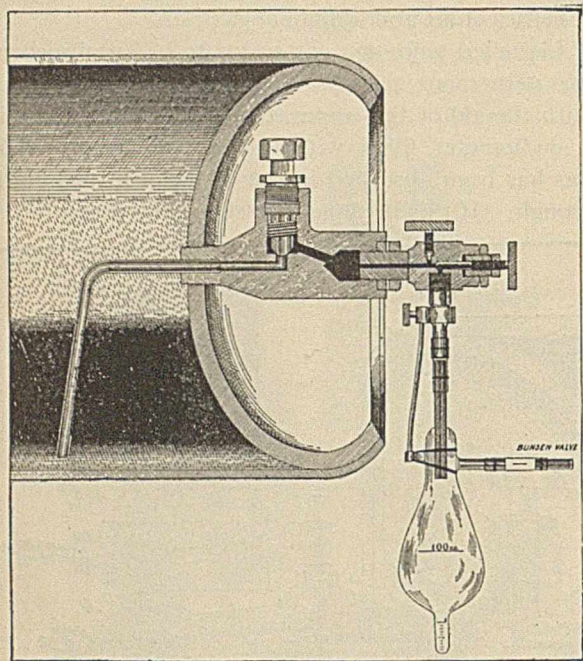


Fig. 10.

flask. If the ammonia has evaporated to 1 cc., and if the air has been expelled the glass tube is inserted into the pneumatic trough and under the opening of the eudiometer and ammonia for the sample is admitted. Hydrogen is evolved at once as a result of the decomposition of water and alcohols contained in the ammonia. If the sample has evaporated to 1 cc. the volume of hydrogen is measured and the amount of ammonia absorbed in the water of the pneumatic trough is determined by titrating, giving the data for calculation. The results obtained by this method are a little too high because ammonia is decomposed slowly by sodium to sodium amide and hydrogen. But it would seem that this secondary reaction is so slow that the result is only slightly affected.

With this apparatus, a series of tests has been made with a view of ascertaining its accuracy in determining the various impurities, which are most frequently contained in commercial liquefied ammonia gas.

As a basis, liquid ammonia was used which had been manufactured in the regular run of the works from sulphate of ammonia previously purified from all volatile carbon compounds, and in

the manufacture of the product great care was taken that all the moisture was eliminated. For this reason it was certain that bodies like benzol, pyridine, alcohols, and other carbon compounds could not be present, and the limit of water was ascertained by analysis to be less than 2 milligrams in 100 grams of liquid ammonia. The determination of water was made by adding an ammoniacal solution of metallic sodium to the liquid ammonia to be analyzed, and collecting the hydrogen, which was evolved by the reaction of the sodium upon the water in the sample. 100 grams of the liquid ammonia treated in this way produced 2.4 cc. hydrogen. If all the hydrogen had been produced from water 2 milligrams of water must have been present, but it is well known that ammonia itself decomposes slowly with sodium into sodium amide and hydrogen and for this reason part of the hydrogen may have come from the ammonia, in which case the water present would be less than 0.002 per cent. As a fact, I will state that about one-half of the hydrogen was developed rapidly right after the sodium solution had been added, while the balance accumulated from minute bubbles during the time of evaporation of the entire hundred grams of the liquid ammonia, and for this reason it is quite possible that the second half comes from decomposition of ammonia. Therefore, under no circumstances could more than 0.002 per cent. water

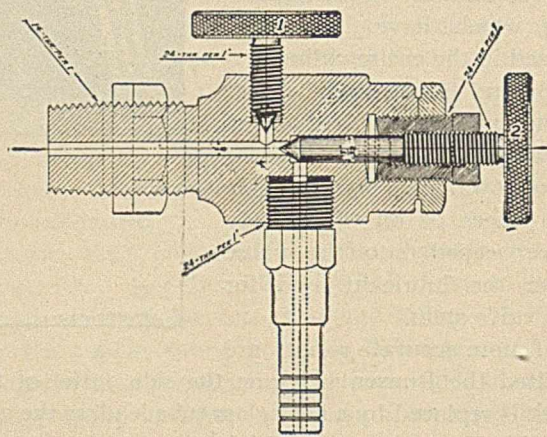


Fig. 11.

be present, and it is possible that the quantity of water in the sample was not more than half of that amount. Table V gives the result of five tests made with this ammonia in the smaller of the two apparatus and from the uniformity of the results and the insignificance of the residue the purity of the liquid ammonia is evident.



TABLE V.

80 grams liquid NH<sub>3</sub> left after evaporation 0.003 gram.  
 160 grams liquid NH<sub>3</sub> left after evaporation 0.003 gram.  
 240 grams liquid NH<sub>3</sub> left after evaporation 0.003 gram.  
 320 grams liquid NH<sub>3</sub> left after evaporation 0.005 gram.  
 400 grams liquid NH<sub>3</sub> left after evaporation 0.007 gram.  
 Residue from evaporation 0.00175 per cent.  
 Ammonia by difference 99.99825 per cent.  
 The residue consisted of iron oxide shown by potassium ferrocyanide, and little lubricating oil.

With ammonia of this purity, the experiments of Lange and Heffter have been repeated. The results I have given in Table III. In addition a series of samples containing variable, but known quantities of benzol, pyridine, alcohol, and water have been evaporated in the new apparatus, and the residues left after evaporation have been ascertained as given in Table VI.

TABLE VI.

Evaporated with liquid NH <sub>3</sub>	Benzol.		Pyridine.		Alcohol.		Water.	
	Gram.	Gram.	Gram.	Gram.	Gram.	Gram.	Gram.	Gram.
	0.527	0.652	0.966	0.676	0.673			
First 100 cc.....	0.258	0.142	0.142	0.099	0.077			
Second 100 cc.....	0.178	0.135	0.131	0.089	0.066			
Third 100 cc.....	0.077	0.084	0.117	0.077	0.061			
With 300 cc. } grams... evaporated } per cent.	0.513 97	0.361 55	0.390 40	0.265 39	0.204 30			

From this table it is evident that the quantities of the additions which evaporate with the ammonia decrease if less of the addition is present.

Table VII shows the same results differently

TABLE VII.

Benzol		Pyridine		Alcohol		Water	
present, per cent. of ammonia.	evap., per cent. of benzol.	present, per cent. of ammonia.	evap., per cent. of pyridine.	present, per cent. of ammonia.	evap., per cent. of alcohol.	present, per cent. of ammonia.	evap., per cent. of water.
1.50	59	1.00	24	1.00	17	1.10	11
0.65	49	0.81	22	0.84	14	0.84	11
0.34	66	0.64	26	0.72	15	0.75	11
0.11	84	0.47	22	0.61	16	0.66	11

arranged. It gives the proportion of the addition evaporated with the ammonia to the total amount of the addition present before the evaporation, and a marked difference is observed between benzol on the one side and pyridine, alcohol, and water on the other. While the percentage evaporated seems to be the same for varying quantities of the addition present in the case of pyridine, alcohol and water, it increases rapidly with a decreasing addition of benzol.

The limit of accuracy of this mode of testing has been ascertained in a special series of experiments as follows:

- For water to 0.002 gram in 100 grams ammonia.
- For alcohol to 0.006 gram in 100 grams ammonia.
- For pyridine to 0.009 gram in 100 grams ammonia.
- For benzol to 0.110 gram in 100 grams ammonia.

In other words, 100 cc. samples of liquid ammonia containing these additions, leave, upon evaporation, a visible residue of at least one milligram.

HERF AND FRERICHS CHEMICAL CO.,  
 ST. LOUIS, MO.



NOTES AND CORRESPONDENCE.

RECENT LEGISLATION RELATIVE TO EXPLOSIVES IN THE MAIL AND TRANSPORTATION.

The act to codify, revise, and amend the penal laws of the United States published as (Public-No. 350) and (S. 2982), approved March 4, 1909 and taking effect January 1, 1910, provides under offenses against the Postal Service.

Sec. 217. All kinds of poison, and all articles and compositions containing poison, and all poisonous animals, insects, and reptiles, and explosives of all kinds, and inflammable materials, and infernal machines, and mechanical, chemical, or other devices or compositions which may ignite or explode, and all disease germs or scabs, and all other natural or artificial articles, compositions, or materials of whatever kind which may kill, or in any wise hurt, harm, or injure another, or damage, deface, or otherwise injure the mails or other property, whether sealed as first-class matter or not, are hereby declared to be nonmailable matter, and shall not be conveyed in the mails or delivered from any post-office or station thereof, nor by any letter carrier; but the Postmaster-General may permit the transmission in the mails, under such rules and regulations as he shall prescribe as to the preparation and packing, of any articles hereinbefore described which are not outwardly or of their own force dangerous or injurious to life, health, or property: Provided, That all spirituous, vinous, malted, fermented, or other intoxicating liquors of any kind, are hereby declared to be nonmailable and shall not be deposited in or carried through the mails. Whoever shall knowingly deposit or cause to be deposited for mailing or delivering, or shall knowingly cause to be delivered by mail according to the direction thereon, or at any place at which it is directed to be delivered by the person to whom it is addressed, anything declared by this section to be nonmailable, unless in accordance with the rules and regulations hereby authorized to be prescribed by the Postmaster-General, shall be fined not more than one thousand dollars, or imprisoned not more than two years, or both; and whoever shall knowingly deposit or cause to be deposited for mailing or delivery, or shall knowingly cause to be delivered by mail according to the direction thereon, or at any place to which it is directed to be delivered by the person to whom it is addressed, anything declared by this section to be nonmailable, whether transmitted in accordance with the rules and regulations authorized to be prescribed by the Postmaster-General or not, with the design, intent, or purpose to kill, or in anywise hurt, harm, or injure another, or damage, deface, or otherwise injure the mails or other property, shall be fined not more than five thousand dollars, or imprisoned not more than ten years or both

It provides under the offenses against Foreign and Interstate Commerce.



Sec. 232. It shall be unlawful to transport, carry, or convey any dynamite, gunpowder, or other explosive, between a place in a foreign country and a place within or subject to the jurisdiction of the United States, or between a place in any State, Territory, or District of the United States, or place noncontiguous to but subject to the jurisdiction thereof, and a place in any other State, Territory, or District of the United States, or place noncontiguous to but subject to the jurisdiction thereof, on any vessel or vehicle of any description operated by a common carrier, which vessel or vehicle is carrying passengers for hire: Provided, That it shall be lawful to transport on any such vessel or vehicle small arms ammunition in any quantity, and such fuses, torpedoes, rockets, or other signal devices, as may be essential to promote safety in operation, and properly packed and marked samples of explosives for laboratory examination, not exceeding a net weight of one-half pound each, and not exceeding twenty samples at one time in a single vessel or vehicle which is intended for the transportation of passengers for hire: Provided further, That nothing in this section shall be construed to prevent the transportation of military or naval forces with their accompanying munitions of war on passenger equipment vessels or vehicles.

Sec. 233. The Interstate Commerce Commission shall formulate regulations for the safe transportation of explosives, which shall be binding upon all common carriers engaged in interstate or foreign commerce which transport explosives by land. Said commission, of its own motion, or upon application made by any interested party, may make changes or modifications in such regulations, made desirable by new information or altered conditions. Such regulations shall be in accord with the best known practicable means for securing safety in transit, covering the packing, marking, loading, handling while in transit, and the precautions necessary to determine whether the material when offered is in proper condition to transport. Such regulations, as well as all changes or modifications thereof, shall take effect ninety days after their formulation and publication by said commission and shall be in effect until reversed, set aside, or modified.

Sec. 234. It shall be unlawful to transport, carry, or convey, liquid nitroglycerin, fulminate in bulk in dry condition, or other like explosive, between a place in a foreign country and a place within or subject to the jurisdiction of the United States, or between a place in one State, Territory, or District of the United States, or place noncontiguous to but subject to the jurisdiction thereof, and a place in any other State, Territory, or District of the United States, or place noncontiguous to but subject to the jurisdiction thereof, on any vessel or vehicle of any description operated by a common carrier in the transportation of passengers or articles of commerce by land or water.

Sec. 235. Every package containing explosives or other dangerous articles when presented to a common carrier for shipment shall have plainly marked on the outside thereof the contents thereof; and it shall be unlawful for any person to deliver, or cause to be delivered, to any common carrier engaged in interstate or foreign commerce by land or water, for interstate or foreign transportation, or to carry upon any vessel or vehicle engaged in interstate or foreign transportation, any explosive, or other dangerous article, under any false or deceptive marking, description, invoice, shipping

order, or other declaration, or without informing the agent of such carrier of the true character thereof, at or before the time such delivery or carriage is made. Whoever shall knowingly violate, or cause to be violated, any provision of this section, or of the three sections last preceding, or any regulation made by the Interstate Commerce Commission in pursuance thereof, shall be fined not more than two thousand dollars, or imprisoned not more than eighteen months, or both.

Sec. 236. When the death or bodily injury of any person is caused by the explosion of any article named in the four sections last preceding, while the same is being placed upon any vessel or vehicle to be transported in violation thereof, or while the same is being so transported, or while the same is being removed from such vessel or vehicle, the person knowingly placing, or aiding or permitting the placing, of such articles upon any such vessel or vehicle, to be so transported, shall be imprisoned not more than ten years.

Section 341, under Repealing Provisions, repeals the act entitled "An Act to promote the safe transportation in interstate commerce of explosives and other dangerous articles, and to provide penalties for its violation," approved May thirtieth, nineteen hundred and eight.

CHARLES E. MUNROE.

#### METHOD OF ANALYSIS FOR MASSECUITES AND MOLASSES.

The analysis of massecuites and molasses is one of the most difficult problems in sugar analysis, for with most methods, even a moderate speed can be attained only with more or less loss in accuracy. In the course of analysis of about two thousand samples by the author, several different methods were tried and found unsatisfactory, and finally the following modification of one of Spencer's methods<sup>1</sup> was devised.

The use of a refractometer for determining the density, or Brix, of the sample is of course preferable to the method described here, but in laboratories where a refractometer is not available, it is hoped this method will be found of use.

Weigh out 125 grams of the sample in an ordinary nickel tare, add a little hot water—20 or 25 cc. if the tare will hold that much without danger of slopping over—and stir thoroughly with a small pestle, made by heating and flattening the end of a heavy glass rod. A molasses will thus be readily thinned down so it can be poured into a flask. A massecuite must be stirred until all lumps are broken up and the molasses adhering to the sugar crystals is all dissolved. The contents of the tare can then be poured into a flask and the tare washed out, using a second portion of hot water if necessary. The flask should be a 500 cc. one, graduated, with as wide a neck as possible. After the contents of the tare are all washed into the flask, fill the flask half full of water and shake till all the contents are dissolved. In a factory laboratory where several samples are run at one time, the samples can all be transferred into the flasks and turned over to the helper to dissolve.

After the sample is completely dissolved, fill the flask to the mark with the coldest water available, and shake thoroughly. Unless a large amount of hot water has been used in dissolving the sample, and transferring it to the flask, the diluted sample will be ready for analysis without further

<sup>1</sup> Spencer's "Handbook for Sugar Manufacturers," p. 147.



cooling. If the temperature of the solution is over 30° C., the flask should be cooled in a stream of cold water from the hydrant, and the solution made up to the mark again. Transfer the solution to a 1 1/4 x 15" glass cylinder and note the Brix with an ordinary Brix spindle, and the temperature with a Centigrade thermometer. The apparent degree

To avoid repeated calculation, in the following table the apparent density or degree Brix of the original sample has been calculated for a sufficient range of temperature and Brix of diluted sample to include all ordinary practice.

By using this table for the Brix, and 52.096 cc. of the diluted sample for the sucrose, the analysis of massecuites

TABLE SHOWING BRIX OF MATERIALS DILUTED 125 GRAMS TO 500 CC.

Note the Brix and temperature of the diluted solution, and read the Brix of the original material at the intersection of the horizontal or vertical columns corresponding to the Brix and temperature as noted.

Brix.	18° C.	19°.	20°.	21°.	22°.	23°.	24°.	25°.	26°.	27°.	28°.	29°.
19.0	82.12	82.38	82.73	83.03	83.33	83.63	83.89	84.19	84.49	84.80	85.10	85.40
19.1	82.59	82.84	83.19	83.49	83.80	84.10	84.36	84.66	84.96	85.26	85.56	85.87
19.2	83.05	83.51	83.66	83.96	84.26	84.56	84.62	85.13	85.43	85.73	86.03	86.34
19.3	83.52	83.78	84.12	84.43	84.73	85.02	85.29	85.59	85.90	86.20	86.50	86.80
19.4	83.99	84.25	84.59	84.89	85.20	85.51	85.76	86.06	86.36	86.67	86.97	87.27
19.5	84.45	84.71	85.06	85.36	85.66	85.97	86.23	86.53	86.83	87.13	87.44	87.74
19.6	84.92	85.18	85.53	85.83	86.13	86.43	86.69	87.00	87.30	87.60	87.91	88.21
19.7	85.39	85.65	85.99	86.30	86.60	86.90	87.16	87.47	87.78	88.07	88.39	88.68
19.8	85.86	86.12	86.46	86.77	87.07	87.37	87.63	87.93	88.24	88.54	88.84	89.15
19.9	86.32	86.58	86.93	87.23	87.54	87.84	88.10	88.40	88.71	89.01	89.31	89.62
20.0	86.79	87.05	87.40	87.70	88.01	88.31	88.57	88.87	89.18	89.48	89.78	90.09
20.1	87.26	87.52	87.87	88.17	88.47	88.78	89.04	89.34	89.64	89.95	90.25	90.56
20.2	87.73	87.99	88.34	88.64	88.94	89.25	89.51	89.81	90.11	90.42	90.72	91.02
20.3	88.20	88.46	88.81	89.10	89.41	89.72	89.98	90.29	90.59	90.89	91.20	91.50
20.4	88.67	88.93	89.28	89.59	89.89	90.19	90.45	90.76	91.06	91.36	91.67	91.97
20.5	89.14	89.40	89.75	90.06	90.36	90.66	90.92	91.23	91.53	91.84	92.14	92.44
20.6	89.62	89.88	90.22	90.53	90.83	91.14	91.40	91.70	92.00	92.31	92.61	92.92
20.7	90.09	90.35	90.70	91.00	91.30	91.61	91.87	92.17	92.48	92.78	93.09	93.39
20.8	90.56	90.82	91.17	91.47	91.78	92.08	92.34	92.65	92.95	93.25	93.56	93.86
20.9	91.03	91.29	91.64	91.94	92.25	92.55	92.81	93.12	93.42	93.72	94.03	94.34
21.0	91.50	91.77	92.11	92.42	92.72	93.03	93.29	93.59	93.90	94.20	94.51	94.81
21.1	91.98	92.24	92.59	92.89	93.29	93.50	93.76	94.07	94.37	94.68	94.98	95.29
21.2	92.45	92.71	93.06	93.37	93.67	93.98	94.24	94.54	94.85	95.15	95.46	95.76
21.3	92.93	93.19	93.53	93.84	94.14	94.45	94.71	95.02	95.32	95.63	95.93	96.24
21.4	93.40	93.66	94.01	94.31	94.62	94.92	95.17	95.49	95.80	96.10	96.41	96.71
21.5	93.87	94.14	94.48	94.79	95.10	95.40	95.66	95.97	96.27	96.58	96.88	97.19
21.6	94.36	94.61	94.96	95.27	95.57	95.88	96.13	96.44	96.75	97.05	97.36	97.66
21.7	94.83	95.09	95.44	95.74	96.05	96.35	96.61	96.92	97.23	97.53	97.84	98.14
21.8	95.30	95.56	95.91	96.22	96.52	96.83	97.09	97.40	97.70	98.01	98.31	98.62
21.9	95.78	96.04	96.39	96.69	97.00	97.31	97.57	97.87	98.18	98.48	98.79	99.10
22.0	96.25	96.52	96.87	97.17	97.48	97.78	98.04	98.35	98.66	98.96	99.27	99.57
22.1	96.73	96.99	97.34	97.65	97.96	98.26	98.52	98.83	99.14	99.44	99.75	...
22.2	97.21	97.47	97.82	98.13	98.43	98.74	99.00	99.31	99.61	...	...	...
22.3	97.69	97.95	98.30	98.61	98.91	99.22	99.48	...	...	...	...	...
22.4	98.16	98.43	98.78	99.08	99.39	99.70	...	...	...	...	...	...
22.5	98.64	98.91	99.26	99.56	...	...	...	...	...	...	...	...
22.6	99.12	99.38	99.74	...	...	...	...	...	...	...	...	...
22.7	99.60	99.86	...	...	...	...	...	...	...	...	...	...

Brix of the original sample can then be obtained from the following formula:

$$\text{Apparent degree Brix} = \frac{S \times B \times V}{W}$$

when B = degree Brix of the diluted sample, corrected for temperature;

S = specific gravity, corresponding to the Brix before correcting for temperature;

V = volume of the solution;

W = weight of material used.

Having used the quantities specified, this formula becomes:

$$\text{Apparent degree Brix} = \frac{S \times B \times 500}{125} = S \times B \times 4.$$

Since this solution contains 0.25 gram of the original sample in each cc., it is only necessary to fill a Spencer pipette to the zero mark, thus measuring out 52.096 cc., to obtain a half normal weight of the original sample for polarization. This is transferred to a 100 cc. flask, lead acetate added to clarify, filled to the mark, filtered and polarized.

and molasses may be made with a very satisfactory degree of speed and accuracy.

G. A. ROUSH.

THE VALUATION OF INSOLUBLE PHOSPHORIC ACID IN COMMERCIAL FERTILIZERS.

It is customary in the majority of states having laws regulating the sale of fertilizers, to place a value on the ingredients that enter into the composition of the fertilizers. These values are expressed as either cost per unit (20 lbs.) or cost per pound, and where the value has been stated as cost per unit, the cost per pound has been calculated and used in the accompanying table. In some states no valuation is made on insoluble phosphoric acid, and in other states a value is placed on insoluble phosphoric acid irrespective of its source; *i. e.*, whether or not it is derived from rock phosphate or from bone.

In the manufacture of mixed fertilizers, the phosphoric acid may be derived from either well-made acid phosphate (low insoluble phosphoric acid), badly made acid phosphate (high insoluble phosphoric acid) or tankage or guano con-



taining bone, or a combination of any of these. In mixtures containing badly made acid phosphate, or tankage or guano containing bone, the insoluble phosphoric acid will be high.

CENTS PER POUND FOR PHOSPHORIC ACID.

	In- soluble from bone.	In- soluble from rock.	In tankage.	Insolu- ble in mixed fertz.	Insoluble in fertz. con- solu- ble.	In tain- ing N.	In bone meal.
Pennsylvania.....	2	1 1/2	3	..	..	..	3
Maryland.....	..	1	3	2	..	..	3
Florida.....	..	..	..	..	1	..	..
Maine, Massachusetts, New Jersey, Rhode Island, Vermont, Con- necticut, New York..	..	..	4	2	..	..	4
Michigan.....	..	..	..	..	..	2	4
Georgia.....	..	..	..	..	..	..	3 3/4
Texas.....	..	..	4	..	..	..	4
Louisiana.....	..	..	1.09	..	..	..	4

It will be seen by glancing at the accompanying table, that in those states in which a value is placed on insoluble phosphoric acid in mixed fertilizers, this value is invariably lower than the value placed on bone in tankages; that is, those manufactures using tankage containing bone in their mixed fertilizers do not obtain a correct valuation, but are placed in the same class with those manufacturers that use badly made acid phosphate. The present method of determining insoluble phosphoric acid does not differentiate between that derived from phosphate rock and that derived from bone. The following method has been worked out and found satisfactory for determining approximately the amount of insoluble phosphoric acid, from each source, present in a mixed fertilizer containing both.

This method depends upon the microscopical examination of the fertilizer, and therefore its accuracy is limited to that usually obtained in a quantitative determination by means of the microscope. The samples are mounted in water in the usual manner by rubbing down between the cover glass and slide. The samples are ground to pass a 40-mesh sieve.

On viewing a sample of bone tankage, by means of a microscope magnifying 150 diameters, the organic fibrous matter is seen to be interspersed with particles of bone. The small pieces of bone are translucent, with a pearly luster. The larger pieces of bone only show this luster on the edges.

The examination of acid phosphate shows it as a uniform, non-crystalline mass, not translucent.

In examining a mixed fertilizer, the method that has been found most convenient is as follows: The sample is mixed, quartered down to about 4 oz., ground to pass a 40-mesh sieve and placed in a stoppered bottle. For a preliminary examination, a small portion is mounted in water and examined under a microscope. The presence or absence of bone is immediately shown. If bone is found to be present the fertilizer may be further examined to determine the proportions of insoluble phosphoric acid from bone and acid phosphate as follows: Two grams of the ground fertilizer are treated according to the official method of the A. O. A. C. for the determination of insoluble phosphoric acid in mixed fertilizers. However, after washing with 250 cc. of distilled water, instead of placing the filter paper with the fertilizer into 100 cc. of ammonium citrate

solution, scrape the washed fertilizer from the paper and proceed as usual, thus eliminating the paper pulp and facilitating the following examination: In filtering the insoluble matter, use a filter paper, the apex of which is supported by either a platinum or parchment cone. After filtering and washing, scrape the insoluble residue from the filter paper, mix, and examine under a microscope. The insoluble phosphate rock can readily be distinguished from the accompanying bone and organic matter. By comparing it with standard mixtures, a fair approximation of the proportion of the insoluble phosphoric acid derived from the acid phosphate may be obtained. By determining the total insoluble in the usual manner the insoluble phosphoric acid from bone may be obtained.

Using the above method, a more nearly accurate valuation of mixed fertilizers can be obtained. It has been my experience that mixed fertilizers made from tankage and well-made acid phosphate will rarely run over 0.5 per cent. of insoluble phosphate rock, and will, in the majority of cases, run below this figure. If, on finally examining a fertilizer, only a small quantity of insoluble phosphate rock is present, the chemist would come very close to the truth if he used the value 0.5 per cent. for insoluble phosphate rock. When it is considered that some fertilizers containing tankage run as high as 4 per cent. insoluble phosphoric acid, the fact can readily be seen that the correct valuation of the insoluble phosphoric acid in a mixed fertilizer is of great importance to the manufacturer using tankage.

R. H. FASH.

SWIFT & COMPANY,  
CHEMICAL LABORATORY,  
FT WORTH, TEXAS.

#### A SYSTEM OF RECORDS FOR THE SCIENTIFIC WORK OF A MANUFACTURING PHARMACEUTICAL ESTABLISHMENT.

The system of reports and records here described is the outgrowth of several attempts to keep complete and convenient records of scientific work. As it became necessary to abandon these systems on account of the growth and branching out of the work it was determined to adopt, if possible, a system which would care for the work of a growing department indefinitely. This requirement seems to be met by the present system, which by modifying certain details, could be adapted to the needs of many laboratories engaged in other lines of work. In order to understand some of the difficulties encountered, and also some of the details of the system under consideration, it will be necessary to present the organization of the scientific work for which the system was designed. This is shown in the following diagram:

Subdivision of Chemistry and Biology.

I. Department of Chemistry.

a. Routine.

(1) Examination of crude materials which can be controlled chemically.

(2) Assaying drugs and finished products, including chemical control of manufacturing processes.

b. Research.

II. Department of Botany—Inspection of crude vegetable drugs and botanical research.



III. Department of Pharmacology and Bacteriology—  
Testing of crude materials and finished products  
on animals and bacteria.

Pharmacologic research.

IV. Library and Scientific Records.

V. Care of Scientific Apparatus and Materials.

Technical supervision of manufacturing and pharmaceutical development form two separate subdivisions and are not provided for in this system of records; however, the subdivision of chemistry and biology must by means of reports keep in close touch with these, as well as with the subdivision of manufacturing (including the stock department), and with the purchasing department. All crude materials, from 1500 to 2000 in number, must be examined and, if approved, reported to the stock department by means of a serially numbered slip to be attached to each package; if rejected, a different form of slip is sent to the stock department, and a report must go to the manufacturing office and to the purchasing department. Many samples of materials are received by the purchasing department which must be examined and reported upon.

Some crude materials and samples are examined by two or more departments, for instance many crude vegetable drugs are examined by the botanical department and assayed by the routine chemical department, yet to avoid confusion only one report must go to the purchasing department. In addition to the work on crude materials many finished preparations are assayed and standardized, and manufacturing processes are controlled by chemical tests. Various lines of research are also carried on.

Complete and permanent records of all of this work must be kept in such a manner as to be readily accessible, at all times, to the director of the scientific work. Each worker should also have a record of his own work for convenient reference.

To meet these requirements, with a minimum amount of clerical work, this system was devised. Nine permanent loose-leaf records are provided:

1. Crude Vegetable Drugs.
2. Other Crude Materials.
3. Chemical Research.
4. Chemical Routine.
5. Botany.
6. Pharmacology and Bacteriology.
7. Manufacturing Losses.
8. Standard Methods of Analysis.
9. Index to Scientific Literature.

Records 1 and 2 are simply lists of lots and samples of crude materials received, each of which is given a serial number by an automatic numbering machine, and has recorded its source, disposition, and the name of the examiner. A page (Fig. 1) is devoted to each crude material, the pages are arranged alphabetically and at the top of each page the requirements for the material in question and references to the "Standard Methods of Analysis" are written. These lists make it easy to determine, the customary source for any crude material, the source of each individual lot, the number of lots received in any specified time, the person responsible for each lot, and whether or not samples have been examined to which subsequent lots from the same source should conform. The entries in these records are made by the person who examines the crude material.

Each worker is supplied with a spring back binder which will hold from one to seventy-five loose leaf sheets (Fig. 2) 4½ x 8 inches in size, one of which is properly headed and inserted in the binder for each subject assigned for work. On the back of this sheet a record of weighings, outline of calculations, and other details are placed, and when the final results are obtained all matter essential to the permanent records is written on the front of the blank; several modifica-

Article _____					
Requirements _____					
	Date 19--	Number	Source	Remarks	Ex. by

Fig. 1.

tions of this report blank, of uniform size, are in use for special lines of work. The reports when finished are taken out of the binder, so that the remaining sheets serve as memoranda of work to be completed. The finished reports are submitted to the head of the department in which the work was done and are then transmitted with the proper notations to the librarian, who enters in the permanent records the data appearing on the front of the report. This

Sheet No. \_\_\_\_\_

Reported by \_\_\_\_\_ Date \_\_\_\_\_

Sample  
Lot

Factory or Laboratory No.	Source and Data

Fig. 2.

entry is made by means of a special typewriter with 96 characters which make it possible to write chemical formulas and other frequently used chemical signs. In the permanent records a page (Fig. 3) is assigned to each subject, and when filled another is inserted so that no matter how voluminous the records may become, all information upon one subject remains together and its alphabetical arrangement is never disturbed. Modifications of this record blank



with headings printed in to facilitate the tabulation of retain data are also in use.

After this entry is made and verified the original report is returned to the author, who files it in alphabetical order in a binder provided for this purpose; this file constitutes his individual record and in it all reports are kept for a reasonable length of time, those over one year old being taken out and stored in boxes.

The blank ends of these sheets serve to carry memoranda to the librarian directing the sending of reports to any other divisions or departments of the business, and these reports are made out at the time that the entry in the permanent record is made. Materials examined by two or more departments are reported upon to the department chiefly concerned in their examination; all reports are then embodied in the report from this department.

Taking the data from the reports on assayed drugs and the standardized preparations made from them, the head of the routine chemical department keeps an interesting record (No. 7) showing the manufacturing losses. A record of each preparation is kept on a sheet of squared paper, the abscissae representing the per cent. of active principles in the drug, and the ordinates the ratio of the yield of finished preparation to the amount of crude material used.

Sheet No. \_\_\_\_\_

Date	

Fig. 3.

A line is drawn showing the yield of finished preparation which would be obtained from crude materials of the various strengths if there was no loss in manufacturing. Each lot of the preparation manufactured is represented by a point which falls somewhat below this line, and after a sufficient number of points are fixed, a line representing the average losses in manufacturing can be drawn. Any carelessness in manufacturing or any improvement in process is then readily shown.

Confusion in the records and in the laboratories is avoided by giving to everything which is made the subject of laboratory investigation a distinctive number, which is used on all reports and records. Several series of numbers are in use, but the different lines of work are so well defined that no confusion results.

Aside from records 1, 2, and 7, and the numbered slips for the stock department, it is only necessary for the laboratory worker to make one report on each subject, which serves as a laboratory memorandum, and from which all necessary reports and records are made without further attention on his part. All reports sent to the purchasing department in regard to crude materials are filed there and form a convenient permanent record for that department.

Records 8 and 9 are kept on the regular record blank (Fig. 2). Record 8 gives methods devised in our laboratories or selected from other sources. The index of scientific literature is kept by the librarian and includes only those articles which have a direct bearing on our lines of work. All journals in the library are represented in the index, the articles being marked, and cross references indicated by members on the scientific staff. Articles treating on the same subject are indexed on the same sheet under a suitable heading and copious cross references are made so that the information indexed is easily found. In all the records cross references are inserted wherever they seem to be of value.

FRANK R. ELDRED.

DEPARTMENT OF CHEMISTRY,  
ELI LILLY & Co.

### THE EFFECT OF THE PROPOSED TARIFF BILL ON AMERICAN CHEMICALS.

It is interesting to study the tariff bill as proposed by the Ways and Means Committee in conjunction with the hearings and briefs submitted by interested manufacturers of chemical with a view to determining to what extent the mind of the legislator is influenced by the manufacturer's arguments.

Again, it is of interest in comparing the proposed tariff bill as submitted by the Finance Committee of the Senate with that of the House to observe the diversity of opinion as to what will best serve this country's good, for such, of course, is the purpose of the act which reads, "to provide revenue, equalize duties, and encourage the industries of the U. S., and for other purposes," and one is inclined to ask what motive or reason can be assigned for such contradictory action by the two bodies of legislation.

One is satisfied, after a perusal of the bills and hearings and observing the hoards of manufacturers, importers and others crowding the hotels of Washington and hounding the congressmen for favor, that Hancock was right when he said that the tariff is a "local question": each for his own interest.

The methods pursued by those interested in holding the present duty or increasing the duty on their manufactured product or getting their raw materials put on the free list, vary greatly. Some appeared in person before the Ways and Means Committee; others preferred to send in briefs, thus avoiding the cross-questioning of the committee; some disregarded entirely the Ways and Means Committee and worked with the Senate Finance Committee. If one had in his favor the captious phrase "for the benefit of the farmer," he had all the Representatives with him.

As tariff revision was a Republican issue at the last election, which no doubt had an important bearing upon Republican success, Congress, which is strictly Republican, is duty-bound to revise the tariff, down and not up. On the other hand, it must have in view the receiving of adequate revenue for governmental expenses. Should such revision downward be made in a manner to satisfy a large number of our people and fulfil the promise made by the Republican party, the cut in revenue would be such as would necessitate the enactment of income or inheritance taxes, which are offensive to many people. Further, if duties on certain articles are lowered too far there is danger of crippling industries which are now prosperous and em-



ploying American labor, which is always paid fifty, and sometimes a hundred per cent., more than foreign labor. Therefore, to give worthy industries necessary protection, and no more, to supply by duty ample revenue for government purposes and at the same time revise (downward) the tariff to fulfil the Republican promise, is not an easy matter.

As the chemical schedule was the first considered by the Ways and Means Committee, considerable notoriety was won in the daily press by those asking for duties on chemicals, to be made for the first time in the United States, and by those who had the temerity to petition for an increase in duty on chemicals now made in this country.

The United States must acknowledge Germany's superiority in chemical products, particularly the finer chemicals. The German government has fostered its chemical industry more than any of its other industries. She realizes more than any other nation that chemistry is the foundation of most of its industries and consequently aids it in all possible ways. By not giving proper protection to infant chemical industries, the United States government not only discourages the development of this particular industry, but to a certain degree retards the progress of many other industries which are founded on chemical reactions, or are users to a greater or less degree of chemical products.

In considering duties on chemicals our legislators should have this in mind, first, that those chemicals should be admitted free of duty which are made from minerals or other natural products which do not exist in the U. S. in exploitable amount, *e. g.*, nitrate of soda and potash. Second, that as soon as a chemical manufacturer gives assurance that he intends to enter into the manufacture of a certain chemical now on the free list, a tariff should be placed on that article sufficient to offset the extra cost of the chemical due to higher cost of labor in this country. This sort of protection is what is needed to develop the chemical industry. It does not mean necessarily an increase in the cost of that particular article to the consumer, but it does prevent the foreign trade combination from dumping its surplus goods in this market at a ridiculously low price for a time to kill the American competition.

There is evidence in the proposed tariff bill that this principle has at least occurred to our legislators, for oxalic acid under the Dingley tariff is on the free list; the importations for 1908 amounted to 8,800,000 lbs. In the last few years American manufacturers have succeeded in producing this chemical and should have some protection. The present bill takes oxalic acid from the free list and gives it one cent a pound duty. On the other hand, our Solons have inconsistently denied the petition made for a duty, equivalent to difference in labor cost, of those who with abundant raw material, benzol, stand ready to manufacture aniline oil and salts and free the American consumer of the foreign trade combination on those articles.

Phosphoric, benzoic, phthalic and carbolic acids, whose imports amount to over half a million dollars in value, all on the free list under the present and the proposed bill, should bear duty if satisfactory assurance is made that American manufacturers can produce them in considerable quantities. Acetic acid remains the same as in the old bill, while boracic acid is reduced from 5c. to 2c. per lb.; lactic

acid is cut from 3c. to 2c.; gallic acid from 10c. to 8c.; tartaric acid from 7c. to 6c. In many instances the Senate bill increases the duty imposed by the House; this is noticeably so in the case of artificial sulphate of lime, bichromate of potash, or soda, orange mineral and red lead. The House attempted to make a reduction in the duty on lead acetate, but the Senate would not assent, retaining the old schedules. The Senate is hardly consistent in totally removing the 25 per cent. *ad valorem* duty which sodium cyanide carried under the Dingley bill and leaving a 12.5 per cent. duty on potassium cyanide. The House bill calls for a 12.5 per cent. duty on both. The retention of \$6.00 a ton on sulphur in both bills seems unnecessary in view of the fact that the Standard Oil Co. is producing it at such a low figure that it has completely shut out the Silician product. The potash salts are naturally still held on the free list. Through the efforts of the fertilizer manufacturers crying "free nitrogen for the farmer," ammonium sulphate was placed on the free list in the House bill, reduced from a duty of three-tenths cent per pound, but it was put back to two-tenths by the Senate. Of the ammonium sulphate consumed in this country, 90 per cent. goes into fertilizers. The fertilizer contains on an average 1 per cent.  $\text{NH}_3$ ; if the duty of \$6.00 a ton on sulphate is removed, it means a possible reduction of 24 cents on a ton of fertilizer which costs the farmer \$35. Will the fertilizer manufacturer or the farmer get this twenty-four cents?

Bicarbonate of soda is reduced one-eighth of a cent per lb. and soda ash one-eighth.

The House was inclined to encourage the aniline color manufacturers of this country by giving them 35 per cent. instead of a 30 per cent. duty, as in the Dingley tariff. This would not more than offset the extra labor cost and would be a just recognition of the splendid work that Schoellkopf and Heller & Merz Co. have done in starting this industry in this country. But the Senate cut it back to 30 per cent. in spite of statements from consumers of the colors favoring the increase. The House also consented to remove from the duty list certain products which are the color makers' raw materials, namely, sulpho acids, paranitraniline, dimethylaniline and others, but again the Senate said no.

What the final bill in its amended form will be one cannot predict. It has been intimated that the President may have something to say about it later.

#### ECONOMY IN THE USE OF OXYGEN FOR COMBUSTION CARBONS.

In the steel works laboratory of to-day the consumption of oxygen gives rise to a considerable item on the laboratory invoice. An idea suggested by Mr. A. G. McKenna, of our company, has led to the development of a system by which the volume of oxygen formerly used to make a single carbon determination is now utilized for from six to eight combustions.

The method is shown in the accompanying illustration and consists simply of two eight-liter bottles on a balancing support, which when in one position causes the gas to flow by hydraulic pressure from one bottle through the system into the other; and, when in the other position, gives rise to an exchange in the opposite direction. This reverse of flow is sent through the combustion tube in the right direction by means of a small switchboard arrangement of four

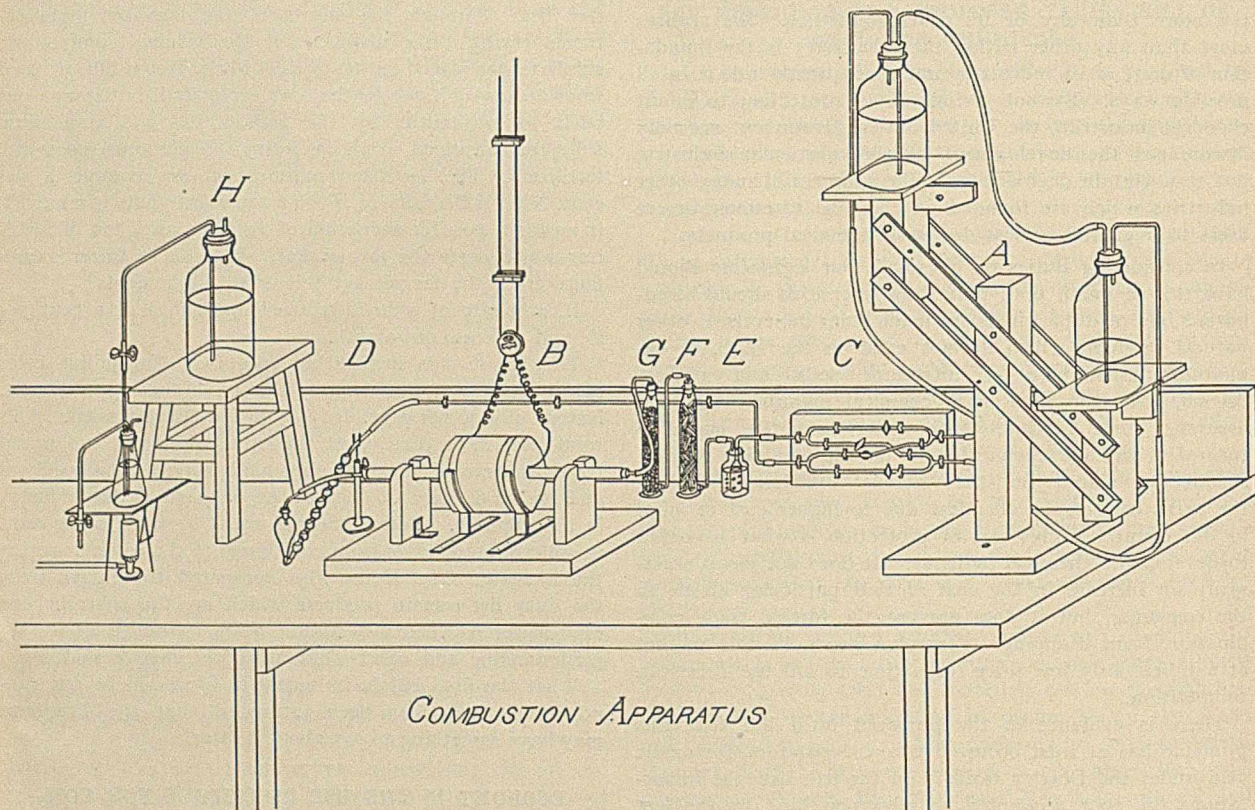


glass stop-cocks so connected that they are operated in pairs and thus leave room for no confusion to the manipulator whenever he finds it necessary to reverse the current. From the bottles the gas passes through a purifying chain consisting of potash solution (1.27 sp. gr.), stick potash, and calcium chloride, and thence through the furnace, which is an ordinary tube of Imperial Berlin porcelain, wound with platinum foil and jacketed with alternate layers of magnesia pipe covering and intervals of air and heated by an electric current of such a strength as to give a constant temperature throughout the day of  $1100^{\circ}\text{C}$ . We have found this temperature to give the most satisfactory results in the direct burning of alloy steels. A small amount of coarse oxide of copper is kept in the tube just beyond the heated area to prevent the possible exit of any carbon monoxide from the tube.

The former method has been used very successfully by the author and easily gives checks within 0.004 per cent. of carbon, but as it is usually necessary to employ untrained labor in steel works laboratories, the latter method has been found to be more reliable in such hands in the long run.

The method in detail:

Weigh two grams of drillings (one gram in the case of furnace tests) into an ordinary platinum boat or filter plug, the former of which should be previously filled with quartz sand or ignited alumina, and the latter covered on the inside with a thin layer of freshly ignited asbestos. Fill the bulb tube with about 100 cc. of the warm barium hydrate solution and insert it in its proper place in the system. It is a good plan to syphon the solution from a large storage bottle into a 500 cc. Erlenmeyer flask, where it may be kept



COMBUSTION APPARATUS

The rest of the train may consist either of the usual purifying tubes and potash bulb as recommended by Blair, or simply of a Meyer bulb tube filled with a warm solution of barium hydrate (15 grams per liter). The barium carbonate which precipitates is filtered off, ignited and weighed as  $\text{BaCO}_3$  containing 6.2 per cent. of carbon. This latter method gives consistent results which may easily be depended on for accuracy within two hundredths of one per cent. It is in common use in the region about Pittsburg and is sufficiently accurate for commercial purposes. It is possible, moreover, by this method to make quick tests for the melters in less than fifteen minutes, which is quite as rapid as, and far more satisfactory in the case of alloy steels than, the colorimetric method which is generally employed.

heated by an Argand burner turned low, and drawn off by a siphon into the bulb tube as needed. Push the platinum plug, or boat, into the hot part of the tube with a nickel rod and quickly put the stopper into the front end of the tube and turn on the two governing stop-cocks, allowing the gas to run at full speed until the white precipitate has just begun to form. Then leaving the return cock on full, turn the other one until the gas bubbles through at the rate of about two bubbles per second and continue the burning for eight minutes—in the case of furnace tests four minutes will suffice, especially if they are low in carbon, as is usually the case. Filter, using gentle suction, through a 11 cm. ashless filter supported by a small platinum cone, and wash with hot water. It is very convenient to have a four-liter flask kept full of hot water and elevated above the desk



near the filtering flask to allow it to be syphoned directly on to the filter, the flow of the stream being controlled by a small pinch-cock. If desired, the first filtrate may be reserved and later used in making up a fresh barium hydrate solution. When thoroughly washed, ignite in a crucible, and when cool brush into a counterpoised watch glass and weigh. This weight multiplied by three—less a correction of 0.01 per cent. found by a blank determination—gives the carbon in the sample. The factor in the case of furnace tests where only one gram is used is six.

In the sketch:

- A. The tilting arrangement.
- B. Electric furnace.
- C. Keyboard for adjusting current of gas.
- D. Meyer bulb tube.
- E. Allihn gas washing bottle containing potash solution.
- F. Calcium chloride cylinder containing stick potash.
- G. Calcium chloride cylinder containing  $\text{CaCl}_2$ .
- H. Barium hydrate solution.

WM. HERBERT KEEN.

LABORATORY OF  
THE FIRTH-STERLING STEEL CO.,  
WASHINGTON, D. C.

#### A SIMPLE COVER FOR ANALYTICAL WEIGHTS.

Probably every analyst is familiar with the annoyance occasioned by floating particles of dust and lint settling on the weights in a balance. The practice of always returning weights to the box has several disadvantages, and is usually considered unsatisfactory. An arrangement devised by the author to obviate this difficulty is in daily use in this laboratory, and may be of service elsewhere.

The base *B* has marked on it the shape, size, place and denomination of each weight. The exact size and shape of the base must, of course, be governed by the available

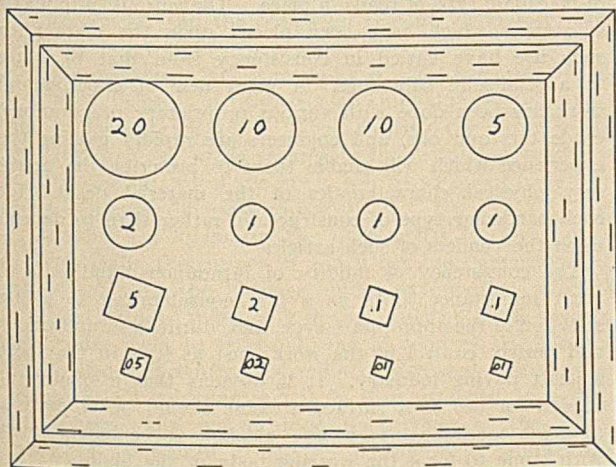


Fig. 1 (B).

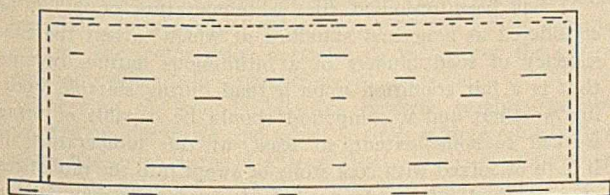


Fig. 2 (C).

space in the balance case, it being kept ordinarily in the space immediately in front of the pillar. The cover *C*, having a glass top with pasteboard sides high enough to clear the tallest weight, is somewhat smaller than the base, and rests on it when the balance is not in use, but is removed whenever the weights are needed. The use of glass in the cover makes it possible to place it over the weights without displacing any of them. This form of base greatly facilitates the reading of the weights, while the cover excludes dust at all times save when using them. As they are left at all times in the balance case, it is only necessary to remove the cover in order to have them in readiness for immediate use. The device may be obtained from Eimer & Amend.

L. T. BOWSER.

OHIO AGRICULTURAL EXPERIMENT STATION,  
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WOOSTER, OHIO.

#### THE "SIZE-WEIGHT" PERCENTAGE.

APRIL 8, 1909.

MR. HARRISON EVERETT ASHLEY,  
NEWELL, WEST VA.

Dear Sir:—I have your letter of March 29th regarding the "size-weight" per cent. mentioned in my article "Accuracy in Sampling Coal." On page 164 of the *JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY*, it says: "The size-weight percentage . . . . . is the percentage of the largest pieces of slate to the total weight of the sample previous to its last division or quartering." If the largest pieces of slate weighed one pound and the sample to be divided weighed one hundred pounds, the "size-weight" per cent. would be one. If the sample had been crushed so that the largest pieces weighed only 0.1 lb. each the "size-weight" per cent. would be 0.1 per cent.

Unfortunately, in the reproduction of Fig. 2 the values of the "size-weight per cent. were omitted. The 4th, 8th, 12th and 16th heavy lines should be numbered 1, 2, 3, and 4, respectively.

Trusting this explanation clears up the matter, I am,

Yours very truly,

E. G. BAILEY.

#### DEFINITION OF "GASOLINE."

NEW YORK, April 13, 1909.

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

DEAR SIR:—On page 261 of the *Journal* for April, 1909, under "Notes on Turpentine," reference was made to "very light naphtha" and "exceedingly light petroleum products" as "not intended to include ordinary gasoline, but merely such unusual products as hexane, petroleum ether, etc."

Would it not be well to have the term "gasoline" or gasolene defined?

1. As to composition, whether composed of pentane, hexane, heptane, etc., and which hydrocarbon predominates.

2. Boiling-point limitations.

3. Whether or not, it is a part of the well-known "petroleum ether," or an entirely separate portion of "naphtha" distillate; and, the relations between it and the hydrocarbon fluid known as "benzine," whether inclusive or not.



Commercially, two or three grades of gasoline are known and the lightest might refer to petroleum ether. Some speak of the "benzines" as coming after the "gasolines."

According to Richter's "Organic Chemistry," the fractional distillation of American petroleum yields various products:

Petroleum Ether—B. p. 50°–60° C.; sp. gr. 0.665–0.67 (Pentane and Hexane).

Petroleum Benzine—B. p. 70°–90° C.; sp. gr. 0.68–0.72 (Hexane and Heptane).

Ligroin—B. p. 90°–120° C.; sp. gr. not given (Heptane and Octane).

Burning Petroleum—B. p. 150°–300° C.; sp. gr. 0.78–0.82 (Kerosene).

Where would "gasoline" come in under such a classification?

In Merck's 1907 Index, gasoline seems to have been omitted. Instead, "Benzine" is described as "Petroleum Ether; Naphtha; Petroleum Naphtha."

According to Thorp's "Outlines of Industrial Chemistry," 1899, p. 292, "The benzine distillate yields:

Cymogene, b. p. = 32° F.; sp. gr. = 0.590–0.610	} Petroleum ether
Rhigoline, b. p. = 60° F.; sp. gr. = 0.625–0.631	
Gasoline, b. p. = 115° F.; sp. gr. = 0.635–0.666	
C Naphtha (Benzine), b. p. 122°–140° F.; sp. gr. 0.678–0.700, etc., etc."	

Judging from this, gasoline would be one of the ingredients of petroleum ether, and "Benzine" a separate commodity.

Gill, in his "Oil Analysis," under Petroleum Products, mentions the "Naphtha Distillate" which is fractionated into four parts. The first, or "Crude Gasoline," is redistilled, giving:

Cymogene, b. p. 32° F. Largely butane for ice machines.  
Rhigoline, b. p. 65° F. Largely pentane for local anaesthesia.

Petroleum Ether, "Sherwood Oil," "Gasoline," b. p. 100°–150° F. Largely hexane for gas machines and fat extractions.

Gasoline, Canadol, b. p. 150°–190° F. For large oil extraction.

In this case, petroleum ether is "gasoline" and does not include cymogene nor rhigolene, as in Thorp's classification. Again, canadol is apparently gasolene but not petroleum ether, although, according to Merck's 1907 Index, canadol is "Very Light Petroleum Ether."

Stillman's "Engineering Chemistry," 1897, p. 364, gives the following:

1. Naphtha group comprises:

Cymogene, a gas, boiling-point 0° C., specific gravity 110° B.  
Rhigolene, liquid, boiling-point 18.3° C., specific gravity 100° B.

Petroleum ether, boiling-point 40° to 70° C., specific gravity 85° to 80° B.

Gasolene, boiling-point 70° to 90° C., specific gravity 80° to 75° B.

Naphtha (Danforth oil), boiling-point 80° to 110° C., specific gravity 76° to 70° B.

Ligroine, boiling-point 80° to 120° C., specific gravity 67° to 62° B.

Benzene, boiling-point 120° to 150° C., specific gravity 62° to 57° B.

Notice the overlapping of the boiling-points for gasolene, naphtha, and ligroine; also, that gasolene has apparently the

same boiling-point (70° to 90° C.) given for "Petroleum Benzine" in Richter's "Organic Chemistry."

It might be interesting to know the distinction between petroleum ether and the gasoline used for automobiles, and whether the latter is a variable mixture of hydrocarbons.

Very respectfully yours,

FREDERIC S. HYDE.

#### CONTROLLING THE CONSISTENCY OF BITUMINOUS BINDERS.<sup>1</sup>

In the asphalt paving industry it was early recognized that some means of measuring the consistency of the asphaltic cement in use was absolutely essential if uniform results were to be achieved, and H. C. Bowen invented a penetration machine in 1888 for this purpose. The asphaltic cement generally employed in sheet asphalt pavements is semi-solid, and must be heated to about 300° F. or more before it can be satisfactorily incorporated with hot sand in a power mixer. A penetration machine is now invariably employed in connection with all such work of importance.

This instrument consists of a weighted needle and a graduated scale upon which the movement of the former, in millimeters, can be measured. The standard conditions for the test are a No. 2 cambric needle under a 100-gram load, which is allowed to penetrate the sample of bitumen under examination for five seconds at 77° F. The New York Testing Laboratory penetrometer and Dow instrument are the two in general use, and operate upon the same principle (see *Proceedings Am. Soc. Testing Materials*, Vol. 7, p. 626).

In the present movement for good roads that will withstand motor-car traffic, a new and urgent demand has arisen for a form of bituminous binder to hold the road metal of macadam type of roads in place. The various bituminous substances which have been placed upon the market for such use have varied in consistency from that of water to a semi-solid substance. A great deal of experimental work has been done with various proprietary preparations, crude tars and oils, and engineers are rapidly gaining the experience which will enable them to prescribe the necessary physical characteristics of the material desired for each particular type of construction, rather than to depend upon the vendors of such articles.

The consistency or fluidity of bituminous binders is of great importance, both as a first consideration to insure selection of the appropriate type, and, during use, uniformity and perfect control of the work, just as it is in the sheet asphalt paving industry. It is obvious that a binder intended for use as a surface dressing should be of quite a different consistency from one which is to be incorporated with stone to form the wearing body of the highway. No single preparation will answer all requirements.

It has been proposed that a temperature of 90° F. be considered as a normal standard at which to test the consistency of road binders of a bituminous nature, because that is a fair condition upon a road during suitable working weather, and a compound should be capable of being worked to some extent, at least, at this temperature, if it is to be mixed with cool stone or swept into the interstices of a roadbed. A much more fluid material is required for

<sup>1</sup> Reprinted from the *Engineering Record*, May 1, 1909.



such use than in the older forms of bituminous pavements, the mixture for which is taken from a central plant in a hot condition, and the standard forms of penetrometers mentioned above are not available for regulating the consistency of the same.

TESTS OF THE CONSISTENCY OF BITUMINOUS BINDERS FOR HIGHWAYS.

Material.	N. Y. T. L. viscosimeter at 90° F.		Engler viscosimeter —100 cc. flow at—	
	Min. Sec.	77° F. Min. Sec.	130° F. Min. Sec.	250° F. Min. Sec.
Refined water gas tar.	0 20	Too stiff	9 46	0 50
Crude coal-tar.....	0 30	Too stiff	9 38	0 50
Tarvia.....	1 30	Too stiff	Too stiff	1 2
Tarina.....	0 16	17 10	3 18	0 34
Texas flux oil.....	0 51	Too stiff	.. ..	1 32
Calif. flux oil.....	3 40	Too stiff	Too stiff	4 14
Headley oil, No. 2....	0 35	Too stiff	Too stiff	1 30
Headley oil, No. 4....	2 53	Too stiff	Too stiff	1 30
Headley oil, No. 4....	6 56	Too stiff	Too stiff	8 42
Genasco compound...	10 54	Too stiff	Too stiff	8 30
Genasco compound... *69	55	Too stiff	Too stiff	Too stiff
Genasco compound...	11 54	Too stiff	Too stiff	9 28
Standard Oil Co.....	0 6	5 38	1 36	0 32
Standard Oil Co.....	0 12	Too stiff	3 0	0 38
Standard Oil Co.....	0 23	Too stiff	Too stiff	0 56
Standard Oil Co.....	0 39	Too stiff	Too stiff	1 18
Standard Oil Co.....	1 24	Too stiff	Too stiff	2 26

The Engler viscosimeter is in general use for testing fluid compounds, such as oils, at any desired temperature, but is not available for the highly cementitious and semi-fluid asphaltic materials now preferred by road engineers, except at temperatures above 200° F.

To provide an instrument for controlling the consistency of semi-fluid compounds intended for road building the author has elaborated a simple form of viscosimeter, originally suggested by Mr. E. C. Wallace, now with the Warren Brothers Company, which fills the gap for substances between those which are sufficiently fluid for the Engler type of viscosimeter and the semi-solid cements heretofore regulated by penetration instruments.

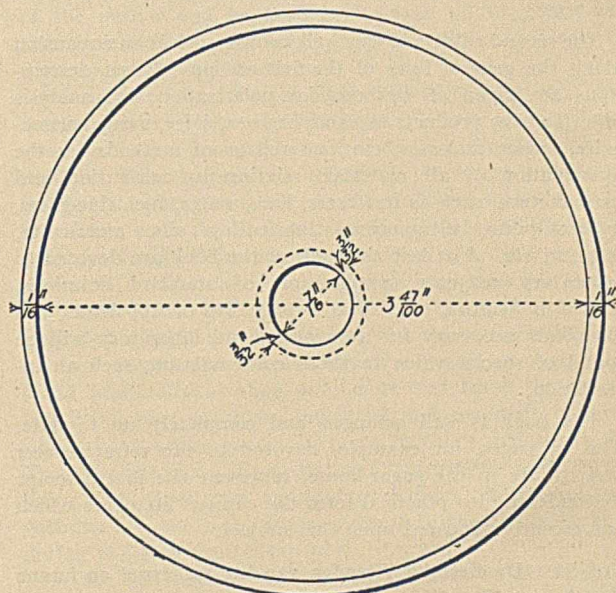
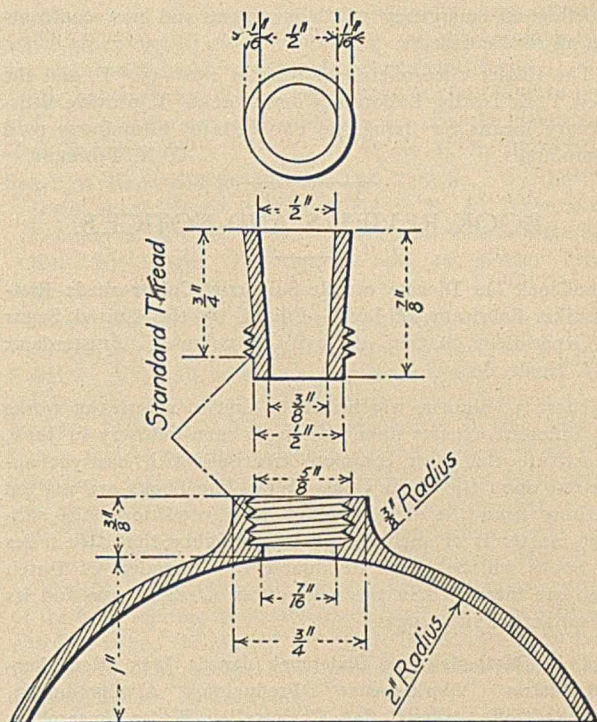
The apparatus, which is made by Howard & Morse, Brooklyn, N. Y., consists of two parts, an aluminum float or saucer and a conical brass collar. The two parts are shown in the drawing, and are made separately for reasons of economy, so that one or two of the floats will be sufficient for an indefinite number of brass collars.

In using the apparatus the brass collar is placed upon a brass plate, the surface of which has been amalgamated, and filled with the bitumen under examination, after it has been softened sufficiently to flow freely by gentle heating. The collar must be level-full, and as soon as the bitumen has cooled sufficiently to handle it is placed in ice water and immediately placed upon the surface of the water, which is maintained at 90° F., or any other temperature desired.

As the plug of bitumen in the brass collar becomes warm and fluid, it is gradually forced out of the collar, and as soon as the water gains entrance to the saucer the entire apparatus sinks below the surface of the same.

The time, in seconds, elapsing between placing the apparatus on the water and when it sinks is determined most conveniently by means of a stop-watch, and is considered as the consistency of the bitumen under examination.

This device has been in general use in the New York Testing Laboratory for some time. An equipment of 12 brass collars, 2 aluminum saucers, a nursery refrigerator for ice water, and an open tank holding about one quart of water and heated by a Bunsen burner is sufficient for testing a great many samples. A thermostat in the water bath will assist in maintaining a constant temperature. From the data presented in the above table, it will



Instrument for determining the consistency of road binders.

<sup>1</sup> N. Y. T. Laby, test at 150° F. = 2 min. 18 sec.



be observed that this device is available at 90° F. for testing almost any compound used in road building except light oils.

In conclusion we should perhaps explain that we appreciate the fact that this device and the proposed method for its manipulation may be open to serious criticism as a strictly scientific method of determining viscosity. It is, therefore, not presented as such, but as a practical tool for meeting an emergency which is urgent and now confronting all road engineers.

The Engler viscosimeter for tests at 250°–350° F. and the New York Testing Laboratory device at 90° F. provide satisfactory means for fixing the two ends of bituminous road compounds.

C. N. FORREST.

## BOOK REVIEWS AND NOTICES.

**Handboek ten Dienste van de Suikerriet-Cultuur en de Riet-suiker-Fabricage of Java.** Edited by the United Sugar Experiment Station of Java. 3 volumes. Amsterdam: J. H. de Bussy.

These 3 volumes, which are the first of a series of works by different experts upon the sugar cane industry in Java, constitute the most complete treatise which has yet appeared upon the sugar cane. While the books are written entirely from the standpoint of Java conditions, the subject matter is of such general applicability that this series of books will be found invaluable to all readers of Dutch who are interested in the cultivation of sugar cane and its manufacture into sugar.

**Vol. I. Methoden van Onderzoek der de Java Rietsuiker-Industrie Voorkomende Producten, Afvalproducten, Brandstoffen, Meststoffen, Chemicalien, Water, Smeeroliën, Enz.** By H. A. P. M. TERVOOREN, Assistent aan het Proefstation voor Suikerriet in West-Java, "Kogok." 2nd. Edit. 1907. 367 pages, 46 cuts. Price, bound, f 7.50.

The second edition of this well known work is an expansion along the general lines of the first edition. Exact descriptions are given of methods for polarization and analysis of sugar cane products as cane, bagasse, juice, syrup, masse-cuite, sugar, molasses, etc., as well as of methods for the examination of all materials relating to cultivation and manufacture, such as fertilizers, lime, water, fuel, flue-gases, hydrosulphite, ultramarine, lubricating oils, molascuite, sulphur, etc. The last 40 pages of the book are devoted to laboratory reagents, preparation of standard solutions, use of indicators, etc. The 8 page list of apparatus and chemicals necessary for a sugar control laboratory will be found of special value to those contemplating such an installation.

The book is well arranged and completely up to date. The 13 pages, for example, devoted to the refractometer and its use in the sugar-house, represent the first attempt to condense into practical form the copious literature which has recently appeared upon this subject.

**Vol. II. De dierlijke Vijanden van het Suikerriet en hunne Parasieten.** By W. VAN DEVENTER, Assistent aan het Proefstation voor Suikerriet in West-Java "Kagok."

1906. 298 pages, 71 cuts, 42 colored plates. Price, bound, f 15.

This treatise upon the animal foes and parasites of the sugar cane is a compilation of the exhaustive researches published in past years by Zehntner, Krüger, and Kobus, together with much new material contributed by the author. It need hardly be stated that the greater part of the work (272 pages) is devoted to the insect enemies of the cane, which are especially prevalent and noxious in Java. The general distribution of the insects described may be seen from the following summary:

Coleoptera (beetles, etc.), 6 families, 19 species.

Lepidoptera (moths, etc.), 8 families, 34 species.

Diptera (flies, etc.), 2 families, 2 species.

Rhynchota (plant-lice), 5 families, 22 species.

Orthoptera (grasshoppers, etc.), 6 families, 29 species.

The remainder of the book is given to a description of a miscellaneous lot of enemies of the cane—apes, rats, mice, wild hogs, birds, crabs, nematodes, etc., of which some 20 different species are described.

The volume is not only an important one from the biological standpoint, but its very full directions for combatting the attacks of the various animal foes and parasites of the cane give the book great practical value to the planter.

The photographic reproductions and colored plates of the volume are splendidly executed and form a most valuable adjunct to the descriptive matter of the text.

**Vol. III. De Fabricatie van Suiker uit Suikerriet op Java.** By H. C. PRINSEN GEERLIGS, Directeur van het Proefstation "Kagok" te Pekalongan. 1907. 449 pages. Price, bound, f. 7.50.

The author of the third volume of this series, whose name is almost synonymous with that of the Java Sugar Industry, needs no introduction to the Sugar World. The present work is an amplification of his previous book, published in 1902, upon "Cane Sugar and the Process of Its Manufacture in Java," which obtained such a wide success.

In his preface Mr. Geerligs states that he has endeavored to condense the results of all chemical researches relating to the constitution of the sugar cane and to the manufacture of sugar therefrom. With this end in view he has avoided so far as possible all discussion of sugar house machinery and equipment, and this more especially since the latter will form the subject matter of Vol. IV in the series of the Handboek.

In the first part of the volume (82 pages) the author discusses the chemical properties of the different constituents of the sugar cane—sucrose, dextrose, levulose, starch, cellulose, pentosans, pectin, acids, wax, nitrogenous substances, mineral matter, etc.—and the distribution of these in the different tissues of the cane.

In the second and principal part of the volume (306 pages) the manufacture of sugar from the cane is taken up. The subject matter of this division is indicated in the following summary:

1. *Extraction.*—Milling, diffusion, composition of juice, composition and fuel value of bagasse, etc.

2. *Clarification.*—Defecation and its influence on the juice constituents, Carbonatation and its influence on the juice constituents, Lime and liming, sulphurous acid, hydrosulphites, phosphoric acid, electrolytic methods, etc. Filtration.



3. *Evaporation.*

4. *Curing.*—Crystallization in motion, purging, working-up of after-products, composition of sugar, conservation of sugar during transport and storage.

5. *Molasses.*—Definition and theory of formation, composition of Java molasses, working up of molasses, etc.

In the third part of the volume (61 pages) the author takes up methods of factory control, calculation of extraction, determination of yields and losses, etc. Specimen pages of forms for factory work are given, together with the tabulation of results obtained in 96 Javan sugar houses.

The whole book is typical of Mr. Geerligs' characteristic breadth of treatment and in his compilations he has given not only the results of his own numerous investigations published in the *Archief voor de Java Suikerindustrie*, but he has also included the researches of investigators in Louisiana, Hawaii, and other cane-producing countries. The completion of this book, coincident with the author's retirement from the directorship of the Sugar Experiment Station Kagok, represents the crowning achievement of 20 years' activity in the Java sugar industry.

Those who are unable to read Dutch will be glad to know that Mr. Geerligs' volume of the *Handboek* is soon to be printed in English. It was the reviewer's privilege recently to go over a part of the proof of this translation with Mr. Geerligs in person. While it has been deemed advisable to omit the more local part of the Dutch volume relating to methods of factory control in the translation, the two more general parts upon the composition of the cane and the manufacture of sugar therefrom will be found to be even more complete than the Dutch original.

Mr. Geerligs' many friends in all parts of the world will wish him continued and greater successes at home in the continuation of his work in the service of his country and of the sugar industry.

C. A. BROWNE.

*Die Raffination des Zuckers.* By WILHELM GREDINGER. Vienna and Leipzig: A. Hartleben. 283 pages with 125 cuts. Price, 12 Marks, bound.

This work, to quote from the author's preface, was written not simply as a book of reference for the practical sugar maker, but also as a guide and handbook for the student.

Owing perhaps to the disinclination of practical sugar men to communicate the knowledge gained by experience, a complete comprehensive work upon sugar refining has up to the present time not yet been written. It was hoped from the preliminary announcement of the publishers that the present work of Gredinger's would fulfil this long-felt need; this expectation, however, has not been fully realized. In the first place the author has limited his treatment of the subject almost exclusively to Austrian conditions with the result that comparatively little is said of the processes of refining followed in other countries. Over 50 pages of the book are devoted for example to the manufacture of cube sugar while that of granulated and other forms of sugar made elsewhere is dismissed with scanty mention. In the second place the language of the author is so full of localisms, unusual expressions of non-Germanic origin (*usuelle*, *praedisposition*, etc.) and unfamiliar idioms that the reader is often unable to grasp the writer's meaning. The criticisms of the book by von Lippmann and other German reviewers are particularly severe as to literary short-comings.

But aside from the two objections just named the present book by Gredinger will be found to contain much matter of interest. The sections upon affining and bone-black are especially well treated. The chapters upon working-up of after-products and molasses are also comprehensive. The various methods of refining without the use of bone-black are briefly described and the questions of refinery losses and yields discussed. The strongest feature of the book is unquestionably the description of machinery and apparatus and the many valuable points of practical value which the author gives concerning refinery management are plainly the result of years of personal experience.

The book is printed upon good paper in plain Roman type and the illustrations and diagrams are for the most part clear and distinct.

C. A. BROWNE.

*Sugar, A Handbook for Planters and Refiners.* By the late JOHN A. R. NEWLANDS and BENJ. E. R. NEWLANDS. London: E. & F. N. Spon. New York: Spon & Chamberlain. 856 pages, 236 figures, and 12 plates. Price, \$10.00, bound.

Every student of sugar and sugar-making is familiar with the work "Sugar" by Lock and Newlands Bros., published in 1888 and now for a long time out of print. The old book in spite of the obsolete character of much of its material had obtained a wide popularity; it was broader than a limited treatise and for a general account of all that pertained to the agriculture and technology of sugar there was no other work with which it could be compared.

In his preface to the new edition the author states that he has endeavored to bring "the subject matter closely up to date." "By leaving out certain parts of the last edition of the book relating to matter which has become more or less obsolete, it has been found possible to introduce a very large amount of new material of a useful character without extending the size of the volume."

The great criticism of the previous edition was that so much of its information was out of date; it was hoped that the new book might be freed from these old encumbrances, and with the injection of new material fulfil the promises of the author and be brought "closely up to date." In the realization of this hope, however, the reader will be more or less disappointed.

Of the 856 pages in the new edition 580 are reprinted without change from the old book. Where methods and processes have remained unaltered the incorporation of material from a previous edition is perfectly justifiable, but in the present case this has been followed with too much literalness, as in the chapter upon Melon Sugar which begins as 20 years ago "The preparation of syrup from the melon (*Cucumis melo*) is fast assuming some importance in America," and so on word for word to the end. The chapters upon Palm Sugar (18 pages), Sorghum Sugar (reduced from 32 to 7 pages) and Maple Sugar (11 pages, 1 page new) stand practically as they did before and leave the reader but little wiser as to the industrial and economic changes which have taken place in these fields of sugar-making. The chapters upon Beet Molasses (13 pages) and Starch Sugar (46 pages) are reproduced without alteration, notwithstanding the many improvements in these departments during the past quarter-century.

Of the 270 pages of new material added to the present edition the 72 pages upon Production and Commerce have



been entirely rewritten; there have also been added 52 pages to the very full chapter on Cane Sugar Factories, 45 pages to the two chapters upon Extraction and Defecation of Beet Juices, 30 pages to Refining and 21 pages to Rum and Alcoholic Liquors. These additions are well written and the text is enriched with many new diagrams and illustrations.

The author in his readjustment of material has unfortunately limited his pruning and grafting almost exclusively to the mechanics and has neglected the chemistry of sugar-making. The excellent bibliography of sugar literature mentioned in the introduction shows that he had the very latest researches at hand upon the chemistry of the sugar cane and sugar beet; it is difficult to understand, therefore, why the author should have reprinted for example the antiquated results of Stenhouse and of Icery (1865) instead of quoting the more recent and far more reliable work of Geerligs. The nomenclature of chemical terms is also in many places archaic and misleading, as for example the continued use of the terms crystallizable sugar for sucrose and uncrystallizable sugar for reducing sugars, globular and granular matter of cane juice, etc. Faulty discrimination is shown in the choice of references as for example (p. 107): "The uncrystallizable sugar present in juice from sound canes consists according to Winter, entirely of dextrose, levulose being found only in diseased and altered canes." The insertion of such matter is doubly unfortunate for it not only misleads the student beginner, but serves to disqualify the book completely in the eyes of the better informed.

Notwithstanding the great progress made in sugar analysis the chapter upon methods is reproduced with but very few additions from the old book. Faulty and obsolete methods of analysis are prescribed and no references at all made to the more recent methods of procedure such as those adopted by the international commission or such well known processes as that of Herzfeld for determining reducing sugars.

The volume, however, as indicated by the title is a handbook for planters and refiners and not a treatise for chemists. For descriptions of agricultural methods and processes of manufacture the reader will not be disappointed; these are all very complete and easily comprehended by means of the numerous plates and illustrations. Much of the material contained in this as in the previous edition is unique in character and for many facts of interest the reader would search elsewhere in vain.

The typography of the volume is good and the illustrations for the most part distinct. In a few cases, however, the cuts (as on page 351) have been reduced to such an extent that there is lack of detail. The binding-in of 32 pages of advertising matter is going somewhat beyond the limit in such matters and in a book already over-weighty only adds to the cumbersomeness of the volume. C. A. BROWNE.

**Laboratory Manual of Dyeing and Textile Chemistry.** By J. MERRITT MATTHEWS, Ph.D. New York: John Wiley & Sons. 363 pp. Price, \$3.50.

This book is intended as an elementary laboratory manual or guide for students in the textile schools, and in other institutions where the many branches of technical chemistry are taught. To more clearly illustrate the points in question the experimental method has been adopted, and is supplemented by additional notes and quiz ques-

tions which serve more firmly to fix the information gained by the experiment.

The subject matter is divided into twenty eight sections, each section corresponding to a weekly apportionment of study. The experimental part will require from eight to twelve hours of laboratory practice each week, though sufficient experiments have been given to allow the teacher considerable latitude as far as selection of material and length of laboratory practice are concerned.

The book starts out with a study of the fibers, and shows the action of acids, alkalies, and salts upon cotton and wool. Next comes a study of the methods employed for scouring of wool, cotton, and silk, followed by the bleaching operations. Thus the various sections follow each other in natural sequence, leading by degrees through the simpler to the more involved processes of dyeing and analytical tests.

In section five is found a classification of the dyes, followed by several sections upon their application and testing, together with a complete list of all the dyestuffs on the market. This last feature is a novel one and is to be highly commended. The sulphur colors are assigned quite an important position, while the sections on mordant dyes, developed dyes, vat dyes, and natural dyes are very ably treated. Considerable space is devoted to the testing of dyes, and the analysis of textile fabrics, while the appendix contains much valuable data for the dyer and textile chemist.

The advent of this book is the fulfilment of a long-felt want for a text on dyeing and textile chemistry. The method of presentation is clear and concise, and as care has been taken to avoid too scientific explanations, it becomes all the more valuable for the purpose for which it is designed. The subject treated is a technical one, and an endeavor has been made to present it in a technical manner; that is to say, definite facts have been presented in a definite form. The book as a whole is well written, and the author deserves great credit for the manner in which he has handled the subject. ALLEN ROGERS.

**Die Theorie der Färbervorgänge.** By DR. PROCOPIOS ZACHARIAS, Privatdozent an der Universität Athen. pp. viii+420. Berlin: Verlag für Textil-Industrie. 1908.

This volume forms an interesting addition to the subject of textile chemistry. It is in reality a German edition, greatly enlarged, of Dr. Zacharias' previous work in Greek. On this account it is especially acceptable, as the latter book (by reason of the language in which it was written) was available to but a limited class of readers.

Though Dr. Zacharias, himself, has done considerable research in the subject of the "Theory of the Dyeing Process," and has embodied his results and theories in his book, nevertheless the chief value of the present volume is its collection of work done by all previous chemists in this field. This compilation is both historical and critical, and extends from the work done by Hellot in 1734 up to and including all work of the present time relative to the subject. Especial attention is paid to the experimental work of Chevreul, Persoz, Bolley, E. J. Mills, Mueller-Jacobs, Knecht, Vignon, Weber, Georgievics, Guehm and Rötheli, Hallitt, and F. Krafft, and a critical discussion is carried on concerning the various theories of dyeing as advanced by these chemists.

The ideas of Dr. Zacharias, himself, have a mathematical



bias with a tendency to reduce the process of dyeing to a differential equation. Though this makes an attractive proposition for the physical chemist, it is not much of an aid to either the practical dyer or the textile chemist.

This book will hardly be of much value to the technical chemist or dyer, but to the broader mind interested in the physical chemistry of textile processes it will prove an interesting and instructive volume. Though the author has not given us much original matter, yet he has assiduously collected a large mass of material relative to dyeing and arranged it in an intelligent and more or less systematic manner. The lack of an index is a serious drawback to the value of the book.

J. MERRITT MATTHEWS.

**Bedeutung der Kolloide für die Technik.** PROF. KURT ARNDT. 40 pp. Dresden: Steinkopff. Price, 1 Mark.

This brochure is a simply and clearly written treatise on recent colloid work connected with a number of technical fields. It is quite full of suggestive matter which must interest chemists working along the lines touched upon. The treatment of each subject is altogether too brief to satisfy more than the general reader. After a brief description of colloids and their characteristics, the application of colloidal chemistry to the following subjects is considered: Ruby and opal glasses, gold and silver mirrors, tungsten lamp filaments, ceramics, cements and glues, color lakes, dyeing, tanning, soaps and fats, sewage purification, and soil fertilization.

W. R. WHITNEY.

**Post's Chemisch-technische Analyse. Handbuch der analytischen Untersuchungen.** Herausgegeben von DR. BERNHARD NEUMANN. Zweiter Band. Braunschweig: F. Vieweg und Sohn. 1909. 800 pp., xiv, 1515, 252 illustrations. Ladenpreis, M. 37.50.

This is the second and final volume of the new edition of an important publication covering Kalk, Kalksteine, Zement und Gips by Drs. Segers und Cramer, pp. 1-80; Touwaren by the same, pp. 81-172; Glas und Glasuren, by the same, pp. 173-208; Rubenzucker, by Dr. R. Frühling, pp. 209-352; Stärke, Dextrin, Traubenzucker, by Dr. E. Parow, pp. 353-424; Bier by Drs. H. Vogel and C. Bleisch, pp. 425-470; Wein, by Dr. P. Kulisch, pp. 471-560; Spiritus und Presshefe, by Dr. H. Hanow, pp. 561-626; Essig, Essigsäure, Verbindungen der Essigsäure, Holzgeist, by Dr. F. Rothenbach, pp. 627-658; Handelsdünger, Stallmist, by Dr. Paul Wagner, pp. 659-680; Bodenarten und Emtesubstanzen, by the same, pp. 681-688; Die Luft, by H. Chr. Nussbaum, pp. 689-716; Ätherische Öle, by Dr. J. Helle, pp. 717-792; Leder und Gerbstoffe, by Dr. Max Philip, pp. 783-850; Leim, by Dr. Richard Kissling, pp. 851-860. Tabak, by the same, pp. 861-870; Kautschuk und Kautschukwaren, 871-908; Spring und Zündstoffe, by Dr. H. Kash, pp. 909-1048; Der Steinkohlentur, by Dr. G. Schultz, pp. 1049-1084; Farbstoffe und Zugehörige Industrie, by the same, pp. 1085-1487; Sachregister, 1490-1514. What was said of Volume I in a previous notice applies also to Volume II.

EDWARD HART.

**Sprechsaal Kalender for the Ceramic, Glass and Allied Industries.** J. KOERNER. 115 pp. Coburg: Mueller and Schmidt.

This publication of 115 pages is one of the most unique attempts in chemical technology. It summarizes all of

the recent work done in Germany in the field of silicates, paying particular attention to the accurate, scientific researches such as the work of Berdel on the vitrification of clay substance, quartz and feldspar, insoluble glazes, testing of glass, Rieke's researches on mica, lime, magnesia and titanitic acid, Simonis' results on mixtures of kaolin, quartz and feldspar, etc. In addition a good deal of general information is given regarding bodies, glazes and glass which is very useful. Dr. Koerner is to be congratulated in producing such a radical change in the makeup of such a publication, and it is to be hoped that this feature will be maintained in the future and include also the results of American research along these lines.

A. V. BLEININGER.

**The Testing and the Properties of Sand Lime Bricks.** H. BURCHARTZ. Royal Materials Testing Station, Gross Lichterfelde. 105 pp. Berlin: Julius Springer.

Exhaustive tests and statistics on the properties of these bricks. This work is probably the most thorough ever done in this field. The recommendations for specifications governing the purchase of sand-lime bricks are especially interesting and are as follows: The bricks must possess good structure and must have a good ring. The water absorption should not be more than 15 per cent. of the weight of the dry brick. When used as backing up bricks or as facing they should have a crushing strength of not less than 2100 pounds per sq. in.; the regular run of hard brick should show a crushing strength of not less than 2800 pounds per sq. in., tested in the dry condition. The bricks must not soften in water and the crushing strength when tested wet should not be lower than when tested dry by more than 15 per cent. They must resist twenty-five successive freezings. The loss in crushing strength on freezing should not be more than 20 per cent. as compared with the dry strength. The crushing strength to be accepted should be the average of ten tests. Variations amounting to 10 per cent. below the minimum requirement in strength are permissible.

A. V. BLEININGER.

**Bulletin No. 9, Illinois State Geological Survey, "Paving Brick and Paving Brick Clays of Illinois.** By C. W. ROLFE, R. C. PURDY, A. N. TALBOT and I. O. BAKER. University of Illinois, Urbana. 1908.

This is a volume of 316 pages, containing three plates and thirty-three illustrations. It covers the Geology and Classification of Clays, 46 pages; Qualities of High-Grade Paving Brick and Tests Used in Determining Them, 87 pages; Qualities of Clays Suitable for Making Brick, 84 pages; Pyro-Physical and Chemical Properties of Paving Brick Clays, 60 pages; Clays Tested which Are Suitable for Use in the Manufacture of Paving Brick, 9 pages; Construction and Care of Brick Pavements. 16 pages. W. D. RICHARDSON.

**The Spanish Translation of the Pharmacopoeia of the United States of America.** Eighth Revision. American Drug-gist Publishing Co., New York. Price, cloth, \$5.00.

The close and friendly relations existing between the United States and Spanish-speaking countries will be further cemented by the appearance of this book, which has been three years in actual preparation. Some delay has been experienced in the completion of the translation, due mainly to the passage of the United States Food and Drugs Act,



June 30, 1906, making necessary some additions and changes in the chemical tests and standards.

The Spanish translation is from the latest revised text and it is confidently hoped that it may prove a potent factor in elevating the professions of medicine and pharmacy by furnishing a guide to the preparation of medicines accurately from drugs of the highest quality.

The Committee of Revision and the editor of the Spanish edition have labored earnestly to produce a standard work which will merit the approbation of scientific men, and science knows no language and no country.

JOSEPH P. REMINGTON,

Chairman of the Committee of Revision  
of the Pharmacopoeia of the United States  
of America (8th Rev.).

The Portland Cement Industry from a Financial Standpoint.

E. C. ECKEL. *Moody's Magazine*. New York. Cloth; 6x9 ins. pp. 93; with two-page map of the United States, showing locations of Portland cement plants. Price, \$2.00 net. C. N. WILEY.

## NEW BOOKS.

Die Bedeutung der Kolloide für die Technik. By PROF. KURT ARNDT. Dresden: Verlag von Theodor Steinkopff. Preis, M. 1.

Reichenbach als Beobachter, S-A aus "Archiv für die Geschichte der Naturwissenschaften und der Technik." By A. BAUER. Leipzig: Verlag F. C. W. Vogel, 1909.

Tabellen für Berechnung von Kalianalysen. DR. R. EHRHARDT. Halle a/S: Wilhelm Knapp's Verlag. 1908. 8°, 69 pp. Price, M. 3.00 net.

Malmaterialienkunde als Grundlage der Maltechnik. By PROF. A. EIBNER. Berlin: Verlag von Julius Springer, 1909. Preis, geh., M.12; geb., M.13.60.

Cement Pipe and Tile; Advantages of Cement for Pipe and Tile, Methods of Manufacture, Tests, Costs, etc. By E. SMITH HANSON. Chicago: Cement Era Publishing Co., 1909. 110 pp., il., cl. Price, \$1.00.

Verdampfen, Kondensieren und Kühlen. Erklärungen, Formeln und Tabellen für den praktischen Gebrauch. By E. HAUSBRAND. 36 fig., 74 tabellen, 4 Aufl. Berlin: Verlag Julius Springer, 1909. Price, geb., M.10.

Untersuchung des Wassers an Ort und Stelle. By HARTWIG KLUT. Berlin: Verlag von J. Springer. Mit 29 figuren und 154 ss. Preis, M. 3.60.

Examination of Water for Sanitary and Technic Purposes by Chemical and Bacteriological Methods. By H. LEFFMAN. 6th ed., rev. and enl., 1909. Phila.: P. Blakiston's Son & Co. il., 12°. Price, \$1.25.

Laboratory Manual of Dyeing and Textile Chemistry. By JAS. MERRITT MATTHEWS. New York: John Wiley and Sons, 1909. 12+363 pp., 8°, cl. Price, \$3.50.

Farbenchemisches Praktikum zugleich Einführung in die Farbenchemie und Farbereitechnik. By RICH. MOHLAN and HANS TH. BUCHERER. Leipzig: Veit & Co. 7 Taf., 8°. Preis, M. 8.

Systematic Treatment of Metalliferous Waste. By L. PARRY. London: *The Mining Journal*, 1909. Price, 5s. net.

A Manual of Volumetric Analysis, for the Use of Pharmacists, Sanitary and Wood Chemists. By H. W. SCHIMPF. 5th ed., rewritten. New York: John Wiley & Sons, 1909. 745 pp., figs., 8°, cl. Price, \$5.00.

## SCIENTIFIC AND INDUSTRIAL SOCIETIES.

### AMERICAN ELECTROCHEMICAL SOCIETY.

Very appropriately the Fifteenth General Meeting of the American Electrochemical Society was held at Niagara Falls, Canada, May 6, 7 and 8, 1909. Thus the society met for the third time at the great American center of the electrochemical industries. The headquarters for the meeting was the Clifton Hotel on the Canadian side and the sessions were held in the Convention Room of the Hotel. The total number of members and guests registered was 316. The principal drawing feature of the meeting was easily the symposium on the Electrometallurgy of Iron and Steel. Unfortunately a number of those scheduled on the program to read papers were not present and their papers were read by others. The program was as follows:

### PROGRAM.

THURSDAY, MAY 6TH.

8.30 A.M.—Registration. Writing room of Clifton Hotel.

9.30 A.M.—Symposium on the Electrometallurgy of Iron and Steel.

(1) Robert Turnbull, Resident American Engineer for Dr. P. L. T. Héroult.

"The Héroult Electric Steel Furnace."

(2) Paul Girod, Director, "S. A. Electrometallurgique Procédés Girod," Ugine, Savoie, France.

(3) Dr. A. Kjellin, Electrometallurgical Engineer Metallurgiska Patent Aktiebolag, Stockholm, Sweden.

(4) Cav. Ernesto Stassano, Director, Forni Ellectrici Stassano, Turin, Italy.

(5) Remo Catani, Electrometallurgist of the "Société Elba," Portoferraio, Elba, Italy.

"Large Electric Steel Furnaces in the Iron and Steel Industry."

(6) Ch. A. Keller, General Manager "Société des Etablissements Keller-Leleux," Livet, Isère, France.

"A Contribution to the Study of Electric Furnaces for the Electrothermic Treatment of Iron and Steel."

(7) Gustav Gin, Electrometallurgist "Société Procédés Gin," Paris.

(8) Henry D. Hibbard, Consulting Engineer, Plainfield, N. J.

"The Present Value of Electric Steel."

(9) F. A. J. FitzGerald, Consulting Electrometallurgist, FitzGerald and Bennie, Niagara Falls.

"The Application of the Lash Process to the Electric Furnace."

(10) P. McC. Bennie, Consulting Metallurgist, Niagara Falls.

"Electric Furnace Pig Iron in California."

(11) Jos. W. Richards, Professor of Metallurgy, Lehigh University.

"The Electric Furnace Reduction of Iron Ore."



- 1.30 P.M.—Intermission for Luncheon.
- 2.45 P.M.—Continuation of Discussion on the Electrometallurgy of Iron and Steel. Reading of papers left over from morning session.
- (12) Furnace Electrode Losses. C. A. Hansen.
- (13) The Working Limit in Electrical Furnaces Due to the "Pinch" Phenomenon. Carl Hering.
- (14) A New Type of Ammeter for the Accurate Measurement of Alternating Currents above 1000 Amperes. E. F. Northrup.
- (15) A New Radiation Pyrometer. Chas. E. Foster.
- (16) The Automatic Registration of Temperatures. E. F. Northrup.
- 8.00 P.M.—Presidential Address, by the retiring President, Mr. E. G. Acheson. Subject: "The Electro-chemist and the Conservation of our National Resources."
- 9.00 P.M.—Smoker.

## FRIDAY, MAY 7TH.

- 9.30 A.M.—Meeting of the Board of Directors. Clifton Hotel.
- 10.30 A.M.—Business Meeting of Society. Announcement of Election of Officers. Reading and Discussion of Papers.
- (17) Addition Agents in Electrolytic Plating Baths. E. F. Kern.
- (18) Electrolytic Precipitation of Lead and Zinc as Affected by the Addition of Certain Organic Compounds. S. A. Tucker and E. G. Thomsen.
- (19) Electro-deposition of Some Metals from Acetone Solution. H. E. Patten and W. R. Mott.
- (20) The Deposition of Aluminium from Aqueous Solutions, using Rotating Cathodes. S. A. Tucker and E. G. Thomsen.
- (21) The Electro-deposition of Platinum. W. J. McCaughey.
- (22) Electrolytic Production of White Lead and Colors. John A. Yunck.
- (23) The Electromotive Force of Cadmium Amalgams. G. A. Hulett.
- (24) Thermodynamics of the Weston Standard Cell. G. A. Hulett.
- (25) The Electrolytic Reduction of Nitric Acid—II. H. E. Patten and W. J. McCaughey.

1.30 P.M.—Adjournment for Luncheon.

3.00 P.M.—Take special cars to the Gate House of the Ontario Power Co. Walk to the plant of the Electrical Development Co., Ltd.; walk to the Canadian Niagara Power Co., thence to entrance of the Ontario Power Company, from which point elevator will take visitors to Power House in the Gorge and to the Transformer Station at the top of the bluff.

7.30 P.M.—Banquet. Clifton Hotel.

## SATURDAY, MAY 8TH.

- 9.00 A.M.—Reading and Discussion of Papers.
- (26) The Probable Electrical Nature of Chemical Energy. A. H. Patterson.
- (27) Bakelite and Its Electrical and Electrochemical Applications. L. H. Baekeland.
- (28) A Graphite Cathode Dish. J. W. Turrentine.
- (29) Relative Efficiency of the Arc and Resistance

- Furnace for the Manufacture of Calcium Carbide. S. A. Tucker, W. A. Alexander and H. K. Hudson.
- (30) Some Physical Properties of Silicon. C. I. Zimmerman.
- (31) Magnetic Tests of Some Iron Alloys. C. F. Burgess.
- (32) Further Experiments with Calcium Alloys as Reducing Agents. O. P. Watts and E. R. Suhm.
- (33) Electrochemical Methods of Soil Analysis. F. K. Cameron.
- (34) A Modified Oxy-hydrogen Gas Coulometer. J. W. Turrentine.
- (35) Rapid Electrochemical Analysis—a Comparison of Several Methods. L. S. Palmer and R. C. Palmer.
- (36) An Answer to Prof. Ganz's Questions on the Corrosion of Iron. W. H. Walker.
- N. B.—Papers (31) and (36) will be called up on Thursday, if time permits.

1.30 P.M.—Adjournment for Luncheon.

2.30 P.M.—Take special cars at Clifton Hotel to plant of Development and Funding Co. (note under announcement of excursions, special conditions under which this invitation is extended to persons in allied lines of manufacture); thence take cars to plant of International Acheson Graphite Co.; thence to Power House of the Niagara Falls Power Company. Thence take cars to Main Street and walk to the Power House of the Niagara Falls Hydraulic Power and Manufacturing Company.

8.30 P.M.—The Niagara Club will tender the Society and its guests an "Open House" at the Club House, corner of Buffalo Ave. and First Street, Niagara Falls, N. Y.

## AMERICAN SOCIETY FOR TESTING MATERIALS.

The Twelfth Annual Meeting of the Society will be held at the Hotel Traymore, Atlantic City, N. J., on Tuesday to Saturday, inclusive, June 29—July 3, 1909.

To avoid the unsatisfactory acoustics in the pavilion used last year for meeting purposes, and in order to be independent of elevator service, the sessions will be held this year in the first-floor parlors of the Hotel Traymore. The program will be arranged with a view of avoiding parallel sessions, and it is expected that provision will be made for an informal dinner and a smoker.

The Fifth Congress of the International Association for Testing Materials will be held in Copenhagen, September 7-11, 1909. Attention is again called to the fact that any member of the Society may become a member of the International Association by forwarding a remittance of \$1.50 to the Secretary of the American Society, and filling out an application blank for membership which will be furnished on request. Members of the International Association are entitled to free copies of all official papers presented at the Congress, including reports of referees and committees on technical subjects. These papers will be printed in German, French and English, and may be procured in any one of these languages.

The non-official papers to be presented at the Congress, consisting of scientific papers by individual members, can be obtained by members of the International Association at a moderate additional charge, which has not yet been fixed. These papers will be printed in their original language only,



followed by abridged summaries in two other languages. A list of the titles of these papers will be forwarded in due course by the Secretary of the American Society to the American members of the International Association.

That the remarkable growth of the Society has been well sustained since the last annual meeting is shown by the following statistics. The total membership reported at the last annual meeting was 1,015. Since then 162 applications for membership have been approved, 28 members have resigned, and there have been two losses by death—V. S. Allien, July 13, 1908, and Thomas Gray, February 12, 1909—making a net gain of 132, and a present membership of 1,147.

#### SUB-COMMITTEES OF GENERAL COMMITTEES ON STANDARD SPECIFICATIONS FOR COALS, AMERICAN SOCIETY FOR TESTING MATERIALS.

##### SUB-COMMITTEE NO. 1.

###### *Coals for Steam Power Plants.*

Goss, Prof. W. F. M., Chairman, University of Illinois, Urbana, Ill.

Stott, H. G., 600 West 59th St., New York, N. Y.

Kolsem, J. C., Indiana Coal Op. Ass'n, Terre Haute, Ind.

McGreath, A. S., 121 Market St., Harrisburg, Pa.

Scholz, Carl, C. R. I. & P. Ry., Old Colony Bldg., Chicago, Ill.

Barrus, Geo. H., 12 Pemberton Square, Boston, Mass.

Capp, J. A., General Electric Co., Schenectady, N. Y.

Gibbs, A. W., Penna. R. R., Altoona, Pa.

Clark, D. O., Union Pacific Coal Co., Omaha, Neb.

Taylor, Ed. H., Old Colony Bldg., Chicago, Ill.

Fleming, H. S., 1 Broadway, New York, N. Y.

Winder, Jno. H., Clinchfield Coal Corporation, Roanoke, Va.

Hume, A. P., Engineer of Tests, American Bridge Co., Pencoyd, Pa.

Garrison, O. L., Big Muddy Coal & Iron Co., Wainwright Bldg., St. Louis, Mo.

Woodwell, J. E., Terminal Bldg., Park Ave. and 41st St., New York, N. Y.

Randall, D. T., U. S. Geological Survey, Pittsburg, Pa.

##### SUB-COMMITTEE NO. 2.

###### *Coals for Producer Gas.*

Fernald, R. R., Chairman, Case School of Applied Science, Cleveland, Ohio.

Carney, F. D., Pennsylvania Steel Co., Steelton, Pa.

Jones, John H., Pittsburg-Buffalo Co., Pittsburg, Pa.

Parr, Prof. S. W., University of Illinois, Urbana, Ill.

Atkinson, John B., St. Bernard Mining Co., Earlington, Ky.

Dulaney, B. L., Black Mountain Coal Land Co., Bristol, Tenn.

Rice, Geo. S., U. S. Geological Survey, Pittsburg, Pa.

##### SUB-COMMITTEE NO. 3.

###### *Domestic and Heating Coals.*

Woodwell, J. E., Chairman, Terminal Building, New York, N. Y.

Lathrop, W. A., 108 South 4th St., Philadelphia, Pa.

Bemont, A., 2114 Fisher Bldg., Chicago, Ill.

Richards, W. J., P. & R. C. & X. Co., Pottsville, Pa.

Toulmin, Priestley, Lehigh Coal Mining Co., Birmingham, Ala.

Moldenke, Richard, Watchung, N. J.

Choney, E. H., 1712 Marquette Bldg., Chicago, Ill.

Dougherty, P. L., U. S. Treasury, Washington, D. C.

##### SUB-COMMITTEE NO. 4.

###### *Metallurgical Coals (for Coke, Gas and Smelting Coals).*

Robinson, C. S., Chairman, Youngstown Sheet & Tube Co., Youngstown, Ohio.

Blauvelt, W. H., Semet-Solvay Co., Syracuse, N. Y.

Eilers, Anton, Am. Smelt. & Ref. Co., 165 Broadway, New York, N. Y.

Moldenke, Richard, Watchung, N. J.

Schluederberg, G. W., Pittsburg Coal Co., Pittsburg, Pa.

Harris, J. R., Tenn. Coal, Iron & R. R. Co., Ensley, Ala.

Belden, A. W., U. S. Geological Survey, Pittsburg, Pa.

Howard, Chas. W., Gen. Mgr., New Etna Coal Co., Chattanooga, Tenn.

Hume, A. P., Engineer of Tests, American Bridge Co., Pencoyd, Pa.

Hahman, William, Lilly Coal Co., Masonic Temple, Altoona, Pa.

Murray, W. J., Victor Fuel Co., Denver, Colo.

Brady, Wm., Chief Chemist, Ill. Steel Co., S. Chicago, Ill.

##### SUB-COMMITTEE NO. 5.

###### *Coals for Illuminating Gas (and Water Gas).*

White, Prof. Alfred H., Chairman, University of Michigan, Ann Arbor, Mich.

Clark, Walton, Broad and Arch Sts., Philadelphia, Pa.

Jones, John H., Pittsburg-Buffalo Co., Pittsburg, Pa.

Fisher, Thomas, Berwind-White Coal Mining Co., Betz Bldg., Philadelphia, Pa.

Page, Wm. N., Gauley Mt. Coal Co., Ansted, W. Va.

Blauvelt, W. H., Semet-Solvay Co., Syracuse, N. Y.

##### SUB-COMMITTEE NO. 6.

###### *Coals for Cement, Drying, Brick Kilns, Etc.*

Orton, Prof. Edward, Ohio State University, Columbus, Ohio.

Hagar, Edward M., Commercial Bank Bldg., Chicago, Ill.

Lesley, R. W., Pennsylvania Bldg., Philadelphia, Pa.

Hutchins, R. G., Jeffrey Mfg. Co., Columbus, Ohio.

Kolsem, J. C., Indiana Coal Op. Association, Terre Haute, Ind.

Mitchell, Robert, Land Title Bldg., Philadelphia, Pa.

Kemmerer, John, Central Bldg., 143 Liberty St., New York, N. Y.

##### SUB-COMMITTEE NO. 7.

###### *Sampling, Testing and Improvement of Coals.*

Randall, D. T., Chairman, U. S. Geological Survey, Pittsburg, Pa.

Haas, Frank, Fairmont Coal Co., Fairmont, W. Va.

Bailey, Edwin G., 93 Broad Street, Boston, Mass.

McGreath, A. S., 121 Market St., Harrisburg, Pa.

Lord, Prof. N. W., Ohio State University, Columbus, Ohio.

Bush, B. F., Continental Trust Bldg., Baltimore, Md.

Wadleigh, F. R., 65 Broadway, New York, N. Y.

Voorhees, S. S., U. S. Geological Survey, Washington, D. C.



Mueller, Frank E., Roberts & Schaefer Co., Old Colony Bldg., Chicago, Ill.

## SUB-COMMITTEE No. 8.

*Forms of Contract (deliveries and acceptances of coals).*

Taylor, E. H., Chairman, Old Colony Building, Chicago, Ill.

Bement, A., 2114 Fisher Bldg., Chicago, Ill.

Bush, B. F., Continental Trust Bldg., Baltimore, Md.

Murray, Wm. J., Colorado Fuel & Iron Co., Denver, Colo.

Dougherty, P. L., U. S. Treasury, Washington, D. C.

Bushnell, Fred. N., c/o Stone & Webster, 147 Milk St., Boston, Mass.

Clark, D. O., Union Pacific Coal Co., Omaha, Neb.

## SUB-COMMITTEE No. 9.

*Educational Work.*

Holmes, J. A., Chairman, U. S. Geological Survey, Washington, D. C.

Dudley, Chas. B., Pennsylvania R. R., Altoona, Pa.

Deems, J. F., N. Y. C. & H. R. R., New York, N. Y.

Wadleigh, F. R., 65 Broadway, New York, N. Y.

Moldenke, Richard, New York, N. Y.

Lathrop, W. A., 108 South Fourth St., Philadelphia, Pa.

Scholz, Carl, Old Colony Building, Chicago, Ill.

Storrs, L. S., New England Inv. & Security Co., Springfield, Mass.

## SEVENTH INTERNATIONAL CONGRESS OF APPLIED CHEMISTRY.

London, 1909.

WEDNESDAY EVENING, MAY 26TH.

Reception by the Lord Mayor and Corporation of the City of London at the Guildhall. (Leveé, Uniform, or Evening Dress.)

THURSDAY, MAY 27TH.

10 A.M.—Meeting of the Joint Organizing Committee.

3 P.M.—Inaugural Meeting, opened by H. R. H. The Prince of Wales, in the Royal Albert Hall. (Morning Dress.)

EVENING.—Reception by the Foreign Office. (Evening Dress.)

FRIDAY, MAY 28TH.

10 A.M. TO 1.30 P.M.—Sectional Meetings.

2.30 P.M.—General Lectures by Professors Haller and Paterno.

EVENING.—Banquet at the Crystal Palace. (Evening Dress.)

SATURDAY, MAY 29TH.

10 A.M. TO 2 P.M.—Sectional Meetings.

AFTERNOON.—Garden Party at the Botanic Gardens by the Ladies' Committee.

EVENING.—Reception by the London Section of the Society of Chemical Industry at the University of London. (Evening Dress.)

MONDAY, MAY 31ST.

10 A.M. TO 1.30 P.M.—Sectional Meetings.

2.30 P.M.—General Lecture by Prof. O. N. Witt.

4 TO 6 P.M.—Sectional Meetings.

EVENING.—Private Receptions. (Evening Dress.)

TUESDAY, JUNE 1ST.

10 A.M. TO 1.30 P.M.—Sectional Meetings.

2.30 P.M.—General Lecture by Sir Boverton Redwood.

4 TO 6 P.M.—Sectional Meetings.

EVENING.—Reception at the Natural History Museum. (Evening Dress.)

WEDNESDAY, JUNE 2ND.

10 A.M.—Official Closing Meeting of the Congress.

AFTERNOON.—Visit to Windsor Castle, by permission of the King.

## UTAH SOCIETY OF ENGINEERS.

On February 19th the Utah Society of Engineers held a public meeting in the hall of the Packard Library, Salt Lake City, for the purpose of discussing the conservation of the natural resources in the intermountain country. Addresses were made by Mr. Clyde Leavitt, U. S. Forest Service, on "Forestry;" Mr. Markham Cheever, Telluride Power Co., on "Water Power;" Mr. William D. Livingston, Irrigated Lands Co., on "Irrigation;" Dr. Robert H. Bradford, University of Utah, on "Mineral Resources;" Mr. Daniel Harrington, formerly of the Utah Fuel Co., on "Fuel Supply;" Mr. E. C. Lackner, Telluride Power Co., on "Water Power from the Standpoint of the Small User;" and Mr. R. W. Saulsbury, Utah delegate to the Conservation Congress. At the March meeting of the Society, held at the University of Utah, papers upon "The Sewer System of Salt Lake City," and "The Filing System in Use in the City Engineer's Office" were read by Messrs. L. H. Krebs and O. H. Skidmore, assistant city engineers. At the annual meeting and banquet, held at the Commercial Club on April 16th, J. F. Merrill, University of Utah, was re-elected *president*; William Ashton, chief engineer Oregon Short Line, *first vice-president*; L. C. Kelsey, city engineer of Salt Lake city, *second vice-president*; D. McNicol, local manager of the Postal Telegraph Co., *secretary*; and J. P. Hornung, resident engineer for the Schott heating system, *treasurer*.

The Fink smelter and smelting process will be explained by Mr. Fink at the May meeting of the Society.

## UTAH ACADEMY OF SCIENCE.

The annual sessions of the Utah Academy of Science were held in Salt Lake City on April 9th and 10th, at the Packard Library. The first session was devoted to papers upon Darwin and the factors of evolution in botany and zoology; and at the second session the regular papers were presented. Officers for the year were elected as follows: *President*, W. C. Ebaugh, Univ. of Utah; *First Vice-president*, E. D. Ball, Agric. Col. of Utah; *Second Vice-president*, W. D. Neal; *Secretary*, A. O. Garrett, S. L. H. S.; *Treasurer*, Philomena Homer, B. Y. U.; and *Members of the Council*, W. W. Henderson, B. Y. C., J. L. Gibson, U. of U., and S. H. Goodwin, Proctor Academy.

## PERSONAL NOTES.

The Arthur D. Little Laboratory announces the reorganization of the coal department with the following personnel:

Dwight T. Randall, M. E.—Engineer in Charge.

Graduate in Mechanical Engineering, University of Illinois. Formerly with R. W. Hunt & Company; Westinghouse, Church, Kerr & Company; in charge of Steam En-



gineering Laboratory, University of Illinois; in charge of steam boiler tests, United States Government Exhibit, St. Louis Exposition; Engineer in charge of fuel tests, Technologic Branch, United States Geological Survey, directing fuel tests, boiler tests, smoke abatement work and gas producer tests.

Perry Barker, M.S.—Assistant Chemical Engineer.

Graduate in Chemical Engineering, University of Illinois; formerly Chemist Peabody Coal Co., Chicago; Research Chemist Illinois State Engineering Experiment Station; Asst. Engineer U. S. Geological Survey at Fuel Testing Plants, St. Louis and Jamestown; later at Experimental Gas Plant, Ann Arbor, in charge of Tests of Gas Coals.

Karl M. Way, E.M.—Assistant Mining Engineer.

Graduate in Mining Engineering, Ohio State University; formerly Assistant Engineer, Fuel Testing Branch, U. S. Geological Survey, engaged in mine inspection, mine sampling, and studies of coal preparation in all important coal fields of the United States.

Charles H. Jumper, B.S.—Assistant Chemist.

Graduate in Chemistry, Rose Polytechnic Institute; formerly with Testing Departments, Pennsylvania and Union Pacific Railroads.

Homer F. Braddock, B.S.—Assistant Chemist.

Graduate in Chemistry, Pennsylvania State College; formerly with Testing Department, Fairmont and Consolidation Coal Companies.

Arthur D. Camp, A.B.—Asst. Gas Chemist and Engineer.

Graduate in Chemistry, Cornell University; formerly Chemist, Mineral Point Zinc Co.; Asst. Superintendent People's Gas Light & Coke Co., Chicago.

Prof. Chas. Baskerville, Director of the Chemical Laboratory, College of the City of New York, sailed for London on May 15th on the steamer Kroonland, to attend the Seventh International Congress of Applied Chemistry. He will be abroad all summer.

R. D. Rhodes, Supt. of the Bingham Junction plant of the U. S. Smelting & Refining Company, has been compelled on account of ill health to retire temporarily from his duties, and Mr. George Metcalf, formerly Asst. Supt. of the Bingham Junction plant, and recently Superintendent of the Yampa Smelter, is now Acting Superintendent for the U. S. Company.

The Chemistry and Metallurgy Class of the Association Institute, conducted by the Y. M. C. A. of Salt Lake City, has been giving a series of public lectures upon Mining Theory and Practice. The following are the subjects and speakers:

April 6.—Geology of Utah, Dr. Fred. J. Pack, University of Utah.

April 12.—"The Bingham Mines," Mr. Louis Cates, Supt. Boston Con. Mine.

April 20.—"Park City Mines," Mr. P. L. Williams, Jr., Gen. Supt. Daly-West and Daly-Judge Mines.

April 27.—"Milling and Concentrating," H. P. Saunders, Consulting Engineer, Salt Lake City.

May 4.—"Copper Smelting," Mr. E. P. Mathewson, Gen. Supt. Anaconda Mining Co.

May 11.—"Lead Smelting," Mr. A. H. Richards, Asst. Supt. A. S. & R. Co. Smelter, at Murray, Utah.

May 18.—"Mining Law," Mr. Frank B. Stephens, Attorney-at-Law, S. L. C.

May 25.—"Mining Stocks," Mr. W. H. Tibbals, Stock Broker, S. L. C.

On Monday, April 5, 1909, the St. Louis Chemical Society sustained a great and painful loss in the death of Mr. Henry August Hunicke, a man whose true worth, untiring energy, enthusiasm and high ideals could be little appreciated except by those associated with him in his scientific pursuits. Mr. Hunicke was born in St. Louis September 21, 1861.

## RECENT PATENTS.

The following patents relating to industrial and engineering chemistry are reported by C. L. Parker, solicitor of chemical patents, McGill Building, 908 G. Street, N. W., Washington, D. C.

908,603. Method of Treating Electrolytic Slimes for the Recovery of Metals Therefrom. ALEXANDER J. MCNAB, Trail, British Columbia, Canada. January 5, 1909.

The method consists in converting one or more of the metals of the slime into soluble sulfids; separating the soluble sulfids, in solution, from the residue; roasting the residue and converting more or less silver therein into sulfate, in solution and precipitating metallic silver from such solution.

908,696. Method of Making Sulfuric Acid. HUGO PETERSEN, Wilmersdorf, Germany. January 5, 1909.

The method consists in generating sulfurous acid gases, passing them in contact with two supplies of nitrous vitriol of different strength in two separate steps, thereby forming two different supplies of Glover acid, submitting the escaping gases to lead chamber action, treating and absorbing the escaping gases which then contain oxids of nitrogen, in two separate steps with the two previously obtained Glover acids, thereby producing two separate supplies of nitrous vitriol and separately treating a new supply of sulfurous acid gases with each of the two supplies of nitrous vitriol.

908,925. Manufacture of Rubber. MEYER WILDERMAN, London, England. January 5, 1909.

This is a process for separating rubber by treating it with a mixture of solvents, of which one when alone is a solvent of all the constituents of the rubber, while another when alone is only a solvent of resins, the mixture being capable of penetrating the whole of the rubber but only a solvent for a part; running off the mixture of solvents containing the inferior constituents of the rubber; evaporating the mixed solvents from the whole of the rubber and resins, first at atmospheric pressure and then under vacuum; and recovering the mixed solvents without separation from the whole of the rubber and resins.

909,167. Waterproofing-paint for Portland Cement Buildings. THOMAS A. EDISON, Llewellyn Park, Orange, N. J. January 12, 1909.

The paint comprises a petroleum residue known as "B. S." dissolved in a suitable solvent, and boiled linseed oil also dissolved in a solvent.



909,168. **Waterproofing Fibers and Fabrics.** THOMAS A. EDISON, Llewellyn Park, Orange, N. J.

The fiber is impregnated with the petroleum residue known as "B. S."

909,169. **Waterproofing Paint for Portland Cement Structures.** THOMAS A. EDISON, Llewellyn Park, Orange, N. J. January 12, 1909.

The paint is produced by dissolving the petroleum residue known as "B. S." in petroleum benzin and removing the insoluble portion.

909,171. **Magnesia Cement.** CARLETON ELLIS, Larchmont, N. Y. January 12, 1909.

This cement is a dry substantially non-hygroscopic powder, comprising oxid of magnesium and saline bodies capable of gradually reacting in the simultaneous presence of water, and said oxid to form chlorid of magnesium; whereby setting qualities are conferred upon the composition, due to the formation of oxychlorid of magnesium.

909,257. **Method of Producing Cellulose Threads.** EDMUND THIELE, Brussels, Belgium. January 12, 1909.

The method consists in permitting a concentrated solution of cellulose in ammoniac copper to issue from comparatively wide apertures into a slowly acting precipitation bath, drawing out the threads so formed in this bath, and subsequently treating them with a boiling caustic lye.

909,277. **Sulfur Color and Process of Making Same.** EMILE T. BUNDSMAN, Point Loma, Cal. January 12, 1909.

The dye is made by heating together a mixture of grape sugar, meta-nitranilin, and a sulfuring agent.

909,343. **Manufacture of Tanning Extracts.** DONALD STEWART, Iverness, Scotland. January 12, 1909.

The extracts are made by passing successively through vats or pits containing tannin-containing material waste sulfite liquor whose ash contains not more than 6 per cent. of iron calculated as FeO, and then settling out difficultly soluble substances and concentrating the clear liquor.

909,536. **Composition of Matter for Generating Hydrogen.** GEORGE F. BRINDLEY, Niagara Falls, Ontario, Canada. January 12, 1909.

The composition contains sodium, an inert substance and an agent for temporarily preventing the oxidation of the sodium.

909,546. **Smokeless Gunpowder.** CONRAD P. H. CLAESSEN, Wilmersdorf, Germany. January 12, 1909.

The powder consists of a mixture of nitrocellulose and nitroglycerin with about 5 per cent. diethyldiphenyl-caramid.

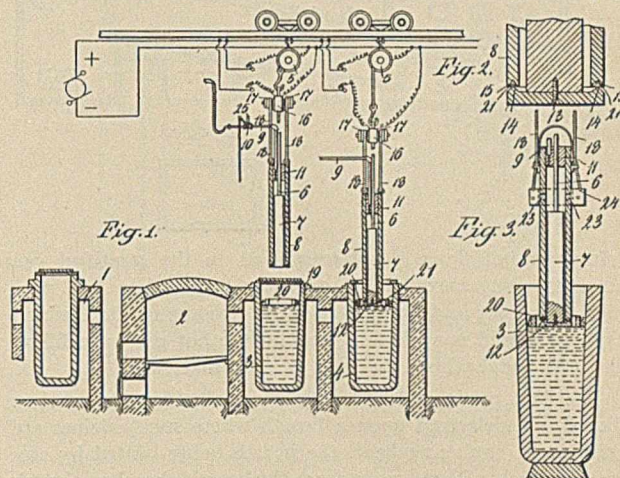
909,667. **Process of Obtaining Nickel from Silicious Ores.** EDGAR F. PRICE, Niagara Falls, N. Y. January 12, 1909.

This is a process of producing ferro-nickel by smelting a charge containing a silicious ore of nickel and iron and carbon by means of an electrically-heated resistance-conductor, withdrawing the slag and product from the furnace and supplying the charge-mixture as required.

909,924. **Compound Metal Body and Process of Producing Same.** JOHN F. MONNOT, New York, N. Y. January 19, 1909.

In the practice of this method, a bar of aluminum is

cleaned thoroughly, and is then heated under conditions precluding oxidation. This heat may be just enough to insure a dry surface, or it may be somewhat higher. The bar, or ingot, is then withdrawn under conditions precluding oxidation and other surface deterioration, and is then plunged momentarily into a bath of silver, copper, or other suitable metal of relatively high melting point, preferably a metal which is not readily oxidizable, such metal being maintained in a very fluid condition, and preferably heated considerably above its melting point. If only a thin coating of copper or other coating metal is desired, the ingot may be withdrawn after a few seconds, under conditions pre-



cluding oxidation, and allowed to cool; and when cool will be found to be coated with a thin cohering film of the coating metal having the desirable properties of a metal solidified from the fluid state. Such coating so formed is very thin. If a thicker coating is desired such coating may be formed by confining a portion of the molten metal, of suitable thickness, in contact with the surface of the aluminum body, and then withdrawing the aluminum body, and confined layer and permitting the latter to solidify. The compound ingot thus produced is then preferably worked, as by rolling, pressing, hammering or like mechanical operation.

The illustration shows apparatus for carrying out the process.

910,049. **Composition for Removing Finish.** CARLETON ELLIS, Larchmont, N. Y. January 19, 1909.

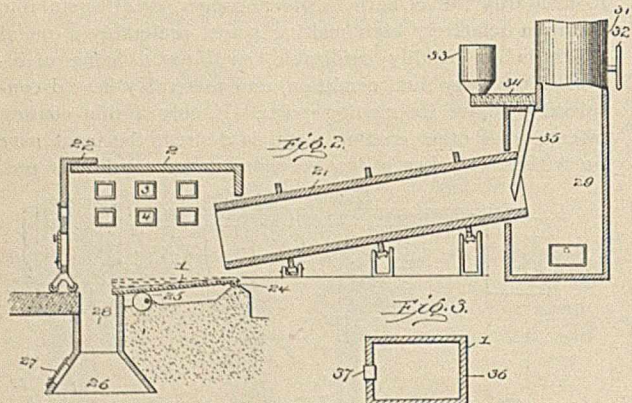
The remover comprises turpentine with which a metallic resinat has been incorporated and acetone added thereto.

910,121. **Process of and Apparatus for Making Cement Clinker.** CARLETON ELLIS, Larchmont, N. Y. January 19, 1909.

In this process of making cement clinker, the two operations of calcining and clinkering are more or less segregated, each being performed in apparatus particularly adapted thereto. The calcining is performed in a rotary kiln with the aid of a stream of hot waste gases substantially filling the whole cylinder and therefore contacting with the stream of cement material instead of being spaced away therefrom as in the ordinary kiln, developing the flame yielding the waste gases elsewhere than in this kiln so that there will be no necessity for instituting draft conditions and consequently stratification of gases therein. In some measure,



the sheer temperature required for calcination is reciprocally related to the percentage of carbon dioxide in the gas mass bathing the material being calcined so that in the ordinary operation where the evolved carbon dioxide tends to flow forward over the cement material as a discrete stream under-



lying the hot flame gas stratum, as in the stratified gas currents going through the ordinary kiln, calcination conditions are not as favorable as in the present method, where evolved dioxide is at once diluted in the hot gas mass filling the kiln and bathing the solid cement material.

The hot calcines, free or substantially free, of carbon dioxide, are delivered upon a hearth where such calcines are raised to a clinkering heat, the hearth being heated by any suitable fuel. Waste gases from the hearth chamber, though preferably but a portion of such gases, are passed upward through the rotary kiln to effect calcination in the manner described. It will be noted that the described operation makes calcination and clinkering wholly independent operations though retaining the advantage of the ordinary practice of using but a single source of heat. There is, of course, no necessary relation between the speed of feed of materials through the rotary kiln and the length of time that materials remain on the hearth, and each operation can be independently regulated.

The illustration shows the apparatus in which the process is carried out.

**910,524. Process for the Production of Soluble Starch.** FERDINAND FRITSCH, Rumburg, Austria-Hungary. January 26, 1909.

The process consists in mixing common starch with a perborate and heating said mixture.

**910,530. Method of Absorbing Oxids of Nitrogen.** BIRGER F. HALVORSEN, Christiania, Norway. January 26, 1909.

This method consists in treating the oxids of nitrogen with water and conducting the resulting gases into an absorbent containing caustic alkali and a carbonate of the alkalis.

**910,662. Process of Making Caustic Alkalies.** WILLIAM T. GIBBS, Buckingham, Quebec, Canada. January 26, 1909.

The process consists in digesting finely divided potash feldspar with milk of lime under steam pressure.

**910,707. Process of Treating Iron Ore.** CHARLES B. MORGAN, Oakland, Calif. January 26, 1909.

The ore is first melted to a liquid state then a sufficient supply of liquid carbonaceous material is added to effect a reduction of the iron.

**910,894. Process of Making Titanium Alloys.** FREDERICK M. BECKET, Niagara Falls, N. Y. January 26, 1909.

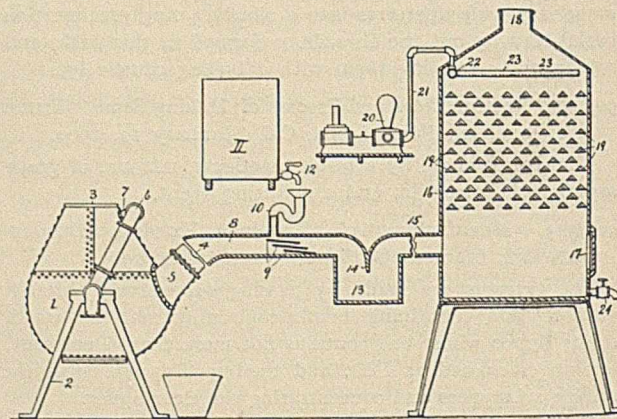
The titanium ore is reduced in an electric furnace by means of carbon in the presence of sufficient silicon to exclude from the product carbon in excess of 10 per cent.

**910,936. Dinitroglycerin Explosive and Process of Making.** ANTON MIKOLAJZAK, Kastrop, Germany. January 26, 1909.

The explosive is produced by dissolving gelatinating media in dinitroglycerin and incorporating solid inorganic oxidizing salt, with the mixture.

**910,982. Process of Forming Metal Salts.** WILLIAM H. ALLEN, Detroit, Mich. January 26, 1909.

In this process furnace gases containing metals in a finely divided or gaseous state, are passed into a retort where a portion is condensed. The gases then pass through a horizontal pipe containing baffle plates over which hydrochloric



or sulfuric acid is allowed to flow, which causes a further separation of metal in the form of chlorides or sulfates. The furnace gases are then passed through a large chamber in which are a number of triangular bars over which water or dilute acid flows to effect a further separation of the melted salts.

**911,140. Leather Product and Process for Making the Same.** HIGH MACKAY, El Paso, Texas. February 2, 1909.

This is a process of surface hardening sole leather by soaking the same in an aqueous mixture of Portland cement and borax.

**911,254. Ammonia-cyanid Process of Treating Ores Containing Precious Metals.** DAVID MOSHER, San Francisco, Calif. February 2, 1909.

The process consists in treating copper, nickel, zinc and cobalt ores and tailings with gold and silver values, by simultaneously extracting all the metals with a solution of ammonia and an alkaline earth-metal cyanid.

**911,269. Process of Producing Certain Proteids from Butter-milk.** LUDWIG H. REUTER, Berkeley, Calif. February 2, 1909.



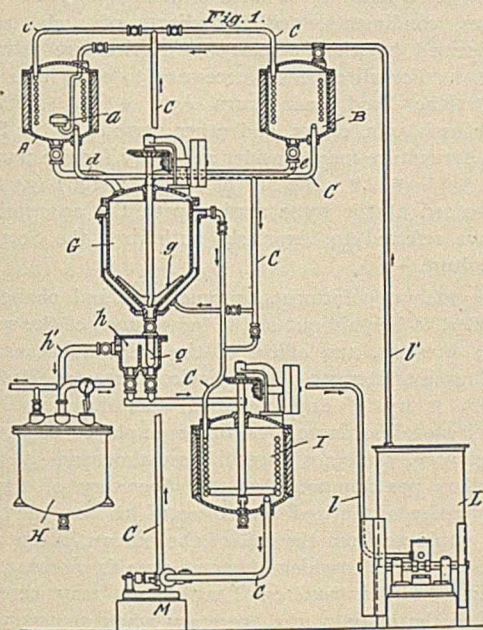
The process consists in treating buttermilk by neutralizing the acid thereof at a low temperature, then moderately heating for a considerable period of time, then heating for a short time almost to the boiling point, thereafter cooling and filtering and subsequently precipitating the casein.

911,283. Process of Making Fertilizers. JOHN R. YOUNG, Norfolk, Va. February 2, 1909.

In this process tricalcium phosphate is mixed with undried press-cake of nitrogenous refuse, and potash containing vegetable matter, and the mixture is then decomposed by strong sulfuric acid, the water for the reaction being supplied from the moisture contained in the press-cake of nitrogenous refuse.

911,553. Process for Purifying Crude Petroleum and Its Distillates. LAZAR EDELEANU, Ploieschi, Roumania. February 2, 1909.

This process consists in treating the crude petroleum, its distillates and its products with sulfur liquefied dioxid. The inventor has discovered that the coloring matters, the disagreeably smelling compounds, and the constituents rich in carbon and poor in hydrogen of the crude petroleum, which injure the quality of the petroleum products, are soluble in liquefied sulfur dioxid, whereas the hydrocarbons



which constitute the refined product are difficulty soluble or altogether insoluble in liquefied sulfur dioxid, supposedly consisting principally of paraffins and naphthenes. This discovery enables the inventor to separate by a physical process the prejudicial constituents of crude petroleum of its distillates merely by using the liquefied sulfur dioxid as a solvent.

The illustration shows one form of apparatus in which the process can be satisfactorily carried out.

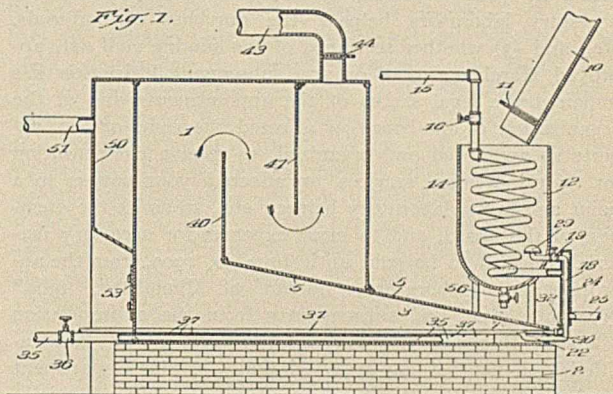
911,606. Surface Finish. PERCY NEYMANN, Chicago, Ill. February 9, 1909.

The composition consists of wax, a solvent, coloring matter, and an ingredient for hardening the superficial

wax, composed of flowers of sulfur, butter of antimony, and acetic acid.

911,735. Process of Making Sulfur Dioxid. CHESTER W. LYMAN, New York, N. Y. February 9, 1909.

This invention relates to a new method of burning sulfur to produce sulfur dioxid and consists essentially in introducing the melted sulfur in the form of spray into a suitable retort and there burning it. The melted sulfur and air are introduced under pressure through nozzles so arranged that the air jet impinges on the inflowing sulfur, whereby the



sulfur is atomized, and at once intimately mixed with the air within the retort. This process is stated to possess the advantage of effecting the result of intimately mixing the sulfur and the air, whereby there is obtained practically perfect combustion of the sulfur and the combination therewith of a larger proportion of the oxygen of the air than usually obtained.

The illustration shows the apparatus in which the process is carried out.

912,245. Solder for Incandescent-lamp Filaments. HANS KUZEL, Baden, near Vienna, Austria-Hungary. February 9, 1909.

This means of connecting incandescent lamp filaments to their supply wires, comprises a solder containing carbide of tungsten.

912,266. Hydraulic Cement. ARTHUR C. SPENCER and EDWIN C. ECKEL, Washington, D. C. February 9, 1909.

This is a mixture containing greensand and calcareous material.

OFFICIAL REGULATIONS AND RULINGS.

FOOD INSPECTION DECISION 107.

*Decision of the Attorney-General in regard to the legality of the Referee Board.*

The decision of the Attorney-General in regard to the legality of the Referee Board is hereby promulgated as Food Inspection Decision No. 107. JAMES WILSON,

*Secretary of Agriculture.*

WASHINGTON, D. C., April 22, 1909.

DEPARTMENT OF JUSTICE,  
WASHINGTON, April 14, 1909.

*The Honorable The Secretary of Agriculture.*

SIR: I am in receipt of your favor of the 23d ultimo,



asking my opinion with respect to (1) the legality of the appointment by you of five scientific consulting experts to give you necessary advice upon questions arising in the enforcement of the Food and Drugs Act, June 30, 1906, whose salaries and expenses you have directed to be paid from the appropriation "Laboratory, Department of Agriculture" (34 Stat., 1271); and inquiring specifically (2) whether you were, on February 20, 1908, authorized to form these five consulting experts into a board, and to pay the expenses incident to the investigations made by such board at your direction, including the compensation of necessary laboratory helpers, the purchase of materials, etc., and (3) whether section 9 of the sundry civil act, approved March 4, 1909, or any subsequent legislation has impaired the legal status of the appointments and of the organization of the board, or affected the right of the experts so appointed and organized, to receive compensation for their individual services, or affected your powers to a point assistants, laboratory helpers, etc., to assist the members of the board, and to incur expenses for necessary material, etc., all to be paid until June 30, 1909, from the appropriation "Laboratory, Bureau of Chemistry, 1909" (35 Stat., 260), and subsequently from the appropriation "General Expenses, Bureau of Chemistry, 1910" (act entitled "An act making appropriations for the Department of Agriculture for the fiscal year ending June thirtieth, nineteen hundred and ten," approved March 4, 1909).

1. As to the legality of the appointment. The Food and Drugs Act, after prohibiting the introduction into any State or Territory, or the District of Columbia, from any other State or Territory or the District of Columbia, or from any foreign country, or shipment to any foreign country of any article of food or drugs which is adulterated or misbranded, within the meaning of the act, enacts in Section 2:

That the Secretary of the Treasury, the Secretary of Agriculture, and the Secretary of Commerce and Labor shall make uniform rules and regulations for carrying out the provisions of this act, including the collection and examination, of specimens of foods and drugs manufactured or offered for sale in the District of Columbia, or in any Territory of the United States, or which shall be offered for sale in unbroken packages in any State other than that in which they shall have been respectively manufactured or produced, or which shall be received from any foreign country, or intended for shipment to any foreign country, or which may be submitted for examination by the chief health, food, or drug officer of any State, Territory, or the District of Columbia, or at any domestic or foreign port through which such product is offered for interstate commerce, or for export or import between the United States and any foreign port or country.

Section 3 enacts:

That the examinations of specimens of foods and drugs shall be made in the Bureau of Chemistry of the Department of Agriculture, or under the direction and supervision of such Bureau, for the purpose of determining from such examinations whether such articles are adulterated or misbranded within the meaning of this Act; and if it shall appear from any such examination that any of such specimens is adulterated or misbranded within the meaning of this act, the Secretary of Agriculture shall cause notice thereof to be given to the party from whom such sample

was obtained. Any party so notified shall be given an opportunity to be heard, under such rules and regulations as may be prescribed as aforesaid, and if it appears that any of the provisions of this act have been violated by such party, then the Secretary of Agriculture shall at once certify the facts to the proper United States district attorney.  
\* \* \* \*

The statutes of the United States do not provide for the creation of the Bureau of Chemistry in the Department of Agriculture. The existence of such Bureau is recognized in the appropriation acts, and in the act entitled "An act to make appropriations for the Department of Agriculture for the fiscal year ending June thirtieth, nineteen hundred and eight" (34 Stat., 1271), under the head of "Bureau of Chemistry" appropriations are made for the salaries of "One chemist, who shall be chief of Bureau," and a certain number of clerks, laborers, messengers, etc., after which, under the subheading of "Laboratory, Department of Agriculture," a lump sum appropriation was made for "necessary expenses in conducting investigations in this Bureau, including \* \* \* work in such investigations, in the city of Washington and elsewhere \* \* \* ; for the employment of additional assistants and chemists, when necessary \* \* \* ; to investigate the composition, adulteration, and false labeling, or false branding of foods, drugs, beverages, condiments, and ingredients of such articles, when deemed by the Secretary of Agriculture advisable \* \* \* . For all expenses necessary to carry into effect \* \* \* [the Food and Drugs Act] \* \* \* employing such assistants, clerks, and other persons as the Secretary of Agriculture may consider necessary for the purposes named \* \* \* ." The act of March 4, 1907 (34 Stat., 1280), passed at the same session with the appropriation act above referred to, expressly authorizes the Secretary of Agriculture.—

to make such appointments, promotions, and changes in the salaries, to be paid out of the lump funds of the several bureaus, divisions, and offices of the Department as may be for the best interests of the service: *Provided*, That the maximum salary of any classified scientific investigator in the city of Washington, or other employee engaged in scientific work, shall not exceed three thousand five hundred dollars per annum. And the Secretary of Agriculture is hereby authorized and directed to pay the salary of each employee from the roll of the bureau, independent division, or office in which the employee is working, and no other: *Provided, however*, That details may be made from or to the office of the Secretary when necessary and the services of the person whom it is proposed to detail are not required in that office; and he is further authorized and directed to submit to Congress each year a statement covering all appointments, promotions, or other changes made in the salaries paid from lump funds, giving in each case the title, salary, and amount of such change or changes, together with reasons therefor. (34 Stat., 1280).

Pursuant to the provisions of Section 2 of the Food and Drugs Act, the Secretary of the Treasury, the Secretary of the Agriculture, and the Secretary of Commerce and Labor, on October 17, 1906, promulgated certain rules and regulations for carrying out the provisions of the act. Regulations 3 and 4 dealt with the collection of samples and the methods of analysis. Regulation 5, "Hearings," is as follows:



(a) When the examination or analysis shows that the provisions of the Food and Drugs Act, June 30, 1906, have been violated, notice of that fact, together with a copy of the findings, shall be furnished to the party or parties from whom the sample was obtained or who executed the guaranty as provided in the Food and Drugs Act, June 30, 1906, and a date shall be fixed at which such party or parties may be heard before the Secretary of Agriculture or such other official connected with the food and drug inspection service as may be commissioned by him for that purpose. The hearings shall be private and confined to questions of fact. The parties interested therein may appear in person or by attorney and may propound proper interrogatories and submit oral or written evidence to show any fault or error in the findings of the analyst or examiner. The Secretary of Agriculture may order a reexamination of the sample or have new samples drawn for further examination.

(b) If the examination or analysis be found correct the Secretary of Agriculture shall give notice to the United States district attorney as prescribed \* \* \*.

The appropriation act of 1908 (35 Stats., 251, 261) made appropriations for the fiscal year ending June 30, 1909, and contained provisions in the lump sum appropriation for "Laboratory, Department of Agriculture" similar to those above quoted from the act of 1907, except that the sentence "for the employment of additional assistants and chemists" was not included in the enumeration of the objects for which the lump sum appropriation was made.

The appropriation act of 1909 (Public No. 330) contains similar provisions to those above cited from the act of 1908. Under these acts, I am clearly of the opinion that the Secretary of Agriculture was empowered to employ in the Bureau of Chemistry such additional assistants and chemists as he should deem necessary to investigate the composition, adulteration, and false labeling, or false branding of foods, drugs, beverages, condiments, and ingredients of such articles, when deemed advisable by him, and such assistants "and other persons" as he might deem necessary to carry into effect the Food and Drugs Act.

The form of appointment which you made, which accompanies your letter, shows that you appointed each of certain persons "consulting scientific expert to the Secretary of Agriculture, to aid in enforcing the provisions of the" Food and Drugs Act, in the Department of Agriculture at a salary of \$25 per day, for days actually employed, to be paid from the appropriation "Laboratory, Department of Agriculture, General Expenses, Bureau of Chemistry," to perform such duties as should be required by the Secretary. While the form of appointment does not expressly specify that the expert is employed as a part of the Bureau of Chemistry, that fact is implied from the specification of the fund from which he is to be paid. In my opinion these appointments were expressly authorized by the acts of Congress referred to.

2. You further inform me that you organized the five persons so appointed into a board called the "Referee Board," and that you imposed upon them the duty to consider and report to you upon the wholesomeness, or the deleterious character of such foods, or of such articles used in foods as you might refer to them. I do not understand from your communication that you conferred upon this so-called

Referee Board any *power*. Their sole function was to investigate and report to you, and their detail to your office is justified in the provision of the act of March 4, 1907, above quoted. The purposes for the employment of these gentlemen, and the organization of them by you into a board, are set forth in your letter. You point out that it was to enable you to have recourse to the disinterested and unbiased advice of eminent and expert chemists whenever a serious conflict of opinion may arise as to the deleteriousness of any particular article or substance added to food. It is, of course, apparent that in the administration of a statute of such far-reaching effect as the Food and Drugs Act, the ordinary investigation and conclusions of the Bureau may be disputed by interested parties, and Section 4 of the act provides for a rehearing by the Secretary of Agriculture whenever the conclusion of the Bureau is disputed. The Secretary would naturally desire to reach a right conclusion as to such matters and not subject the owners of articles affected by the ruling to litigation if any error should have been committed by the Bureau, and Congress would seem to have had that in mind in providing in the lump sum appropriations of 1907 and 1908, for the employment of "such assistants, clerks, and other persons, as the Secretary of Agriculture may consider necessary for the purposes named," *i. e.*, the investigation of the composition, adulteration, and false labeling, or false branding of foods, drugs, beverages, etc., when deemed by him advisable. Your right to appoint any one of these men for that purpose can scarcely be seriously disputed under the provisions of the act above referred to, and, in my opinion, you were entirely justified in directing them to confer and act as a committee or board in advising you with respect to the enforcement of the act.

3. The act entitled "An act making appropriations for sundry civil expenses of the Government for the fiscal year ending June thirtieth, nineteen hundred and ten, and for other purposes," approved March 4, 1909 (Public No. 328), contains the following provision:

SECTION 9. That hereafter no part of the public moneys, or of any appropriation heretofore or hereafter made by Congress, shall be used for the payment of compensation or expenses of any commission, council, board, or other similar body, or any members thereof, or for expenses in connection with any work or the results of any work or action of any commission, council, board, or other similar body, unless the creation of the same shall be or shall have been authorized by law; nor shall there be employed by detail, hereafter or heretofore made, or otherwise personal services from any executive department or other government establishment in connection with any such commission, council, board, or other similar body.

You inform me that since this enactment a question has been raised as to your right to cause payments to be made to the above-mentioned experts, and you ask my opinion as to whether or not such objections are well founded. In my opinion this section last quoted does not repeal the provisions of the appropriation act passed at the same session, authorizing the Secretary of Agriculture to employ "such assistants, clerks, and other persons as he may consider necessary" to enable him to carry into effect the provisions of the Food and Drugs Act, nor to submit to a number of persons appointed pursuant to that act, to consider



jointly as a committee or board, and report to him for his information, any question upon which he is by law required to take action arising under that act. The commissions or boards referred to in Section 9 of the act of March 4, 1909, are commissions or boards constituted without authority of law, and I can not conceive that it could ever be construed to prohibit the head of a Department from submitting to the concurrent investigation and report of several employees of his Department any question which he might submit for investigation to any one of them. Inasmuch, therefore, as the employment of experts of the character referred to by you is authorized by law, and appropriations made out of which they may be paid for their services, as above set forth, I am of the opinion that neither Section 9 of the sundry civil act, approved March 4, 1909, above referred to, nor any other legislation to which my attention has been called, has affected your right to employ such experts or submit to their joint investigation and report, any question of fact affecting the adulteration or misbranding of articles concerning which any party from whom such articles have been obtained is entitled to be given an opportunity to be heard under the provisions of Section 4 of the Food and Drugs Act.

Respectfully,

GEO. W. WICKERSHAM,  
*Attorney-General.*

The following Judgment Notices have been issued under the Food and Drugs Act:

MARCH 16, 1909.

- 39. Misbranding of corn and beans (Underweight).
- 40. Misbranding of canned corn (As to presence of saccharin).
- 41. Adulteration of water (Great Bear Spring).
- 42. Misbranding of butter (Renovated butter).

APRIL 12, 1909.

- 50. Adulteration of coffee (Coated with lead chromate).

APRIL 15, 1909.

- 51. Misbranding of bottled beer (As to place of manufacture).
- 52. Misbranding of canned corn (Underweight).
- 53. Misbranding of canned corn (Underweight).

APRIL 29, 1909.

- 54. Misbranding of a drug (Muco-Solvent).
- 55. Misbranding of coffee (As to geographical source).

MAY 7, 1909.

- 56. Misbranding of lemon extract (As to presence of oil of lemon).
- 57. Misbranding of canned apples (Underweight).

(T. D. 29701). *Chrome Alum.*

KUTTROFF *v.* UNITED STATES.

U. S. Circuit Court of Appeals, Second Circuit. April 13, 1909. No. 154 (suit 5024).

CHROME ALUM—CHEMICAL SALT—"CRUDE STATE."

Chrome alum, which after production has been subjected to a crystallizing process, being thereby freed from incidental impurities, is by reason of this process removed from the

provision in paragraph 482, tariff act of 1897, for articles in a "crude state," used in dyeing, and is dutiable as a chemical salt under paragraph 3.

APPEAL from the Circuit Court of the United States for the Southern District of New York.

[Decision in favor of the Government.]

The decision below (T. D. 29003) affirmed a decision by the Board of United States General Appraisers, G. A. 6647 (T. D. 28346), which had affirmed the assessment of duty by the collector of customs at the port of New York on merchandise imported by Kuttroff, Pickhardt & Co.

*Curie, Smith & Maxwell* (W. Wickham Smith of counsel), for the importers.

J. Osgood Nichols, assistant United States attorney, for the United States.

Before LACOMBE, COXE, and WARD, Circuit Judges.

This cause comes here upon appeal from a decision of the Circuit Court, affirming a decision of the Board of General Appraisers, which affirmed the collector. The importation is commercially known as chrome alum, and was classified under paragraph 3, act of 1897, as a "chemical salt." It is concededly a chemical salt, but the importers contend that it is entitled to free entry under paragraph 482:

Articles in a crude state used in dyeing or tanning not specially provided for.

Per CURIAM: The only question in the case is whether the article is "in a crude state." It is derived as a by-product from the process of manufacturing certain coal-tar colors. In such process chromium sulphate and potassium sulphate are formed, and these two products combined with water constitute chrome alum; but, as the evidence shows, it remains at close of the process, "as a grayish, greenish paste." This seems to be its crude state, but the paste is thereafter treated with sulphuric acid, and from the solution the chrome alum of the importation crystallizes out, being thereby freed from incidental impurities. We concur with the Board and the Circuit Court that chrome alum thus crystallized is not in a crude state.

(T. D. 1483). *Alcohol for Scientific Purposes.*

Alcohol classified and branded as "commercial alcohol" may be withdrawn from bond, free of tax, under the provisions of Section 3297, Revised Statutes, for scientific purposes.

TREASURY DEPARTMENT.

OFFICE OF COMMISSIONER OF INTERNAL REVENUE

Washington, D. C., April 14, 1909.

SIR: In reply to your letter of the 8th instant, requesting that you be advised whether, in view of the reclassification of distilled spirits, "commercial alcohol" may be withdrawn from bond, free of tax, for scientific purposes, under the provisions of Section 3297, Revised Statutes, you are informed that the honorable Attorney-General, to whom this question was recently submitted, holds that spirits as above designated may be so withdrawn for the purpose stated.

Respectfully,

J. C. WHEELER,  
*Deputy Commissioner.*

Mr. AUGUST E. MUENTER,  
*Collector First District, San Francisco, Cal.*



## INDUSTRIAL AND TRADE NOTES.

FEDERAL COURT.

The completion April 24 of the custom zinc concentrator of former United States Senator W. A. Clark marked an epoch in the mining industry of Butte.

This plant, the first of its kind in the Northwest, will mean the successful and profitable mining of large bodies of low-grade silver-zinciferous ores, in which the mines of Butte abound and which, heretofore, have defied successful treatment. In fact, it is believed that the Clark method of handling zinc ores will in time bring Butte to the front as one of the principal zinc-producing districts in the world, in addition to its enormous copper output.

The Clark concentrator, the result of long and elaborate experiments, will begin receiving custom zinc ores this week, starting on shipments from the Butte & Superior and the Elm Orlu mines. The plant has a capacity of 300 tons of ore a day, and will produce about 125 tons of about 40 per cent. zinc concentrates, carrying in addition about 25 ounces of silver, about 4 per cent. copper and \$1 in gold. About two or two and one-eighth tons of crude ore will be concentrated into one ton of product, the grade of the crude ore determining in a measure the degree of concentration.

On May 4th, the Senior Engineering students of the University of Utah leave for their trip of inspection through Utah and neighboring states. The more important mining camps and smelting districts of Utah, Colorado, Montana and Nevada will be visited.

Work is progressing upon the plant of the International Smelting Company at Tooele. Early in April there was held an auction sale of the lots in the new smelter town, and about \$100,000.00 worth of property was sold within a few hours. The company is conducting this enterprise along the lines followed at Anaconda, Mont., with so much success.

## NITRIC ACID FROM AMMONIA.—OSTWALD—BRAUER PROCESS.

Notes on lecture by Morse, of Harvard, before the American Chemical Society, April 8, 1909.

The work on this process started in Ostwald's laboratory at Leipsic but was afterwards given a commercial trial by Griesheim. Brauer afterwards put up works devoted entirely to this process. As finally worked a mixture of air and ammonia gas must pass through a catalyzer which consists of spirally wound sheet platinum of roughened surface. The temperature is held at a very dull red heat. After the reaction is started the temperature takes care of itself. No definite reaction need be given as different oxids of nitrogen may be produced. From the catalyzer the gas passes to condensation chambers. Five condensing towers in series are used. The weak acid is used as a wash water in the first tower. The best acid is drawn from the second tower and usually runs about 60 per cent.  $\text{HNO}_3$ . 85 per cent. of the nitrogen of ammonia is converted into nitric acid. The nitric acid obtained is treated with ammonia to give ammonium nitrate. This solution is evaporated and dry ammonium nitrate is obtained.

Judge William H. Hunt, in the Butte federal court, April 26th, dismissed the bill in the action brought in the name of Fred. J. Bliss against the Anaconda and Washoe companies to close down the great smelting plant at Anaconda. Each side will pay its own costs, which are said to aggregate about half a million dollars. This order disposes of the famous "smoke" case begun three years ago by the farmers of the Deer Lodge Valley, who claimed they were being damaged by the fumes from the smelter, and who demanded an injunction closing down the work. The main issue was decided last February when Judge Hunt denied the injunction.

In supplementary proceedings, however, the court heard testimony on the part of the smelter company with a view to ascertaining whether everything was being done to eliminate the arsenic from the smelter smoke at Anaconda. Counsel for Bliss had an opportunity on April 19th to combat the evidence introduced on Feb. 15th, but offered no proof. Instead, they submitted an offer to arbitrate, provided that the defendants would be bound to include all the farmers who had contributed to the prosecution in the case, Bliss alone being the complainant of record.

Judge Hunt declared the offer to arbitrate wholly irrelevant. He held that the decision in the Highland Boy case in Utah, on which Bliss relied, was not applicable, and that the defendants were doing all that science could suggest to abate the smoke trouble. The decision is entirely in favor of the defendants, and the only relief to which the complainant is entitled, if entitled to any, is through action for damages or other proper suit.

*European Zinc Trust.*—According to reports appearing in some of the leading European industrial organs, Consul H. Albert Johnson, of Liege, says that an international agreement has been concluded between a number of prominent zinc works, resulting in the organization of what is designated as the "Syndicat des Usines a Zinc."

The limit of production of each one of the firms having been established, the principal clause of the agreement stipulates that should the current price of zinc fall below £19 (\$92.46), or should the accumulation of stocks show an unusual increase, the limit of production will be proportionately reduced. If, on the other hand, the current price of zinc continues to be above £19, this limit of production may be increased as arranged in virtue of an agreement between delegates from each group of concerns making up the trust, which includes German, Belgian, and English concerns, although it seems that agreements have not yet been concluded with the last mentioned.

It is asserted that the works of Heritiers Georg von Giesche, a concern that is said to produce one-fifth of the total output of zinc in Silesia, is not included in the new combine, whose main object is to regulate production, but competition from this quarter apparently is not feared. Steps have been taken to induce the five most important zinc works in England to join the trust; as to the zinc works of the United States, it is believed that anything like extensive exports of zinc from that part of the world are too far in the future to meet serious consideration at the present time.



It is rumored that one of the earliest results of the formation of this trust will be an increase in the present price of zinc, such an advance having already been definitely decided upon.

Referring to his former report regarding the formation of a European zinc trust, Consul H. Albert Johnson writes from Liege that it now appears from more recent reports that the organization of a syndicate composed mainly of German zinc manufacturers was concluded the latter part of February under the form of a limited liability company. The capital is said to be \$509,400, and the management is placed in the hands of Director-General Lob, of the Hohenlohe Works. The sale of zinc will be effected through the agency of Beer, Sonheimer & Co., the Metal Gesellschaft, of Frankfurt-on-the-Main, and by Aron Hirsch, of Halberstadt. It is stated that this new combine includes a large number of Belgian, French, Dutch, and Austrian works.—*U. S. Consular Report*, March 23, 1909.

*Growth of the German Potash Industry.*—Consul-General Richard Guenther writes from Frankfort that the potash production of Germany at present represents about 750,000,000 marks (\$178,500,000) of capital and employs 28,000 miners and work people. The yearly sales of potash are \$24,000,000, the trade being controlled by a trust. In 1908 this combine added 9 new mining companies to its membership, while this year 13 new works will go into operation, and also controlled by the potash trust. Germany alone consumes for soil fertilization nearly half the potash salts annually produced. There has been much speculation among German investors in shares of potash-production companies.—*U. S. Consular Report*, March 25, 1909.

*Radium in Cornwall.*—A report from Consul Joseph G. Stephens, of Plymouth, states that considerable interest is manifested throughout England in the announcement of the existence in large quantities in Cornwall of pitchblende, from which radium is produced after the extraction of uranium. The consul describes the sources of the valuable article and probability of securing further supplies:

Pitchblende ore is largely found in the Erzgebirge, in Bohemia, and in Cornwall, and so valuable are such finds of pitchblende that the Austrian Government, exercising a legitimate right, has prohibited further export from that country. The world, therefore, must be searched for new sources of supply, and Cornwall appears likely to furnish the deficiency. Portugal has also been mentioned as a likely field.

The process of extraction is extremely tedious and expensive, tons of pitchblende (oxide of uranium) yielding only the minutest quantities of the precious mineral. However, the fact that a pound of radium is theoretically worth over \$36,000,000 is an inducement to produce it, and science makes its production a necessity. [The total world production of radium has not yet equaled one gram.—B. of M.] It is known that radium is widely disseminated throughout the earth's crust, but the occurrence of radioactive minerals in a formation sufficiently permanent to render mining at any depth a paying proposition is still very rare. Radium to be appreciably cheaper must be derived from uranium minerals, when obtainable in large quantities

from surface deposits at little expense. Even where so found the cost of its separation from other minerals and waste is enormous. Great interest therefore naturally attaches to the refuse heaps at the mines of Cornwall.

The particular mine in which the existence of radium has been found is in the St. Austell district, but St. Ives is sharing the public attention by reason of the investigations which have been made for some time past. At present the Cornish mines are fitted with machinery for dealing with the residuum as ore, but not for dressing it, and persons are engaged to crush it into powder. Their market thus far has been for uranium oxide, which has been sent to Germany, where it is chemically treated to produce sodium uranite for the colored glass and porcelain trade, and nitrate of uranium for the photographic trade. Hitherto the tailings from the mines have been regarded as useless, but it has recently been discovered that the residuum is the mother of radium. The Cornish Company contemplates the establishment of works for the treatment of pitchblende at Trenwith mine, near St. Ives, and in the St. Agnes district. It is asserted that the works will afford employment to some 400 workmen, and will constitute a new industry of considerable benefit to the country.—*U. S. Consular Report*, March 26, 1909.

*Nitrate Combine Broken.*—Consul Rea Hanna, of Iquique, advises that at a meeting held on March 29, 1909, it was definitely decided to discontinue the combination among the Chilean producers of nitrate of soda, for the control of the production and exportation of that article. The consul writes:

Forty-one producers, representing 77 "officinas" (nitrate works), accepted quotas of production aggregating 40,480,000 Spanish quintals of 101.61 pounds each, and 46 producers, representing 77 oficinas, refused to accept the quotas assigned to them. Those refusing to accept demanded quotas aggregating 10,800,000 quintals more than was proposed to them.

As a consequence the nitrate combination ceased to exist on March 31, 1909. In the future, or until a new combination be formed, there will be unlimited production and exportation by the several oficinas. This condition is desired by many of the producers who possess rich deposits and whose cost of production is low, and the general opinion is that the nitrate market will suffer at least only a temporary fluctuation as a result. The "Association Salitrera de Propaganda," however, still continues as an advertising medium, and as a statistical department for the industry.—*U. S. Consular Report*, May 10, 1909.

#### CORRECTION.

On page 300 left-hand column, about the 24th and 25th lines for

"Ground October, 1907" insert "Ground October, 1906."

"Analyzed October, 1907" insert "Analyzed October, 1907."