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### ALBERT ERNEST LEACH.

April 7, 1864.

August 22, 1910.

Instances are not uncommon of men who have become famous in youth and died in early manhood, or who did not find their calling until late in life and achieved fame in old age, but it is rare indeed that men begin their life work well along in manhood and end a distinguished career in middle life.

Albert Ernest Leach belongs in the last-named class. Although he did not join the chemical fraternity until after he had become an expert in another calling, he soon won for himself a foremost place among food chemists and in less than ten years accomplished work that well might have been distributed through a long life.

Mr. Leach was born in Boston, April 7, 1864, and died in Denver, August 22, 1910.

He was graduated from the Newton (Mass.) High School in 1882, and from the Massachusetts Institute of Technology, with the degree of S.B., in 1886, after completing the course in mechanical engineering. Although specializing in mathematical and mechanical studies, he devoted considerable time to chemistry, thus laying the foundation for his subsequent career.

In 1887 he entered a patent law office, where after five years' service he acquired the reputation of an expert in mechanical electrical and chemical cases. Although these years in the law office were lost so far as laboratory experience was concerned, they were of inestimable value in developing the remarkable ingenuity afterwards displayed in devising analytical methods and in acquiring the art of clearness and exactness of expression, of which he became a master.

In 1892 he gave up patent law and entered the laboratory of the Massachusetts State Board of Health as assistant analyst. Feeling the need of further chemical training, he found time during the years 1894, 1895 and 1896, without giving up his position with the Board of Health, to complete two years of postgraduate study at his Alma Mater, the subjects covered being industrial, theoretical and sanitary chemistry and quantitative analysis. This work was not done with a view of attaining an advanced degree, as he took no examination whatever during his graduate study.

His position with the Massachusetts State Board of Health was exceptionally fortunate. Not only was this institution the pioneer in the country in food inspection, but it numbered among its officers such men as Dr. Abbott, Secretary of the Board, Dr. Brown, the well-known sanitary chemist, and Dr. Worcester, analyst in charge of the Food and Drug Laboratory, all men of high attainment and noble ideals, who had brought the Board up to a high degree of efficiency. For these men Mr. Leach acquired not only respect but affection, as evidenced by the dedication of his "Food Inspection and Analysis" to Dr. Worcester and the acknowledgments in the preface.

After the untimely death of Dr. Worcester from the very disease that later claimed Mr. Leach as a victim, the Board hesitated in its selection as a successor. Since up to that time the position of analyst had been filled by a doctor of medicine, he who was destined to fill soon a unique position among food chemists was all but rejected in the vain search for a medical man with training quite foreign to his profession. Mr. Leach was, however, appointed in 1899.

The amount of work accomplished by Mr. Leach during the few years he served as analyst of the Massachusetts State Board of Health is indeed extraordinary. In addition to the official inspection, involving the examination of some ten thousand samples annually, and frequent appearance in court,

he found time for devising important analytical processes, the publication of papers on the composition and analysis of foods in scientific journals and official reports, the instruction of pupils in the analysis and microscopy of food and drugs, the delivery of courses of lectures on food inspection and analysis at the Massachusetts Institute of Technology and the Brooklyn Polytechnic Institute, as well as popular talks before various clubs and organizations, the study of methods as a referee of the Association of Official Agricultural Chemists, and finally the Herculean task of writing a comprehensive work of over eight hundred pages—"Food Inspection and Analysis."

He joined the Bureau of Chemistry, U. S. Department of Agriculture, in 1907, as Chief of the Denver Food and Drug Laboratory, although his health was such that for some time he was not able to enter fully on the duties of the office.

At the time the American Chemical Society organized This Journal, Mr. Leach was chosen a member of the Board of Editors. He served on the board until his death, reading all papers connected with food chemistry and analysis.

At the time when his recovery seemed an accomplished fact, he was stricken by a new form of the disease that previously had laid him low, and died after a wasting illness of some months, retaining his mental powers until the very end.

He is survived by his wife, nee Martha Hughes Thompson, to whom he was married on Sept 2, 1890, three daughters and one son, the latter a Sophomore in the University of Colorado, specializing in chemistry.

Mr. Leach's methods of analysis were at once ingenious and timely. His refractometric method of detecting wood alcohol in liquor was devised at the time when this adulterant was causing havoc among the consumers of cheap liquors and at once superseded the laborious and unsatisfactory methods then in vogue. He also applied the different forms of refractometer to various other new lines of investigation not thought of by the originators of the apparatus, thus greatly enhancing the value of the instrument in the food laboratory.

His procedure in milk analysis was not only rapid, permitting the inspection of large numbers of samples daily, but complete in details for the detection of watering, skimming, preserving and coloring of the product. The fraud of coloring blue milk yellow might long have escaped detection had it not been for his analytical scheme. He was a pioneer in the examination of condensed milk and the methods proposed by him are to-day widely used for purposes of inspection. His work on vinegar was also path-breaking and of special value in diagnosing skilfully concocted imitations of cider vinegar.

Among the other products which he specially studied were molasses, glucose, corn oil and various other oils and fats, mustard and other spices and drugs.

As a man, Mr. Leach was at once strong and gentle; honorable in all his dealings with members of the profession and the outside world, sympathetic with all in trouble, a true friend and conscientious adviser. A follower of Swedenborg, his work and life were a part of his religion.

Following is a partial list of his publications:

"Character and Extent of Food Adulteration in Massachusetts." Read before the Society of Arts, Boston, and published in the *Technology Quarterly*, Vol. 13, p. 22.

"Foreign Coloring Matter in Milk." *Jour. Am. Chem. Soc.*, Vol. 22, p. 207.

"Estimation of Fat in Condensed Milk." *Loc. cit.*, supra, p. 589.

"Microscopical Examination of Foods for Adulteration." Illustrated with 15 plates of photomicrographs. An. Rep. Mass. Board of Health, 1900, p. 679.

"Report on Arsenic and Other Poisons in Manufactured Goods." *Loc. cit.*, supra, p. 695.

"Systematic Inspection of Milk for Preservatives." *The Analyst* (London), Vol. 26, p. 289.

"Foods: Methods of Inspection and Analysis." Signed article in *Reference Handbook of the Medical Sciences*, Vol. 3, p. 180, et seq.

"Provisional Methods for the Analysis of Saccharine Products of the Assn. of Official Agrl. Chemists." U. S. Dept. of Agric., Bur. of Chem., *Bull.* 65, p. 43.

"The Determination of Commercial Glucose in Molasses, Syrups and Honey." *Jour. Am. Chem. Soc.*, Vol. 25, p. 982.

"The Determination of Commercial Glucose in Some Saccharine Products." Proc. of 20th An. Convention of the A. O. A. C., U. S. Dept. of Agric., Bur. of Chem., *Bull.* 81, p. 73.

"Reports as Associate Referee on Dairy Products of the A. O. A. C." *Loc. cit.*, supra, p. 25. Also Bur. of Chem., *Bull.* 90, p. 75, and *Bull.* 99, p. 78.

"Composition and Adulteration of Ground Mustard." *Jour. Am. Chem. Soc.*, Vol. 26, p. 1203.

"The Composition of Turmeric." *Loc. cit.*, supra, p. 1210.

"Cider Vinegar and Suggested Standards for Purity." (With H. C. Lythgoe, assistant analyst.) *Loc. cit.*, supra, p. 375.

"A Comparative Refractometer Scale for Use with Oils and Fats." (With H. C. Lythgoe.) *Loc. cit.*, supra, p. 1193.

"The Detection of Watered Milk." (With H. C. Lythgoe.) *Loc. cit.*, supra, p. 1195.

"Some Phases of Food Adulteration and Inspection." Read before the Dept. of Health of the Am. Social Science Assn. of Boston, 1904. Published in *Jour. Am. Soc. Sci. Assn.*, No. 42, p. 110.

"Food Inspection and State Control." Read before the Vermont School of Health Officers at Burlington, 1904. Pub. in the Sept. *Bull. VI. State Board of Health.*

"The Adulteration of Drugs." Address before the International Pure Food Congress at St. Louis, 1904. Pub. in the *Proc. of the 8th An. Convention of State Dairy and Food Depts.*

"The Detection and Determination of Ethyl and Methyl Alcohols in Mixtures by the Immersion Refractometer." (With H. C. Lythgoe.) *Jour. Am. Chem. Soc.*, Vol. 27, p. 964.

Reports of the Analyst of the Mass. State Board of Health, 1899-1906, inclusive.

"Food Inspection and Analysis." Published by John Wiley & Sons, New York and London, 1st edition, 1904, pp. 786. 2d edition, 1909, pp. 954.

A. L. WINTON.

## EDITORIALS.

## CAST COPPER.

THOSE who were fortunate enough to attend the Chicago Meeting of the American Electrochemical Society had the privilege of inspecting samples of copper castings made by the process worked out by Dr. Weintraub in the Research Laboratory of the General Electric Company at West Lynn, Mass. Dr. Weintraub's paper (which was read by Mr. Marvin, of Chicago, Dr. Weintraub not being present at the meeting) appears in this number. The castings exhibited at the meeting appeared to be as perfect as the most critical could desire, homogeneous, dense and free from flaws—of very different appearance from castings made in the old ways. Dr. Weintraub obtains his results by the use of boron, the preparation of which in the pure state has also been worked out by himself during recent years. Copper is one of the most beautiful of metals, and in the form of castings made by this process its attractiveness appears to be enhanced visually, as well as by the knowledge of its peculiar physical properties, including a greatly increased conductivity. The new uses to which copper cast by this process can be put are probably very great. But immediately its greatest field of usefulness will be in applying uniform cast metal where formerly joints, brazed and otherwise, were the rule. In this field alone the use of copper will naturally increase and also naturally it will be more valuable insofar as it is better adapted to a particular purpose. This invention will probably prove one of the greatest contributions to the arts in recent times.

## PHOTOGRAPHY.

THE increase in the use of photography and the improvements in the practice are astonishing. Without dwelling upon the development of the portable camera, adapted to a great variety of uses including ultra-rapid photography and for which no better name has been devised than the "hand camera," it is only necessary to observe that no modern family is complete without one of the numerous forms. Neither do we care to consider at length the domain lying between ordinary photography and art—art photography, if you please—where the tendency is toward softness of outline, contrasted effects, separation into planes and wherein the lenses are purposely left more or less uncorrected to produce such effects. What the technical man and chemist want is photographic detail and usually they can not get too much of it. A long series of papers might be written on the modern technical and scientific uses of photography, which would lead from simple detail photography of apparatus, machinery, plant and equipment to the photomicrography of tissues, mineral sections, metal surfaces and the applications of the wet plate and

Lumière color plate. Photo-engraving is a branch by itself full of the most elaborate and intricate details and also full of possibilities in the line of more perfect development. The moral of all this is that the chemist and technologist will find it to their advantage to be photographers. Some institutions of learning include photography among their courses. But the curricula are already generally full and until some general agreement as to what is most advisable in the curriculum for the education of a chemical engineer, we do not advise adding photography to the courses of study. What we do advise, is, if it is not advantageous or expedient to take a course in photography, that each man take it up for himself. It will be found one of the most fascinating and profitable avocations which could possibly be undertaken. We recommend further that the study be not taken up piecemeal, haphazard, irregularly, or in a dilettante spirit. Let the amateur cover the whole ground of cameras, lenses, diaphragms, shutters, exposures, wet and dry plates, developers and prints! There be photographs without pith or merit, smudgy, cloudy, distorted and out of focus which could well be spared by many an editor and printer. It were well to eliminate these altogether. The technical or scientific photograph, which is clean cut, detailed, properly lighted and properly developed and printed, is a joy to the engraver, printer and beholder.

## ORIGINAL PAPERS.

## THE ACTION OF SUNLIGHT AND AIR UPON SOME LUBRICATING OILS.

By C. E. WATERS.

Received October 1, 1910.

In 1885, Schaal<sup>1</sup> patented a process for the manufacture of soaps by the action of air upon mineral oils in the presence of alkaline substances. This was based on an earlier observation of Engler's.

In 1891, Zaloziecki<sup>2</sup> studied the oxidation of petroleum by means of air. By blowing cold air through the oil the oxidation was slight, but was greatly increased in the presence of sodium hydroxide. The acidity shown by methyl orange, as opposed to phenolphthalein, he believed to be due to the oxidation of mercaptans and organic sulphides, and not to traces of the acid used in the refining.

Schestopal<sup>3</sup> found that illuminating oils absorbed several volumes of oxygen at room temperature, when enclosed in sealed tubes laid horizontally. He pointed out the bearing this had upon the treatment of kerosene in agitators.

<sup>1</sup> D. R. P. 32,705. *Chem.-Ztg.*, **9**, 1520 (1885).<sup>2</sup> *Z. angew. Chem.*, **1891**, 416-9.<sup>3</sup> *Chem.-Ztg.*, **15**, 352-4 (1895).

Holde<sup>1</sup> exposed mineral and vegetable oils to the air for a long time, studying changes in viscosity, specific gravity, etc. He found that the changes were much less for mineral than for vegetable oils.

Haack<sup>2</sup> believed that the oxidation observed in refining oils was due to the combined action of concentrated sulphuric acid and air. This idea was upheld by Kern.<sup>3</sup> Other papers bearing upon the question of oxidation by atmospheric oxygen were published.<sup>4</sup>

Zaloziecki<sup>5</sup> studied the influence of light and air upon the acid treatment of kerosene. In some of his experiments he used colored glass. The general conclusion reached by him was that light and air should be excluded. Ostrejko<sup>6</sup> confirmed this work as to the effect on the color of refined petroleum, and also determined the increase in acidity due to oxidation. Wischin<sup>7</sup> thought that the darkening in color of kerosene was perhaps due to a molecular rearrangement of dissolved asphalt under the influence of actinic rays.

Hirsch<sup>8</sup> passed air through mineral oils in the presence of soda lye and, even after 12 hours at 70°, could detect no change in the alkali, concluding from this that there was no oxidation. Aisinmann<sup>9</sup> found that the absorption of oxygen was very slight. It was also stated by Künkler and Schwedholm<sup>10</sup> that there was no increase in weight when mixtures of mineral oil with bone oil, olive oil, etc., were rubbed up with sand or pumice and exposed to the air. The resinification noticed was believed to be due solely to polymerization.

There can be no doubt, however, that oxidation really takes place. Charitschkow,<sup>11</sup> who states that the resinous products formed in petroleum on long standing under the combined influence of light, air and moisture are nearly identical with those of Schaal, obtained sirup-like acids<sup>12</sup> by the action of air in the presence of alkalis. These acids, which are nearly insoluble in benzine and petroleum ether, he calls "polynaphthene" or "asphaltogenic" acids, the latter name being self-explanatory. Meyerheim,<sup>13</sup> in this connection, thinks the increase in asphalt with lapse of time is due to polymerization or to a change from the original colloidal state to the insoluble condition. His experiments were carried out in closed bottles, two-thirds filled with oil. The changes in asphalt content in two oils, kept in the dark, in diffused light and

under the influence of radium emanations, were determined. After 8 months no difference due to the action of radium could be detected. In diffused light one oil showed a higher asphalt content than a control kept in the dark; the other oil showed a somewhat higher content of asphalt in diffused light.

Molinari and Fenaroli<sup>1</sup> obtained unstable ozonides from petroleum hydrocarbons. They were precipitated from their solutions in benzene, ether or chloroform by means of petroleum ether. They decomposed at 20°, changing at 35–50° into a soft, red substance which was converted into a resinous mass at 105°.

An interesting observation bearing on the work described in this paper was made by Ackerman.<sup>1</sup> A sample of naphtha from Russian Turkestan was entirely thickened on exposure to the sun's heat for a whole year in an open vessel. Fresh samples of naphtha yielded 76.68 per cent. of distillate between 20° and 310°, while the thickened oil yielded only 15.67 per cent. of distillate.

In a paper entitled "Studies on the Carbenes," Mackenzie<sup>2</sup> shows that the action of light upon solutions of asphalt in carbon tetrachloride greatly increases the percentages of "carbenes" or insoluble bitumens. This is shown to be due to the formation of hydrochloric acid, which precipitates the carbenes.

The present writer was at the time determining asphaltic material in certain lubricating oils, and the above-mentioned paper, though on a somewhat remotely related subject, suggested the experiments herein described.

Three samples of oil were used. B and C were straight mineral oils; B was moderately dark in color, while C was much lighter colored and less viscous. D resembled B in color and viscosity, but contained approximately 0.5 per cent. of fatty oil.

Duplicate samples of 20 grams each were weighed into 150 cc. Erlenmeyer flasks, loosely covered with caps to exclude dust and rain, and were exposed outside a window facing south for 23 days. Within 2 or 3 days the samples began to show a slight cloudiness, and after a week or ten days the insoluble matter formed a cake on the bottom. After 23 days the contents of each flask were diluted with 50 cc. of ligroin and after 36 hours the insoluble matter was filtered off in Gooch crucibles, washed with ligroin, dried at 100° and weighed. The following percentages of insoluble matter were found:

Sample.	B.	B.	C.	C.	D.	D.
Insoluble per cent. ....	0.77	1.03	2.42	2.86	1.40	1.91

The discrepancies between duplicate samples are believed to be due to their having received unequal amounts of sunlight, as it was necessary to place the flasks in a crystallizing dish to prevent their blowing

<sup>1</sup> Ber., 41, 3704 (1908).

<sup>2</sup> This Journal, 2, 124–7 (1910).

<sup>1</sup> *Mith. kgl. techn. Versuchsanst.*, 10, 85–90 (1892); *J. Soc. Chem. Ind.*, 11, 619 (1892).

<sup>2</sup> *Chem.-Ztg.*, 16, 694 (1892).

<sup>3</sup> *Ibid.*, 17, 706 (1893).

<sup>4</sup> Zaloziecki, *Chem.-Ztg.*, 16, 591 (1892). Holde, *Ibid.*, 18, 794, 1737 1835 (1894). Hirsch, *Ibid.*, 18, 1824.

<sup>5</sup> *Ibid.*, 19, 875–7, 956–8 (1895).

<sup>6</sup> *J. Soc. Chem. Ind.*, 15, 345 (1896).

<sup>7</sup> *Chem. Rev. Fett.-Harz.-Ind.*, 1895, Nos. 26 and 27; *J. Soc. Chem. Ind.*, 14, 1039 (1895).

<sup>8</sup> *Chem.-Ztg.*, 19, 41 (1895).

<sup>9</sup> *Dingl. pol. J.*, 294, 658 (1894); *J. Soc. Chem. Ind.*, 14, 282 (1895).

<sup>10</sup> *Seifensiederztg.*, 35, 341; *Chem.-Zentr.*, 1908, I, 1859.

<sup>11</sup> *Z. angew. Chem.*, 21, 1155 (1908).

<sup>12</sup> *Chem.-Ztg.*, 33, 1165 (1909).

<sup>13</sup> *Ibid.*, 34, 454 (1910).

away, and late in the afternoon those outside were in the sun longer than the others.

Another test was then started with approximately 10 gram samples, the flasks being weighed at intervals. After each weighing the flasks which showed the least gain were placed so as to receive the maximum of sunlight. For this reason the last figures given in the next table are more concordant than they would otherwise have been.

Sample.	B.	B.	C.	C.	D.	D.
Weight of oil.....	10.002	10.012	10.025	10.023	10.023	10.003
Total gain after:						
31 days (gram).....	0.176	0.152	0.187	0.183	0.154	0.139
46 days (gram).....	0.192	0.174	0.215	0.207	0.176	0.159
80 days (gram).....	0.233	0.223	0.274	0.278	0.221	0.201
110 days (gram).....	0.277	0.263	0.327	0.340	0.274	0.243
132 days (gram).....	0.290	0.289	0.358	0.369	0.297	0.268

That the increase in weight is somewhat influenced by the temperature and is not entirely due to rays of short wave length, seems clear from the results obtained when two lots of oil B were exposed in very shallow dishes made by cutting off the bottoms of two beakers of the same diameter. One was covered with a thick watch-glass. The other was uncovered and so shallow that hardly any light passed through glass before falling on the oil.

Dish.	Uncovered.	Covered.
Weight of oil.....	10.001	10.001
Gain after 6 hrs., bright sun (gram).....	0.0354	0.0354
Further gain after 6 hrs., bright sun.....	0.0183	0.0198
Further gain after 6 hrs., bright sun.....	0.0154	0.0179
Further gain after 6 hrs., partly cloudy.....	0.0085	0.0094
Further gain after 4 hrs., partly cloudy.....	0.0050	0.0061
Further gain after 6 hrs., dull $\frac{1}{2}$ of time.....	0.0059	0.0068
Further gain after 6 hrs., dull $\frac{1}{3}$ of time.....	0.0063	0.0071
Further gain after 6 hrs., dull $\frac{1}{2}$ of time.....	0.0049	0.0065
Total gain (gram).....	0.0997	0.1090

In the cool, windy weather then prevailing, the oil in the covered dish must have been warmer than that in the uncovered one. On the first five days the dishes rested on white paper. This was replaced by black paper on the last three days, but only a slight change in the results was noticed. The increasing opacity of the oil due to the formation of insoluble matter and the increasing saturation with oxygen would both tend to mask any effect due to a slight rise in temperature. Traces of precipitate were noticed at the end of the first six hours.

The observed increase in the weight does not truly represent the amount of oxygen involved, for carbon dioxide, water and volatile organic matter are lost. Twenty-five grams of oil B were exposed in a flask having a glass stopper with sealed-in tubes so that a slow current of purified air could be passed over the surface of the oil, carrying the carbon dioxide into baryta water. In seven days, only three of which were sunny, 0.0249 gram of carbon dioxide was found. On exposing the same sample for seven days longer, about four and a half of which were sunny, there was the further formation of 0.0223 gram of carbon dioxide.

In a final experiment, the oil was exposed in an

Erlenmeyer flask with glass stopper and delivery tubes. The water formed was carried by a slow current of dry air, passing over the surface of the oil, into a calcium chloride tube and finally weighed. It was noticed that the calcium chloride where the air entered was stained yellow, and that there was a whitish coating on the inside of the tube leading in through the window from the flask. Evidently some organic matter was given off. The carbon dioxide was absorbed in baryta water as before, finally set free by dilute hydrochloric acid, absorbed in a potash-bulb and weighed. The change in weight of the oil was also determined as well as the amount of insoluble oxidation product.

The following data were obtained after 22 days constant exposure on the window-sill:

	Per cent.
Gain in weight of oil.....	1.83
Water formed (corr. for H <sub>2</sub> O in oil).....	0.89
Carbon dioxide.....	0.37
Total.....	3.09
Oxidation product.....	1.47

The figures for water and gain in weight of the oil are slightly in error on account of the volatile oily matter mentioned. However, the total of 3.09 per cent. agrees closely with the figure 2.89 per cent., obtained by adding the gain in weight of the oil to the oxygen in the water and carbon dioxide found.

The "insoluble" oxidation product dissolves to only a slight extent in ligroin. It is readily filtered on a Gooch crucible, and when washed with ligroin and dried is a light brown powder. It dissolves in strong alcohol and more easily in dilute alcoholic potash and in ether. Attempts to crystallize it and to obtain a crystalline potassium salt were fruitless.

The amount found evidently accounts for only a small part of the 1.83 per cent. of oxygen taken up by the oil. The following analyses show that a large part of it remains in solution in the oil, or that most of the oil is too slightly oxidized to form the insoluble product.

Found.	C.		H.		O.	
Original oil.....	86.40	86.34	13.83	13.63	...	...
Oxidation product....	72.87	72.58	7.83	7.81	19.30	19.61
Oil filtered off.....	84.55	84.44	13.45	13.38	2.00	2.18

To determine changes in the acidity of the oils, 20-gram samples of each were exposed, in duplicate, for 23 days. After dilution with benzene, they were titrated with N/10 alkali, the results being calculated as percentages of oleic acid.

Sample.	B.	B.	C.	C.	D.	D.
Acidity of original oil.....	0.08	0.08	0.03	0.03	0.07	0.07
Acidity after exposure.....	3.66	3.75	3.29	3.30	3.00	..

Attempts to obtain concordant figures for the permanganate value of the oil were fruitless, as the permanganate was decomposed catalytically, no true end-point being obtained. The oils were shaken with dilute sulphuric acid and heated on the steam-bath,

standardized permanganate being added from time to time. Apparently the temperature was too high, for it has been shown quite recently by Sarkar and Dutta<sup>1</sup> that at or above 81°, permanganate is decomposed by manganous sulphate, with formation of manganese peroxide, even in the presence of considerable sulphuric acid.

BUREAU OF STANDARDS,  
September 29, 1910.

## COMPARATIVE ANALYSES OF WATER FROM GREAT SALT LAKE.

By W. C. EBAUGH AND WALLACE MACFARLANE.

Received September 5, 1910.

From about 1900 until 1904 fears were expressed that the Great Salt Lake was doomed to extinction, and that it would be a matter of only a few years until its site would become a salt desert. The recession of the shore line and sinking of the lake level continued until the autumn of 1903. Since that time there has been a rise in the level of the lake, and during the year just ending new fears have arisen—fears that large engineering works like the Lucin cut-off of the Southern Pacific and the roadbed of the Western Pacific railroad would have to be abandoned. A succession of years with abnormally high rainfall is responsible for the condition now existing.

TABLE I.

	Date of collection.	Specific gravity.	Total solids.		Authority.
			Per cent. by weight.	Grams per liter.	
	1850	1.170	22.282	260.69	L. D. Gale
Summer,	1869	1.111	14.9934	166.57	O. D. Allen
Aug.,	1873	1.102	13.42	147.88	H. Bassett
Dec.,	1885	1.1225	16.7162	187.65	J. E. Talmage
Feb.,	1888	1.1261			J. E. Talmage
June,	1889	1.148			J. E. Talmage
Aug.,	1889	1.1569	19.5576	226.263	J. E. Talmage
Aug.,	1892	1.156	20.51	238.12	E. Waller
Sept.,	1892	1.1679	21.47	250.75	J. E. Talmage
	1893		20.05		J. T. Kingsbury
Dec.,	1894	1.1538	21.16	244.144	J. E. Talmage
May,	1895	1.1583	21.39	247.760	J. E. Talmage
June,	1900	1.1576	20.90	241.98	H. N. McCoy and Thos. Hadley
July,	1900	1.1711	22.89	268.09	H. W. Sheley
Aug.,	1900	1.1805	23.36	275.765	H. W. Sheley
Oct.,	1900	1.1860	24.03	285.020	H. W. Sheley
Sept.,	1901	1.1979	25.221	302.122	L. J. Seckles
Oct.,	1903	1.2206	27.72	338.36	Wm. Blum
June,	1904	1.1905	25.196	299.96	J. E. Talmage
Nov.,	1904	1.2120	26.71	323.71	Wm. Blum
Oct.,	1907	1.1810	22.92	270.685	W. C. Ebaugh and Kenneth Williams
Oct.,	1909	1.1561	20.887	242.25	Wallace MacFarlane
Feb.,	1910	1.1331	17.681	200.32	Wallace MacFarlane

NOTE.—The above values are taken in part from "The Great Salt Lake," by J. E. Talmage, and all the analyses during recent years have been made in the laboratories of the University of Utah.

An inspection of the results of analyses of the lake water will be of interest. In Table I are shown the specific gravity and total solids obtained by investigators at various times during the last forty or more years, and in Table II more complete results of the latest analyses are recorded. In this connection it should be remembered that the annual variation

of the lake water shows a minimum of total solids in the spring, following the winter and spring precipitation, and a maximum in the autumn.

TABLE II.

Sample collected.....	Oct.,	Nov.,	Oct.,	Oct.,	Feb.,
	1903	1904	1907	1909	1910
Specific gravity.....	1.2206	1.2120	1.1810	1.1561	1.1331
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Total solids.....	27.72	26.71	22.92	20.88	17.68
Constituents.					
Chlorine (Cl).....	15.27	14.54	12.67	10.91	9.48
Sulphate (SO <sub>4</sub> ).....	1.86	1.82	1.53	1.39	1.05
Magnesium (Mg).....	0.155	0.43	0.45	0.447	0.391
Calcium (Ca).....	0.045	0.055	0.04	0.080	0.055
Sodium (Na).....	9.58	8.77	7.58	7.25	5.79
Potassium (K).....	0.73	0.89	0.72	0.76	0.88

UNIV. OF UTAH,  
SALT LAKE CITY.

[CONTRIBUTION FROM THE TEXAS AGRICULTURAL EXPERIMENT STATION.]

## CONSTITUENTS OF CANDELILLA WAX.

By G. S. FRAPS AND J. B. RATHER.

Received September 16, 1910.

The wax is from the Candelilla, or Mexican wax plant, which grows in a number of Mexican states. The sample was secured for us by Dr. H. H. Harrington, Director of the Texas Experiment Station. It is a hard wax, opaque, and almost colorless. A description and constants of this wax have been given by Hare and Bjerregaard.<sup>1</sup> According to an editorial in the *Journal of the Royal Society of Arts*,<sup>2</sup> the following uses have been suggested for it; candles, shoe polish, phonographic records, insulation of electric wires, and as a bees' wax substitute.

*Constants.*—The following constants were determined by A. C. Deiler in the spring of 1909. For the sake of comparison, the same constants as ordinarily secured for bees' wax are given, and also those obtained by Hare and Bjerregaard.<sup>3</sup>

	Candelilla wax.		
	Deiler.	Hare.	Bees' wax.
Specific gravity at 100° C..	0.870	....	...
Specific gravity at 15/15° C. ....	....	0.9825	....
Iodine number.....	14.0	36.8	6-13
Acid number.....	19.0	12.4	19-21
Ester number.....	40.7	....	73-76
Saponification number....	59.7	64.9	....
Melting point.....	66° C.	67-8° C.	....
Unsaponifiable matter, per cent.....	....	91.17	....

Our sample of wax was completely soluble in chloroform and carbon bisulphide. Ether dissolved 0.12 gram in 100 cc. and alcohol 0.048 gram, wax and solvent being brought together for fifteen minutes at room temperature.

*Isolation of a Hydrocarbon.*—The wax was powdered and saponified with alcoholic potash and the alcohol evaporated off. It was then transferred to a Kutscher and Steudel extraction apparatus with hot water and extracted with ether. After extracting

<sup>1</sup> THIS JOURNAL, 2, 204 (1910).

<sup>2</sup> 57, 644.

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

for 40 hours (5 days) the extract was washed twice with water and evaporated to dryness. About 40 per cent. of the wax was extracted. The extract was dissolved in hot ether, cooled, and allowed to crystallize. The crystals were washed on a filter paper with cold ether; the mother liquid and washings were evaporated to dryness. The crystals were recrystallized from ether three times. Both crystals and residue from mother liquor then had a melting point of  $68^{\circ}$  C. The crystals were recrystallized from chloroform and melted at  $68^{\circ}$  C. The mother liquid, on evaporation to dryness, left a residue which melted at the same temperature. This was taken to indicate the purity of the product.

The substance appears as fine white crystals. It is readily soluble in hot ether, fairly soluble in cold ether, insoluble in cold 95 per cent. alcohol and slightly soluble when heated. It is soluble in cold carbon tetrachloride. It is more easily soluble in chloroform than in ether.

A small portion of the substance was distilled and the solidified material had the same melting point as the original substance.

Tested by Hell's method,<sup>1</sup> by heating with soda lime, the sample did not appear to be an alcohol. This substance appears to be the hydrocarbon hentriacontane, which is found also in bees' wax.

Analyses gave the following results:

	Calculated for $C_{30}H_{62}$ Per cent.	Found.
Carbon.....	85.30	85.48-85.16
Hydrogen.....	14.70	14.10-14.28

*Other Substances.*—The mother liquors from the above hydrocarbon were combined, recrystallized from ether and then heated with 95 per cent. alcohol, filtered, allowed to cool, and filtered again. The alcohol was evaporated off and the resulting solid dissolved in ether. On evaporation no crystals separated out. The product is a transparent, yellowish, resinous, solid—brittle when dry and melting at  $55^{\circ}$  C. It is readily soluble in cold ether and alcohol. It was evidently not completely pure. This substance is not a hydrocarbon, but contains oxygen. Tested by Hell's method, it gave practically no gas (0.12 per cent.), and we believe this gas to be hydrocarbon produced by the cracking of the substance, and not hydrogen. This substance, therefore, does not appear to be of alcoholic nature.

Analysis gives the following results:

	Per cent.
Carbon.....	83.85
Hydrogen.....	12.33

After extracting the original material with ether for 40 hours it was salted out and extracted with boiling ether. About 30 per cent. of the original substance was secured. The product was recrystal-

lized from ether four times. This product was heated with alcohol and filtered hot. The filtrate was evaporated to dryness and the product recrystallized twice from alcohol and three times from ether.

The resulting solid was a shining, white crystalline substance, very similar to the hydrocarbon first mentioned, but more easily soluble in alcohol. It is slightly soluble in cold ether, more soluble in hot ether. It melts at  $85^{\circ}$  and is probably a hydrocarbon.

From the mother liquor we secured further quantities of the hydrocarbon melting at  $68^{\circ}$  C. Other substances are also present in this wax, and it is our intention to investigate it further, but we are at present unable to do so.

#### SUMMARY.

Candelilla contains a hydrocarbon hentriacontane melting at  $68^{\circ}$ , and probably one melting at  $85^{\circ}$ . It also contains other substances.

### AN EXTRACTION APPARATUS.

By SAMUEL M. BAIN.

Received August 25, 1910.

In connection with cotton breeding experiments for the U. S. Department of Agriculture, the writer has for several years been studying the oil content of the cotton seed as an hereditary factor, and has developed an outfit for extraction with ether which has after long use proved very efficient.

Fig. 1 shows a general view of the apparatus resting on the small table support. The heating device is essentially that described by Hopkins.<sup>1</sup> It consists of a wooden base provided with connections for five 32 c. p. incandescent globes, with a double-walled

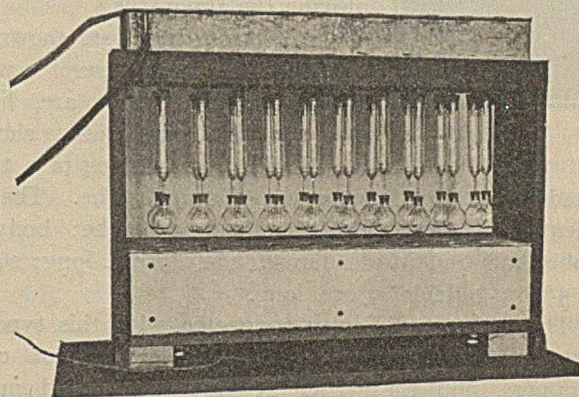


Fig. 1.

galvanized iron box, fitted with lid carrying 20 holes serving as rests for 20 extraction flasks. In addition to the asbestos packing between the double vertical walls of this box, as recommended by Hopkins, it was found desirable also to cover the sides with heavy asbestos sheeting, and to apply a thinner asbestos

<sup>1</sup> *Ann. Chem. (Liebig)*, **223**, 269.

<sup>1</sup> *J. Am. Chem. Soc.*, **21**, 645 (1899).

cover with holes corresponding to those in the galvanized iron lid. This heater rests, as shown in the figure, on two blocks  $5.8 \times 10.5 \times 23.5$  cm. By lifting the heater, which moves freely between the ends of the upright stand, and turning these blocks edgewise, the flasks come into position over the holes in the lid.

At the top of the stand is a galvanized iron box  $16 \times 23.3 \times 97.8$  cm., into which are soldered the condenser worms in proper position over the flasks. The worms are made of block-tin tubing (6.5 mm. inside) and make two coils each within the box. Each pair of worms is braced across the top of the box with galvanized iron strips 2.8 cm. wide and projects below 7.5 cm. to a beveled end, as shown in detail in Fig. 2.

The worms are kept cool by a current of water flowing through the box, the inflow being led by a lead pipe to the bottom and at the extreme right of the box, the exit taking place at the left, as shown in Fig. 1. For convenience in moving about the room, the worm tubes should pass through the braces and extend 2 or 3 cm. above the top of the box, and the box should be made 2 or 3 cm. deeper than the dimensions above indicated. This would permit the location of the off-flow pipe at a sufficient distance below the rim of the box to prevent the tipping over of water on moving the apparatus.

The support tubes are shown in detail in Fig. 2, and are of a well-known pattern. They are, however, shown longer than they should be, the illustration being of a type made to fit another pattern of sample tube. The dimensions of these tubes should therefore be: body of tube, 2.8 cm. inside diameter, 15.3 cm. long; stem, 7.5 mm. outside, 9 cm. long.

The greatest difficulty connected with this type of extraction apparatus is the requirement of cork stoppers, and the writer at first experienced much difficulty in this direction. A method of fitting them was, however, finally devised which overcomes nearly all the inconveniences of the cork stopper. In the first place, a large stock of corks of the best obtainable quality should be available from which those best suited to the purpose may be chosen. By plunging for two or three minutes in vigorously boiling water, a cork softens and may with a little care be forced while still hot and wet into a comparatively

small tube or neck. If allowed to remain for a few hours in this tube, then withdrawn two or three millimeters at a time at intervals extending over several days, the cork is molded, so to speak, to fit its tube. It has then a gradual slope from its smaller end fitting the tube, and may be forced further as required. This fitting should be done before the hole is bored for adjusting on the worm tube. After the cork has dried in its tube the hole should be bored a little smaller than the tube it is to fit, and before adjusting to this tube a jet of steam is allowed to pass through the hole and the cork at once forced over the worm tube, the cork meanwhile remaining in position in the glass tube. This is then allowed to dry a few days as in the former case.

In a similar way the corks are fitted to the flasks below, as shown in Fig. 2. After drying, the apparatus is set in operation and ether allowed to play upon the corks for a day or two, so as to remove all their readily soluble constituents. If any leaks occur, as will be indicated by loss of ether or by extracted soluble material accumulated around holes on the exposed surfaces of the corks, defective ones are replaced as before.

In order to facilitate the replacement of parts accidentally broken, it is important to have each flask neck and each part of the extraction tube forming a cork connection made uniform in size and perfectly cylindrical. This means that a cork fitting one connection of a set will fit all the others.

Another difficulty to be overcome in using tightly fitted corks is the occasional cracking of the tubes due to sudden changes in temperature and the non-yielding qualities of the cork. This can be largely overcome by leaving the connections somewhat loose until the corks are wet and softened by the warm, freshly condensed ether. At the close of an experiment it is also well to disconnect the parts before the corks have dried out. To avoid the effect of air currents it is wise to cover the apparatus while in use. When a good quality of glass is used and these precautions are taken, very few tubes are broken.

The most distinctive feature of this apparatus is the combined weighing and extraction tube shown in Fig. 3. It consists of four parts: (1) The upper cap, ground on the outside of the body of the tube;

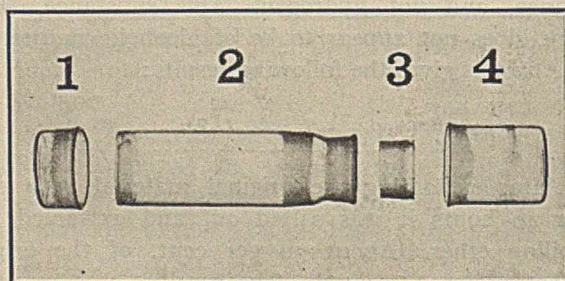


Fig. 3.



(2) the body of the tube 25 x 75 mm. narrowed to a neck, as shown; (3) a hollow cone ground to fit the neck and squared at either end, diameter at small end 18.5 mm., length 12 mm.; (4) lower cap ground to fit on outside of body of tube as shown, outside diameter of flared part 27 mm., length 32 mm. Extreme length of tube with caps, 90 mm.

In use a disk of filter paper 21.5 mm. diameter (C. S. & S. "Blue Label" gave best results) is wet with distilled water, centered on the small end of the cone, and carefully forced into the neck. The whole is then placed in the oven to dry, and after drying, the cone readjusted to take up the shrinkage of the paper, and the caps replaced. The tube is then weighed and the sample poured in and weight of sample obtained by difference. The whole is now put into the oven and dried, in which operation the caps are both removed and the tube allowed to stand on end, the cone serving as base. The writer uses a copper tray 26 x 193 x 226 mm., divided into 12 partitions, which serves to carry twelve tubes with their caps, each set by itself. After drying, the caps are replaced and the dry weight obtained. The caps are now removed and the tube carrying the sample placed in position for extraction, as shown in Fig. 2, resting on a copper wire.

After extraction the filtrate may be weighed directly in the flask below, or the caps may be replaced after driving off the ether and the tube containing the residue weighed.

In Fig. 2 is shown a sealed glass bob containing a bit of iron wire suspended within the extraction tube and attached to the end of the tin tube by means of a silk cord. This leads the condensed ether down near to the sample and prevents spattering. If by accident any of the sample has become attached to the wall of the tube, a magnet applied to that side will pull this bob over and cause the ether to run down the wall over the adhering bit of sample. By attaching a few short, loose strands of silk cord to this bob it may be made to adhere permanently to any desired portion of the tube. There is, however, seldom need of directing the ether current down the wall; for the ether soon accumulates for 30 or 40 mm. above the sample and remains constantly at this level. Of course, the rate of ether distillation may be controlled at will. This combined weighing and extraction tube <sup>1</sup> has been in use in the writer's laboratory for about a year, and has proved entirely satisfactory. The advantages in its favor are:

(1) The sample may be directly weighed, the moisture taken, the extraction made, and all necessary manipulations carried out without transfer of sample.

<sup>1</sup> The writer wishes to acknowledge his indebtedness to Mr. H. H. Hampton, late assistant chemist of the Tennessee Experiment Station, for valuable suggestions in developing this tube.

(2) Owing to the perfect fit of the filter joint, the extract comes through quite clear.

(3) All manipulations are quite simple, and considerable time is saved as compared with the use of the prevailing forms of extraction apparatus.

(4) By properly regulating the rate of ether distillation the sample is kept constantly covered with the solvent.

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## CONTRIBUTION ON JELLY-MAKING.

(SECOND PAPER.)

By N. E. GOLDTHWAITE.

Received August 30, 1910.

The previous report<sup>1</sup> founded upon experiments with fall fruit juices established the following general results: The essential constituents of fruit juices suitable for jellies are pectin and acid. A desirable accessory in jelly-making is cane sugar. Too much sugar causes deterioration of the quality of the jelly. Over-dilution of fruit juices is a frequent cause of the use of too much sugar. The physical constants of jellies ready to jell are practically, boiling point 103° C. and specific gravity 1.28. Jelly-making is so controlling conditions by means of acid and sugar that the pectin of the fruit juices may be precipitated evenly throughout the volume allotted to it.

The present report embraces some work on apple and quince juices, also such summer fruits as currant, raspberry, strawberry, blackberry, blueberry and cherry, and in general it strengthens the preceding conclusions.

*Extraction of Juices from Fruits.*—The juices of these summer fruits were extracted, in most cases, by adding to four or five quarts of the prepared fruit the least possible amount of distilled water (perhaps 200 cc.) just enough to prevent burning, then simmering very slowly, mashing, and finally draining through double cheesecloth. These first juice extractions then, unlike most of the fall fruits, were very nearly undiluted material. Further juice extractions were made by slowly cooking up the remaining pulp with a relatively large supply of distilled water, then draining as before. Although several such extractions with any given pulp were made, yet the alcohol test always proved that the limit of pectin extraction had not been reached; however, the proportion of it present was constantly diminished. From an economic standpoint, perhaps, it is not always practical to use the last very dilute extractions for jelly, although scientifically it would be interesting to ascertain the limit of pectin in various fruit pulps. Juices extracted as described were heated to boiling, sealed into fruit jars, and kept for future experiments.

*Proportion of Sugar to Juice.*—An effort was made

<sup>1</sup> THIS JOURNAL, 1, 333.

to discover a relationship between the specific gravity of juices and the quantity of sugar they can utilize in jelly-making. That such a relation does exist for a given juice in hand seemed probable from the results obtained, but these results did not indicate such a relationship between the juices of different fruits. Juices of different fruits (and even of the same fruit), apparently, are too variable in composition, depending upon acidity and proportion of solids present—due in part to the method of extraction and perhaps also to the condition of the fruit when used. If fruit juices were acid solutions of pectin alone, *i. e.*, contain no admixture of other solutes, probably the relation sought would be found to exist.

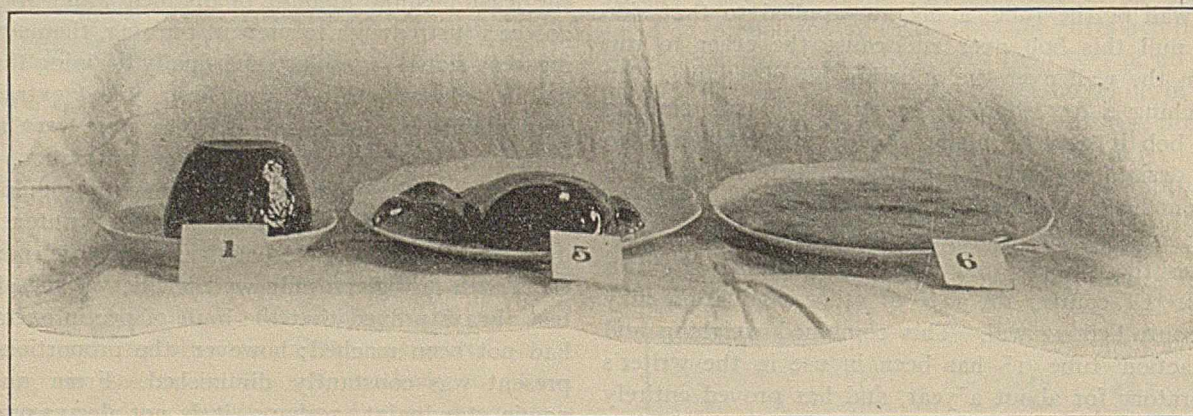
a quantity greater than the pectin of the juice can utilize. The preceding record shows some results of experiments having as their object the determination of the amount of sugar preferable for the various juices.

Examination of this table emphasizes the following facts: With increasing proportion of sugar, the time necessary for boiling decreases, the volume of jelly increases, while the texture of the jelly constantly changes from tough to a condition so soft that it will not hold together. Fig. 1 shows photographs of samples of 191, 192, 194. In general, with juices of specific gravity and acidity here considered, a three-fourths volume of sugar to one of juice was preferable, though currants and blueberries yielded jellies of good texture when the proportion of sugar to juice by volume was 1:1. Unquestionably, in the ordinary practice of jelly-making much depends upon the proportion of water added to extract the juice from the fruit, and probably something depends upon the condition of the fruit when used. In any case, too little rather than too much sugar should be the rule in jelly-making. A superabundance of sugar leads to a crystallization of sugar throughout the whole mass. Samples of 194 and 24 within a month from making were filled with large crystals.

*Inversion of Sugar in Jelly-making.*—Considerable further work has been done to determine the proportion of sugar inversion preferable in jelly-making. For this purpose jellies were made from blueberry, currant, red raspberry, blackberry, apple, quince, and plum juices—the general method of procedure being as follows: After having determined the proportion of sugar to use, then three samples of each jelly were made, the first having juice and sugar cooked together from start to finish, the second having the

TABLE I.

No.	Kind.	Sp. gr. of juice.	Per cent. of acid calculated as H <sub>2</sub> SO <sub>4</sub> .	Vol. of sugar to juice.		Time of boiling juice (min.).		Relative vol. of jelly shown by opening of angle.	Texture of jelly.
				Before adding sugar.	After adding sugar.	Before adding sugar.	After adding sugar.		
190	Sour apple	1.042	0.860	1/2 : 1	15	20		Tough	
191		1.042	0.860	3/4 : 1	15	15		Excellent	
192		1.042	0.860	1 : 1	15	12		Slightly soft	
193		1.042	0.860	1 1/2 : 1	15	8		Very soft	
194		1.042	0.860	2 : 1	15	4		Pectin pptd. in lumps.	
21	Currant <sup>1</sup>	1.038	1.892	1/2 : 1	10	3		Tough	
20		1.038	1.892	3/4 : 1	10	2		Excellent	
22		1.038	1.892	1 : 1	10	1		Very good	
23		1.038	1.892	1 1/2 : 1	10	1		Soft	
24		1.038	1.892	2 : 1	10	4		Gummy and soft	
311	Blueberry	1.020	0.483	1/4 : 1	10	15		Tough	
310		1.055	1.332	3/4 : 1	2	6		Excellent	
314		1.055	1.332	1 : 1	2	5		Good	
313		1.055	1.332	1 1/2 : 1	..	..		Soft	
281	Red raspberry	1.048	1.809	3/4 : 1	15	20		Excellent	
280		1.048	1.809	1 : 1	15	10		Soft	
292	Blackberry	1.050	1.208	3/4 : 1	15	12		Excellent	
291		1.050	1.208	1 : 1	15	5		Soft	

 $\frac{3}{4} : 1$ 

1 : 1

2 : 1

See Nos. 191, 192, and 194, Table I.  
Fig. 1.

Continued experiments proved beyond question that a most frequent cause of failure in jelly-making lies in the use of an over-abundance of sugar,<sup>2</sup> *i. e.*,

sugar added (hot) so that juice and sugar should boil together about one-half the total time of cooking, and the third having the hot sugar so added that the two should be cooked together from two to four minutes only. The results are tabulated in Table II:

<sup>1</sup> Expts. done by Miss Grace Stevens.<sup>2</sup> THIS JOURNAL, 1, 336.

TABLE II.

No. of experiment.	Kind of fruit.	Sp. gr. of juice.	Per cent. of acid in juice (calc. as H <sub>2</sub> SO <sub>4</sub> ).	No. of juice extraction.	Wt. of juice (grams).	Proportion of cane sugar to juice (by vol.).	Wt. of cane-sugar (g.).	Time boiling (min.).	Before adding sugar.	After adding sugar.	Temp. at which jelly-test was observed.	Wt. of jelly (grams).	Wt. of skimmings (grams).	Wt. of jelly and skimmings.	Per cent. of cane sugar put into jelly = E.	Saccharimeter readings.	Before inversion.	After inversion.	Temp. at which saccharimeter readings were taken.	Per cent. of cane-sugar found in jelly by Clerget's formula: $S = \frac{100(a-b)}{144 - 1/2}$	Per cent. of cane-sugar inverted = E.	Per cent. of cane-sugar inverted $100 - \frac{S(100)}{100 - E}$
400	Blueberry	1.065	1.332	I 214	1:1165.6	0	12	102°+	297.5	21.0	318.5	52.00	12.54	-9.90	22°	33.75	64.90	35.10				
401		1.065	1.332	I 214	1:1165.6	5	5	102°+	312.5	5.0	319.5	52.00	18.40	-12.10	21°	45.69	87.67	12.33				
402		1.065	1.332	I 214	1:1165.6	8	2	102°+	303.0	6.0	309.0	53.00	21.80	-10.30	22°	48.27	91.07	8.93				
403	Blueberry	1.039	0.962	II 211	3/4:1124.2	0	15	102°+	228.5	22.0	250.5	51.64	15.00	-9.70	23°	37.28	72.19	27.81				
404		1.039	0.962	II 211	3/4:1124.2	8	7	102°+	239.7	16.5	256.2	48.08	17.60	-9.20	22°	40.30	83.81	16.18				
405		1.039	0.962	II 211	3/4:1124.2	13	2	102°+	240.5	15.0	255.5	48.61	20.90	-9.70	23°	46.18	95.00	5.00				
406	Currant	1.045	2.417	I 210	3/4:1124.2	0	17	103°	211.3	23.0	234.3	53.00	4.00	-10.00	25°	21.29	40.17	59.83				
407		1.045	2.417	I 210	3/4:1124.2	9	8	103°	215.5	16.5	231.5	53.65	11.20	-10.00	26°	32.36	60.31	39.69				
408		1.045	2.417	I 210	3/4:1124.2	15	2	103°	223.0	9.0	232.0	53.53	20.55	-11.20	18°	47.03	87.85	12.15				
409	Red raspberry	1.047	1.809	I 262	3/4:1187.5	0	19	103°	320.8	18.9	339.7	55.19	9.00	-10.55	18°	29.00	52.54	47.46				
410		1.047	1.809	I 262	3/4:1187.5	10	9	103°	322.0	12.5	334.5	56.02	16.50	-11.00	18°	40.74	72.72	27.28				
411		1.047	1.809	I 262	3/4:1187.5	15	4	103°	319.0	8.7	327.7	57.21	21.40	-10.50	19°	47.43	90.84	9.16				
412	Blackberry	1.051	1.208	II 262	3/4:1187.5	0	28	103°+	304.5	19.0	323.5	58.00	16.00	-11.00	19°	40.00	69.00	31.00				
413		1.051	1.208	II 262	3/4:1187.5	18	10	103°+	298.0	18.0	316.0	59.00	24.30	-12.10	19°	54.12	91.73	8.27				
414		1.051	1.208	II 262	3/4:1187.5	25	2	103°	306.0	10.0	316.0	59.00	27.50	-11.50	19°	58.00	98.24	1.76				
415	Apple	1.029	0.484	II 255	1/2:1103.5	0	27	103°	168.0	4.0	172.0	60.00	16.00	-13.40	19°	43.71	72.85	27.15				
416		1.029	0.484	II 255	1/2:1103.5	17	9	103°	168.0	5.5	173.5	59.65	24.00	-13.75	19°	56.13	94.09	5.91				
417		1.029	0.484	II 255	1/2:1103.5	23	4	103°	175.0	6.0	181.0	57.17	23.70	-12.60	18°	53.85	94.20	5.80				
418	Quince	1.024	0.723	III 250	3/4:1187.5	0	25	103°	297.0	7.0	304.0	61.70	16.50	-11.55	18°	41.55	67.34	32.66				
419		1.024	0.723	III 250	3/4:1187.5	15	6	103°	291.0	18.0	309.0	60.70	25.20	-11.55	18°	54.44	89.68	11.32				
420		1.024	0.723	III 250	3/4:1187.5	20	3	103°	290.0	22.0	312.0	60.00	27.00	-11.44	18°	56.94	94.90	5.10				
421	Plum	1.053	1.483	I 260	3/4:1187.5	0	20	102°+	328.0	21.0	349.0	53.72	13.00	-9.00	18°	32.59	65.67	39.33				
422		1.053	1.483	I 260	3/4:1187.5	10	10	103°	320.0	18.0	338.0	55.47	20.20	-9.90	18°	44.59	80.38	19.62				
423		1.053	1.483	I 260	3/4:1187.5	17	3	103°	329.0	13.0	342.0	54.82	23.60	-9.30	18°	48.74	88.90	11.10				

Examination of this table emphasizes the general principle previously considered<sup>1</sup>, that the inversion of the sugar depends upon the time of boiling sugar and juice together and also upon the acidity of the latter. In all cases the percentage of sugar inverted increases with the time juice and sugar are boiled together. It should be stated that each of these juices was examined for sucrose, none of which was found, except a trace in red raspberry, and about 0.75 per cent. in currant—so little comparatively that no account was taken of it in the calculations. The absence of sucrose in these juices is probably accounted for by its hydrolysis through the slow cooking of the fruit in extracting the juices and by the boiling in preparation for canning them.

In making these three jelly samples from each fruit, it was expected that some indication, concerning which one of the three might be preferable, would be shown through a difference in texture, color, etc., of the jellies produced, and through some form of sugar finally crystallizing from one or more of the three samples, even though in each case an excess of sugar had been carefully avoided. However, three months after making (the surfaces of the jellies having been carefully paraffined) no trace of crystals of any sort has appeared in any sample. These jellies will be kept for future examination on this point. The jellies of each fruit seemed of uniformly good texture among themselves—the only difference discernible being a slight increase in depth of color depending upon the longer time juice and sugar had been boiled together.

In this connection an interesting economic point appears, on consideration of the columns (Table II) showing weights of skimmings and weights of jelly. In general, the greatest waste of the jelly in the form of skimmings occurs when juice and sugar are boiled together from the beginning. Conversely, the yield of jelly is likely to be greater when the sugar is added near the end of the operation.

The boiling-point at which the jelly-test was observed, and at which the jelly was removed from the fire, was approximately 103° C., with the exception of blueberry, which was found to be slightly lower. This is interesting in connection with the boiling point of cherry and strawberry jellies discussed further on.

*Use of Beet Sugar Compared with Cane Sugar.*—Several experiments were carried out comparing beet sugar and cane sugar in jelly-making. When an equal weight of pure beet sugar was substituted for that of cane sugar to a like volume of juice, the only difference observable was that the volume of jelly produced in the case of beet sugar was slightly less than the volume when cane sugar was used. No difference in the texture or taste of the two jellies was discernible.

*Hydrolysis of Pectin.*—That pectin can be hydrolyzed on sufficiently long boiling in acid solution was shown from the work of Haas and Tollens,<sup>1</sup> who found that pectin heated for eight hours with four per cent. sulphuric acid was hydrolyzed. That the pectin of fruit juices can also be hydrolyzed by the acid present through sufficiently long boiling under a reflux condenser, is probable from the results shown in Table III:

<sup>1</sup> Bureau of Chem., Bull. 66, 52. THIS JOURNAL, 1, 338.

<sup>1</sup> Ann. Chem. (Liebig). 286, 278.

TABLE III.

No.	Juices.	Sp. gr. of juice.	Percent. of acid calculated as $H_2SO_4$ .	Time of boiling (hours.)	Sugar.	Result.
1	Currant	1.050	3.212	5.3/4	3/4 : 1	No test for pectin
2	Currant	1.048	1.925	10	None	No test for pectin
3	Red raspberry	1.050	1.925	10	3/4 : 1	No test for pectin
4	Red raspberry	1.050	1.925	10	None	No test for pectin
5	Blueberry	1.055	1.332	16	None	No test for pectin
6	Apple	1.042	0.860	16	None	No test for pectin
7	Plum	1.053	1.483	12	None	No test for pectin
8	Quince	1.024	0.723	18	None	No test for pectin

From these results it would appear that the acidity of the juice modifies the time necessary to destroy the pectin as such (compare 1 and 2). Doubtless the percentage of pectin in the juice modifies the result also—*e. g.*, for blueberry and quince (5 and 8) two juices known to be particularly rich in pectin, a long time was necessary, 16 to 18 hours; though in the case of quince doubtless the low acidity also increased the length of time. Apple juice (6) of moderate acidity comparatively, but of high specific gravity, also required 16 hours. Apparently the presence of sugar in these juices (see 3 and 4, whose acidity is nearly the same) had little or no effect on the time.

From what has been shown, then, it would appear that it is possible to spoil jelly through long heating. In practice this would be unusual on account of the hours of time necessary to hydrolyze the pectin, but a case of this sort did come to the writer's attention through the failure of one home jelly-maker to make her grape jelly "jell." Hours of simmering on the back of the stove had destroyed the pectin.

Further, in the light of these experiments, it may be questioned whether or not it is wise to attempt to use the later juice extractions mentioned earlier in this article, for jellies. It was found, however, that these later extractions could be used for jellies either by first concentrating to a specific gravity approximating that of the first extraction, before determining upon the volume of sugar to be used, or by proportioning the sugar with due regard for the specific gravity and the acidity of the juice. The comparatively short time necessary for concentration, and the low relative acidity (insufficient for hydrolysis under the time condition) did not affect the jelly-making qualities of these later extractions.

The results shown in Table II possibly indicate that sufficiently refined methods for the determination of the percentage of pectin in a fruit juice will reveal the fact that this percentage varies with the condition of the fruit.

*Absence of Pectin from Pine-apple.*—No pectin could be found in cooked pine-apple—the raw juice was unexamined. This lack of pectin in pine-apple explains, of course, the failure to produce jellies from

this juice, which was experimentally demonstrated in the course of these experiments. If the pine-apples in hand, then, were fair samples of their kind, then, pine-apple jellies must contain some other juice (apple?) to supply pectin, the jelly-making basis—the pine-apple supplying the flavor.

*Absence of Pectin from Juices of Uncooked Fruits.*—The absence of pectin from the juices of certain uncooked fruits was observed. Little was found in apple, none in quince, lemon, or orange—these being all the raw juices so far examined.<sup>1</sup> As is well known, the juice of cooked apple or of cooked quince each contains an abundance of pectin and these two fruits are among the standards for jelly-making. Of course, it is a simple matter to obtain the raw juices of oranges and of lemons. To obtain the raw juices (cider) of apples or quinces the fruit was cut (pulp with skins and seeds) into quarters, then passed through a meat grinder, and the ground mass drained through cloth. The flavor of these juices was excellent, but no pectin could be detected in the quince juice, and only a small amount in that of the apple. The results of attempts to make jelly from this apple juice led to an opaque, gummy mass, having a strong flavor of apples (see 202 and 203, Table IV). The ground mass above mentioned of either apple or quince, when cooked slowly with water sufficient to cover, yielded a juice containing an abundance of pectin as usual—the quince a superabundance of it. However, such an apple juice refused to make a clear jelly (see 204, Table IV) though the low proportion of sugar ( $1/4 : 1$ ) here used might have had something to do with this, as it certainly did with the tough texture—a low proportion of sugar was here used, however, on account of the low specific gravity (1.020) and the low acidity (0.154) of this juice (most of the acid of the fruit having appeared in the cider, acidity 0.633).

*Extraction of Apple Juice by Use of Double Boiler.*—To avoid any dilution of apple juice the prepared fruit was cooked in a double boiler until soft, then mashed and drained as usual. However, this juice (sp. gr. 1.090 and acidity 0.967) refused to produce a clear jelly even when an equal volume (1:1) of sugar was used (206, Table IV), though the taste was excellent. The remaining pulp, after cooking with water by the usual method, still failed to produce a clear jelly (207, Table IV), though the texture was good.

Nos. 211 and 212, Table IV, show the data of two quince jellies, the second of which was artificially acidified. Such addition of acid (tartaric) improved the texture of this jelly much, and in this instance the taste also:

<sup>1</sup> Since this paper was written, the raw juices of currants and of blackberries have been examined. Each was found to contain pectin, but in less proportion than the juice of the cooked fruit. The jelly made from raw currant juice was not clear. That from blackberry (sugar  $1/2 : 1$ ) was exceedingly clear and of excellent quality.

TABLE IV.

No.	Fruit.	Extraction.	Sp. gr. of juice.	Per cent of acid calculated as H <sub>2</sub> SO <sub>4</sub> .	Vol. of sugar to juice.	Time of boiling (min.).		Texture of jelly.	Remarks.
						Before adding sugar.	After adding sugar.		
202	Northern Spy apple	Cider	1.057	0.633	3/4 : 1	10	15	Gum	Strong taste of fruit, muddy
203	Northern Spy apple	Cider	1.057	0.633	1/2 : 1	10	20	Gum	Strong taste of fruit, muddy
204	Northern Spy apple	Ext. after cider	1.020	0.154	1/4 : 1	40	20	Rather tough	Not clear
205	Northern Spy apple	II	1.027	0.385	1/2 : 1	20	30	Rather tough	Clear, excellent flavor
206	Northern Spy apple	Double boiler	1.090	0.967	1 : 1	..	12	Rather tough	Not clear, flavor excellent
207	Northern Spy apple	Ext. after double boiler	1.0375	0.434	1/2 : 1	20	8	Good	Not clear, flavor excellent
211	Quince	II	1.016	0.589	3/4 : 1	..	..	Soft	
212	Quince	II	1.016	0.877	3/4 : 1	..	..	Good	

*Orange and Lemon Jellies.*—Quite as interesting as the absence of pectin from the raw juices of oranges and lemons is the fact that on cutting either of them into thin slices, adding distilled water and cooking slowly for some hours, an abundance of pectin was found in the resulting drained-out juice. Further experiments located this pectin in the white inner skin. The method of its extraction was as follows: A thin peeling sufficient to remove the yellow, oily portion was cut off, then the thick white skin removed from the pulp. This thick white skin was passed through a fine meat grinder, soaked in distilled water (sufficient to cover) over night, cooked very slowly for several hours, and then drained. This first extraction was particularly rich in pectin. Subsequent extractions of this pulp always showed a considerable proportion of pectin, but decreasing in amount. These extractions, on addition of some of the clear juice from the pulp, for acidity and flavor, yielded excellent jellies. If the thin outer yellow skin had been carefully removed they had little or no bitter taste. The following table comprises some data concerning jellies of oranges and of lemons.

it was acidified either by the raw juice (249, 302) or by some other acid as tartaric (249a).

Then, in what is usually a waste product (the white inner skins of oranges and lemons) we have an abundant source of pectin from which excellent jelly can be made if properly acidified.

*Isolation of a Pure Pectin.*—It was noteworthy that the purest pectin yet prepared in this research was obtained from oranges and lemons. It was isolated by the method heretofore used<sup>1</sup> and was reprecipitated three times. By long manipulation of the precipitated pectin (supported on a very fine cloth suspended from the corners) the liquid was so completely worked out of the substance that a powdery white body, somewhat starch-like in appearance, was obtained. This was dried in a current of dry hydrogen over sulphuric acid.

Ash determinations of orange pectin so obtained showed less than 0.5 per cent. of ash—of lemon pectin about 3.5 per cent. Compare Haas and Tollens. No melting-point of this pectin could be obtained, but the substance, when out of contact with air, chars strongly at 170° C.

TABLE V.

No.	Kind.	Juice extraction.	Sp. gr. of juice.	Per cent of acid calc. as H <sub>2</sub> SO <sub>4</sub> .	Vol. of sugar to juice.	Time of boiling (min.).		Results and remarks.
						Before adding sugar.	After adding sugar.	
239	Orange	Raw juice	1.048	1.932	..	..	..	No pectin, hence no jelly
240	Orange	Cooked whole fruit	1.037	0.482	1/2 : 1	10	10	Thick gum, finally set to fair jelly (bitter, marmalade-like)
242	Orange	Cooked whole skins	1.018	0.123	1/2 : 1	20	20	Thick syrupy gum, bitter
245	Orange	Cooked inner white skin	1.017	trace	1/2 : 1	10	8	Syrup
246	Orange	Cooked outer yellow skin	1.015	trace	1/2 : 1	10	5	Syrup, bitter
247	Orange	Cooked fruit exclusive of yellow skin	1.050	1.031	3/4 : 1	5	8	Good jelly, good flavor
249	Orange	Cooked inner white skin + raw juice	..	1.031	3/4 : 1	5	10	Tender jelly
249a	Orange	Cooked inner white skin + tartaric acid	..	1.031	3/4 : 1	5	10	Good jelly
249b	Orange	Cooked outer yellow skin + tartaric acid	..	1.031	3/4 : 1	5	10	Gum
300	Lemon	Cooked fruit exclusive of yellow skin	1.008	3.988	3/4 : 1	10	10	Firm jelly (sour)
301	Lemon	Cooked inner white skin	1.005	trace	1/2 : 1	15	8	Syrup
302	Lemon	Cooked inner skin + raw juice	1.008	1.039	3/4 : 1	10	5	Excellent jelly

Results given in this table show that if the yellow outer skin is used the product is more or less gummy (240, 242, 246, 249b). Good jellies from the cooked whole fruit, exclusive of the yellow skin, could be obtained (247, 300). The juice from the cooked inner white skin contained a large percentage of pectin, but no jelly (245, 301) could be obtained from it till

It is hoped to continue this work on the isolation and examination of pure pectin.

*Cherry and Strawberry Jellies.*—Although the juices of these fruits contain pectin in abundance and are quite acidic, yet they offer much difficulty if jellies

<sup>1</sup> THIS JOURNAL, 1, 339. Haas and Tollens: *Ann. Chem.* (Liebig), 286, 278.

from them are attempted. The cause of this difficulty appears to lie in a more or less gummy substance which accompanies the pectin and which interferes with its nice action. An increase of the acidity of the natural acid of the strawberry (by addition of tartaric acid) aided materially in the precipitation of a jelly from this fruit, but the characteristic flavor of the strawberry was destroyed just as in the case of pear and peach jellies. Cherry juice is so strongly acid that further acidification of it was not attempted. (Wild red cherries were found to yield an excellent jelly.)

Experiment finally proved that in making strawberry and cherry jellies reliance could not be placed upon the first indication of the jelly-test as is usual in other jellies, but that the concentration must be continued beyond that point, and the results were best, although not ideal, when the temperature of the boiling mixture was allowed to reach 105° C. No attempt is made at present to account for this—merely the fact is stated. If the concentration was continued beyond this temperature merely a sticky gummy mass resulted. Very interesting in this connection is the statement by Belling,<sup>1</sup> who, in making guava jelly, found that the boiling point must be raised to 113° C. Evidently the temperature is no infallible criterion for determining the jelly point.<sup>2</sup> Of course, to a certain degree undoubtedly, different experimenters would reach different conclusions regarding the point at which jelly is to be considered done, since the personal equation must enter more or less into any decision regarding the texture of jelly which is to be called ideal.

#### SUMMARY.

1. A very frequent cause of failure in jelly-making is the use of too much sugar.
2. The percentage of inversion of sugar most desirable in jellies has not been determined.
3. Beet sugar and cane sugar may be used interchangeably in jelly-making.
4. Pectin of fruit juices may be hydrolyzed by the acids present if boiled sufficiently long.
5. The raw fruit juices, so far examined, contain less pectin than do those of the cooked fruit. Sometimes the former are pectin-free.
6. The white inner skin of oranges and of lemons is a prolific source of pectin.
7. To insure results approximately good, the boiling point of strawberry and of cherry jellies must be allowed to increase (as much as two degrees) above that at which the jelly-test is observed.
8. Owing to variations in specific gravity and acid-

<sup>1</sup> Florida Ag. Exp. Sta. Record, 106, (1908).

<sup>2</sup> The work of Miss Snow (*Farmers' Bull.* 388, 30) regarding the density of jelly at its boiling point may here be cited.

ity of juice, good jellies cannot be made by rule o' thumb.

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### THE CHEMICAL AND MINERALOGICAL EXAMINATION OF SOME CHINESE TEA SOILS.<sup>1</sup>

By W. O. ROBINSON AND W. J. MCCAUGHEY.

[ Received June 3, 1910.

Three samples of Chinese tea soils were submitted to this laboratory for analysis by Dr. Rodney H. True, of the Drug Plant, Poisonous Plant, Physiological and Fermentation Investigations, Laboratory of the Bureau of Plant Industry, U. S. Department of Agriculture. These soils were collected by Vice-Consul H. E. Nightingale, from the northern central part of the Province of Fukien, about 150 miles north of Foochow, at Kienning fu and in the Wu I Shan district.

In this district a tea of very superior quality is grown. The varieties are known as the "Dragons Pool" and "White Coxcomb." These teas are not sold but are given as tribute to the Chinese throne. The growth and picking of the tea is attended with religious ceremony.

The soil is held by the Chinese to be the most important factor in tea cultivation. The object is to produce a rather stunted shrub, for it is held that under these conditions a more desirable flavor is developed in the tea. The culture is very simple, being restricted to digging around the plants twice a year and keeping the weeds pulled. They are seldom, if ever, fertilized.

It is interesting to compare the composition of American soils that have been under cultivation for a comparatively very short time, with these Chinese soils, especially when we consider how very little has been added to these latter by way of fertilizers, and that for thousands of years they have been under a clean cultivation, the tea leaves and weeds having been removed from their place of growth and not returned to the soil. The following analyses represent the compositions of soils that have been under cultivation longer probably than any soils of which we are aware. These soils were weathered from Middle Paleozoic formations. The present altitude is 500-1000 feet. The formation has shown a decided tendency to stand high and probably has been subjected to denudations.

Soil No. 1 came from the Wu I Shan district, which is just south of the Tayuling Mountains.

Soils Nos. 2 and 3 came from Kienning fu, a town on the junction of the Min and Sing ki rivers, about 100 miles above Foochow, in the province of Fukien in South China.

<sup>1</sup> Published by permission of the Secretary of Agriculture.

The following examinations have been made:

1. Chemical, consisting of an analysis for the total constituents and the constituents soluble in hydrochloric acid according to the official method given in Bulletin No. 107 of the Bureau of Chemistry, U. S. Department of Agriculture.
2. Mechanical analysis.
3. Mineralogical examination.
4. An examination made for the purpose of correlating these soils with recognized American types.

The chemical examination shows that the composition is much the same as that of fertile American soils. The phosphoric acid is fairly high, both the total and the acid-soluble. The acid-soluble potash, lime and magnesia are much the same as in the American soils.

The analyses are given in Tables I and II.

TABLE I.—COMPOSITION OF CHINESE TEA SOILS.  
Ultimate analysis. Analyses made by W. O. Robinson.

	No. 1. Per cent.	No. 2. Per cent.	No. 3. Per cent.
Loss on ignition.....	10.14	6.69	8.29
SiO <sub>2</sub> .....	54.75	73.97	67.36
Fe <sub>2</sub> O <sub>3</sub> .....	9.50	3.65	5.26
Al <sub>2</sub> O <sub>3</sub> .....	22.02	12.48	13.72
TiO <sub>2</sub> .....	0.95	0.53	0.81
MnO.....	0.023	0.014	0.105
CaO.....	0.18	0.28	0.58
MgO.....	0.14	0.52	0.45
Na <sub>2</sub> O.....	0.26	0.34	0.42
K <sub>2</sub> O.....	1.26	1.71	2.32
P <sub>2</sub> O <sub>5</sub> .....	0.51	0.24	0.59

TABLE II.—COMPOSITION OF CHINESE TEA SOILS.  
Acid digestion. Official method. Analyses made by W. O. Robinson.

	No. 1. Per cent.	No. 2. Per cent.	No. 3. Per cent.
Loss on ignition.....	10.14	6.48	8.01
Insoluble.....	63.98	81.27	79.28
Fe <sub>2</sub> O <sub>3</sub> .....	8.82	3.03	4.41
Al <sub>2</sub> O <sub>3</sub> + TiO <sub>2</sub> .....	16.43	7.83	6.63
MnO.....	0.022	0.012	0.095
CaO.....	0.11	0.10	0.23
MgO.....	0.09	0.40	0.41
Na <sub>2</sub> O.....	0.09	0.08	0.09
K <sub>2</sub> O.....	0.39	0.44	0.31
P <sub>2</sub> O <sub>5</sub> .....	0.24	0.14	0.32
Nitrogen by moist combustion.	0.052	0.068	0.154

The solubility of the potash in these soils is interesting. It will be seen that soil No. 3 is highest in total potash and lowest in acid-soluble potash. By inspection of Table IV it is evident that soil No. 3 differs quite widely in mineralogical composition from No. 1 and No. 2, the most striking difference being the large amount of microcline in No. 3. This

mineral is found in the soil with sharp, unweathered faces and is acid-resistant, facts that account for the low solubility of potash.

The mechanical analysis given in Table III shows that soil No. 1 is a heavy clay and that soils Nos. 2 and 3 are loams.

The results of the mineralogical examination are given in Table IV. Examination by W. J. McCaughey:

TABLE IV.

No. 1. Clay soil. The sands are largely quartz; minerals other than quartz relatively low; of these zircon and mica are most abundant. The mica, for the greater part, is biotite. Hornblende, chlorite and tourmaline are present. Hornblende and biotite show weathering and transformation into chlorite.

No. 2. Loam. Minerals other than quartz in the sands are fairly abundant. Micas (biotite and muscovite) very abundant. Orthoclase abundant, being largely kaolinized. Tourmaline is present. Microcline and zircon are common accessories.

No. 3. Loam. Kaolin, quartz, mica (muscovite and phlogopite), microcline, and magnetite are quite abundant. Hematite is present in some quantity, generally as a coating on soil grains. Partly kaolinized orthoclase is a common accessory. Hornblende, epidote, tourmaline and zircon are present. The mica carries inclusions of apatite and magnetite.

It is the opinion of Professor G. P. Merrill, who has kindly looked over the rock fragments in these soils, that Soils Nos. 1 and 2 have probably resulted from the decay of sandstones, the material having been transported but little and being originally derived from granitic material. Soil No. 3 has probably resulted from the decay of a volcanic mud of the trachyte type. The rock fragments contain quartz and are plainly secondary.

There are no soils yet described in the United States that correspond in general type characteristics to these Chinese soils. The nearest approach are the soils of the Cecil, Chester or Durham series. They most resemble the soils in the southern Piedmont plateau and those in Oklahoma, and this fact, in view of the conditions of the climate in these regions, would appear to make these areas desirable for experimental work on tea culture.

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### NOTES ON THE DETERMINATION OF NITROGEN BY THE KJELDAHL METHOD.

By P. L. HIBBARD.  
Received July 29, 1910.

The routine chemist usually follows some official or prescribed method, with little thought as to the reasons for each step, or the effect of variations. He should know the effect of varying time, temperature, quantity of reagents, and size of apparatus on his results. In view of these needs the following experiments were made. It is to be understood that they cover only part of the ground and that only a few of the possible variations have been tried. Naturally the practice in different laboratories varies to some extent.

*Conditions of the Experiments.*—Substance taken—1.401 grams (ground to pass 1 mm. sieve) with

TABLE III.—MECHANICAL ANALYSES OF SOILS FROM CHINA, MADE BY C. C. FLETCHER.

No.	Description.	Fine earth.						
		Organic matter. Percent.	Fine gravel 2 to 1 mm. Per cent.	Coarse sand, 1 to 0.5 mm. P. ct.	Medium sand, 0.5 to 0.25 mm. P. ct.	Fine sand, 0.25 to 0.1 mm. P. ct.	Very fine sand 0.1 to 0.05 mm. P. ct.	Silt 0.05 to 0.005 mm. Percent.
(1)	Red clay.....	1.8	5.0	3.4	7.8	4.2	19.4	58.3
(2)	Loam.....	5.9	14.3	8.4	12.2	8.4	24.0	26.8
(3)	Loam.....	2.4	6.5	6.4	18.1	14.2	30.6	21.7

25 cc. sulfuric acid and 10–12 grams of a mixture of potassium sulfate with 5 per cent. of copper sulfate. The substance to be analyzed is placed in a 500 cc. round-bottomed Jena flask, neck about 6 inches long, 1 inch diameter. The potassium sulfate mixture is added and mixed with the substance to avoid balling, then the acid is added and mixed and the flask supported at an angle of  $45^\circ$  on an asbestos ring 2 inches in diameter of opening. It is heated by a Bunsen flame, regulated so that the acid boils gently, and volatilizes slowly. During three hours' digestion it may lose one-third the volume of acid. After the digestion it is cooled, 250 cc. are water added, a little granulated pumice and enough strong caustic soda, 50–60 cc., to make alkaline. After connecting with the condenser, the flask is shaken to mix the contents. It is then heated by direct flame so that about 200 cc. are distilled off in course of about one hour. The ammonia is collected in standard half normal hydrochloric acid, the excess of which is titrated back with standard tenth-normal ammonia with cochineal indicator. Thus, the result is found as percentage of nitrogen without tables or calculations.

1. *Time for Digesting Dried Blood.*—Used an ordinary sample, which contained, as an average of five determinations by regular method, 13.27 per cent. nitrogen.

Time digested. Hour.	Per cent. nitrogen found.
$\frac{1}{2}$	12.80
1	13.00
$1\frac{1}{2}$	13.10
2	13.22
3	13.27
5	13.27

Three hours seem to be sufficient time.

2. *Time for Digesting Bone Meal.*—Used a sample of steamed bone, rather high in nitrogen.

Time digested. Hour.	Per cent. nitrogen found.
$\frac{1}{2}$	3.87
$\frac{3}{4}$	3.88
1	3.93
$1\frac{1}{4}$	3.98
2	3.99
3	4.04, 4.05 Two determinations
4	4.05
5	4.04, 4.05 Two determinations

Time needed, about three hours.

3. *Time for Digesting Nitrate of Soda to Reduce it to Ammonia by Ulsoh-Street Method.*—Used same flasks as for Kjeldahl work, in which were placed 25 cc. of a solution containing 0.5 gram of sodium nitrate, added 5 grams iron powder (reduced by hydrogen), and 20 cc. of 1 : 3 sulfuric acid. After standing for varying time, added 50 cc. water, heated to boiling for 30 minutes. Cooled, added 200 cc. water, 30 cc. caustic soda, and some pumice, distilled as in Kjeldahl method.

Time digested before heating. Hour.	Per cent. of nitrogen.
0	15.44
$\frac{1}{4}$	15.55
$\frac{1}{2}$	15.53
1	15.53
2	15.53
4	15.47
6	15.36

It appears that very little time is required for digestion before heating. On account of the long time in the two last experiments all the acid was used up by the excess of iron present so that on boiling 30 minutes some basic iron sulfate was formed and some ammonia thereby expelled. Hence, it is concluded that an excess of acid should be used, at least 25 cc. 1 : 3, and thus prevent loss of ammonia.

4. *Time Required to Expel Ammonia in Distillation of Kjeldahl Digestion.*—One gram ammonium sulfate placed in flask same as for regular Kjeldahl distillation. Added water and soda, etc., distilled as usual. Some cochineal was placed in the receiver and tenth-normal hydrochloric acid added gradually as was needed to neutralize the ammonia distilled over.

Time. Min.	Volume distilled.	cc. N/10 HCl used.
2.....	...	42
4.....	...	85
6.....	...	122
8.....	...	137
10.....	51	145
14.....	...	148.5
19.....	100	149.0
24.....	...	149.0
29.....	160	149.0
45.....	220	149.0

Thus shown that 15 minutes' boiling will expel nearly all the ammonia, which comes over with about 75 cc. of water. The amount of ammonia present at the start was as much as is found in the ordinary Kjeldahl determination.

5. *Time Required to Expel Ammonia by Distilling with Magnesium Oxide.*—Experiment conducted like the preceding, but using 5 grams MgO instead of the soda solution and having present 0.5 gram ammonium sulfate. On account of the greater tendency to foam, the distillation with magnesia must go slower than with soda, yet this experiment shows no great difference between the two. A parallel experiment with soda was made following this, and the results are presented together.

Time. Min.	MgO.		NaOH.	
	Volume distilled. cc.	N/10 HCl used. cc.	Volume distilled. cc.	N/10 HCl used. cc.
2.....	...	30	...	34
4.....	...	46	...	54
6.....	...	58	...	63.5
8.....	...	64	...	68.5
10.....	31	69	34	71.5
12.....	...	72	...	73.3
17.....	...	73.8	59	74.5
22.....	...	74.4	...	74.7
27.....	96	74.5	...	74.7
42.....	146	74.5	155	74.7
52.....	196	74.5	...	....



Thus shown that it is not necessary to distil nearly to dryness with MgO to obtain all the ammonia.

6. *Optimum K<sub>2</sub>SO<sub>4</sub> for Kjeldahl Digestion.* Same Apparatus as for Experiment 1.—1.401 grams dried blood, 25 cc. H<sub>2</sub>SO<sub>4</sub> and varying amount of K<sub>2</sub>SO<sub>4</sub>.

K <sub>2</sub> SO <sub>4</sub> . Grams.	Time till clear.	Time digested.	Color of diluted solution.	Per cent. nitrogen.
2.....	did not clear	6 hours	Yellow	12.98
4.....	3 hours	3 "	Yellow	12.95
6.....	2½ "	3 "	Yellowish	13.00
8.....	2 "	3 "	Colorless	13.15
10.....	1¾ "	3 "	"	13.33
12.....	1½ "	3 "	"	13.32
14.....	1¼ "	3 "	"	13.40
16.....	1 "	3 "	"	13.41

On account of difficulty during distillation, it seems not desirable to use more than 10–12 grams K<sub>2</sub>SO<sub>4</sub> and this is not sufficient to secure complete oxidation within three hours, so copper sulfate is added, hence the following experiments with CuSO<sub>4</sub>.

7. *Optimum CuSO<sub>4</sub> for Kjeldahl Digestion.*—Similar to last experiment using 10 grams K<sub>2</sub>SO<sub>4</sub> in each test, but varying CuSO<sub>4</sub>.

CuSO <sub>4</sub> . Grams.	Time till clear. Min.	Total time heated. Hours.	Per cent. nitrogen.
0 .....	70	2	13.10
0.2.....	45	2	13.35
0.4.....	38	2	13.37
0.6.....	35	2	13.38
0.8.....	30	2	13.34
1.0.....	30	2	13.34
1.5.....	30	2	13.34
2.0.....	30	2	13.27

In order that the effect of the different amounts might be more easily seen the digestion was continued for only 2 hours, which was not long enough to obtain all the nitrogen.

ANOTHER SET.

CuSO <sub>4</sub> .	Time till clear. Min.	Total time heated. Hours.	Per cent. nitrogen.
0.5.....	35	3	13.50
0.5.....	35	3	13.47
1.0.....	35	3	13.36
1.0.....	35	3	13.27
2.0.....	35	3	13.27
2.0.....	35	3	13.23

With 0.5 gram CuSO<sub>4</sub> and 10 grams K<sub>2</sub>SO<sub>4</sub>, and heating three hours, the results were pretty nearly correct. Cause of low results with upward of 0.5 gram CuSO<sub>4</sub> is unknown. However, more than 1 gram is useless, as it is simply dehydrated and remains undissolved in the acid. The equal time required to become clear, with varying amounts of CuSO<sub>4</sub>, indicates that the larger amounts do not hasten oxidation.

It seems desirable to use not much more than half a gram of CuSO<sub>4</sub>. The third set was run with 0.5 gram CuSO<sub>4</sub> and varying K<sub>2</sub>SO<sub>4</sub>.

K <sub>2</sub> SO <sub>4</sub> . Grams.	Time till clear. Min.	Total time heated. Hours.	Per cent. nitrogen.
2.....	120	3	13.12
4.....	65	3	13.28
6.....	45	3	13.43
8.....	40	3	13.45

Results not high enough.

Using 0.8 gram CuSO<sub>4</sub>.

6.....	55	3	13.48
8.....	36	3	13.54
10.....	32	3	13.56
12.....	28	3	13.54

The larger quantity of copper seems able to replace some of the K<sub>2</sub>SO<sub>4</sub>. From all of these results it seems that 10 grams K<sub>2</sub>SO<sub>4</sub> with 0.5 gram CuSO<sub>4</sub> is the most suitable combination. Using this mixture the following results were obtained:

Heated 3 hours.....	13.53, 13.52 per cent. nitrogen.
Heated 6 hours.....	13.48, 13.53 per cent. nitrogen.
Heated 10 hours.....	13.55, 13.56 per cent. nitrogen.

Three hours' heating seems sufficient.

8. *Prevention of Bumping.*—In this connection, it is interesting to note that presence of the excess of CuSO<sub>4</sub> which remains insoluble in the boiling acid prevents the bumping which often accompanies this method of digestion, but as it tends to cause low results, CuSO<sub>4</sub> cannot be used. FeSO<sub>4</sub> crystals were tried and found to prevent bumping as well as CuSO<sub>4</sub>, but without causing low results for nitrogen. Probably Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> would be better, but not being at hand it was not tried. In case a digestion bumps badly, a gram or two of crystallized ferrous sulfate dropped; it nearly always produces quiet boiling.

9. *Loss of Ammonia during Digestion, through Dissociation of Ammonium Sulfate.*—It has been said that in the Gunning nitrogen determination there is danger of loss of ammonia, if the flame strikes the flask above the level of the acid, thereby dissociating (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> with loss of NH<sub>3</sub> due to high temperature. In order to settle this question several experiments were made.

(1) One gram (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> plus 25 cc. H<sub>2</sub>SO<sub>4</sub> in 300 cc. flask connected with condenser. Distilled off two-thirds of the acid. The distillate was neutralized and distilled; only a trace of NH<sub>3</sub> found in it. Flask heated with bare flame.

(2) Similar to (1) but added 10 grams K<sub>2</sub>SO<sub>4</sub> CuSO<sub>4</sub> mixture as for regular nitrogen determination. Distilled off two-thirds of the acid with bare flame. Found in distillate nitrogen = 0.020 gram = 2 per cent. on ordinary determination. Presence of K<sub>2</sub>SO<sub>4</sub> seems to cause loss of nitrogen along with the acid distilled over.

(3) Used same materials as in last experiment but placed in regular 500 cc. flask and heated as for regular nitrogen determination. The vapors coming from the mouth of the flask were collected, neutralized, and distilled for NH<sub>3</sub>. For the first two hours' heating the distillate contained only a trace of NH<sub>3</sub>. For the

third hour, found in distillate  $\text{NH}_3 = 0.015$  gram  $\text{N} = 1.5$  per cent. Stopped heating as most of the acid was distilled out.

(4) Used a sample of dried blood, run as for regular nitrogen determination, except heating faster and collecting acid and other vapors distilled out. First hour, found in distillate  $\text{NH}_3 = 0$ , during this time about half the acid was distilled out. After 15 minutes more, stopped heating as nearly all free acid was expelled. Found in second distillate  $\text{N} = 0.008$  gram; in digestion flask  $\text{N} = 0.051$ . Out of 13.5 per cent. present at start only 5.9 per cent. were recovered. The other 7.6 per cent. were lost through dissociation or some other cause; unknown. These extreme and unusual conditions proved the possibility of loss. It was next determined that under ordinary conditions there is no loss.

(5) Same as last experiment but heated 3 hours supported by large asbestos rings as in usual manner for nitrogen determination, except that the rings were large enough so that a portion of the flask was exposed to the flame above the level of the acid. Lost about 20 per cent. of total acid. Found in the digestion, two tests 13.52, 13.53 per cent. nitrogen.

(6) Like the last but heated 6 hours on small rings, boiled gently. Nearly half the acid was lost; found, two tests,  $\text{N} = 13.48, 13.53$ , hence no loss occurred.

(7) Similar to last, but used large asbestos rings; heated at ordinary rate. After 3 hours' heating added 10 cc. more acid, after next two hours' heating added 10 cc. acid again. Continued heating 5 hours longer. Total time heated 10 hours. Total acid used 45 cc. Acid remaining in flasks, about 30 cc. Found  $\text{N} = 13.55, 13.56$  per cent.

From this it is concluded that if the volume of acid does not fall below 20 cc. there is no danger of loss of nitrogen even though the flame strikes the naked flask above the acid.

#### SUMMARY.

(1 and 2) About three hours are required to obtain all the nitrogen in dried blood or bone meal by sulfuric acid digestion with 10 grams  $\text{K}_2\text{SO}_4$  plus 0.5 gram  $\text{CuSO}_4$ .

(3) Using the Ulsch-Street method, only a few minutes are required to change the nitrogen of nitrates to ammonia.

(4 and 5) In distilling ammonium salts with soda or with magnesia, nearly all the ammonia, together with 50–75 cc. water, is obtained during the first 10–15 minutes.

(6) About 10 grams  $\text{K}_2\text{SO}_4$  is the most satisfactory quantity for aiding the acid digestion of nitrogenous substances.

(7) One-half gram  $\text{CuSO}_4$  added with the  $\text{K}_2\text{SO}_4$  hastens oxidation about as well as larger amounts. Much more than this seems to cause loss of nitrogen; the reason for such loss is not known.

(8) Bumping of the digestion is prevented by addition of 1–2 grams ferrous sulfate.

(9) Loss of ammonia during the acid digestion occurs when a large portion of the acid has been driven out by too much or too long continued heat; but not because the flask is heated by the bare flame above the level of the boiling acid. Or, it may be said that loss occurs when, owing to loss of acid, the boiling point of the residue rises too high.

STATE FERTILIZER CONTROL, UNIVERSITY OF CALIF.,  
BERKELEY, CALIF.

## ADDRESSES.

### THE ELECTROLYTIC PREPARATION OF CALCIUM.<sup>1</sup>

By ARDEN R. JOHNSON.

It is the main purpose of this paper to set forth the most essential results of considerable experimental work upon the electrolytic preparation of metallic calcium, carried out in the electrochemical laboratories of the University of Wisconsin during the school year 1905–1906. The work was written up and presented as a thesis toward the baccalaureate degree in engineering, but was never published in a scientific journal.

Several good papers have been published in the last three or four years on the preparation of calcium, some by members of this society. It is therefore my object to present only data and statements that will largely supplement or reinforce much that has already appeared in print in leading journals.

While the thesis as originally written up four years ago contains an historical review of experimental endeavors in this line, I shall forego the presentation of the same here for the reason that the work has already been well given by Mr. F. C. Frary.<sup>2</sup> But my observations and data obtained upon two or three typical styles of apparatus much used—apparatus with typically inherent faults, and also an electrolyte with all the common capricious properties, will, I think, be of some value to the many electrochemists who, of late, have become interested in the chemical and metallurgical properties of this element.

The main efforts to produce calcium seem to have been through the medium of a correctly designed apparatus for electrolyzing, and if we glance over the different designs we shall recognize just two different types: First, the apparatus of the Borchers type for utilizing an extremely high current density and consequently obtaining the calcium in a molten condition, in which it rises to the surface and is skimmed off, and secondly, the Rathenau type, which requires that the calcium come out in a compact form, that is, plated out at a moderately low current density.

*Experiments with Apparatus No. 1.*—In view of the successful results which have been attained by the above mentioned experiments, using the Rathenau type of apparatus, I decided also to begin the study of the electrolysis of  $\text{CaCl}_2$  with an apparatus of this class. Accordingly the design shown in Plate II was followed, making use of such data concerning dimensions of anode and cathode as could be gleaned from the published description of Goodwin and Woehler's apparatus.

While a goodly number of trials with this apparatus were carried out, most of which were not over two to three hours duration, only three or four performed under different conditions will be described.

A run was made using pure  $\text{CaCl}_2$  of the very best grade, made by Baker & Adamson. The cylindrical, graphite vessel was filled with  $\text{CaCl}_2$  and then by the use of the auxiliary elec-

<sup>1</sup> Read before the Chicago meeting of the American Electrochemical Society, October 13–15, 1910.

<sup>2</sup> *Trans. Am. Electrochem. Soc.*, **16**, 185 (1909).

trode an arc was struck with the inner surface of the anode container, and the  $\text{CaCl}_2$  gradually melted down in the vicinity of the arc until a small pool of fused  $\text{CaCl}_2$  was formed. The

under the influence of the convection currents. It was immediately recognized that a furnace of the Borchers type was at hand, so the current was gradually cut down as low as 40 to 50 amperes (10 volts drop across cell), at which point a reguline deposit was noticed forming at the end of the rod. But it was now found impossible to keep all the bath fused next to the anode, and finally there was only a comparatively narrow channel of molten salt between anode and cathode. A glowing phenomenon, like a sheet of small arcs, was noticed at the anode. After the bath had been in operation under these conditions some time the iron rod was raised a trifle and electrolysis continued. The calcium "rod" which was forming was very irregular in cross-section, and continued in spite of all efforts to "neck down" until the "rod" suddenly melted, causing an arc and interrupting electrolysis. Part of the calcium formed was skimmed out before the bath solidified and the remainder gotten afterwards by loosening the solid  $\text{CaCl}_2$  from the walls of the anode vessel by immersion in  $\text{H}_2\text{O}$  and then breaking the solid chunk to pieces.

It was also noticed in this bath, as well as in all others, that the current had passed through the bath comparatively near the surface and hence the bath was melted to only a shallow depth, while at the bottom of the anode-containing vessel the bath had remained solidified and almost pure white, though the upper portion through which the current had passed for a long time during electrolysis was very dark colored.

Woehler used a mixture of 100 parts of  $\text{CaCl}_2$  and 17 parts of  $\text{CaF}_2$  by weight for his bath, thus getting an electrolyte which

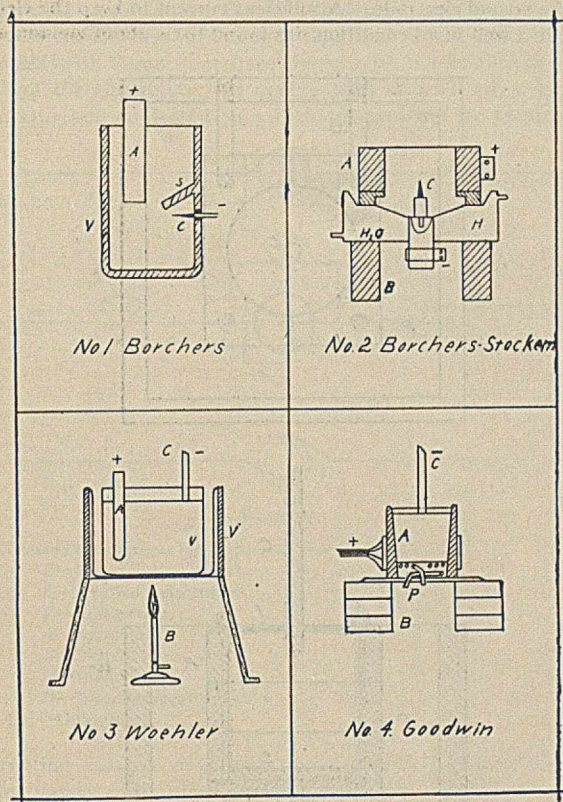


Plate I.

## No. 1. Borchers Apparatus.

- V—Iron container for electrolyte.
- A—Carbon anode.
- C—Iron needle cathode insulated from container.
- S—Shelf under which molten calcium may collect.

## No. 2. Borchers-Stockem Apparatus.

- A—Circular graphite anode.
- C—Iron needle cathode.
- H—Water-cooled bottom insulated from anode.
- B—Brick support.

## No. 3. Woehler Apparatus.

- A—Graphite anode.
- C—Iron cathode.
- V—Iron electrolyzing vessel.
- V'—Sheet-iron jacket.
- B—Bunsen burner for melting bath.

## No. 4. Goodwin Apparatus.

- A—Graphite, circular anode container.
- C—Iron cathode (fastened to stationary upright screw).
- F—Space filled with asbestos-cement.
- B—Brick support.

auxiliary electrode was now inserted directly into the fused  $\text{CaCl}_2$  and the current caused to flow through the bath directly. When sufficient electrolyte was melted the iron cathode  $R$  was lowered into the molten mass by means of the screw ( $R'$ ) and electrolysis begun. After a period of three or four minutes at most the odor of chlorine was noticed, and there was considerable cracking and snapping at the cathode and bursting forth of yellow flames. The current was gradually increased until finally the whole bath was in a state of fusion and 90 to 100 amperes were flowing. After waiting some minutes the iron cathode was cautiously raised from the bath, but nothing was observed until an arc was struck between the end of the rod and the surface of the bath. The rod was again lowered and electrolysis continued for 15 to 20 minutes, at the end of which time bright beads of metallic calcium were noticed to coalesce around the iron rod and finally break away and swim around in the bath

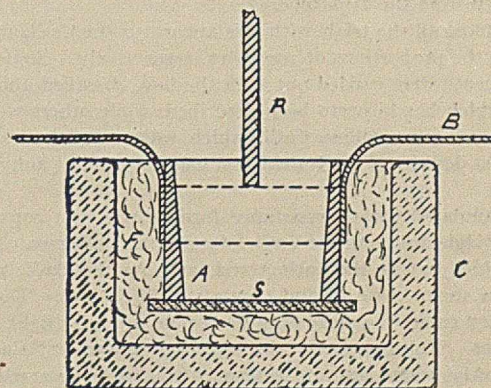
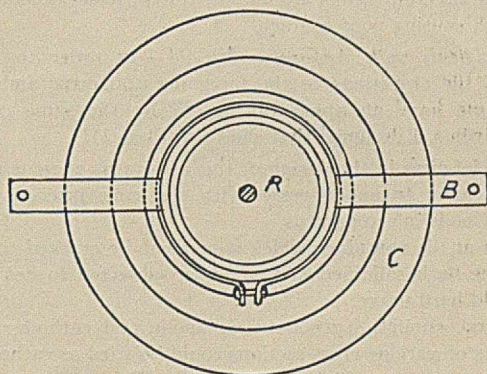


Plate II.

## Electrolyzing Apparatus No. 1.

- A—Circular graphite anode.
- B—Metallic conductor connecting with bands on anode vessel.
- C—Soapstone bottom for anode.
- B—Portland cement vessel.
- F—Space filled with asbestos-cement.
- R—Iron and cathode (fastened to a stationary upright screw).

fused at a much lower temperature. Hence several trials were carried out in the apparatus described, using such an electrolyte. The  $\text{CaCl}_2$  was from the same bottle as that used in the previous trials while the  $\text{CaF}_2$  was ordinary powdered fluorspar.

The two salts were intimately mixed together and then fused down as previously described. After adjusting the current to 40 to 50 amperes it was noticed that a larger portion of the bath was kept in a fluid condition than when  $\text{CaCl}_2$  only was used as electrolyte. Again the melt seemed to be more liquid. Calcium was noticed to form quite rapidly at the cathode but not in a good, compact form. It grew out from the end of the iron rod in a very irregular manner, tending to extend across the anode and short-circuit the bath. Difficulty was experienced in getting a piece even an inch long on the iron cathode, an arc always being struck and melting off all the calcium.

While no attempt will be made to discuss here the chemical phenomena of the baths tried, it may be well to point out the mechanical defects of the apparatus as viewed from an engineering standpoint:

1. The parts of the apparatus (anode container and cathode) are wrongly proportioned to obtain proper cathode current density and still keep the electrolyte in a proper state of fusion.
2. The space intervening between anode and cathode is too small, allowing short circuits to occur.
3. The upper portion of the graphite anode vessel exposed to the air rapidly disintegrates, allowing carbon to fall into the bath.
4. When the bath solidifies the graphite anode container is cracked to pieces.
5. The metallic bands around the anode vessel become badly corroded, causing poor contact.

*Experiments with Apparatus No. II.*—In order to obviate some of the objections to the foregoing apparatus and at the same time have an apparatus practically the same capacity as Goodwin's, I designed that shown in Plate III.

My chief aims in the design of this apparatus were as follows:

1. To have an anode vessel with the same internal dimensions as Goodwin's apparatus.
2. An anode container which would not be cracked to pieces when the bath solidified and could be subjected to heavy currents and hard usage.
3. Large enough space between anode and cathode so that iron rods of various cross-sections could be tried as cathode.
4. An apparatus which would have as little radiating surface as possible.
5. To have the exposed portions of the graphite container protected from the atmosphere.

In almost all the trials with this apparatus the  $\text{CaCl}_2$  and  $\text{CaF}_2$  were in the proportions of 100 to 17, respectively. Some of the experiments were carried out with the pure so-called anhydrous  $\text{CaCl}_2$  which has hitherto been described, while others were performed using crystalline  $\text{CaCl}_2$  which was first dehydrated. I shall first describe a run for calcium using the pure "anhydrous"  $\text{CaCl}_2$ .

A granulated carbon resistance furnace was first constructed and a weighed amount of  $\text{CaCl}_2$  placed in a Battersea clay crucible which sat in the carbon resister. As the  $\text{CaCl}_2$  warmed up, some steam came off and then a very little  $\text{HCl}$ . The whole mass then came to a state of quiet fusion at a very light cherry-red color. Approximately the requisite amount of  $\text{CaF}_2$  was now slowly added and its action in the bath observed. The  $\text{CaF}_2$  seemed to gradually and easily dissolve in the bath. At the same time the bath as a whole grew more and more fluid and limpid. When all of the  $\text{CaF}_2$  had been added the bath seemed to have a cloudy appearance, some portions being rather transparent, while light yellow clouds floated about in other portions. After agitation and maintenance of bath in fused condition for some time it became fairly homogeneous and opaque in appearance.

From the Battersea crucible the fused mass was poured into the electrolyzing apparatus. An alternating current was now passed through the bath, using a carbon rod an inch in diameter as the second electrode. A sufficient current to keep the electrolyte in a well fused condition was found to be about 200 amperes.

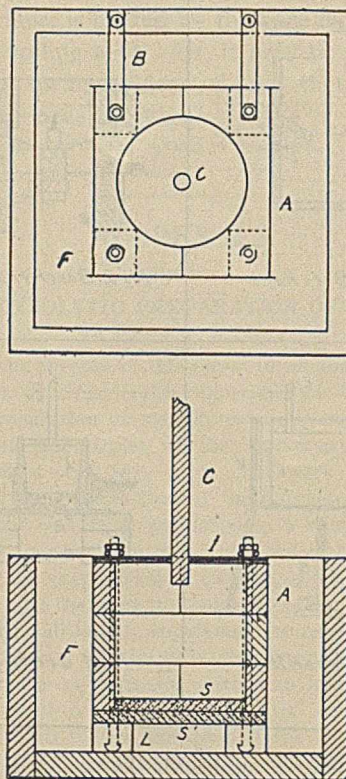


Plate III.

*Electrolyzing Apparatus No. 2.*

- A—Graphite anode built up of six sections, and held together by bolts in corners.  
 W—Wooden case.  
 F—Intermediate space packed with asbestos cement.  
 S—Loose soapstone bottom.  
 S'—Soapstone bottom bolted fast to anode.  
 L—Carbon legs.  
 C—Iron cathode (fastened to stationary upright screw).  
 B—Copper conductors leading to iron covering, I, which is held down on the top of anode by the corner bolts.

At the surface of contact of the large electrode with electrolyte all was quiet excepting perhaps now and then a faint glow. The small electrode, however, was completely enshrouded with first a glowing sheet of flames and outside of this a circle of yellow flames bursting from the surface of the bath. In the space intervening, only now and then appeared a small yellow flame.

The alternating current was now switched off and the direct current switched on after first replacing the small electrode with an iron one 1 centimeter ( $3/8$  inch) in diameter. Chlorine was immediately detected coming off, and after some time calcium was noticed at the cathode, but it was soon in a molten condition and had to be skimmed from the bath. The current was gradually cut down to 75 or 80 amperes before calcium would adhere to the end of the rod in a reguline condition. With this current, however, only about  $1/4$  to  $1/3$  of the bath was fused. A sort of irregular mass of metallic calcium plus adhering  $\text{CaCl}_2$  was built out from the end of the rod, after an hour's run.

The presence of water in the  $\text{CaCl}_2$  has an important influence upon the results attained, and in view of this fact several runs were tried, using  $\text{CaCl}_2$  which had been subjected to dehydration as follows:  $\text{CaCl}_2$  loaded down with water of crystallization

was placed in the Battersea crucible of the electric resistance furnace above described, and gradually "boiled down." When the mass had reached a pasty condition, a considerable volume of HCl came off, and then it soon began to melt down into a cherry-red, pasty fluid. Small bubbles of gas were noticed rising to the surface, exploding with a loud report and bursting in a yellowish flame sometimes tinged with a bluish color.

All of the electrolyte was next poured into the electrolyzing apparatus and subjected to a high temperature by passing an

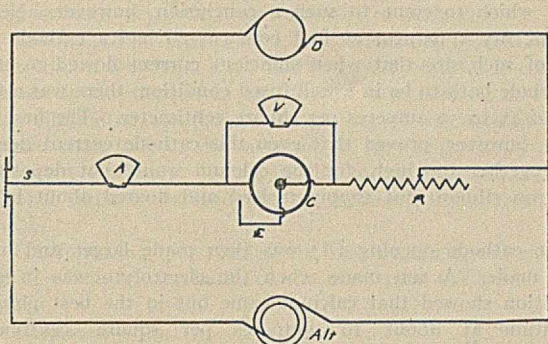


Plate IV.

Showing Connections for Apparatus Nos. 2 and 3.

- C—Electrolyzing vessel.
- E—Auxiliary electrode.
- V—Voltmeter.
- A—Ammeter.
- S—Switch.
- R—Rheostat.
- D—Direct current dynamo.
- Alt.—Alternator.

alternating current of about 200 amperes. A profusion of yellow flames was noticed bursting forth from around the small electrode and the whole surface of the electrolyte became covered with large transparent bubbles, up to 2.5 cm. (1 inch) in radius, of gas (hydrogen) which burst into highly colored flames with a loud report. If the heating be continued these transparent bubbles almost entirely disappear after a time, but no matter how long we continue to heat the electrolyte (or electrolyze) we never get rid of the small yellow flames around the smaller electrode (cathode, if electrolyzing).

Now there are two conditions under which we may electrolyze the above mentioned electrolyte: (a) Electrolyze after all of the gas bubbles (excepting those giving yellow flames) disappear; or (b) electrolyze before the formation of bubbles ceases.

In the first case chlorine is noticed coming off immediately after switching on the direct current. Metallic calcium also appears at the cathode and comes out regularly, if proper conditions of temperature and current are maintained.

On the other hand, if we attempt to electrolyze before the  $\text{CaCl}_2$  is as thoroughly "dehydrated" as possible, that is, before the cessation of gas formation, we get entirely different results. If the bath has been heated little previously, we get almost no chlorine given off at the anode, while at the cathode nothing is deposited if the temperature and cathode current density are high, and if within proper limits, a black, crispy mass, resembling charcoal in appearance, and having seemingly good electrical conductivity, is formed. By long continued electrolysis one may succeed in finally getting a poor reguline deposit, but the bath acts very capriciously at all times.

In a preceding paragraph was set forth the chief aims in the design of apparatus No. 2, with which the above experiments were performed. These experiments have shown the apparatus to fulfil most of the requirements, but before proceeding with the chief objections to this general type of apparatus (Rathenau principle) for use either as a commercial apparatus of for carry-

ing on research work, it is first desirable to give some facts concerning the physical nature of the deposit of calcium and the manner in which it forms in reguline condition.

Of course it will be assumed that the electrolyte is in good working condition chemically and otherwise, all conditions being such as to contribute to the best formation of metallic calcium. My experiments have shown that whether the metal comes out with a coarse crystalline structure (as it usually does), or in a compact, dense form, the deposit cannot form to any great thickness before becoming nodular on the surface, or even growing out in arborescent forms.

From analogy with the nature of deposits from aqueous solution (and also because of the particular design of apparatus used), the above results might be anticipated. In the refining of metals from aqueous solutions by electrolysis it has been found practically impossible to obtain a smooth deposit deeper than 4 cm. (1.5 inches), and in most cases the deposit becomes very nodular and uneven by the time the deposit is 1.25 cm. (1/2 inch) thick.

There is no reason to believe that a reguline deposit from  $\text{CaCl}_2$ , or any other fused electrolyte should come out in a smooth compact form for a practically indefinite thickness than there is in an aqueous solution. A glance at a calcium deposit which has been sawed through will show that these formations have a core of fairly solid metallic calcium. This core is particularly solid near the base of the iron rod, but as we leave the center of this core and approach the outside of the formation we find the metal becoming more and more irregular and mixed mechanically with more or less  $\text{CaCl}_2$ . Still farther from the center of the core the metallic calcium and  $\text{CaCl}_2$  are hopelessly mixed together. (This fact has an important bearing upon the values obtained for current efficiency, as will be shown later.) To be sure, an irregular cathode deposit of considerable length can be formed but there is a greater waste of calcium and  $\text{CaCl}_2$  and hence lower and lower efficiency as the "rod" grows longer, on account of the increased mixing of the electrolyte with the metallic calcium formed. It must be borne in mind that a piece of this deposit, consisting of metallic calcium and  $\text{CaCl}_2$  so intimately mixed mechanically as to appear almost homogeneous, can still be a very good conductor of the electric current and hence may act as a cathode surface for a further deposit of metallic calcium, or a mixture as described, so that a stick of "metallic calcium" such as this is easily obtained.

Again, as intimated above, the particular design of electrolyzing apparatus used is such as to cause the formation of a more or less irregular calcium deposit. When we pass an electric current through this apparatus the current will seek that path which offers the least resistance, and of course we find the lines of current flow as near the surface of the electrolyte as possible. Indeed, as a consequence, the bath remains comparatively shallow in spite of a considerable increase of current. We cannot put the end of an iron rod into the electrolyte to act as cathode without exposing a small portion of the side of the rod, as well as the bottom, to the electrolyte. Consequently more current will enter the edge, or side, of the rod, and hence a tendency for more calcium to deposit around the edge of the rod than directly on the flat end.

Cathode current density has been found to have an important effect upon the structure and nature of the deposit and hence upon current efficiency also. As it is a great objection to this "calcium rod" idea that nothing definite concerning the effects of current density can be determined, because, as the deposit grows the actual cathode surface is continually changing. However, since the current density is continually changing, the conditions under which a reguline deposit is formed are continually changed.

In regard to the use of iron rods of different diameters

starting cathodes, it may be stated that with the apparatus in hand it was found that if a rod much less than 1 cm. ( $3/8$  inch) in diameter was used the current density and hence the heating effect was so great that calcium would not form in the solid condition, while if a much larger rod were used the deposit was very crystalline and irregular and mixed with  $\text{CaCl}_2$  even near the base of the iron rod.

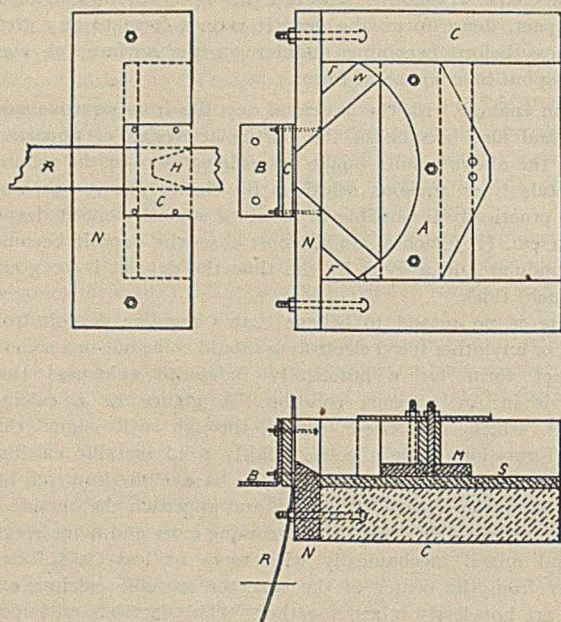


Plate V.

*Electrolyzing Apparatus No. 3.*

- C—Portland cement casing.
- N—Soapstone end.
- S—Soapstone bottom.
- W—Soapstone sides.
- M—Soapstone base for anode.
- A—Graphite anode.
- H—Aperture in soapstone end N.
- R—Iron ribbon as cathode, to slide between soapstone end N and iron block C.
- B—Copper conductor fastened to iron block C.

Connections for apparatus same as for apparatus No. 2.

*Experiments with Apparatus No. III.*—Before taking up the question of current efficiency and in view of the inherent faults in the design of the apparatus used up to this time, I shall describe an electrolyzing apparatus of my own design in which I have endeavored to overcome some of the difficulties.

In the design of this apparatus I have endeavored to attain the following:

1. An apparatus in which the anode and cathode surfaces shall be definite under all conditions.
2. To be able to vary the current density and note the effects upon the electrolysis.
3. To accomplish the removal of the deposit of calcium as fast as formed without interfering with the uniformity of conditions.
4. Construction such that it would stand many "runs."
5. To confine the current to a definite amount of electrolyte, and to an invariable course through the same.

As in the preceding experiments, 100 parts of  $\text{CaCl}_2$  and 17 parts  $\text{CaF}_2$  were fused down in a Battersea crucible by means of a resistance furnace and after being thoroughly mixed were poured into the apparatus. Here, after first being thoroughly heated for some time with an alternating current (a carbon block was attached to the ribbon cathode in the opening H), the direct current was switched on and electrolysis started. As

the metallic calcium deposited to a sufficient thickness the iron ribbon was gradually raised, presenting new cathode surface.

Paul Woehler states that in his experience electrolyzing  $\text{CaCl}_2$  and  $\text{CaF}_2$  by the Rathenau method that a cathode current density of from 45 to 250 amperes per square centimeter can be used and a stick of calcium obtained. He based his figures upon the fact that the amount of current flowing through his apparatus was constant, but the cross-section of the calcium stick varied at different parts. This is obviously a wrong basis upon which to come to such a conclusion, however. Nevertheless, my apparatus at first constructed had a cathode surface of such size that when sufficient current flowed to cause the whole bath to be in a well fused condition, there was a density of 45 to 50 amperes per square centimeter. The first two trials, however, proved that even this cathode current density is altogether too high, for the calcium would not deposit on the iron ribbon, but simply melted and floated about in the bath.

The cathode opening (A) was then made larger and other runs made. A run made when the electrolyte was in good condition showed that calcium came out in the best physical condition at about 10 amperes per square centimeter. To be sure, calcium will come out at a considerable higher current density, but under the particular set of conditions under which these experiments were performed the above current density was found to give the best results. At a lower current density the calcium carries up more  $\text{CaCl}_2$  with it, which tends to solidify just above the surface of the bath and cause the iron ribbon to be cemented fast.

If the calcium does not sufficiently alloy with the iron (which seldom happens), the two will break apart upon cooling some distance above the bath on account of difference in rate of contraction of the two metals. When the calcium does alloy with the iron ribbon, the latter expands at these places, giving rise to little "hills" in the ribbon. Examination of the iron at these points after removal of the calcium shows it to be softer and rotten.

Anode current density is not of so great importance as is cathode current density. We simply need sufficient anode surface over which the  $\text{CaCl}_2$  bath can fuse as far as it likes with the given current in use. If the anode density is unduly great, arcing or glowing phenomena take place at the junction of the carbon and fused bath increasing the total resistance offered by the cell.

*Current Efficiency.*—While the current efficiency at which calcium can be reduced from its chloride is of great importance from an engineering standpoint, yet if the exact conditions under which efficiency is obtained can be maintained are not known, the figures have lost much of their value. Still, although we do not understand fully the nature of the process, to know what efficiency can be attained at times is interesting and encouraging.

Both Goodwin and Woehler made current efficiency determinations, and not only their results, but also their methods of determining them were very different. Goodwin determined the current efficiency for a run of several hours duration, using only  $\text{CaCl}_2$  as electrolyte. He separated the metallic calcium from the  $\text{CaCl}_2$  by pounding loose the encasing  $\text{CaCl}_2$  which had solidified about the "rod" of calcium. The calcium thus mechanically separated was weighed and the efficiency figured. His highest efficiency was 40 per cent.

On the other hand, Woehler, who electrolyzed 100 parts of  $\text{CaCl}_2$  and 17 parts of  $\text{CaF}_2$ , made a mark on the forming calcium rod at the level of the bath, electrolyzed five minutes (gradually raising the rod) and made another mark at the level of the bath. This section was cut out after the run and dissolved in a weak acid solution, the hydrogen collected, and from this the amount of calcium computed. He claims an

efficiency of 82.2 per cent. While Woehler would probably obtain a higher current efficiency than Goodwin by employing a mixed electrolyte, yet the method of Woehler is open to criticism.

Goodwin's method gives us the efficiency for the actually available metallic calcium, while Woehler's efficiencies are too large, because, from my description of the nature of the deposited calcium, much of the metal was in a non-available form; that is, so intimately mixed with  $\text{CaCl}_2$  that it would be almost impossible to separate it mechanically or otherwise.

The method which I used in determining current efficiency was that of Goodwin, but I had to be very careful to completely break up the deposit in order to be sure that all of the  $\text{CaCl}_2$  was eliminated, for the specific gravity of the latter is so much higher than that of the metal as to very appreciably raise the apparent efficiency. Some of the pieces were even boiled in alcohol (anhydrous) in order to facilitate the removal of the  $\text{CaCl}_2$ .

The following four results, the best obtained, are submitted, all of which are for baths of 100 parts pure  $\text{CaCl}_2$  and 17 parts  $\text{CaF}_2$ .

*Apparatus No. 3.*

- No. 1. Current, 76.4 amperes; time, 70 minutes; current efficiency, 67.65 per cent.
- No. 2. Current, 51.5 amperes; time, 57 minutes; current efficiency, 75.6 per cent.
- No. 3. Current, 45 amperes; time, 12 minutes; current efficiency, 84.6 per cent.
- No. 4. Current, 70 amperes; time, 10 minutes; current efficiency, 93.3 per cent.

*Chemical Changes in Electrolyte.*—Above was described the action of the bath during electrolysis, both when water was present and when the bath had been previously dehydrated. If the bath had been thoroughly heated for some time previous to the passage of the direct current, calcium was formed quite readily, other conditions being favorable. Now it will be remembered that the dehydration proceeded with the following successive results after the application of the heat to the  $\text{CaCl}_2 + \text{H}_2\text{O}$ : Firstly, steam came off; secondly,  $\text{HCl}$  fumes, then bubbles of hydrogen appeared all over the bath and lastly only the

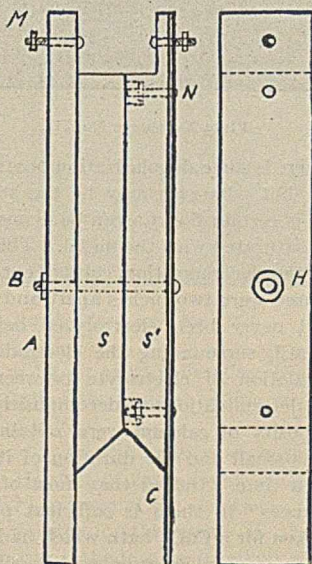


Plate VI.

- A—Graphite anode.  
 C—Iron cathode.  
 H—Large hole through iron cathode.  
 S and S'—Soapstone separators.  
 M—Binding screw.  
 B—Bolt for holding pieces together.  
 N—Bolts for holding iron cathode to support S'.

yellow flames remained. As stated before, these yellow flames were never gotten rid of entirely under any conditions. They exist only in the very hottest part of the bath, *i. e.*, at the cathode if direct current is used, and if alternating current is used only at the smaller electrode. For the most part they appear to be burning hydrogen but no doubt some other substances are present also.

In view of the fact that it is almost impossible to get such data as would give an exact insight into the chemical compo-

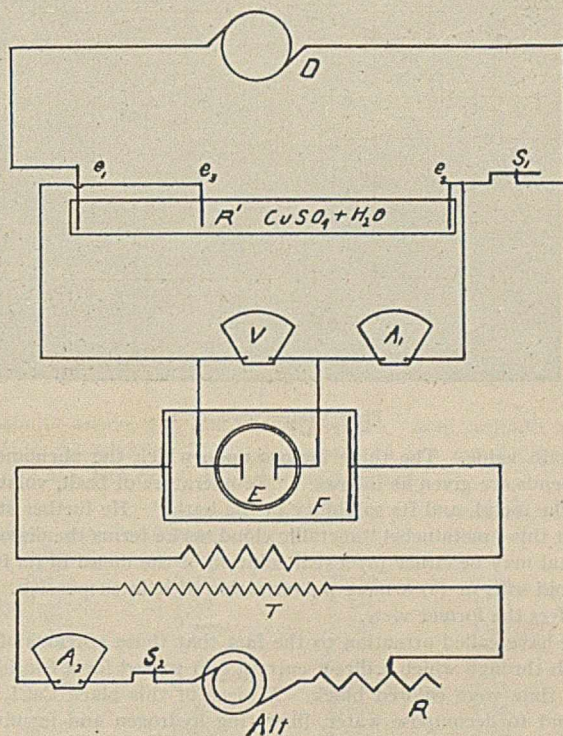


Plate VII.

Showing disposition of apparatus for obtaining decomposition voltage curves.

- E—Electrodes in  $\text{CaCl}_2$  in Battersea crucible.  
 E—Liquid rheostat provided with three electrodes,  $e_1, e_2, e_3$ .  
 S—Switch.  
 D—Direct current dynamo.  
 V—Low-reading Weston voltmeter.  
 A<sub>1</sub>—Low-reading Weston ammeter.  
 T—Step-down transformer.  
 A—Ammeter.  
 S<sub>2</sub>—Switch.  
 Alt.—Alternator.  
 R—Rheostat.

sition of the bath, I can give only my belief as to what actually occurs. And in this I am influenced by the views of Woehler, who has made similar observations. Woehler claims that after the temperature of the bath has reached a certain value the  $\text{H}_2\text{O}$  which still remains begins to combine with the  $\text{CaCl}_2$  according to the following equation:  $\text{CaCl}_2 + \text{H}_2\text{O} = \text{Ca} - \text{OH}; -\text{Cl}$ .

This compound releases water with difficulty, and furthermore during electrolysis would probably react with the metallic calcium to form anhydrous oxychloride and hydrogen. This theory, while worthy of close consideration, does not explain why hydrogen bubbles and the yellow flames come off during the heating with alternating current. Nevertheless, the above equation probably states the condition of affairs somewhere nearly right. So far as the whole truth is concerned, we need much more data of a kind which is very difficult to get.

It is a well known fact, that if in the electrolysis of  $\text{NaCl}$ , the temperature of the electrolyte becomes too high the sodium will

redissolve in the electrolyte to form a hypothetical subchloride, and but little sodium will be obtained no matter how high the current density may be.

Such an assumption has been made by some regarding the electrolysis of  $\text{CaCl}_2$ :  $\text{CaCl}_2 + \text{Ca} = 2\text{CaCl}$ .

Richard Lorenz has shown that lead and zinc will dissolve in their respective chlorides if the temperature is raised to a

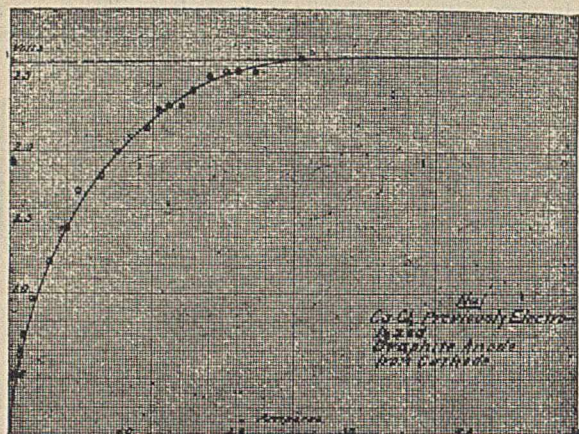


Plate VIII—Curve No. I.

certain value. The three factors upon which the phenomenon depends are given as follows: "Temperature of bath, volatility of the metal, and its solubility in the bath." He further states that this "metanebel," metallic cloud, as he terms the dissolved metal may be either (a) a real solution of the metal in its fused haloid salt, or (b) a mere pulverization of it in suspension. He prefers the former view.

I have called attention to the fact that those portions of the bath through which a direct current had passed for a considerable time were colored black. A piece of this black  $\text{CaCl}_2$  was found to decompose water, liberating hydrogen and forming a white precipitate of  $\text{Ca}(\text{OH})_2$  suspended in a clear solution, but leaving no black residue. This showed that the coloration of the  $\text{CaCl}_2$  was not due to the disintegrated carbon but to either finely divided metallic calcium or the subchloride.

Since  $\text{CaCl}_2$  is soluble in alcohol (anhydrous) and metallic calcium is not, a piece of the solidified electrolyte was placed in the solvent with the result that the  $\text{CaCl}_2$  was dissolved and a finely divided deposit formed at the bottom of the test tube. The alcohol was poured off and some water then poured on the

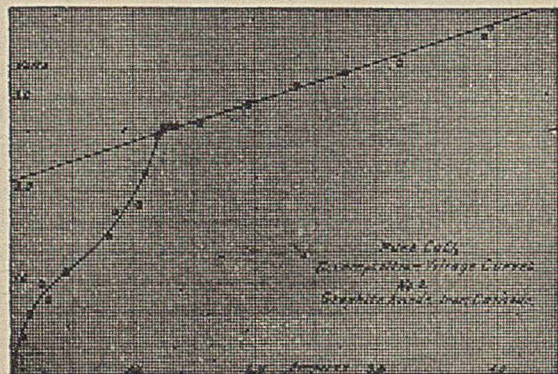


Plate IX—Curve No. II.

precipitate. Hydrogen immediately began coming off quite briskly. A drop of  $\text{H}_2\text{SO}_4$  was next added to the solution and instantly the whole black precipitate was changed to snow-white  $\text{CaSO}_4$ , and the heat developed from the reaction was sufficient to cause the ignition of the hydrogen liberated.

The two experiments just described do not prove absolutely that the dark particles are metallic calcium instead of  $\text{CaCl}$ ; but from the physical qualities, as metallic appearance and specific gravity observed, it was concluded that these particles must exist as finely divided calcium mechanically suspended in the bath when the latter is solidified.

Now from analogy it is very likely that calcium dissolves to some extent in  $\text{CaCl}_2$  at a high temperature the same as lead or zinc have been found by Lorenz to dissolve in their respective chlorides. However, there are grounds to believe that the solution of calcium in  $\text{CaCl}_2$  ought to be regarded as taking place in a manner exactly parallel to the solution of  $\text{CaCl}_2$  in  $\text{H}_2\text{O}$ , rather than to assume that a sub-chloride is formed. As such a compound has never been formed under any condition by any one so as to be recognized as a definite chemical compound, surely there is no reason to state without any qualifications that such a compound is formed in the electrolysis of  $\text{CaCl}_2$ . If we assume that the calcium is really soluble in the  $\text{CaCl}_2$ , while the latter is at a high temperature, as stated, we can also naturally infer that as the  $\text{CaCl}_2$  cools down that the solubility of the calcium in  $\text{CaCl}_2$  decreases and hence crystallizes out as it were.

In discussing the effects of dissolved metallic zinc or lead in their respective chloride baths which are undergoing electrolysis, Lorenz states that the metal acts as a depolarizer, and further, that no metal will be deposited at the cathode until the bath has become saturated with the metal.

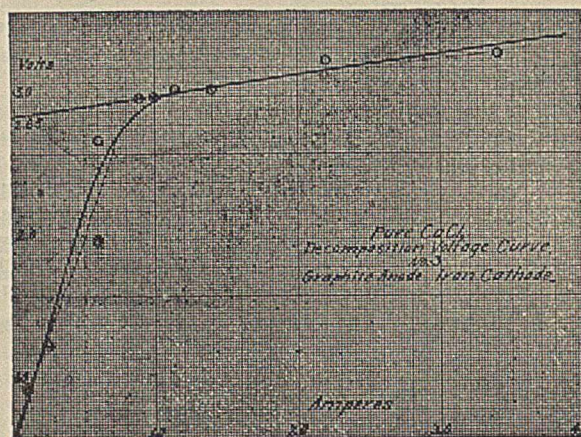


Plate X—Curve No. III.

To be sure, there is some depolarization occasioned in the case of calcium also, but whatever may be the state of affairs for zinc and lead, it is certain that Ca can be deposited from a  $\text{CaCl}_2$  bath before it is saturated with the metal. This fact was proved while obtaining the decomposition voltage curve which follows. The electrodes used were two inches apart and placed in a  $\text{CaCl}_2$  bath which had never been electrolyzed before. There was much molten  $\text{CaCl}_2$  surrounding the electrodes and plenty of chance for circulation of electrolyte between electrodes. In several of these determinations of decomposition voltage, easily recognizable deposits of calcium were obtained, although the current used was small and the duration of its passage only a few minutes at a time. Indeed that most of the curves have well defined "knees" in them is sufficient proof of the fact. Curve II was taken for a  $\text{CaCl}_2$  bath which had metallic calcium suspended in it from previous runs, and it will be noticed that there is no well defined "knee" in the curve.

*Decomposition Voltage Curves.*—As just intimated, the study of the decomposition voltage curves which would be obtained under various conditions would throw much light on the best chemical composition of the bath as well as best physical conditions, correct temperature, etc., at which the electrolysis ought to be carried on for most successful results. Although



prevented by lack of time and the necessary pyrometer for determining temperatures, experiments along this line were not carried on as far as desirable. However, a few results have been obtained which are of at least some value. On Plate V is shown a diagram of the apparatus used, and on page 470 a description and data concerning same.

Details of electrodes for obtaining decomposition voltage curves are shown on Plate VI.

It must be borne in mind that the value of the decomposition voltage for a fused salt will depend not only upon the chemical composition of the electrolyte and its temperature, but also upon the nature of the electrodes used and to a slight extent upon their geometrical disposition with respect to each other.

In the experiments carried out a  $\text{CaCl}_2$  electrolyte only was used and in each case the data taken for the curve when the bath had just reached a nicely molten condition.

Curve No. II was obtained with an anode of graphite and a cathode of polished sheet iron, each 2.5 cm. (1 inch) square and separated 2.5 cm. (1 inch). After the current had been passed and the data obtained the electrodes were removed and examined. The anode, of course, was unchanged; while the cathode

resistance line to the Y axis we obtain 2.62 volts as the decomposition value.

Curve No. IV shows a very steep resistance line, due to the fact that only the tip ends of the electrodes were immersed in the molten electrolyte. The electrolyte was pure  $\text{CaCl}_2$ . Extension of the resistance line shows a critical voltage of about 2.65.

Since in many of the experiments performed calcium seemed to possess the power of forming a superficial alloy with the iron cathode, this fact may help to lower the critical voltage. (Theoretical 3.727.) If we could employ two electrodes which would be entirely inert toward the products of electrolysis and the electrolyte, we might reasonably expect to get results agreeing fairly closely with the theoretical value, if from this we also subtracted the voltage corresponding to the heat supplied to the  $\text{CaCl}_2$  to raise it from the ordinary temperature to the fusion point. This latter step is necessary since the heat of formation is given for solid  $\text{CaCl}_2$ .

However, the above values are probably approximately correct for electrodes of graphite and iron—the two electrode materials which would be used in the electrolysis of  $\text{CaCl}_2$  on an industrial scale.

## DATA FOR DECOMPOSITION VOLTAGE CURVES.

## Curve No. I.

Graphite anode, 2.5 cm. (1 inch) square. Iron cathode, 2.5 cm. (1 inch) square. Electrodes separated 2.5 cm. (1 inch). Bath of  $\text{CaCl}_2$  had previously been electrolyzed.

Volts.	Amperes.	Volts.	Amperes.
0.433	0.05	2.300	1.30
0.560	0.10	2.330	1.40
0.600	0.10	2.330	1.50
0.717	0.10	2.434	1.60
0.966	0.20	2.535	1.75
1.233	0.35	2.567	1.91
1.466	0.50	2.567	2.00
1.734	0.60	2.567	2.15
1.834	0.80	2.660	2.55
2.067	0.95	2.660	2.80
2.167	1.20	2.660	3.20

## DATA FOR DECOMPOSITION VOLTAGE CURVES.

## Curve No. II.

Graphite anode, 2.5 cm. (1 inch) square. Iron cathode, 2.5 cm. (1 inch) square. Electrodes separated 2.5 cm. (1 inch). Pure  $\text{CaCl}_2$  electrolyte.

Volts.	Amperes.	Volts.	Amperes.
0.10	0.025	2.7	1.3
0.40	0.10	2.7	1.35
0.65	0.15	2.725	1.55
0.95	0.25	2.95	1.95
1.1	0.45	3.15	2.35
1.5	0.80	3.30	2.75
1.75	0.85	3.40	3.20
1.85	1.05	3.70	3.90
2.2	1.10	3.80	3.95
2.6	1.20	4.10	4.30
2.7	1.25	4.20	4.50

## DATA FOR DECOMPOSITION VOLTAGE CURVES.

## Curve No. III.

Graphite anode, 2.5 cm. (1 inch) square. Iron cathode, 2.5 cm. (1 inch) square. Electrodes, 2.5 cm. (1 inch) apart. Pure  $\text{CaCl}_2$  bath.

Volts.	Amperes.	Volts.	Amperes.
0.95	0.10	3.05	1.15
1.25	0.25	3.05	1.40
2.00	0.60	3.25	2.20
2.70	0.60	3.30	3.4
3.00	0.90	3.75	4.15
3.00	1.00	3.90	5.00

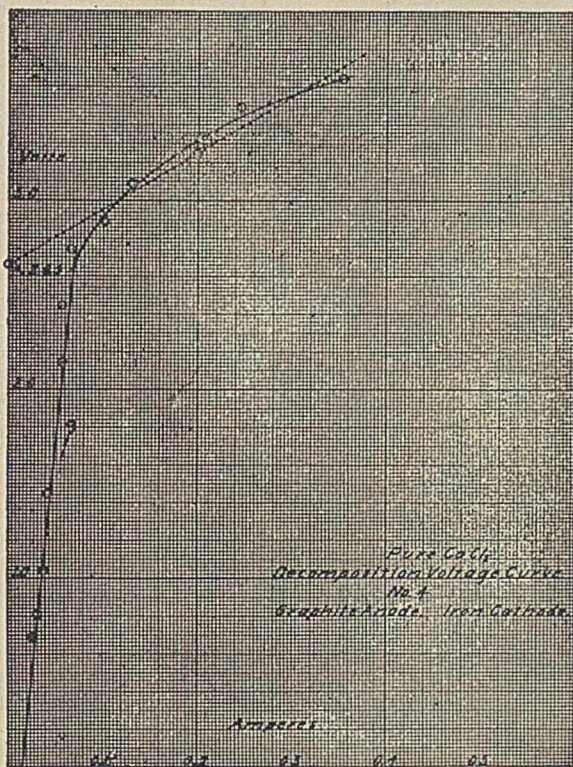


Plate XI—Curve No. IV.

showed no deposit of Ca, or any Ca-Fe alloy, it was appreciably corroded. By extending the resistance line we get 2.04 volts as the decomposition voltage.

Practically the same conditions are maintained in getting data for curve No. III, except for a slight increase of temperature as evidenced by the slope of the resistance line of the latter curve. The critical voltage for this curve is about 2.85 volts. An examination of the cathode after the run showed it to be covered with a thin bright deposit of calcium which decomposed water. After the calcium was dissolved off the iron was left in a soft corroded condition. No doubt an alloy was formed with the iron at its surface.

As previously explained, curve No. I was obtained for a bath which had been previously electrolyzed and contained much metallic calcium in the electrolyte. If we extend the very flat

## DATA FOR DECOMPOSITION VOLTAGE CURVES.

*Curve No. IV.*

Graphite anode and iron cathode. Only tip ends immersed in pure  $\text{CaCl}_2$  bath. Electrodes, 2.5 cm. (1 inch) apart.

Volts.	Amperes.	Volts.	Amperes.
0.30	0.0	2.15	0.05
0.44	0.0	2.45	0.05
0.55	0.0	2.75	0.06
0.70	0.025	2.90	0.10
0.81	0.03	3.10	0.13
1.05	0.03	3.25	0.20
1.10	0.03	3.50	0.24
1.45	0.04	3.65	0.35
1.755	0.04		

TRADE SCHOOL FOR THE LEATHER INDUSTRY.<sup>1</sup>

BY ALLEN ROGERS.

It is with extreme pleasure and gratification that I am permitted the honor of appearing before this joint meeting of the three American societies which stand for the growth, future development and higher aims of our leather industry. The coming together of such a representative body clearly signifies that we have passed beyond the rule of thumb period, realize the importance of technical education, and appreciate the benefit to be derived from coöperation. To-day, therefore, in my opinion, marks the beginning of a new era in our industrial advancement, and one which will go down in the pages of its history.

An attempt to enumerate all of the influences which have tended to bring about this condition of affairs would indeed be a difficult undertaking, but at the same time it might be of interest to note a few of the causes which have aroused us from a state of lethargy, and have made this wonderful scientific art one of the most important manufacturing interests of the country. The first great step in this direction was, no doubt, the introduction of chrome tannage by the Schultz process; this being a purely chemical treatment, paved the way for the application of chemistry along other lines. The second radical change has been the gradual breaking away from bark tannage, with the consequent replacement by liquid and solid extracts. The third advance has been due to the knowledge gained in the manufacture and use of mineral and sulphonated oils, either employed alone or in conjunction with vegetable, animal, and fish oils. The fourth cause should be ascribed to the perfection attained in the manufacture of leather-working machinery. Among the many other causes responsible for this growth should be included the application of chemistry. Indeed, we cannot give too much credit to those who have labored so strenuously to solve the many difficult problems which have constantly confronted them. Let it be borne in mind that the American Leather Chemists' Association, as well as the International Leather Trader Chemists' Association, have done and are still doing a work which should gain for them the everlasting gratitude of the tanner, the currier, the shoe manufacturer, the other users of leather, and the public at large.

And last, but by far not least, should be mentioned the manufacturers themselves. These men of sterling character, who have worked by day and planned by night to bring order out of chaos, who have been to the leather industry what Lavoisier was to the science of chemistry: The organizers and producers of results.

We have come now, however, to the fork in the road, and it remains for us to choose the path. On the one hand is the old beaten trail, full of pitfalls and obstructions, while on the other hand we see a smooth, wide thoroughfare, scientifically constructed, freely oiled with the lubricant of technical skill, and

<sup>1</sup> An address delivered at the meeting of the Leather Chemists' Association, Chicago, October 6, 1910.

with no signs to intimidate those who desire to travel faster than the regulated speed limit.

Which road will be our choice? This question is easily answered when we consider that we are traveling in a modern conveyance, and not with the ox team of our grandfathers. To travel this road, however, we must be skilful in the manipulation of our vehicle, so as to avoid the danger of an upset should we lose control of the steering gear. Equally disastrous would be the result of a rear-end collision should we go too slow. We must, therefore, keep up with the procession or drop out of the race.

As we hesitate in our decision the cloud of doubt slowly rises, exposing to our vision the wonderful truth that others before us have already taken the modern route, and that weeds and bushes are growing in the beaten trail. With the dawning of this fact upon our senses we take new courage, so with a steady hand on the lever and a sharp eye on the road, we start out in pursuit of those who are ahead, trusting that with our knowledge, skill, and energy we will eventually gain the lead.

Thus it is that American manufacturers have been standing at the fork in the road. They have seen the new route, but have hesitated until they were convinced that England, Germany, France and Italy were no longer following the beaten trail. There are many, however, who have realized this condition for several years, and many more also who will not realize the condition for years to come; but let us be thankful that it has been realized and that we are headed in the right direction. Without throwing any bouquets at himself, the speaker has appreciated for some time that eventually we would break away from the old régime. That his dreams might be realized seemed possible when in September, 1905, he was appointed to his present position, and he could attempt the inauguration of a course in tanning which would be to America what some of the foreign schools are to their respective countries. That this attempt has not been futile is evidenced by the fact that twelve of our graduates are at present employed in the leather industry. Yet it has not been all smooth sailing by any means, and there have been times when the undertaking seemed hardly worth the candle. But we are told to cast our bread upon the water, and I feel in this instance at least it has returned again a thousandfold, for there is no doubt that our small beginning had much to do with the recommendations of the committee that the proposed Tanners' Institute be founded at Pratt Institute. It is, therefore, of Pratt Institute, its methods, its aims, its past, its future, and what it hopes to accomplish for the leather industry that I wish to speak.

The founder of this institution, Mr. Charles Pratt, was born of parents in moderate circumstances, and was deprived in his youth of the advantages of an education. As a young man he worked at various trades, and finally became stillman in one of the oil refineries. While engaged in the duties of his occupation he noticed that if a certain fraction of the distillate was separated he obtained the highest grade of illuminating or kerosene oil. Taking advantage of this observation, he started in a small way to put the product on the market, with the result that Pratt's Astral Oil became so widely known that the business rapidly developed into one of vast proportions. In time the Pratt refinery became part of the Standard Oil Company. As prosperity smiled upon him, Mr. Pratt saw the opportunity of realizing his great ambition, and that was to establish a school which should give to others what had been denied him in his own youth. His chief aim was to reach out and help all practical workers, and being a man of wide experience, he fully appreciated the shortcomings of the ordinary schools in this connection. He, therefore, sought for some means whereby young men and young women might be given a liberal education, and at the same time become proficient along some definite line of work. Although he met with but very

little sympathy, he was not shaken in his belief, so in 1887 he founded the institute which bears his family name. At the opening of the school in the fall of 1888 there were no definite plans arranged as to the nature of the courses to be followed, it being his idea to first learn what the students desired, and then endeavor to give it in the most practical and systematic manner. On the opening day there appeared twelve students, each one wanting something different from the rest. They were told that the necessary teaching force would be secured to meet all of their demands, and in a few days the work was undertaken.

It would be too long a story to follow the growth and development of the institution, suffice it to say that since 1888 the enrolment has increased from twelve students to over 4,000, and during that time instruction has been given to over 65,000 individuals. During the early years of its growth Mr. Pratt gave a large part of his personal time and attention to its management, and since his death the work has been carried on by his five sons and one daughter, who have liberally added to its endowment.

The range of subjects covered may be judged from the fact that at present there are offered over sixty distinct courses, many of them day courses and others evening courses. The students enrolled are drawn from nearly every state in the Union, and from a number of foreign countries. It requires nine different buildings to house these classes, and they are taxed to their full capacity.

It is a significant fact that Mr. Pratt did not, in his will, lay down any hard and fast rules for the institute. The trustees have, therefore, made changes in the courses whenever they seemed necessary, but have never changed its purpose, though this is also in their power. It is owing to this elastic condition of affairs, and to the increased demand for young men with technical training, that it was deemed advisable to introduce a course in applied chemistry. Consequently, in September, 1905, the work was undertaken, at which time the speaker accepted the position as instructor in charge of industrial chemistry, and was told to organize his course and equip his laboratory for practical instruction. This may seem a very simple proposition, but rest assured it was no small task. Being an entirely new departure in this method of teaching the subject, there was no school which could be visited in order to gain information. It was, therefore, largely a matter of originality, although our simple creed, as outlined by the founder, was constantly before us. That simple creed is:

Show men how to do something, and then insist that they do it as well as honestly, as economically as it is possible to be done.

Show men why certain combinations produce the desired results, see that they understand the simple principles and apply them to their work.

Consequently, in order to teach young men the methods employed in the manufacture of commercial products, it is necessary to bring them in as close relationship as possible with the actual working conditions. This method shows them the how. The why is gained by class-room instruction. To carry this idea into practical operation, we started out with five miniature factories, equipped in such a manner as to be able to manufacture on a fairly large scale, each plant being a unit by itself and worked independently of the others. They are as follows: Chemical works, soap works, tannery, dry color and paint works and dye works.

What the institute desires is to meet the needs of the large number of young men who cannot afford the time or money for a four-year course with college requirements, and hence is made but two years in length. It is designed especially to give the necessary technical knowledge and practical training to those who wish to become foremen, superintendents or heads of

departments in the important chemical industries. We are not, as some suppose, a trade school; that is, we do not claim to graduate men who are fitted to do only one kind of work. On the other hand, we are far removed from the ordinary technical school or university. Although we may teach the same or similar subjects as given in trade or technical schools, we approach them in an entirely different manner, while the attitude of our students toward their work is also quite different. Our field, therefore, may be said to lie half way between the two.

The method of selecting our students is one which is characteristic of Pratt, and may be of interest to mention at this time. On appearing for examination the young man is given a personal interview by the instructor in charge of the department. This interview constitutes the major part of the examination, as by it we judge his mental and physical ability to handle the course. He is then given a test in English and arithmetic. This you will note is quite a bit different from college requirements. Having been admitted to the course, his first year is devoted to the study of general chemistry, qualitative analysis and quantitative analysis, together with the necessary amount of physics, mathematics and mechanical training. The last two subjects extend over two years and include mechanics, strength of materials, steam and power transmission, pattern-making, forge, foundry, machine work and design.

In the second year of the course the student is brought into contact with commercial processes, the major part of his time being devoted to industrial chemistry and technical analysis. During the fall term one hour each day is given over to class-room instruction in inorganic industrial chemistry, by means of which he becomes familiar with the details involved in the various branches of industry. Not only is he made familiar with the processes and relationship which exist between them, but is shown where chemical knowledge and control are very beneficial, and often absolutely essential. In the winter term the class-room instruction covers general organic chemistry, while the spring term is devoted to organic industrial chemistry. On completing each industry one or more trips are made to such factories as best illustrate the points which have been studied, thus more firmly fixing the knowledge gained by class-room instruction.

It is on our laboratory work, however, that we place special emphasis, as it is through this method of training that we hope to prepare young men to fill the positions mentioned above. At the beginning of the school year one man is assigned as foreman of the chemical works, and is given four of his classmates as assistants. He is instructed in this assignment to make an estimate for the manufacture of some chemical; to see that his machinery is put in perfect condition; to inspect his shafting, belts and motors; to see that everything is in order, and that his factory is kept clean. In order to make his estimates in an intelligent manner he has all raw materials tested out by the laboratory force, takes into consideration the cost of raw materials, freight f. o. b. New York, wear and tear of machinery, interest on investment and cost of containers. All of these details are taken into account before he attempts to make the product, for he must be satisfied that it can be made at a profit. Having determined that it is a profitable operation, he endeavors to produce as much high-grade material as possible. Although the student may be called upon in after-life to manufacture chemicals, the training in this plant is very beneficial, as it teaches him to handle the steam-jacketed kettle, vacuum pan, vacuum filter, vacuum pump, filter press, and centrifugal machine. Having spent one week at this kind of work, the entire crew are transferred to analytical problems, and a new gang takes their place.

When the chemical works have been in operation for about three weeks the first gang are assigned to the soap factory, the second in line becoming foremen. Here the same idea is

carried out, with the result that the students become familiar with the various processes employed in the manufacture of soap. Starting out with the raw materials they follow the numerous operations even to that of wrapping, boxing and shipping. During the past year we made over thirty-five hundred pounds of toilet soap in our miniature factory. Next in order comes our miniature paint factory, in which during the past year we made about four hundred gallons of ready-mixed paint. In this connection I might add that owing to coöperation with the American Society of Testing Materials we have arranged to grind one hundred and sixty different kinds of white paints during the coming year, all of which will be tested out on fences erected for the purpose. To carry out this work and increase our efficiency we have added two twenty-inch burr-stone mills, a roller mill and post mixer, making an equipment with a maximum capacity of one hundred gallons per day.

In our miniature tannery the same procedure holds good as for the chemical, soap and paint works. The equipment for this plant consists of three pits, one paddle, one drum, a union splitter, one set of buffing wheels, rolling and glazing jack, slating table, and beams, with the necessary tools for hand work. During the past year we tanned 350 sheepskins, 45 calfskins and several hides. What we plan to add to this equipment I will mention later.

Having given a partial outline of our methods as they now exist, before taking up what the future may have in store for us, I would like to mention a few facts which show the phenomenal growth of this department. In the fall of 1905, when the course was started, there were eight men who applied for chemistry. By taking men who had been refused admittance to other courses, the class was finally made up to twenty-seven. In 1906 about fifteen applied for chemistry, and that year the class was made up to thirty. In 1907 there were twenty-five applied for chemistry. In 1908 about the same number applied as the previous year. In 1909 forty applied for the course, and the class was made up with thirty-five of them. This year there were fifty-one applied for the course in chemistry, of which we took thirty-three, making the class up to our limit of thirty-five, with two men who were required to repeat the first year. I think that you will agree with me that this is quite a rapid growth. Out of the twenty-seven admitted to the first class we graduated sixteen. Of the thirty in the second class we graduated seventeen. The following class graduated eighteen. Last June we graduated twenty-three, while next June we expect to graduate twenty-three. These figures are interesting, as they tend to prove the old adage, "You can lead a horse to water, but you can't make him drink." That is to say, we admitted men to our first three classes who were not desirous of taking the course, with the consequent result that our mortality was very great. That we have gained somewhat of a reputation in the past five years, and that our graduates as a rule have made good, is indicated from the fact that of the twenty-three men who finished the course last June there were twenty-one placed in good positions before graduation. Of the seventy-four graduates and eight special students who have taken this course, there are seventy-eight engaged at present along chemical lines. Those of you who have read the report of the Committee on the Tanners' Institute are aware that after considering the advantages of several institutions of learning, all agree on Pratt Institute as being the most desirable school for the kind of work to be undertaken.

Although we have not decided upon the details of the courses to be offered, I feel justified in presenting a few tentative suggestions, and trust that there may be many valuable points brought up in the discussions which I hope will follow. From our experience in the past, it is safe to assume that there will be three types of men to apply for this work. The first class

will be those who are at present engaged in the industry and want to obtain some knowledge of the theoretical and technical side of the subject. The second class will be those who are graduates from technical schools and universities, who desire to know something of the practical operations involved in the manufacture of leather. The third class will be those who have taken our two-year course in Applied Chemistry, and wish to spend an additional year in a more advanced study of the practical, technical and theoretical problems. There is also another class of men who will want to follow some special line, such as coloring beamhouse practice, chrome tannage, etc. This last type, however, presents a difficult problem, and one which I think at present would be almost impossible to handle. It therefore seems evident that we must present two courses—one in which the principal feature is theory, and the other in which the principal feature is practice. We must consequently start out with a definite object in view, and so organize the work that it will do the greatest good to the greatest number, and above all we must have system. The courses which it would be most natural for Pratt Institute to offer would be those in which the practical and theoretical are so blended as to fill the needs of the above-mentioned classes. It would thus appear wise to offer two courses, each being one year in length.

Course A should be designed for men who have already been employed in the leather industry. What these men want is a broader knowledge of the practice of tanning, together with an idea of the most important scientific principles upon which the industry rests. Some of the subjects which should be included in this course would be the following:

- (a) Elementary principles of chemistry as related to the tanning industry.
- (b) Chemical laboratory practice in conjunction with the above.
- (c) Lecture course in industrial chemistry covering such industries as are closely related to tanning.
- (d) Laboratory practice in industrial chemistry covering such subjects as soap-making, boiling of oils, dyeing, etc.
- (e) Technical analysis, such as water, coal, oils, fat, egg yolk, extracts, barks, etc.
- (f) The principle and practice of tanning, using an improved text on the subject.
- (g) Practical tanning and finishing in school tannery.
- (h) Brief course in mechanical drawing.
- (i) Power plant operation, and mechanical transmission of power.

Course B should be designed for men who have graduated from a four-year chemical course, from our two-year day course in applied chemistry, and for other men who are properly prepared. Some of the subjects which might be included in the course would be the following:

- (a) The principles and practice of tanning, using an approved text.
- (b) Practical tanning and finishing in school tannery.
- (c) Technical analysis, including such substances as water, oil, grease, fat, wax, egg yolk, extracts and barks.
- (d) Chemical research. This would include the investigation of such problems as constantly arise, and the publication of the same from time to time.
- (e) Industrial chemistry: A lecture course covering such industries as are closely related to tanning.
- (f) Industrial laboratory practice, including the manufacture, use, and testing of dyestuffs, soap-making, oil-boiling preparation of varnishes, finishes, etc.
- (g) Problems in the design and construction of tanneries, with arrangement of power plants and equipment.
- (h) Power plant operation, including electrical and mechanical transmission.

For the benefit of all students there should be conducted during the year a series of lectures, which should be given by men who are specialists in their line of work.

The equipment required for the two courses would, of course, be identical, and much of the practical instruction for both groups would be the same. Each group of men would profit by the close association with the other. The men from the factory would learn much from the men of scientific training, while the men with theoretical knowledge would become broader by working shoulder to shoulder with men of practical experience. In the technical and theoretical portion of the work, however, the courses should be kept entirely separate and distinct.

The plans as just outlined, therefore, offer three courses of different length:

(I) A one-year course for those who have had practical experience, and cannot afford the time or money for the longer courses.

(II) A three-year course, consisting of two years in our day course in applied chemistry, and one year in the special course in tanning.

(III) A five-year course, consisting of four years in some technical school or university, and one year in the special course in tanning.

In order to carry out the plans for this course we have been engaged, during the summer, in making extensive alterations in our chemistry building. We have provided for about six times as much floor space as originally occupied by our school tannery, have greatly enlarged our analytical laboratory, and added materially to our other industrial plants. To facilitate the instruction in tanning we have planned for the installation of seven more pits, an 8-foot drum, a 4-foot paddle, fleshing machine, shaving machine, staking machine, a new finishing room and two drying rooms.

In order to properly carry on the work, it will be necessary for us to increase our teaching force, which would require at least two more men. One of these men should be a high-grade tanner and finisher who has had practical experience in all of the different branches of the industry. The other man should be a well-informed chemist who has had both teaching and practical experience. This addition with our present staff of five in the chemical department would amply provide for the instruction which would be necessary. In the School of Science and Technology, of which the Tanning Course would be a part, we have nearly fifty instructors, so that there would be no difficulty in the other departments.

To permanently provide for the annual expenses of these additional salaries, to cover the operating expenses, the maintenance of equipment, the heat, light, janitor service, etc., an endowment of something over \$100,000 would be necessary on the part of Pratt Institute.

That the Institute is deeply interested in this project is evidenced from its proposition to the Tanners' Association. That it is not from any financial consideration which might accrue is also apparent. We are taking it up because we believe that the tanners are interested, and that it is a field in which we hope to be of service.

That the tanners are very much interested in the founding of a school where young men may be trained along the proper lines needs no proof. The establishment of a fund for the promotion of research, for scholarship, for publications and other uses will be of the greatest benefit to the advancement of the industry. Above everything else, however, we must have coöperation, encouragement, and support. Let me impress upon the National Association of Tanners, the American Leather Chemists' Association, and the Extract Manufacturers' Association, as well as the manufacturers of dyestuffs, chemicals, machinery, oils and specialties that we need your help. For

if we are to succeed we must join our forces so as to work in unison and harmony to achieve the one great end.

ALLEN ROGERS.

### CAST COPPER OF HIGH ELECTRICAL CONDUCTIVITY.<sup>1</sup>

By DR. E. WEINTRAUB.

The production in the foundry of pure copper castings mechanically sound and possessing an electrical conductivity comparable to that of pure forged copper has been an important problem ever since the development of electrical machinery and apparatus. The literature as well as the patent files are replete with descriptions of methods for the accomplishment of this result. Secret compositions, warranted to give high conductivity copper, were and are being sold to foundrymen, and there are but few foundry foremen to be met with who do not claim to have some time or other obtained the desired result by adding "phosphorus" or similar material. Obstinate, however, the casting of high conductivity copper refused to make its entrance in the foundries and we may assume that there were good reasons for it.

The cause of the difficulty of producing sound pure copper castings has been sufficiently well understood for a long time. Molten copper has the property of dissolving gases such as oxygen and of setting a part of these gases free on cooling. This produces pin-holes and even big cavities. The casting obtained is therefore mechanically unsound and has naturally a low electrical conductivity.

The elimination of these dissolved gases presents but little difficulty. It is sufficient to add one of the well-known deoxidizers such as zinc, magnesium, phosphorus, etc., in small quantities to bind the oxygen chemically. Copper castings free from blow-holes are thus easily produced. The electrical conductivity of the copper thus produced is, however, as a rule, low; and this because all the *deoxidizers used had also the property of combining with copper*, and the remarkable rate at which the conductivity of copper drops when small amounts of impurities are alloyed with it is well known. The amount of oxygen dissolved in copper during the process of melting is a variable quantity and is distributed throughout the whole mass. It is therefore practically impossible to add the deoxidizer in such a way as to eliminate oxygen completely without, at the same time alloying it to a certain degree with the copper. Accordingly the conductivity of the resultant product was a matter of accident. In the laboratory sometimes good results were obtained but they were hard to duplicate and the transfer to the foundry was always a disastrous experiment.

It seemed to me that what was needed for the solution of the problem was the finding of a deoxidizer which would have no affinity for copper (at least at the melting point of the latter) and which, therefore, could be added in excess with impunity.

While carrying on the work of the isolation of the pure element boron, and its fusion, the lack of affinity of this element for copper has forcibly impressed itself on my mind. Boron has, at the melting point of copper, a great affinity for oxygen, nitrogen, etc., and it occurred to me, therefore, that boron ought to be the ideal substance for deoxidizing copper.

Pure boron as prepared by methods described by me in paragraphs 2 and 4 of my article<sup>2</sup> can, by this time, be produced in large quantities and is rather inexpensive for uses based on its electrical properties. For casting copper, however, its price would probably be too high.

In the course of the investigation mentioned I had prepared, however, an amorphous powder which contained as essential impurity only a certain percentage of oxygen and which I termed "boron suboxide." This substance is prepared by a

<sup>1</sup> An address read before the Chicago Meeting, American Electrochemical Society, October 13, 1910.

<sup>2</sup> *Trans. Am. Electrochem. Soc.*, **16**, 165.

modification of the reduction-process of boric anhydride by magnesium. Obviously for the purpose of eliminating oxygen from copper without introducing any impurity capable of alloying with copper, this boron suboxide would be as good as pure boron or perhaps better on account of its being obtained in form of a very fine powder.

This substance was used in the first experiment. The very first attempt was successful. A perfectly sound bar was obtained with a conductivity of 94 per cent. 0.1 per cent. of the weight of the copper was added. Further experiments showed that the amount of boron suboxide could be reduced to  $\frac{1}{30}$  per cent. and also that the addition of a large excess, say of 1 per cent., did not diminish the conductivity or change in any way the mechanical properties of the cast copper.

Here was, therefore, a method which could be put into the hands of any foundry man and which would always give good results provided the copper (ingots or scrap) melted down was pure metal.

A further simplification could, however, be introduced into the process. The reduction of boric anhydride by magnesium, the first being in excess, gives a product containing magnesium borate, boric anhydride and boron suboxide. While the last one is the active agent, the first two are harmless. In fact, the presence of boric anhydride and magnesium borate is even an advantage as the addition of a larger mass of material makes the operation easier; also the boric anhydride would probably dissolve the copper oxide and bring it to the surface as copper borate. It is therefore unnecessary to submit the direct product of the reaction to chemical treatment for the purpose of isolating the boron suboxide. It is sufficient to grind up the mixture into coarse grains and use an amount of it which contains the necessary amount of boron suboxide.

The foundry practice at present is to add 1 per cent. to  $1\frac{1}{2}$  per cent. of this material which is equivalent to 0.08 to 0.1 per cent. of boron suboxide.

The properties of the material obtained are as follows:

The electrical conductivity obtained can be as high as 97 $\frac{1}{2}$  per cent. if the copper melted down is perfectly pure. In the foundry, where the scrap copper which is melted down cannot always be very carefully selected, a conductivity of 91 to 95 per cent. is obtained and a guarantee of 90 per cent. is at present given.

The resultant casting is readily machined; fears which were entertained on this subject did not materialize and the engineers using the material have not complained on this account.

The mechanical properties are as follows:

Tensile strength.	Elastic limit.	Elongation.	Reduction in area.
24350	11450	48.5%	74.49%

Castings are being made of all shapes and sizes with no more difficulty than in the case of brass. Sand and iron moulds can be used and for standard articles the latter are preferred.

The use of cast copper lies in two different directions: 1. Replacing forged copper. In this case a great saving in cost is often obtained. (2) Replacing alloys of copper. The gain in conductivity allows the reduction in bulk and thus a saving in material.

The use of this material is spreading as the engineers get better acquainted with it and is already of very considerable magnitude. It is interesting to note that in spite of the fact that cast copper has lower conductivity than forged copper, in many cases, as for instance, in case of current transformers, the effect of higher conductivity is obtained by substituting it for forged copper. This is due to the fact that in casting a chance is given to eliminate riveted, screwed or soldered joints whereby a number of poor contacts are avoided. Not only this but the elimination of joints often insures a better operation.

The cost of the boron added is small; at the present prices

of magnesium and boric anhydride the cost per pound of copper is less than 0.73 of a cent.

The success with "boronizing" copper, as it is now called in the foundry, suggests new lines of work which I have taken up lately.

Among these I mention:

First, the addition of boron to different copper alloys.

Second, the replacing of the poling operation in the process of refining copper by a "boronizing" operation.

In conclusion, I want to express my appreciation of the excellent work done by my assistant, F. A. Kroner, without whose energy and foundry experience the technical and commercial success of the process described would have been greatly delayed.

CONTRIBUTION FROM THE RESEARCH  
LABORATORY, GEN. ELEC. CO.,  
WEST LYNN, MASS.

## NOTES AND CORRESPONDENCE.

### BAKELITE AND RESINIT.

*Editor Journal of Industrial and Engineering Chemistry:*

On page 441, you publish a consular note about Resinit Compound. For the information of your readers, I beg to mention that the use of sodium sulphite, ammonium sulphite, tertiary sodium phosphate, sodium acetate, and all bases or basic salts, with which these Resinit compounds are made, as well as the impregnation of wood and other cellular or fibrous materials like infusorial earth, starch, etc., is broadly covered by my United States Bakelite patents:

- No. 939,966, filed Jan. 28, 1909. Issued Nov. 16, 1909.
- No. 941,605, filed Feb. 1, 1909. Issued Nov. 30, 1909.
- No. 942,699, filed July 13, 1907. Issued Dec. 7, 1909.
- No. 942,700, filed Dec. 4, 1907. Issued Dec. 7, 1909.
- No. 942,808, filed Oct. 26, 1907. Issued Dec. 7, 1909.
- No. 942,809, filed Oct. 15, 1907. Issued Dec. 7, 1909.
- No. 942,852, filed July 13, 1907. Issued Dec. 7, 1909.
- No. 949,671, filed Feb. 18, 1907. Issued Feb. 15, 1910.
- No. 954,666, filed Oct. 15, 1907. Issued Apr. 12, 1910.
- No. 957,137, filed Oct. 8, 1909. Issued May 3, 1910.

Several other patents are pending here and abroad, and will be published later on.

For further information, I may tell you that the former manufacturers of Resinit have discontinued the manufacture of this article, and have purchased an interest in the Bakelite Gesellschaft, m. b. H., in Berlin, who are the owners of my European patents, and who, for certain purposes, will supply also Resinit compounds.

On this occasion, permit me to state that the first public mention of Resinit was made in Heidelberg, about one year after my French patents were published, and several months after I had read my paper on Bakelite before the American Chemical Society, and after it had appeared in this Journal, and translations thereof had been published in Germany and other foreign countries.

I am pleased to state that since a few days, my United States, Canadian, Mexican and Japanese patents have been acquired by the General Bakelite Company, 100 William Street, New York City, and that henceforth, Bakelite and the raw materials thereof will be obtainable from that source.

Respectfully yours,

L. H. BAKELAND.

### TRIBO-LUMINESCENCE.<sup>1</sup>

The term "Luminescence" has been applied to the light which is emitted by various substances at a temperature much lower

<sup>1</sup> Read at Chicago Meeting of Am. Electrochem. Soc., October 13-15, 1910.

than that which is necessary to show visible incandescence, as illustrated for example in the light of the firefly, decaying wood, and many natural and artificial mineral compounds.

The general phenomenon of luminescence has been divided into several classes by Dr. Sylvanus P. Thompson, as shown in the following tabulation which is copied from his book entitled "Light Visible and Invisible," page 175 (1893).

Phenomenon.	Substance in which it occurs.
1. Chemi-luminescence.....	Phosphorus oxidizing in moist air; decaying wood; decaying fish; glow-worm; firefly; marine organisms, etc.
2. Photo-luminescence	
(a) Transient: Fluorescence....	Fluor-spar; uranium-glass; quinine; scheelite; platino-cyanides of various bases; eosin and many coal-tar products.
(b) Persistent: Phosphorescence..	Bologna-stone; Canton's phosphorus and other sulphides of alkaline earths; some diamonds; etc.
3. Thermo-luminescence.....	Fluor-spar; scheelite.
4. Tribo-luminescence.....	Diamonds; sugar, quartz; uranyl nitrate; pentadecyl-paratolyketone.
5. Electro-luminescence	
(a) Effluvio-luminescence.....	Many rarefied gases; many of the fluorescent and phosphorescent bodies.
(b) Kathodo-luminescence.....	Rubies; glass; diamonds; many gems and minerals.
6. Crystallo-luminescence.....	Arsenious acid.
7. Lyo-luminescence.....	Sub-chlorides of alkali metals.
8. X-luminescence.....	Platinocyanides; scheelite; etc.

Tribo-luminescence, to which the present remarks particularly refer, is the property possessed by certain bodies of emitting light when rubbed or scratched.

A number of natural mineral exhibit different degrees of this property, an interesting investigation of which is described in a paper by Mr. Wallace Gould Levison, published in *Science*, N. S., Vol. XIX, No. 491, pp. 826, 827.

A simple but rather faint illustration of the phenomenon may be easily produced by rubbing together two lumps of white sugar in the dark.

While experimenting in the production of fluorescent and phosphorescent materials several years ago, the writer accidentally discovered that one of his preparations exhibited tribo-luminescent properties, and two or three subsequent trials led to improved results. As an easily prepared artificial product of this kind does not appear to be generally known, some brief directions for making it may be of possible interest.

The following mixture was selected as the best after a series of experiments.

Zinc carbonate (C. P.).....	70 parts by wt.
Flour sulfur.....	30 parts by wt.
Manganese sulfate.....	trace.

The zinc carbonate in fine powder should first be mixed with the flour sulphur. Then a small particle of manganese sulphate should be dissolved in distilled water and enough of the solution added to the powdered materials to make a thick cream. After being thoroughly triturated in a mortar, the mixture can be poured into a shallow glass or earthenware dish and allowed to dry at a gentle heat. When quite dry it should be again reduced to a fine powder, and packed *hard* into a porcelain or Battersea crucible with a tight cover, and subjected to a bright red heat for twenty minutes. The mixture shrinks considerably and sinters together into a stone-like mass, which possesses the peculiar property of tribo-luminescence to a marked degree, so that when scratched with a knife blade or sharp point, it emits a train of minute sparks showing a yellowish light. This light appears to have little or no heating effect as it will not ignite any inflammable vapor or gas, so that in this

regard, it differs essentially from the light and sparks produced by striking together flint and steel, or the scratching of a pyrophoric alloy of iron and cerium.

W. S. ANDREWS.

#### RAPID SAPONIFICATION OF FATS FOR TITRE DETERMINATION.

There have been many detailed methods proposed for the rapid and complete saponification of fats for titre work, all of which divide themselves into two classes, one the water-caustic method and the other the alcoholic-caustic method.

The water-caustic saponification is slow and liable to be incomplete unless well stirred. Where strong caustic is used over a flame, there are the objections of scorching the soap.

In alcoholic saponification, we have the expense, the danger of fire and necessity of expelling all alcohol. It has been decided to try out some less volatile solvent. Glycerine has been used for saponification, but never for titres. The method proved so rapid, easy and complete, that it was thought advisable to give a detailed description of same.

Experiments were tried with C. P. Glycerine (95 per cent.). This was found not to "take hold" of the stock well, especially when the fat was neutral. Also some tedious foaming occurred and the 5 per cent. of water had to be expelled to get saponification to start. H. G. glycerine was substituted. The following gives the details of method as finally adopted.

About 120 grams of H. G. glycerine are placed into a 6½" porcelain casserole, a stick and a half or about 25 grams of caustic potash added and the whole heated over a flame. The potash dissolves very easily; now the fat is added (100 grams) and stirred while heating over flame. In a minute the fat dissolves in the caustic. Slight foaming occurs in the case of pure and refined fats. Heating is continued until a quiescence appears, shown when caustic solution and fat become a homogeneous mixture, indicating complete saponification. This point is not indicated so well by low-grade fats. In this case 10 to 15 minutes' heating was always found to complete same. Now the melt is taken off the flame and about 15-30 cc. cold water added, bit by bit, from a wash bottle, allowing each foaming to subside. Dilute acid is added to break up the fatty acids. The fatty acids precipitate in a milky form so that it only takes slight heating to coalesce them into a layer.

It is important not to get too much water into the melt after saponification, as this would hydrate the soap and when the acid is added, it would take much longer to boil out same so as to get clear fatty acids. At times we have been able to add just enough water so that when acid was added, the heat generated did the boiling out and fatty acids were clear. Insufficiency of water will cause the formation of too much heat when acid is added to the melt. The acid must be added from the cylinder in small quantities, stirring in each addition. Now a larger quantity of hot water is added and the whole placed on steam bath ready for washing. The whole operation takes less time than it does to write it. We have completed the operation of saponification and breaking up soap in ten minutes on a single sample or in one hour and forty minutes on nine samples of high-grade stock.

There is no long and tedious stirring of semi-solid soaps or prolonged dissolving of same in water and no long boiling out as in water and alcoholic methods. The method requires some experience and care in the case of low-grade stocks.

It has been tried out thoroughly against other methods of saponification and found to hold good concordance. The cost of the glycerine is at most three and a half to four cents per titer. The saponification is rapid and complete and the breaking up of the soap likewise rapid. It requires a little experience to strike the right point of dilution of the melt, but this is soon acquired by the operator. It is excellent where factory con-

trol requires rapid work. The method has been checked against the other standard methods by Mr. W. C. Marckworth and Mr. H. C. Knight, 1st and 2nd assistants. CHAS. V. ZOUL.

THE PROCTER AND GAMBLE LABORATORY,  
IVORYDALE, OHIO

#### IMPROVEMENT OF THE WILEY METHOD FOR DETERMINING THE MELTING POINTS OF FATS.

During the course of an extended investigation on the chemical and physical properties of animal fats the writer had occasion to use the Wiley method for determining the melting point of fats. This exceedingly ingenious method was found difficult of manipulation under all but perfect conditions. However, with a few modifications the operation may be considerably improved.

As is well known to all who have had experience with this method the main difficulty is encountered in securing fat disks perfectly free from occluded air. During the course of the determination, the smallest trace of air is sufficient, by its expansion on heating, to carry the fat disk to the surface of the liquid and thus ruin the determination. With high-melting fats, as tallows, the difficulty is especially great on account of the great change in temperature and therefore great expansion of the air. It is true that the number of air bubbles may be appreciably decreased by preparing the fat disks on ice floating in cold, recently boiled, distilled water. If the water is not near the freezing point, some of the ice will melt and thus introduce air into the water. Some ice used was found to be so full of occluded air that even with these precautions fat disks prepared on it invariably were found to be useless for a melting point determination.

With a view of correcting these difficulties the writer prepared fat disks by dropping the melted fat on cold mercury. By proper adjustment of the height from which the fat is dropped, together with proper control of the temperature of the fat and mercury, disks of very regular shape were obtained. When thoroughly cool and hardened, they were removed with a cold steel spatula and thrown into a beaker containing cold, dilute (50 per cent.) alcohol. The beaker was then set in a vacuum desiccator and the air exhausted until bubbles ceased to be given off. Usually this was obtained in less than an hour. Disks thus prepared were found to be always free from occluded air and satisfactory in every way for the determination.

HARRY STEENBOCK.

LABORATORY OF AGRICULTURAL CHEMISTRY,  
UNIVERSITY OF WISCONSIN.

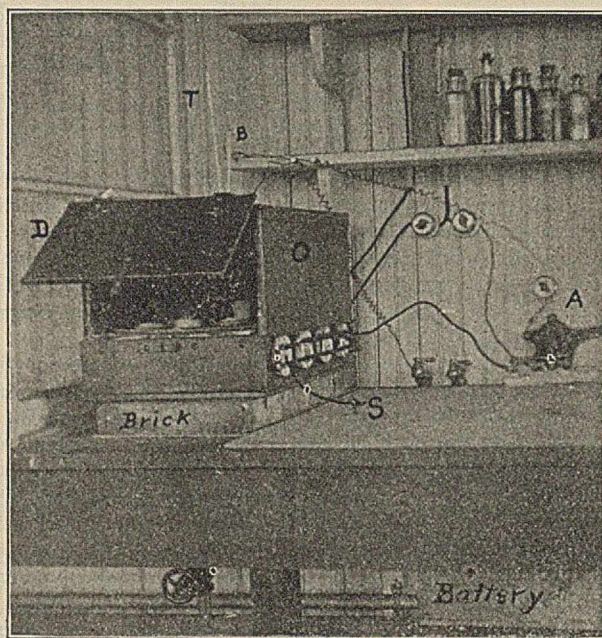
#### A CONVENIENT DRYING OVEN.

To some of the younger chemists in small laboratories, who have had trouble in finding a satisfactory drying oven for various temperatures, this device in use in our laboratory may have some suggestion.

We make no claims for originality in this device, and the ideas were suggested from various sources and concentrated in this oven. It consists of an oblong box "O" as shown in the cut  $12'' \times 12'' \times 18'' \times \frac{3}{8}''$  thick. The box itself is made of two thicknesses of thin black sheet-iron with  $\frac{1}{4}''$  transite asbestos board (obtained from H. W. Johns-Manville Co., of Detroit) between. All joints are bent and overlapped so that rivets or other fastenings are unnecessary. The oven is heated by electric lights in two circuits, one set being on continuously, and the other connected through a home-made thermostat device. The thermostat consists of a piece of fine capillary glass tubing (B) about 8" long, with a  $\frac{1}{2}''$  to  $\frac{3}{4}''$  thin bulb, blown on one end, and full of clean mercury. A small piece of platinum wire is sealed at this end, and another piece of wire is thrust in the other open end, to close the circuit as the mer-

cury expands. A consists of the magnet coils of an old worn-out electric bell. The soft iron bar of the bell was soldered to the end of a single knife switch, as short as possible, and four dry cells connected with the capillary tubing and coils, all in series. One set of lamps, put in staggered (all sockets on one side can be seen in cut), consists of four 16 candle power incandescent lamps continuously on, while the other set, for our use, three 16 candle power and one 32 candle power, is connected through the knife switch. The wire in the capillary tubing can be so adjusted that when the mercury expands to the desired temperature, the battery circuit will close, the magnet will attract the soft iron (that is, throw out the switch) and the other set of lights will go out. When the oven cools below the desired temperature, the mercury falls, the battery circuit breaks, and when the switch engages again (instantaneously), the lights go on.

Two 1" angle irons are fastened to the inner sides of the oven, forming a support for the oven shelf, which should be of thin iron and contain plenty of holes for circulation. The lamp sockets (S) extend through the oven and are made of porcelain. The door (D) is of same material as rest of the box, and is fastened with riveted hinges. Two 1" openings are left in top for



thermometer (T), thermostat (B) and vent. Care must be taken to have the knife switch as light as possible to work quickly without doing too much mechanical work to compromise accuracy of working, and the bore of capillary small enough to allow considerable expansion for each degree of temperature.

This makes a very satisfactory automatic oven, and will run very close to the desired temperature, depending on the skill in glass-blowing and adjustment of switch. A thermometer which we purchased specially constructed to cut off at  $95^\circ$  did not work as delicately as our own device. Any temperature may be obtained easily up to  $150^\circ$  C., depending on the number of watts consumed. We happen to use  $95^\circ$  C. with a maximum variation of about one degree each way. At  $105^\circ$  to  $110^\circ$  the variation is much smaller. The cost of the whole of this oven, including labor, was below \$10.00. The writer happens to know of no satisfactory automatic device similar to this on the market, and has seen none in the literature. There are no resistance coils to get out of order or burn out, and when the lights burn out it is only necessary to replace them by screwing new ones in the socket. Another, but not as satisfactory a



method is to have each light turn off and on separately and thus regulate the temperature according to radiation.

M. M. MACLEAN.

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#### DETERMINATION OF MERCURY IN THE MERCURY SALTS OF THE PHARMACOPOEIA.<sup>1</sup>

Nearly all of the mercury salts of the Pharmacopoeia are provided with standards of purity, known as Purity Rubrics; but none of them is correspondingly equipped with a quantitative method of analysis by which one may determine whether the salt is up to its required standard. The reasons for this state of affairs and the undesirability of such a condition will not be entered upon here. It is only desired at this time to call attention to quantitative methods of assay that have proved valuable in the examination of these chemicals and to recommend such methods for consideration by those interested. Some of the processes to be described here contain little that is new in the way of chemistry, but the application of them for pharmacopoeial testing may be new. Others of the processes may have a grain of new material in them.

If the mercury preparations of the U. S. P. are first converted into mercuric nitrate they are then readily subjected to electrolysis to determine the per cent. of mercury. This is practical with mercury oxide red, mercury oxide yellow, metallic mercury, and with mercury with chalk. Solution of mercuric nitrate U. S. P. may also be assayed for the mercury present after proper dilution to bring it to the usual concentration. The general statement of the method is to dissolve about 0.5 gram sample in about 1 cc. of nitric acid (sp. gr. 1.20), dilute to about 20 cc. and subject the solution to electrolysis. Use the mercury cathode and the rotating anode, the latter revolving about 700 to 800 times per minute. The voltage should be 10 to 12, the amperage 3, and 15 minutes at room temperature will be found sufficient time for the electrolysis to become complete. The mercury cathode is then washed with water, alcohol, and ether in the usual way, any increase in weight being due to the metallic mercury from the weighed sample with which the analysis began. Three-quarters of an hour or one hour easily covers the entire analysis from beginning to end.

In the case of mercury with chalk the mercury cathode and rotating anode have not been sufficiently tried, but with a stationary anode and a platinum dish as cathode the method is excellent. On account of the size of the cathode dish the sample is diluted to greater volume. It is well to weigh off a sample of about 1 gram, dissolve it in nitric acid, dilute to about 125 cc. and electrolyze aliquots of about 25 cc. each. Pass the current through the solution for two hours, keeping the temperature at about 70° C., the amperage about 0.05, and the voltage about 2. Wash the cathode dish in which the mercury is deposited in mirror-form (not in globules if done right) with water, alcohol, and ether as usual and weigh for increase in weight of cathode. The calcium present does not interfere with the electrolysis.

For ammoniated mercury, mercury iodide red, mercury iodide yellow, and mercurous chloride, a different procedure is advisable. These salts are all dissolved in solution of sodium sulphide and from this the mercury is deposited. The samples, about 0.3 to 0.5 gram, are weighed directly into the tared mercury cathode dish. 10 cc. of solution of sodium sulphide (sp. gr. 1.18) are added and a current of 0.5 to 0.75 ampere and 4 to 5 volts is passed for half an hour. Use the rotating anode running about 500 revolutions per minute. The mercury from the samples is deposited on that of the mercury cathode and all is washed and dried in the usual way with water, alcohol,

and ether. If any amalgam of sodium and mercury forms during the electrolysis allow the wash water to stand on it until it is all decomposed. The results are excellent.

Mercuric chloride is best treated in a simpler manner. To determine the mercury quickly in this place a sample of about 0.3 gram in a weighed mercury cathode dish, dissolve in 20 cc. of water, and, after adding a layer of about 10 cc. of toluene to protect the apparatus from chlorine gas, pass a current of about 1 ampere and 10 to 11 volts for fifteen minutes. The speed of the rotating anode to be used in this case is about 500 revolutions per minute. At the end of fifteen minutes the liquids may as usual be removed by decantation and the mercury cathode washed, dried and weighed. The method is accurate, and the time consumed by the entire analysis is about one-half to three-quarters of an hour.

It is perhaps advisable to add that in carrying out these tests no unusual dexterity or skill in manipulation is required, only a very elementary knowledge of electricity is needed, and the cost of the entire apparatus, exclusive of the platinum dish used in the one test, need not exceed thirty or thirty-five dollars. The work can therefore be carried on in any laboratory reasonably well equipped.

B. L. MURRAY.

#### THE OCCURRENCE OF SUCROSE IN GRAPES.

It has been positively stated by the most eminent writers on enology the sucrose does not occur in the fruit of grapes. For several of these statements consult the following:

Dr. J. W. L. Thudicum, "A Treatise on Wines," London, 1896, p. 65.

Babo und Mach, "Weinbau und Kellerwirtschaft," Berlin, 1896, Vol. 11, p. 2.

M. Victor Sebastian, "Traite Pratique de la Preparation des vins de Luxe," Montpellier, 1909, p. 84; and other statements of like tenor occur in the literature.

However, in 1908 Mr. H. C. Gore, of the Bureau of Chemistry, discovered sucrose in two varieties of Scuppernong grape, as in a communication to THIS JOURNAL.<sup>1</sup>

During the season of 1909 while conducting for the Bureau of Chemistry, the work on the chemical investigation of the grapes grown in the Lake Erie district, we found sucrose in several varieties of grapes belonging to the species commonly cultivated in the United States, but more especially in a new seedling grape discovered growing near Sandusky, O., which was until then unknown to horticultural literature.

The known varieties in which sucrose was found to occur are Hays, Pocklington and Worden. These showed sucrose in quantities varying from 4.49 grams to 5.66 grams per 100 cc. of juice. The new seedling, however, showed sucrose varying at different dates from 7.06 grams to 9.73 grams per 100 cc. of juice with a total sugar content varying from 15.93 grams to 19.52 grams per 100 cc. of juice.

While these determinations of sucrose on several cases have been checked by polariscope readings and are apparently correct, the full data will not be published until the whole subject can be re-examined the present season.

The occurrence of sucrose in large quantity in the fruit of grapes belonging to the types usually cultivated for wine and table use is so unexpected and important an observation that it is proper to exercise due caution before printing a detailed statement of the results. The purpose of this note is simply to record an interesting discovery.

However, it should not be presumed from the above statement that sucrose will probably be found in any considerable number of varieties of our common grapes. On the contrary, we have now examined in the laboratory a large series of samples of practically all the wine and table grapes grown in the Eastern States and in no case other than mentioned above have we

<sup>1</sup> Read at San Francisco Meeting, American Chemical Society, July 12-16, 1910.

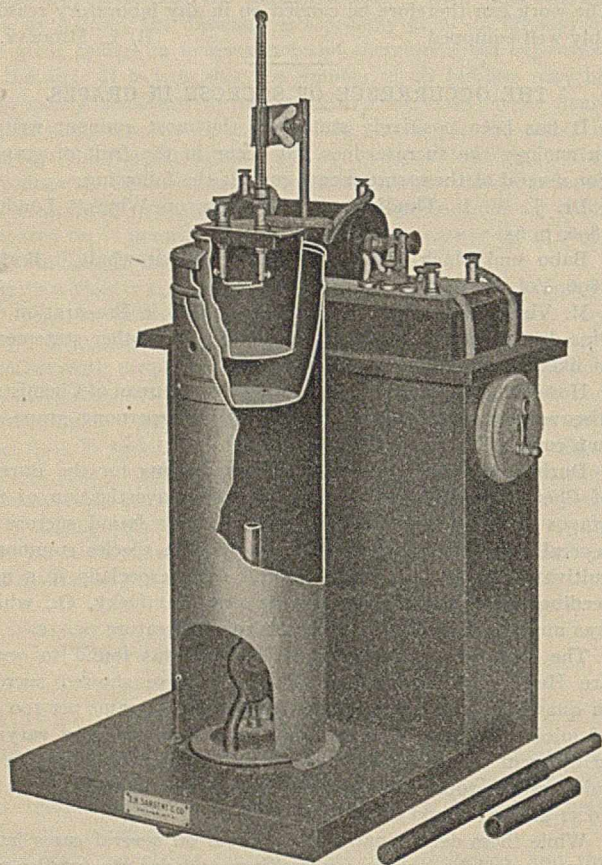
found sucrose in appreciable quantity. The analyses were made by Dr. B. G. Hartmann and Mr. John R. Eoff.

WM. B. ALWOOD.

STONEHENGE LABORATORIES, CHARLOTTESVILLE, VA.,  
July 15, 1910.

### THE SCOTT OIL TESTER.

Scott's oil tester is a flash point apparatus for oils, varnishes, ether, alcohol, gasoline, turpentine, etc. While it has just made its appearance on the open market it has been in constant use for several years in the laboratory of its designer, Mr. D. G. Scott, of Chicago. It is electrically ignited and does away with the errors of personal equation, every determination being made under exactly the same conditions. One of the peculiar features of this apparatus lies in the electrodes which are so fashioned that when properly adjusted the spark travels backward and forward across a considerable portion of the surface of the oil so fast as to practically form a ribbon of fire about a quarter of an inch wide. The combustion or flash chamber



is so arranged that determinations may be made in freezing mixtures or high flash point determinations may be made over a naked flame. The amount of sample needed is exceedingly small, precluding danger from fire and thus making it particularly desirable for use in paint, oil and varnish laboratories.

The whole apparatus is self-contained, including batteries, etc., and occupies a space of only 8 by 10 inches on the table and is about 16 inches high. Its operation is very simple and its convenience apparent. It is durable and easily cleaned and its compactness and stability of design are such that it is as easily placed on the table ready for use or removed to the wall case for storage as a bottle or jar of the same size.

The first notice of this apparatus occurs in the new catalog of Messrs. E. H. Sargent & Co., of Chicago, who manufacture the instrument under the direction of Mr. Scott.

### CHROME PAINT ANALYSIS.

The chrome yellow paints on the market very frequently contain a number of constituents other than lead chromate, and the lack of any definite procedure in analytical works dealing with paints has led to the following attempt to arrive at a satisfactory scheme of analysis. A mixture containing the following ingredients was made:  $\text{BaSO}_4$ ,  $\text{SiO}_2$ ,  $\text{PbCrO}_4$ ,  $\text{PbO}$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{ZnO}$ ,  $\text{CaSO}_4$  and  $\text{CaCO}_3$ . While it is not likely that all of these constituents would ever be present in a single paint, yet all of them have been found in chrome paints, and the following scheme of analysis can be applied to determine any or all of them.

(1) The silica and barium sulphate are separated by dissolving 1 g. of the paint in conc. HCl and evaporating to dryness twice with HCl. A few drops of HCl are then added and about 100 cc. hot water and the solution brought to a boil, allowed to settle, decanted through a filter and the process repeated. The insol. residue, which should then be white (if not a few drops of HCl are added and the process repeated until it is), is filtered off, washed with hot water several times and ignited in the blast in a platinum crucible and weighed. This gives  $\text{BaSO}_4$  and  $\text{SiO}_2$ . A few drops of  $\text{H}_2\text{SO}_4$  and some hydrofluoric acid are added and the  $\text{SiO}_2$  volatilized, and the  $\text{BaSO}_4$  weighed. The  $\text{SiO}_2$  is estimated by the loss in weight.

(2) The calcium present as sulphate is obtained from the filtrate from the  $\text{BaSO}_4$  and  $\text{SiO}_2$ . This is brought to a boil and the sulphate precipitated with  $\text{BaCl}_2$  in the usual manner. After settling, the  $\text{BaSO}_4$  is filtered off and washed thoroughly with very hot water (care must be exercised to wash out all the  $\text{PbCl}_2$ ), ignited and weighed. The  $\text{BaSO}_4$  is calculated to  $\text{CaSO}_4$ .

(3) The zinc oxide and calcium carbonate are obtained by treating 1 gram of sample with strong acetic acid, boiling, then diluting and filtering. The zinc oxide, all calcium salts and most of lead present as oxide pass into solution. The zinc and lead are precipitated with  $\text{H}_2\text{S}$  and filtered off, redissolved in hot dilute HCl and several grams of sodium acetate added; the lead is then precipitated as chromate by adding small excess of  $\text{K}_2\text{CrO}_4$ . This is filtered off and the zinc precipitated in the filtrate as carbonate by adding an excess of  $\text{Na}_2\text{CO}_3$  and digesting. The precipitate is filtered off, ignited and weighed as  $\text{ZnO}$ . The Ca which is in the filtrate from the Zn and Pb is precipitated as oxalate after making alkaline with  $\text{NH}_4\text{OH}$ , filtered, ignited and weighed as oxide. From total CaO is subtracted CaO present as  $\text{CaSO}_4$ , and the remainder calculated to  $\text{CaCO}_3$ .

(4) The ferric oxide is obtained by treating 1 g. of the paint with about 10 g. NaOH and 100 cc. water, heating to the boiling point, filtering and washing with hot water. The filter paper containing  $\text{Fe}_2\text{O}_3$ ,  $\text{BaSO}_4$ ,  $\text{SiO}_2$  and  $\text{PbO}$  is then ignited slowly in a porcelain crucible (to prevent its sticking to sides of crucible) and the residue digested with 5 cc. hot conc. HCl and transferred to a beaker with 50 cc. of water. The  $\text{BaSO}_4$  and  $\text{SiO}_2$  are filtered off and the filtrate almost neutralized with  $\text{NH}_4\text{OH}$  and the lead precipitated as sulphide and filtered off. The  $\text{H}_2\text{S}$  is boiled from the filtrate, the iron oxidized to ferric condition with a little  $\text{HNO}_3$ , precipitated with ammonia and weighed as  $\text{Fe}_2\text{O}_3$ . The filtrate from  $\text{Fe}_2\text{O}_3$ ,  $\text{BaSO}_4$ , etc., is used for the estimation of lead chromate. It is acidulated with acetic acid, when the insol.  $\text{PbCrO}_4$  is precipitated, filtered through Gooch crucible, dried on hot asbestos plate and weighed as  $\text{PbCrO}_4$ .

(5) To obtain  $\text{PbO}$ , 1 gram of paint is treated as for  $\text{BaSO}_4$  and  $\text{SiO}_2$ , the filtrate evaporated to dryness, taken up with a few drops of HCl, about 100 cc. hot water added and the solution boiled until clear green, adding more HCl if necessary. About 5-10 grams sodium acetate are then added to remove free hydrochloric acid. At this point the color of the solution

changes to reddish brown on account of the ferric acetate formed. A few cc. of acetic acid are now added and then 1 gram of potassium chromate, and the solution stirred vigorously. The lead is precipitated as chromate which is filtered off and washed thoroughly with cold water on a weighed Gooch crucible, dried on the asbestos plate and weighed. The weight of the  $PbCrO_4$  found in previous sample is subtracted from this and the difference is calculated to  $PbO$ . When white lead is present it is necessary to make a  $CO_2$  determination, and after deducting the  $CO_2$  due to  $CaCO_3$  the remainder is calculated to white lead and the excess of lead then figured as  $PbO$ .

The mixture analyzed had the composition shown in column 1 and the results obtained are given in column 2.

	1.	2.
	Per cent.	Per cent.
$SiO_2$ .....	4.76	4.52
$BaSO_4$ .....	4.76	4.70
$Fe_2O_3$ .....	4.76	4.88
$CaSO_4$ .....	4.76	4.90
$ZnO$ .....	9.52	9.54
$CaCO_3$ .....	4.76	4.64
$PbCrO_4$ .....	57.14	57.04
$PbO$ .....	9.54	9.68
	100.00	99.90

E. F. SCHERUBEL.  
E. S. WOOD.

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## BOOK REVIEWS AND NOTICES.

**The Corrosion and Preservation of Iron and Steel.** By ALLERTON S. CUSHMAN AND HENRY A. GARDNER. Large 8vo. pp. xv + 373. New York: McGraw-Hill Book Company, 1910. Price, \$4.00 net.

A book was needed which would bring together from the scattered literature the investigations and conclusions of the last few years on the corrosion and preservation of iron and steel. Although this work is still in progress and many investigations are engaged upon it, nevertheless it is high time that the conclusions reached thus far be presented in a single volume. This the authors have done in a way which is on the whole satisfactory and commendable. The development of the electrolytic theory of corrosion has been due largely to American workers, among whom the names of Whitney, Cushman and Walker stand pre-eminent, although others have contributed to no small degree. The development of the theory of corrosion is a triumph of which American chemists may well be proud. The first chapter, stating the problem of corrosion, is a satisfactory presentation of the question. Mention is made of the iron column of Kutab Minar at Delhi, India, supposed to have been erected 900 B. C., which has withstood atmospheric conditions for so many years without corrosion. A good deal of sentiment has been wasted on this column during recent years. It seems a pity that the Indian government has not investigated the physical and chemical properties of the column, or granted permission to somebody to do so. Chapter 2 takes up the Theory of Solution, and while it may be criticized on the ground that the statements are somewhat too absolute for dealing with a theory, the exposition is nevertheless good. Chapter 3 considers the Theory of Corrosion: discusses the carbonic acid theory, the peroxide theory, and finally the electrolytic theory. The development of the feroxyl indicator by Walker and Cushman and its application are given in detail. Chapter 4 deals with the Application of Electrolytic Theory; Chapter 5 is on The Inhibition and Stimulation of Corrosion; Chapter 6 is on The Technical Protection of Iron and Steel; Chapter 7, Relation of Pigments to the Corrosion of Iron; Chapter 8, Recent Field Tests on Protective Coatings for Iron and Steel; Chapter 9,

Paints for Various Purposes; Chapter 10, The Testing and Design of Protective Paints; Chapter 11, The Properties of Pigments; Chapter 12, The Properties of Paint Vehicles. This ends the body of the book. There is an appendix on The Corrosion of Water Jackets, and a bibliography of the subject reprinted from the bibliography issued by the Carnegie Library of Pittsburg. The index covers 8 pages single column. The work is well printed and bound; the paper and illustrations excellent. All students of the subject will necessarily find the work desirable. It is a creditable addition to the literature.

**Soil Fertility and Permanent Agriculture.** By CYRIL G. HOPKINS. Cloth, 8vo. pp. xxiii + 653. Boston: Ginn & Company, 1910. Price, \$2.75.

Dr. Hopkins' name has appeared frequently in various recent scientific publications as an advocate of certain ideas in regard to soil fertility in opposition to those held by another school. It is, therefore, interesting to quote the first words in the introduction of the present book which appears to lay down Dr. Hopkins' fundamental doctrine: "It is the first business of every farmer to reduce the fertility of the soil, by removing the largest crops of which the soil is capable, but ultimate failure results for the land owner unless provision is made for restoring and maintaining productiveness. Every land owner should adopt for his land a system of farming that is permanent—a system under which the land becomes better rather than poorer." A little further along in the introduction, the author speaks so pointedly in regard to dealing with a technical subject, technically that his remarks are worth quoting for the benefit of writers in other lines of work: "In the preparation of this book free use has been made of such technical terms as are necessary to the discussion of fundamental principles with scientific correctness. No apology is offered for this. Farmers and agricultural students have at least as good intellects as other classes of people; and if when they leave the farm they can learn to understand and manage successfully such lines of business as banking, contracting, building, operating railroads, factories, and other commercial establishments—which they are doing everywhere—they can also understand their own business, if they will, when they remain on the farm or in control of land." This introduction is most interesting reading for the student, farmer or for the average man. The body of the book is divided into four parts and an appendix. Part 1, Science and Soil; Part 2, Systems of Permanent Agriculture; Part 3, Soil Investigations by Culture Experiments; Part 4, Various Fertility Factors. The book is evidently designed primarily as a text-book, and begins with the fundamentals of science and chemistry. The chemical part of the book is open to criticism on the ground that it is too artificial in its treatment. There is a tendency to make absolute statements where absolute statements cannot be made. It is too abstract, too academic, too much concerned with signs and symbols and too little with the realities of the science. "The bond between atoms may be likened to the hand clasp between persons, except that under normal conditions the hand of one atom always grasps the hand of another atom." (P. 4.) "The nitrogen atom has five bonds, or hands, but in some compounds only three bonds are used to hold other atoms. It might be conceived in this case that the other two hands are clasped together, and this conception might even be extended to cover a molecule composed of a single bivalent atom (such as mercury and, possibly, argon)." (P. 6.) In general it is doubtful if the use of fanciful similes in chemical theory has been of great benefit to the student or the science, certainly not now. The science of chemistry has been obscured by a jumble of bonds, hands, hooks, claws, oranges and peas, shot and cannon balls, smooth spheres and billiard balls until the

student has lost sight completely of the realities and becomes lost on a maze of obscure imagery. Pray let us reform this altogether! In the chapter on the earth's crust (in general good) there is some ground for remark. On page 48 the statements about gneiss are entirely misleading or untrue.

Dr. Hopkins is most at home in the chapters actually dealing with soil composition, surveys and analyses, sources of plant food, the uses of plant foods by plants, theory of soil fertility, the Rothamsted experiments and other field experiments. The chapters dealing with these subjects are in general excellent. They are, of course, colored by Dr. Hopkins' views on soil fertility, but he has nevertheless given a fair statement of the views held by his opponents, largely in the form of quotations. Naturally a considerable portion of the book is devoted to discussion of the Rothamsted experiments; a smaller portion to Ohio, Illinois, Minnesota, Canadian and other field experiments. Under Various Fertility Factors he discusses Manufacture of Commercial Fertilizers, Crop Stimulants and Protective Agents, Critical Periods in Plant Life, Farm Manure, Losses of Plant Food from Plants, Losses of Plant Foods from Soils, Analyzing and Testing Soils, Relation of Fertility to Appearance of Soils or Crops, Factors in Crop Production, Essential Factors of Success in Farming, The Value of Land, and Two Periods of Agricultural History. The last chapter mentioned consists of quotations from the ancient Roman writers down to Abraham Lincoln and Daniel Webster on the subject of fertility of the soil. The appendix consists mainly of statistics and tables of analyses of soils. There is a good index. The book is well printed and bound, and illustrated principally by reproductions from photographs of well-known agricultural chemists and directors of experiment stations.

There is a two-page map of the United States and one of Illinois showing the soil provinces, which are examples of good printing. The book is a welcome addition to the literature of the subject and barring a few features of the chemical and geological chapters the presentation is excellent.

**The Chemists' Pocket Manual.** By RICHARD K. MEADE. Second Edition. Black flexible leather.  $4 \times 6\frac{1}{2}$  inches. pp. xii + 443. Easton: The Chemical Publishing Co. 1910. Price, \$3.00 net.

This little book is really a chemists' pocket book. It might also be called a chemical engineers' pocket book on account of its inclusion of much engineering data seldom inserted in chemical books of this sort. Since the first edition was issued several years ago much material has accumulated and the present volume appears to be of about double the contents of the former. The book is subdivided by the following headings which give a good idea of its plan and makeup: Metric and United States Weights and Measures; Mathematical; Mensuration, International Atomic Weights, 1908; Stoichiometry; Graphic Methods for Saving Calculation; Conversion Tables; Specific Gravities of Solids and Liquids; Weight and Volume of Substances; Standard Tables of Specific Gravity of Sulphuric Acid, Nitric Acid, Hydrochloric Acid and Ammonia, Adopted by the Manufacturing Chemists' Association of the United States; Lunge's and Other Tables of the Specific Gravity of Acids and Alkalies; Physical Properties of Gases; Hygrometers; Solubility; Boiling-Point and Melting-Point; Standardizing Weights; Calibration of Chemical Glassware; Temperature; Heat; Combustion; Radiation; Electricity; Mechanics; Mineralogy; Determinative Mineralogy; Geology; Volumetric Solutions; Standardizing Volumetric Solutions; Reagents Used in Quantitative, Qualitative and Gas Analysis; Indicators for Volumetric Analysis; Test Papers; Special Reagents; Solutions for Gas Analysis; Miscellaneous; Scheme for the Qualitative Analysis of a Substance; Assaying; Analysis of Iron Ores; Determination of Copper in Ores, Mattes, Etc.; Determination of Lead in Lead Ores; Determination of Zinc in Zinc Ores; Determination of Sulphur in Lead, Copper and Zinc Ores; Analysis of Iron and Steel;

Analysis of Blast-Furnace Slags; Analysis of Alloys of Tin, Antimony, Lead, Copper and Zinc (Bearing Metals); The Analysis of Coal; Analysis of Fuel Gases; Analysis of Blast-Furnace Gas, Illuminating Gas, Producer Gas, Etc.; Determination of the Hardness of Water; Complete Examination of Water for Boiler Purposes; Rapid Analysis of Water for Boiler Purposes; The Examination of Potable Water; Chemical and Physical Examination of Portland Cement; Analysis of Limestone, Cement Rock, Lime, Rosindale Cement, Etc.; Analysis of Clay, Fire-Clay, Fire-Brick, Etc.; Examination of Lubricating Oils; The Analysis of Soap; Chemical Analysis of Mixed Paints; The Chemical Analysis of Mixed White Pigments; The Analysis of Fertilizers; Determination of Carbon and Hydrogen in Organic Substances; Determination of Nitrogen; Index.

The binding and print are good and the paper, which at first appears somewhat thick, is found to be tough and of good wearing quality—most desirable in a work of this sort. The book will find many users among works chemists, who want just the sort of information covered in a single compact volume.

## SCIENTIFIC AND INDUSTRIAL SOCIETIES.

### AMERICAN ELECTROCHEMICAL SOCIETY.

Chicago Meeting, October 13-15, 1910.

The eighteenth general meeting of the American Electrochemical Society, which was held in Chicago October 13, 14 and 15, 1910, was a splendid success. The registration showed a total enrolment of about 230 members and guests present at the meeting. The arrangements of the local committee and the officers of the Society were complete and well perfected, so that each event took place according to schedule and to the complete satisfaction of those in attendance at the meeting.

The first session was called to order by President William H. Walker, of Boston, at 10.00 A.M., October 13th, in the Florentine room of the Congress Hotel. The program of papers has already been published in these columns.<sup>1</sup> The papers of greatest interest to our membership will be published in THIS JOURNAL. Dr. E. Weintraub's paper on cast copper (not on the printed program), read by Mr. A. B. Marvin, of Chicago, on account of Dr. Weintraub's absence, excited great enthusiasm. Samples of cast copper made by the process were exhibited to the Society. Dr. Richards read a paper on the Hiorf electric furnace, also not on the printed program. A third paper, not previously announced, was by Mr. F. T. Snyder, entitled "The Flow of Heat through Furnace Walls."

In the afternoon there were several excursions, largely participated in. The evening was left free and was utilized by the members mainly for promoting acquaintanceship and good feeling.

On Friday morning, October 14th, the reading of papers was resumed in the Florentine room at 9.00 A.M. One of the most pleasing and vital features, characteristic of all the sessions was the general discussion of papers heartily entered into by the audience.

On Friday afternoon there were excursions to the plants of the Illinois Steel Co. at South Chicago, the Indiana Steel Co. at Gary, Ind., and Sears, Roebuck & Co.

### ILLINOIS STEEL CO.

At the Illinois Steel Co.'s plant the party witnessed the operation of the 15-ton Héroult electric steel furnace, which is working on blown metal from the Bessemer converters. This furnace has been in operation over seventeen months. Its electrical equipment consists of three 750 kw. transformers to reduce the three-phase twenty-five cycle power from 2200 volts to approximately 90 volts, so as to obtain approximately 50 volts at each arc. This trip was made particularly interesting by the presence of Dr. Héroult. After the electric furnace,

<sup>1</sup> See THIS JOURNAL, October, 1910.

other parts of the plant were visited. While the Gary plant in time is destined to be the largest plant in the world, the South Chicago plant has up to the present time held its own remarkably well. No single thing shows the development of the plant better than the increase in the use of electrical power during recent years. Twelve years ago the electrical power generating equipment consisted of a 400 kilowatt unit. Six years later this had been replaced by a 3000 kw. plant. Then followed rapidly one increase after another until now three power generating stations are in operation with a total capacity, including that under construction, of 33,000 kw., the same comprehending steam engine driven, gas engine driven and high-pressure and low-pressure turbine driven, units varying in size from 400 kw. to 7000 kw. Several sub-stations for transforming the energy to lower voltage are in service; likewise numerous local transformer stations. Plans under discussion will no doubt result in more than doubling the present capacity within two or three years.

The electrical energy is utilized for every conceivable purpose in every department of the plant. A few years ago problems were solved by means of hydraulic or steam power. One need but enter one of the newer mills to perceive the absence of such power. An application of electrical power is invariably resorted to in solving mill problems.

The generating stations of this plant supply power for 2000 motors, aggregating 90,000 h. p., an electric reducing furnace requiring 1500 kw., and the necessary plant illumination. A rail mill for rolling light sections has two 2000 h. p. motors on the main rolls. A universal plate mill has a 6000 h. p. motor on the rolls. A new structural mill will have 4000 h. p. on the main rolls. From such sizes down to fan motors are found in this plant.

At the Gary plant the visitors were shown the facilities for handling ore, the blast furnaces, gas washers, power plants and mills.

On Friday night the banquet was attended by 85 members and guests.

Saturday morning the reading of papers was continued in Kent laboratory, at the University of Chicago. Among others, Prof. Milliken spoke on the "Isolation of an Ion and the Precision Measurement of Its Charge," detailing his recent fundamental researches in this field. Prof. Milliken's personality, as well as his subject matter, aroused the greatest interest and enthusiasm in his listeners.

Luncheon was served at the University Commons.

#### CHICAGO DRAINAGE CANAL AND POWER PLANT.

In the afternoon the Society left by special train for Lockport to inspect the Sanitary and Ship Canal and Power Plant of

the Chicago Sanitary District at that place. The combination of sewage disposal canal, ship canal, and electric power plant is unique and the visitors were delighted with the splendid engineering achievement.

This excursion was the last feature of the program and the visiting members, for the most part, left Saturday night for their homes.

#### TAYLOR INSTRUMENT COMPANY'S EXHIBIT.

One of the interesting features of the meeting was the pyrometer exhibit of the Taylor Instrument Company, arranged near the meeting place. They classify their instruments into three groups:

1. Radiation Pyrometers.
2. Resistance Pyrometers.
3. Thermo-couple Pyrometers.

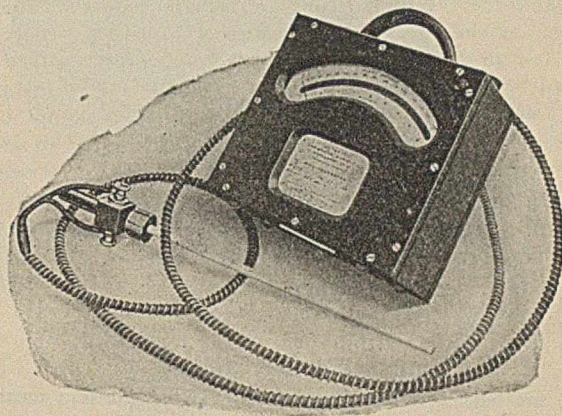
The application of these various instruments depends chiefly on the temperature they are to measure and for convenience we will describe their limits as follows:

Class 1. For temperatures of 1500° F. and upward.

Class 2. For temperatures from the lowest to 2200° F.

Class 3. For temperatures up to 2500° F.

The most satisfactory form of radiation pyrometer is one so constructed that error in bringing the object under observa-

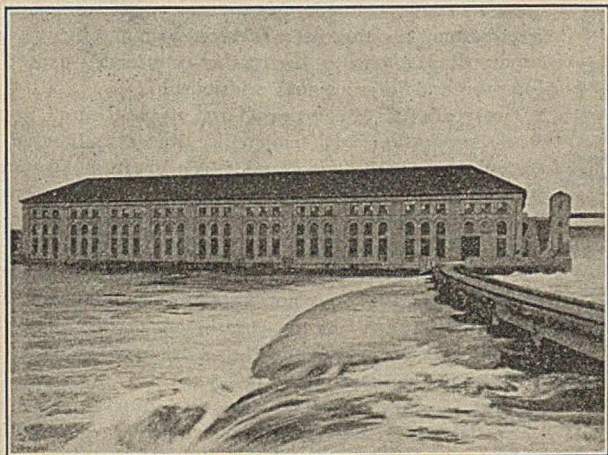


Thermo-couple.

tion is impossible. It must not depend upon color, as it is a well-known fact that one and the same hue will not appear identical to two observers.

The Fery radiation pyrometer overcomes this objection by having a mechanical focusing device which insures proper observation and is entirely independent of color comparison. The instrument is virtually a reflecting telescope with the hot junction of a sensitive thermo-couple at the focus of the concave reflecting mirror. The radiant heat emanating from a body is collected by the mirror and focused upon the couple generating electrical energy of sufficient magnitude to be measured on a millivolt meter. As the thermo-couple has both its hot and cold junctions very close together, the instrument is practically independent of surrounding temperature. Furthermore, within wide limits it is unaffected by the distance at which it works. It may be used as an indicating instrument and the complement of the outfit in this case comprises the telescope, cable, tripod and indicator with two scales, viz., 1000° F. to 2400° F. and 1600° F. to 3600° F., or their centigrade equivalents, or if records of temperature are desirable the thread recorder is supplied. This instrument is a highly sensitive galvanometer entire free of friction. It gives a record with rectangular coördinates. As the record is written in ink it is permanent and requires no further fixing.

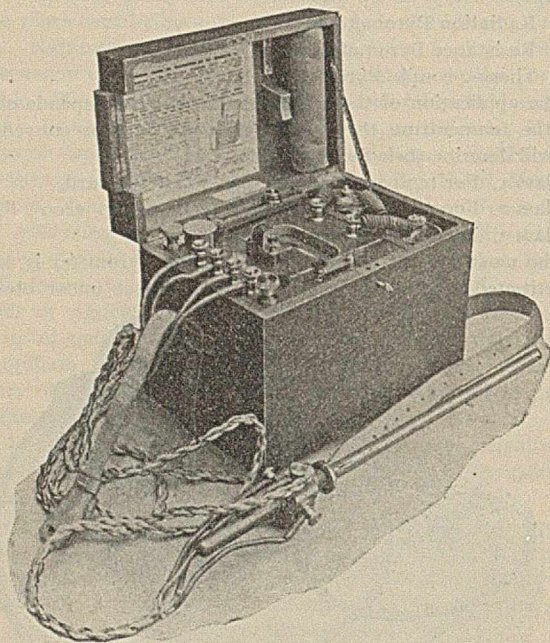
The resistance thermometer consists of a mica frame upon which is wound fine platinum wire. This "bulb" is about 3 to 4 inches in length and the fine wire is connected to stouter wire at its upper extremities. If the temperatures under con-



Chicago Sanitary District Power House at Lockport, Ill.

sideration are high, these lead wires are platinum; for lower temperatures, however, they are usually copper. The lead wire and bulb are protected by a suitable covering sheath. As the bulb becomes heated its electrical conductivity is lessened, while conversely the lower its temperature the less resistance it offers to the flow of current.

Measurements of resistance can be made very accurately on the Wheatstone bridge, hence by applying this principle in the construction of the Whipple indicator and suitably calibrating the resistance scale to read directly in temperature units, an instrument of extreme precision is obtained. As a balanced method is used, two additional wires (making 4 in

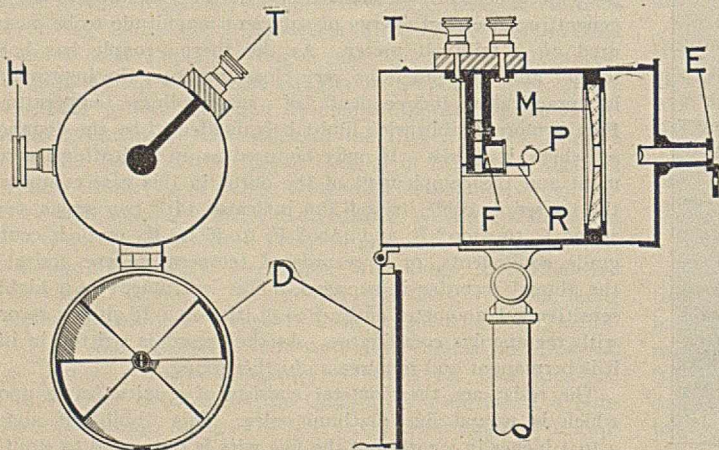


Whipple indicator.

all) are led from the thermometer. This affords a means of comparison so that the correct operation of a thermometer is assured regardless of the distance from the Whipple indicator, hence the suitability for temperature measurements of most inaccessible locations as mines, water tanks, chill rooms, hot blast mains, etc.

A Callender Recorder is used for obtaining continuous records of temperature. This instrument also embodies the Wheatstone Bridge principle and is exceedingly rapid in following the slightest changes of temperature. A very open rectangular scale is therefore provided.

As both the Whipple Indicator and Callender Recorder



Féry pyrometer.

operate on a battery circuit they may be used in isolated places where no source of electrical supply is available.

This method of temperature measurement is the most accurate as yet devised.

The Thermo-electric Thermometer or Thermo-couple is one of the most convenient forms of apparatus for measuring temperatures. Its bulk is small, and it can be made up in various shapes to suit widely different conditions.

The Platinum, Platinum-Rhodium Couple is capable of use for temperatures up to 2500° F. and even higher, the alloy is stable and deterioration in use very slow. The melting points of the two component metals are very high, over 3000° F., and the wires are strong and tough. If, after long use, the electromotive force of a couple has fallen, it can be almost completely restored to its original value by electrically annealing the wires in air at a bright yellow heat.

The actual electromotive force measured is the difference between that of the two junctions of the couple; for accurate work it is therefore important to keep the so-called cold, or comparison, junction at or near a constant known temperature.

For high temperature measurements in industrial work it is usually sufficient if the comparison junction in the thermo-couple head is kept fairly cool in the air outside the furnace, and this consideration frequently decides the length of the couple.

As with the Féry pyrometer, the thermo-couple may be used with either indicator for instantaneous value of temperature or with the thread recorder where continuous records are desired.

#### THE AMERICAN IRON AND STEEL INSTITUTE.

New York Conference, October 14 to 22, 1910.

Friday, October 14th, 10.30 A.M., Myrtle Room, Waldorf Astoria Hotel.—Reading and discussion of papers. Address by the President, E. H. Gary. "Foreign Relations," by James A. Farrell. "Betterment of Labor Conditions," by William B. Dickson. "Contract Obligations," by Willis L. King. "The International Metallurgical Congress at Düsseldorf," by Charles Kirchhoff.

7.30 P.M.—Banquet in the Astor Gallery.

October 15th to 22nd.—Special excursion to inspect the large steel industries of the country, Buffalo, Gary, Chicago, Pullman, Pittsburg and Washington being included in the itinerary.

The recent meeting in New York of the American Iron and Steel Institute and the subsequent tour through the country, inspecting the great steel centers, is unique. There have been several previous tours in this country of representatives of the large iron and steel industries, notably those of 1890 and 1904, at the invitation of the American Institute of Mining Engineers, but never before in this country or in Europe has an international meeting been gathered together, the primary import of which is to discuss the commercial aspect of domestic and international relationship.

Two years ago, Chairman Gary, of the United States Steel Corporation, was tendered a dinner in London by the officers of the Iron and Steel Institute of Great Britain. At that dinner Judge Gary outlined a certain policy which he thought could be adopted to the greatest economic advantage by the great steel industries. In his own words this doctrine was "competition, but not hostility; rivalry, but not antagonism; progress and success for all, but not the punishment or destruction of any." And this meeting in New York may be taken as a direct furtherance of this policy.

Thirty foreign representatives, including some of the most important masters of the iron and steel manufacturers of Great Britain and Europe, attended this meeting as the guests of the Institute.

The first day, Friday, October 14th, was given up to the reading and discussion of papers, the very nature of which indicates the general purport of the whole meeting.

"Foreign Relations," "The Betterment of Labor Conditions," and "Contract Obligations" suggest that this meeting is not a mere metallurgical conference but rather a desire for a better international understanding, a binding together of foreign sympathies and the wish for more complete co-operation in the handling of the great problems of international trade and competition.

And then, too, after the formal meeting and the opening dinner in New York, the subsequent tour through the country with its attendant inspection of the leading industrial centers was but another step toward the same end.

By special train and special arrangement, handled by committees composed of representatives of the leading commercial interests in each great city to be visited, about seventy-five members and guests visited the following centers in turn. Leaving New York Sunday evening, the party proceeded to Buffalo. Here the plant of the Lackawanna Steel Company was visited and later the Mammoth Lock at Black Rock was seen. From Black Rock the party went to Niagara Falls, where several hours were spent in a tour of the gorge and a visitation of several of the power plants.

From Niagara Falls the party left for Gary, Indiana, where Tuesday morning was spent in a tour through the wonderful new plant of the Illinois Steel Company.

On Tuesday afternoon the party inspected the South Works of the Illinois Steel Company, and in particular the Héroult Electric Furnace and the Gayley Dry Blast.

Tuesday evening a brilliant dinner was held at the Blackstone Hotel, with President Gary in the chair.

Wednesday was occupied in visits to the plants of the International Harvester Company, the Pullman Company, and the stock-yards.

Thursday, at Pittsburg, was spent in a steamboat trip up the Monongahela River to view in outline the great mills clustered on its banks. The Jones & Laughlin and the Carnegie Steel Company plants were visited, and in particular the Homestead Works.

Friday morning was devoted to an inspection of the Carnegie Institute, and the afternoon to a visit to the Westinghouse interests in East Pittsburg.

Finally the tour was brought to a fitting conclusion by a visit to Washington and a reception tendered the Institute and its guests by President Taft.

The importance of a great conference of this kind cannot be overestimated. Not only are the leading manufacturers of this country brought into closer and more intimate relationship with those of Great Britain and Europe but an understanding of local conditions, an appreciation of local sympathies, and an insight into the magnitude of our steel industries are obtained from a trip of this kind that can be obtained in no other way.

The United States for some time has been the leading manufacturer of iron and steel products, and the need for some such body as the Iron and Steel Institute has long been felt. The technical side of this great industry has been adequately dealt with by the Institute of Mining Engineers, the Society of Mechanical Engineers, the Chemical, Electrochemical and similar societies, but the commercial aspect and the executive aspect has been, till now, somewhat neglected.

The commercial and business aspects of the iron and steel industry have just as much need for discussion by the executives of the great companies as have the technical points by its engineers and scientists. And it is this aspect of the iron and steel industry that the American Iron and Steel Institute will embrace, in addition to bringing together the work of the scientists and technical societies.

CHAS. GLIDDEN OSBORNE.

## AMERICAN LEATHER CHEMISTS' ASSOCIATION.

The Seventh Annual Meeting, Chicago, 1910.

The seventh annual meeting of the American Leather Chemists' Association was held in conjunction with the convention of the National Association of Tanners at the Hotel La Salle, Chicago, Ill., October 6th, 7th and 8th, convening at 9.30 o'clock, A.M., Thursday, the 6th. The program was as follows:

Thursday, October 6th, Morning Session.—Opening remarks by the president. Report of the Secretary-Treasurer. Committee reports.

Afternoon Session.—Address by Dr. Allen Rogers, "Trade School for the Leather Industry." Address by Mr. George A. Kerr, "Manufacture of Extracts in the United States." Address by Mr. F. C. Rose, "Leather Oils." Address by Mr. John H. Yocum, "Disinfection of Hides."

Friday, October 7th, Morning Session.—Meeting of the National Association of Tanners. Members of the A. L. C. A. are invited to attend this meeting.

Afternoon Session.—Visit to the stock-yards: Armour & Co., Swift & Co., Libby, McNeil & Libby.

Evening.—Joint banquet at the Hotel La Salle. The National Association of Tanners have cordially invited the active members of the A. L. C. A. to attend this banquet as their guests.

Saturday, October 8th, Morning Session.—Address by Mr. F. P. Veitch, subject to be announced. Address by Mr. F. E. Atteaux, "The Proper Bating of Hides." Address by Mr. J. H. Russell, "Tannage of Heavy Leather in California." Address by Mr. George A. Riker, "Extraction of Leather by Various Solvents."

Afternoon Session.—Address by Mr. Thomas A. Faust, "The Necessity of Proper Sampling." Address by Mr. R. C. Jacobsen, subject to be announced. Committee reports. Election of officers.

H. C. REED,

Secretary.

## AMERICAN CHEMICAL SOCIETY.

Joint Meeting, Cincinnati, Nov. 12, 1910.

The Cincinnati Section of the American Chemical Society will hold a joint meeting with all the neighboring Sections on November 12th, in Cincinnati.

Dr. W. D. Bancroft, President of the National Society, has been secured as the main speaker, and his subject will be "The Measurement of High Temperatures in the Electric Furnace."

An honorary banquet will be given by the local section to all the visiting members of the American Chemical Society and side trips have been arranged to all points of interest in our city.

Guides will be appointed to escort the visitors to any industrial plant or section of the city that they wish to go to, and will see that all the wishes of the visitors are gratified.

ALFRED SMITH, Secretary.

October 7, 1910,  
RING PLACE, PRICE HILL,  
CINCINNATI, OHIO.

## PERSONAL NOTES.

Dr. Charles A. Crampton, for many years chief chemist in the U. S. Govt. Internal Revenue Service, has established a commercial laboratory in the Evans Building in Washington, D. C., and left the government service.

Mr. Robert E. Divine, analytical and consulting chemist of Detroit, Mich., has moved from the Park Building into more commodious quarters at 138 Monroe Ave.

Dr. Heinrich Caro, industrial chemist and recently director of the Badische Anilin- und Soda-Fabrik, died on a journey to Dresden on Sept. 11, 1910, at the age of 76 years.

## RECENT INVENTIONS.

The following patents relating to Industrial and Engineering Chemistry are reported by C. L. Parker, Solicitor of Chemical Patents, McGill Building, 908 G St., N. W., Washington, D. C.

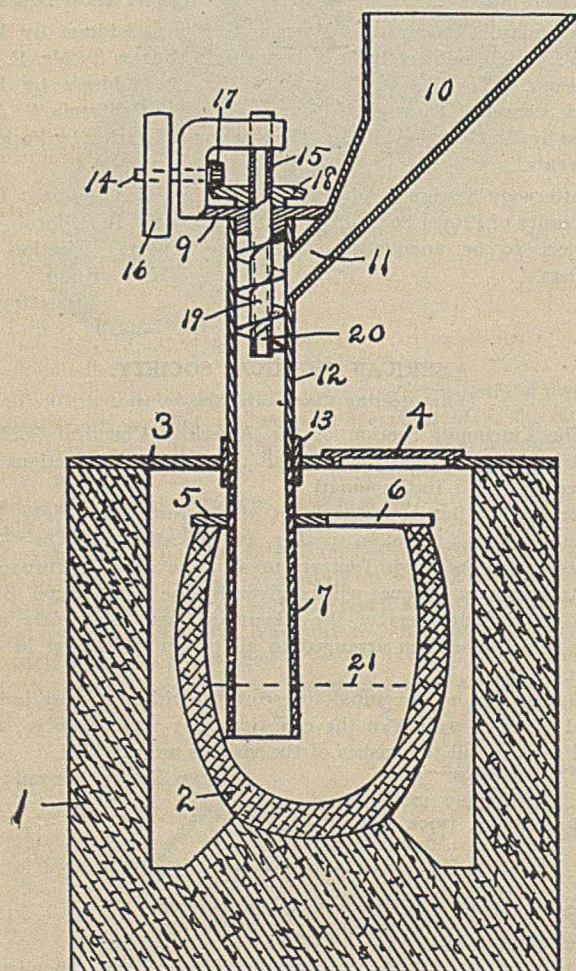
Only a few patents which are deemed of greatest importance are abstracted in this department.

Abstracts of all patents appear regularly in *Chemical Abstracts*, to which publication the reader is referred.

969,253. Process for Recovering Metal. ARCHIBALD W. DIACK, of Detroit, Michigan, Patented Sept. 6, 1910.

This invention comprises a process for recovering metals from the skimmings, and other dross, and for melting down borings and other small particles and pieces of such metals as readily oxidize when heated.

While this process is not necessarily carried out by any particular type of mechanism, that shown in the accompanying illustration embodies features necessary to the carrying out of this process. An oil, gas, or solid fuel furnace, 1, of well-known construction, contains the crucible 2 and is closed by



the cover 3, having an opening closed by the lid 4. A top, 5, partly covers the crucible and has an opening, 6, through which access is had to the contents. A refractory tube, 7, unacted upon at the temperature employed, preferably of carbon, extends down into the crucible below the line of the molten contents.

Above the furnace is mounted a hopper, 10, having a spout, 11, connecting with the pipe 12, which pipe connects to the tube 7 in any desirable manner, a connecting sleeve, 13, being shown. At the upper end of the pipe 12 may be mounted a frame, 9, in which are revoluble the shaft 14 and a tube, 15. On the shaft 14 may be secured a pulley, 16, and a pinion, 17, which latter meshes with the gear 18 on the tube 15. A screw-conveyer, 19, is secured to the tube 15 and will be turned by the shaft 14. The parts above the furnace top 3 may all be of cast iron, and any other mechanism may be employed to force the material down the tube 7. Arrangements may also be made for preheating the material in its passage from the hopper to the molten metal in the crucible.

Dross such as skimmings, from melted aluminum and of light-weight alloys, contain large amounts of metal. When such dross is packed into a crucible and heated to the melting point of the metal or alloy, the oxides and other impurities do not separate readily, as their specific gravity is so nearly that of the metal or alloy. In view of the fact that these metals readily oxidize at the melting point, much not already an oxid becomes so during this heating.

By thrusting the mingled metals, oxid and other impurities below the surface of a heavier metal with which it readily forms an alloy, the oxid is released, and, because of the great difference in specific gravity, immediately rises to the surface, covering the same and retarding further oxidation.

When the apparatus used in the drawing is employed, the mixed oxid and metals are thrown into the hopper 10, and slip down to the pipe 12 through the spout 11. The screw 19 slowly feeds down this material at a constant speed, forcing it down into the molten mass which fills the lower end of the tube 7. By the time the material is forced out of the lower end of the tube, the metal will have been melted and absorbed by the molten mass in the crucible and the oxid will float to the surface, 21, of the mass. As more metal is added to the amount already in the crucible, and the surface rises, the oxid will come near to the opening 6, where it can be skimmed off.

This process may be modified to recover the metal of borings and scraps of such metals as readily oxidize near the melting point. A quantity of the metal is first melted, after which the borings and scraps are forced down into the molten mass through the tube 7. This tube prevents the oxidizing flame reaching the finely divided metal, which really does not melt until under the surface of the main mass. The tube 7, or its equivalent, can be filled with an unoxidizing or neutral atmosphere so as to further protect the metal.

It is sometimes desirable to mix a proper flux, such as ammonium chlorid, with the mixed metal and oxides and with the metal borings before submerging the same in the molten metal.

969,773. Process of Producing Alloys and the Separation of Metals. PERCY F. COWING, of New York, N. Y., Sept. 13, 1910.

This process relates to the electrolytic treatment of alloys containing principally copper and nickel, and in certain cases iron, the object being to separate the copper and to recover nickel and iron economically and by simple operations. The nickel and iron are obtained in the form of a compact thick deposit on the cathode.

In carrying out the process the original alloy is placed as an anode in an electrolytic bath, which, upon the passage of the current, has the property of dissolving substantially all of the metals of the original alloy. That portion of the electrolyte forming the resultant solution is then treated in such manner as to replace the copper by iron, or nickel and iron, and nickel and iron are thereafter electrolytically deposited.

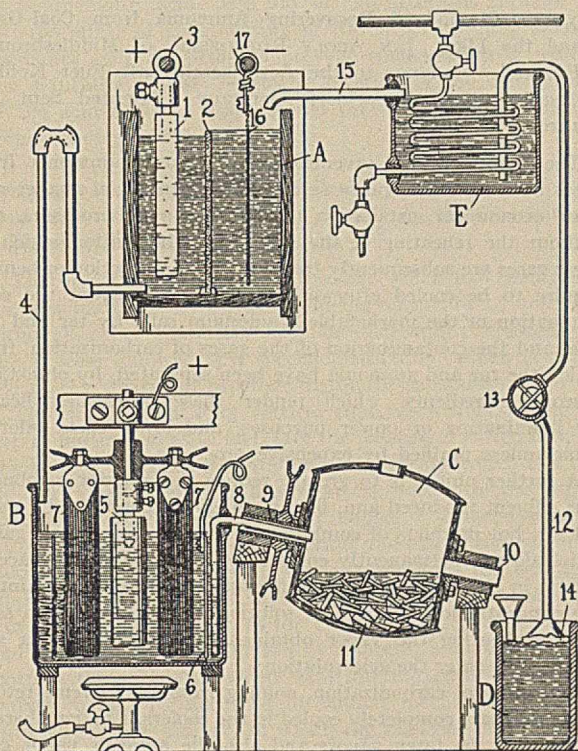
A warm, rather concentrated, substantially neutral aqueous



solution of nickel chlorid is a suitable initial electrolyte for carrying out the process. The very soluble nickel chlorid dissolved in water forms, especially when hot, an electrolyte of low electrical resistance and permits the use of high current densities.

The first vat, A, comprises an anode compartment and a cathode compartment, wherein the copper-bearing alloy 1 to be treated is dissolved as the anode, and nickel and iron are deposited on the cathode. The anode and cathode compartments are separated by a diaphragm, 2. The rest of the apparatus is for modifying the copper-containing solution which is withdrawn from the anode compartment of this vat, A, and for discharging it, after proper treatment for the removal of the copper therefrom, into the cathode compartment thereof. The anode and cathode and the electrolytes in the two compartments of this vat constitute a divided electrolytic cell in which the alloy to be treated is dissolved and the desired metal or metals obtained. The dividing diaphragm of the vat is of that type which is sufficiently non-porous to prevent

solution to replace the copper. It is not expected that all of the copper in the solution be so replaced. It is obvious that a series of vats for selective deposition of copper may be used. The solution is withdrawn from the vat for selective deposition, or from the last of these vats if more than one is used, through a conduit, 8, which communicates with the interior of a receptacle or series of receptacles, C, wherein the copper still remaining in solution is removed by cementation on iron. It is here shown as a tumbling barrel mounted on an inclined axis by means of hollow trunnions, 9 and 10, the conduit entering through the upper trunnion. This tumbling barrel contains loose pieces or fragments of iron, 11, which it is desired to substitute for the copper remaining in solution after leaving the selective deposition vat. While the substitution is herein being effected by cementation, the tumbling action is of advantage, as it dislodges some of the cemented copper from the iron fragments in the receptacle and prevents their adherence to each other and, by agitation, continually brings fresh solution into contact with the fragments. As already indicated, a series of these cementation receptacles may be used. Sufficient time is allowed during the passage of the solution through the vat for selective deposition, B, and the cementation receptacle C to permit the replacement of the copper. When this replacement has taken place sufficiently, the solution is discharged from the cementation receptacle, or the last of these if more than one is used, through the lower trunnion into a settling tank, D, in which any undissolved material carried over mechanically is allowed to settle. The solution now freed from the salts of copper, and containing the salts of iron, or iron and nickel, substituted therefore, is conveyed from the settling tank to a heating tank, E, through a conduit, 12, by any suitable means, a pump, 13, being illustrated. A filter, 14, can be introduced between the settling tank and the heating tank, if desired. This solution is then conveyed through a conduit, 15, from the heating tank to the cathode compartment of the first vat A, the temperature of the solution being raised to a sufficient degree in the heating tank or in the vat A to maintain in the cathode compartment that temperature of the electrolyte at which the electrolytic process is most efficiently carried on. The bath in this cathode compartment contains nickel and iron in solution. A cathode, 16, is immersed therein upon which metals are deposited from the solution. This cathode is suspended from a negative bus-bar, 17. When the desired amount of metal has been deposited upon the cathode, the cathode is removed. The solution in the cathode compartment is allowed to flow over the diaphragm 2 into the anode compartment, thereby completing the circuit flow. The deposit or the cathode and deposit constitute the produce which my process is designed to secure. By depositing predetermined weights of nickel, or nickel and iron, upon a cathode of known weight and material, as iron, the relative weights of these metals in the cathode and its deposit may be varied as desired.

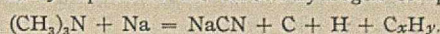


the mixing of the solutions on either side thereof to any appreciable degree, but at the same time has sufficient porosity to allow the electric current to be conducted therethrough by the electrolyte in its pores without too greatly increasing the resistance. The original alloy is connected as the anode to a positive bus-bar 3. When the electric current is passing, the original alloy goes into solution in the electrolyte in the anode compartment of the vat A. The solution thus formed is carried from the bottom of this compartment through a conduit, 4, into a second electrolytic vat, B, in which selective deposition is carried on. The anode 5 in this vat is composed of nickel or iron or both, and the cathode 6 may be composed of copper; but this is not essential. The electrolyte is preferably agitated by stirrers, 7, and may be heated. By maintaining a sufficiently low impressed voltage between the anode and cathode, the copper-containing solution from the anode compartment of the first vat A in its passage through the second vat, B, is caused to deposit copper selectively upon the cathode, 6, an equivalent amount of the anode 5 going into

969,885. Process of Making Sodium Cyanid. OTTO LIEBKNECHT, of Frankfort-on-the-Main, Germany. Assignor to the Roessler & Hasslacher Chemical Co., of New York. Patented Sept. 13, 1910.

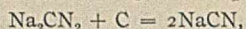
This invention relates to a process of making sodium cyanid from metallic sodium by reaction with trimethylamin.

The patentee has discovered that if dry vapors of trimethylamin are passed into highly heated molten metallic sodium the formation of sodium cyanid takes place in connection with the formation of various carbon-hydrogen compounds, carbon and hydrogen, as may be illustrated by the following equation, in which  $C_xH_y$  represents the carbon-hydrogen compounds:

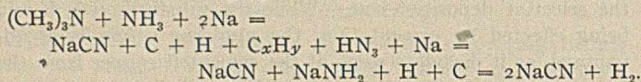


The sodium cyanid may subsequently be freed from the

carbon by any of the usual mechanical means as, for instance, by filtering the mixture, the carbon-hydrogen compounds and the carbon-hydrogen compounds passing off at the high temperature used, as gases. The formation of carbon in this free state may also be avoided chemically, for example, by the addition of disodium of cyanamid which combines with the carbon to form sodium cyanid, according to the equation



disodium cyanamid being added until a sample does not show any more carbon; or ammonia may be added to the trimethylamin whereby a separation of carbon is prevented, in accordance with the following equations:



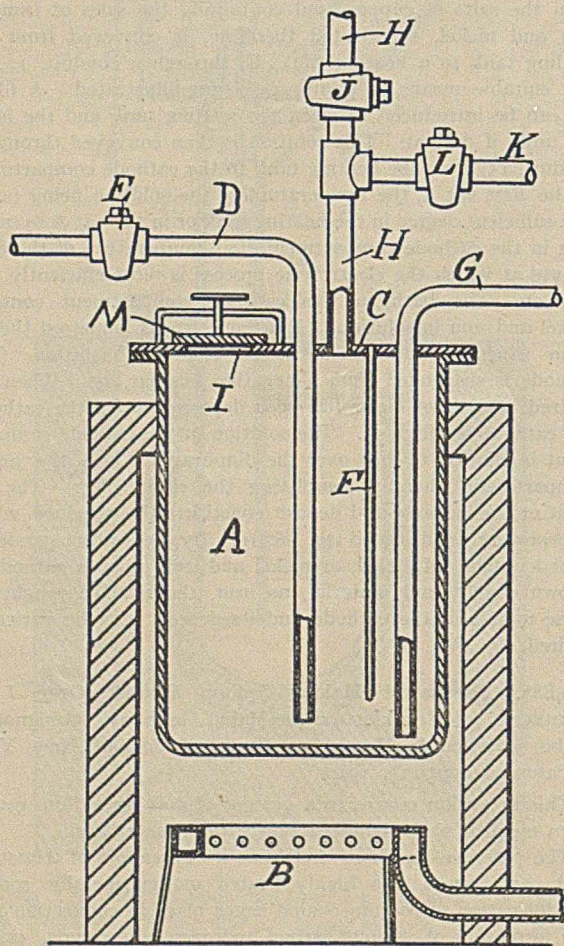
The formula  $\text{C}_x\text{H}_y$ , as before stated, represents the carbon-hydrogen compounds which at the high temperature used exist in gaseous form and pass off as a gas, together with the hydrogen.

The accompanying illustration shows diagrammatically a form of apparatus with which the process may be practiced.

In the drawing, A represents a pot, preferably of iron, set in a suitable furnace.

B is a burner for heating the furnace and pot and may be of any suitable construction, preferably in the form of a gas burner.

The pot A is provided with a cover, C, in which are suitable



apertures through which intake pipe D, exhaust pipe H, thermometer pipe F, and pressure pipe G communicate with the interior of pot A.

I is a hand-hole closed by a suitable cover, M.

Intake pipe D is provided with a cock, E, and exhaust pipe H is provided with a cock, J. Connecting with the exhaust pipe H is a pressure pipe, K, controlled by a cock, L, and leading to any suitable source of pressure.

In practicing, the process in connection with apparatus of the above type, 7 kilograms of sodium may be heated in pot A to about 700° Centigrade, the temperature being determined by a thermometer inserted in thermometer pipe F, and about 18 kilograms of trimethylamin passed in through intake pipe D. Cock L should be closed and cock J opened so that the waste gases may pass off through exhaust pipe H. When the reaction is finished and sodium cyanid formed the cocks E and J are closed, cock L opened and pressure applied through pipe K, which forces the finished charge up through pipe G to a suitable filter. The sodium cyanid formed, as stated, contains free carbon, which may be removed by filtration or otherwise, as stated above, so that the resulting sodium cyanid will be white.

969,907. Method of Recovering Ammonia from Coal-Gases and the Like. JAN ADOLF ROELOFSEN, of Middlesbrough, England. Assignor to the Actiengesellschaft Fuer Kohlendestillation, of Düsseldorf, Germany. Patented Sept. 13, 1910.

The object of this invention is to recover ammonia from gases as a salt, preferably sulfate, by means of a smaller and more economical installation than in the prior processes, and without the reheating of the gases of carbonization which, if these gases are subsequently treated for the extraction of benzol, require to be cooled afterward; further, to prevent the contamination of the marketable ammonium salts by tar and the like, and the contamination of the gases of carbonization, from which the tar and ammonia have been separated, by obnoxious gaseous ingredients, which render these gases inapplicable for illuminating or power purposes, and lower their calorific value unless purified by expensive processes.

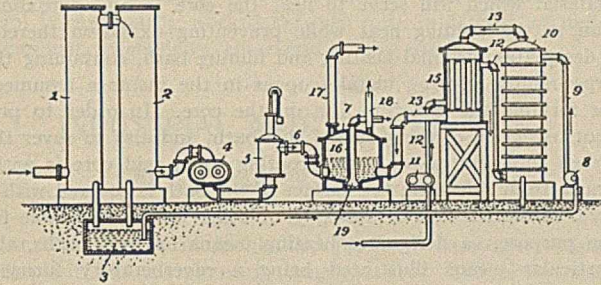
A further object is to greatly reduce the amount of obnoxious effluent produced and, finally, to effect economics by using hot air, hot products of combustion, or other hot neutral gases, which do not permanently combine with ammonia, in place of steam, in the operation of the ammonia still, and to maintain the concentration of the acid solution of the ammonium salts by superheating the vapor obtained from the ammonia still before they enter the acid solution.

The gases of carbonization, coming from coke ovens, retorts or the like, are completely cooled in the coolers, 1 and 2, thereby condensing the tarry vapors and also the aqueous vapor containing most of the ammoniacal and other fixed salts, as well as some free ammonia; these oily and aqueous condensates are collected and saturated by gravity in tank 3. The gases of carbonization, after having thus been cooled, pass through a gasblower, 4, and a Pélouze tar-extractor, 5, which separates the residual tarry vapor, and without being reheated, are conducted through pipe 6 into a lead-lined saturator, 7.

The condensed ammoniacal liquor from tank 3 is delivered, by means of pump 8, through pipe 9, into ammonia still 10, an alkali, such as lime, being also introduced, and into which still, the blower 11 forces, through pipe 12, and superheater 15, a current of hot air, hot products of combustion, or any other hot neutral gases which do not permanently combine with ammonia. The ammonia thus liberated passes through pipe 13 and superheater 15 into saturator 7.

The saturator 7 is divided into two compartments by means of the diaphragm 16, which dips into the acid solution of ammonium salts contained in the saturator 7. The gases of carbonization enter into one of these compartments through the perforated end of pipe 6, pass up through the acid solution, and leave the saturator, after having thus been freed from resid-

ual ammonia, by means of pipe 17. The vapors from the ammonia still 10, enter the other compartment of the saturator 7 through the perforated end of pipe 13. The contained ammonia is thereby absorbed, while the aqueous vapor and noxious gases which may contain hydrogen sulfid and other gaseous sulfur compounds, hydrocyanic acid, carbonic acid and traces



of hydrochloric acid pass out of the saturator through pipe 18; the effect of superheating of the vapors coming from the ammonia still 10 being to prevent condensation of water in the saturator 7, and thereby a lowering of the concentration of the acid solution which would prevent the ammonium salt from separating in the solid state.

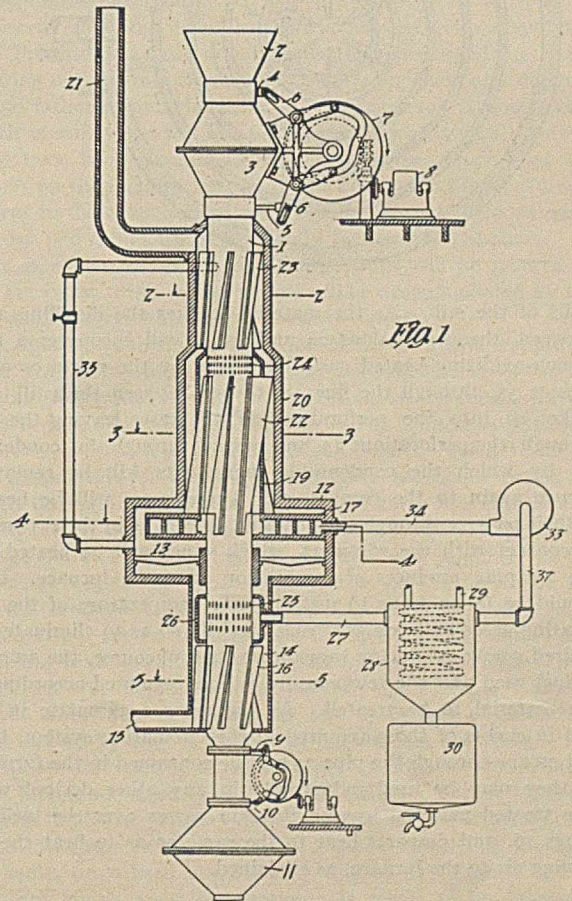
970,364. Process of Distilling Coal. JONAS W. AYLSWORTH, of East Orange, and FRANK L. DYER, of Montclair, New Jersey. Patented Sept. 13, 1910.

This invention consists in a process which has been devised for eliminating the smoke-producing elements of soft coals, although other materials may be successfully treated, since the process followed is a special distillation, which is applicable to the treatment of other distillable substances.

The still 1 is a long vertical tube, preferably flaring from top to bottom, so as to facilitate the passage of material through the same. At the top is a hopper, 2, beneath which is a buffer hopper, 3, gate valves, 4 and 5, at the bottom of the hopper 2 and at the bottom of the buffer hopper 3, respectively, being alternately operated so as to permit successive charges of solid material to enter the still, without breaking the seal at the upper end. These gate valves are intermittently operated by any suitable mechanism, as for example, the levers 6, 6, actuated by a cam 7, slowly driven by a motor, 8, as shown. At its bottom the still is provided with intermittently operated gate valves, 9 and 10, actuated by similar mechanism, and opens into a receiving bin, 11, in which the material may cool somewhat before being discharged into the atmosphere.

Surrounding the still is a furnace, 12, having grate bars, 13, and supplied with air through a flue, 14, which envelops the still, and having an inlet, 15, leading to the atmosphere. A series of inclined wings, 16, made preferably of copper, extend through the walls of the still and project into the flue 14 and within the still (see Fig. 5), whereby the material passing through the still will be checked in its descent, and pressure on the still walls, due to the height of the column of material, will be relieved and the material will be more or less agitated. The principal function, however, of these inclined wings, is to act as heat conductors for carrying off the heat from the material into the flue 14, so as to heat the air passing through the flue to the furnace. This acts as a regenerative device, heat being abstracted from the treated coal, or other material, and being imparted to the incoming combustion air. The inclined wings are, therefore, made preferably of copper or aluminum, so as to readily conduct heat. Mounted in the furnace 12 is a drum, 17, having a spiral flue, 18, which opens into a space between the still and a jacket, 19. Outside of the jacket 19 is a second jacket, 20, acting as a stack for carrying off the products of combustion from the furnace 12 and connect-

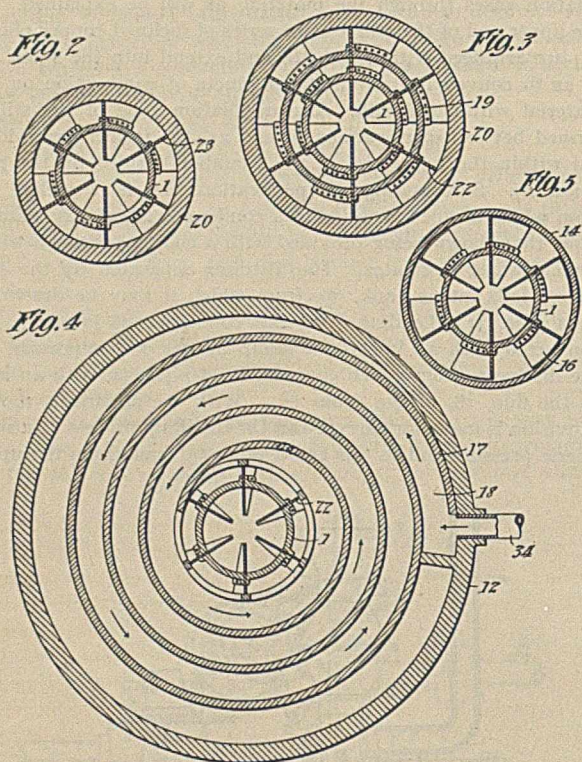
ing at its upper end with a stack flue, 21. Inclined wings 22, similar to the wings 16, extend into the still and pass through the wall 19, so that heat from the products of combustion passing through the stack will be conducted and imparted to the material undergoing distillation. The inclined wings 22 also serve to check the passage of the material through the still, to partially support the same, and to keep the material in movement, so as to permit the more ready passage of the distillation gases through the material, as will be explained. At the upper end of the still a third series of inclined copper plates, 23, are arranged within the still and extend into the stack 20, so as to convey heat from the products of combustion on the material within the still. The distillation zone of the still is formed between perforations 24 and 25, the former being located within the upper end of the chamber, formed by the partition 19. Surrounding the perforations 25 is a jacket, 26, from which a pipe, 27, leads to a condenser, 28, of any suitable type, that shown being provided with a coil, 29, through which cooling water circulates. The distillate separated by the condenser falls into a tank, 30, from which it may be drawn off by a pipe, 31. Leading from the condenser 28 is a pipe, 32, connected with an inlet to a pump blower or compressor, 33, the discharge from which connects by a pipe, 34, with an inlet to the flue, 18, of the drum 17. With the apparatus shown, provision is made for carrying on the distilling process at atmospheric pressure and to this end a pipe, 35, leads from the upper



part of the still to the furnace 12, so as to carry off any excess gases accumulating in the still. The operation of the still may be carried on below or above atmospheric pressure if desired.

In carrying the process into effect, and assuming the apparatus to be used for the treatment of soft coal, so as to

eliminate the smoke-producing ingredients therefrom, we charge the still with the material through the gate valves 4 and 5, whereby the material will slowly progress through the still, being withdrawn at the lower end through the gate valves, 9 and 10. As the material passes over the inclined wings 23, heat will be imparted to the same, since these wings will be kept hot by the products of combustion from the furnace 12, and, of course, the material will also be heated through the



walls of the still. As the material reaches the distilling zone between the perforations 24 and 25, it will encounter a flow of non-oxidizing heated gases circulated by the pump or compressor 33, through the flue 18, thence between the still and jacket 19, into the perforations 24, the gases leaving the still through the perforations 25, and passing through the condenser 28, by which the condensable ingredients will be removed, thence again to the compressor. These gases will be heated in their passage through the drum 17, as well as in their passage in contact with the wings 22, which wings will be heated by the escaping products of combustion from the furnace. Care should be taken so as to maintain the temperature of the circulating gases between 550° and 800° F., so as to eliminate the desired smoke-producing ingredients, but, of course, the amount of fuel used and the temperature will be regulated according to the material to be treated. As the gases accumulate in the still in excess of those required in the circulating system, they will escape through the pipe 35 and be consumed in the furnace, or they may be used industrially in any other desired way. The treated material leaving the still passes over the inclined wings 16, and imparts heat to the same so as to heat the incoming air to the furnace, as explained.

971,136. Process of Producing Compound Metal Bodies. JOHN FERREOL MONNOT, of New York, N. Y. Assignor to Duplex Metals Company, of New York. Patented Sept. 27, 1910.

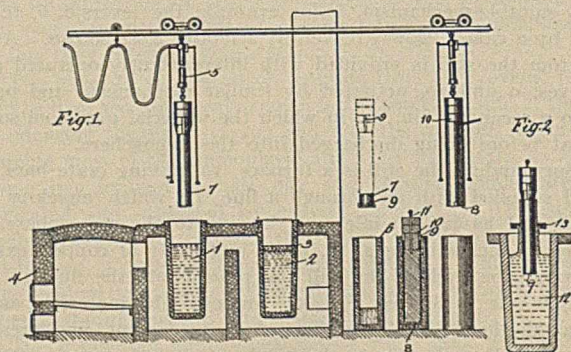
This invention relates to a process of producing compound metal bodies, such articles comprising ingots, plates, sheets, rods, tubes, wire and other articles, composed of layers or

strata of unlike metals welded together (such as iron or steel, autogenously and completely welded to copper, silver, gold, brass, bronzes, aluminum, etc.).

In the accompanying illustration, in which is shown apparatus for carrying out this process, 1 designates a heating bath termed, ordinarily, the flux bath, containing molten cuprous chlorid, borax, fluor spar, cryolite, or other fusible material which will serve to heat the core to an approximation to the welding heat while preventing oxidation thereof. 2 designates the final heating and filming bath, containing the super-molten coating metal (copper in the instance assumed) for forming the cohering film on the core. In order to prevent oxidation of the metal of this bath, and also to cover the filmed core with a protecting coating when said core is withdrawn from this bath, the surface of the bath is covered with a layer of molten flux or borax, 3, or other material suitable for the purpose. 4 designates heating means for said baths, the particular means illustrated being a reverberatory furnace, though in lieu thereof any suitable heating means may be employed. 5 designates elevating and traversing means for handling the core, the particular means illustrated being power hoist and traveling crane. 7 designates the core.

In carrying out the process, the core 7, which has first been cleaned so that it presents an actual metallic surface entirely uncontaminated and chemically clean, is immersed in the heating bath 1, preferably of cuprous chlorid and allowed to remain therein until heated to the proper temperature.

The core, when heated in the flux bath to the proper degree, is withdrawn therefrom and immersed in the filming bath 2, the flux on the surface of the core rising to the surface of the bath as the core descends, thus exposing an absolutely clean surface to the supermolten coating metal, excluding air from the surface of the core, and avoiding the carrying of the air down into the molten metal. Said molten metal being in the supermolten condition, above described, in which it appears to be chemically active, raises the core to the welding temperature (if said core is not already at that temperature), and what may be an alloy film is formed next the core, the molten metal apparently uniting chemically or by a solvent action with the steel. The core so filmed is then withdrawn from bath 2, dipped in a bath of molten metal, 12, maintained near its point



of solidification, and then withdrawn from such bath, 12, with the adhering mass of metal from said bath, 12, any excess of metal from said bath being scraped off if desired by passing the ingot through a suitable scraping gage, 13.

Instead of dipping the filmed core into a bath of molten metal maintained at such temperature that upon withdrawal a considerable mass of metal from such bath will adhere thereto, the filmed core may be placed within an ingot mold, 6 (see Fig. 1), such mold having preferably been charged previously with the required quantity of molten coating metal required for forming the predetermined thickness of coating, such molten metal rising up around the core as it ascends into the mold

and completely filling the space between the core and the inner surface of the mold. Union between the molten metal and the film coating takes place very rapidly, and such union is assisted and indeed compelled by the pressure due to contraction of the coating during and after solidification, reinforced usually by pressure due to passing the ingot between the rolls or by hammering or the action of a press. A complete weld and sound coating is thereby formed.

After the formation of the coating as thus described, the ingot is customarily worked between rolls, in a press, or under a hammer to condense the metal of the coating.

## INDUSTRIAL AND TRADE NOTES.

### NEW JAPANESE PAPER MILL.

[From Consul-General Thomas Sammons, Yokohama.]

At Tomakamai, in Hokkaido, Japan, a \$4,000,000 paper-mill plant was completed in August, 1910. This is the most important individual commercial enterprise of this nature in the Far East. Only one foreigner is employed, being an American, who was sent out to assist in installing the paper-making machinery.

The total cost of the machinery of this modern plant is placed at \$1,000,000. Of this all but 3 per cent., it is stated, was purchased in America. An inspection of the plant shows that all the heavy machinery—in fact, practically all of the machinery proper—was manufactured by well-known paper-mill machinery establishments in the United States, mostly by the Bagley Sewall Company, of Watertown, N. Y. A few of the smaller parts, including shafting, were made in Japan, and constitute the 3 per cent. referred to.

The cost of the electrical machinery in this plant, all of which was imported from America, is placed at about \$1,000,000. This does not include the cost of the reservoir and power-house plant, located 14 miles distant, at Lake Shikatsu. This lake is 800 feet above sea-level, and as the paper plant at Tomakamai is only a few feet above the near-by Pacific Ocean, excellent power facilities have been provided. Already a 15,000 horse-power plant has been developed, to which 7,500 horse-power will be added. As the paper plant requires only 8,000 horse-power, it is proposed to sell the surplus (14,500 horse-power) for commercial purposes.

The daily output of the Tomakamai paper-mill plant is upward of 70 tons per twenty-four hours. This is considerable over 50 per cent. of the total consumption of news and book paper in the Empire of Japan. Moreover, it is estimated that the timber supply available for this plant will not be exhausted for half a century or more. Some experts claim that the timber supply in this case will not be used up for over one hundred years. The timber areas include state and railroad lands in addition to privately owned tracts.

Those concerned in this enterprise, being all Japanese, state that the selling price on the tonnage basis of their product will, for the present, be placed at 2.5 to 3 cents per pound. It is evident that this will net a handsome profit, and that the reduced cost of production will enable the Japanese manufacturer to cut prices if necessary to secure a ready market for the output. It is also evident that Japan will export large quantities of paper in the future, the wages paid the ordinary laborer in the Tomakamai plant being approximately \$15 per month.

The Tomakamai plant was planned by a Japanese subject, Mr. Naoki Takata, chief engineer and manager of the manufacturing and construction departments of this plant. He has been engaged in the manufacture of paper in Japan for sixteen years, and for six years has directed all of his time and energies to evolving this new industrial property. He has visited the paper manufacturing centers of the world, and is of the opinion

that the manufacture of paper will become an important industry in Japan.

Mr. Takata states that he has heretofore pointed out to foreign capitalists the inviting possibilities of this industry, but thus far with no practical results, no foreigners becoming interested.

After examining modern American paper-mill plants, Mr. Takata decided that if sufficient water power could be developed in Hokkaido, where coal and timber are plentiful, this industry would prove successful. After locating the necessary water power possibilities at Lake Shikatsu his plans were approved by Japanese capitalists.

Mr. Takata has placed in the Tomakamai plant his own personal inventions for washing the pulp. This plant is also provided with another Japanese invention, the Miyabara naval boiler, of which there are 10, each of 300 horse-power, or a total of 3,000 horse-power boiler capacity.

The electrical machinery, all of American manufacture, includes five 750 horse-power motors; four 450 horse-power motors; forty-three from 100 to 200 horse-power motors.

The floors are all concrete and the buildings are of brick.

### PHOSPHATE DEVELOPMENTS IN OCEANIA.

[From Consul Julius D. Dreher, Tahiti, Society Islands.]

As previously reported by this consulate, deep-sea moorings were purchased in San Francisco by the French Phosphate Company, of Oceania, to replace those lost in the wrecking of the new Norwegian ship, *Ocean Queen*, at the island of Makatea, on September 16, 1909.

Heavier moorings being necessary, another Norwegian ship, the "*Promise*," has succeeded, under the superintendence of the mooring master from Ocean Island, in laying these at a distance of 150 fathoms from the shore and in water 225 fathoms deep, which is said to be the greatest depth at which such moorings have ever been anchored. To prevent the slipping of the anchor on the inclined bottom of the ocean, the immense buoy, 11 feet in diameter and 16 feet high, is securely held in position by two cables which are firmly fastened to the shore. As there is no harbor at Makatea, large ships will be moored to the big buoy, while other vessels will be accommodated by the four smaller boys which support the two cables.

A difficult problem at Makatea is an adequate supply of labor. As native labor is scarce, it was supposed from the beginning that when the operations at Makatea reached a certain stage it would become necessary to import Chinese or Japanese laborers, as has been done at Ocean Island. As a result of the importation of Chinese to cultivate sea-island cotton in Tahiti during the civil war in America, there are now in this colony about 800 of these industrious people. They control a large share of the retail trade, of curing and shipping vanilla, or raising vegetables for market, and of baking bread for the natives throughout the island.

Recently a special tax has been imposed on Chinese and also upon the value of buildings occupied by them for business purposes, but still they continue to come to this island, 177 having arrived since the first of the year, and 67 by way of San Francisco and 27 by way of Sydney within the last six weeks. The Phosphate Company entered into negotiations to obtain labor from Japan, but it has been found impossible to import Japanese laborers under a contract binding them to deportation in a specified number of years. In the last few months 27 Japanese mechanics have been imported by way of San Francisco, and they are giving great satisfaction. It seems to be necessary for the company to import ordinary laborers from Japan.

The total working force on Makatea, including superintendents, clerks and craftsmen, numbers a little more than 300, for all of whom this company has to furnish board and lodging.

The island of Makatea, which is situated in the extreme

northwestern part of the extensive Tuamotu Archipelago, has an area of about 10 square miles, one-fifth of which is covered with deposits of phosphate estimated at 10,000,000 tons, of an average grade of 82 per cent. So far as is known here, nothing further has been done to develop the phosphate discovered on Henderson (or Elizabeth) Island, a British possession lying 300 miles east of the limits of this consular district, as was reported by this consulate in *Daily Consular and Trade Reports* of March 12, 1909. As that island is of an elevated coralliferous limestone formation like Makatea, Ocean, Pleasant, and Angaur, it is highly probable that further investigations will show conclusively that the deposits on Henderson are large and valuable.

The world's annual production of phosphate rock is about 5,000,000 tons, the United States being the largest producer, with an annual output of more than 2,000,000 tons. Tunis, which ranks second, produced phosphate of a rather low grade to the amount of \$6,117,000 in 1908. In 1909 the great Gafsa Company, which owns its own railroad, mined 907,000 metric tons (metric ton = 2,204.6 pounds). It pays a large dividend on its capital of \$7,750,000, as is shown by the fact that the stock of this Tunisian company is selling in Paris at a premium of more than 600 per cent. The Pacific Phosphate Company, of London, which owns deposits of 50,000,000 tons of high-grade phosphate on Ocean and Pleasant islands, is mining some 250,000 long tons a year at a profit of more than 50 per cent. on its capital stock of \$1,216,600. A German company has recently begun to mine phosphate to a considerable extent on the island of Angaur, which lies in the western part of the Carolines at no great distance from the Philippines.

As the Pacific Phosphate Company, which has not yet mined 4 per cent. of its immense deposits, could largely increase its annual output; as the production at Angaur will become large in a few years; as Makatea ought to send to the markets of the world several hundred thousand tons of phosphate in the near future; and as the exploitation of deposits in Tunis and Algeria, as well as in other countries, is being carried on actively and extensively, the question arises as to whether the production of phosphate may not soon exceed the world's demand.

But it may be said with some degree of confidence that the increasing attention given to scientific agriculture in most countries of the civilized world will result in a demand for larger and larger quantities of the commercial fertilizers manufactured from phosphate rock, and hence it may be regarded as fortunate that the world's supply of phosphate is being increased from year to year. Even if competition should cause a considerable reduction in the price of phosphate, there would still remain a fair margin of profits for the great operating companies.

#### A NEW ALLOY.

[From Consul Charles N. Daniels, Sheffield, England.]

The Sheffield newspapers announce the discovery and patenting by a Sheffield concern of a new metal or alloy to which the name of "Duralium" has been given, and that it will be manufactured in Birmingham and put upon the market in October.

They believe this alloy to be superior to anything heretofore discovered. Being slightly heavier than aluminum and as strong as steel, it can be rolled, drawn, stamped, extended, or forged at suitable temperatures; it is less corrosive than other high aluminum alloys under the usual corrosion tests, and it is only one-third the weight of brass.

It is expected that the new metal will find a steadily increasing demand for aviation uses, while it is likely to prove an important factor in the construction of motor cars, owing to the difficulty of securing aluminum castings sufficiently reliable not to break under the strain of sudden jars and shocks caused by quick stoppages, changes of speed, and jolts upon bad roads.

#### HIGH-VOLTAGE POWER FROM NIAGARA.

[From Consul Harry A. Conant, Windsor, Canada.]

The feasibility of the Ontario hydro-electric commissioner's plan of transmitting electric current from Niagara Falls at a high voltage, on which Windsor's hope of securing current from the government line depended, has been thoroughly demonstrated by severe tests which have been made by the commission on the line between Niagara Falls and Dundas.

Power at a voltage of 165,000 was turned on to the wires, and it is declared that not a spark was noticeable, although the test was 55,000 volts higher than the voltage which will regularly be used.

It is said that the first time the experiment of transmitting electricity at 165,000 volts has been successfully carried out.

The success of the test indicates that the hydro-electric commission has overcome the last of the difficulties in the way of carrying out its power project, and it is a settled fact that municipalities will get power at a lower rate than was at first promised.

#### RESINITE AND BAKELITE INVENTIONS.

The inventor of bakelite, Dr. L. H. Baekeland, of New York, with a view to preventing infringements, requests the Bureau of Manufacturers to invite attention to the fact that bakelite, which, as has been stated in the *Daily Consular and Trade Reports*, has some analogy with the German product called resinite, is protected in the United States by patents Nos. 939,966, 941,605, 942,699, 942,700, 942,808, 942,809, 942,852, 949,671, 954,666, 957,137, to which patents those interested are referred.

#### GUAYULE RUBBER SUPPLY IN MEXICO.

[From Consul Luther T. Ellsworth, of Ciudad Porfirio Diaz.]

Robert W. Dowe, United States collector of customs for the ports on the Mexican border between San Antonio and El Paso, Tex., verifies my statement in *Daily Consular and Trade Reports*, of August 13th, regarding guayule rubber shrub supply in this consular district by saying:

"Most of it is being used by the American manufacturers of automobile tires. A number of big guayule factories in Coahuila and Chihuahua are working night and day.

"It has been estimated that the value of the guayule shrub now standing on the lands of Coahuila is \$65,000,000. There is as much more in Chihuahua and other mountainous Mexican states. The Mexican owners of guayule land are seeing that the shrub is gathered in such a way that the supply will not be exhausted. Plants are being left in sufficient quantities to reseed and replace those which are cut down and taken to market. The demand for the shrub is so great that thousands of men are employed in gathering it.

"The demand for rubber was never so active and the supply is by no means equal to the demand. The Mexican factories have more orders than they can fill, even by operating day and night, and others are being built. The land on which the guayule grew a few years ago was practically valueless. It is now in big demand. With proper conservation it is believed Mexico has an inexhaustible supply."

#### DRIED BANANAS IN EUROPE.

[From Consul-General Robert P. Skinner, Hamburg, Germany.]

Dried bananas and what are known here as dried banana chips have been offered for sale in Germany with increasing commercial success for over two years.

It is impossible to obtain reliable statistics as the import figures are included with those relating to fresh bananas, both the dried and the fresh fruit being free of duty. Importers consulted state that they have been able to sell without diffi-

culty all the bananas shipped to them, and that the difficulty has been rather to obtain the goods than to find a market.

The consumers of dried bananas and banana chips were originally vegetarians who discovered attractive ways of serving them, but there is now a considerable demand for the dried fruit in all classes of society and one susceptible of being extended materially.

Thus far the best dried bananas have been received from Jamaica, which also seems to be the chief country of exportation. The whole fruit is shipped in wooden cases weighing 25 kilos, or 56 English pounds, and chips are received in sacks. Importers are paying 25 marks (\$5.95) per 100 English pounds for goods delivered in Hamburg, at which price a fairly remunerative business can be carried on. Any substantial increase in the wholesale price would react unfavorably upon the consuming market. (A list of Hamburg firms who might be addressed in regard to dried fruit may be secured from the Bureau of Manufacturers.)

#### ENGLISH SLAG PAVING BLOCKS.

[From Vice-Consul H. Nixon, Newcastle-on-Tyne.]

Scoria bricks, or slag paving blocks, have been manufactured in the Middlesbrough district for many years, and considerable quantities have been shipped to the United States since 1894. (A shipload was recently received at Washington, D. C.)

The bricks are manufactured from molten slag from the blast furnaces. The slag is drawn from the furnace into iron caldrons lined with fire-brick. These are mounted on bogies, for transmission to the brick plant. The slag is poured from these caldrons into iron molds. These molds are made with a hinged bottom and are mounted on the circumference of a circular iron framework. This revolves and allows the molds to be filled separately. As the bricks solidify they are removed and placed in a beehive oven, where the residual heat anneals the whole of the brick.

#### AIR-NITRATES IN THE UNITED STATES.

Mr. W. S. Lee, Jr., Vice-President of the Southern Power Company, of Charlotte, N. C., has gone to Europe, where he will spend several weeks in the interests of the plants his company proposes to establish for the manufacture of air nitrate fertilizers. The *Charlotte Chronicle* states that the contract for the first plant, which will be of 4,000 horse-power, has already been let, and some of the machinery for it is being manufactured abroad at this time. The Southern Power Company has acquired the right to use the "Geneva process" of abstracting nitrogen from the air and also rights to use a newer process, and it is in connection with the latter that Mr. Lee goes abroad.

The first plant to be built by the Charlotte Company will be used to a great extent for experimental purposes, and the second, which will require 24,000 horse-power to operate, will be used for the manufacture of nitrate fertilizers on a large scale

not contain more than 300 milligrams of tin per kilogram, or salts of tin equivalent thereto. When the amount of tin, or an equivalent amount of salts of tin, is greater than 300 milligrams per kilogram, entry of such canned goods packed subsequently to January 1, 1911, will be refused, and if found in interstate commerce proper action will be taken.

It is the opinion of the board that the trade will experience little hardship in adjusting itself to this condition, as the results of examinations made by the Bureau of Chemistry of various types of canned goods indicate that in a very large majority of cases inconsiderable quantities of tin are found, well within the limit herein set.

H. W. WILEY,  
F. L. DUNLAP,  
GEO. P. McCABE,

*Board of Food and Drug Inspection.*

Approved:

JAMES WILSON,  
*Secretary of Agriculture,*

WASHINGTON, D. C., September 22, 1910.

#### NOTICE OF JUDGMENT NO. 539, FOOD AND DRUGS ACT *Adulteration and Misbranding of Turpentine.*

On or about June 11, 1909, the Gulf Manufacturing Company, Limited, a corporation, New Orleans, La., shipped from the State of Louisiana to the State of Texas a consignment of alleged spirits of turpentine, the packages containing which each bore the following label: "Crescent brand spirits of turpentine. Guaranteed by Gulf Manufacturing Company, Ltd., under the Food and Drugs Act of June 30, 1906, Gulf Manufacturing Company, Ltd., New Orleans, La."

Samples from this shipment were procured and analyzed by the Bureau of Chemistry, United States Department of Agriculture, and as it appeared from the findings of the analyst and report thereon that the product was adulterated and misbranded within the meaning of the Food and Drugs Act of June 30, 1906, the Secretary of Agriculture afforded the Gulf Manufacturing Company, Limited, and the party from whom the samples were procured, opportunities for hearings. As it appeared after hearings held that the said shipment was made in violation of the act, the Secretary of Agriculture reported the facts to the Attorney-General, with a statement of the evidence upon which to base a prosecution.

In due course a criminal information was filed in the Circuit Court of the United States for the Eastern District of Louisiana, charging the above shipment, and alleging that the product so shipped was adulterated, in that mineral oil had been added to the so-called turpentine, and that the product differed from the standard of strength, quality, and purity of oil of turpentine as determined by the test laid down in the United States Pharmacopoeia or National Formulary official on the date of said shipment, the words "Spirits of Turpentine" occurring in the label above set forth being synonymous with and used indiscriminately in this country to designate the same drug as the name oil of turpentine just mentioned; and in that the strength and purity of said product fell below the professed standard and quality indicated by the label aforesaid; and further alleging that the product was misbranded, in that the label above set forth was false and misleading, and such as to deceive the purchaser into believing that the contents of said packages were pure spirits of turpentine, whereas in truth and in fact said contents were not pure spirits of turpentine but a mixture of spirits of turpentine with mineral oil; and in that said label was meant and intended and calculated to convey, and did convey, the impression that the said drug was pure spirits of turpentine, which was false, since the said drug was a mixture of spirits of turpentine and mineral oil; and in that the product was an imitation and offered for sale under the distinct-

## OFFICIAL REGULATIONS AND RULINGS.

FOOD INSPECTION DECISION NO. 126.

#### *Salts of Tin in Food.*

The attention of the board has been directed to canned goods which contain salts of tin derived from the solvent action of the contents of the package upon the tin coating. Pending further investigations on the question all canned goods which are prepared prior to January 1, 1911, will be permitted to enter and pass into interstate commerce without detention or restriction in so far as their contents of tin salt is concerned. All foods which are canned subsequently to January 1, 1911, will be permitted importation and interstate commerce if they do

ive name of another article, to wit, spirits of turpentine, which it was then and there represented to be by the label, though in truth and in fact it was not spirits of turpentine for the reason that mineral oil had been mixed therewith.

On June 29, 1910, the defendant entered a plea of guilty to the above information, and the court imposed a fine of \$10 and costs.

This notice is given pursuant to Section 4 of the Food and Drugs Act of June 30, 1906.

W. M. HAYS,  
*Acting Secretary of Agriculture.*

WASHINGTON, D. C., July 29, 1910.

NOTICE OF JUDGMENT NO. 550, FOOD AND DRUGS ACT.

*Adulteration and Misbranding of Spirits of Camphor*

On or about February 9, 1909, the Dow & Snell Company, Incorporated, Toledo, Ohio, shipped from the State of Ohio to the State of Michigan a consignment of drug product labeled "Triumph Brand, Pure Spirits of Camphor, manufactured by the Dow & Snell Company, Toledo." A sample from this shipment was procured and analyzed by the Bureau of Chemistry, United States Department of Agriculture, and as it appeared from the findings of the analyst and report thereon that the product was adulterated and misbranded within the meaning of the Food and Drugs Act of June 30, 1906, the Secretary of Agriculture afforded the Dow & Snell Company, Incorporated, and the dealer from whom the sample was purchased, opportunities for hearings. As it appeared after hearings held that the said shipment was made in violation of the act, the Secretary of Agriculture reported the facts to the Attorney-General, with a statement of the evidence on which to base a prosecution.

In due course a criminal information was filed against the said Dow & Snell Company, Incorporated, in the District Court of the United States for the Northern District of Ohio, charging the above shipment and alleging that the product was adulterated in that it was sold under a name "Spirits of Camphor," recognized in the United States Pharmacopoeia, but that the article did not comply with the standard prescribed by that authority and did not have the standard of strength, quality, or purity stated upon the package in which it was contained, and further that the product was misbranded in that the package containing the same was so labeled as to cause the purchaser to believe that the article was spirits of camphor when as a matter of fact it was not spirits of camphor as recognized by the United States Pharmacopoeia, and also that the label in no manner declared the presence of alcohol in the product, when as a matter of fact a quantity of alcohol was found therein.

On June 22, 1910, the defendant entered a plea of *nolo contendere* and the court imposed a fine of \$25 and costs.

This notice is given pursuant to Section 4 of the Food and Drugs Act of June 30, 1906.

W. M. HAYS,  
*Acting Secretary of Agriculture.*

WASHINGTON, D. C., August 5, 1910.

NOTICE OF JUDGMENT NO. 572, FOOD AND DRUGS ACT.

*Adulteration and Misbranding of Gum Tragacanth.*

On or about September 3, 1909, The National Aniline and Chemical Company, a corporation of the Borough of Brooklyn, New York City, shipped from the State of New York to the State of New Jersey a quantity of a drug product labeled "Powdered Gum Tragacanth."

Samples from this shipment were procured and analyzed by the Bureau of Chemistry, United States Department of Agriculture, and as the findings of the analyst and report thereon indicated that the product was adulterated and misbranded within the meaning of the Food and Drugs Act of June 30, 1906, the Secretary of Agriculture afforded said National Aniline and

Chemical Company and the dealer from whom the samples were procured opportunities for hearings. As it appeared after hearings held that said shipment was made in violation of the act, the Secretary of Agriculture reported the facts to the Attorney-General, with a statement of the evidence upon which to base a prosecution.

In due course a criminal information was filed in the Circuit Court of the United States for the Eastern District of New York against the said National Aniline and Chemical Company, charging the above shipment and alleging the product so shipped was adulterated, in that it differed from the standard of strength, quality, and purity of gum tragacanth as determined by the test laid down in the United States Pharmacopoeia or National Formulary official at the time of investigation, and further alleging that said product was misbranded, in that it was sold and shipped under and by the name of "Powdered Gum Tragacanth" when in truth and in fact it was not "Powdered Gum Tragacanth" but a different article, to wit, "Powdered Indian Gum."

On June 1, 1910, the defendant entered a plea of guilty and the court imposed a fine of \$25.

This notice is given pursuant to Section 4 of the Food and Drugs Act of June 30, 1906.

W. M. HAYS,  
*Acting Secretary of Agriculture.*

WASHINGTON, D. C., August 20, 1910

NOTICE OF JUDGMENT NO. 583, FOOD AND DRUGS ACT.

*Misbranding of Asafetida.*

On or about January 21, 1909, Albert Bruen, William P. Ritchey, and Charles C. Bruen, doing business under the first name of Bruen, Ritchey & Co., New York City, shipped from the State of New York to the State of New Jersey a quantity of a drug product contained in a package labeled: Gum Asafetida (*Foetida ferula*) 1 pound Bruen, Ritchey & Co., New York. Guaranteed under the Food and Drugs Act, June 30, 1906. Serial No. 1063.

Samples from this shipment were procured and analyzed by the Bureau of Chemistry, United States Department of Agriculture, and as the findings of the analyst and report made indicated that the product was misbranded within the meaning of the Food and Drugs Act of June 30, 1906, the said Bruen, Ritchey & Co., and the party from whom the samples were procured were afforded opportunities for hearings. As it appeared after hearings held that the shipment was made in violation of the act, the Secretary of Agriculture reported the facts to the Attorney-General, with a statement of the evidence upon which to base a prosecution.

In due course a criminal information was filed in the Circuit Court of the United States for the Southern District of New York against the said Bruen, Ritchey & Co., charging the above shipment and alleging that the product was misbranded, in that the label above set forth was false and misleading because it represented the product to be 'asafetida' when, as a matter of fact, it contained much foreign material.

On April 5, 1910, the defendants entered a plea of guilty to the above information and the Court imposed a fine of \$50.

This notice is given pursuant to Section 4 of the Food and Drugs Act of June 30, 1906.

W. M. HAYS,  
*Acting Secretary of Agriculture.*  
WASHINGTON, D. C., August 27, 1910.

CORRECTION.

An error was made in reporting the carbonates in the Columbia River water which appeared in the June number of THIS JOURNAL. The CO<sub>2</sub> should read 11.0 parts per million instead of 1.1 as reported.  
C. E. BRADLEY.





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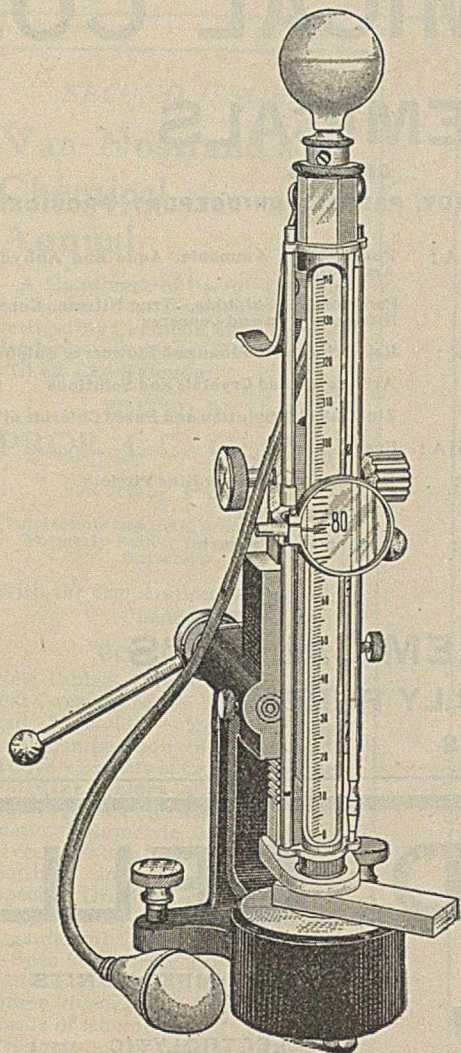
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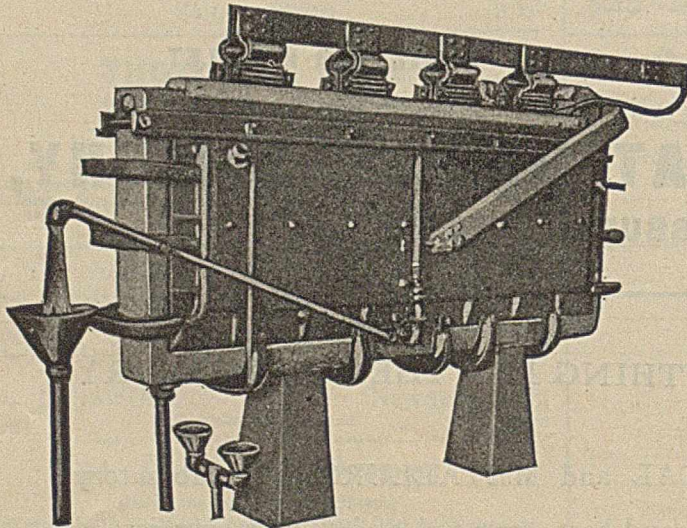
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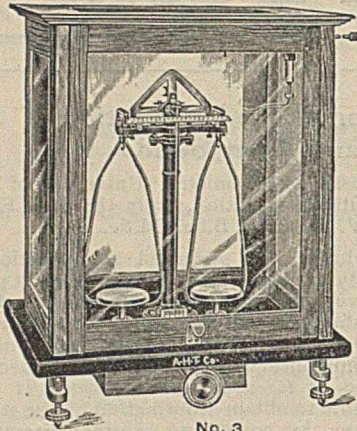
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