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Vol. I.

OCTOBER, 1909. No. 10

## EDITORIALS.

## THE MOISTURE DETERMINATION.

THAT scientific training and thought do not eliminate altogether the judgment based on sentiment, custom and tradition is true, yet nevertheless the man of scientific habits is probably on the whole freer from unreasoned and snap judgment than the average man. The remains of these things in him link him to the past and the conservation of heredity and custom prevent his toorapid evolution.

The necessity for standard methods of analysis worked out and followed in greater or lesser detail by chemists is everywhere recognized. Their greatest drawback is that they tend to make the analyst more of a machine and less of a reasoning being. A method once adopted and generally recognized attains with use and age a peculiar prestige. Results obtained by it, not infrequently go unquestioned and unchallenged by the analyst. Such a method is the moisture determination "at the boiling point of water." This, the least accurate of methods, as ordinarily performed, is considered by the majority of chemists to yield all but perfect results. It is a strange commentary on analytical chemistry that of the two commonest substances on the earth, water and oxygen, the one is ordinarily determined with a very low degree of accuracy and the other not determined at all.

The Association of Official Agricultural Chemists directs that the moisture in fertilizers be determined by heating 2-5 grams of the sample for five hours at the temperature of boiling water. The loss of weight is reported as moisture. The National Fertilizer Association sent out, some time ago, four check samples of phosphate rock which were analyzed by some thirty-two chemists. On sample No. 1 the returns for moisture showed: average 1.10 per cent., maximum 1.50, minimum 0.69, difference 0.81; sample No. 2 average 0.48, maximum 0.73, minimum 0.25, difference 0.48; sample No. 3 average 0.56, maximum 0.87, minimum 0.27, difference 0.60; sample No. 4 average 0.31, maximum 0.60, minimum 0.06, difference 0.54. These are enormous differences for small percentages and of course would not be tolerated in many kinds of analysis.

Instances might be multiplied. Stranger figures than those given above have been yielded in coöperative work on coal, meat extracts, fertilizers, stock foods and organic and inorganic materials of the most varied sorts. The inaccuracies are great enough to warrant attention and invite correction. They are of every-day occurrence in industrial and commercial laboratories. They may be positive or negative, resulting from loss of volatile substances other than water, or from the addition of oxygen by the oxidation of fats and oils and other readily oxidized substances.

In spite of the known insufficiency of the moisture determination by loss, even in the case of check samples sealed with all precautions chemists are accustomed to calculate results to the "dry basis," when differences in other determinations occur, for purposes of comparison. This is worse than folly and adds error to error. It would seem that the inaccuracy of the ordinary moisture determination should be recognized although sanctioned officially. This is the least thing which could be asked or done. If it is necessary to have and use methods of a low order of accuracy, the fact ought to be well understood, clearly recognized and the methods used for what they are worth and no more.

## ORIGINAL PAPERS.

## THE BAG HOUSE AND ITS RECENT APPLICA-TIONS.

## By W. C. EBAUGH.

Received August 20, 1909. The recovery of dust and fume from metallurgical operations is one of the most important problems confronting the works' manager. Escaping fume means loss in efficiency of metal extraction and becomes the cause of injury to surrounding property, thereby resulting in suits for damages or for injunctions. The literature relating to smoke damage1 is quite extensive, and the numbers of lawsuits in California, Montana, Tennessee and Utah growing out of the question need only to be drawn to our attention in order to impress us with the magnitude of the subject. An excellent résumé of the legal status of the smoke question is given in a recent number of the Engineering and Mining Journal.<sup>2</sup>

In his testimony before the Federal Court for the District of Montana Mathewson gave the following summary of the methods that have been tried to remove fume from smoke:<sup>3</sup>

#### A.-Cooling processes.

- 1. Water spray.
- 2. Admission of air.
- 3. Radiation.
- 4. Freezing.

B.-Filtering processes.

- I. Bag house.
- 2. Friction.
- 3. Centrifugal gas cleaners.

<sup>1</sup> See bibliographies in "Rauch-beschädigung," Haselhoff und Lindau (Bornträger, Leipzig); pp. 395-400; Persifor Frazer, Trans. A. I. M. E., **38**, 520-555 (1907); Haywood, Bulletins **89** and **113**, Bureau of Chemistry, U. S. Dept. of Agric.; Haywood, Jour. Amer. Chem. Soc., **29**, 998-1009 (1907); Ebaugh, Jour. Amer. Chem. Soc., **29**, 951-970 (1907); Harkins and Swain, Jour. Amer. Chem. Soc., **29**, 970-998 (1907); **30**, 915-946 (1908); Widtsoc, Bulletin **88**, Utah Agri. Expt. Sta.

<sup>2</sup> Baskerville, Eng. Min. Jour., 87, 884-886 (1909).

<sup>3</sup> Eng. Min. Jour., 87, 87 (1909).

## C.—Electrostatic process. 1. Cottrell process.

For reasons that need not be discussed here the cooling processes enumerated above have not been satisfactory when applied to lead and copper smelter smoke. The Cottrell electrostatic process is still in an experimental stage. Greater success has attended the various filtering processes. The Theisen centrifugal purifiers1 and the Osborn purifiers combine the centrifugal action with water spray, and are successful in removing dust from gases, but experiments made in the Salt Lake Valley indicate that such processes do not remove the fume from lead and copper smelter smoke. The various friction devices, such as Cowper flues, Freudenberg plates, Roesing screens, zigzag flues, etc., also seem to aid in settling the dust, but not in removing fume. It is said that by permitting flue gases to enter expansion chambers<sup>2</sup> the solids can be settled out if the velocity be reduced sufficiently, but here too the fume escapes. In fact when treated by any of the above mentioned methods the minute particles that constitute the fume seem to act more like insoluble gases than like solids.3

For lead and zinc furnaces the bag house has long been recognized as a most efficient means for removing solids from smoke. Beginning with the work of Jones,<sup>4</sup> who used large bags (8x70 ft.) for filtering zinc fume, Wetherill, and Hall,<sup>5</sup> who substituted screens in the side of a house for the bags previously employed as filters, we find Percy<sup>6</sup> suggesting that a fabric might be used for lead fume. The inventions of Lewis and Bartlett (1878, *et seq.*), Ward,<sup>7</sup> Batchman<sup>8</sup> and Bartlett<sup>9</sup> led up to the modern bag house as perfected by

<sup>3</sup> The difference in composition between the dust deposited in a flue at a given point and the fume (probably mixed with dust) drawn from the flue at the same point and collected by bag filtration is shown by the following analyses:

- (a) Fume from copper blast furnaces.
- (b) Dust from copper blast furnaces.
- (c) Fume from copper converters.

9 U. S. Pat. 470,970 (1892).

(d) Dust from copper conv	erters.			
Sample number	a	Ь	с	d
Density in lbs./cu. ft	12.75	77.0	not det	ermined
Gold in ozs./ton	0.16	0.33	0.20	6.0
Silver in ozs./ton	19.24	6.17	41.40	129.60
Copper in per cent	0.49	2.65	2.20	65.86
Lead in per cent	31.70	3.65	40.40	2.75
<sup>4</sup> U. S. Pat. 8,756 (1852). <sup>5</sup> U. S. Pat. 72,032 (1867).				
6 " Metallurgy," p. 449 (18	70).			
7 U. S. Pat. 381,301 (1888).				
8 U. S. Pat. 463,184 (1891).				

<sup>&</sup>lt;sup>1</sup> Eng. Min. Jour., 70, 607 (1900).

<sup>&</sup>lt;sup>2</sup> Ibid., 80, 695 (1905).

Iles and his associates.<sup>1</sup> During the succeeding sixteen years it was generally accepted that the bag house had reached the limit of its development and could be used only for lead and zinc furnace products—not for copper furnaces or for roasters.<sup>2</sup> Minor mechanical improvements were proposed by Rourke,<sup>3</sup> Hannay,<sup>4</sup> Picher and Boss<sup>5</sup> and others.

The construction of the modern bag house for the Lewis and Bartlett process is so well known that no description of it need be given here.<sup>6</sup> The lead smelter bag house is somewhat different.7 It consists essentially of two chambers, the lower or dust chamber being twelve to sixteen feet from floor to ceiling, and the upper or bag compartment ranging from thirty to forty-five feet in height. The walls and partitions are usually of brick, properly buttressed or held together by tie rods. The dust chamber or smoke cellar is divided by brick partitions into as many compartments as there are flues leading to the bag house, thus enabling a part of the bag house to be shut down without interfering with the rest of it. The ceiling of the smoke cellar is made of iron or steel plate and is provided with a number of nipples or rings projecting into the bag compartment. From supports near the top of the building bags of cotton or wool are hung, and the lower ends of the bags are tied on to the nipples by means of twine, wire, or hoop bands provided with strap clamps. The older bag houses, designed simply to recover the solids because of their value to the smelter, were built with ventilators along the walls and on the roof; but the more recent ones, intended for the protection of adjacent property as well as for the recovery of values, are provided with stacks that carry the filtered gases two or three hundred feet into the air.

Fans capable of moving 150,000 to 250,000 cubic feet of gas per minute force the flue gases, laden with dust and fume, through a number of large pipes or flues into the smoke cellar and through the bags, and the solids are removed so perfectly that the escaping gases are invisible. The ordinary bag house is provided with 3,000 to 4,500 bags,

<sup>6</sup> U. S. Pat. 470,970 (1892); Collins, "Metallurgy of Lead" (1899), pp. 72-73; Hofman, "Metallurgy of Lead" (1906), pp. 131-148; Eng. Min. Jour., 83, 937,1033 (1907).

<sup>7</sup> U. S. Pat. 484,017 (1892); Eng. Min. Jour., 80, 55 (1905); 83, 1246 (1907); 84, 527,575 (1907).

each bag being about 18 inches in diameter and 30 to 33 feet long. Whereas the earlier bag houses had 750 to 1,100 square feet of filtering surface per ton of charge, modern practice finds 300 to 500 square feet per ton of charge ample. (In zinc filtration a ratio of 150 to 200 square feet of filtering surface per square foot of hearth is customary.)

As a filtering medium cotton cloth having 42 to 50 threads to the linear inch and weighing 0.4 to 0.7 ounce per square foot was largely used. The objection to this material was that when used at a temperature approximating 200° F. it became brittle and broke, and if used at temperatures near 100° F. it became weakened by the acid substances in the filtered solids. It was soon learned that bags made of woolen material, like flannel, not only withstood the action of the corrosive agents better than did cotton, but that the woolen fabric retained its elasticity even when used at temperatures of 250° to 270° F. Perhaps this is due to the relatively non-volatile oil contained in the wool, or to the nature of the nitrogenous fiber itself. Modern practice, therefore, is to use the expensive woolen bags rather than the cheaper ones made of cotton. To obtain the benefits of wool, but at a less cost, Iles1 proposed that a fabric be used in which the warp should be wool and the weft cotton, or vice versa, and Sheedy<sup>2</sup> suggested that the lower section of a bag should be made of flannel (wool) and the upper portion of muslin (cotton). The most recent experience has shown that vegetable fibers should not be used at all. Even linen thread for stitching is unsatisfactory.

When woolen bags are used a wide range of temperature is permissible. Perhaps 150° to 175° F. is safest, but a minimum of 70° F. and a maximum of 270° F. have been used. The best practice is to so adjust dampers at the furnaces and in the flues as to maintain the temperature as near 160° F. as possible. Under these conditions the material collected in the bag will be dry and filtration can go on rapidly. Lead blast-furnace gases are usually so cool that their treatment in a bag house can take place directly, but the gases from copper furnaces and most roasters ordinarily require cooling. This is effected either by a suitable flue system, which must be as elastic as possible, or by the admission of cold air into the flues with the gases.

Until the investigations of Rhoades and Sprague

<sup>&</sup>lt;sup>1</sup> U. S. Pats. 475,774, 480,834, 484,016, 484,017 (1892).

<sup>&</sup>lt;sup>2</sup> Eng. Min. Jour., 80, 55, et seq. (1905); Iles, "Lead Smelting" (1902), pp. 2, 170, 203 and 204.

<sup>&</sup>lt;sup>8</sup> U. S. Pat. 530,553 (1894).

<sup>&</sup>lt;sup>4</sup> U. S. Pat. 729,492 (1903).

<sup>&</sup>lt;sup>5</sup> U. S. Pat. 735,904 (1903).

<sup>&</sup>lt;sup>1</sup> U. S. Pat. 485,797 (1892). <sup>2</sup> U. S. Pat. 480,834 (1892).

(1907-1908) had shown the possibilities of filtering neutralized gases, the bag house was looked upon as not suited for copper furnace and roaster gases (cf. p. 687 above). These experimenters, impressed with the fact that the bag houses at zinc works were sometimes managed in what seemed to them, as lead and copper smeltermen, a very reckless fashion, yet with little apparent ill effects, conducted an extensive series of investigations with a miniature bag house of the type illustrated by Iles,1 but devised independently by them. A variable speed motor drove a fan that drew gases from a flue and then forced them through a four-inch pipe into a cylindrical dust chamber two by five feet. Upon this cylinder were placed two nipples over which bags were tied. Necessary thermometers, manometers and other instruments were provided, and a house of corrugated iron enclosed the whole apparatus. This experimental bag house was set up at various parts of the flue system of a lead and copper smelter, and the effects of the flue gases with their accompanying solids were determined. During this series of tests neutralizing copper blast furnace and lead roaster gases by means of zinc oxide were tried, and the results were

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so satisfactory that now the lead plant is operated under conditions suggested by them for filtering all the smoke from the blast-furnaces, hand roasters working on matte and converter or pot roasters charged with ore. Stacks, which under ordinary conditions emit large volumes of dense smoke, send only colorless gases and no solids into the atmosphere when the bag-filtration plant is used. An

<sup>1</sup> Eng. Min. Jour., 69, 647 (1900).

observer would judge from the appearance of the stacks that the smelter was not running.

Under this system of operation zinc roasting furnaces for the treatment of zinciferous middlings have been erected, and the flue from these furnaces leads directly into the main roaster flue. The middlings, mixed with fuel, are roasted on a grate and the liberated sulphur dioxide and zinc oxide pass into the roaster flue, where the latter oxide neutralizes the corrosive sulphur trioxide that comes from the roasters. Provision is made at the bag house fan for adding ordinary zinc oxide constantly, if need be, by an automatic feeding device.

It would not be economical to use this method, under ordinary conditions, with roasters of the hand or McDougal types, because of the large quantities of sulphur trioxide produced by them; but the treatment of the gases coming from pot or converter roasters presents no difficulties. In these the amount of sulphur trioxide is relatively much less than in the hand or McDougal roaster gases, and zinc oxide neutralization is commercially possible. Were this small amount of sulphur trioxide not to be neutralized, however, the filtering bags would soon be destroyed.<sup>1</sup> Gases from copper blast-furnaces, reverberatories and converters are amenable to treatment under the conditions herein given.

An item of importance in bag house operation is the means adopted for shaking the bags. Originally the method used was to shut off the gas from a set of bags, open the doors in the bag room, and after the noxious gases had escaped shake the bags by hand. This was at best a disagreeable-even dangerous-task and consumed much time. At East Helena a device was provided whereby an attendant could move a lever on the outside of the bag house and have a set of beaters strike against the rows of bags within. The scheme was abandoned, both because the shaking was ineffectual and the wear and tear on bags was great. Rourke<sup>2</sup> attached ropes or wires to rings placed around the centers of bags so that a whole row could be shaken by jerking the end of the rope extending outside the building. The most satisfactory device is one designed by Benedict,3 in

<sup>3</sup> Patent pending.

<sup>&</sup>lt;sup>1</sup> Hand roasters present a double difficulty. Not only is the production of sulphur trioxide large, but the products from coal firing seem to make the bags and their contents liable to spontaneous combustion—a combustion that is not accompanied by flame, but is more like that which one observes in the burning of punk.

<sup>&</sup>lt;sup>2</sup> U. S. Pat. 530,553 (1894).

which the bags are suspended from short levers attached to a central shaft. The outer end of this shaft projects beyond the wall and is provided with a larger lever, so that when this lever is moved to and fro the tops of the bags are given a similar motion, and a wavy motion is imparted to the bags as a whole.

The bag house product, collected in the dust chambers or smoke cellar, usually has a black or dark gray color, is quite light (density less than water) and can be handled with difficulty when in its original state. Therefore it is burned. Some oily waste is lighted and thrown upon it, the doors to the dust chambers are opened, and a fan draws the fumes through a flue situated at the back of the chamber and discharges them into the main flue leading to the bag house. Here the fume evolved during the burning is caught in a part of the house that is operating as usual. The material on the floor of the dust chamber burns slowly like punk, gradually sinters to a product resembling soft lava, and loses some of its arsenic and sulphur. The burning continues a number of days, and then the sintered product goes to an arsenic refinery. The white arsenic is volatilized and used as such, and the residual cinder is charged again into the lead blast-furnace. Thus the arsenic, lead, copper and precious metals are recovered.

The labor question has been a serious one in bag house management, and the expenditure of thought and care upon the welfare of the men is imperative. It will not do to put any kind of a laborer in charge of a bag house. The foreman should be at least as able as the charge floor foreman or the head furnaceman, and his helpers should be capable of understanding orders and executing them implicitly. A wash room divided into three parts should be provided. The laborer enters the "clean room," disrobes and places his clothing in sanitary lockers, passes through the "wash room" into the third room and there dresses in heavy underwear, socks, overalls, jumper, cap, gloves and shoes provided by the company. His shift completed he leaves his working clothes where he got them originally, bathes thoroughly from head to foot, using plenty of soap and hot water, and then passes from the wash room to the locker room he entered at first, and there clothes himself. Constant watchfulness on the part of the management is necessary to make sure that the washing is done regularly and thoroughly. Careful attention must be given to the health of the men, and at the first appearance of

arsenical or lead poisoning proper antidotes should be administered and the men ought then to be transferred to some other kind of work until recovery is complete. When regulated as here indicated, the same crew of men can work at a bag house for months at a stretch without discomfort or danger.<sup>1</sup>

The objections to the use of a bag house are (1) cost of installation;<sup>2</sup> (2) cost of maintenance; (3) artificial draft must be substituted for natural draft; (4) danger to operatives; and (5) the arsenic saved as a by-product is sometimes disposed of with difficulty.

To offset these disadvantages we must consider (1) the greater recovery of metals; (2) regularity in draft and ventilation; (3) greater safety and less discomfort to the inhabitants of the region surrounding the smelter, and consequently less expense for lawsuits.

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## A COMPARISON OF METHODS FOR THE DE-TERMINATION OF SULPHUR IN COAL.

By S. W. PARR, W. F. WHEELER AND RUTH BEROLZHEIMER. Received July 10, 1909.

Advantage has been taken of a comparative series of tests on thirty-five coals for the determination of calorific values,<sup>3</sup> to make a further comparison of methods for the determination of sulphur. These methods involve the use of the residues from the bomb calorimeter of the Mahler Atwater type and from the Parr calorimeter, using sodium peroxide as the fusion medium. To these two methods were added the usual Eschka method for the determination of sulphur in coals and the photometric method recently described in the *Journal of the American Chemical Society*.<sup>4</sup>

While this work was in progress, the article by Holliger<sup>5</sup> on the estimation of sulphur in coal appeared, in which the same comparison of methods was made, excepting the photometric process. In that article the fusion method by the use of sodium peroxide is discredited as being subjected to too great personal errors. The result of the work described in this paper goes to show the method

<sup>&</sup>lt;sup>1</sup> Some idea as to the dangerous nature of the bag house dust may be gained from the fact that it contains from ten to thirty per cent, of arsenic, and from fifteen to fifty per cent, of lead.

<sup>&</sup>lt;sup>2</sup> A bag house of 4000 bags will cost approximately \$150,000.

<sup>&</sup>lt;sup>3</sup> "A Series of Parallel Determinations with the Mahler and Parr Calorimeters," THIS JOURNAL, 1, 673.

<sup>4</sup> Jour. Am. Chem. Soc., 26, 1139-43

<sup>&</sup>lt;sup>5</sup> Zeit. angew. Chem., 22, 436-49, 493-7.



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to be exceedingly reliable. It is entirely possible that the method of carrying on the fusion may have much to do with this difference of opinion. In the method as practiced by us the grade of sodium peroxide as especially prepared for the determination of heat values was used and a uniform amount of one gram of potassium chlorate was added to the charge, one-half gram being the amount of coal used. The fusion was carried on in the calorimetric bomb as illustrated in Fig. 1, and was exceedingly uniform and complete so far as the combustion of the material was concerned. The fusion was dissolved and acidified in such a manner as to give approximately 5 cc. of free acid in a volume of 250 or 300 cc. of liquid. Under these conditions the interference of salt or iron was not appreciable. This method has been in constant use and has been subjected to very many tests in this laboratory during the last five or six years and has given at all times evidence of thorough reliability.

Fig. 1. In the table presented herewith the gravimetric results obtained by the method of fusion with  $Na_2O_2$  are given in column (1). In



column (2), comparison is made with the results obtained by the usual Eschka method modified, however, in that the residue, after burning and dissolving out the soluble sulphates, was found almost without exception to contain sulphur which was not soluble by the usual method of washing with water. This residue, therefore, was treated with acid and a precipitation of the sulphate obtained which was added to the main precipitation from the water solution. Only by this means could a duplication of results be obtained and, when so modified, the results are seen to be in good agreement with the fusion values as obtained under column (1). The differences between the values in columns (1) and (2), using the values in column (1) as a basis, are shown in column (5).

COMPARISON OF METHODS FOR THE DETERMINATION OF SULPHUR.

1.		, i d	a	do.	fu-	iff.	iff.	iff.
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		20.	· 문편	H N II	Va <sub>2</sub> Va <sub>2</sub>	r p	r p	p
		Na	Me 2	Me 33.	Act	ho	ho	ho
	No.	W		Children Mil	Pl	fet	fet	fet
ž	e	Gr	avimetr	1C.	Photo-	4	*	A
ıb.	[q]	(1)	(2)	(3)	(4)	(5)	. (6)	(7)
La	Ť	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
2111	1	1.82	1.72	1.65	1.71	-0.10	-0.17	-0.11
2112	2	1.26	1.21	0.90	1.08	-0.05	-0.36	-0.18
2113	3	0.92	0.81	0.82	0.63	-0.11	0.10	-0.29
2114	4	4.41	4.29	4.05	4.48	0.12	-0.36	+0.07
2115	5	1.79	1.81	1.77	1.75	+0.02	-0.02	-0.04
2116	6	3.83	3.78	3.60	3.68	-0.05	0.23	-0.15
2117	7	3.48	3.421	3.21	3.32	-0.06	-0.27	-0.16
2118	8	3.76	3.841	3.62	3.68	+0.08	0.14	-0.08
2119	9	4.80	4.811	4.31	4.80	+0.01	-0.49	±0.00
2120	10	3.20	3.13	2.89	3.00	-0.07	-0.31	-0.20
2121	11	5.57	5.811	5.25	5.60	+0.24	0.32	+0.03
2122	12	2.68	2.621	2.48	2.54	-0.06	-0.20	-0.14
2123	13	3.44	3.311	3.20	3.34	-0.13	-0.24	-0.10
2124	14	3.09	3.22	2.85	3.14	+0.13	-0.24	+0.03
2125	15	0.93	0.89	0.76	0.66	-0.04	-0.17	-0.21
2126	16	0.82	0.81	0.72	0.62	-0.01	-0.10	-0.20
2127	17	0.97	1.02	0.94	0.72	+0.05	-0.03	-0.23
2128	18	1.12	1.14	1.09	0.82	+0.02	-0.03	-0.30
2129	19	1.00	0.95	1 01	0.85	-0.08	+0.01	-0.24
2130	20	0.72	0.92	0.70	0.51	-0.03	-0.02	-0.21
2131	21	0.72	0.63	0.64	0.53	-0.07	-0.06	-0.16
2132	23	1 41	1 30	1.36	1 32	-0.02	-0.05	-0.09
2134	24	1 26	1 16	1 20	1 16	-0.10	-0.06	-0.10
2135	25	1 63	1 53	1.50	1.73	-0.10	-0.13	+0.10
2136	26	0.52	0.50	0.54	0.31	-0.02	+0.02	-0.21
2137	27	1.31	1.23	1.21	1.15	-0.08	-0.10	-0.16
2138	28	2.64	2.64	2.50	2.50	±0.00	0.14	-0.14
2139	29	0.80	0.77	0.64	0.70	-0.03	-0.16	-0.10
2140	30	1.20	1.23	0.95	1.16	+0.03	-0.25	-0.04
2141	31	0.64	0.67	0.61	0.52	+0.03	-0.03	-0.12
2142	32	1.25	1.29	1.22	1.18	+0.04	-0.03	-0.07
2180	33	5.07	4.97	4.62	4.95	-0.10	-0.45	-0.12
2193	34	2.54	2.58	2.40	2,58	+0.04	-0.14	+0.04
2194	35	3.01	2.99	2.80	3.16	-0.02	-0.21	+0.15
								-
Avera	age	2.13	2,11	1.97	2.02	-0.02	-0.16	-0.11

In column (3) are listed the values obtained in

<sup>1</sup> The magnesium oxide and carbonates and residue insoluble in water were not dissolved in hydrochloric acid as they were in all other determinations by the Eschka method. SULPHUR PHOTOMETER CURVE



the washings from the Mahler-Atwater bomb. These values are not in such close agreement to the values in column (I) and it may be argued in explanation that the sulphur thus obtained represents the combustible sulphur. The values are almost without exception lower. The differences between the values in columns (I) and (3) are shown in column (6).

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In column (4) the values have been obtained by means of the photometer as illustrated in Fig. 2.

The standard light (L) is made to read through the precipitated material in the graduated tube (A), the lower end of which is submerged in the cup (B). By this means a lens effect is secured and the end point is made very sharp and distinct. Aside from the improved form of apparatus shown in Fig. 2, the chief modification of the process made use of in the present series was the use of a small amount of oxalic acid in conjunction with barium chloride, which precipitates the barium sulphate in a very nearly colloidal condition, at least it does not settle readily and is uniform in its action towards the transmission of light. The curve as shown in Fig. 3 affords a ready means for determining the weight of sulphur in milligrams to correspond with the depth of the solution in millimeters.

It cannot be expected that such a method will have the same degree of accuracy that should accompany a gravimetric method, but for technical purposes it is admirably adapted as a quick and ready process for determining the sulphur in the fusion residues from calorimetric determinations by the use of sodium peroxide. Comparison as between this method and the gravimetric fusion method of column (1) is shown in column (7).

It may be well to call attention to one advantage in this series over the tests made by Holliger, in that three times the number of coals are tested, sufficient to bring out any variations that inhere in the various processes. It should be borne in mind that the fusion method No. 1 yields the total sulphur, while the tendency of method No. 3 is to yield the burnable sulphur rather than the total sulphur. If this latter distinction were sharply drawn, that method would have an advantage. On the other hand, method No. 1 has the advantage of certainty of oxidation when conducted as herein described, and there can be no question about the completeness of the process. It is, therefore, given preference in the table and the others are arranged in the order of what seems to be their respective merits. The coals are widely distributed; their source, as may be seen by reference to the article on calorimetric values above referred to, is from Pennsylvania, West Virginia, Kentucky, Alabama, Maryland, Kansas, Indiana and Illinois.

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## STUDIES IN THE USES OF FINELY DIVIDED SILICA.<sup>1</sup>

By S. W. PARR, T. R. ERNEST AND W. S. WILLIAMS. Received August 7, 1909.

The extensive deposits of finely divided silica found in Southern Illinois have furnished, in very large measure, the incentive for some work in the Department of Applied Chemistry of the University of Illinois during the last two years. This work has consisted mostly of experiments on compounds such as are formed at steam temperatures between this material and lime.

It was found that bricks made in this way possessed, after steaming, good texture and strength. This suggested a series of experiments to determine the best ratio of lime to silica. Then the idea of making briquettes by moulding without pressure suggested itself, and with it came the question as to the best conditions for maximum strength. In the first case the briquettes were made by compression and tested for crushing strength while in the second a cement briquette mould was used and briquettes were tested for tensile strength. In the first case the best lime silica ratio was found to be one mol. of lime to one of silica. while in the latter the best results were attained when 20 per cent. of lime was used with 80 per cent. of amorphous silica as is shown by the following tables:

		TABLE I.			
Data:	Steam pro	Steam pressure 105 fbs. per sq. in.			
	Time exposed to steam, 10 hrs.				
	Per cent.	Per cent.	Tensile strength,		
	lime.	silica.	lbs. per sq. in.		
	10	90	135		
	20	80	265		
	30	70	202		
	40	60	189		
	50	. 50	150		
		TABLE II.			
Data:	Steam press	ure 150 lbs. per s	sq. in.		
	Time expo	sed, 10 hrs.			
	Per cent.	Per cent.	Tensile strength,		
	lime.	silica.	lbs. per sq. in.		
	10	90	134		
	20	80	278		
	30	70	204		
	40	60	169		
	50	50	148		

<sup>1</sup> An examination of this material made some years ago showed it to be of chalky appearance sometimes stained by iron. It consists of

ALC: NO		TABLE III.	
Data:	Steam pre	ssure 50 lbs. per	sq. in.
	Time expo	osed, 10 hrs.	
	Per cent.	Per cent.	Tensile strength,
	lime.	silica.	lbs. per sq. in.
	10	90	102
	20	80	182
	30	70	116
	40	60	94
	50	50	71

An attempt was made to ascertain whether or not pressure was needed to bring about this reaction, or whether it was caused by the heat of saturated steam together with the moisture. To do this, briquettes were made in the usual way and placed in a chamber that could be heated from without to a temperature corresponding to that of saturated steam at 150 pounds pressure. Steam was then generated in a flask and superheated to the temperature of the chamber after which it was passed over the briquettes. The time of exposure varied from ten to forty-eight hours, but in no case was there any noticeable reaction. From this it was concluded that pressure as well as heat and moisture is required to bring about the reaction.

The effect of heat on these briquettes is rather interesting, as is shown by the following table:

	TABLE IV.			
Test number.	Drawn at tem- perature, ° C.	Tensile strength, lbs. per sq. in.		
0	20	278		
1	300	45		
2	400	0		
3	500	101		
4	600	131		
5	700	124		
6	750	27		
7	800	71		
8	850	186		
9	1190	260		
10	1390	263		

As this behavior is not what one would expect, the work was repeated several times with substantially the same results.

After finding the best proportion in which to mix the lime and silica to give the best tensile strength, experiments were made in which other substances were introduced into the briquette. Magnesia was found to work about the same as lime. It was found that the introduction of about four parts of sharp sand into a mixture of equal parts of lime and silica (one part) gave a product with much higher tensile strength than when no sand was used. The effect of the addition of fibrous material was next studied, and the first to be tried was slag wool. The addition of varying per cents. of this material did not materially affect the strength until enough was added to dilute the original material so much that it resulted in a weakening of the bond. There appeared to be no union between the fiber and lime. A cheap grade of asbestos was tried next. The results of the tests are shown in the following table:

		TABLE V.	· AND AND STREET
Data:	Steam	pressure, 150 fbs. p	er sq. in.
	Time of e	xposure, 10 hrs.	
	Per cent.	Tensile strength,	Increase in tensile
	asbestos.	lbs. per sq. in.	strength.
	3	285	7
	4	297	19
	5	300	22
	10	343	65
	12	377	100
	14	302	24
	20	242	-36
	40	173	-105

From the table it will be observed that the tensile strength rises with the addition of asbestos until 12 per cent. is added, when the drop is sudden. It will be noticed, too, that the strength of the material may be increased very materially by the addition of this fiber.

The effect of the addition of colloids was next studied. To the regular mixture of lime and silica (20 per cent. CaO to 80 per cent.  $SiO_2$ ), varying per cents. of sodium silicate and of Portland cement were added and the briquettes made and tested in the usual way. The results showed a decrease in the strength in every case, which in the case of the sodium silicate was about 66 per cent. of the original strength when only 5 per cent. was added.

Mixtures of lime and silica in the proportions for best tensile strength were found to possess good plasticity, this property being possessed in about the same degree as in the case of Georgia kaolin. This discovery led to an attempt to make a vase of this material. The regular mixture of lime and silica was used, enough water being added to give the best plasticity, and the vase moulded by hand in a plaster of Paris mould. When dry, the vase could be removed and the joints finished. When hardened by steam it had, when struck with a pencil, the true hard ring of vitrified clay biscuit. The material easily took the form and markings of the mould; there was no shrinkage that was noticeable and the color was a good clear white. No attempt was made to prepare a vase by making the mixture up with enough water so that it could

minute particles of silica, from 50 mm. to 0.2 mm. in diameter, of crystalline structure, transparent and irregular in shape, loosely cemented together by a small amount of clay. An analysis showed: Moisture, 0.15%; silica, 98.00%, magnesium oxide, 0.20%; aluminium oxide, 1.21%; undetermined, 0.44%.—ED.

be poured into a mould in the form of a slip, but there can be little doubt as to the possibility of making ware in this way. Small trials were also made by jiggering, which were very successful.

These experiments suggest the possibility of using this process for making architectural building material to be used in a way similar to terra cotta. The manufacturer of terra cotta has many obstacles to overcome in the process of burning. His ware may warp or be of the wrong color; it may shrink excessively or be defective in some other way. The manufacture of artificial stone that might be used similarly to terra cotta might be made, it seems, very advantageously by this process.

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#### CYANIDATION OF SILVER MINERALS.

By THEO P. HOLT. Received July 17, 1909.

This paper embodies some results of an investigation carried out during the past year, under the Wall Research Fellowship at the Utah State School of Mines. From a mechanical standpoint, the adaptation of the cyanide process from gold to silver ores has been very satisfactorily worked out, but chemically the development has been slow, and to quote a well-known writer, "It is difficult to differentiate between fact and fancy in dealing with the available evidence on the subject." It was with the hope of clearing up some of these mooted questions that this investigation was undertaken.

The experiments, which are represented in graphic form, were conducted in 8-oz. bottles. Ten of these were clamped in a suitable frame which was rotated by a small water motor. The speed of the motor was so regulated that the contents of the bottle would fall from one end to the other during each half revolution. The bottles were only partly filled so that an excess of oxygen was assured. The conditions favoring extraction are as good as obtained with air agitation, while variations due to gases present in the air, evaporation, etc., are largely eliminated. Thus it is possible to estimate closely the consumption of cyanide due to the minerals present in the ore, and determine the amount of any metal dissolved from solution assays.

The value of experimental tables showing variations in results, secured by changes in treatment, is largely determined by the accuracy of our knowledge of the active agents at work in the test. In case several unknown factors enter into an experiment, we simply get a mass effect, and are unable to judge with safety the action of each agent. For this reason the significance of a set of tests on any particular ore is limited. The results on another ore of similar chemical composition may be quite different, due to mineralogical and physical differences. Thus it has been aptly said "that each ore is a problem in itself."



In consideration of the above facts I have made most of my experiments on chemical compounds and artificial ores of known mineralogical composition. Active agents, met with in cyanide practice, have been added singly. One example will serve to illustrate the general procedure. About 1.6 gms. of a crystallized specimen of pyrargyrite (Ag<sub>3</sub>SbS<sub>3</sub>) were crushed with a few grams of quartz sand in a porcelain mortar, to pass a 100-mesh screen. This was then mixed with 550 grams of quartz sand, which had been ground on a stone bucking board to avoid the introduction of iron. After adding 1.6 gms. of lime the sample was thor-

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oughly mixed and quartered. The method of preparation favors a large number of mineral particles approaching the maximum-sized grain that will pass the screen. This will in a measure



offset the fact that none of the mineral is encased in the gangue. No doubt the surface of mineral exposed to the action of the cyanide solution is much less than obtains in modern slime practice.

All solutions were titrated with a standard silver nitrate solution. In cases where the silver mineral is the only cyanicide present, it is possible to calculate quite closely the extraction secured by the amount of KCy consumed. The per cent. of silver dissolved was computed in all cases from fire assays. The original ore was assayed in duplicate, and the result checked by assays on both the tail and the solution.

Compounds, other than potassium and sodium cyanide, used to assist in the solution of silver, are termed "chemical salts" by cyanide mill men. In treating ores containing their silver chiefly as argentite, these are of great practical importance. The degree to which they are capable of influencing the amount of silver dissolved, may be appreciated when we inspect the graphic results of a few tests. Take the line for argentite for an example. A 0.5 per cent. KCy solution dissolves 38 per cent. in 17 hours. The addition of 0.3 per cent, lime brings this up to 58 per cent. By making the solution strongly alkaline with KOH almost 97 per cent, of the silver is dissolved in the same time. A small amount of litharge is about equally effective.

To explain this action requires reference to the primary equation for the solution of silver sulphide:

$$Ag_2S + 4KCy \leq 2KAgCy_2 + K_2S$$

This reaction proceeds only to a slight extent before equilibrium is established between the  $S^{--}$  ions, the Ag<sup>+</sup> ions and the undissociated Ag<sub>2</sub>S. Litharge (PbO), although it enters the solution as a plumbite, still dissociates a sufficient number of lead ions to effectively remove the  $S^{--}$  ions



as insoluble PbS. Thus the presence of the lead salt prevents the equilibrium indicated above being established and the solution of silver sulphide proceeds. Any metal whose sulphide is but slightly soluble in cyanide solution will produce a similar effect.

The increased extraction observed on adding potassium hydroxide to the solution, is also in



accordance with the mass law. In this case we have

 $(K_2S)k = (2K^+) \times (S^{--}).$ 

Upon adding a highly dissociated base as KOH, we greatly increase the number of K ions, and hence the product on the right of the equation. To establish ionic equilibrium some of the K ions must unite with S ions to form undissociated  $K_2S$  and thus effectively removing S ions from the solution. Theoretically any highly ionized compound capable of forming a slightly dissociated compound with sulphur, will increase the solubility of silver sulphide.

From a practical standpoint the use of some metal to precipitate the sulphur in an insoluble form is much to be preferred, as any other method results in the fouling of the solution by the accumulation of sulphur compounds. Lead acetate is the salt most extensively used in practice for the precipitation of soluble sulphides. Litharge has been substituted in one or two instances to advantage. It is less expensive both on account of the higher lead content, and less cost per pound. Litharge has the further advantage that a slight excess does not seriously affect the results, as is the case with lead acetate.

Mercury compounds are also effective precipitants for soluble sulphides, but are more expensive than lead salts. Hobson<sup>1</sup> experienced very beneficial results on a certain Mexican silver ore by the addition of mercurous chloride to an alkaline cyanide solution. He attributes his success to the property of "mercurous potassic cyanide" to dissolve argentite, pyrargyrite, stephanite and



proustite. It has been pointed out later by both Hunt<sup>2</sup> and Sharwood<sup>3</sup> that the compound "mercurous potassic cyanide" does not exist. The

<sup>3</sup> Ibid., Sept. 26, 1908.

<sup>&</sup>lt;sup>1</sup> Min. and Sci. Press, Aug. 8, 1908.

<sup>&</sup>lt;sup>2</sup> Ibid., Aug. 29, 1908.

formation and action of mercury compounds are discussed rather fully in these two articles. I have tested out the chemical combination given by Hobson on several of the silver minerals, and



with the exception of argentite, have experienced no beneficial results. The general conclusion would seem to be that the mercury is effective chiefly by virtue of its ability to remove soluble sulphides.

Bromocyanide is an energetic oxidizer, and its property of dissolving gold is well known. At the present time it is being successfully applied to gold ores which cannot be treated by plain cyanide. Some statements have been made regarding the adaptation of bromocyanide to silver ores. I find that in the absence of free cyanide it is not a solvent for the silver minerals. At any rate its solvent power is so slight as to be of no value. The addition of a limited quantity of bromocyanide to a cyanide solution is quite effective in increasing the extraction, in some cases, due to its oxidizing power.

A chloridizing roast of silver sulphide ores is an essential preliminary to successful treatment by the old processes. With cyanidation it has been found possible to treat successfully a great many such ores without roasting, and hence one of the strong points favoring the cyanide process. It is likely, however, that many cases exist where roasting could be employed to advantage. In fact in some cases it seems the only means of securing a satisfactory extraction.

A number of the "ore samples" were given a one-hour roast in an open muffle at a dull red heat. It is evident from the graphs that a chloridizing roast is about equally effective in all cases, the silver being converted into a chloride which is readily dissolved. For an oxidizing roast one hour is too brief a period to change materially the state of the silver minerals.

There are many interesting points suggested by the tabulations and graphs that are not mentioned in this discussion. For instance in the "variations in treatment," it is remarkable how closely the silver of ore "13" follows the line for argentite.



It was proven by independent tests that the silver present in this ore is practically all present in the form of argentite.

In conclusion it may be well to summarize some

of the more important points regarding the chemistry of the cyanide process as applied to silver ores.

Each silver mineral presents its own peculiar difficulties. The fundamental laws of chemistry explain in a remarkable way many of the reactions otherwise obscure. Results obtained from experiments on ores are often misleading due to their complex nature. It is therefore advisable to work on artificial ores of known mineralogical composition, before making generalizations regarding the several minerals.



The halogen compounds of silver are the most soluble form in which it occurs, and no oxygen is required for their solution in potassium cyanide. Native silver is next in order of solubility in an aerated solution, and it dissolves in accordance with Ellsner's equation.

The silver sulphide, argentite, requires the removal of soluble sulphides to insure good extraction. Lead salts are applied for this purpose, and litharge, though less used, presents decided advantages over lead acetate.

Mercury compounds are effective by virtue

of their ability to form insoluble sulphides, and not by their property to dissolve the "rebellious minerals."

Oxygen or an oxidizer is essential to the continued solution of silver sulphide in cyanide, even in the presence of lead salts.

The presence of lead or mercury compounds decrease the solubility of the complex antimony and arsenic silver minerals, in a cyanide solution.

Bromocyanide is not a solvent for the silver minerals. In the presence of free cyanide it may give beneficial results by virtue of its power as an oxidizer.

A chloridizing roast is about equally effective on all the silver minerals, and subsequent treatment by cyanidation will give good extraction.

The problem of interfering minerals is more serious in the cyanidation of silver than it is for gold. This is due to the stronger solutions used, and the greater chemical activity of silver.

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## THE ESTIMATION OF PYRIDINE IN AQUA AMMONIA.

By A. C. HOUGHTON. Received July 17, 1909.

Owing to the increasing use of anhydrous ammonia for refrigerating purposes, the manufacturer of aqua ammonia has been called upon to furnish a very pure product. The presence of pyridine has proved especially troublesome to the maker of anhydrous ammonia, as, being basic in character, it escapes the purifying trains that serve to hold back the impurities of acid character, such as carbon dioxide and sulphuretted hydrogen, and goes over with the ammonia gas to the compressor, where it separates out before the ammonia is liquefied, and causes trouble by filling up the traps that serve to catch any moisture remaining in the gas, finally going over to the anhydrous ammonia. Even an aqua ammonia testing as low as 0.2 gram per liter in pyridine will cause considerable trouble, as in working off a carload of 25 cubic meters, all the pyridine present, amounting to 5,000 grams, eventually finds its way to the compressors. The elimination of pyridine from aqua ammonia presents no particular difficulty, but it is important to have an accurate and reliable method of estimating small amounts of pyridine in the presence of a large excess of ammonia, in order that the liquors in the different

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stages of manufacture may be followed, and the amount of pyridine present controlled. As none of the methods given in the literature are sufficiently accurate or reliable for this purpose, the following work was undertaken in the hope of finding a satisfactory means of estimation.

The property that pyridine has of forming a double compound with mercuric chloride may be made the basis of a very satisfactory determination. If an excess of mercuric chloride is added to a mixture of pyridine and ammonia, the double compound  $C_5H_5N.HgCl_2$  is formed and the ammonia reacts with mercuric chloride according to the equation

 $_{2}HgCl_{2} + _{4}NH_{4}OH = NHg_{2}Cl + _{3}NH_{4}Cl + _{4}H_{2}O.$ The precipitate consisting of NHg<sub>2</sub>Cl and C<sub>5</sub>H<sub>3</sub>N. HgCl, is filtered off, and on thoroughly washing with water at about 60° C., the pyridine compound, which is soluble in hot water, is dissolved out. The filtrate and washings then contain ammonium chloride, the double compound of pyridine with mercuric chloride, and excess of mercuric chloride, and on long-continued boiling the pyridine compound is completely dissociated into pyridine and mercuric chloride, pyridine passes over as the only free base, and may be titrated with standard acid.1 The distillate, however, always shows a test for ammonia with Nessler solution, owing, no doubt, to the slight dissociation of ammonium chloride, and further, the method requires considerably more time and more careful work than the one now to be described.

While working on this problem of the estimation of pyridine, Mr. Carl Sundstrom, of our Detroit laboratory, called my attention to the sodium hypobromite reaction with ammonia. As is well known, sodium hypobromite oxidizes ammonia according to the equation

 $2NH_3 + 3NaOBr = 3NaBr + N_2 + 3H_2O.$ 

It was found to have no action on pyridine, however, as would be expected from the fact that there is no oxidizable hydrogen combined with the nitrogen atom in pyridine, and as the following experiment shows.

Varying amounts of a tenth-normal solution of pyridine were added to 50 cc. of a tenth-normal solution of ammonium chloride. An excess of sodium hypobromite solution was then added, the flask shaken until no more gas was evolved, and after adding a little caustic soda, the liquor distilled for pyridine, and the distillate titrated with tenth-normal acid. The following results were obtained:

Grams pyridine	Grams pyridine
taken.	found.
0.000	0.0002
0.006	0.008
0.013	0.014
0.025	0.025
0.032	0.027
0.038	0.039
0.044	0.042
0.051	0.047
0.057	0.057
0.063	0.063

In testing aqua ammonia the method of procedure is as follows:

100 cc. of aqua ammonia are put into a liter flask with about 150 cc. of distilled water, a few drops of methyl orange indicator added, and the liquor carefully neutralized with dilute sulphuric acid (1-3) until just acid, the flask being kept cool under the tap to prevent escape of pyridine. The liquor is now brought to the neutral point with normal alkali, made alkaline with 5 cc. of normal caustic soda, and distilled into 10 cc. of normal acid with a little water in a quart flask, twenty minutes' boiling being sufficient. Pyridine being a weaker base than ammonia is liberated first by the caustic soda, and the distillate contains all the pyridine present in the 100 cc. of aqua. and some ammonia. With aquas containing large amounts of pyridine, it would, of course, be necessary to make the neutralized aqua alkaline with more than 5 cc. of normal caustic soda, but in this case it would be better to start with a smaller amount than 100 cc. The ammonia in the wellcooled distillate is now destroyed by adding 100 cc. of sodium hypobromite solution (made by dissolving 100 grams caustic soda in 1000 cc. of distilled water, cooling, and adding 25 cc. of bromine), the liquor is shaken for a few minutes until no more gas is evolved, and the pyridine distilled over into an excess of tenth-normal acid, a Kjeldahl distilling bulb being used to prevent any mechanical carrying over of caustic soda. The excess of acid in the distillate is titrated back with tenth-normal caustic, using methyl orange indicator. 1 cc. tenth-normal acid is equivalent to 0.0079 gram pyridine. When only small amounts of pyridine are present, the end point with methyl orange indicator is quite sharp, and the titration can easily be made to within o.1 cc. of tenth-normal

<sup>&</sup>lt;sup>1</sup> Since the above work was done, I have found that Kinzel in the *Pharmaceutische Centralhalle* (1890), **31**, 239, has already worked out a method for the quantitative estimation of pyridine in ammonia based on this same property of the easy dissociation of the double compound of pyridine with mercuric chloride.

acid, but with larger amounts of pyridine the end point is more or less indefinite, owing to the acid character of pyridine salts. Even in this case, however, it is possible to get the end point within 0.5 cc. of tenth-normal acid, and this does not introduce an error of more than 0.04 gram per liter pyridine.

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To check up the method finally, known amounts of pyridine were added to aqua ammonia absolutely free from pyridine. As ordinary chemically pure ammonia always contains pyridine, it was necessary to prepare an aqua ammonia entirely pyridine-free, which was done by heating Merck's c. p. ammonium chloride with calcium hydroxide, neglecting the first portions of ammonia given off, and then absorbing the evolved gas in well-cooled distilled water. The aqua thus prepared contained 24% NH<sub>2</sub>, and when carefully neutralized with acid gave absolutely no odor. To this pure ammonia was added weighed amounts of chemically pure pyridine, which had been dried over solid caustic potash and carefully re-fractioned, and the estimation carried through exactly as described above. The following results were obtained, which leave nothing to be desired in the way of accuracy.

rams per liter pyridine	Grams per liter pyridine
present.	found.
0.00	0.008
0.05	0.05
0,10	0.10
0.15	0.14
0.20	0.21
0.40	0.38
0.60	0.57
0.80	0.81
1.00	1.06

Duplicates of some of the above tests were also run through, but instead of adding methyl orange and titrating the final distillate, it was tested with Nessler solution. In no case, however, was even a slight coloration obtained, showing that the ammonia had been completely destroyed, so that the only alkaline body that passes over in the final distillation is pyridine and its homologues.

Since the above method was worked out, it has been slightly modified by Dr. D. A. Morton, of this laboratory, who found that it was possible to carry out the estimation with only one distillation, with no loss in accuracy. After the aqua is neutralized and the 5 cc. of normal caustic added, it is distilled through hypobromite solution. An ordinary one-pint sample bottle is fitted with a two-hole rubber stopper, through one hole of which passes the delivery tube from the distillation flask, reaching nearly to the bottom of the bottle, and through the other a Kjeldahl bulb, connected, of course, by rubber tubing with the flask containing tenthnormal acid to absorb the pyridine. 100 cc. of sodium hypobromite solution are placed in the bottle, the stopper inserted tightly, and the distillation commenced. The bottle is not cooled, and the steam from the distillation soon heats the contents to boiling, the ammonia coming over being destroyed, and the pyridine passes over to the receiver. With this modification a single test may easily be made in half an hour.

The method described above has been in use in this laboratory for several years, and has proved itself very satisfactory and reliable.

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## FLOUR BLEACHING, ITS RELATION TO BREAD PRODUCTION AND NUTRITION.

By JOHN A. WESENER AND GEORGE L. TELLER. Received July 29, 1909.

This question has received much attention of late, both in this country and in Europe, in its relation to economics, bread production and nutrition. It has also found a prominent place in the courts, and recent rulings by the Department of Agriculture indicate that it will receive still further attention in this connection. The questions involved are so imperfectly understood, not alone by laymen but also by most scientists, that they should be clearly outlined, and it is the province of this paper to bring this data before the reader in an accurate and concise manner. Not only is the interest of the general public at stake, but there is also involved the rights of the manufacturer, as relating not only to the economics from his view point, but also, as these economics may have a direct bearing upon the consumer.

From an early date in the production of wheat flour the bleaching of the color has been practiced by millers before putting their product upon the market. Up to within the last decade the method employed was to store the flour for a period of several months whereby the more or less marked yellow color of the flour was removed.

Not many years ago, a Frenchman, Frichot,<sup>1</sup> discovered that the gas produced by discharges of electricity through air were capable of whitening the flour. He did not explain properly the cause of this whitening, but attributed it to the production of ozone (which it is now known does not readily bleach flour).<sup>2</sup> It remained for two Eng-

lishmen by the name of Andrews<sup>3</sup> to discover that the bleaching was brought about by the action of certain oxides of nitrogen which are produced when nitric acid is brought in contact with a reducing agent such as ferrous sulphate. Numerous processes have been brought forth for applying this principle to practical bleaching of flour in the mill. The process, which has up to the present time gained the greatest ascendency in this country, is what is known as the Alsop process, where air is pumped through a chamber in which a flaming electric arc is discharging. It can readily be shown that the air discharged from the pump contains small quantities of oxides of nitrogen. In more recent times the use of nitrosyl chloride has been introduced into the art of flour bleaching and possesses advantages over the oxides of nitrogen in the character of the bleach which it produces as well as in the simplicity of application to the flour. This gas can readily be liquefied and bought in small bulk for convenience of transportation and storage. The pressure is ideal for the purpose, being at normal temperature from 15 to 20 pounds to the square inch, and is an important point, as it materially aids in the methods of applying the gas to the flour.

Color is probably one of the most important factors entering into foods, because it plays an important rôle in the mind of the consumer, bringing in a physiological factor which not only affects the palatableness of the food, but also the health of the consumer. The United States government took advantage of that factor in passing the Oleomargarine Act, imposing a tax of 10 cents a pound on this product when colored.

In the earlier milling process where the wheat was ground between stones and the resulting meal separated through a simple bolting device, the flour was often improved by the method of storing which was called ageing, and which ageing process, while maturing the gluten to some extent, was clargely used for the purpose of whitening the flour. By the introduction of the roller process, which became general only within the last half century and only after considerable opposition, a flour was produced which was much whiter in color than was generally produced by the use of millstones. Some consumers of this flour complained that while it was whiter in color it would not make good bread, which was probably due largely to their prejudice against the new flour, commonly known as patent flour. In spite of this prejudice, the desire for the new flour became general and every mill was eventually compelled either to go out of business or adopt this system.

In the roller process of milling the wheat is first run lightly through a pair of rolls so that it is broken into fragments, and care is taken that the fragments be made as large as possible. The products from the roll are put upon the bolting machine and the coarser portion, including the bran and many larger fragments of the interior of the grain. is bolted off and carried to a second pair of rolls. where the process is repeated. This is again repeated and so the bran finds its way through several pairs of rolls. Each time a considerable quantity of the fragments of the interior portion of the grain are broken off, and by successive bolting and purification the fragments are obtained comparatively free from bran. These fragments are divided into various grades according to their purity, size, etc., and crushed to fragments between various pairs of smooth rolls. The fragments are called middlings. They can be made very pure, and when crushed to flour make an article which is very free from the material which commonly finds its way into the feed. It is to flour produced in this way that the name patent flour is given. Not all of the flour contained in the grain can be obtained in the form of middlings of such character that they can be sufficiently purified to make a better grade of flour. The remaining portion is mixed with fine fragments of bran and often of the germ which is the embryo of the kernel. This portion of the flour is generally named clear grade flour. It is darker in color than the patent flour, and when separated in this way is inferior for bread-making purposes because of the impurities which it contains. When the clear flour and the patent are united in one, they constitute a straight grade flour, which, while superior to the flour made by the old stone mill process, has more nearly its same general character. The improved process of milling has enabled the miller to produce a greater quantity of flour from the wheat as well as flour of a superior quality, for by the old stone method it was impossible to remove all the flour from the bran without also grinding up with the flour an excessively large portion of the bran. From a food and economic standpoint, the clear flour is superior to the patent flour because it contains more of the protein, more of the fat and less of the starch. The same is true of the straight grade flour, for it contains

the excess of these ingredients found in the clear, and at the same time the objectionable qualities of the clear are largely removed, because the impurities thus more diluted are, except as to color, more beneficial than injurious. The color of the flour which is removed by the bleaching process is distributed throughout the entire flour-producing portion of the wheat grain, but is more abundant in the portion nearest to the bran, which is also the most nutritious. It is the purpose of the modern bleaching process to remove the yellow color instantly. It should be clearly borne in mind, in this connection, that the yellow color which is removed by the bleaching process is entirely independent of any dark or inferior color produced by the presence of the bran which is ground up with the lower grades of flour.

The writers were given the opportunity to examine the first samples of flour bleached by the Andrews process which were brought to this country. A careful comparison of the bleached and unbleached flour was made by us and published in milling journals.4 Our examination of these flours was very thorough, and although we set about the analysis with somewhat prejudiced minds, we were unable to find any objections whatever to the sample of bleached flour, or any material differences between the two flours except that which was apparent in the color. Our findings with regard to this matter were put before the millers by one of the writers at the Operative Millers Convention, at St. Louis, and by the other at the meeting of the Federation of Millers at Buffalo. Shortly after this time and since, other investigators have made a careful study of the subject of bleaching flour and all, with the exception of two, have arrived at substantially the same conclusions that we gave in our first article and public lectures. Those prominent in these investigations are Fleurent,5 of France, Prof. Harry Snyder,6 of the Minnesota Agricultural Experiment Station, Prof. Ladd and Mr. Stallings,7 of North Dakota, Dr. Avery8 and later Prof. Alway,9 both of the University of Nebraska, and lately Dr. Ludwig Weil,10 Strassburg, Germany.

It has already been stated that the active agents in the bleaching of flour are nitrogen oxides and nitrosyl chlorid. Both of these substances result in decomposition products which give the usual test for nitrites. The test which is commonly used for detecting small quantities of this substance is known as the Griess-Ilosvay test, which is among the most delicate tests for any substance which is known to chemists. So delicate is this test that, according to Warrington, it is capable of detecting one part of nitrous anhydride in a billion parts of water. It was because of this very delicate test that it was possible to discover the residues of these gases in the flour.

The chemistry involved in the bleaching of flour has received much attention by us. We have isolated the coloring matter and have identified it or at least have established a very close relationship for it with other natural vegetable colors. It is not the province of this article to go into this phase of the question. The chemistry of this reaction will be taken up by us in a later article. The coloring matter of flour is the one material which shows the great avidity for uniting with the gases which are used in bleaching. The action on the coloring matter is instantaneous, and the amount required for the purpose of bleaching the flour is very minute. If an excess of the gas is used, it will eventually combine with other substances, primarily the gluten and finally the oil. It is only when the flour is overbleached and ruined that the action on these constituents of the flour is sufficient for it to be detected either by chemical or by physical properties.11 It was conceded in the flour bleaching case conducted in North Dakota, as well as at the hearing in Washington, D. C., that the average amount of nitrite-reacting nitrogen in commercially bleached flours is practically one part per million. In our own experiments we have often found much less than this amount. It has been still further conceded12 on the part of even those opposed to the bleaching of flour that only a small proportion, and in some instances not any, of the nitrite-reacting nitrogen is present in the bread. We have found this disappearance of nitrite-reacting nitrogen to be due in a large measure to the action of the yeast which is used in the bread.

The nitrite-reacting material of flours is also removed by the action of heat. This may be clearly shown by the tables given below. Quantities of spring patent flour were dried over night, and in the morning examination for nitrites was made in the usual way.

Temperature of oven.	Part per million nitrite- reacting nitrogen.
105° C.	None.
75° C.	0.22
40° C	0.37
Not dried	0.37
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Four lots of spring patent flour or 20 grams each were made into a sponge dough and put in the proving case over night. In the morning it was found the temperature had risen somewhat above 100° F. The flours were tested in the morning for nitrites in the usual way.

Zeast in dough.	Nitrite-reacting nitroge
None,	None
1 Gram	None
2 Grams	None
1 Gram plus chloroform.	0.37
In original flour	0.37

Four lots of spring patent flour of 20 grams each were made into dough as on the preceding day and allowed to stand over night at room temperature. Determinations of nitrites were made in the morning.

Amount of yeast.	Parts per million nitrite- reacting nitrogen.
None	0.37
1 Gram	0.031
2 Grams	0.018
1 Gram plus chloroform	0.37
In original flour	0.37

It will be noted that in the first experiment where the flour is dried over night at a temperature of 105° C. that all of the nitrite-reacting nitrogen disappears. At a temperature of 75° C. it is reduced from 0.37 to 0.22. At a temperature of 40° C. no change takes place. In experiment No. 2 it will be noticed that from flour made into a dough with water and allowed to ferment in a proving case all of the nitrite-reacting nitrogen disappears. The same is true when I gram and 2 grams of yeast are added to the flour before making a sponge. When, however, chloroform was added to stop the action of yeast and bacteria, the nitrites remained as in the original flour. In the third experiment we notice that this change does not take place in the sponge made directly from flour without the addition of yeast when kept at ordinary room temperature. Better action takes place, however, when one and two grams of yeast are added to the flour. There is a reduction then from 0.37 to 0.018. In the third experiment the nitrite-reacting nitrogen would have all disappeared had the correct incubating temperature been used, except when chloroform was added. From the results of this experiment we see that drying at sufficiently high temperature dispels all of the nitrite-reacting nitrogen and that living bacteria and yeast consume it.

That the presence of even considerable amounts of nitrite-reaction nitrogen in flour acts favorably to the development of yeast in dough and not as a preservative is apparent from the following:

TABLE SHOWI	NG THE IN	FLUENCE O	of SMALL QI	UANTITIES .	OF ADDED
Sodium	NITRITE O	N THE EX	PANSION OF	SOFT DOU	GHS.

0	Added nitrite nitrogen		Total volum	Total volume of dough.		
		of flour.	H. S.	L, S.		
Blank		0.0	800	860		
1 cc. 1% ni	itrite of se	oda. 20.3	900	860		
5 cc. 1%	" "	101.5	860	900		
10 cc. 1%	ы <i>и</i> и	203.0	840	900		
20 cc. 1%	u u u	406.0	300	260		

In making the above expansions 100 grams of Pillsbury's best flour were used in each dough in connection with the following other ingredients; water, 75 grams; yeast, 10 grams; sugar, 3 grams. Total dough, 188. In those doughs in which the solution of nitrite was used it was made to take the place of the other water used so that all the doughs contained the same amount of liquid. Expansions marked L. S. were at slightly higher temperature than those marked H. S., the temperature of the two lots being about L. S. 95° F. and H. S. 90° F.

It will be seen from the above that no effect injurious to the expansion of the dough is apparent when a quantity of nitrite-reacting nitrogen is added to the flour in quantities 200 times as great as is present in commercially bleached flour. On the other hand, we find this larger amount to be beneficial to such expansion. It was also found that when bleaching gases are added to the flour sufficient to produce nitrite-reacting nitrogen to the extent of 60 parts per million, the flour was entirely destroyed for commercial purposes because of the well-known xanthoproteic reaction whereby the flour was highly discolored, producing a reddish yellow shade, and because the character of the gluten was changed by the action of this large amount of the acid-producing gases upon the gluten. Even in this aggravated condition the growth of the yeast was not prevented and the bread-making property of the flour was not destroyed, although the bread was of inferior quality. Attention should be called to the fact that in the experiment noted above sodium nitrite was used, while in the gas-treated flour the sodium was not added, the acid in this case being in part at least united with the gluten, forming with it the well-known class of syntonin bodies. It is known13 that bodies of this character are continually formed during the process of bread-making by yeast, and are an essential step in such process of bread-making.

One of the contentions of those who are opposed to the bleaching of flour is that it leaves a deleterious substance in the nature of a preservative in the flour. The experiment cited above in which we have shown that the bacteria and yeast consume the nitrite-reacting material of the flour is sufficient answer to this contention

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We have been making daily examinations of flour received from all parts of the United States and from foreign countries, including Canada, England and South America. This has naturally brought us in contact with flours of all kinds, from all classes of wheat, and representing all methods of milling. This work has been done for the purpose of ascertaining the bread-making and commercial value of these flours. During the last five years probably 90 per cent. of the flours which we have received have been bleached, and we have not in a single instance found any flour which appeared to be in any way injured by the process of bleaching. The analyses which we make of these flours are very thorough, as the commercial value, which depends on the quality of the bread, is based on our reports. In addition to this general analytical work on flours, we have made a thorough and exhaustive study of many flours, bleached and unbleached, to determine any effects which might have been produced by the action of bleaching gases upon the bread-making qualities of the flour. We have found that in every instance the only essential change in the character of the flour was in a whitening of the color and in some instances an increase of the water-absorbing capacity of the flour. Where there has been any slight difference in the volume of the bread, which could not be attributed to natural variation in the work, it has been, if anything, slightly in favor of the bleached flour. The same is true of the number of loaves of bread which are produced from a barrel of flour. The amount of yeast necessary for the production of bread from bleached flour, which we always call the fermenting period, was in no way increased over that required for the unbleached flour. The quality of the gluten, as shown by its capacity for expanding into a good-sized loaf of bread, has not been found to be in any manner injured. The average value, which in our report is based upon the color of the flour, the character of the bread, the amount of bread and the size of the loaf, was a little higher for the bleached flour than for the unbleached, because the color of the flour and the color and quality of the bread were improved.

The flavor of bread is subject to much variation, due to the methods of bread-making, and is also one of the most difficult points to determine in the comparison of breads under examination. We have made careful comparisons along this line and have been unable to detect any deterioration of flavor when both flours, bleached and unbleached, were submitted to the same process of bread-making. We have already called attention to the fact that the patent flour, as compared with the clear, is relatively deficient in protein, fat and mineral matter, and for this reason possesses less nutritive value than the clear flour. We have also pointed out that when these two flours are combined they constitute the straight grade flour which is intermediate in character between the patent and the clear. While an experienced eye can readily distinguish between the grades of flour used when a straight grade flour from which the color has been removed is baked into bread and compared with bread from a patent flour from which the color has not been removed, it is our experience that preference will be given to bread made from the straight grade flour.

#### TYPICAL ANALYSIS OF BLEACHED AND UNBLEACHED FLOUR.

	Standard spring patent bleached.	Spring patent not bleached.	Hard winter wheat bleached.	Hard winter wheat not bleach- ed.	Durum bleached.	Durum not bleached.
Gluten, per cent	11.0	11.0	11.5	11.5	10.5	10.5
Ash, per cent	0.42	0.42	0.40	0.40	0.60	0.60
Absorption, per cent	: 61.0	61.0	62.0	62.0	61.0	61.0
Color	100.0	99.0	100.5	99.0	97.5	95.0
Loaves, per bbl	100.0	100.0	100.6	100.6	100.0	100.0
Size of loaf	100.0	100.0	100.0	100.0	98.0	98.0
Quality of loaf	100.0	99.7	100.3	99.7	99.0	98.0
Average value	100.0	99.7	100.4	99.8	98.6	97.7
Fermenting period	100.0	100.0	102.2	102.2	97.7	97.7
Quality of gluten	100.0	100.0	97.8	97.8	102.4	102.4

#### NUTRITIONAL EXPERIMENTS.

Twenty-four white rats were purchased of a rat dealer in Chicago during the month of July, 1908, the lot including 10 females and 14 males. These were put in several cages, weighed after dividing, and arranged on shelves in a cool, well-ventilated room, where all were given similar treatment as to food, water and ventilation. Boxes suitable for their protection and comfort were put in each cage. On the 28th of July the rats were arranged so that each of two cages should contain seven males, and each of two others should contain five females. An effort was made to have the weight of males in the two cages about equal and also the weights of females of the two cages about equal. The experiment in feeding was begun on the 28th of July. Within two or three days one of the rats died from causes common to rats and in no way connected with the experiment. The weights of the rats at the beginning and end of the experiment are shown in the table given below:

WEIGHTS AND GAIN OF RATS.

Flour bleached.			Flour not bleached.			
	5 Females.	6 Males	11 Total.	5 Females.	7 Males.	12 Total.
July 28	325.0 g.	482.0 g.	807.0 g.	329.0 g.	553.0 g.	882.0
Oct. 14	425.0 g.	572.0 g.	997.0 g.	439.0 g.	670.0 g.	1102.0g.
Gain	100.0 g.	90.0 g.	190.0 g.	110.0 g.	117.0 g.	227.0g.
Gain %	30.8%	, 18.6%	23.5%	33.4%	21.1%	25.7%

Figures show weight in grams and also gain in per cent. of the original weight.

A barrel of flour was procured from the Berger-Crittenden Milling Company, of Milwaukee, which was known at that time to have no bleacher, and which was fully borne out by tests to be entirely unbleached. Soon after receiving at the laboratories, the flour was put into four 50-pound tight tin flour bins for preservation. The flour from one of these bins was passed through the gases of the Alsop bleaching machine in a manner identical with that used in the flour mills. The time required for the passing of the flour through the gases produced from a generator suitable for a 100-barrel mill was two minutes. Tests were made for the amount of nitrite in the flour bleached in this manner, and the nitrite-reacting nitrogen was found to be three-tenths parts in a million. The flour was again run through the bleaching machine in the same manner as at first and the nitrites again determined. The amount found after this second bleaching was 0.56 part per million of nitritereacting nitrogen. On the following day biscuit was made from the bleached and unbleached flour. No nitrites whatever were found in either the flour or the biscuit unbleached. In the biscuit from the bleached flour the amount of nitrites found was equivalent to 0.3 part per million of nitrite-reacting nitrogen. It was considered that the bleaching of this flour had not been sufficient to meet the objections which might be raised as to its having been given sufficient treatment, and it was put through the bleacher twice more in the same manner as already described. After this last bleaching the nitrite determination was again made upon the flour on July 31, and was found to be, on the flour four times bleached by the Alsop machine, equivalent to 0.79 part of nitrite-reacting nitrogen per million. The flour was now put aside in the laboratory for use in the making of bread and biscuit for the experiment. The composition of these flours before and after bleaching was determined in the usual manner in which we determine the characteristics of different flours in making our commercial laboratory reports, and the results are given herewith:

	Spring		6796
	patent.	6796	bleached.
Gluten, per cent	10.5	11.0	10.9
Ash, per cent	0.42	0.49	0.49
Absorption, per cent	62.0	62.0	62.0
Color	100.0	99.0	99.5
Loaves, per barrel	100.0	100.0	100.0
Size of loaf	100.0	100.0	100.0
Quality of loaf	100.0	98.7	99.0
Average value	100.0	99.4	99.6
Fermenting period	100.0	102.5	102.0
Quality of gluten	100.0	97.5	98.0
Moisture		12.85	12.0
Acidity, as lactic acid		0.117%	0.117%

The bread used in these experiments was made substantially as follows: 500 grams of each flour were mixed with 310 cc. of water, 12 grams of compressed yeast, 15 grams of sugar, 7.5 grams of salt and 15 grams of lard. The dough was allowed to develop for two hours with one working, which working was done at the end of the first hour. At the end of the two hours the dough was molded into a loaf and put in the pan, where it was allowed to prove until it was ready for baking in the oven. It was baked usually in the electric oven until sufficiently done, requiring about 45 minutes. It was then taken out of the oven, cooled, and subsequently put in suitable tin boxes to preserve for the experiments. A portion of the time biscuits were substituted for the bread, so that the rats might have some variety of diet, and so that the teeth of the rats might be kept in somewhat better condition by their gnawing the harder biscuit. The biscuits were made substantially as follows:

Two hundred and fifty grams of each flour, 115 grams of water, 2 grams of salt, and 5 grams of Dr. Price's cream baking powder were mixed together, rolled to suitable thickness, cut with a tin biscuit cutter, put in a pan and baked in the electric oven. The amount of nitrites remaining in the bread and biscuit was determined on many of the samples, and the average amount found was 0.03 part per million of nitrite-reacting nitrogen for the bread and 0.24 part for the biscuit from the bleached flour. The average amount of nitritereacting nitrogen in the bread from the unbleached flour was none and in the biscuit from the un-

bleached flour was 0.02 part per million. The bread in these experiments was purposely made by an unusually short process, which would tend to leave in it much more nitrite-reacting nitrogen than would remain when the bread is given the usual amount of fermentation. Even in this short process of bread-making the nitrites were in many instances entirely removed. The rats were fed night and morning of each day and were supplied with sufficient water in a suitable tin vessel placed in their cage. The amount of bread or biscuit given at each feeding was equivalent to about 5 per cent. of the weight of the rats daily. This amounted to about 50 grams for the rats fed bleached flour and a trifle more for the rats fed unbleached flour.

The supply of bleached flour used in feeding these rats was exhausted about the 3rd of October, and another quantity of the same flour was run through the bleaching apparatus three times, in the same manner as on the previous occasion. The amount of nitrite-reacting nitrogen in the flour thus bleached was 1.56 parts per million, which was much in excess of what would possibly be introduced into the flour by a single bleaching. The rats were fed on bread and biscuit from this flour for about one week. The amount of nitrite-reacting nitrogen in the biscuit from this flour was 0.6 part per million and from the bread was 0.3 part per million.

At the end of the feeding period a considerable number of the rats were dissected by the Biological Division of the Columbus Laboratories under the direction of Dr. Adolph Gehrmann, and the data obtained in this manner is given below:

Notes on individual rats as dissected: The rats were killed with chloroform, stretched in a proper manner, and opened by a ventral incision extending the entire length of the body. Examinations were made on the following points, and where normal no mention is made of them in the following notes:

Nutrition and blood supply of intestines; general condition of intestines, stomach, liver, kidneys, spleen, heart, lungs, and also of amount of fat in mesentery and omentum. Portions of organs, as follows, were removed and put into a seven per cent. solution of formaldehyde for subsequent examination under microscope: Anterior portion of right lobe of liver; one-half of left kidney; middle portion of stomach including portions of the cardiac and pyloric ends; one-half of spleen and such other special portions as occasion required. The remaining portions of each rat were also preserved in formaldehyde for any future examinations which might be desired.

NOTES ON SERIES FED BLEACHED FLOUR.

## Cages I and 2.

Rat No. 1. Stomach partially distended with food.

Rat No. 4. Tapeworm in intestines.

Rat No. 7. White nodule of fat within capsule of left kidney. 1/2 right kidney taken for microscopic examination.

Rat No. 11. Nothing abnormal found.

Rat No. 13. Much congestion of abdominal vessels. Stomach distended with gas; small ulcer on interior wall. Tapeworm in intestines; hydatid cyst in liver. Hardly any fat in mesentery. This was the most unthrifty rat in the experiment.

Rat No. 16. Intestines somewhat congested; hardly any fat in the mesenteries.

Rat No. 23. One of the most thrifty-looking rats in the entire experiment. No defects of any kind whatever found.

SERIES FED FLOUR NOT BLEACHED.

## Cages 3 and 4.

Rat No. 2. Stomach fairly well distended with food. Examination showed nothing abnormal.

Rat No. 5. Tapeworms in small intestines. Spleen appeared somewhat small.

Rat No. 8. Normal appearance throughout.

Rat No. 10. Apparently abnormal contraction between cardiac and pyloric ends of stomach causing hour-glass shaped organ.

Rat No. 14. Some congestion of abdominal blood vessels. Small intestines contained three very large tapeworms. Stomach somewhat distended with gas. Darkened spots on anterior portion of anterior lobe of liver. Spleen presented irregular mottled appearance. Slight consolidation in upper lobe of left lung.

Rat No. 17. Small tapeworm in small intestine.

Rat No. 18. Some gas in intestines. Some tendency toward diarrhea. Small infiltration of fat into capsule of left kidney.

Rat No. 19. Slight congestion of small intestines. One very large tapeworm in small intestines.

IRREGULARITIES FOUND CLASSIFIED AS TO ORGANS

#### EXAMINED.

Nutrition and Blood Supply of Intestines.-Some congestion of abdominal blood vessels found in

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rat No. 14, unbleached series, and much in rat No. 13 of bleached series.

Abnormal Condition of Intestines.—Tapeworms found in rats Nos. 4 and 13 of bleached flour series; in Nos. 5, 14, 17 and 19 of unbleached series. Some inflammation in rats Nos. 13 and 16 of bleached series; also in rats Nos. 14 and 19 of unbleached series. Tendency to fermentation and diarrhea were noticeable in rat No. 18 of unbleached series.

Stomach.—Stomachs were more or less distended with gas in rats No. 13, of bleached series, and No. 14, of unbleached series. Rat No. 10 of the unbleached series showed marked hour-glass contraction between cardiac and pyloric ends.

*Liver.*—Hydatic cysts were found in rat No. 13 of bleached, series. Darkened spots were found on the anterior portion of the anterior lobe of No. 14 of the unbleached series.

*Spleen.*—That of No. 5 of unbleached series was unusually small and that of No. 14 of the same series presented an irregular mottled appearance.

*Kidneys and Renals.*—Whitish nodules of fat were found within the capsules of one of the kidneys of No. 18, of unbleached series, and No. 7, of bleached series.

Heart .- No abnormalities were apparent.

Lungs.—Slight areas of consolidation in upper lobe of left lung of No. 14 of unbleached flour series.

Fat in Mesenteries and Omentum.—Deficient in mesenteries of No. 13 of bleached flour series, and in No. 16 of same series. Also in No. 14 of unbleached series.

SERIES	FED	ON	FLOUR	Not	BLEACHED.
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No. of rat.	Sex.	Weight in grams.	General appearance.
. 2	Male	132	thrifty
5	Female	82	fairly thrifty
8	Male	92	fairly thrifty
10	Male	83	not thrifty
14	Male	71	not thrifty
17	Male	79	not thrifty
18	Female	84	fairly thrifty
19	Female	76	fairly thrifty
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The remaining rats of this series were thrifty in appearance except one female which did not appear thrifty.

Sec. 1 March	SERIES FEI	D ON BLEACHED F	LOUR,
No. of rat.	Sex.	Weight in grams.	General appearance.
1	Male	110	thrifty
4	Female	110	thrifty
7	Male	120	fair
11	Female	83	not thrifty
13	Male	47	not thrifty
16	Male	74	not thrifty
23	Female	129	very thrifty

The unkilled rats of this series were of thrifty appearance.

A series of feeding experiments was conducted with rats covering a period of several months. The bread which they received was made from flour which had been bleached with nitrosyl chlorid gas. The general findings were substantially the same as in the experiment detailed above. There were noticed no nutritional disturbances of any kind, and the post-mortem examination showed no lesions which in any way could be attributed to the bread made from this flour. In addition to the feeding experiments conducted with the rats, we have fed the guinea pigs, which we have always on hand for experimental purposes, bread from bleached flour covering a period of more than six years. During this time we have never once noticed any symptom or sign in the health of the animals which could in any way be traced to the bread made from bleached flour. Post-mortem examinations never revealed any lesion which could in any way be associated with the food which they had been receiving.

Ladd<sup>14</sup> has given results of experiments where alcoholic extracts of bleached and unbleached flours were evaporated under specified conditions and the residue administered to rabbits. The extracts from the bleached flour were reported by him to have produced death in a short time, the *post-mortem* findings being those of a corrosive poison. These experiments have been carefully repeated by Prof. Haines and Prof. Snyder,<sup>12</sup> who were not able to obtain results showing any indication of poison in the rabbits.

In working in conjunction with Prof. Haines at the Columbus Laboratories in November, 1908. several lots of flour were taken for extraction with 90 per cent. alcohol following the same general lines of investigation as laid down by Ladd. The flours used in these experiments were Sunnyside, marked 6796 unbleached, the same bleached by passing through Alsop gases four times, and the same bleached by very excessive treatment with nitrosyl chlorid. The flour bleached by the Alsop process contained 2.8 parts of nitrite-reacting nitrogen per million; that excessively bleached by the nitrosyl chlorid was entirely destroyed for commercial purposes and contained 30 parts nitritereacting nitrogen per million. Of the three samples of 6796 each flour in lots of 750 grams each was put in each of two bottles and 1750 cc. alcohol added to each bottle, making a total of 1500 grams of each flour and 3500 grams of alcohol for each. These were agitated thoroughly during the afternoon of the day, were allowed to settle over night, and the clear supernatant fluid was poured from each on the following morning. This liquid was filtered to remove any starch which might be carried over. The total amount of liquid obtained for the flour treated with nitrosyl chlorid was 1280 cc.; that for the flour treated by the Alsop process 1210 cc.: that for the flour not bleached was substantially the same. Each of these liquids was introduced into a heavy glass globe of about 1500 cc. capacity. Those having the larger amounts were divided in two portions and a part of the liquid evaporated off before the remainder was added. The globes were connected with Liebig condensers at the lower end of which was an Erlenmeyer flask with a wide neck and side tube such as is used for vacuum filters. The whole was then connected with the small vacuum pan in the laboratories. Two of the glass globes were put in a large dishpan filled with water and so arranged that heat could be applied. Two others were put in another pan in a similar manner. The evaporation was then done by the assistance of the heated water and the vacuum pan. The time required for the evaporation was six hours. The maximum temperature of the liquid in the several flasks was 48° C. The average vacuum as shown by the manometer connected with the vacuum pan was from 23.5 to 25.5 inches. The removal of the alcohol in this manner was quite complete, but to still further remove small quantities which were in the flask, each flask was closed with a pledget of cotton and placed in the incubator heated by one 16-candle power electric light so that the temperature was about 37° C. and allowed to remain at this temperature over night. In the morning there was no odor of alcohol in any of the flasks. The residue in the several flasks was of an oily consistency with some sediment which was not entirely soluble in the clear oil. The oil was carefully poured from the flasks containing the Alsop process bleach and the nitrosyl chlorid bleach on the samples of Sunnyside flour, and allowed to drain into beakers until no more oil would flow from the flasks. These two flasks were carefully broken and the material remaining in them scraped from the glass. The weight of the total residue obtained from the flask treated with nitrosyl chlorid was 8.9 grams, and that from the flask of which the flour was treated with the Alsop process was 6.746 grams. 3.266 grams of that taken from the nitrosyl chlorid flour were weighed

and mixed with water and 2 cc. of 95% alcohol so that the total volume of the mixture was about 10 cc. This amount of fat corresponded to nearly 250 grams of flour. It was taken up by means of a small glass syringe and fed to a rabbit weighing 2130 grams. No ill effects were apparent the rabbit. It went to eating vigorously in in the course of 15 minutes and showed no symptoms of distress of any kind. The oil from the Alsop process bleached flour was divided into two portions as nearly equal as possible. The first of these was treated in the manner described above and given to a rabbit, but unfortunately the soft catheter at the end of the syringe was introduced into the trachea which led to disturbance in the breathing of the rabbit, so that it was considered advisable to kill it shortly after and find the cause of the distress. This was done, and a post-mortem examination showed the lungs to contain much foreign matter and naturally highly congested. The other organs were found to be perfectly normal. The remaining portion of the Alsop process flour extract was fed to another rabbit in the same manner as that fed from the nitrosyl chlorid bleached flour, and no indications of distress whatever were occasioned by this flour. The weight of the rabbit which was fed was substantially that of the other, 2100 g. Both rabbits were kept under observation for a considerable time and no indication of distress or unusual symptoms were apparent. The acidity of the alcohol extract was identical for the three samples of Sunnyside flour, the unbleached, that bleached by the Alsop process, and that strongly overbleached by nitrosyl chlorid.

In the flour bleaching trial<sup>15</sup> in England, Wilcox and Luff, working independently, repeated experiments with rabbits as outlined by Ladd using unbleached, commercially bleached and strongly commercially bleached flour, making all told more than 50 experiments and report that in these experiments they were in no instance able to kill the rabbits because of any poisonous substance in the extracts, or to produce any toxic symptoms other than such as might come from the alcohol which was administered in connection with the extract. Judge Pollock, of North Dakota, and Judge Warrington, of England, both having listened to the testimony detailed by Ladd and the other experimenters, as mentioned above, sum up in their decision that bleached flour does not contain a deleterious substance.

If we calculate that the total amount of nitrous and nitric acid which might be present in one of the flours which was used by Ladd14 for his test, based upon double the amount of nitrite-reacting nitrogen found (3 parts per million) and all calculated to nitric acid, it would be equivalent to only 5.4 milligrams (1/10 drop) of nitric acid. This amount of nitric acid is practically 1/100 the medicinal dose of nitric acid as given in the U.S. Dispensatory, and according to Ladd's testimony the liquid administered to the rabbits was much greater in amount than necessary to produce the dilution specified in that work for internal doses of nitric acid. Under the circumstances we could not expect any corrosive action from this amount of acid, even assuming that it could have been separated out of the flour as free acid without in any manner combining with the organic matter of the flour and alcohol.

It has been asserted by Prof. Ladd and others testifying against the bleaching of flour that proof of the action of the bleaching gases upon the oil of commercially bleached flours was apparent from the altered iodin absorption of the oil. Careful work upon this point had been done by the United States Department of Agriculture, and the results of the work submitted as testimony in the North Dakota flour bleaching trial14 in which it was shown that in nine cases out of ten in which the "unbleached" samp'e did not show the presence of nitrites the difference between the iodine absorption of the fat of the bleached and unbleached flours was within the limit of experimental error, and this, notwithstanding the limit of experimental error on this test for iodine absorption, was considered to be not greater than the very close figure of one-half of one per cent. The readiness with which oil of wheat takes up oxygen of the air in its preparation for analysis is a potent factor in altering the iodine absorption of the oil.

The same table of analysis also gives detailed results of a considerable number of the determinations of the acidity of the bleached and unbleached flours in which the difference between the considerable number of samples examined was again within the limit of experimental error. We ourselves have never found such increased acidity in our own experiments with commercially bleached flours. Even assuming it to exist to a slight extent, it would not act injuriously on the breadmaking quality of the flour, for as already stated, the changes brought about by the action of acids produced during fermentation are among the constant and necessary steps in the preparation of dough for baking.

As throwing some light on the influence of the bleaching gases upon the digestive action of pepsin on the proteids of flour, we will give the results of the following digestion experiments: In each case 10 grams of flour were used, one unbleached, the other bleached, showing 2.8 parts of nitritereacting nitrogen per million. Each was treated with 50 cc. of the official pepsin and hydrochloric acid mixture and kept at animal temperature for one hour with occasional agitation. Then both were made up to 250 cc. with cold water and 50 cc. were filtered from each. These were examined by the Kjeldahl method in duplicate and the unbleached gave 0.0369 g. nitrogen, the bleached 0.038 g. in one set, and in the other set 0.0361 g. for unbleached, and 0.0375 g. for the bleached. We thus see that the peptonizing action of this enzyme was in no way retarded because of the presence of the nitrites.

Diazo Test.—100 grams of unbleached flour were introduced into a flask of about one liter capacity, and carbon dioxide passed into it for 1 1/4 hours with frequent vigorous agitation. Dilute hydrochloric acid which had been recently boiled was added warm and the mixture agitated. The evolved gas was then swept by a stream of carbon dioxide into a Schiff azotometer containing the usual solution of caustic soda. A small amount of gas passed to the top which could not be absorbed by repeated agitation. The volume of this gas was 1.4 cc. It was tested with a lighted paper. It did not burn nor did it support combustion when tested as indicated by Prof. Ladd.14 The above experiment, carried on in coöperation with Prof. Haines, was carried out as detailed by Ladd14 and used by him as testimony in North Dakota to show the presence of diazo compounds of the nature of tyrotoxicon resulting in bleached flour from the action of the bleaching gases upon the constituents of the same. The amount of gas which he obtained from the bleached flour is substantially the same as we obtained in the above experiment from unbleached flour, and is undoubtedly air which adheres to the particles of flour and which cannot be removed even by the most careful and persistent treatment with carbon dioxide. That the experiments tend to show the presence of tyrotoxicon, or that such tyrotoxicon would be formed by the action of bleaching gases

on flour is not probable when we remember that this material, as found in cheese and other milk products, is wholly the result of bacterial growth, and that it is of an exceedingly unstable nature.

Nitrites and nitrite-reacting substances, which form the chief basis of the contentions of the opponents of bleached flour against that material, are very widely distributed throughout the mineral, vegetable and animal kingdoms. We have entered more into detail in the question of this distribution in a former paper,16 and will summarize the facts only very briefly here. Nitrites are found in many drinking waters. They are objected to in that connection because their presence is an indication of the bacteriological or sanitary condition of the water, and not because of poisonous qualities of the nitrites themselves. Nitrites are found in considerable quantities in soils and in such common foodstuff as salt, baking soda, and baking powder. They have been found in small quantities in green vegetables including celery and other articles of food. They have been found in considerable quantities in preserved meats, especially those in the preparation of which saltpetre is used and the formation of nitrites is apparently an essential step in the preservation of meats by saltpetre. We have found in ham bought in the open market as high as five parts per million of nitrite-reacting nitrogen, or five times the average maximum amount found in commercially bleached flour. Nitrite-reacting nitrogen is found in all parts of the animal body, and is nearly always present in comparatively large quantities in the human saliva, including the normal saliva of healthy nursing infants. We have found in the saliva of man as high as 13 parts per million of nitritereacting nitrogen, which is 13 times that found in bleached flour. While nitrite-reacting nitrogen can nearly always be found in flours which have been bleached with oxides of nitrogen, the presence of nitrites in flour is not a proof that they have been bleached, as shown by Weil10 who has obtained reaction for this substance by the Griess-Ilosvay test in samples of unbleached flour and in wheat which he has examined.

After having devoted much time during the past several years to a careful study of the question of flour bleaching in all its relations,<sup>17</sup> we would sum up our findings briefly as follows: The essential action of the bleaching of flour is to remove from it a small amount of yellow color which in itself is in no wise a valuable constituent in the flour

from a food standpoint, and the presence of which is objectionable because it detracts from the flour in the eyes of the consumer by whom the demand for flour is created. The best results are obtained by its use only when thorough purification and cleaning are adopted, and it in no way contributes to the covering up of an unsound or damaged condition in the wheat. Any imperfections in purification or cleaning are made more apparent by the bleaching because these defects are in a manner covered by the coloring matter when it is present. Any improvement in quality brought about by removing an unusually large amount of color present in a flour which was inferior because of the presence of such excess of color certainly cannot be looked upon as in any way injuring or deceiving the consumer, as has been contended by some, for the cause which produced the inferiority now no longer exists. The purpose of the bleaching is to remove and not to conceal the inferiority. The prohibition of the bleaching of flour will curtail the use and cut down the price of durum wheat and all wheats which have an intense yellow color in spite of the fact that aside from this some of these wheats produce flour of the very highest quality. The effect of this is naturally felt more by the producer of wheat and the consumer of flour than by the miller whose prices are regulated by market values and competition. The most searching investigations have failed to show the presence in the commercially bleached flour of any substance that in the minute quantities in which it is present is in any way injurious to the bread-making qualities of the flour, or is in any way poisonous or has any toxicological or preservative action, or any action which is prejudicial to digestion or nutrition.

Oct., 1909

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THE COLUMBUS LABORATORIES, CHICAGO.

#### THE TITANIUM ARC.

### By ISADOR LADOFF. Received July 17, 1909.

I was fortunate to be present when Dr. E. Weintraub succeeded in striking an arc between two mercury terminals *in vacuo* by his ionization method at the Research Laboratory of the General Electric Co., then just called into existence by the sagacity of Mr. C. P. Steinmetz.

The mere fact that metallic vapors, even under such artificial conditions as a high vacuum, could give so much light was a revelation to me. I, of course, noticed the undesirable color of the arc and even at that time considered the mercury of very limited importance as a source of illumination. However, I realized fully the immense scientific interest of this arc. The idea occurred to me that some metals, or combinations of metals, might be found which would give satisfactory arc light illumination in the free air.

Since that time the idea of a metallic arc in the open air for lighting purposes turned into a fixed idea with me. In the spring of 1902 I was occupied in eliminating from a molten bath of copper the occluded gases. The color of the copper vapors, by a very natural association of ideas, recalled to my mind the mercury arc. I then started to study the voltaic arc, with the view of advancing my scheme of a metallic source of illumination in the open space.

It is a matter of common knowledge that carbon possesses the highest point of electro-vaporization among all-non-metallic elements known to chemistry and that carbon was therefore considered as the only material so far available or arc lighting. The connection between the point of vaporization and intensity of the arc light could be easily explained by the following familiar analogy:

Water subjected to heating in a vessel absorbs the heat supplied to it until it reaches the vaporization point. From the time this temperature is attained all the additional supply of the heat energy is consumed in the work of evaporization. It is obvious that the heating of a black body like carbon, whose vaporization point is very high, must result in the manifestation of some other form of energy expenditure than mere vaporization. This form of energy is light. The higher the vaporization point of the body heated-the more intensely luminous must be its arc. In fact, its light must increase with the temperature. This is known to be strictly true of so-called black bodies, of which carbon is the nearest approach to theory. This line of reasoning compelled me to look for some metallic compound or metal having an electrovaporization point nearest to that of carbon.

I ound that among all metals titanium possesses the highest point of vaporization,<sup>1</sup> and that this point is the nearest to the point of vaporization of carbon.

Another consideration in favor of using titanium as a material for arc light illumination was, that among all metals it furnishes the richest spectrum as far as the number of lines is concerned. Indeed, titanium excels all non-rare metals in the nature and extent of the light-giving part of the spectrum. Thalen counted 201 lines in the red part of the spectrum out of 6,556; about 4,163 in the violet. Leveyng and Dewar counted between the same four new ones and Corner 25 in the ultraviolet, together 230 lines, two in the red, 17 in the orange, 32 in the yellow, 70 in the green, 35 in the blue, 45 in the indigo and 4 violet lines. 118 Fraunhoffer lines were recognized in the photosphere of the sun; 32 of these were artificially restored by Leveyng and Dewar to their actual state.

One drawback possessed by titanium as a source of illumination is its comparatively low electric conductivity.

With this conclusion in my mind, I made a great effort to find out whether my purely theoretical considerations were corroborated by facts. I ransacked my memory and tried to get

<sup>1</sup> See Mendelejéff's tables.

as many compounds of titanium as I could and tried them in the arc. I recall especially a piece of metal, or rather alloy of metals, containing iron and titanium in unknown proportions. The arc was very brilliant and white. Evidently there was more than a mere fancy behind my theoretical conclusions. Unfortunately, I was at this time again prevented from following up this line of investigation.

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Titanium belongs to that class of rare metals that are not rare. As a matter of fact, titanium is probably as abounding in nature as copper. The reason why titanium enjoyed the reputation of being rare is most likely the fact that its technical application was rather limited. Titanium compounds are used in coloring porcelain and artificial teeth, leather, etc. There were attempts made by English metallurgists, among others Mr. Mushet, to use titaniferous iron ores for smelting. Moissan was the first to prepare some impure metallic titanium. Mr. Auguste J. Rossi was the first to use titanium alloys for the purpose of improving steel.

In the spring of the year 1903 I started to investigate the best methods of manufacturing solid magnetite arc light pencils. The main defect of these pencils consisted in their high resistance to electricity at ordinary temperatures.

The pencils belonged to the second class of conductors, *i. e.*, oxidic bodies conducting electricity only at comparatively high temperatures. The first step toward improving the solid magnetite pencils consisted in reducing their electric resistance. The encasing in metallic (iron) tubes was a classical method. The iron tube served the same purpose as the glower or heater in the Nernst lamp. However, the defects of this method were many and varied. The resistance of the body of the pencil remained unaffected and the arc had the tendency to run from the body of the pencil, according to the line of least resistance, on the iron tube, causing a sudden drop of luminosity.

In the light of my investigations of the ferrotitanium arc, it appeared to me clear that the only way to perfect the magnetite pencils is to radically change their physical and chemical properties by reducing them to the metallic state. My first step in that direction was the elimination of the hematite from the composition of the body, which proved, among other things, the mythological nature of the "binding properties" of the red oxide of iron. The experiment leading to that elimination was conducted as follows: I made up three sets of pencils under identically the same conditions and of the identical material and composition with the difference that in one case I put in the customary 20 per cent. of hematite, in the second case I omitted this 20 per cent. of hematite, and in the third case I made the body of the pencil entirely of the red oxide of iron. All pencils contained equal quantities of titanic acid. The best photometric results were obtained from the pencil principally composed of magnetite. The ordinary mixture containing 20 per cent. red oxide of iron in addition to magnetite furnished intermediary results.

I then followed up these experiments with a series of similar tests, proving conclusively that the deoxidation or metallization of the electrode must lead to technical and commercial success. The tests made proved that a reduced pencil gave twice the luminous efficiency of a non-reduced one, both containing equal quantities of titanic acid. Besides this the reduced pencils had the advantage that they could contain comparatively high quantities of titanium without turning inoperative. The metallic pencils possessed greater strength, uniformity, and a better appearance. Their resistance was reduced to negligible values. The process of oxidation going on in the arc liberated an immense quantity of heat, and in this way relieved the electric field of the necessity of generating it through the process of overcoming useless resistance.

The magnetite pencil was oxidic from start to finish. The metallic pencils presented three phases: The first phase was presented by the electrolytic or oxidic material used for the manufacture of the pencils—the oxygen compounds of iron and titanium; the second phase was covered by the metallic ready-made pencil; the third phase and final one consisted of the electrolytic or oxidic material resulting from the process of arcing.

The process of deoxidation or metallization is practically a process of storing up energy in the body of the electrode, the energy of combustion. The positive coefficient of the metallic pencil, *i. e.*, its property of increasing in electric resistance with rise in temperature was helpful in steadying the arc. In short, the magnetite pencil was by the process of metallization turned a technical and commercial success equal to that of the ferro-titanium electrode prepared from the alloy manufactured in the electric furnace.<sup>1</sup>

<sup>&</sup>lt;sup>1</sup> The color of the respective arcs was, however, different. The ferro-titanium made in the furnace was bluish white, white in the arc of the reduced pencil was rather yellowish white.

In order to determine the difference in the behavior of the pencils according to their conductivity, a number of them were partially reduced in carbon at 1,150° C. Their resistance varied from 11 to 28.56 ohms. Pencils heated without carbon at the same temperature at the same time showed a resistance varying from 5,400,000 to 9,830,-000 ohms. As the degree of reduction seemed to me to play some rôle in the arc, I made a few experiments to determine the influence of the process of oxidation in the arc. Electrodes for these experiments were prepared in the usual way and manner. One batch contained the usual proportion of hematite and magnetite, the second batch did not contain any hematite at all, and the third batch did not contain any magnetite at all. All of these three batches contained an identical quantity of titanium oxide (of 7.5 parts) and all were treated at the same time under exactly the same conditions, as far as practicable. The most luminous arc was produced by the pencils containing only magnetite and titanium oxide; the least luminous arc was produced by pencils composed only of hematite and titanium oxide. The mixture composed of 80 parts of magnetite and 20 parts of hematite produced an arc of medium luminosity. As hematite represents the final product of oxidation of iron, the conclusion was near at hand that oxidation is a factor favorable for the luminosity of the arc. i. e., the more space there is left for the oxidation of the material of the electrodes in the arc, the higher must be the luminosity of the arc.

For the purpose of verifying this conclusion, additional experiments were conducted. Electrodes of the identical batch were subjected to various degrees of reduction and the luminosity of their arc tested. In each case the more reduced pencils gave a higher luminosity, and the luminosity of the perfectly reduced pencils was about double that of the luminosity of the non-reduced pencils of the same batch. A batch containing 50 per cent. magnetite and 50 per cent. titanium oxides was subjected to various degrees of reduction with the following results:

Well reduced pencils produced an arc of a lumi-

nosity equal to	1271	candle	powe
Partly reduced pencils produced an arc of a lumi- nosity equal to	1000		
Poorly reduced pencils produced an arc of a lumi- nosity equal to	868		
Non-reduced pencils produced an arc of a luminosity equal to	700		

All these are horizontal measurements. The current used was a direct one of 3.4 amperes,

with 75-80 volts drop across the arc. The volts of the standard were 30.1 of 63 candle power. The reduction not only about doubled the efficiency of the arc, but allowed the use of high percentages of titanium oxide in the mass of the pencils. In the non-reduced or partially reduced pencils an addition of about ten parts of titanium oxide was excluded, because it caused the arc to flicker to such an extent that no photometric reading could be taken with any degree of accuracy, if at all. The arc of the reduced pencils did not flicker very badly even when as high as 90 per cent, of titanium oxide was used in addition to 10 per cent. of magnetite as the mass for the pencils.

There appears, however, to be a limit beyond which any further addition of titanium oxide is not advantageous. The maximum efficiency of the arc is obviously not the only thing to be considered in our case. The best results obtained was with pencils containing about 50 per cent. of titanium oxide and 50 per cent. magnetite. The perfectly reduced electrodes were metallic through and through, harder than tool steel, and took an excellent polish. When ready for use they looked like ordinary steel cylinders.

From Auguste J. Rossi I procured technically important quantities of iron and titanium and also copper and titanium alloys. A series of tests convinced me of the high technical value of titanium alloys for arc light illumination. A patent, No. 840,634, was granted to me Jan. 8, 1907. The claims allowed are as follows:

1. An arc light pencil consisting for the most part of an alloy of titanium with another metal, possessing greater conductivity than titanium.

2. An arc light pencil consisting for the most part of an alloy of titanium with iron.

3. An arc light pencil comprising an alloy of titanium with other metals possessing greater electrical conductivity than titanium, said alloy constituting more than ten per centum of said pencil.

4. An arc light pencil comprising an alloy of titanium with iron, said alloy constituting more than ten per centum of said pencil.

5. An arc light pencil comprising an alloy of titanium with iron, said titanium constituting more than five per centum of said pencil.

6. An arc light pencil comprising an alloy of titanium with other metal possessing greater electrical conductivity than titanium, said titanium constituting more than five per centum of said alloy. 7. An electrode for arc lighting consisting of an alloy of titanium and other metals possessing greater electrical conductivity than titanium.

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8. An electrode for arc lighting consisting of an alloy of titanium.

9. An electrode composed of ferro-titanium.

10. An electrode containing ferro-titanium.

11. An electrode containing considerable amounts of iron and titanium in metallic state.

12. An electrode formed from any alloy of iron and titanium containing about thirty-five parts of titanium and sixty-five parts of iron.

13. An electrode formed from an alloy of iron and titanium containing less than thirty-five parts of titanium and more than sixty-five parts of iron.

In order to ascertain the technical value of my invention I investigated a few samples of my arc light pencils. The samples were designated as follows:

B—2 pieces marked F. T.—80 per cent. ferrotitanium.

C—1 piece marked 41-L—30 per cent. rutile, 70 per cent. magnetite.

 $3^{-1}/_{2}$  piece National plain carbon.

The tests were conducted at the laboratory of the Lamp Testing Bureau, now "Electrical Testing Laboratory." Tests were first made in a handfed arc lamp on a direct current circuit from a storage battery of 60 cells, giving 120 volts, approximately. Owing to the peculiar nature of the arc it was found impossible to maintain an arc in this style of lamp much above 3/8 inch in length, and from 48 to 55 volts.

The results of the candle power measurements and life tests of the ferro-titanium (B) and rutile (C) pencils, as well as tests made on a plain open arc domestic carbon, are summarized in Tables I, II and III. These tests are principally valuable as illustrating the nature of the pencils and relative illumination and life, as compared to an ordinary carbon when operated under the same conditions. The results as tabulated are the averages obtained.

The average results given were mostly obtained with a positive carbon pencil above. The results with a positive copper pencil above were slightly lower in candle power, but not materially so, and were averaged with the other results obtained.

The positive copper pencil is evidently only slowly consumed in the arc when used in connection with the titanium pencils. There is a slight loss in weight probably due to oxidation and scaling, at the surface of the copper owing to its high temperature. The rutile pencil was further tested in a constant potential arc lamp operating on a current of 4.5 amperes approximately. In this lamp the rutile pencil (C) was placed below, ordinary carbon above, and the test made first with the carbon as a negative and then with it as a positive. As the rutile pencil could not be placed in the upper holder in the lamp for purposes of comparison, a further test was made in the hand-fed lamp, using rutile pencils both above and below, with the upper pencil positive. Two sets of measurements were made, one at 60 volts, approximately, and the other at 70 volts. The results are tabulated in Tables I, II and III:

TABLE I RESULTS OF TESTS IN	HAND-FED	LAMP AT 3.5	AMPERES.
	"B."		"F."
	Ferro	"C." Rutile	Plain
Mean spherical candle power	510.0	283.0	84 0
Watts per candle power	0.579	0.701	2.5
Amperes	3.51	3.51	3.5
Volts	48.3	55.8	49.1
Watts	169.4	195.4	171,4
Length of arc, inches (estimated)	0.375	0.350	0.125
Life of negative, hours per inches	3.85	11.1	2.8
Diameter of pencils, inches	0.57	0.52	0.50
Resistance per inch, ohms	0.00164	0.00284	0.0846

#### TABLE II .- LAMP AT 6.6 AMPERES.

	"B." Ferro- titanium.	"C" Rutile.	"F." Plain carbons.
Weight per inch, grams	9.64	14.33	4.83
Mean spherical candle power	968.0	737.0	243.0
Watts per candle power	0.341	0.436	1.35
Amperes	6.6	6.6	6.6
Volts	49.5	48.7	50.1
Watts	326.7	321.4	330.3
Length of arc, inches (estimated)	0.400	0.375	0.180
Life of negative, hours per inch	1.1	1.96	1.7

#### TABLE III .- LAMP AT 9.6 AMPERES.

	"B." Ferro- titanium.	"C." Rutile,	"F." Plain carbons.
Mean spherical candle power	1135.0	Fused	485.0
Watts per candle power	0.330	excessive-	0.986
Amperes	9.6	ly with	9.6
Volts	39.0	9.6 amp.	5.0
Watts	374.4		480.0
Length of arc, inches (estimated)	0.375		0.190

TABLE IV .- RESULTS OF TESTS WITH RUTILE PENCILS.

At 60 Volts.	Carbon upper—, rutile lower+.	Carbon upper+, rutile lower	Rutile upper+, rutile lower
Mean sperical candle power	. 544.0	448.0	624.0
Watts per candle power	. 0.521	0.61	0.41
Amperes	. 4.52	4.18	4.25
Volts	. 62.8	64.8	61.2
Watts	. 283.9	270.9	260.1
Length of arc, inches (estimated)	. 0.75	0.75	0.70

The light distribution in the tests of the ferrotitanium, rutile and carbon pencils at 3.5 amperes and from 50 to 55 volts are shown in Fig. I (left side). The light distribution of the test of the rutile pencil in a constant potential lamp at 4.2 amperes and 70 volts is shown in Fig. 1 (right side).

TABLE VAT 70	VOLTS.		
	Carbon upper —, rutile lower+.	Carbon upper+, rutile lower	Rutile upper +, rutile lower
Mean spherical candle power	469.0	590.0	1015.0
Watts per candle power	0.663	0.56	0.30
Amperes	4.35	4.35	4.30
Volts	71.5	72.0	70.0
Watts	311.0	314.0	301.0
Length of arc, inches (estimated)	0.875	0.875	0.75

All candle power measurements were made on the arc photometer designed by Prof. Matthews. The life of the results is computed from a burning test of from one and one-half hours to two hours, the pencils being first burned to shape and then weighed on a chemical balance before and after the test.

For the purpose of comparison, was plotted on Fig. 2 a curve "F" taken from the report of Prof. C. P. Matthews, included in the report of the Committee on the Photometric Measurements of Arc Lights to the National Electric Light Association, May 20, 1902. This curve is the result of many measurements and represents series enclosed lamps in common use in this country for street and interior illumination. The measurements were made on a constant current of 6.8 amperes with 70 volts at the arc, with an opalescent inner and a clear outer globe. In interior illumina-



tion an opal or opalescent outer is usually substituted for a clear outer. The constant potential enclosed arc lamp is also used for inside illumination, but its light distribution curve for the same energy consumption does not materially differ from that shown.

The series enclosed arc lamp gives 303 mean spherical candle power at 476 watts or 1.57 watts per mean spherical candle power. They burn approximately 100 hours for one trimming.

The light produced by ferro-titanium and rutile (oxide of titanium and magnetite) pencils is a yellowish white as judged by the eye. The spectrohand-fed lamp without globe. At from 50 to 55 volts the familiar sound of a "hissing" carbon is produced. As the voltage and the length of the arc is increased this sound is diminished and the only sound produced is the one of escaping steam, but this is not of an objectionable character.

Under both the above conditions of operation the point of contact of the arc with the upper and lower pencils is constantly shifting, which causes a flutter in the light emitted. The objectionable features of this are largely eliminated with a long arc, and when protected by a globe.

The life tests were made from 50 to 55 volts, a



scope shows the entire spectrum with special brilliancy in the bright yellow portion and the neighboring light green and orange. The size and length of the arc, which produces most of the light, softens the sharpness characteristic of the open carbon arc. The arc itself presents many peculiar characteristics. The normal arc with a positive copper or carbon pencil above and the ferro-titanium or rutile pencil below is approximately 1/2inch in length at from 50 to 55 volts, and increases to 3/4 or 7/8 inch at 70 volts. It is from 3/32to 1/8 inch in diameter and seems to emanate from the titanium pencil. At the higher voltage the arc is very unstable and is easily broken in a

much lower voltage than that at which the pencils are designed to operate. Therefore, while these tests are indicative of the results to be expected at 75–80 volts, the life at 75–60 volts cannot be predicted upon them. From these results, however, and from actual tests made with the necessary special apparatus by the writer and certified to by others engaged in the tests, it seems safe to estimate the life of a rutile pencil 1/2 inch in diameter to be from 10 to 12 hours per inch—perhaps higher, later tests showed 25 hours to one inch.

There should be no difficulty, therefore, in proportionating the size and length of the pencil to attain a life of 200 hours, the normal life of the enclosed carbon lamp with one trimming. As the arc under proper conditions can be maintained at from 3/4 to I inch in length by increasing the diameter of the titanium pencil, the burning hours for one trimming could be very materially increased. This has actually been done and a very much higher life obtained.<sup>1</sup>

As already mentioned, when the titanium pencil is made the negative and placed below, at 70 volts, the arc is from 3/4 to 7/8 inch long and about 1/8 inch in diameter, and of an unusually white color, slightly more light being produced at or near the surface of the electrodes, owing to the concentration of the arc and the incandescence of the surfaces. The arc itself is clean cut and well defined, and is a true arc and not a flaming arc, as is generally understood by that term.

The same results were obtained with either a copper or carbon positive. As already mentioned, the arc fluttered and shifted constantly. The apparent trend of the arc and the gases and material carried by it was, under normal operation, from the negative to the upper positive pencils. Under the same current reversed, that is, the lower titanium pencil made the positive, an arc of entirely different character was obtained. The diameter of the arc was increased from 1/8 to 1/4 inch or 5/16 inch. At the same voltage the length was also slightly increased, approximately to an inch. The "flutter" as mentioned was absent, although the arc lazily shifted from one point to another. The direction of the arc still seemed to be from the lower positive to the carbon or copper negative above, but possessed very little illuminating power, and had a decidedly reddish tinge. Frequently, for short intervals, the arc became suffused with the white light characteristic of titanium. On account of its steadiness, lack of "fluttering" and very large diameter and length, the arc presents many attractive features.

By placing the titanium pencils on both sides of the arc and making the upper electrode positive, a third arc of a distinctly different character was obtained; though differing from either, it seemed to be practically a combination of the two above mentioned. The arc resembled an inverted cone in shape with an apex on the lower rutile negative pencil, and the base on the upper pencil. Through the center ran a core fairly representing the arc obtained in the first case above mentioned. Around it was a zone representing the second arc described but increased in diameter to possibly 5/16 or 3/8 inch and suffused with the white titanium incandescent gases. The arc thus obtained was absolutely steady with no fluttering or shifting. Both upper and lower pencils were fused at their surfaces, but contrary to expectations, the molten material upon the upper pencil did not drop. The light is softened somewhat by the presence of what might be called the negative arc, but possesses all the color characteristics of the titanium arc. The measurements made of these three arcs have been given in Tables IV and V, from which it is seen that the candle power efficiency is nearly doubled at 70 volts by placing the titanium pencil above as well as below.

An efficiency of 0.3 watt per mean spherical candle power was obtained in this test, and it seems entirely probable that this can be improved.

In Fig. 2 I have plotted the watts for mean spherical candle power obtained at 50 volts approximately, with 1/2 inch pencils, and at various current values. It is apparent from the curves there shown that there is very little increase in efficiency resulting from an increase of current above 6 amperes, under the conditions observed in the test, *viz.*, with 1/2 inch pencils and about 50 volts at the arc.

In Fig. 3 there were plotted the results obtained at 60 and 70 volts with a rutile lower negative, and a positive electrode of carbon and rutile, respectively. Unfortunately, the number of observations was not sufficient to construct a reliable



curve, but the cut indicates graphically the great increase in efficiency due to using a titanium positive as well as negative, and from the direction of the curves it indicates that an increased efficiency would be obtained at a higher voltage, especially in the case where the titanium is used for both positive and negative electrodes.

A very interesting phase of the investigation

of the titanium pencils was opened up by the chemical analysis to which the raw material and the ready-made pencils were subjected. The general opinion is, that titanium oxides can be reduced to the metallic state only at the high temperature of the electric furnace. However, the behavior of the reduced magnetite-rutile pencils was such as to suggest that at least a part of the titanic oxide was actually reduced to the metallic state.

The author thought that the oxygen compounds of iron may facilitate the reduction of the titanic oxide homogeneously intermixed with them to metallic titanium at a temperature of about  $1200^{\circ}$  C., in the presence of a reducing agent, like carbon, hydrogen, etc. For the purpose of ascertaining whether or not this hypothesis was correct, the analyses of the following bodies were carefully executed:

A. Ilmenite ore. B. Arc light pencil prepared as described above. C. Magnetite rutile pencil made of rutile and magnetic oxide of iron prepared as above described. D. Another sample of ilmenite in the shape of pellets. E. Pencils pre-



Fig. 4.-Comparative photometric values of the improved titanium electrode.

The analyses of these materials were as follows:

#### ANALYSIS A .--- ILMENITE.

		Per cent.
Silica	(SiO <sub>2</sub> )	3.40
Titanic oxide	(TiO <sub>2</sub> )	41.70
Ferrous oxide	(FeO)	44.24
Alumina	(Al <sub>2</sub> O <sub>3</sub> )	3.64
Oxide of manganese	(MnO)	0.74
Oxide of chromium	(Cr <sub>2</sub> O <sub>3</sub> )	0.16
Lime	(CaO)	1.20
Magnesia	(MgO)	3.96
Oxide of copper	(CuO)	0.40
Phosphorus anhydride	$(P_2O_5)$	0.22
Sulphuric anhydride	(SO <sub>3</sub> )	0.20
Alkalies	$(Na_2O.K_2O)$	1.07
		100 93

ANALYSIS B .- ORE PENCIL FROM ILMENITE.

a second a second second second second		Per cent.
Silica	(SiO <sub>2</sub> )	3.87
Alumina	(Al <sub>2</sub> O <sub>3</sub> )	2.35
Manganese	(Mn)	0.50
Oxide of chromium	(Cr <sub>2</sub> O <sub>3</sub> )	0.24
Lime	(CaO)	1.32
Magnesia	(MgO)	3.93
Copper	(Cu)	0.32
Phosphorus	(P)	0.10
Sulphur	(S)	0.05
Alkalies	(Na <sub>2</sub> O.K <sub>2</sub> O)	1.12
Carbon	(C)	0.50
Metallic iron	(Fe)	46.28
Metallic titanium	(Ti)	3.54
Oxides of titanium	$(TiO_2.Ti_2O_3)$	35.88 by dif
		100.00
Total titanium calculated to	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	
higher oxide, TiO <sub>2</sub>	all a state of a	46.52

ANALYSIS C.—RUTILE PENCIL MADE FROM MIXTURE OF THE MINERAL RUTILE WITH MAGNETIC OXIDE OF IRON.

		Per cent.
Silica	(SiO <sub>2</sub> )	3.25
Alumina	(Al <sub>2</sub> O <sub>3</sub> )	1.77
Manganese	(Mn)	0.40
Oxide of chromium	(Cr <sub>2</sub> O <sub>3</sub> )	0.12
Lime	(CaO)	1.25
Magnesia	(MgO)	1.04
Copper	(Cu)	0.08
Phosphorus	(P)	0.10
Sulphur	(S)	0.05
Alkalies	(Na <sub>2</sub> O.K <sub>2</sub> O)	1.80
Carbon	(C)	0.35
Metallic iron	(Fe)	48.93
Metallic titanium	(Ti)	3.24
Oxide of titanium	(TiO <sub>2</sub> .Ti <sub>2</sub> O <sub>3</sub> )	37.62 by diff.
		100.00
Total titanium calculated to TiO2		48.45%

BEEN ACTED UPON BY	THE ELECTRIC	ARC.
		Per cent.
Silica	(SiO <sub>2</sub> )	2,60
Alumina	(Al <sub>2</sub> O <sub>3</sub> )	2.45
Manganous oxide	(MnO)	0.50
Oxide of chromium	(C.O.)	0.20
Lime	(CaO)	1,60
Magnesia	(MgO)	4.17
Copper oxide	(CuO)	0.30
Phosphoric anhydride	(P <sub>2</sub> O <sub>5</sub> )	0.03
Ferrous oxide	(FeO)	50.08
Titanium oxides, plus alkalies	by difference	38.07 by diff
		100.00
Total titanium calculated to		
higher oxide, TiO2		40.90%
ANALYSIS E ILMENIT	E PELLETS.	
		Per cent.
Silica	$(SiO_2)$	3.50
Alumina	(Al <sub>2</sub> O <sub>3</sub> )	2.55
Ferrous oxide	(FeO)	44.68
Titanic oxide	(TiO <sub>2</sub> )	37.90
Lime	(CaO)	1.02
Magnesia	(MgO)	3.46
Alkalies	(Na <sub>2</sub> O.K <sub>2</sub> O)	0.60
Chromic oxide	(Cr <sub>2</sub> O <sub>3</sub> )	0.35
Oxide of copper	(CuO)	0.05
Suphuric anhydride	(SO <sub>3</sub> )	0.41
Manganous oxide	(MnO)	0.25
Phosphoric anhydride	$(P_2O_a)$	0.035
Loss in ignition		4.93
		100.00
A State of the second second second	in the second	
ANALYSIS F,ILMENITE PE	INCIL FROM PI	LLETS.
		Per cent.
Silica	$(SiO_2)$	4.05
Alumina	$(AI_2O_3)$	2.40
Manganese	(Mn)	0.40
Oxide of chromium	$(Cr_2O_3)$	0.35
Lime	(CaO)	0.91
Magnesia	(MgO)	3.09 .
Copper	(Cu)	0.12
Phosphorus	(P)	0.03
Sulphur	(S)	0.06
Alkalies	$(Na_2O.K_2)$	1.30
Carbon	(C)	1.33
Metallic iron	(Fe)	45.00
Metallic titanium	(Ti)	5.22
Oxides of titanium	(TiO <sub>2</sub> .Ti <sub>2</sub> O <sub>8</sub> )	35.10
		99.36

otal titanium calculated	to	
TiO2		43.80%

To

ANALYSIS G .- REDUCED ILMENITE PELLBTS.

		Per cent.
Silica	(SiO <sub>2</sub> )	4.88
Alumina	(Al <sub>2</sub> O <sub>3</sub> )	3.40
Manganese	(Mn)	0.59
Oxide of chromium	(Cr <sub>2</sub> O <sub>3</sub> )	0.35
Lime	(CaO)	0.82
Magnesia	(MgO)	4.18
Copper	(Cu)	0.28
Phosphours	(P)	0.10
Sulphur	(S)	0.30
Alkalies	(Na <sub>2</sub> .K <sub>2</sub> O)	1.19
Metallic iron	(Fe)	45.40
Carbon	(C)	0.43
Metallic titanium	(Ti)	3.00
Oxides of titanium	(TiO2.Ti2O3)	35.08 by diff.
		100.00
Total titanium calculated to		
higher oxide	(TiO <sub>2</sub> )	40.40%

NALYSIS HILM	IENITE PENCILS	FROM REDUCED	PELLETS.
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		Per cer	ıt.
Silica	(SiO <sub>2</sub> )	3.95	
Alumina	(Al <sub>2</sub> O <sub>3</sub> )	1.80	
Manganese	(Mn)	0.14	
Oxide of chromium	(Cr <sub>2</sub> O <sub>3</sub> )	0.22	
Lime	(CaO)	0.65	
Magnesia	(MgO)	3.11	
Copper	(Cu)	0.16	
Phosphorus	(P)	0.02	
Sulphur	(S)	0.10	
Alkalies	(Na <sub>2</sub> O.K <sub>2</sub> O)	1.28	
Carbon	(C)	0.82	
Metallic iron	(Fe)	50.60	
Metallic titanium	(Ti)	5.64	
Oxides of titanium	(TiO <sub>2</sub> .Ti <sub>2</sub> O <sub>3</sub> )	31.51	by diff.
		100.00	
Total titanium calculated to			
higher oxide	(TiO <sub>2</sub> )	43.05	

The mineral ilmenite is a ferrous titanate present in the sample under examination to the amount of 85.94 per cent., the remaining 14.05 per cent. represents the total percentage of the other minerals present in the chemical combinations shown in the report.

The ilmenite pencil consists of 46.28 per cent. of metallic iron, 3.54 per cent. metallic titanium and 35.88 per cent. of the lower and higher oxides of titanium, respectively  $Ti_2O_3$  and  $TiO_2$ . The sum of the percentages of metallic iron, metallic titanium, and the oxides of titanium comprises 85.70 per cent. of the compounds reported in the analysis. The method of arriving at these figures is as follows:

The metallic iron was determined by treating the powdered pencil with a 20 per cent. solution of sulphuric acid with exclusion of air, namely, in a flask through which a current of carbonic acid gas was continually flowing. The ferrous sulphate solution thus obtained was titrated for iron with a solution of permanganate of potash by the wellknown method. The presence of metallic iron was corroborated by the occurrence of small particles of metal which were found while powdering the pencil, these particles flattening out under the pestle and resisting reduction to the powdered state. These particles were also magnetic.

Another proof of the presence of a metal was the evolution of gas when the powdered pencil was treated with hydrochloric acid. Further evidence of the presence of metallic iron in the ilmenite pencil was the precipitation of metallic copper on treating the powdered pencil with a solution of copper sulphate.

The metallic titanium was found by treating the powdered pencil with strong hydrochloric acid, filtering the liquid and determining the titanium

in the clear solution. That this was metallic titanium that was dissolved, and not the oxide, is borne out by the investigations of Henry Moissan, who, in his "Treatise on the Chemistry of Minerals," Paris, 1905, states that ignited oxides of titanium Ti<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> are not soluble in hydrochloric or nitric acids. The same facts are reported in published results of the investigations of Ebelman (see Roscoe and Schorlemmer's "Chemistry," Volume II), where it is stated that the ignited oxides of titanium Ti2O3 and TiO2 are not soluble in hydrochloric acid. Again, in Watts' "Dictionary of Chemistry," 1878, Volume III, page 419, the same authority, 1894, Volume IV, page 774, the fact is stated that ignited titanium oxide Ti<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> are insoluble in hydrochloric acid.

Fresenius, in his "Treatise on Qualitative Analysis," states that ignited  $\text{TiO}_2$  is insoluble in hydrochloric acid.

Prescott and Johnson, in their "Qualitative Analysis," state that metallic titanium is soluble in hydrochloric acid.

As shown by the results of the analysis, there was formed in the pencil made from ilmenite 3.54 per cent. of metallic titanium, this percentage being obtained from the hydrochloric acid solution of the metal. By adding together the percentages of the metallic iron, metallic titanium and the foreign minerals found in the ilmenite pencil, we obtain 74.12 per cent.; the difference between this figure and 100 per cent. is 35.88 per cent, which shows the percentage of the mixed oxides of titanium  $Ti_2O_3$  and  $TiO_2$  present. The percentage of the respective oxides when mixed cannot be determined as there is no method known by which it can be done.

The total titanium found in the ilmenite pencil if calculated as TiO, gives 46.53 per cent.; the metallic iron plus the other compounds in the pencil gives 60.58 per cent.; the total of these two percentages is 107.10 per cent. The excess over 100 per cent. shows that the titanium cannot all be present as the higher oxide TiO2, but must occur in the pencil as part metal and part oxide. If titanium found in the ilmenite pencil were all calculated as the lower oxide Ti2O3, this oxide would be present in the amount of 41.87 per cent., which added to 60.58 per cent., the sum of the percentages of iron and the other compounds, would give 102.45 per cent., which still shows an excess of oxygen, indicating that the titanium is present in the ilmenite partly as metal and in part as oxide.

Corroborating this theoretical deduction is the fact that 3.54 per cent. of metallic titanium was found by direct determination.



In the rutile pencil by the same method of calculation, I find that the sum of the percentages of metallic iron and compounds, other than titanium, is 59.14 per cent.; in adding to this the titanium found calculated as higher oxide TiO2, namely, 48.45 per cent., the sum 107.59 per cent. is obtained, indicating again an excess of oxygen. If the titanium is all calculated as lower oxide Ti<sub>2</sub>O<sub>2</sub>, we get 43.60 per cent. of Ti<sub>2</sub>O<sub>3</sub>, which, added to 59.14 per cent., gives 102.74 per cent., a result which still shows an excess of oxygen. As stated above, this indicated excess of oxygen shows that the titanium cannot all be present in the form of oxides. Corroborating this theoretical conclusion is the fact that 3.24 per cent. metallic titanium was found by direct determination.

The metallic iron present in the pencil was determined directly by solution in dilute sulphuric acid with exclusion of air subsequent titration with permanganate of potash, the result showing 45.00 per cent. of metallic iron.

The metallic titanium was determined directly by solution in hydrochloric acid. The result showed 5.22 per cent.

The sum of the metallic iron percentages, namely, 45.00, and the gangue constituents is 59.04 per cent.; if to this is added the titanium found, all calculated as higher oxide TiO<sub>2</sub>, we get 102.84 per cent., the excess of oxygen indicating again

the reduction of the titanium which is corroborated by the metal found on direct determination.

<sup>1</sup> The metallic iron and metallic titanium were each determined as previously described, the iron showing 45.40 per cent. and the metallic titanium 5.00 per cent.

The sum of the percentages of metallic iron and gangue matter is 60.92 per cent.; upon adding to this figure the percentage of titanium found, all calculated as higher oxide  $TiO_2$  we get 102.32. The excess of oxygen again indicates the presence of metallic titanium, which is corroborated by the metallic titanium found on direct test, namely, 3.00 per cent.

The pencil made from the reduced ilmenite pellets shows metallic iron 50.60 per cent. and metallic titanium 5.64 per cent. The sum of the metallic iron and gangue matter percentages is 62.85; when to this is added the titanium found calculated to higher oxide TiO<sub>2</sub>, the figure 105.90 is obtained. The excess of oxygen again indicated the presence of metallic titanium, which is corroborated by the metallic titanium found on direct determination. The conclusion was forced upon the author:

*First.*—That there was actually a reduction of titanic acid into metallic titanium along with the reduction of the oxygen compounds of iron taking place under the conditions of the manufacture of arc light pencils as above described.



Second.—That the process of reduction to which the raw material, out of which the arc light pencils were manufactured, led to a storing up of energy in the arc light pencils. The oxides reduced by the above-described process to the metallic state were then again oxidized in the arc to their previous condition. These processes of oxidation supplied the pencils with the heat necessary for the ionization that caused the selective radiation and luminosity of the arc.

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This storing up of energy is the principal distinctive feature differentiating the metallic rutile pencil from the mere conglomeration of the oxygen compounds of iron with oxygen compounds of titanium known under the name of magnetite arc light pencils now on the market. Consequently the author considers himself justified in claiming that his arc light pencil prepared of a mixture of oxygen compounds of iron and titanium, subjected to a process of reduction, are actually ferrotitanium pencils, inasmuch as they contain the metallic titanium and metallic iron, conglomerated and intermingled with each other in one solid cylindrical body.

In order to verify the above formulated conclusion, another electrode, made as above described, was analyzed with the following results:

	Per cent.
Metallic iron	64.89
Oxides of titanium	26.69 (by diff.)
Silica	1.99
Metallic titanium	1.82
Alumina	1.08
Magnesia	1.08
Lime	0.80
Alkalies	0.75
Manganese	0.50
Carbon	0.32
Sulphur	0.08
Phosphorus	Trace
Chromium	None
Metallic titanium soluble in hydro-	
chloric acid	1.86

This pencil contains 3.60 per cent. of a 50 per cent. ferro-titanium, assuming that the alloy of iron and titanium exists here in the proportion of one part of metallic titanium to one part of metallic iron.

The reason for a lower percentage of metallic titanium in this pencil than that found in the pencil reported a year ago, is that in the former pencils there was present a total of about 45 per cent. of titanium calculated as titanic acid, whereas in this pencil there is present a total of only 29 per cent. of titanium calculated as titanic acid. The presence of iron in metallic state is demonstrated beyond doubt and the fact that titanium was obtained in solution by treatment with hydrochloric acid shows, according to recognized authorities

in chemistry, that metallic titanium is also present.

Report of Life Tests of Improved Titanium Electrodes. Duration of Each Test 100 Hours.

Pencil $E_3 - 5/8''$ diam. $\times 8''$ long:	
1	57.14
2	43.23
3	43.96
Average hours per inch	48.11
Pencil D <sub>3</sub> $-9/16''$ diam. $\times 8''$ long.	
1	53.33
2	44.44
3	44.83
Average hours per inch	47.53
Pencil $E_3$ —9/32" diam. $\times$ 8" long.	
1	50.00
2	30.76
3	37.18
Average hours per inch	39.31
Pencil E <sub>3</sub> — $5/8''$ diam. $\times 8''$ long.	
4 1	57.14
2	43.23
3	43.96
Average hours per inch	48 11

Comparative data as to the life of the improved titanium arc light electrodes and carbon electrodes (at their respective normal current density per square mm.):

C	m. per one hour.
Life of the D titanium pencil 9/16 in d	0.075
Life of the averaged cored carbon	1.300
Life of the solid carbon	1.450
Life of the enclosed carbon	0,152
Life of the flaming carbon	3.00
Life of the Bremer type carbon	4.00
Life of the Blondel type carbon	2.00

The increased life of the titanium pencil in hours per inch in comparison with the following carbons, is:

Cored carbons	17.35	times
Solid carbons	19.33	
Enclosed carbons	2.03	44
Flaming carbons	40.00	
Bremer type carbons	53.33	
Blondel type carbons	26.67	••

Comparative specific resistivity of the titanium arc light electrode and various carbon electrodes (1 m. long, 1 mm.<sup>2</sup> in diameter):

	Ohms.
Solid carbons. Brand Conradty C	69.0
Solid carbons. Brand Henrion (Nancy)	69.0
Solid carbons. Brand Plania	79.0
Average	72.3
Cored carbons. Brand Siemence A	72.0
Cored carbons. Brand Plania	81.0
Average	76.5
Enclosed lamp carbons. Brand Plania, solid	81.0
Enclosed lamp carbons. Brand Eos, solid	89.0
Average	85.0

	Onms,
Bremer type carbons. Brand Plania, white	84.0
Bremer type carbons. Brand Plania, with	
nickel-plating	58.0
Bremer type carbons. Brand Plania Rosa	88.0
Bremer type carbons. Brand, Plania, yellow	87.0
Bremer type carbons. Brand Siemence, white	93.0
Bremer type carbons. Brand Siemence, yellow.	106.0
Bremer type carbons. Brand Siemence, yellow,	
with copper-plating	29.3
Bremer type carbons. Brand Siemence Con-	
radty Noris, yellow, with copper-plating	60.5
Same, with wire in the core	81.0
Siemence, yellow, with metallic wire in the core	39.0
Average	72.58

The resistivity of the titanium pencil is 2.02 ohms. Consequently:

The resistivity of the average solid carbon pencil is 36 times.

The resistivity of the average cored carbon pencil is 38.25 times.

The resistivity of the average enclosed carbon pencil is 42.5 times.

The resistivity of the average Bremer type carbon is 36.2 times that of the titanium pencil.

## A RAPID METHOD OF DETERMINING THE PERCENTAGE OF CASEIN IN MILK.

BY T. BRAILSFORD ROBERTSON. Received May 10, 1909.

In a recent paper<sup>1</sup> I have shown that the difference between the refractive indices of two solutions of a caseinate, which differ only in their casein-content, is proportional to the difference between the percentages of casein which they contain; in other words, that

## $n-n_1=a \times c$ ,

where n is the observed refractive index of the solution, c is the percentage of casein which it contains, n, is a constant, the value of which depends upon the concentration and nature of the alkaline (or acid) solution employed as solvent, and a is a constant numerically equal to the change in the refractive index of the solvent which is brought about by the addition to 100 cc. of 1 gram of casein. I have also shown that by means of the above formula the concentration of casein in a solution can be very accurately determined, the deviations from accuracy rarely exceeding 2 per cent. of the quantity of casein contained in 100 cc. of the solution (provided that quantity exceeds 0.5 gram) and that the change in the refractive index of a given volume of a solution of a base which is brought 1 T. Brailsford Robertson: Journ. of Physical Chem., 13, 469

(1909).

about by the introduction of a given weight of casein is independent of the concentration of the base and of the nature of the base; if the volume be 100 cc. and the weight of casein 1 gram, the change in the refractive index is 0.00152.

It occurred to me that these facts might be applied to the determination of the percentage of casein contained in fluids such as milk.1 The procedure of the determination was as follows:

Fifty cc. of fresh, unskimmed milk were diluted to 250 cc. and 75 cc. of N/10 acetic acid (made up by diluting 10 cc. of glacial acetic acid to 1750 cc.) were slowly added, the mixture being continuously and rapidly stirred during the addition. The precipitate was then allowed to settle and the supernatant fluid was poured through a 15 cm. S. & S. No. 589 "white band" paper. The precipitate was then washed by decantation with distilled water several times, the washings, and, subsequently, the precipitate being transferred to the same filter. The filter and precipitate were then allowed to drain for about 1 hour and were then transferred to a dry beaker and 100 cc. (accurately measured) of N/10 NaOH were added and the filter and precipitate were macerated (by the aid of a stirring rod protected at the tip by rubber) until the filter paper was transformed into a fine pulp and the casein was completely dissolved. As the casein particles are of a different color to the particles of paper, the point of complete solution can be readily determined; complete solution of the amounts dealt with in this determination is usually attained within ten minutes. The mixture was then filtered<sup>2</sup> and the refractive index of the filtrate determined, by means of a Pulfrich refractometer reading accurately to within 1' of the angle of total reflection, if possible at 20° C. Since *n* for N/10 NaOH is 1.33444 (at 20° C.),<sup>3</sup> the results are calculated as follows:

Grams case in 50 cc. milk =  $\frac{n - 1.33444}{3.3444}$ 

0.00152

where n is the refractive index of the final solution, obtained as described above.

An obvious source of error is the water associated with the precipitate and filter paper when they are transferred to the N/10 NaOH. As the succeeding determinations show, however, with the

<sup>3</sup> Cf. the paper cited above.

<sup>[</sup>FROM THE RUDOLPH SPRECKELS PHYSIOLOGICAL LABORATORY OF THE UNIVERSITY OF CALIFORNIA.]

<sup>&</sup>lt;sup>1</sup> Since writing the above, I have found that Reiss (Arch. f. Exper. Pathol. und Pharm., 51, 18 (1903); Beitr. z. chem. Physiol. und Pathol., 4, 150 (1904)) has previously employed the refractive indices of bodyfluids as a measure of their protein-content.

<sup>&</sup>lt;sup>2</sup> This filtration can be omitted provided the solution be allowed to stand for a sufficient time so that the particles of paper settle to the bottom of the beaker.

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amounts of casein likely to be obtained from 50 cc. of milk, and provided the precipitate is allowed to drain for a sufficient time this error is, for all practical purposes, negligible. It could, of course, be eliminated, if desired by diluting the final solution, before filtration, to an accurately measured volume with N/10 NaOH; in that case it would be advisable to start with 100 cc. of milk instead of 50 cc., completely dissolve the precipitate in 100 cc. of N/10 NaOH and then make up the volume of the mixture to 200 cc. by the addition of N/10 NaOH. The slight dilution of the sodium hydroxide caused by this procedure does not affect the accuracy of the determination, since the refractive index of a dilute sodium hydrate solution varies very much less with its concentration than that of a solution of casein of the concentrations emploved above.

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For comparison, determinations were carried out by the official method<sup>1</sup> as follows:

Twenty cc. of fresh unskimmed milk were diluted to 100 cc. and 30 cc. of N/10 acetic acid were slowly added, the mixture being continuously and rapidly stirred during the addition. The precipitate was then allowed to settle and the supernatant fluid was poured through a 15 cm. S. & S. No. 589 "white band" paper. The precipitate was then washed by decantation with distilled water several times, the washings, and, subsequently, the precipitate being transferred to the same paper. The filter and precipitate were then digested with 20 cc. of H<sub>2</sub>SO<sub>4</sub>, to which a trace of metallic mercury had been added, as in the ordinary Kjeldahl method for the determination of nitrogen, and the process of determining the nitrogen was completed by the official Kjeldahl method. The number of grams of casein in the 20 cc. of milk was estimated by multiplying the number of grams of nitrogen thus determined by 6.25.

Four determinations by the new method yielded the following results:

	Grams casein in 100 cc. milk.
a	2.84
b	2.84
c	2.84
d	2.84
Average	2.84

Four determinations by the official method,

 $^1$  U. S. Department of Agriculture, Division of Chemistry, Bulletin 46, Revised Edition (1899), p. 55. The details of the precipitation were slightly modified and the quantity of milk employed in the determination was double that recommended by the Association of Official Agricultural Chemists. using the same milk, yielded the following results:

· · · · · · · · · ·	Grams casein in 100 cc. milk.
	2.69
	2.81
	2.78
	2.69
Average	2.74

The agreement is sufficiently satisfactory. The factor 6.25 by which the nitrogen is multiplied to obtain the equivalent in casein is calculated on the assumption that the percentage of nitrogen in casein is 16. If, however, we take 15.65 as the true percentage of nitrogen in casein, which, according to the results of Hammersten,<sup>1</sup> Lehmann and Hempel,<sup>2</sup> and Ellenberger,<sup>3</sup> would appear to be the more accurate figure, then the factor by which nitrogen is multiplied becomes 6.4 and the agreement between the official and the new methods is even more satisfactory, the above four determinations by the official method yielding the results:

C	Frams casein i
	100 cc. milk.
a	. 2.75
b	. 2.88
c	. 2.85
d	. 2.75
	ter and the second
Average	. 2.81

In order to further test the accuracy of the method, weighed amounts of casein were dissolved each in 100 cc. of N/50 NaOH and were precipitated, with constant stirring, by the addition of 30 cc. of N/10 acetic acid. The casein employed in these experiments was the c. p. product manufactured by Eimer & Amend and further purified by trituration with large volumes of distilled water alcohol (absolute) and ether (ueber natrium dist.); it was dried for 24 hours at 36°. The properties of the product thus obtained have been fully described by me in a previous paper;4 it gives every indication of being a pure product, being insoluble in distilled water (save in traces which adhere to the undissolved particles) and completely precipitated by acetic acid. It neutralizes to phenolphthalein exactly the quantity of base determined by Laqueur and Sackur and by Van Slyke and Hart;5 it is free from appreciable water. The first

<sup>1</sup> O. Hammersten, Zeitschr. f. Physiol. Chem., 7, 227 (1883); 9, 273 (1885).

<sup>2</sup> W. Hempel, Arch. f. d. ges. Physiol., 56, 558 (1894).

<sup>3</sup> Ellenberger, Arch. f. Anat. und Physiol., Physiol. Abt. Suppl., p. 313 (1902).

4 T. Brailsford Robertson, Journ. of Biol. Chem., 2, 317 (1907).

<sup>5</sup> Laqueur and Sackur, Beitr. z. Chem. Physiol. u. Path., 3, 193 (1902). Van Slyke and Hart, Amer. Chem. Journ., 33, 461 (1905). two determinations were carried out in exactly the same manner as those of the casein in milk, described above. The last three, in which larger quantities of casein were determined, were determined in the same manner save that the final solution, before filtration, was diluted to 250 cc. (accurately measured) so as to eliminate the error due to water associated with the precipitate and paper. The results follow:

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	A second s
Weighed.	Determined.
1.0	1.02
2.0	1.88
3.0	` 2.90
4.0	3.88
5.0	5.03

The agreement is as close as could be desired.

If the refractive index of the final solution be determined at temperatures above  $20^{\circ}$  and below  $30^{\circ}$ , 0.0001 must be subtracted from the value of the constant  $n_1$  (= 1.33444 at  $20^{\circ}$  when the solvent is N/10 NaOH) for every degree by which the temperature exceeds  $20^{\circ}$ . If it be determined at temperatures below  $20^{\circ}$  and above  $10^{\circ}$  0.00007 must be added to the value of the constant  $n_1$  for every degree by which the temperature is less than  $20^{\circ}$ .<sup>1</sup>

The advantages of the method herein described over the official method for the determination of casein are manifest. Not only is the time consumed in the determination much reduced, the time actually occupied in manipulation, exclusive of that allowed for drainage of the precipitate, being not much over 1/2 hour, but the whole procedure is much simplified and the accuracy of the determination is unimpaired.

For further details concerning the dependence of the refractive index of casein solutions upon their concentration, the nature of the solvent, the temperature, etc., I must refer the reader to my previous paper, cited above.

[CONTRIBUTION FROM THE DEPARTMENT OF AGRICULTURAL CHEMISTRY, UNIVERSITY OF MISSOURI.]

## CHANGES IN THE COMPOSITION OF THE SKELETON OF BEEF ANIMALS.

#### (FIRST PAPER.)

By P. F. TROWBRIDGE AND F. W. WOODMAN. Received July 1, 1909.

The study of the composition of the skeletons of beef animals reported in this paper constitutes a portion of the general study of the "Uses to which

<sup>1</sup> Cf. my previous paper, referred to above.

the Animal Puts Its Food," now in progress in the Experiment Station of the University of Missouri. In the work during the past two years, fifteen animals have been slaughtered and analyzed. Of these the six mentioned in this paper were used chiefly to study the resorption of fat which takes place when an animal in good condition of nourishment is unable to obtain a usual food supply. Only the skeleton is considered in this paper.

The housekeeper regards the skeleton as merely so much waste material. To the animal husbandman it is of very great importance, determining, to a large extent, the size and value of the animal. To him an exact knowledge of the conditions of growth of the skeleton and to what extent it is affected by adverse conditions is very important.

Lawes and Gilbert, in their classic researches at Rothamsted, made a special study of the ash content of the carcasses of beeves and other animals. They pointed out especially the variation of the proportion of mineral matter in the bones to that in all other parts of the body, dependent upon the degree of fatness of the animal.

Wildt,<sup>1</sup> working with rabbits, made a special study of the effect of age on the chemical composition of the skeleton of young and of full-grown animals. He concluded that age had little effect after the animal had reached full growth, the composition of the skeleton varying with the condition of the animal. Mann<sup>2</sup> drew the same conclusions, though from rather indefinite data.

Düring<sup>3</sup> gives tables showing the difference in composition of the bones of various parts of birds. He showed that the composition of the air-dry bone varies in different parts of the skeleton.

Brookman<sup>4</sup> made comparative analyses of the skeletons of cow, sheep, horse and swine.

Gabriel<sup>5</sup> studied especially the methods of analysis of bones. His investigation of the composition of the bone ash was very thorough and he gives a probable composition of bone ash.

The phosphorus in the marrow is largely in organic form, according to Otolsky.<sup>6</sup> He found 0.13 per cent. to 0.15 per cent. lecithin in the marrow of the leg bones of the horse. W. Gliken<sup>7</sup> obtained similar results with both human and animal skele-

<sup>&</sup>lt;sup>1</sup> Landw. Versuchsst., 15, 404 (1872).

<sup>&</sup>lt;sup>2</sup> Chem. News, **51**, 132 (1887). <sup>3</sup> Zeit. physiol. Chem., **7**, 446 (1883).

<sup>&</sup>lt;sup>4</sup> Ber., 388 (1882).

<sup>&</sup>lt;sup>5</sup> Zeit. physiol. Chem., **19**, 257 (1894).

<sup>&</sup>lt;sup>6</sup> Abs. in Jahresb. u. Tier. Chem., **37** (1908).

<sup>7</sup> Biochem Ztschr., 4, 235 (1908).

tons. He also found that the per cent. of lecithin in the marrow decreases with age.

The effect of nourishment upon the skeleton has been studied extensively, the investigations following three general lines: (1) one effect of feeding rations normal in other respects, but poor in one or more necessary mineral constituents; (2) the comparative effect of organic and inorganic compounds as sources of phosphorus; (3) the effect of starvation upon the composition and weight of the skeleton. The results of the various investigators<sup>1</sup> seems to show: Foods poor in calcium have little effect upon the skeleton. Foods low in phosphorus have a pronounced effect, producing weakness, brittleness and actual loss in weight of the skeleton.

Whether the form of combination of the phosphorus has any effect is still disputed.

Inanition decreases the fat and increases the moisture in the skeleton but does not appear to effect the mineral composition to any noticeable extent.

## EXPERIMENTAL PART.

## Objects of this Investigation.

In the investigations noted above the variations in the kind and conditions of the animals used, the object and methods of the work, and the varying manner of reporting make it hard to compare the results. In the course of the series of experiments upon the effect of breed, feed and age upon the composition of animal flesh, and the distribution of the food materials in the various parts of the animal body, which investigations are being carried on at the Missouri Agricultural Experiment Station, it became desirable to know the composition of the skeleton of the animals under consideration. With this object in view, the following work was undertaken, having special reference to the distribution of phosphorus in the different parts of the skeleton.

## Condition and Age of Steers at Time of Slaughtering.

The six steers reported in this paper were all from the group known as "special maintenance steers." They were from the same sire and herd of cattle, grade Herefords; of about the same age, spring calves of 1907, and had run on grass until purchased for the experiment in fall of 1907. At the outset all were put upon full feed, consisting of 2.5 parts grain (composed of 8 parts corn to 1 linseed meal) to one part alfalfa hay, until in nearly prime condition.

In February, 1908, steer 594 was killed as a check animal. He was a fat yearling, showing Shorthorn blood, and was in a nearly finished condition. His carcass graded as "baby beef."

Steers 591, 593 and 597 were killed in September, 1908, after having been kept on "special maintenance" for six months.

Steer 591 was eighteen months old when killed, and had been kept on "submaintenance" for six months, being made to lose one-half pound daily. He was very thin, his carcass being graded as a "canner."

Steer 597 was eighteen months old when slaughtered. He had been kept on "maintenance," being kept at the same weight, neither gaining or losing. His carcass graded as No. 3 beef, being too thin for No. 2.

Steer 593 had been kept on "supermaintenance" for six months, being made to gain one-half pound per day. He was eighteen months old when slaugh-tered and graded as No. 2 beef.

Steer 592 was killed in January, 1909, being about 22 months old when killed. He had been kept on "submaintenance" for eleven months, was exceedingly thin and graded as a "poor canner." The appearance of the skeleton of this steer was remarkable, the marrow having nearly all disappeared, and in its place was a watery, ill-smelling liquid, having none of the ordinary properties of normal marrow, and no greasy or fatty appearance whatever.

Steer 595 was slaughtered in February, 1909, after having been kept on "maintenance" for a year. He was graded as a "canner."

The feed of all these animals was exactly the same in quality, differing only in quantity from the beginning, so that whatever differences appear in the skeleton are due to amount of food and individuality of the animals.

## Method of Obtaining Samples.

The samples were obtained at the time of slaughtering and when the carcass was cut up, fortyeight hours later. The carcass of each animal was cut into the regular wholesale cuts, and the right

<sup>&</sup>lt;sup>1</sup> Weiske, Zeit. physiol. Chem., **20**, 595 (1895). Lehman, Jahresb. u. Agr. Chem., **20**, 382 (1877). Sedlmair, Zeit. f. Biol., **37**, 25 (1898). Gusmitta, Jahresb. u. Thier. Chem., **1884**, 401. Forbes, Bull. **201** (1909). Ohio Agr. Expt. Station. Wiley, "Composition of Carcass of Pigs," U. S. Dept. Agr., Division of Chemistry, Bull. **53**, page 70 (1898). Hart, McCullum and Fuller, Am. Jour. Physiol., **23**, 246 (1909). Hart, McCullum and Humphrey, Ibid., **24**, 86 (1909).

half taken for all chemical analyses, being hand-separated into lean, fat and bone.

The bone samples were made up for analysis as follows: (1) head and tail; (2) feet minus hoofs; (3) shin and shank; (4) chuck and neck; (5) flank and plate; (6) ribs; (7) rump; (8) loin; and (9) round. The reasons for combining the samples in pairs in some cases was to reduce the number of samples.

The whole bone was taken for analysis, including such adhering fat, lean and tendon as could not be removed with a knife. The bones were not boiled nor heated to remove the adhering flesh in any case. The marrow was thus included in the sample.

In the case of 594, the bones of the various cuts were not analyzed separately, but a composite of the whole was made and analyzed as one sample. The analysis of this animal will therefore be comparable only with the averages of the others.

## METHODS OF ANALYSIS.

## Preparation of Sample for Analysis.

As the samples of bone were removed from the carcass they were weighed quickly to avoid loss of moisture, then broken up, and ground in a Mann's bone grinder. The ground sample was mixed thoroughly and quartered down to the desired size of sample. This portion was put into closed jars and sent at once to the laboratory or kept in the cooler until it could be weighed out for analysis.

## Moisture.

Samples for moisture and fat were weighed out rapidly, in triplicate, in tared porcelain evaporating dishes, the size of the sample varying according to the fat content, as judged by the appearance of the bone. For fat, greasy samples, 25–40 grams were considered sufficient while for those with little fat 100 grams or even more were sometimes taken.

The dishes containing the weighed samples were at once placed in vacuum desiccators and dried over sulphuric acid, at room temperature, by means of the Benedict vacuum method, as modified for use in this laboratory.<sup>1</sup>

This method has proved very satisfactory, agreeing very closely with the older methods, and leaving the sample in perfect conditions for further analyses.

Fat.

The determination of fat was made upon the same samples as for moisture. The dry substance was transferred carefully to Soxhlet extractors prepared by placing a pad of absorbent cotton in the bottom to filter the ether extract.

Redistilled anhydrous ether was used for the extraction, the fat being collected in tared flasks and weighed after drying for 24 hours in vacuum

After drying and extraction the triplicate samples were combined in one and ground in a steel mill until fine enough to pass through a millimeter mesh sieve. This ground, air-dry sample was used for the determination of ash and phosphorus.

## Ash.

Two gram samples were weighed out in tared porcelain crucibles dried at 100–110°, and the moisture determined. The samples were ashed by igniting over bunsen burners until practically free from carbon, the ignition being completed in the muffle at dull red heat. A clear, white ash could be readily obtained by this means in a short time.

## Phosphorus.

The ash from the above determination was dis solved by digestion in hot, dilute nitric acid and the solution made up to 250 cc. Aliquots of 25 cc. were taken and the phosphorus determined gravimetrically as in fertilizers by the official methods of the Association of Official Agricultural Chemists (U. S. Dept. Agr., Bureau of Chem., Bull. 107 (revised).

## DISCUSSION OF THE RESULTS.

In an investigation conducted, as this one was, with animals which must of necessity differ in many characteristics, much depends upon selecting animals which are as nearly alike in all respects as it is possible to obtain them. The steers in the "special maintenance" group, of which these six steers were a part, were all selected with the greatest care to obtain animals of the same age, size, breed, and general condition. However, even after this careful selection there were individual characteristics which influenced the results, especially the weight and proportion of the skeleton.

The weights of the skeleton as shown in Tables I and II are almost solely a matter of the individual characteristics of the animals. No. 594, killed at the outset, was the smallest of all the steers and the total weight of his skeleton is also the smallest. Measurements made during the feeding period show that the special maintenance steers all con-

<sup>&</sup>lt;sup>1</sup> P. F. Trowbridge (Nov., 1908), Proceedings of the Association of Official Agricultural Chemists (U. S. Dept. of Agriculture, Bureau of Chemistry, Bull. 122). L. F. Shackell, Am. Jour. Physiol., 24, 325 (June, 1909).



Plate 1.



Plate 2.

S. S. C.

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tinued to grow in height, those on maintenance and submaintenance, as well as the supermaintenance animals, indicating that the animal continues to build up the skeleton even when insufficient food is supplied for maintenance. The total weight of the skeleton is higher in all of the older steers than in 594, the young animal killed as a check. The difference is only slight in the case of 591. Steers 593 and 597 were very much alike in empty weight and weight of skeleton, though in physical condition, before slaughtering 593, was considerably superior to 597.

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Steer 597 was, in some respects, unlike the other two animals killed in September, being not only larger but also coarser boned. The skeleton of this an mal shows a higher fat content and a correspondingly lower moisture content than does that of 593, the former having been on maintenance for six months and the latter on supermaintenance for the same length of time (Tables III and IV).

The ratio of weight of skeleton to the empty weight of the animal varied with the condition of the animal at slaughtering (Table I). The term "empty weight" meaning the live weight of the animal minus the contents of the digestive and excretory system.

## Moisture and Fat.

The moisture and fat content of the skeleton of each animal is shown in Tables III and IV. The check steer, 594, was low in fat and high in moisture content, as compared with the other four animals, 591, 593 595 and 597. This can be accounted for by the age of the animal, Wildt<sup>1</sup> and others having shown that the fat content of the skeleton in growing animals increases with age. The four steers named above were, apparently, so fat as moisture and at content of the skeleton is concerned, normal animals. The variations between the different steers was in no case greater than could be explained by individuality.

The extreme effect of poor nutrition was shown in 592. The condition of this animal was approaching that of starvation. Practically all the fatty tissue had disappeared from his muscles, and even the marrow had gone. This is proved by the extremely low per cent. of fat in the skeleton, 3.09 per cent., as contrasted to that of the others, which averaged 17.09 per cent. The moisture content of the skeleton of 592, 52.46 per cent., contrasts as sharply with the average for the other five steers, 36.54 per cent.

<sup>1</sup> Landwirts, Versuchs Stationen, 15, 404 (1872).

Steer 595, after being on maintenance for a year, illustrated an important point sought in this investigation. This animal had continued to grow in size while remaining constant in weight. The growth had been at the expense of the fat in the muscles and tissues of the body, and when slaughtered almost no fat could be separated by hand from the muscles. At the same time the fat content of the bones was only very little, if at all, below that of normally fed animals. It is thus seen that the poorly nourished animal resorbed practically all of the fat from the muscles and tissues of his body before drawing upon the fat contained in the skeleton.

The distribution of the fat and water in the individual animals is shown also in Plates 1*a* and 1*b*. In the plates the cuts are so arranged as to give an ascending curve for one animal, steer 591 being taken as the basal animal. The arrangement of the cuts of this steer to give an ascending curve for the moisture content of the separate cuts gives a similar curve for the other steers, with the exception of 592. The variation in the moisture tent is not uniform in all cases, single cuts of each animal deviating from the general curve, but in general the order is the same for all of the four steers.

The fat curve is almost an exact reverse of that for moisture, the only general exception being in the case of the flank and plate cut. This cut containing more cartilage is highest of all in moisture, but also contains a fairly high per cent. of fat.

Steer 592 shows a striking exception in the distribution of moisture in the different cuts of the skeleton. The moisture curve for this animal instead of being a rapidly ascending curve is an irregular line having no semblance to the curve of the other animals. The curve representing the fat content of this animal is a comparatively horizontal line, with an extreme variation of less than two per cent. This shows that in the struggle to maintain life this steer had resorbed nearly all the marrow, until he had reached the extreme, where all portions of his skeleton contained practically the same amount of fat, averaging 3.09 per cent. of the whole weight of the skeleton.

The figures obtained by adding the per cent. of moisture to that of fat vary widely in the different parts of the skeleton of each animal, according to the nature of the bone in the cut. The variation is not constant in the different animals, and could not be plotted to show any similarity in curves. The sum of the per cents. of moisture and fat is, however, nearly the same for the composite of the whole skeleton in all of the animals as shown in Table I. The fact that in steer 592 the sum of the moisture and fat in the skeleton is a little higher than in the other steers indicates that when fat is resorbed water is deposited in its place, and the resame for the different animals, the per cent. of ash in the original sample will be of little value, since it will be entirely dependent upon the sum of fat and moisture.

The per cent. of ash in the original sample varied with no regularity in the separate cuts of the individual animals. The only cuts in which all the

TABLE	I.		
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	and the second second		SUMMAR	x or co.	mPOSITIC	JN OF SP	CELEION	OF SIX	SPECIA	L MAINT	ENANCE	STEERS.	
	Weight of skele- ton, kilos.	Empty weight of steers, kilos.	Per cent, skeleton to empty weight.	Moisture in skele- ton. Per cent.	Ether-soluble (fat) in skeleton. Per cent.	Sum of moisture and fat in skele- ton. Per cent.	Ash in skeleton Per,cent.	Phosphorus in ash of skeleton. Per cent.	Phosphorus in skeleton. Per cent.	Ash in skeleton freed from mois- ture and fat. Per cent.	Phosphorus in skeleton freed from moisture	Organic matter in skeleton not fat, Per cent.	Remarks.
Steer No. 594	39.270	247.52	15.86	39.89	14.68	54.57	23.81	18.48	4.40	52.40	9.11	21.63	Slaughtered as check animal, Feb., 1908
Steer No. 593	51.104	317.92	16.07	35.36	17.63	52.99	25.29	18.15	4.59	53.81	9.77	21.72	Six months supermaintenance, slaughtered, Sept., 1908
Steer No. 597	51.012	302.56	16.86	34.32	19.53	53.85	24.72	18.65	4.61	53.57	9.99	21.43	Six months maintenance, slaughtered, Sept., 1908
Steer No. 595	44.912	230.24	19.51	36.11	16.47	52.58	26.42	18.89	4.99	55.72	10.53	21.00	One year maintenance, slaughtered, Feb., 1909
Steer No. 591	40.396	190.31	21.22	37.02	17.32	54.34	23.81	18.45	4.39	52.15	9.62	21.85	Six months submaintenance, slaughtered, Sept., 1908
Steer No. 592	48.430	190.20	25.46	52.46	3.09	55.55	22.76	18.29	4.16	51.19	9.37	21.68	Eleven months submaintenance, slaughtered, Jan., 1909

verse may very probably be true during the fattening of animals and may explain why a steer may be getting fat but not gaining weight.

## Organic Matter not Fat.

The organic matter other than fat was calculated for the individual cuts of each animal, and it seems to be practically constant for all animals, varying in the cuts calculated between the limits 20.5 per cent. and 22.5 per cent. in the different parts of the same steers. For the composite of each of the six animals this content of organic matter, not fat, varied between the limits of 21.00 per cent. and 21.95 per cent., or is practically constant. This was calculated by difference, being the loss on ignition of the dry and fat-free substance. The constant proportion of this organic matter which forms the framework of the skeleton shows that in none of the animals was the ratio of the organic portion other than fat changed by feeding during the period. The slight differences in the per cent. of this organic substance are no greater than can be attributed to the variations in sampling or other causes.

## Ash.

The ash represents the mineral portion of the skeleton and is a large part of the total weight (Tables I and V). Since, as has been shown, the organic matter other than fat is practically the animals were similar were the flank and plate, which was lowest in ash in all cases, and the head and tail which analyzed very close to 25.00 per cent. of ash in all animals.

#### TABLE II.

WEIGHT OF SK	ELETON	OF THE	SEPARA	TE CUT	s, Gram	<b>IS.</b>
No. of steer	593	597	595	591	592	594
Head	6302	6357	5620	4888	4454	4740
Tail	222	270	280	146	342	146
Feet	5574	5378	4584	4570	5112	4576
Shins	4132	4114	3344	3572	4014	2974
Shanks	4650	4503	4032	3668	4316	3362
Chucks	9259	9820	8006	7248	8906	6890
Neck	756	932	952	796	1112	720
Flanks	63	32	92	80	76	78
Plates	4187	4240	3650	3324	4432	2998
Ribs	4325	4382	4290	3378	5050	3696
Loins	5001	5172	4690	3905	4446	4654
Rumps	2164	1644	1582	1335	1788	1120
Rounds	4470	4168	3790	3516	4380	3312
Total	51105	51012	44912	40396	48430	39266

#### TABLE III.

PER CENT. MOISTURE IN SKELETONS OF SPECIAL MAINTENANCE STEERS.

No. of steer	593	597	595	591	592	
Head and tail	43.70	43.83	45.12	45.58	50.77	
Feet	38.58	38.66	39.34	40.55	47.29	
Shins and shanks	33.31	28.69	29.74	31.94	50.37	
Chucks and neck	34.24	34.00	34.90	37.33	53.81	
Flanks and plates	48.19	46.99	46.43	47.46	57.21	
Ribs	30.86	35.42	33.01	35.12	55.95	
Loins	31.20	29.92	35.14	32.87	51.67	
Rumps	34.86	31.01	34.93	32.84	53.28	
Rounds	22.71	23.93	28.69	28.10	55.13	
Total skeleton	35.36	34.32	36.11	37.02	52.46	39.86

The per cent. of ash in the dry and fat-free substance (Table VI) is a better indication of the ash content than the per cent. in the fresh sample. Steer 595, the oldest of the lot when killed, was highest in ash content, this being not true of all parts of the skeleton, but of the skeleton as a whole.

#### TABLE IV.

PER CENT. FAT IN S	KELETONS	OF	SPECIAL	MAINTE	NANCE	STEERS
No. of steer	593	597	595	591	592	594
Head and tail	8.68	9.05	9.14	7.18	3.15	·
Feet	. 12.23	14.82	14.03	14.42	3.43	
Shins and shanks	21.92	21.56	20.36	22.03	2.25	
Chucks and neck	14.53	18.91	14.13	15.26	3.13	
Flanks and plates	11.87	15.85	11.95	11.68	2.25	
Ribs	18.79	19.31	16.59	15.56	2.69	
Loins	22.92	23.57	18.74	23.10	2.87	
Rumps	21.30	24.76	20.68	20.09	2.15	
Rounds	30.48	35.29	28.62	30.39	3.81	
Total skeleton	17.63	19.53	16.47	17.32	3:09	14.68

#### TABLE V.

DISTRIBUTION OF ASH IN SKELETONS OF SPECIAL MAINTENANCE STEERS; EXPRESSED IN PER CENT. OF FRESH WT.

No. of steer	593	597	595	591	592
Head and tail	25.00	25.00	25.61	25.29	25.08
Feet	25.32	23.73	25.78	22.14	24.42
Shins and shanks	24.41	27.81	28.76	24.91	26.28
Chucks and neck	27.42	25.83	28.05	24.23	21.45
Flanks and plates	17.24	15.36	19.63	17.71	17.62
Ribs	27.86	23.69	27.44	26.17	22.36
Loins	24.75	25.81	26.83	23.59	20.66
Rumps	25.94	23.66	24.13	26.20	23.57
Rounds	28.12	24.01	26.03	23.60	22.30
Total skeleton	25.29	24.72	26.42	23.81	22.76

#### TABLE VI.

PER CENT. OF ASH. REFERRED TO SKELETON FREED FROM MOISTURE AND FAT.

No. of steer	593	597	595	591	592	594
Head and tail	52.51	53.07	56.00	53.54	54.36	
Feet	51.48	51.00	55.29	49.19	49.56	
Shins and shanks	54.54	55.91	57.62	54.11	55.47	
Chucks and neck	53.52	54.85	55.03	51.10	49.83	
Flanks and plates	43.17	41.35	46.92	43.34	43.47	S
Ribs	55.34	52.33	54.46	53.07	49.20	
Loins	53.95	55.58	58.17	53.59	49.95	
Rumps	58.67	53.50	54.37	55.07	52.90	
Rounds	59.95	58.87	60.98	56.85	54.31	
Total skeleton	53.81	53.57	55.72	52.15	51.19	52.40

#### TABLE VII.

DISTRIBUTION OF PHOSPHORUS IN SKELETONS OF SPECIAL MAINTE-NANCE STEERS; IN PER CENT. OF FRESH WEIGHT.

No. of steer	593	597	595	591	592
Head and tail	4.59	4.63	4.99	4.48	4.42
Feet	4.59	4.38	4.72	3.89	4.34
Shins and shanks	4.44	5.21	5.56	4.54	4.90
Chucks and neck	4.89	4.71	5.18	4.75	3.99
Flanks and plates	3.12	3.09	3.63	3.35	3.17
Ribs	5.06	4.47	5.22	4.75	4.11
Loins	4.61	4.84	5.18	4.25	3.82
Rumps	4.32	4.55	4.49	4.68	4.34
Rounds	5.28	4.49	4.83	4.45	4.05
Total skeleton	4.40	4.61	4.99	4.41	4.16

Although steer 592 was the second oldest of the six, the ash content of his skeleton was lowest, in the skeleton as a whole. This may be a mere coincidence or it may point to a resorption of mineral matter from the bones during a long period of insufficient nutrition. No conclusion can be drawn from only one animal. Compare the recent work of Hart<sup>1</sup> and others at the Wisconsin Agricultural Experiment Station.

## Phosphorus.

The percentages of phosphorus, reported in Tables I and VII, are those obtained by determination of the phosphorus in the ash.

In order to show that very little phosphorus is dissolved by the ether, determinations of phosphorus were made upon each sample of fat extracted from the skeletons of steers 591, 593 and 597. The results show that the amount of phosphorus lost in this way is entirely negligible. The amount of phosphorus in the fat was less than 0.06 per cent. in all cases, averaging about 0.02 per cent. of the weight of the fat, or calculated to the fresh skeleton was less than 0.003 per cent.

The phosphorus content reckoned upon the original substance gives about the same indication as does the ash. Those steers having the highest total moisture and fat content, or, in other words, those having the least solid matter are lowest in phosphorus. Calculated to the dry and fat-free basis, the phosphorus content also corresponds to that of the ash.

The composition of the ash, as indicated by the per cent. of phosphorus, varies in the different parts of the skeleton. The per cent. of phosphorus in the ash of the single cuts varies differently in the several steers and no conclusion will be attempted from the present data.

#### CONCLUSIONS.

(1) Young, growing steers continue to grow in height and build up skeleton even when losing in weight.

(2) The skeleton is unaffected by poor nutrition until practically all of the fat has been removed from the muscles and other organs.

(3) The principal effect of poor nutrition upon the skeleton is the removal of the fat or marrow and the replacement of this with water.

(4) The per cent. of organic matter other than fat is practically constant for the whole skeleton, under different conditions of nourishment.

(5) No evidence was obtained to warrant the conclusion that the mineral matter is resorbed or affected in amount due to lack of proper nourishment, although in steer 592 there is some indication that this may have taken place.

<sup>1</sup> Hart, McCullum and Fuller, Am. Jour. Physiol., 23, 246 (1909). Hart, McCullum and Humphrey, Ibid., 23, 86 (1909).

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(6) The proportion of fat and moisture in the corresponding parts of the skeleton is fairly constant for normally fed steers. In steers which have suffered from insufficient nutrition for a long period the fat may be nearly all resorbed from the skeleton, and this resorption takes place from all parts of the skeleton.

(7) The proportion of organic and mineral matter in the skeleton varies with the age. This proportion varies in the different parts of the skeleton, according to the nature of the bone.

(8) The per cent. of phosphorus in the ash of the skeleton of steers is nearly constant. The per cent. of phosphorus in the ash of different parts of the skeleton of the same steer varies, but the average for the corresponding cuts of the five steers is fairly constant, showing a variation of not more than 0.7 per cent.

UNIVERSITY OF MISSOURI, June, 1909.

# EMIL CHR. HANSEN.

MAY 8, 1842-AUGUST 27, 1909.

Last year, on the 12th of November, an anniversary of no little importance was celebrated by the Carlsberg breweries in Copenhagen. On that day it was 25 years since the first pure culture yeast was introduced in the brewery of "Gamle Carlsberg." And to Emil Chr. Hansen justly went all the honors of the day.

Before Hansen made his epoch-making discoveries the brewing industry had been in the dark in regard to the causes of the very disturbing and often fatal troubles which occurred in the beer during fermentation and storage. Pasteur ascribed the diseases to the influence of bacteria and prescribed methods for the purification of the yeast, methods which in many cases not alone did not improve the conditions but even made them worse. Pasteur's methods were to cultivate the yeast in a sugar solution to which had been added tartaric acid or in beer-wort containing a small percentage of carbolic acid in order to destroy the bacteria. In many, and perhaps the most cases, however, the diseases of the beer were not due to bacteria but, as Hansen showed, to certain species of yeast, wild yeasts, and as the addition of tartaric acid and carbolic acid to the fermenting liquid did more damage to the good yeast than to the wild yeast, the result was the opposite of what was expected. The only solution which might

prove satisfactory seemed to be the elimination of the wild yeast as well as the bacteria. But how? This Hansen accomplished by introducing his pure culture method. Hansen devised methods by which it was possible to distinguish between the different species of wild and culture veast. Hansen, and not Robert Koch, was the first to introduce nutrient, transparent gelatine as a highly suitable medium in which to grow cultures. Hansen proved that by using pure cultures in the fermentation of the beer-wort, it was always possible to obtain the same result. If the starting yeast was kept pure and precautions were taken that no infection was introduced during the different stages of manufacture, the brewer would always be sure that he would obtain the same good and stable product. In connection with Kühle, the late director of "Gamle Carlsberg," he constructed a pure culture apparatus by means of which it was possible to work the pure cultures on a scale large enough for practical purposes.

From the time the pure culture yeast was first introduced in "Gamle Carlsberg" brewery, it has been a great success. With the generosity which always has characterized the Jacobsens (J. C. Jacobsen the founder of "Gamle Carlsberg," and his son, Carl Jacobsen), the discoveries of Hansen were given to the world, and every brewery in the old as well as in the new world has benefitted more from these than from any other discovery of modern times.

Not the breweries alone, but also the other branches of the fermentation industry: the distilleries, the compressed yeast factories, the wine factories have benefitted more or less from these same discoveries of Hansen. Also other industries have taken advantage of the pure cultures. Thus it was due to the introduction of the pure culture of lactic acid bacteria in the dairies that the Danish butter has reached the highest grade of perfection and won the reputation of being the best in the world.

Hansen limited his research work to the yeasts and to the bacteria which occur in the fermentation of beer, but these fungi he also pursued wherever and whenever he found them, not alone in the beer but also in the air, in the water, in the earth until they revealed their last life-secret to him. It is a fact that his results are final, at least, nobody has ever yet succeeded in disproving any of his printed statements.

Emil Chr. Hansen came from a poor family. His highly eccentric father was a painter by trade, and the son learned the same trade before he came to Copenhagen. Here he first started to study with the intention of becoming an artist, but soon he changed and took up scientific studies. During his years of study he had to provide for his own living by teaching school and drawing pictures and writing stories for the magazines. In 1876 he won the University's gold medal for a treatise on certain fungi found in manure, and a few years later he took his degree as Phil. Dr. At this time he also participated in the translation of Darwin's books.

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He was first connected with the "Ny Carlsberg" brewery, but as soon as the "Carlsberg Laboratory" (a research laboratory, founded by J. C. Jacobsen and maintained by "Gamle Carlsberg" brewery) was instituted, he was made director of one of its two departments, the physiological department, and here he worked until his death.

In the first years after his revolutionizing discoveries he met much opposition from both inside and outside Denmark, and the adversity seemed to weigh heavily on his shoulders, but even the faintest mumbling has stopped long ago and Emil Chr. Hansen has received the highest honors and recognition which it is possible to bestow on a man both from European governments and from the most important scientific bodies in the world.

His discoveries brought great fortunes to others. Hansen himself got nothing but his far from royal salary, but he was satisfied with a modest living and with small trips to the great art galleries of Europe; during his whole life he was a passionate lover of art.

At his death he did not leave much worldly goods, but he left a name for himself which will go down in history side by side by the names of Louis Pasteur and Robert Koch. NIELS C. ORTVED.

## NOTES AND CORRESPONDENCE.

## ELECTROTITRIMETER.

The "Electrotitrimeter" is an apparatus devised for testing the concentration of an electrolyte. It is based upon the fact that for many acids, alkalies and salt solutions the electrical conductivity varies with the concentration sufficiently to make the electrical test a method of accurate quantitative measurement. Temperature is such an important factor in conductivity work that a thermostat is usually used and the solution to be tested is brought to a given temperature before making the reading. According to Kohlrausch's method the results thus obtained are expressed in electrical units, the readings being made with a carefully calibrated rheostat. In the application of conductivity measurements for technical purposes, such apparatus is undesirable and by the use of the "Electrotitrimeter" it is unnecessary.

The difficulty of controlling the temperature has been overcome by the use of a compensating tube filled with a solution similar to the one to be tested, and when the apparatus is used for one purpose, such as for testing sulphuric acid of a certain strength, the tube is permanently sealed to prevent changes in concentration. This tube and the test tube are kept at the same temperature by immersing in a bath consisting of the solution which is being tested, or in another form of the apparatus the bath may be water.

The compensating tube and the test tube are each provided with two platinum electrodes and these are connected in such a way that each tube represents one arm of a Wheatstone bridge, and a high resistance calibrated wire represents



the other two arms. By means of a telephone and induction coil, a point is found on the bridge wire which shows the solutions in the two tubes to be in electrical equilibrium and any change in the concentration of the solution in the test tube causes this point to move. The wire is calibrated so as to read directly in percentage content of the solution to be tested.

The apparatus shown in Fig. 1 represents one form in which the electrolyte to be tested flows continuously through the test tube and then around both tubes forming the temperature equalizing bath.

In another form as shown in Fig. 2, the solution to be tested is poured into the test tube, the temperature equalizing bath being water in this case.

These tubes are each provided with glass stopcocks so that the solution in the compensating tube as well as in the test tube may be changed when different kinds of solutions are to be tested with the same apparatus.

When it is desired to control the strength of a product which flows continuously, a portion of the product is made to pass through the test tube, and with long connections the reading part of the apparatus may be a considerable distance from the point to be tested. Any number of points may thus be connected with one reading apparatus. All

that is necessary to make a determination at any point is to throw a switch making the connection, start the induction coil and with the telephone receiver at the ear, the pointer is moved along the bridge wire until the point of minimum sound is reached. The strength is then read off direct, the wire having been previously so calibrated when standardized.

With this simple apparatus, an unskilled laborer secures in a few seconds time results generally closer to the truth than those obtained by refined volumetric methods in the hands of a skilled ana-



lyst. This method is applicable for testing acids, water, solutions of various salts and alkalies. The sensitiveness in a few cases has been found to be approximately as follows:

	Per cer	ıt.	Per	cent.
Oleum .	30 Free	SO3	0.01	Total SO3
Sulphuric acid		100	0.01	H <sub>2</sub> SO <sub>4</sub>
		99	0.03	
		98	0.05	
		97	0.15	
Nitric acid		96-98	0.04	HNO3
Salt solution	1-3 NaCl		0.02	NaCl
11 II	3-5 NaCl		0.05	
Sea water	(Atlantic)		0.0004	sp. gr.
Free HCl in bi	chloride of ti	n	0.04	HC1

This last is only slightly affected by a change of  $1^{\circ}$  Bé in the strength of the solution.

Patent rights have been secured in Canada and France, and applications are pending in other European countries and United States. H. B. BISHOP.

#### THE TECHNICAL DETERMINATION OF CAOUTCHOUC IN GUAYULE.

The rubber chemist is frequently required to determine the amount of caoutchouc in rubber-producing plants. As a rule the assay offers but little difficulty. Woody plants like guayule and pingué often give trouble Guayule is especially troublesome.

Where only small quantities are available an extractor, usually of the Soxhlet type, is used. A rapid economical way, suitable for technical work, is to make a double extraction; firstly, with acetone to dehydrate and to remove resin and, secondly, with some rubber solvent (carbon bisulfid, carbon tetrachlorid, chloroform, naphtha, etc.). The acetone extraction offers no difficulties excepting a possible carrying through of a little of the finely ground material. This can be remedied by efficient packing, and by returning the first portions of the percolate. Trouble begins after the addition of the rubber solvent when the thick rubber solution clogs the apparatus. The clogging can be remedied by the proper packing of the material in the barrel of the extractor. Use a large extractor. Pack firmly into the bottom a thick layer of clean, short-cut cotton waste. Take a metal rod of the required length, wrap it with several layers of flannel, securing the cloth with fine wire. Place one end of the prepared rod in the layer of waste, fill in the substance to be extracted—distributing it evenly around the core. Cover with a layer of waste. Place a small glass rod in the reservoir to prevent superheating and start the extractor slowly. The action of the syphon is a rough indicator of the richness of the sample and also of the progress of the extraction.

Operating the extractor by direct heat usually results in a broken flask. Single extractors are best heated with a water-bath, using either gas or electricity.

Extractors in battery formation give best results with steam.

Where a large number of determinations are to be made, and where it is desirable to control factory methods, small (I pint or I quart size) ice-cream freezers in battery arrangement and operated by a small motor are desirable. One hundred grams are used as a charge. Benzol is used as the solvent; the resulting solution allowed to settle until clear and then alcohol added to complete precipitation. After settling of the precipitate decant the alcohol-benzol liquors, wash the precipitate, first, with a mixture of benzol and alcohol (precipitant in action), then with strong alcohol.

If quantity only is desired, the precipitated rubber is dried (vacuum preferred) on watch glasses or metal pans and weighed. Metal pans (copper) are preferred as they can be readily cleaned by burning. Scoring for quality requires that the precipitate be boiled with water to drive off solvents, then sheeted on the washing rolls and finally allowed to dry in a dark cool place. Clear, bright solutions give fine light rubber when dry. Cloudy, muddy solutions give black rubber.

Using finely cut material, duplicates give very uniform yields. Results are slightly low, owing to slight solubility of the caoutchouc in the benzol-alcohol mixture.

The most important part of the performance is the preparation of the sample. The guayule is a shrub, 10 to 30 inches high, and varying from 1 to 2 inches in diameter. The wood is very hard and bark thick. About 90 per cent. of the rubber is in the bark. With such conditions, to get a uniform sample for analytical purposes is a problem.

Usually, in a rubber factory the only available machine for pulverizing, is the common washing rolls with corrugated rolls.

The following experiments with this mode of grinding were made to determine the best stage of grinding. From the same shipment of shrub, five samples were prepared.

A. Coarsely ground. Many large pieces.

B. Ground medium fine. Obtained by re-grinding. Product more uniform than A.

C. Finely (?) ground. B reground until a sheet was obtained. Product not a uniform mass.

D. Medium fine. Sifted through a 1/4-inch mesh screen. Tendency towards separation between woody and barky portions. E. Coarsely ground, ball-milled for two hours. Wood reduced to powder; bark reduced to small scales.

These samples were extracted in a Soxhlet with acetone, and then by carbon bisulfid, the extracts dried at 100° C. and weighed. The table shows the results.

		Ace	tone ex	tract,	resin.		extrac	i bisuli t, rubb	id er.
	Preparation.	(1)	(2)	Av.	Var.	(1)	(2)	Av.	Var.
<b>A</b> .	Coarsely ground	9.91	9.84	9.88	0.07	9.77	10.95	10.41	1.18
в.	Medium fine	é9.16	8.84	9.00	0.32	9.34	9.44	9.39	0.10
c.	Finely (?) ground	9.90	9.74	9.82	0.16	7.33	8.71	8.02	1.38
D.	Medium ground,								
	sifted	9.97	10.77	10.42	0,88	8.68	9.88	9.28	1.20
E.	Coarsely ground,								
	ball-milled	9.63	9.54	9.74	0.21	7.88	9.29	8.58	1.41

An examination of these results reveals:

rst. That none of the methods of preparation are perfect. 2nd. That the regrinding system, (B) when carefully carried out, gives best results.

3rd. That the rubber content seems inversely proportional to the degree of fineness.

This is probably due to a more even distribution of the rubbery (barky) portion throughout the entire mass. In coarsely ground sample the barky portions, due to gravity, are usually on top and an undue proportion gets into the assay charge.

4th. That the tendency of the bark particles on account of their rubber content, as determined by the initial grinding or crushing, is to amalgamate in opposition to heavy milling or ball-milling and sifting combined, and that a cutting system is demanded. CHAS. P. Fox.

#### RECENT INVESTIGATIONS OF FATS AND OILS.

In the first number of THIS JOURNAL<sup>1</sup> I contributed a brief account of some work, carried out by Professor Paal, of Erlangen, on the catalytic reduction of fats and oils. The number of letters on the subject which I have received suggests that a further report on some subsequent investigations may prove to be of interest. The method of experiment consisted in emulsifying the fat or oil with an aqueous solution of gum arabic adding a little colloidal palladium, also dissolved in water. The remainder of the vessel was filled with hydrogen and it was shaken at the ordinary temperature, until the volume of gas remained constant. The relative quantities of fat and palladium which were employed differed within rather wide limits, in one case, which may be taken as fairly typical, the ratio of metal: fat was 1:500. In their most recent communication Paal and Roth<sup>2</sup> describe experiments with castor, croton, olive, sesame, cottonseed and linseed oils and with butter fat, lard and oleomargarin (from beef suet). In some cases complete reduction was only attained by a second or even a third treatment with hydrogen and colloidal palladium. The reduced fats and oils are in general, colorless solids, which melt at definite temperatures, ranging from about 45-70°. The compounds from croton and castor oils fail to exhibit any physiological activity. As a rule these reduction products are without taste or odor, but that from butter has a pleasant nutty flavor and a faint odor of the mother substance is noticeable in the castor oil derivative.

One of the most interesting points brought out in the course of the work is the lack of connection between the quantity of iodine and that of hydrogen with which a given fat or oil can combine.

It has been generally supposed that the additive power of a fat for hydrogen, iodine, or iodine chloride was dependent on the double (ethylene) linkages which were present. This, however, is by no means the only factor to be considered and it is necessary to recognize clearly that the addition of hydrogen and of iodine are quite independent reactions which proceed on parallel, but by no means on identical lines.

All the fats and oils mentioned above absorbed more hydrogen than the quantity calculated from their iodine numbers and the excess was quite considerable, amounting to over 25% in the case of linseed oil, to more than 50% in that of lard and, with the cottonseed oil, to almost 100%.

The reduced fats, although containing this "excess" of hydrogen are, in some cases, still capable of combining with iodine. The product from sesame oil and also that from oleomargarin, for example, have iodine numbers of 2 and 1.2, respectively.

This compound from sesame oil (iodine number 2) exhibits Baudouin's test only very feebly. After remaining during eight months the fat gives the Baudouin color quite strongly, although its iodine number is not changed. It is evident that the particular substance which is present in sesame oil, and on which the Baudouin test depends, is reduced by the hydrogen but is regenerated, to some extent at least, on exposure to air. The reduced fat from cotton-seed oil fails to show either the Becchi or the Halphen tests. In general, these reduced fats are quite stable in air and exhibit no sign of rancidity.

In another paper,<sup>1</sup> Paal and Hartmann show that a minute quantity of colloidal palladium solution is able to bring about the combination of ethylene and hydrogen. The liquid was shaken in a closed vessel, containing the two gases, at the ordinary temperature. The union of the gases was quantitative. This result is of considerable interest in connection with the reactions involved in the reduction of fats and oils.

The problem of the fats has been attacked in another manner by Quensell,<sup>2</sup> who has synthesized a number of the glyceryl esters of stearolic acid and also those of behenolic acid. From some of these esters he has prepared a number of halogen addition compounds. The first molecule of halogen is added by the acid radicle without difficulty, but combination with a second molecule takes place only slowly and in presence of light. In these respects the glyceryl esters resemble the free acids. In dealing with the diacylated glycerols, Quensell unfortunately fails to state which of the isomers, HOCH<sub>2</sub>CHOXCH<sub>2</sub>OX or XOCH<sub>2</sub>CH(OH)CH<sub>2</sub>OX (X =  $C_{18}H_{31}O$  or  $C_{22}H_{39}O$ ), he employed in his experiments with halogens, so that the value of his results is lessened. I. BISHOP TINGLE.

MCMASTER UNIVERSITY, TORONTO, CANADA, Aug., 1909.

## CORRECTED QUALITY FIGURES.

In the July number of THIS JOURNAL, the writer criticizes Turner's rule for getting quality figures as being

<sup>&</sup>lt;sup>1</sup> Vol. 1, 47. <sup>2</sup> Ber., 42, 1541.

<sup>&</sup>lt;sup>1</sup> Ber., **42**, 2239. <sup>2</sup> Ibid., **42**, 2440.

wrong in principle and giving, therefore, misleading results, and proposed a rule of his own which, however, upon further study, is seen to be also wrong (although to a lesser degree) when any factor happens to be abnormally low, and he therefore begs to substitute the following:

Find what per cent. the elastic limit of the steel under consideration is of the very best elastic limit that could be possible (always thereafter adhering to this best elastic limit as a standard, and so also of the other factors), what per cent. the shock or alternation figure is of the best, what per cent. the hardness is of the best. Add them together and divide by three.

According to this rule the quality figures given in the above-mentioned paper come as follows: Standard, elastic limit, 200. Shock, 20. Hardness, 600.

QUENCHED VANADIUM NICKEL STEELS.

	CHARGE ST		
Cor	npositi	on. (	Quality figur
C 0.20	Ni 6	Va 50	0.5570
C 0.20	Ni 6	Va 70	0.4433
0	1.194		
Q	UENCH	ED CHROMIUM, ETC. NICKEL STEE	LS.
C 0.20	Ni 6	Cr 0.50	0.5653
C 0.20	Ni 5	Cr 1.0	0.6583
C 0.20	Ni 6	Cr 1.0	0.5422
C 0.20	Ni 5	Cr 3.0	0.5055
C 0.20	Ni 6	W 2.0	0.6050
C 0.20	Ni 6	Wo 30	0.5900
C 0.20	Ni 5	Wo 30	0.5939
C 0.20	Ni 6	Mo 1.0	0.5072
C 0.20	Ni 6	Mo 0.50	0.6030
C 0.20	Ni 6	Mo 2.0	0.5317
C 0.20	Ni 6	Si 0.50	0.5839
C 0.20	Ni 6	Si 0.80	0.6133
	QUEN	CHED VANADIUM NICKEL STEELS.	
C 0.20	Ni 2	Va 30.0	0.5600
C 0.20	Ni 2	Va 1.0	0.4636
	QUE	ENCHED SILICON NICKEL STEELS.	
C.0.20	Ni 2	Si 1 5	0.4603
C 0.20	Ni 2	Si 1 0	0.5252
C 0.20	Ni 2	Si 0.50.	0.6131
	Nor	MAL VANADIUM NICKEL STEELS.	
C 0.20	Ni 6	Va 30.0	0.3693
C 0.20	Ni 6	Va 50.0	0.3609
C 0.20	Ni 6	Va 70.0	0.3897
	NOR	MAL CHROMIUM NICKEL STEELS.	
C 0.20	Ni 5	Cr 1.0	0.5628
C 0.20	Ni 6	Cr 1.0	0.4639
C 0.20	Ni 6	Cr 2.0	0.5467
	NOR	MAL TUNGSTEN NICKEL STEELS.	
C 0.20	Ni 6	W 1.0	0.4517
C 0.20	Ni 6	W 2.0.	0.4100
C 0.20	Ni 6	W 6.0	0.4319
	NORM	AL MOLYBDENUM NICKEL STEELS.	
C 0 20	Ni 6	Mo 0 50	0.5203
C 0 20	Ni 6	Mo 1 0	0.5064
C 0 20	Ni 6	Mô 2 0	0.4561
C 0 20	Nic	Mo 5.0	0.4311
00120	in o		
	NOR	MAL SILICON NICKEL STEELS.	0.5001
C 0.20	Ni 6	St 0.50	0.5094
C 0.20	Ni 6	Si 0.80	0.3144
C 0.20	Ni 6	Si 1.0	0.2822
C 0.20	Ni 5	Si 2.0	0.2722
C 0.20	Ni 6	Si 2.0	0.3511

Mr. Turner's Tests.—Standard, 200,000 elastic limit. 2000 alternations. Not comparable with preceding. Vanadium Chrome Steels.--No. 15, 0.5568; No. 17, 0.5203; No. 23, 0.6083; No. 24, 0.5333; No. 27, 0.6170.

Nickel Chrome Steels.--No. 9, 0.3858; No. 21, 0.3907; No. 28, 0.4810. George Auchy.

#### A MODIFIED KJELDAHL CONNECTING BULB.

A modification of the Kjeldahl connecting bulb, which is not only useful but also tends toward greater accuracy in ammonia determination, is represented in the drawing. This was designed primarily for ammonia determinations in water but will, no doubt, find favor in other ammonia determinations as well.

The drawing speaks for itself, so there is little need for an explanation of the modified apparatus. Chemists making an ammonia determination in water with a Kjeldahl flask connected to a condenser by a Kjeldahl bulb pursue



a method which is capable of greater experimental error than it should be. In an effort to lessen this experimental error, the above modification was made. With the old style of bulb it was necessary, after freeing the apparatus of ammonia, to remove the rubber stopper containing the connecting bulb and pour it through the neck of the flask, the sample of water to be analyzed. The stopper had to be removed again when introducing the alkaline permanganate for the determination of the albuminoid ammonia. This makes possible the introduction of ammonia into the apparatus which has previously been carefully freed of ammonia. With the modified connecting bulb, the opening in the bulb is much smaller and is far removed from the body of the flask, and the danger of contamination with ammonia when the stopper is out of this opening while introducing the water sample and the alkaline permanganate solution is reduced to a minimum.

The writer has used this form of the Kjeldahl connecting bulb for more than a year and has found it very satisfactory. They were made for the writer by E. H. Sargent & Co., of Chicago. C. A. JENNINGS.

CHIEF CHEMIST AND SUPERINTENDENT OF FILTRATION, UNION STOCK YARD AND TRANSIT CO., CHICAGO.

#### A SIMPLIFIED COMBUSTION CRUCIBLE.

In 1899 the writer published a paper<sup>1</sup> describing an ordinary platinum crucible which, for combustion purposes, could be closed by means of a water-cooled brass stopper fitted with a rubber band and provided with inlet and outlet tubes. In 1901 he described a special water-jacketed crucible<sup>2</sup> to be used in connection with the water-cooled stopper. This latter form has come into rather extensive use. A disadvantage, however, is its rather high cost, the 60 cc. size weighing about 92 grams; it is also rather difficult to manufacture and repair.

In the latest form of the apparatus<sup>3</sup> the crucible is a simple one, without the water jacket. It is 60 cc. capacity, rather deep in form, and weighs only 50 grams. When provided with an ordinary cover, to be used when making fusions, the weight is 57 grams. The crucible is accurately fitted with a rigid knurled ring around its upper part, easily removable. The water-cooled German silver stopper, instead of having a rubber band to secure a tight joint, is ground into the crucible. This joint may easily be kept tight by an occasional grinding in, using for this purpose a little glass, finely ground in the agate mortar with oil.

A very good way to secure absolute tightness and, at the same time, a certain lubrication, is to melt the rubber at the extreme end of a "policeman" in the flame and apply the minutest amount possible at intervals around the upper edge of the crucible. Now insert the stopper tightly with a turning motion. One milligram of rubber is enough for the purpose and the water circulation prevents this from being affected by the heat.

The advantages of this form of apparatus over the older form are:

1. The 60 cc. crucible weighs about 40 grams less and, on account of its simple form, it is less expensive to make.

2. When provided with an ordinary platinum cover it is a fusion crucible, on removal of the ring. Its deep form is desirable for fusions. This ready adaptability for both combustions and fusions is an advantage in any laboratory, especially the small one in which platinum is limited.

3. By doing away with the rubber band there is no risk of losing a determination in case the water supply fails temporarily.

4. The knurled ring protects the mouth of the crucible from injury and stretching, supports the crucible at the proper height in the asbestos board, and helps to secure a tight joint

<sup>1</sup> Jour. Am. Chem. Soc., 21, 1899.—"Carbon Combustions in a Platinum Crucible."

<sup>2</sup> *Ibid.*, 23.—"A Spiral Crucible for Carbon Combustions." <sup>3</sup> Patented 1909. 5. It is more convenient than the older form because it may be removed easily from its support for charging; this is especially advantageous in direct combustions of steel.

6. Smaller cost of repairs and renewals.

The writer, nearly ten years ago, tried nickel in place of platinum, but found it more expensive in the long run and otherwise less desirable. It is estimated that one carbon

## FIG.I.



combustion in a 60 cc. platinum crucible costs about 1/3 of one cent for platinum, counting interest on the investment, repairs and final scrapping. The short life of a nickel crucible makes each combustion cost about 3 cents in nickel, with risks of faulty results toward the end of the life of the crucible.

The train for carbon determination is as follows: 1. The oxygen tank 2. KOH bulbs. 3. A small guard bulb. 4. The

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crucible. 5. The burner. 6. A water-jacketed brass CuO tube. 7. The zinc tube according to Johnson. 8. Calcium chloride tube. 9. Soda-lime U-tube or other absorbent. 10. A small guard tube of CeCl., 11. An empty bottle provided with inlet and outlet tubes, the latter dipping under the surface of water contained in a small beaker for the purpose of noting the rate of flow of oxygen in direct combustions. The CuO tube is used only when making carbon determinations by the solution method and for organic combustions; for direct combustion of steel it is not needed. As a source of heat, a good Bunsen burner with a hot "roaring" flame is sufficient for most purposes. In many combustions, however, the upright blast lamp is necessary. For absorbent, a soda lime U-tube weighing, when filled, anywhere from 50 to 100 grams, is used. The soda lime should be about 20-mesh in size and free from powder.

Drillings of steel for direct combustion should be thin and should be between 20- and 60-mesh in size. As a base for the drillings a great many substances have been proposed and the writer has tried most of them, among them sesquioxide of iron both in the form of rich hemetite ore and chemically pure sesquioxide. With the latter good results may be obtained, but it is rather hard to get it reliably free from "blank." Very satisfactory results may be obtained by using sea sand previously washed and well ignited. The grains are rounded; it holds the fused oxide well and leaves the crucible clean.

In charging the crucible, put in not less than 1/4 inch deep of the sand, hollow out a little depression and pour the drillings into the middle of it through a funnel with wide and short stem. Cover the drillings with a mixture of finely divided ignited asbestos and powdered CuO. Onehalf the factor weight—1.3635 gram—is the usual weight of steel used. Combustion should be active in from 1 to 3 minutes and should be complete in 20 minutes or less.

Carbon in pig-iron may be determined by direct combustion on the sand, without admixture, but it is best to use the blast lamp. When red lead is used it is, of course, necessary to use a porcelain crucible, with sand, inside the platinum crucible.

In determining carbon in ferro-chrome, very good results were attained by burning on the sand a very intimate mixture (mixed on glazed paper with spatula) of 0.6818 gram of the finely ground ferro-chrome with four times its weight of powdered CuO.

A little experience and observation will soon indicate the best way to make direct combustions in the various steels and alloys, but it is always best to avoid coarse or thick drillings and brittle alloys should be finely pulverized. It is always well, when in any doubt as to the completeness of the combustion, to examine the fused mass for metallic particles by grinding in a mortar.

A few results follow:

	By solution and combustion.	By direct combustion.
	(0.137	0.137
	0.240	0.239
	0.424	0.420
Straight steels	0.600	0.603
	0.829	0.823
and the second	1.036	1.035
	0.627	0.628
	0.452	- 0.456
Pig iron	3.62	3.64

				By	direct combust	10n.
	Ferro	-chron	me	7.09	7.10	7.12
	Alloy	steel-	-W, Cr, V		0.450	0.444
	и	u	Ni, Cr, V		0.920	0.916
	u	u	Ni, Cr. V		0.660	0.666
	"	u	W. Cr. V		0.542	0.550
	"	u	Ni, Cr		0.194	0.196
	u	"	Cr, V		0.998	1.000
			· .		PORTER W	. SHIMER
F	ASTON	. PA.				

## NOTE ON A RAPID COOLING ELECTRICALLY HEATED COMBUSTION TUBE.

The object aimed at in designing the furnace described below was to devise a combustion tube which would combine the advantage of rapid and equal heating of an electrically heated combustion tube with the rapid cooling of a gas-heated combustion furnace. Most electrically heated furnaces are so carefully insulated against loss of heat by radiation that when the heating current is cut off the cooling of the furnace is very slow. Fused silica combustion tubes have been used so that a cold combustion boat could be pushed into a hot tube without danger of cracking the latter. Experience in this laboratory has gone to show that if the combustion boat, containing the sample, particularly iron or steel, can be put into a cold or nearly cold furnace. and the temperature raised to about 900°, that complete combustion of the carbon can be readily effected without danger of the residual oxides spattering, thus endangering the tube. Porcelain combustion boats, provided with a thin sheet platinum lining, are usually preferred by the author.

The combustion furnace, which has given very satisfactory results in this laboratory, is made from a porcelain combustion tube, 78 cm. long, 24 mm. inside, and 30 mm. outside diameter, provided with a side arm about 5 cm. from one end. A tube of fused silica might easily be substituted for the porcelain tube, but the silica tubes are not made with side arms, and are usually not made true enough to be easily stopped gas-tight with a rubber stopper. With the system of winding used in the furnace to be described, there is not much danger of cracking a porcelain tube by too rapid heating.

For electric heating, the tube is wound with a "Nichrome" ribbon, 4.7 mm. (3/16 inch) wide and 0.1 mm. (0.004 inch) in thickness. The general construction of the furnace is shown in the accompanying sketch. The wound portion in the central part of the tube is about 38 cm. in length. Before winding with the nichrome ribbon, this portion of the tube was wrapped with two thicknesses of thin, pure asbestos paper, laid on wet with water only. The nichrome ribbon was bent at right angles and at the bent portion was wired to the asbestos with a piece of nichrome or nickel wire. The tube was then wound spirally, the space between adjoining spirals being about two mm. When this first winding was complete, the end was again wired, the ribbon bent at right angles, and carried along the top of the tube for connection with the binding posts, B. When the inner winding had been put on, this was wrapped with four thicknesses of thin pure asbestos paper, extending about one cm. beyond the beginning of the spiral wind. The four outer layers of asbestos were put on wet with water only,

and outside of this a second winding of nichrome ribbon was wound. This outer layer of nichrome ribbon is wound with the edges close together, thus forming a practically continuous thin metal jacket, insulated from the inner nichrome winding, but serving to prevent the asbestos from crumbling and falling off after heating. At each end of the nichrome jacket, which was prevented from unwinding by wiring with nichrome or nickel wire, a collar, A, about 30 mm. wide and 8 mm. thick, was made by winding that portion with a strip of thin asbestos wet with a dilute solution of sodium silicate. The binding posts, B, were first soldered to a strip of copper, the width of the binding post, and about 30 mm. long. The second strip of copper, of the same size, could be fastened to the under side of the binding post by means of a screw. Connection with the nichrome ribbon is made by inserting it between these two copper strips before tightening the screw. Each binding post was attached to the porcelain tube by first wrapping the latter with a strip of asbestos, wet with dilute sodium silicate, thus making a collar about 40 mm. wide and 3 or 4 mm. thick. The binding post was then placed on this collar and firmly fastened by means of a couple of wires. After the nichrome windings were complete, and connection made to the binding posts, the tube was thoroughly dried

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to the porcelain tube by packing the space, J, with a stiff mixture of talc and diluted sodium silicate. When this latter mixture has been thoroughly dried in the drying oven, the tube was evacuated and the joint at J made permanently gas-tight by applying hot rubber cement. When in use, the portion of the porcelain tube from the nichrome jacket to the outlet end is filled with pieces of porous ware, and the heated portion of the tube, for a distance of about 10 cm., is filled with similar pieces which have been platinized by first saturating with a dilute solution of platinic chloride, and subsequently drying and igniting at a bright red heat. After the boat containing the sample has been pushed into the furnace, a porcelain plug, K, about 12 cm. long is put in in order to prevent radiated heat from affecting the rubber stopper, L. The plug, K, consists of a piece of porcelain tubing 12 mm. inside diameter, provided with a nickel wire loop at one end and silicated asbestos collars at both ends, these collars increasing the diameter almost to that of the inside diameter of the combustion tube. The rubber stopper, L, used for closing the open end of the combustion tube, contains a piece of clear glass rod, M, cut the length of the stopper and polished at both ends. Through this glass, and the hollow plug K, the color of the interior of the combustion tube may be readily observed.



in a drying oven, in order to harden the asbestos collars at the ends of the nichrome jacket, and under the binding posts. In order to prevent loss of heat, the removable mantle, C, was made, by thoroughly wetting with sodium silicate three or four thicknesses of heavy sheet asbestos, and shaping it over a piece of tubing a little larger in diameter than the collars, A. Asbestos soaked in a solution of sodium silicate, diluted with twice its volume of water, after drying, became quite hard and strong, and has been found very useful in this laboratory for a good many years. The mantle, C, is provided with a loosely fitting bottom, D, made from a strip of heavy asbestos board hardened with sodium silicate. The mantle bottom, D, when the furnace is in use, rests upon three heavy wire pins, E, passing through the mantle. If these pins are pulled out, the bottom and mantle can be easily removed, thus permitting the furnace to cool quickly; at the same time the asbestos and nichrome jacket are sufficiently thick to prevent the tube from cracking. When in use, the combustion tube is supported by means of two adjustable supports, F.

Oxygen for combustion is admitted through the side arm, G, and the products of combustion pass out through the thick-walled glass tube, H. This glass tube is cemented The heating current is controlled and measured by means of a rheostat and ammeter, kept in the circuit during the working. When testing the rate of heating and cooling, a standardized platinum rhodium thermocouple was inserted into the furnace in place of the combustion boat. With the heating current maintained at 8.3 amperes the thermocouple showed a temperature of  $500^{\circ}$  in five minutes and thirty seconds,  $600^{\circ}$  in seven minutes and fifteen seconds,  $700^{\circ}$  in nine minutes and fifteen seconds,  $800^{\circ}$  in twelve minutes,  $900^{\circ}$  in fifteen minutes and fifteen seconds,  $950^{\circ}$  in nineteen minutes and thirty seconds. Nine hundred degrees is hot enough for direct carbon combustions in iron and steel, and 7.9 amperes was the current required to maintain the furnace at this temperature.

Starting with a temperature of 934°, the rate of cooling is shown in the following table, which gives the temperature at intervals of two minutes:

Time in minutes.	Temperature in degrees centigrade.
0	935.0
2	800.0
4	650.0
6	529.0
8	435.0

· Time in	Temperature in
minutes.	degrees centigrade.
10	368.0
12	314.0
14	271.0
16	238.0 .
18	206.0
20	180.0
22	158.0
24	140.0
26	125.0
28	112.0
30	100.0
	Edward DEMILLE CAMPBELL,
	EDGAR NATHANIEL GOTT.
EMICAL LABORATORY,	
IVERSITY OF MICHIGAN;	
ANN ARBOR.	

#### HOME-MADE ELECTRIC FURNACE.

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In view of the fact that the writer has received a great many inquiries as to the method of making the electric furnace referred to in the article entitled "Economy in the Use of Oxygen, Etc.," <sup>1</sup> he believes that a note describing the same may be of interest to others. project far enough for a connection and wind the strip of platinum tightly around the tube, leaving about 1/4" or more between turns and fasten with the clamp at the other end, allowing it to project in a similar manner for connection with the current. The length of the tube wound with resistance should be about 9" and not more than 10". Cover this with a plaster about 1/8" to 1/4" thick made of broken up pipe covering mixed with water and enough fire clay to act as a binder, putting it on in much the same way as frosting is put on a cake, thus enclosing the resistance in an infusible casing which proves very efficient in preventing a short circuit. Next cut off a piece of magnesia pipe covering (1 3/4" inside diam.) 9" long and put it around the tube, replacing the cloth cover with asbestos and fasten it on with a few turns of iron wire. Prepare a stand similar to that in the illustration (scale: 1/2'' = 1') in THIS JOURNAL and place on the iron brackets of the stand two pieces of pipe covering, one inside the other, the outside one having an inside diameter of 7", and the inside one an outside diameter of 7".

Slip the tube into its position and strap it down with pieces of leather thong to the two uprights—these should be of such a height above the brackets as to support the



#### FURNACE

#### LAMPS

The furnace is strictly "home-made" and gives excellent service. As before brefly explained, it is a porcelain tube wound with a platinum resistance and jacketed with pipe coverings. A porcelain tube is to be preferred to one of quartz on account of the repeated annealing it has to undergo in cooling off every night. The condition of the former is improved by such treatment, but the latter assumes a crystalline structure and becomes very brittle. The tube should be about two feet long and one inch in diameter. The resistance is a strip of platinum foil, 8 feet long, 4 mm. wide, and about 10 mm. thick, with the last four inches of each end left about 10 mm. wide in order to have metal enough to make a good cool connection with the circuit.

To make up the furnace, moisten a piece of sheet asbestos 16'' long by 1/16'' thick, and wide enough to encircle the tube, and wrap it once around the tube, having first covered the latter with a little water glass solution. Make a couple of clamps of sheet nickel. These are bands about 1/2'' wide and 1/32'' thick clamped together with small bolts and nuts and are intended to hold the two ends of the foil. Put them on about six inches from each end of the tube and clamp one end of the foil under one of them, letting it

<sup>1</sup> THIS JOURNAL, 1, 375.

tube independently of the outside jacket—then plaster up each end of the jacket with a paste of pulverized pipe covering or of asbestos cement and the furnace is ready for use.

GENERATOR

SWITCH

Sometimes the heat fuses the inside of the tube a little, but when this is the case, magnesium oxide spread out on the inner surface will soon prevent any further trouble. It is necessary to use a nickel rod or wire to push the boat into the tube, as an iron rod becomes magnetized and will pull the drillings from the boat on being withdrawn.

We are at present using this furnace on a 220 volt direct current circuit in series with a lamp back of twelve to fourteen one ampere (100 c. p.) lamps. This gives a current of about 12 amperes with a drop across the furnace of 30 to 35 volts and a temperature of 1100° to 1200° C. This, of course, can be regulated to almost any degree by changing the number of lamps. It is possible to attain a temperature of 1500° C., but it is not advisable to run it as high as that, both on account of the liability of a burn-out and the loss of platinum by volatilization. In case a burn-out occurs it is a simple matter to weld the foil electrically, thus making quick repairs possible. In fact it is possible to entirely dismantle and rebuild this, furnace in less than an hour. The cost of the furnace need not exceed \$30, and it may be made for much less and operated with much less power if a thinner foil is used as a resistance. The dimensions given, however, are preferable for furnaces in continuous operation. The ideal method of using such a furnace is in conjunction with a motor-generator set, but the following sketch will show the method of wiring above described. WM, HERBERT KEEN.

LABORATORY OF THE FILTH STERLING STEEL CO , WASHINGTON, D. C.

## BOOK REVIEWS AND NOTICES.

The Lead and Zinc Pigments. By CLIFFORD DYER HOLLEY, M.S., PH.D., Chief Chemist, Acme White Lead and Color Works. 12mo. xv + 340 pp., 85 figures. Cloth, \$3.00 net. New York: John Wiley & Sons.

The thirty-one chapters of this book contain much that is of interest and value. Much of the matter is of a historical and descriptive nature. The analytical methods are in the main adequate. Among the defects of the work are selection of illustrations of superficial rather than intrinsic merit (notably the frontispiece), and the addition of an appendix containing tables which are already in the possession of all chemists in books of more convenient form.

The Scientific and Industrial Bulletin of Roure, Bertrand Fils, of Grasse, for April, 1909 (Series 2, No. 9), while appearing rather late contains much matter of interest in connection with essential oils. Among other articles are these: "The Resin Industry of the Landes;" "Contribution to the Study of Perfume-Yielding Plants;" "The Suppression of Fraud So Far as Essential Oils Are Concerned;" "Review of Recent Work on the Perfumes and Essential Oils." 8vo. pp. 160.

The Mineral Content of Illinois Waters. Bulletin No. 4 Illinois State Water Survey. Prepared in Coöperation with the State Geological Survey. By Edward Bar-Tow, J. A. Udden, S. W. Parr, and George T. Palmer, Urbana, 1909.

This volume contains an introduction by Edward Bartow; Geological Classification of the Waters of Illinois, by J. A. Udden; Classification of Mineral Waters, According to Physical and Chemical Properties, by Edward Bartow; Methods and Interpretations, by Edward Bartow; Boiler Waters, by S. W. Parr; Mineral Springs of Illinois, by George T. Palmer, M.D.

## NEW BOOKS.

#### This list was prepared by Mr. Paul Escher, 1319 Pearl Street, Alameda, California.

#### Agricultural Chemistry.

- Moisture Content and Shrinkage in Grain. By J. W. T. DUVEL. pp. 13. (Circular 32, Bureau of Plant Industry.)
- Agriculture for Southern Schools. By JOHN FREDERICK DUGGAR, Louisiana edition, MacMillan Co., New York, Ill., 12. \$.60x. Cloth, xi. 340 pp. xlii pp., index.

#### Fertilizers.

- Artificial Manures: Their Chemical Selection and Scientific Application to Agriculture. By M. GEORGE VILLE, translated and edited by SIR WILLIAM CROOKES. New edition revised by SIR WILLIAM CROOKES, D.Sc., F.R.S., and JOHN PERCIVAL, M.A. 6 plates. 8vo. pp. xl, 348. Longmans, Green & Co., London and New York. Price, \$3.00 net.
- Chemical Conversion Tables. For use in the analysis of fertilizers, fertilizing materials, iron and its products, etc. By H. B. BATTLE, PH.D., and W. J. GASCOYNE, PH.D. Pocket size, indented reference index. Williams & Wilkins Pub. Co., Baltimore, Md. Price, \$2.50.

### Mining and Metallurgy.

- Antimony. By WANG CHUNG YU. J. B. Lippincott Co., Philadelphia. 1909. Price, \$4.00.
- The Nature of Ore Deposits. Translated and revised by W. H. WEED. R. BECK. 1909. Hill Publishing Co., New York. \$6,00.

#### Fuel.

- On the Rate of Formation of Carbon Monoxide in Gas Producers. By J. K. CLEMENT, University of Illinois, Urbana. 1909.
- Das Erdöl, Seine Physik, Chemie, Geologie, Technologie, Etc. By C. ENGLER and H. HOFER. 5 bände. Band 2. Geologie, Gewinnung und Transport. Mk. 46, geb. 50.

#### Paints.

Lead and Zinc Pigments. By C. D. HOLLEY. John Wiley & Sons. New York. Price, \$3.00.

#### Industrial Chemistry.

- Chemistry for Engineers and Manufacturers. By B. BLOUNT and A. G. BLOXAM. Vol. II. The Chemistry of Manufacturing Processes. 8vo., cloth. Ill., 514 pp. J. B. Lippincott Co., Philadelphia. Price, \$4.00.
- Cane Sugar and Its Manufacture. By H. C. PRINSEN GEER-LIGS. Large 8vo., pp. 350. 128. Norman Rodger, Altrincham, Manchester.
- Tables and Diagrams of the Thermal Properties of Saturated and Supersaturated Steam. By LIONEL S. MARKS, M.M.E., and HARVEY N. DAVIS., PH.D. Large 8vo., pp. 106. Longmans, Green & Co., London and New York. \$1.00 net; by mail \$1.10.
- Die Fabrikation der Lacke, Firnisse und des Siegellacks. By ERICH DETTMERS. (116 Band. Bibliothek der gesamten Technik.) 8 dbb. Mk. 2.60. M. Janecke, Hannover.
- Chemisches Auskunftsbuch für Fabrikanten, Gewerbetreibende und Landwirke. By HUGO KRAUSE. A. Hartleben, Leipzig und Wien. Mk. 4.80.

## SCIENTIFIC AND INDUSTRIAL SOCIETIES.

## ASSOCIATION OF STATE AND NATIONAL FOOD AND DAIRY DEPARTMENTS.

The thirteenth annual convention of the Association of State and National Food and Dairy Departments was held at the Brown Palace Hotel, Denver, Colo., August 24-27, 1909. On the opening day, August 24, addresses of welcome were made by the Governor of Colorado, the Mayor of Denver, and the Secretary of the State Board of Health, followed by a response by Food Commissioner A. C. Bird, of Michigan. President J. Q. Emery, of Wisconsin, then delivered his address in which he noted the progress made by the association during the year. He discussed at length the question of benzoate of soda and criticized the findings of the Referee Board appointed to investigate its effect on health when present in food products. By general request Hon. James Wilson, Secretary of Agriculture, then addressed the Association, defending the Referee Board and calling for its fair treatment at the hands of the Association.

During the afternoon session five minute speeches were made in the most salient features in the food, dairy and drug control work of the respective states, by the various commissioners, executive officers and visiting chemists representing the states that follow: California, Colorado, District of Columbia, Georgia, Illinois, Idaho, Indiana, Iowa, Kansas, Kentucky, Louisiana, Maine, Maryland, Massachusetts, Michigan, Minnesota, Missouri, Nebraska, New Jersey, New York, North Carolina, North Dakota, Ohio, Oklahoma, Oregon, Pennsylvania, South Dakota, Tennessee, Texas, Utah, Wyoming and Wisconsin. A food chemist's conference was listed for Tuesday afternoon, but was not held.

On Wednesday the delegates and guests enjoyed a complimentary, all-day mountain trip through Clear Creek Canyon over the Georgetown Loop and to the summit of Mt. McClellan, over 14,000 feet above the sea.

On Wednesday evening a meeting was held at which Commissioner E. F. Ladd, of North Dakota, addressed the Association on a Model State Food Law, and Commissioner H. E. Barnard, of Indiana, spoke on Sanitary Inspection of Food Products and Distribution.

Thursday's program opened with a discussion of the report of the Referee Board on sodium benzoate, beginning with short addresses by Doctors Ira Remsen, R. H. Chittenden, John H. Long and C. H. Herter, members of the Board. This was followed by a brief report of a committee of State Food Chemists, appointed by the executive committee to review the experiments and conclusions of the Referee Board, the members of that committee being Messrs. M. A. Scovell, Chairman, Lexington, Ky., W. M. Allen, Raleigh, N. C., E. H. S. Bailey, Lawrence, Kansas, H. E. Barnard, Indianapolis, Ind., Richard Fischer, Madison, Wis., J. Hortvet, St. Paul, Minn., M. E. Jaffa, Berkeley, Cal., E. F. Ladd, Agricultural College, N. D., Floyd W. Robinson, Lansing, Mich., Jas. H. Shepard, Brookings, S. D., and Chas. D. Woods, Orono, Me. Dr. Chas. A. L. Reed, of Cincinnati, Chairman of the Legislative Committee of the American Medical Association, then read a paper on "Some Queries Suggested by the Referee Board on Benzoate of Soda," in which he criticized the methods employed by the Board, claiming that dietaries were selected of a nature specially adapted to add weight to the subjects experimented on, and suggested that the experiments were conducted by subordinates not under the immediate supervision of the members of the Board. Doctors Remsen, Chittenden and Long made further remarks in reply to Dr. Reed and ended the discussion. A paper was read by Dr. D. R. Lucas, of Columbia University, on "Some Effects of Sodium Benzoate." A resolution was then introduced to place the Association

on record as endorsing the findings of the Referee Board. The discussion that followed developed the fact that two sides were sharply drawn on this issue, the resolution barely passing by the preponderance of one state, the vote being 57 to 42.

On Thursday afternoon the following papers were read: The Chemistry of Bleached Flour, by Dr. H. W. Wiley, Chief Bureau of Chemistry, Chairman Food and Drug Inspection Board, U. S. Department of Agriculture, Washington, D. C.

The Need of State Laws to Protect the Consumers' Meat Supply, by Miss Alice Lakey, Chairman Food Committee, National Consumers' League, Cranford, N. J., read by Mrs. Florence Kelley, Executive Secretary, National Consumers' League, New York.

Organization and Work of the U. S. Branch Food Laboratories, by Dr. W. D. Bigelow, Chief of Division of Foods, Bureau of Chemistry, Department of Agriculture, Washington, D. C.

An address was also made by Wm. Hudson, of Michigan, representative of the National Wholesale Grocers of the U. S.

On Friday morning papers were presented as follows:

Soda Fountain Syrups and Crushed Fruits without Preservatives, by W. P. Anderson, Secretary, J. Hungerform Smith Co., Rochester, N. Y.

The Preparation of Condiments without Preservatives, by Chas. F. Louden, President, the Louden Packing Co., Terre Haute, Ind.

The Advance toward Higher Food Standards, Some Helps and Some Hindrances, by L. S. Dow, of H. J. Heinz Co., Pittsburg, Pa.

Some Important Facts Worthy of Consideration, by A. F. Merrell, President the Oyster Growers and Dealers' Association of North America, New York.

By resolution adopted on a previous day, the discussion on the controversial whiskey question, due for Friday's session, was omitted.

Friday afternoon was devoted almost entirely to the election of the following officers:

George L. Flanders, N. Y., President.

L. P. Brown, Tenn., 1st Vice-president.

D. Harvey Dillon, La., 2nd Vice-president.

Andrew French, Minn., 3rd Vice-president.

W. M. Allen, N. C., Secretary.

James Foust, Penn., Treasurer.

EXECUTIVE COMMITTEE.

C. D. Woods, Me,

S. H. Crumbine, Kas.

A. N. Cook, S. D.

New Orleans was selected as the place of the next meeting.

The social features of the convention were most enjoyable. Besides the all-day complimentary mountain trip to Mt. McClellan already spoken of, at which, by the way, eatables, drinkables and smokables were liberally provided during the day, a sunset banquet was tendered delegates and guests on Thursday evening, at Lakeside, and an automobile trip around the city on Friday morning.

## ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

The twenty-sixth annual convention of the Association of Official Agricultural Chemists met at the Brown Palace Hotel, Denver, August 26–28, 1909. Seventy-five members and guests registered during the convention, which met at virtually the same time as the Association of State and National Food and Dairy Departments and in adjacent rooms of the same hotel. The main order of business was as "follows:

## THURSDAY, AUGUST 26.

MORNING SESSION.

Phosphoric Acid, W. F. Hand, Mississippi.

Nitrogen, C. H. Jones, Vermont.

Potash, B. B. Ross, Alabama.

Soils, S. D. Averitt, Kentucky.

Inorganic Plant Constituents, F. W. Robison, Mich.

Additional papers prepared by Messrs. Trescot, Fraps, Stevens and Withers were presented.

#### AFTERNOON SESSION.

Appointment of committees (constitution, resolutions, etc.).

Insecticides, C. C. McDonnell, Washington, D. C.

Water, J. K. Haywood, Washington, D. C.

Committee A on Recommendations of Referees, J. K. Haywood, Washington, D. C.

Reports of committees:

On Unification of Terms, R. J. Davidson, Blacksburg, Va.

On Fertilizer Legislation, H. W. Wiley, Washington, D. C.

On Testing Chemical Reagents, L. F. Kebler, Washington, D. C.

On Revision of Methods, J. K. Haywood, Washington, D. C.

FRIDAY, AUGUST 27.

MORNING SESSION.

Food Adulteration, H. E. Barnard, Indiana.

Colors, H. M. Loomis, Washington.

Saccharine Products, C. D. Howard, New Hampshire.

Fruit Products, C. B. Cochran, Pennsylvania.

Wine, Julius Hortvet, Minnesota.

Beer, H. E. Barnard, Indiana.

Distilled Liquors, L. M. Tolman, Washington, D. C.

Vinegar, R. W. Balcom, Tennessee.

Flavoring Extracts, E. M. Chace, Washington, D. C.

Spices, A. F. Seeker, New York.

Baking Powders and Baking Chemicals, E. C. Clark, Massachusetts.

Meat and Fish, F. C. Weber, Washington, D. C.

Fats and Oils, T. J. Bryan, Illinois.

Dairy Products, H. C. Lythgoe, Massachusetts.

President's Address (special order for 12 o'clock), W. D. Bigelow, Washington, D. C.

In addition to the regular program an address was presented by Hon. James Wilson, Secretary of Agriculture, and papers were read by Messrs. Barnard, Ross, Horne and Smith.

AFTERNOON SESSION.

Cereal Products, E. F. Ladd, North Dakota,

Vegetables, W. L. Dubois, New York.

Condiments Other Than Spices, H. E. Bishop, Indiana. Cocoa and Cocoa Products; Tea and Coffee, A. G. Woodman, Massachusetts. Preservatives, P. B. Dunbar, Washington, D. C.

Water in Foods, P. F. Trowbridge, Missouri.

Separation of Nitrogenous Bodies: Meat Proteids, P. F. Trowbridge, Missouri.

Milk and Cheese Proteids, G. E. Patrick, Washington, D. C. Vegetable Proteids, R. Harcourt, Canada.

Committee C on Recommendations of Referees, A. L. Winton, Illinois.

Committee on Unification of Methods of Analysis of Fats and Oils, L. M. Tolman, Washington, D. C.

Committee on Standardization of Alcohol Tables, L. M. Tolman, Washington, D. C.

Papers were presented at this session by Messrs. Smith and Hiltner,

SATURDAY, AUGUST 28.

MORNING SESSION.

Dairy Products, J. M. Bartlett, Maine.

Foods and Feeding Stuffs, J. P. Street, Connecticut.

Sugar, Chemical Methods, A. H. Bryan, Washington, D. C. Molasses Methods, A. P. Agee, Louisiana.

Committee B on Recommendations of Referees, F. W. Woll, Wisconsin.

· Special Committees (resolutions, constitution, etc.).

Tannin, F. P. Veitch, Washington, D. C.

AFTERNOON SESSION.

Drugs and Medicinal Plants, L. F. Kebler, Washington, D. C.

Medicinal Plants, C. H. LaWall, Pennsylvania; H. H. Rusby, New York.

The officers for the coming year are as follows:

EXECUTIVE COMMITTEE, 1909-1910.

W. A. Withers, Raleigh, N. C., President.

F. W. Woll, Madison, Wis., Vice-President.

H. W. Wiley, Washington, D. C., Secretary.

ADDITIONAL MEMBERS.

James M. Bartlett, Orono, Me. J. T. Willard, Manhattan, Kans.

The members of the Association enjoyed the same social features that were tendered to the Association of State and National Food and Dairy Departments, and in addition a complimentary smoke talk was held by the Western Association of Technical Chemists and Metallurgists in their honor.

The second International Congress for the Suppression of Fraud will be held under the auspices of the White Cross Society of Geneva, at Paris, during October, 1909. An elaborate program has been prepared which covers the following subjects: Food Stuffs, Chemical Products, Pharmaceuticals, Essential Oils and Aromatics, and Mineral Waters.

The Second International Road Congress will be held at Brussels, July 31 to August 6, 1910. The first congress was held in Paris during 1908. ALBERT E. LEACH.

## RECENT PATENTS.

The following patents relating to industrial engineering chemistry are reported by C. L. Parker, solicitor of **che**mical patents, McGill Building, 908 G Street, N. W., Washington, D. C.: 926,352. Method of Purifying Coal-Gas and Collecting By-Products. THOMAS MCWATTERS, Hawkins Station, Pa. June 29, 1909.

The process consists in purifying coal gas and collecting in the by-products thereof, passing the gas at a temperature of 300° Fahrenheit or thereabout, through a washer and thereby reducing the temperature of the gas to a temperature sufficient to eliminate substantially all the tar from the gas, then lowering to and maintaining the gas at a temperature sufficient to prevent the ammonia present assuming gaseous form, and finally by a further reduction in temperature eliminate the lighter tar and the liquor containing the remaining ammonia.

926,413. Process for Oxidizing Atmospheric Nitrogen by Electricity. DEMETRIO HELBRIG, Rome, Italy. June 29, 1909.

This is a process of oxidizing atmospheric nitrogen by electricity in which compressed air is projected by two opposite nozzles arranged in line, against an arc produced between suitable electrodes, whereby two sheaves of flame are produced which spread out along the electrodes in a plane normal to the direction of the nozzles.

925,988. Process of Hardening Tantalum. WERNER VON BOLTON, Charlottenburg, Germany, a corporation of Germany. June 22, 1909.

The process consists in adding to tantalum 3 per cent. of silicon.

926,729. Manufacture of Producer Gas. WILLIAM J. CROSSLEY, Openshaw, Manchester, and THOMAS RIGBY, Fairfield, Manchester, England, July 6, 1909.

This is a process of manufacturing producer gas by subjecting the fuel to a primary combustion while being mechanically agitated, continuously and automatically passing the resultant coke to the gas producer proper and passing the primary products of combustion through the coke in the gas producer.

926,820. Refractory Material and Process for Producing the Same. CHARLES L. NORTON, Manchester, Mass., July 6, 1909.

The process consists in roasting a mixture of magnesium carbonate and a silicious sand until the carbonate is thoroughly calcined, mixing the calcined magnesium and sand with asbestiform fiber and hydraulic cement, adding water to the aforesaid mixture of solids in quantities sufficient to form a wet pulp, and then subjecting the pulp to pressure while hydration of the cementitious materials is in progress, expressing the surplus water and compacting the entire mass.

## 926,980. Process of Making Alloys. JOHN T. H. DEMPS-TER, Schenectady, N. Y. July 6, 1909.

The process consists in melting a quantity of nickel with a decarbonizing agent in a clay lined crucible to reduce the carbon, adding iron and chromium in suitable proportions, then adding manganese and pouring the mixture before the manganese attacks the clay lining.

926,999. Process of Producing Digestible Substances from Keratin. CARL. NEUBERG, Charlottenburg, Germany. July 6, 1909.

This is a process for converting keratin or keratin sub-

stances into digestible albumoses and peptones by subjecting keratin substances to the action of mineral acids for a time and at a temperature to transform the keratin until a diluted sample of the reaction liquid to which a precipitant for albumoses has been added after filtration shows no amino-acid nitrogen, whereupon the formed albumoses and peptones are isolated.

## 927,021. Process of Treating Arsenical Ores and Speiss. ANSON G. BETTS, Troy, N. Y. July 6, 1909.

This is a process of treating speiss by dissolving it as anode in a solution of a salt of a metal depositable more readily than hydrogen, while depositing said metal on the cathode and utilizing the full cathode effect of the current in metal deposition, and recovering metal of the speiss from the solution.

## 927,046. Method of Treating Ores. HASCAL A. HOGEL, New York, N. Y. July 6, 1909.

This process is designed for the treatment of refractory sulfid ores, more especially those containing sulfids of iron, lead, silver, and gold. The process depends upon the action of heat, sulfur, oxygen and salt. The process consists in subjecting the ores to the action of heat in the presence of air, mixing sulfur therewith (unless there is enough sulfur in the ore), mixing salt therewith, heating the mixture in the presence of air to a degree sufficient to volatilize the metals or other compounds which it is desired to recover



passing the volatilized products into a condenser containing a liquid, intimately mixing said volatile products with said liquid and with air, causing the liquid to repeatedly pass through the condenser, allowing the excess of air to escape from said liquid during its passage, cooling the liquid at one period of its passage through a part of said condenser, collecting the solid products deposited in said liquid, and treating the liquid and solid products to obtain the metals, lead, copper, silver, and gold in the usual way.

927,135. Process of Making Acetate of Lime. ROBERT W: ERWIN, St. Louis, Mo. July 6, 1909.

This is a process of making acetate of lime by evaporating a solution thereof in vacuum until the liquid reaches a specific gravity of substantially 1.08, continuing said evaporation in another vessel until the specific gravity of the liquid reaches substantially 1.12, further evaporating the liquid in an independent vessel until the specific gravity thereof reaches substantially 1.16 or at the point of crystallization, and subsequently drying said crystals in vacuum.

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927,198. Process for the Production of a Leather Substitute. RUDOLF WEEBER, Vienna, Austria-Hungary. July 6, 1909.

This is a process for producing a leather substitute by adding fibrous material to a solution of balata, caoutchouc and gutta percha, precipitating the agglutinant on to the fibrous material, separating the fibrous material from the solution, adding an oil to make the fibrous material soft and flexible, and forming said material into a sheet.

927,292. Method of Uniting or Welding Metals. WIL-LIAM S. SIMPSON, London, England. July 6, 1909.

This is a method of welding metals fusible at different temperatures, including iron or steel and copper, by subjecting the metals to heat at least equal to the fusing temperature of the metal that is fusible at the lower temperature and simultaneously subjecting the same to a carbonaceous material.

927,342. Process of Recovering Sulfur. WALTHER FELD, Zehlendorf, and ANTON JAHL, Honningen-on-the-Rhine, Germany. July 6, 1909.

This is a process for obtaining sulfur from sulphur dioxid and sulfuretted hydrogen by passing these gases through liquid having the power of dissolving the sulfur as it separates.

927,374. Process of Manufacturing Incandescent Mantles. ARTHUR MULLER, Furstenwalde-on-the-Spree, Germany. July 6, 1909.

The mantles are manufactured by impregnating the fabric of the latter with a colloidal solution of an oxid-hydrate of a rare earth.

927,418. Process of Manufacturing Gas from Wood. BURDETT LOOMIS, Hartford, Conn. July 6, 1909.

This is a process of making fixed producer gas from wood by placing in the base of a cupola a regulated body of hard carbon, then igniting and burning a body of wood on top of the hard carbon, drawing the resulting flame down through the hard carbon, and heating it to incandescence, feeding wood as required and drawing air down into the body of wood and drawing the resulting flame, hot gas and vapors down through said body of incandescent hard carbon, thereby converting the vapors and carbonic acid into fixed combustible gas.

927,457. Process of Bleaching. FRANZ FUHRMANN, Berlin, Germany. July 6, 1909.

The process consists in heating the goods to be bleached with a liquor containing magnesium perborate in presence of alkali salts of fatty acids.

927,495. Art of Producing from Foundry Iron a Product Having Cutting and Other Properties of High-grade Tool and High-speed Steel. EDGAR A. CUSTER, Philadelphia, Pa. July 13, 1909.

This is a process of producing tool and other metal from foundry iron, by chilling molten foundry iron in a permanent metal mold swiftly to the point of setting in a solid structure and thereafter chilling quickly in a fluid bath. 927,644. Process of Producing Oxids. HENRY L. DOHERTY. New York, N. Y. July 13, 1909.

The process consists in oxidizing moist finely divided iron by a current of air until the observed temperature begins to rise, replacing the air by a current of air diluted to carry less than the normal amount of oxygen, the dilution being directly as such observed tendency to rise in temperature and finally finishing by a current of air as temperatures begin to sink.

927,678. Preparation of Iron or Steel for Casting. PAUL RAKOWICZ, Griesheim, Germany. July 13, 1909.

This is a method of alloying iron or steel with magnesium by first passing iron or steel from the solid into a semifluid or thickly liquid state, then introducing magnesium into the thick mass.

927,758. Process of Treating Silicates of Aluminium for the Purpose of Obtaining Valuable Products. ALF SIND-ING-LARSEN, Christiania, Norway. July 13, 1909.

The process consists in feeding material containing aluminium silicate to one end of a long electric furnace and a mixture of iron and a reducing agent to the other end thereof, and passing an electric current between the electrodes extending into the furnace.

927,822. Manufacture of Lithographic Stone Artificially. THOMAS M. THOM, Woodlands, Cheshunt, England. July 13, 1909.

This is a process of manufacturing lithographic stone by powdering slag, mixing it with lime, molding the mixture into slabs and submitting the molded slabs to the action of carbonic acid gas gradually admitted thereto until the entire mass is hardened.

927,843. Process of Making Alloys of Zinc with Copper. STERLING ELLIOTT, Newton, Mass. July 13, 1909.

The process consists in producing an alloy of zinc with copper by first melting the zinc, then adding the copper to the molten zinc and raising the temperature to the melting point of the alloy which is to be produced, or thereabout, which is less than the melting point of the copper and also less than the boiling point of the zinc.

927,916. Process of Recovering Volatile Metals and Metalloids from Lead and Copper Slags. WILHELM WITTER, Hamburg, Germany. July 13, 1909.

The process consists in bringing the products or ores, when in a molten liquid state, into close contact with incandescent carbonaceous matter in the presence of an air current, consuming the carbonaceous matter for reducing the metals and metalloids to be recovered, and for maintaining the reduction and volatilization temperature, reoxidizing the reduced volatile metals and metalloids, and causing the so-formed oxids to escape with the current of combustion gases.

927,950. Process of Manufacturing Wood Pulp. CHARLES B. CLARK, Bangor, Me. July 13, 1909.

This is a process of digestively treating wood by the "sulfite process," so called, in a bath of digesting liquor contained in a digester to form wood pulp, by removing from the digester during the period of digestion an appreciable quantity of the bath containing resinous matters and extractives of the wood for the purpose of recovering an appreciable portion of the bath after the passing of the resinous matters and extractives into it upon a resolution of the wood, and afterward continuing the process of digestion in the liquor left in the digester.

928,103. Process of Manufacturing Gas. B. BRAZELLE, St. Louis, Mo. July 13, 1909.

This is a process of manufacturing both fuel gas and illuminating gas by alternately admitting and cutting off a supply of air to a bed of hot coal, passing the products of combustion of such coal through a separate mass of mineral carbonates and thence through a second mass of coal, and thence through a mass of refractory material, and collecting



the fuel gas thus produced, and in passing the gas, which distils from the hot coal while the air supply is cut off through the mass of mineral carbonates and the second mass of coal, and then injecting volatile hydrocarbon into it and separately collecting the illuminating gas thus produced. The accompanying illustration shows apparatus in which the process is carried out.

928,235. Process of Making Patent-Leather or Artificial Patent-Leather. GEORGES R. DE MONTLORD, Boston, Mass. July 13, 1909.

The process consists in preparing a solution of nitrocellulose, a solvent, a vegetable oil, alcohol glycerin, spirits of turpentine, and a suitable pigment or dyestuff, immersing the leather or fabric into ethyl acetate, coating the surface of a glass-plate with one or more layers of the solution, uniting the leather or fabric with the coatings on the glassplate by pressure, then drying the leather or fabric while on the glass-plate, and finally removing the coated leather or fabric from the glass-plate.

# 928,412. Method of Treating Armor Plates. SAMUEL S. WALES, Munhall, Pa. July 20, 1909.

The armor plate is produced by heating that face of the plate which is to be hardened to a hardening temperature, simultaneously artificially cooling the back of the plate, periodically interrupting the cooling action or effect, and controlling the length of each period of interruption.

928,476. Process of Manufacturing Silicon Nitrid. ALF SINDING-LARSEN, Christiania, Norway. July 20, 1909.

The process consists in producing vapors of silicon and causing the same to be acted upon by gaseous nitrogen in heated condition. 928,512. Process of and Apparatus for Burning Cement and Like Materials. BYRON E. ELDRED, Bronxville, N. Y. July 20, 1909.

The process consists in mixing air with products of combustion and combustible gas in controlled proportions and causing the mixture to undergo combustion in proximity to pulverulent calcareous materials contained in a hot walled zone,

928,545. Process of Making Nitric Acid. FRIEDRICH L. SCHMIDT, Charlottenburg, Germany. July 20, 1909.

The process consists by causing sulfuric acid in molecular proportions to react on nitrate of calcium and separating the nitric acid thus obtained from the sulfate of calcium by filtration and afterward washing the sulfate of calcium with dilute nitric acid and water.

928,551. Method of Refining Iron. PAUL SEJOURNET, Paris, France. July 20, 1909.

The process consists in cooling low-carbon steel from a molten condition until it is at least in part solidified and the carbon therein has been oxidized by the contained oxygen and the oxid or oxids of carbon driven off, and remelting in the absence of carburizing conditions.

928,580. Process for Producing Incandescent Mantles. GEORG BUHLMANN, Gross-Lichterfelde, near Berlin. July 20, 1909.

This is a process for the impregnation of incandescent mantles consisting in dissolving a salt of an illuminating rare earth metal, adding to said solution a substance capable of reducing the acidity of said solution, shaking said solution in order to redissolve the precipitate, repeating the addition of the substance for reducing the acidity and shaking until the precipitate formed can no longer be dissolved and dipping the mantle fabric in the impregnating bath so formed.

298,734. Process for the Electrolysis of Alkali Chlorids. JEAN BILLITZER, Vienna, Austria-Hungary. July 20, 1909.

This is a process for electrolysis of alkali chlorids, by compelling a part of the cathode product to form a thin layer of the electrolyte and the constant removal of said layer as it is formed.

928,844. Manufacture, Concentration, and Simultaneous Purification of Sulfuric Acid. GASTON C. DE BRIAILLES, Paris, France. July 20, 1909.

This is a process of manufacturing sulphuric acid from sulphurous acid by electrolysis of a solution of sulphurous acid by keeping the solution at substantially the same specific gravity as the solution of sulphurous acid by adding the requisite quantity of sulphuric acid.

928,974. Process of Obtaining By-Products from Gases. GUSTAV HILGENSTOCK, Dahlhausen-on-the-Ruhr, Germany. July 27, 1909.

This is a process for obtaining by-products from gases of dry distillation of coal and other materials by dividing the first quantities of gases from the retorts or coke ovens, containing the greater part of the tar yield from the latter quantities of the gas from the retorts or coke ovens, containing relative little tar, removing the tar from the said first quantities of gas and then combining the substantially tar-free gas so produced with the said latter quantities of the gas.

929,131. Prepared Carbid of Calcium. ZAVIER HERME. Fruitvale, California. July 27, 1909.

This product is produced by mixing one part of sugar and one hundred parts of carbid of calcium; then successively mixing with said substances one-fourth of one part of petroleum, one part of sugar, four and one-half parts of glucose, and two and one-half parts of sugar.

929,152. Method of Making Cement, Mortar, and Concrete Water-tight. PAUL MECKE, Stettin, Germany. July 27, 1909.

The method consists in adding to the cement or lime mixture a pulp produced by the transposition of basic oleate of lime with sulfate of aluminium.

929,219. Method of Treating Aluminous Materials. ALDUS C. HIGGINS, Worcester, Mass. July 27, 1909.

The method consists in fusing an aluminous material in an electric furnace at a temperature substantially above the state of quiet fusion and sufficient to prevent substantial segregation of impurities, and recovering the resulting product in solid form.

929,276. Electrolytic Diaphragm and Method of Making the Same. Anson G. BETTS, Troy, N. Y. July 27, 1909.

This is a method of preparing asbestos board for use as a diaphragm in aqueous electrolysis, by cementing together the fibers of the board by means of an easily fusible substance insoluble in the solution in which the diaphragm is to be used.

929,383. Process of the Manufacture of a Disinfectant. ALEXANDER BRICK, Vienna, Austria-Hungary. July 27, 1909.

The process consists in mixing a soap solution containing alcohol with ethereal oils, adding formaldehyde to this mixture, heating this mixture combined with formaldehyde, allowing this mixture to cool, and adding to the cooled mixture incompletely saponified oils containing alcohol.

929,517. Method of Treating Aluminum Ores. FRANK J. TONE, Niagara Falls, N. Y. July 27, 1909.

The process consists of making a mixture of aluminum silicate, emery and carbon, the amount of carbon being sufficient to reduce the silica and all compounds in the mixture except alumina, but insufficient to reduce the alumina, interposing this mixture as a resistance conductor between electrodes, passing current through the same sufficient to fuse the alumina and reduce the other constituents to metallic form and keeping the temperature of the charge below the reduction temperature of alumina,

## 929,518. Process of Treating Aluminum-Silicate Ores. FRANK J. TONE, Niagara Falls, N. Y. July 27, 1909.

The process consists in forming a charge of aluminum silicate, hase metalliferous material, and carbon, subjecting such charge to electrically developed heat, partially reducing the silica thereby, and alloying the reduced silicon with the reduced metalliferous material, then increasing the percentage of metalliferous material and subjecting the charge to sufficient electrically developed heat to fuse the alumina, reduce the silica and alloy the silicon with the reduced metalliferous material.

929,687. Clad Metal and Process of Producing the Same. JOHN F. MONNOT, New York, N. Y. August 3, 1909.

This is a process of producing clad metal objects comprising unlike metals weld-united, by casting molten bodies of unlike metals into contact with opposite surfaces of a separator of material capable of weld-uniting with both the metals so cast, and progressively cleaning one or both said surfaces by the action thereon of a deeper layer of cleansing material progressively displaced by the molten metals so cast.

929,726. Method of Obtaining Purified Ammonia Gas from Ammonia Liquors. CHARLES M. STINE, Chester, Pa. August 3, 1909.

The method consists in forcing a current of air through ammoniacal liquor, and gradually increasing the temperature of the same to a point at, or above, the boiling point of water.

929,845. Art of Improving the Odor of Vegetable Oils. JOHN H. FILBERT, Baltimore, Md. August 3, 1909.

<sup>4</sup> This is a process of improving the odor of cottonseed oil, by mixing with the oil fresh animal crackling so as to imbue the oil with the essence, odor or flavor residing in the crackling, then separating the oil and crackling.

929,861. Process for Obtaining Starch and Gluten from Wheat Flour. FRIEDRICH A. V. KLOPFER, Leubnitz, near Dresden, Germany. August 3, 1909.

This is a process for separating large-grained and valuable starch from the albumen pulp containing the small-grained starch, the entire albumen, the soluble carbohydrates, and the soluble salts, by adding to the water while stirring it, together with the wheat flour to be centrifuged, chlorid of sodium in such small quantities up to one per cent. so that the gluten cellular texture is not dissolved, but only its structure loosened, whereby, after the mixture is finally treated in a separator, the gluten, the starch and a wheat flour extract containing the extractive wheat flour substances soluble in water are obtained without destroying the lecithin bound to the albumen and the ferments of the flour.

929,925. Art of Improving the Odor of Lard Substitutes. JOHN H. FILBERT, Baltimore, Md. August 3, 1909.

The process consists in improving lard substitutes containing a stiffening fat and vegetable oil by imbuing the same with the essence of heated animal tissue,

929,962. Manufacture of Lead Arsenate. ELLERSLIE E. LUTHER and WILLIAM H. VOLCK, Watsonville, California. August 3, 1909.

The process consists in heating white arsenic in contact with a suitable lead compound, and continuing the heating and roasting the combined material in the presence of oxygen.

### 930,028. Method of Reducing Ores. FREDERICK M. BECKET, Niagara Falls, N. Y. August 3, 1909.

This is a method of reducing ores of refractory metals, by smelting a charge containing such ore and a borid, while maintaining therein a temperature sufficient to secure substantial elimination of the boron from the reduced product.

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930,057. Process for Making White Lead. JOHN A. ERBS-LOW, Los Angeles, Calif. August 3, 1909.

The process consists in causing lead in divided condition to pass alternately into a body of water and of gas containing carbon dioxid and oxygen, to cause corrosion and attrition of the lead and to form a foam containing white lead, drawing off said foam and drying the foam to obtain white lead.

930,274. Method of Treating Wood during Distillation. HOWARD W. DOUGHTY, Amherst, Mass. August 3, 1909.

The process consists in hydrolyzing the cellulose with sulfuric acid and steam under pressure and driving off the volatile oils by the same heat, treating the solid residue with sodium hydroxid and after filtering bringing the acid and alkali solutions together in equivalent proportions, whereby the sulfuric acid is neutralized and the resinous acids are precipitated, then filtering, fermenting the filtrate and distilling off the alcohol.

930,344. Process of Treating Titaniferous Iron Ores. WIL-HELM BORCHERS, Aix-la-Chapelle, Germany. August 10, 1909.

The process of treating titaniferous iron ores, consisting in initially smelting such iron ores in presence of carbon directly, drawing off the crude iron, reducing the slag produced during the smelting operation by carbon to a reducing agent, and utilizing said reducing agent for reducing subsequent charges of titaniferous iron ores.

930,376. Method of Treating Aluminous Materials. ALDUS C. HIGGINS, Worcester, Mass. August 10, 1909.

This is a method of treating artificially prepared aluminous materials by subjecting such materials, suitably subdivided, to heat under oxidizing conditions, and thereby oxidizing impurities therein.

930,442. Method of Making Acids. JOHN L. TUFTS, Boston, Mass. August 10, 1909.

This is a continuous method of producing volatile acids by commingling a salt of such acid and an acid body capable of decomposing the same, working the mixture under the influence of heat until portions of the same become solid, continuously displacing such solid portions from the remaining pasty portions, continuing the working of said pasty portions and subjecting the displaced solid portions to the action of heat.

930,471. Process of Manufacturing Sulfuric Acid. WIL-LIAM HALLOCK, New York, N. Y. August 10, 1909.

The method consists in subjecting sulfurous oxid to the influence of an ionizing agent in the presence of oxygen.

930,575. Process Relating to Refractory Substances. ROB-ERT TRIPMACHER, Schweidnitz, Germany, August 10, 1909.

This is a process of binding refractory substances by treating the same in pulverized condition with a liquid which consists in a saccharine solution, and a boron and a chromium compound, in which naphtha is emulsified.

930,666. Process of Treating Ores. PAUL L. T. HEROULT. La Proz, France. August 10, 1909.

This is a method of treating copper ore containing sulfur

which consists in feeding it into an electric furnace and passing it gradually through a zone of fusion in said furnace, and simultaneously injecting oxygen into the ore above the zone of fusion to oxidize part of the sulfur.

930,697. Waterproof Cement and Method of Making the Same. JACOB F. SCHOELLKOPF, Buffalo, N. Y. August 10, 1909.

The cement comprises a mineral cement and an insoluble resin salt of lime uncombined with material foreign to the base of said salt.

930,723. Process of Forming Electric-incandescent Lamp Filaments. WERNER VON BOLTON, Charlottenburg, and FELIX HARTMANN, Berlin, Germany. August 10, 1909.

The process consists in fitting into a tube of ductile metal a tube of a different, very ductile metal, inserting tungsten powder into the latter tube, subsequently working the tubes and the tungsten powder into filamentary form, removing the outer tube, and subsequently removing the inner tube from around the tungsten metal.

930,764. Method of Treating Iron Ore. JOHN T. JONES, Iron Mountain, Mich. August 10, 1909.

The method consists in mixing carbonaceous material therewith, then advancing the mixture through a highly heated non-oxidizing atmosphere, to consume the carbonaceous material and support its combustion with oxygen from the ore, thus reducing the ore, then advancing the reduced mass through a still more highly heated non-oxidizing atmosphere to fix the metal constituent without melting the slag-making constituents.

930,801. Method of Manufacturin, a Porous Material. HERMANN SENN, Szczakowa, and DAVID KLUGER, Trzebinia, Austria-Hungary. August 10, 1909.

This is a method of manufacturing a porous material which consists in calcining slate containing small quantities of organic substances whereby the material swells and assumes a cellular structure and becomes suitable for use as an insulating and filtering medium.

930,815. Electrolyte. Abraham Van Winkle, Newark, N. J. August 10, 1909.

This is a zinc electrolyte containing fluosilicate of zinc, and a salt of aluminum.

930,874. Cellulose Substitute. OSCAR MULLER, Cologne, Germany. August 10, 1909.

The material consists of cottonseed shells from which the binding materials, dye substances and cotton fibers have been eliminated.

930,900. Process of Making Extracts. WILHELM WIEG-AND, Mersburg, Germany. August 10, 1909.

This is a process for drawing extractive matter from different materials such as tannic material, dye-wood, beet and like by advancing the solid material in a horizontal direction from one end of a horizontal elongated receptacle and the liquor from the other end, permitting the liquor to enter said receptacle in such a manner that the solid material will always stand at a higher level than the liquor, and stirring the material in a vertical direction while feeding it in a horizontal direction, whereby those parts which first lie beyond the level of the liquor are dipped into it, and other parts are raised out, no mixing of the strata of liquor of different degrees of concentration being effected thereby.

930,968. Process of Obtaining Pure Metallic Copper from a Solution of a Salt of Copper. LUCIEN JUMAU, Paris, France. August 10, 1909.

The process consists in obtaining pure copper from a solution containing salts of copper by subjecting said solution to heat and pressure in the presence of a suitable quantity of wood used in any form, such as sawdust, shavings, waste wood and the like.

930,976. Composition of Matter. SAMUEL A. LEAVITT, Gorham, N. H. August 10, 1909.

This is an expansile acid-proof cement for lining digesters or the like, consisting of litharge, quartz, calcined plaster and lampblack, all brought to a plastic condition by the admixture of glycerin.

931,015. Insulating Composition. MINDELIN MCGERRY, Kalamazoo, Mich. August 10, 1909.

The composition consists of asbestos pulp, slippery elm, a vegetable acid, glue and starch dissolved in water and cooked to a paste.

## OFFICIAL REGULATIONS AND RULINGS.

## FOOD INSPECTION DECISION 109.

#### THE LABELING OF WINES.

On June 30, 1909, a hearing was held before the Secretary of Agriculture and the Board of Food and Drug Inspection on the labeling of Ohio and Missouri wines. After giving full consideration to the data submitted, the board is of the opinion that the term "wine" without modification is an appropriate name solely for the product made from the normal alcoholic fermentation of the juice of sound ripe grapes, without addition or abstraction, either prior or subsequent to fermentation, except as such may occur in the usual cellar treatment for clarifying and aging. The addition of water or sugar, or both, to the must prior to fermentation is considered improper, and a product so treated should not be called "wine" without further characterizing it. A fermented beverage prepared from grape must by addition of sugar would properly be called a "sugar wine," or the product may be labeled in such a fashion as to clearly indicate that it is not made from the untreated grape must, but with the addition of sugar. The consumer is, under the Food and Drugs Act, entitled to know the character of the product he buys.

Evidence was offered on the preparation of "wine" from the marc. In these cases it appeared customary to add both water and sugar to the marc and sometimes to use saccharin, coloring matter, preservatives, etc., to make a salable article.

In the opinion of the Board no beverage can be made from the marc of grapes which is entitled to be called "wine" however further characterized, unless it be by the word "imitation." The words "Pomace Wine" are not satisfactory, since the product is not a wine in any sense, but only an "imitation wine" and should be so labeled.

> H. W. WILEY, F. L. DUNLAP, GEO. P. MCCABE, Board of Food and Drug Inspection.

Approved:

W. M. HAYS,

Acting Secretary of Agriculture. WASHINGTON, D. C., August 21, 1909.

## JUDGMENT NOS. 83-91 FOOD AND DRUGS ACT.

83. Misbranding of wine (fermented solution of commercial dextrose, artificially colored and preserved with benzoic acid).

84. Misbranding of baked beans and tomato sauce (underweight).

85. Misbranding of canned tomatoes (underweight).

86. Misbranding of a drug product (saltpetre).

87. Misbranding of evaporated apples (as to quality).

88. Adulteration of milk (added water).

89. Misbranding of evaporated apples (underweight).

· 90. Misbranding of canned peas (underweight).

91. Misbranding of lemon, raspberry and strawberry extracts.

#### (T. D. 29941.) Drawback on Aluminium Ingots.

TREASURY DEPARTMENT, August 6, 1909. SIR: The Department's regulations of November 10, 1906 (T. D. 27697), providing for the allowance of drawback on aluminium sheets, bars, plates, and tubing manufactured by the Pittsburg Reduction Company, of Pittsburg, Pa., wholly from imported aluminum in the pig, are hereby extended, so far as applicable, to cover aluminum ingots manufactured by the Northern Aluminum Company (Limited), of New Kensington, Pa., from imported crude aluminum, in accordance with the sworn statement dated April 3, 1909, transmitted herewith for filing in your office.

In liquidation, the quantity of imported crude aluminum which may be taken as the basis for the allowance of drawback may equal the quantity used as declared in the drawback entry, after official verification of exported quantities, provided it shall not exceed the net weight of the exported material with an addition thereto for worthless waste not to exceed 1 per cent. of such waste.

Respectfully,

JAMES B. REYNOLDS,

Assistant Secretary.

COLLECTOR OF CUSTOMS, Philadelphia, Pa.

(63567.)

## ERRATA.

July number, page 476, second column:

In the heading read; "The Determination of Zinc," instead of "The Determination on Zinc,"

Line 9, from bottom, page 476, read: "heated to faint redness," instead of "heated to redness."

Line 8 from bottom, page 476, read: "cooled," instead of "colored."

Footnote I, page 456, read: See Waring on precipitation from formic acid solution, instead of See Waring, "On Precipitation from Formic Acid Solution."