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## EDITORIALS.

### ADVANCE NEW ENGLAND CHEMISTRY.

ADVANCE NEW ENGLAND is a monthly magazine, published by the Boston Chamber of Commerce, devoted to the commerce, industry and public interests of Boston and New England, whose editor is Mr. Richard J. Walsh. Some of the best known New England chemists have seized upon the August, 1910, number of this journal and with bold hands have hoisted their chemical colors and proclaimed to all their readers what chemistry may do for New England. On the outside front cover appears a cut of the plant of the Badische Anilin- und Soda-Fabrik at Ludwigshafen-on-the-Rhine and we are told editorially that in the development of this plant New England should find an inspiration. "For when the Fatherland was bare of raw materials, German genius imported them from England, manufactured them at home and sent them back to England, killing that country's own aniline industry." New England is to a large extent devoid of raw materials for the chemical industries but she is thrifty. She is already largely dependent on outside sources of raw material for her mechanical manufacturers. In applied chemistry why may she not do what Germany or any country has done?

Alan A. Claflin contributes "The Relation of Chemistry to Industry" and "Extension of Chemical Industry in New England;" F. G. Stantial, "The Chemical Resources of New England;" S. W. Wilder, "Chemical Industry in New England To-day;" Arthur D. Little, "Chemistry and Dividends" and "Chemical Fakes;" William H. Walker, "Achievements of Some New England Chemists;" Henry P. Talbot, "New England's Share in Training Chemists." In addition to this admirable array of authors and papers are several excellent editorials, all on chemical subjects. This is a chemical number indeed and a mine of information for the layman in regard to the nature of the operations of modern applied chemistry. We hope the magazine will find a wide circle of readers and that other journals will follow the same idea and issue a chemical number now and again.

### CHEMICAL ENGINEERING EDUCATION.

THE report of the Committee of the American Institute of Chemical Engineers on Chemical Engineering Education,<sup>1</sup> written by the chairman of the committee, Dr. F. W. Frerichs, of St. Louis, is of more than usual interest and offers a number of suggestions for careful consideration. The difference between American and European conditions of manufacture are ably set forth in the report and the accompanying letters; also the differences in methods and results in European and American schools and universities. Always susceptible to criticism and with abundant critics, curricula can be progressively modified only after the most careful consideration of all the facts. A committee of this sort, however, speaks with authority. It is composed of university-trained men who have had the benefit of long experience in the schools of business, manufacture, and the practising professions. Dr. Frerichs himself is a type of the scholarly chemical manufacturer. The report is worth the careful study of both educators and chemists in the industries.

## ORIGINAL PAPERS.

### ALLOYS OF NICKEL AND COBALT WITH CHROMIUM.

By ELWOOD HAYNES.

Received August 12, 1910.

The metals nickel and cobalt have always possessed a peculiar interest for the chemist. Like nearly all

<sup>1</sup> See page 430 of this issue.

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of the more recently discovered metals, their compounds were known before the metals themselves were discovered. Indeed, it has been known for centuries that certain substances were capable of giving a blue color to glass, and there is but little doubt that this peculiar power was due to some crude compound of cobalt.

About two centuries ago, the ores of cobalt and nickel were encountered in the mining of copper. It was at first supposed that they were ores of the latter metal, but the miners, after vainly striving to smelt the ore, and failing to obtain any copper from it, designated it as "kupfernicks," and from this expression the word "nickel" originated.

Attempts were also made to smelt the ores of cobalt, but as no useful metal resulted, they decided that the goblin, or "kobold," supposed to inhabit the mines, had placed a ban upon the ore, and thus rendered it incapable of producing valuable metal. From this designation, by the German miners, the name "cobalt" was derived.

It was not until 1751 that Cronsted published the results of an investigation which he had made upon an ore obtained from the mines of Helsingland. This ore yielded a brittle metal, and as it occurred most abundantly in "kupfernicks," he suggested for it the name of nickel.

A few years later, in 1776, it was discovered that nickel was evidently one of the constituents of a Chinese alloy, known as "packfong."

The use of nickel in German silver began about the year 1823. It was not, however, until 1857 that Messrs. Deville and Debray, the celebrated French chemists and metallurgists, prepared pure nickel by heating its oxalate in a lime crucible. From the pure metal thus obtained wires were made, which showed a tensile strength superior to that of wrought iron. The wires also showed considerable toughness, and when polished presented a bright, silvery appearance, and retained their luster for an indefinite period, under all ordinary atmospheric conditions.

A few years later the art of electroplating nickel was discovered and has since received a very wide application. Tons of the double sulphate of nickel and ammonium are used for this purpose every year.

Besides this latter important application of the metal, it is now used in large quantities for the manufacture of nickel steel, which has become a common substance in the making of naval guns, projectiles, armor plate, and high-class automobiles.

The history of cobalt is similar to that of nickel, excepting that the compounds of cobalt were used in the arts, instead of the metal itself. In fact, but little was known of the metal until 1857, when Deville produced the pure metal in practically the same manner that nickel was prepared. It was found

that cobalt was even stronger than nickel, possessing a tensile strength of about 65,000 pounds per square inch. Indeed, up to that time, it was the strongest pure metal yet discovered, and it still holds this position, with the possible exception of tantalum.

About the year 1895 I made a number of tentative experiments relating to the production of alloys of nickel with iron, chromium, and other metals. The fusions were made in small graphite crucibles, which were heated in a blast furnace of the Fletcher type, operated by natural gas. I had at that time the advantage of natural gas at a pressure of forty pounds or more per square inch. With a suitably arranged furnace of this character, temperatures ranging up to the fusing point of the most refractory Missouri fire clay were readily obtained. I succeeded in obtaining by this means alloys of nickel and chromium, which contained, however, a considerable amount of carbon and silicon. A small quantity of aluminum was sometimes added to the alloy in order to improve its quality. By this means I obtained an alloy of chromium, nickel, and aluminum, which was hard and brittle, but possessed fairly good color and luster. For this alloy a knife blade was formed, which showed fair cutting qualities and considerable resistance to atmospheric conditions. It was readily soluble in nitric acid and after long exposure to the atmosphere of a chemical laboratory it became tarnished, showing a greenish coating on its surface.

Later, I attempted to produce alloys of nickel and chromium with titanium, by means of an electric furnace, made from blocks of quicklime. This proved unsatisfactory, but I continued experimenting with the gas furnace and, finally, succeeded in producing an alloy of nickel and chromium entirely free from carbon by heating the pure mixed oxides of the two metals with powdered aluminum, in a crucible lined with pure oxide of aluminum. The reaction was so violent that most of the metal was thrown from the crucible. A few small pellets were saved, and these showed great malleability, flattening readily under the hammer, without cracking. The alloy possessed a fine color, and when polished exhibited a beautiful luster. Larger pellets were soon obtained, and it was found that when the chromium content much exceeded ten per cent. that the alloy showed remarkable resistance to chemical reagents, particularly to nitric acid.

At about the same time I reduced a mixture of the sesquioxides of cobalt and chromium with powdered aluminum and thus obtained minute pellets of an alloy of cobalt and chromium. The little particles thus produced were not much larger than pinheads. They were found to be remarkably hard and could scarcely be scratched by a file. A few of them were ground off on one side by means of carborundum and showed a brilliant luster. It occurred to me that



the metal might become serviceable for cutting instruments.

A short time after the above experiments were made I was called actively into the automobile business, and thus compelled to abandon further experiments along this line for some time.

In 1905 I repeated some of the former experiments, with a view to utilizing the alloys of cobalt and nickel with chromium for ignition points, in connection with gas engines. I was soon able to produce both the alloys of nickel and cobalt in considerable quantity. I ascertained that the nickel-chromium alloy could be worked cold, while the cobalt-chromium alloy must be worked hot in order to obtain any degree of satisfaction.

The first pellets of the cobalt-chromium alloy, weighing from fifteen to thirty grams, were obtained by heating mixtures of aluminum with the oxides of cobalt and chromium in crucibles lined with the oxide of aluminum. Some of these pellets were heated to redness and flattened out under the hammer, and while this could be done without cracking the alloy, the metal was found to be very hard even at red heat.

I soon found that it was impracticable to reduce the alloy in this manner in large quantities. I accordingly purchased a considerable amount of pure cobalt and pure chromium and had a mixture of nearly equal parts of these metals fused in an electric furnace, the metals being placed in a carbon crucible lined with pure magnesia. The alloy was cast into a small bar, about one-fourth of an inch square, and five or six inches long. This alloy exhibited most of the characteristics of that obtained by reduction with aluminum, but it could not be drawn to any extent under the hammer without cracking. Whether this was due to the high percentage of chromium, or to slight impurities in the metals employed, I am as yet unable to say.

In order to determine what the effect of alloying the cobalt with smaller percentage of chromium would be, I again had recourse to the gas furnace and succeeded in melting a mixture containing 75 per cent. cobalt and 25 per cent. chromium in a crucible made of a very refractory material, which I compounded for the purpose. Much to my satisfaction, I succeeded in melting this alloy to a perfect fluid and poured it into an ingot mold, which gave me a bar of metal about one-half an inch square and five or six inches in length. This metal proved to be very sonorous and elastic, and if some care were used it could be hammered out into a rough strip.

After a considerable amount of experimenting with various purifying agents I finally succeeded in producing a very tough and malleable alloy, which could be hammered out into the thinnest sheet at a bright red heat without showing any sign of cracking. A razor blade was made from a bar of this alloy,

and while it did not prove equal to the best steel for this purpose, it has been used hundreds of times for shaving purposes, and after a year and a half shows practically no signs of wear, though, of course, it has been necessary to strop it frequently in order to keep it in good condition. The bar was made about two years ago, and I am sure that I have since produced metal that would be much more satisfactory for the purpose.

A test of tensile strength and elastic limit of this material was made, with results as follows: Elastic limit, 79,000 lbs., tensile strength 96,000 lbs. Elongation, 3 per cent. It will be seen from the above that both the elastic limit and tensile strength of this material are superior to those of untreated steel, resembling more nearly those of good nickel steel. The elongation is quite low, but this is to be expected on account of the great hardness of the alloy, which is equal to that of mild tempered tool steel. A test was also made of the modulus of elasticity of this material, which was found to be about equal to that of steel. This is a very significant fact, since heretofore it has not been possible, so far as I am aware, to form an alloy of non-ferrous metals, which would show a modulus of elasticity comparable to that of iron and steel. And it is lack of this valuable property in various non-ferrous mixtures which renders them inferior to iron for many important purposes.

A pocket knife blade and several table knife blades were made from this material, and were found to be very satisfactory in every respect. One of these table knife blades has now been in use for more than two years in the kitchen, where it was used for all sorts of purposes, such as cutting bread, turning griddle cakes, peeling and paring vegetables, and for various other purposes such as are known only to the culinary art. After all this use and abuse, the knife shows not the slightest trace of tarnish, and has held its luster so well that when exposed to the sun it shows a reflection which dazzles the eyes.

By mixing the alloy with small quantities of other substances, its properties may be modified to a remarkable degree. By this means I have obtained alloys, or combinations, which, while very brittle, would readily scratch quartz crystal.

By reducing the quantity of chromium to some extent, and adding certain other materials, alloys which are practically proof against nitric acid can readily be obtained, which are sufficiently soft and malleable to be worked cold, having a hardness not much greater than that of mild, untempered steel.

Between these two extremes a great variety of combinations can be made which are suitable for use for various purposes. For example, I have produced an alloy of sufficient hardness that when it is formed into a small bar, say one-half an inch wide, one-fourth



an inch thick, and three inches long, and one of the ends shaped for a cold chisel, a 20-penny nail can be cut in two without marring the edge of the chisel in the slightest degree. I have formed another alloy into a small lathe tool, about one-fourth an inch square and three inches long, which showed cutting qualities comparable to high-speed tool steel. In fact, in some respects, especially under high speed and light cuts, it has stood the test for a long time, where high-speed steel failed almost instantly, on account of the intense heat generated. I wish it distinctly understood, however, that I do not recommend this material as yet for lathe tools, though it would have a high value for this purpose if it were not obliged to compete with alloy steels.

An alloy of 75 per cent. cobalt and 25 per cent. chromium, to which small quantities of other metals are added, is not only sufficiently hard for good edge tools, but is quite tough, and can be bent much beyond its elastic limit without cracking, resembling in this respect the alloy steels, but, generally speaking, it is much harder. A bar of the alloy, one-fourth inch square, can be bent at right angles without showing any signs of cracking.

*Chemical Properties.*—When the mixture of cobalt and chromium is heated to whiteness in a crucible, the cobalt first commences to fuse and immediately begins to combine with the chromium, and if the metals are mixed in the proportion of about three parts, by weight, of cobalt to one of chromium, a eutectic is formed which seems to possess a lower melting point than either cobalt or chromium.

The color of the alloy lies between that of steel and silver, and is especially pleasing in bright light. The alloy is also readily polished, but requires special treatment in order to develop its highest luster.

The most remarkable property of this combination, however, is its resistance to corrosion. It is equaled in this respect only by gold and the metal of the platinum group. It is attacked slowly by dilute hydrochloric acid and somewhat vigorously by the strong acid, especially when heated. Momentary exposure, however, to either dilute or strong hydrochloric acid has practically no effect on the metal. Both strong and dilute sulphuric acid attack it very slowly when cold, and not very rapidly even when heated. Nitric acid is totally without action upon it, and a polished piece of the alloy may be boiled in that substance for hours without affecting the luster of the metal in the slightest degree.

Solutions of the caustic alkalies are also totally without action upon it, even when boiled for hours. The alloy is likewise proof against all atmospheric influences, whether the air be moist or dry, and retains its brilliant luster for months, or even years, under severest conditions. Even sulphuretted hydro-

gen, when present in the atmosphere in large quantity, is totally without action upon it.

Its resistance to culinary operations has already been mentioned.

When the metal is heated in contact with the atmosphere it retains its color up to a temperature approaching a dull red, or about 500°, when it shows a faint straw color, which deepens as the temperature rises, passing through bronze-yellow, purple, blue, and finally terminating in blue-black.

The alloy shows no scale, even when heated to bright orange, and the film of oxides does not seem to increase in thickness after prolonged heating.

It can readily be melted in an open crucible in a gas furnace with practically no oxidation, so long as a slightly reducing flame is maintained. This is all the more remarkable on account of its high melting point, which seems to be about 1650 cc. for the 25 per cent. alloy. Indeed, the metal has been melted in this manner with a loss of less than one-half of one per cent.

*Uses.*—The uses for any substance may be limited in several particulars: first, by the limitations of its fitness; second, by the possibility of producing it in proper form; and third, by its cost. This material is particularly suitable for all kinds of small cutting instruments, since it takes an edge comparable to that of tempered steel. It is especially adaptable to the manufacture of pocket knives, on account of the beauty of its color, and the brilliancy of its luster, both of which remain permanent under all circumstances, thus giving the blades a particularly attractive appearance. Knives of this description may be used for cutting fruit without danger of marring their luster in the slightest degree.

Alloys in certain proportions will also doubtless find a wide use for surgical instruments, since they resist perfectly all sterilizing solutions.

The alloy is perhaps better adapted for table cutlery than anything that has ever yet been produced. We all know too well that a silver-plated knife, for example, is ill adapted for cutting meat, and it cannot be sharpened without destroying the plating. Steel knives, on the other hand, while they cut well, require endless labor to keep them in presentable condition, and at best, they are unsightly in appearance.

The alloy is also of considerable interest to the chemist and physicist. It is admirably adapted for the manufacture of fine weights for balances, scrapers, spatulas, and other laboratory appliances. To the physicist, it furnishes a material which is at once hard, lustrous and untarnishable, and hence well adapted for the manufacture of fine weights, measuring instruments, and various small tools.

The alloy is also particularly well adapted for the manufacture of standard weights and measures, such as the gram, kilogram, meter, etc., and it is



difficult to see in what respect it is inferior for this purpose to the expensive platinum-iridium alloy now in use.

The alloy could readily be made into laboratory vessels, cooking utensils, spoons, forks, etc., and it is limited in this respect only by its cost.

*Cost.*—Regarding the cost of production and manufacture, I am not at present prepared to make definite statements. I have succeeded, however, not only in obtaining the raw material at lower prices, but have also reduced the cost of production to a considerable degree, so that it is now possible to produce the alloys with as much despatch and precision as is possible in the production of common alloys.

KOKOMO, IND.

### THE DETERMINATION OF SUGAR LOST BY ENTRAINMENT FROM EVAPORATORS.

By R. S. NORRIS.

Received August 5, 1910.

Wherever evaporating apparatus that was constructed twenty years or more ago is in use in sugar factories, there is likely to be a continual loss, more or less, of sugar by entrainment. It was only about that time that the importance of having large vapor pipes and considerable space above the steam chest or tubes was beginning to be realized. Even recently constructed evaporators may also entrain at certain times. Some reliable method of determining how much sugar is being lost in this way is therefore of importance.

In a multiple effect evaporator the entrainment is most pronounced from the last body, on account of the greater volume of the vapor and the greater viscosity of the juice. And as whatever juice is carried

out with the vapor from this effect finds its way into the condenser water, it becomes necessary to determine the sugar in a very dilute solution and to know the weight of the solution, that is, of the condenser water. Various devices have also been used for collecting a sample of the vapor, leaving the last effect representing an aliquotic portion of it, and determining the sugar in this; but these are of doubtful value.

As no detailed description of a method for determining the sugar in the condenser water is known to the writer, he has thought it advisable to publish a description of a method used by him in testing a number of evaporators.

The apparatus necessary for the determination is a stove or heater of some kind that can evaporate a considerable volume of water in a short time, a large vessel for evaporating the water in a small evaporating dish holding about two hundred cubic centimeters, a piece of sheet asbestos about eight inches (20 cm.) square, a measuring cylinder or flask holding five hundred cubic centimeters or a liter, a centigrade thermometer, and the usual sugar laboratory outfit for polarizations.

Five liters of condenser water are collected, a liter at a time, at intervals of half an hour, as it leaves the condenser and as near to it as possible, and placed in a perfectly clean vessel—preferably one that has not had anything containing sugar in it before. The evaporation had best be begun as soon as the first sample is collected, a few drops of sodium carbonate solution being first added. When the water has been evaporated down to about 150 cc. it is transferred to the small evaporating dish, the last traces being washed out of the large dish into the smaller one.

WEIGHT OF CONDENSER WATER CORRESPONDING TO A UNIT WEIGHT OF WATER EVAPORATED FROM JUICE.

(For Single Effects, i. e., Vacuum Pans)  
Temperature of Water Coming Out of Condensers in ° C.

Temp. of water going into condenser in ° C.	30.	32.	34.	36.	38.	40.	42.	44.	46.	48.	50.	52.	54.	56.	58.	60.
5	24.7	22.8	21.3	19.9	18.7	17.6	16.7	15.8	15.0	14.3	13.7	13.1	12.6	12.1	11.6	11.2
6	25.6	23.7	22.0	20.0	19.2	18.1	17.1	16.2	15.4	14.7	14.0	13.4	12.8	12.3	11.8	11.4
7	26.7	24.6	22.8	21.2	19.8	18.6	17.6	16.6	15.8	15.0	14.3	13.7	13.1	12.5	12.0	11.6
8	27.9	25.6	23.6	21.9	20.4	19.2	18.0	17.0	16.1	15.3	14.6	13.9	13.3	12.8	12.3	11.8
9	29.2	26.8	24.5	22.7	21.1	19.8	18.6	17.5	16.6	15.7	14.9	14.2	13.6	13.0	12.5	12.0
10	30.6	27.8	25.5	23.5	21.8	20.4	19.1	18.0	17.0	16.1	15.3	14.6	13.9	13.3	12.7	12.2
11	32.1	29.1	26.5	24.4	22.6	21.0	19.7	18.5	17.4	16.5	15.7	14.9	14.2	13.6	13.0	12.5
12	33.9	30.5	27.7	25.4	23.4	21.8	20.3	19.0	17.9	16.9	16.0	15.2	14.5	13.9	13.3	12.7
13	35.8	32.0	29.0	26.5	24.3	22.5	21.0	19.6	18.4	17.4	16.4	15.6	14.8	14.2	13.5	12.9
14	38.0	33.7	30.4	27.6	25.3	23.4	21.7	20.2	19.0	17.9	16.9	16.0	15.2	14.5	13.8	13.2
15	40.4	35.7	31.9	28.9	26.4	24.2	22.5	20.9	19.6	18.4	17.3	16.4	15.6	14.8	14.1	13.5
16	43.2	37.8	33.6	30.3	27.5	25.2	23.3	21.6	20.2	18.9	17.8	16.8	15.9	15.1	14.4	13.8
17	46.5	40.3	35.6	31.8	28.8	26.3	24.2	22.4	20.8	19.5	18.3	17.3	16.3	15.5	14.7	14.1
18	50.3	43.1	37.8	33.5	30.2	27.4	25.1	23.2	21.6	20.1	18.9	17.7	16.8	15.9	15.1	14.4
19	54.8	46.3	40.2	35.4	31.7	28.7	26.2	24.1	22.3	20.8	19.4	18.3	17.2	16.3	15.4	14.7
20	60.1	50.1	43.0	37.6	33.4	30.1	27.3	25.1	23.1	21.5	20.0	18.8	17.7	16.7	15.8	15.0
21	66.7	54.6	46.2	40.0	35.3	31.6	28.6	26.1	24.0	22.2	20.7	19.3	18.2	17.2	16.2	15.4
22	74.9	59.9	49.9	42.8	37.5	33.3	30.0	27.2	25.0	23.1	21.4	20.0	18.7	17.6	16.7	15.8
23	85.5	66.5	54.4	46.0	39.9	35.2	31.5	28.5	26.0	23.9	22.2	20.6	19.3	18.1	17.1	16.2
24	99.6	74.7	59.7	49.8	42.7	37.3	33.2	29.9	27.2	24.9	23.0	21.3	19.9	18.7	17.6	16.6
25	119.3	85.2	66.3	53.2	45.9	39.8	35.1	31.4	28.4	25.9	23.9	22.1	20.6	19.2	18.1	17.0
26	148.8	98.2	74.4	59.5	49.6	42.5	37.2	33.1	29.8	27.1	24.8	22.9	21.3	19.8	18.6	17.5
27	198.1	118.9	84.9	65.8	54.0	45.7	39.6	35.0	31.3	28.3	25.8	23.8	22.0	20.5	19.2	18.0
28	...	148.3	98.9	74.2	59.3	49.4	42.4	37.1	33.0	29.7	27.0	24.7	22.8	21.2	19.8	18.5
29	...	197.4	118.5	84.6	65.6	53.9	45.6	39.5	34.8	31.2	28.2	25.8	23.7	21.9	20.4	19.1
30	...	...	147.8	98.6	73.9	59.1	49.3	42.2	37.0	32.9	29.6	26.9	24.6	22.7	21.1	19.7



## WEIGHT OF CONDENSER WATER CORRESPONDING TO A UNIT WEIGHT OF WATER EVAPORATED FROM JUICE.

(For Double Effects.)

Temp. of water going into condenser in °C.	Temperature of water coming out of condenser in °C.															
	30.	32.	34.	36.	38.	40.	42.	44.	46.	48.	50.	52.	54.	56.	58.	60.
5	13.2	12.2	11.3	10.6	10.0	9.4	8.9	8.4	8.0	7.6	7.3	7.0	6.7	6.5	6.2	6.0
6	13.7	12.6	11.7	10.9	10.3	9.7	9.1	8.6	8.2	7.8	7.5	7.2	6.9	6.6	6.3	6.1
7	14.3	13.1	12.1	11.3	10.6	9.9	9.4	8.9	8.4	8.0	7.6	7.3	7.0	6.7	6.4	6.2
8	14.9	13.6	12.6	11.7	10.9	10.2	9.6	9.1	8.6	8.2	7.8	7.4	7.1	6.8	6.6	6.3
9	15.6	14.2	13.1	12.1	11.3	10.5	9.9	9.3	8.8	8.4	8.0	7.6	7.3	7.0	6.7	6.4
10	16.3	14.8	13.6	12.5	11.7	10.9	10.2	9.6	9.1	8.6	8.2	7.8	7.4	7.1	6.8	6.5
11	17.2	15.5	14.2	13.0	12.1	11.2	10.5	9.9	9.3	8.8	8.4	8.0	7.6	7.2	6.9	6.6
12	18.1	16.3	14.8	13.5	12.5	11.6	10.8	10.2	9.6	9.0	8.6	8.1	7.7	7.4	7.1	6.8
13	19.1	17.1	15.5	14.1	13.0	12.0	11.2	10.5	9.8	9.3	8.8	8.3	7.9	7.6	7.2	6.9
14	20.3	18.0	16.2	14.7	13.5	12.5	11.6	10.8	10.1	9.5	9.0	8.5	8.1	7.7	7.4	7.1
15	21.6	19.0	17.0	15.4	14.1	12.9	12.0	11.2	10.4	9.8	9.2	8.7	8.3	7.9	7.5	7.2
16	23.1	20.2	17.9	16.2	14.7	13.5	12.4	11.5	10.7	10.1	9.5	9.0	8.5	8.1	7.7	7.3
17	24.8	21.5	19.0	17.0	15.4	14.0	12.9	11.9	11.1	10.4	9.8	9.2	8.7	8.3	7.9	7.5
18	26.8	23.0	20.1	17.9	16.1	14.6	13.4	12.4	11.5	10.7	10.1	9.5	8.9	8.5	8.1	7.7
19	29.1	24.7	21.4	18.9	16.9	15.3	14.0	12.9	11.9	11.1	10.4	9.7	9.2	8.7	8.2	7.8
20	32.1	26.8	22.9	20.1	17.8	16.1	14.6	13.4	12.3	11.5	10.7	10.0	9.4	8.9	8.4	8.0
21	35.6	29.1	24.7	21.4	18.8	16.9	15.3	13.9	12.8	11.9	11.1	10.3	9.7	9.2	8.7	8.2
22	40.0	32.0	26.7	22.9	20.0	17.8	16.0	14.5	13.3	12.3	11.4	10.7	10.0	9.4	8.9	8.4
23	45.6	35.5	29.0	24.6	21.3	18.8	16.8	15.2	13.9	12.8	11.8	11.0	10.3	9.7	9.1	8.6
24	53.2	39.9	31.9	26.6	22.8	19.9	17.7	15.9	14.5	13.3	12.2	11.4	10.6	10.0	9.4	8.9
25	63.7	45.5	35.4	28.9	24.5	21.2	18.7	16.8	15.2	13.8	12.7	11.8	11.0	10.3	9.6	9.1
26	79.5	53.0	39.7	31.8	26.5	22.7	19.9	17.7	15.9	14.4	13.2	12.2	11.4	10.6	9.9	9.3
27	....	63.5	45.3	35.1	28.8	24.4	21.2	18.7	16.7	15.1	13.8	12.7	11.8	10.9	10.2	9.6
28	....	79.2	52.8	39.6	31.7	26.4	22.6	19.8	17.6	15.8	14.4	13.2	12.2	11.3	10.6	9.9
29	....	....	63.3	44.7	35.0	28.7	24.3	21.1	18.6	16.6	15.1	13.7	12.6	11.7	10.9	10.2
30	....	....	78.9	52.6	39.5	31.6	26.3	22.6	19.7	17.5	15.8	14.3	13.1	12.1	11.3	10.5

## WEIGHT OF CONDENSER WATER CORRESPONDING TO A UNIT WEIGHT OF WATER EVAPORATED FROM JUICE.

(For Triple Effect.)

Temp. of water going into condenser in °C.	Temperature of water coming out of condenser in °C.															
	30.	32.	34.	36.	38.	40.	42.	44.	46.	48.	50.	52.	54.	56.	58.	60.
5	9.1	8.4	7.9	7.4	6.5	6.5	6.2	5.8	5.6	5.3	4.9	4.9	4.7	4.5	4.3	4.2
6	9.5	8.7	8.1	7.6	7.1	6.7	6.3	6.0	5.7	5.4	5.2	5.0	4.7	4.6	4.4	4.2
7	9.9	9.1	8.4	7.8	7.3	6.9	6.5	6.1	5.8	5.5	5.3	5.1	4.8	4.6	4.5	4.3
8	10.3	9.5	8.7	8.1	7.6	7.1	6.7	6.3	6.0	5.7	5.4	5.2	4.9	4.7	4.5	4.4
9	10.8	9.9	9.1	8.4	7.8	7.3	6.9	6.5	6.1	5.8	5.5	5.3	5.0	4.8	4.6	4.4
10	11.3	10.3	9.4	8.7	8.1	7.5	7.1	6.7	6.3	6.0	5.7	5.4	5.1	4.9	4.7	4.5
11	11.9	10.8	9.8	9.0	8.4	7.8	7.3	6.8	6.5	6.1	5.8	5.5	5.3	5.0	4.8	4.6
12	12.5	11.3	10.2	9.4	8.7	8.1	7.5	7.0	6.6	6.3	5.9	5.6	5.4	5.1	4.9	4.7
13	13.2	11.8	10.7	9.8	9.0	8.3	7.8	7.3	6.8	6.4	6.1	5.8	5.5	5.2	5.0	4.8
14	14.0	12.5	11.2	10.2	9.4	8.6	8.0	7.5	7.0	6.6	6.2	5.9	5.6	5.4	5.1	4.9
15	15.0	13.2	11.8	10.7	9.8	9.0	8.3	7.7	7.2	6.8	6.4	6.1	5.8	5.5	5.2	5.0
16	16.0	14.0	12.4	11.2	10.2	9.3	8.6	8.0	7.5	7.0	6.6	6.2	5.9	5.6	5.3	5.1
17	17.2	14.9	13.2	11.8	10.6	9.7	8.9	8.3	7.7	7.2	6.8	6.4	6.0	5.7	5.5	5.2
18	18.6	15.9	14.0	12.4	11.2	10.1	9.3	8.6	8.0	7.4	7.0	6.6	6.2	5.9	5.6	5.3
19	20.3	17.1	14.9	13.1	11.7	10.6	9.7	8.9	8.3	7.7	7.2	6.8	6.4	6.0	5.7	5.4
20	22.2	18.5	15.9	13.9	12.4	11.1	10.1	9.3	8.6	8.0	7.4	7.0	6.5	6.2	5.9	5.6
21	24.7	20.2	17.1	14.8	13.1	11.7	10.6	9.7	8.9	8.2	7.7	7.2	6.7	6.4	6.0	5.7
22	27.7	22.2	18.5	15.8	13.9	12.3	11.1	10.1	9.2	8.5	7.9	7.4	6.9	6.5	6.2	5.8
23	31.6	24.6	20.1	17.0	14.8	13.0	11.7	10.5	9.6	8.9	8.2	7.6	7.2	6.7	6.3	6.0
24	36.8	27.6	22.1	18.4	15.8	13.8	12.3	11.1	10.1	9.2	8.5	7.9	7.4	6.9	6.5	6.1
25	44.1	31.5	24.5	20.1	17.0	14.7	13.0	11.6	10.5	9.6	8.8	8.2	7.6	7.1	6.7	6.3
26	55.1	36.7	27.5	22.0	18.4	15.7	13.8	12.2	11.0	10.0	9.2	8.5	7.9	7.4	6.9	6.5
27	73.3	44.0	31.4	24.4	20.0	16.9	14.7	12.9	11.6	10.5	9.6	8.8	8.2	7.6	7.1	6.6
28	....	54.9	36.6	27.4	22.0	18.3	15.7	13.7	12.2	11.0	10.0	9.2	8.6	7.9	7.3	6.9
29	....	....	43.8	31.3	24.3	19.9	16.9	14.6	12.9	11.5	10.4	9.5	8.8	8.1	7.6	7.1
30	....	....	54.7	36.5	27.4	21.9	18.2	15.6	13.7	12.2	11.0	10.0	9.1	8.4	7.8	7.3

The small dish is placed on the stove over the sheet of asbestos with a two and a half-inch (6 cm.) hole in it. The evaporation is continued until only about 75 cc. remain—it is not safe to go beyond this on account of the risk of decomposing some of the sugar. The solution is then transferred to a 100 cc. sugar flask, about 1 cc. of lead subacetate solution and a little aluminum cream added, made up to 110 cc., filtered and polarized. The polarization of the solution can be found from Schmitz's table, taking the first reading on the left in the table. To find the polarization of the condenser water from this, divide

by fifty, since the water was evaporated to one-fiftieth of its original volume.

For instance, suppose the polariscope reading were 3.0, the 100 cc. of evaporated solution would then have a polarization of 0.85 and the original condenser water a polarization of 0.017.

In order to interpret this in pounds of sugar lost in condenser water per day, it is only necessary to know the weight of condenser water. This can be calculated from the volume measured with a weir, or it can be found in terms of the weight of juice by taking the temperature of the water going in and



WEIGHT OF CONDENSER WATER CORRESPONDING TO A UNIT WEIGHT OF WATER EVAPORATED FROM JUICE.

(For Quadruple Effects.)

Temp. of water going into condenser in ° C.	Temperature of water coming out of condenser in ° C.															
	30.	32.	34.	36.	38.	40.	42.	44.	46.	48.	50.	52.	54.	56.	58.	60.
5	7.0	6.5	6.0	5.6	5.3	5.0	4.7	4.5	4.3	4.1	3.9	3.7	3.6	3.4	3.3	3.2
6	7.3	6.7	6.2	5.8	5.5	5.1	4.9	4.6	4.4	4.2	4.0	3.8	3.6	3.5	3.4	3.2
7	7.6	7.0	6.5	6.0	5.6	5.3	5.0	4.7	4.5	4.3	4.1	3.9	3.7	3.6	3.4	3.3
8	7.9	7.3	6.7	6.2	5.8	5.4	5.1	4.8	4.6	4.4	4.1	4.0	3.8	3.6	3.5	3.3
9	8.3	7.6	7.0	6.4	6.0	5.6	5.3	5.0	4.7	4.5	4.2	4.0	3.9	3.7	3.6	3.4
10	8.7	7.9	7.2	6.7	6.2	5.8	5.4	5.1	4.8	4.6	4.3	4.1	3.9	3.8	3.6	3.5
11	9.1	8.3	7.5	6.9	6.4	6.0	5.6	5.3	5.0	4.7	4.4	4.2	4.0	3.9	3.7	3.5
12	9.3	8.7	7.9	7.2	6.7	6.2	5.8	5.4	5.1	4.8	4.6	4.3	4.1	3.9	3.8	3.6
13	10.2	9.1	8.2	7.5	6.9	6.4	6.0	5.6	5.2	4.9	4.7	4.4	4.2	4.0	3.8	3.7
14	10.8	9.6	8.6	7.8	7.2	6.6	6.2	5.7	5.4	5.1	4.8	4.5	4.3	4.1	3.9	3.7
15	11.5	10.1	9.1	8.2	7.5	6.9	6.4	5.9	5.6	5.2	4.9	4.7	4.4	4.2	4.0	3.8
16	12.3	10.7	9.6	8.6	7.8	7.2	6.6	6.1	5.7	5.4	5.1	4.8	4.5	4.3	4.1	3.9
17	13.2	11.4	10.1	9.0	8.2	7.5	6.9	6.4	5.9	5.5	5.2	4.9	4.6	4.4	4.2	4.0
18	14.3	12.2	10.7	9.5	8.6	7.8	7.1	6.6	6.1	5.7	5.4	5.0	4.8	4.5	4.3	4.1
19	15.6	13.2	11.4	10.1	9.0	8.1	7.4	6.8	6.3	5.9	5.5	5.2	4.9	4.6	4.4	4.2
20	17.1	14.2	12.2	10.7	9.5	8.5	7.8	7.1	6.6	6.1	5.7	5.3	5.0	4.7	4.5	4.3
21	18.9	15.5	13.1	11.4	10.0	9.0	8.1	7.4	6.8	6.3	5.9	5.5	5.2	4.9	4.6	4.4
22	21.3	17.0	14.2	12.2	10.6	9.5	8.5	7.7	7.1	6.5	6.1	5.7	5.3	5.0	4.7	4.5
23	24.3	18.9	15.4	13.1	11.3	10.0	8.9	8.1	7.4	6.8	6.3	5.9	5.5	5.1	4.9	4.6
24	28.3	21.2	17.0	14.1	12.1	10.6	9.4	8.5	7.7	7.1	6.5	6.1	5.7	5.3	5.0	4.7
25	33.9	24.2	18.8	15.4	13.0	11.3	10.0	8.9	8.1	7.4	6.8	6.3	5.8	5.5	5.1	4.8
26	42.3	28.2	21.1	16.9	14.1	12.1	10.6	9.4	8.5	7.7	7.0	6.5	6.0	5.6	5.3	5.0
27	56.3	33.8	24.1	18.7	15.3	13.0	11.3	9.9	8.9	8.0	7.3	6.8	6.3	5.8	5.4	5.1
28	.....	42.2	28.1	21.1	16.9	14.0	12.0	10.5	9.4	8.4	7.7	7.0	6.5	6.0	5.6	5.3
29	.....	.....	33.6	24.0	18.6	15.3	12.9	11.2	9.9	8.9	8.0	7.3	6.7	6.2	5.8	5.4
30	.....	.....	42.0	28.0	21.0	16.8	14.0	12.0	10.5	9.3	8.4	7.6	7.0	6.5	6.0	5.6

WEIGHT OF CONDENSER WATER CORRESPONDING TO A UNIT WEIGHT OF WATER EVAPORATED FROM JUICE.

(For Quintuple Effects.)

Temp. of water going into condenser in ° C.	Temperature of water coming out of condenser in ° C.															
	30.	32.	34.	36.	38.	40.	42.	44.	46.	48.	50.	52.	54.	56.	58.	60.
5	5.8	5.4	5.0	4.7	4.4	4.1	3.9	3.7	3.5	3.4	3.2	3.1	3.0	2.8	2.7	2.6
6	6.0	5.6	5.2	4.8	4.5	4.3	4.0	3.8	3.6	3.4	3.3	3.1	3.0	2.9	2.8	2.7
7	6.3	5.8	5.3	5.0	4.7	4.4	4.1	3.9	3.7	3.5	3.4	3.2	3.1	3.0	2.8	2.7
8	6.6	6.0	5.5	5.1	4.8	4.5	4.2	4.0	3.8	3.6	3.4	3.3	3.1	3.0	2.9	2.8
9	6.9	6.3	5.8	5.3	5.0	4.6	4.4	4.1	3.9	3.7	3.5	3.3	3.2	3.1	2.9	2.8
10	7.2	6.5	6.0	5.5	5.1	4.8	4.5	4.2	4.0	3.8	3.6	3.4	3.3	3.1	3.0	2.9
11	7.5	6.8	6.2	5.7	5.3	4.9	4.6	4.3	4.1	3.9	3.7	3.5	3.3	3.2	3.1	2.9
12	8.0	7.2	6.5	6.0	5.5	5.1	4.8	4.5	4.2	4.0	3.8	3.6	3.4	3.3	3.1	3.0
13	8.4	7.5	6.8	6.2	5.7	5.3	4.9	4.6	4.3	4.1	3.9	3.7	3.5	3.3	3.2	3.0
14	8.9	7.9	7.1	6.5	5.9	5.5	5.1	4.8	4.5	4.2	4.0	3.8	3.6	3.4	3.2	3.1
15	9.5	8.4	7.5	6.8	6.2	5.7	5.3	4.9	4.6	4.3	4.1	3.9	3.7	3.5	3.3	3.2
16	10.2	8.9	7.9	7.1	6.5	5.9	5.5	5.1	4.7	4.4	4.2	4.0	3.7	3.6	3.4	3.2
17	10.9	9.5	8.4	7.5	6.8	6.2	5.7	5.3	4.9	4.6	4.3	4.1	3.8	3.6	3.5	3.3
18	11.8	10.1	8.9	7.9	7.1	6.4	5.9	5.5	5.1	4.7	4.4	4.2	3.9	3.7	3.5	3.4
19	12.9	10.9	9.4	8.3	7.5	6.7	6.2	5.7	5.2	4.9	4.6	4.3	4.0	3.8	3.6	3.5
20	14.1	11.8	10.1	8.8	7.9	7.1	6.4	5.9	5.4	5.0	4.7	4.4	4.2	3.9	3.7	3.5
21	15.7	12.8	10.9	9.4	8.3	7.4	6.7	6.1	5.6	5.2	4.9	4.6	4.3	4.0	3.8	3.6
22	17.6	14.1	11.7	10.1	8.8	7.8	7.0	6.4	5.9	5.4	5.0	4.7	4.4	4.1	3.9	3.7
23	20.1	15.6	12.8	10.8	9.4	8.3	7.4	6.7	6.1	5.6	5.2	4.8	4.5	4.3	4.0	3.8
24	23.4	17.5	14.0	11.7	10.0	8.8	7.8	7.0	6.4	5.8	5.4	5.0	4.7	4.4	4.1	3.9
25	28.0	20.0	15.6	12.7	10.8	9.3	8.2	7.4	6.7	6.1	5.6	5.2	4.8	4.5	4.2	4.0
26	35.0	23.3	17.5	14.0	11.7	10.0	8.7	7.8	7.0	6.4	5.8	5.4	5.0	4.7	4.4	4.1
27	46.6	27.9	20.0	15.5	12.7	10.7	9.3	8.2	7.4	6.7	6.1	5.6	5.2	4.8	4.5	4.2
28	.....	34.9	23.2	17.4	13.9	11.6	10.0	8.7	7.7	7.0	6.3	5.8	5.4	5.0	4.6	4.4
29	.....	.....	27.8	19.9	15.4	12.7	10.7	9.3	8.2	7.3	6.6	6.1	5.6	5.2	4.8	4.5
30	.....	.....	34.7	23.2	17.4	13.9	11.6	9.9	8.7	7.7	6.9	6.3	5.8	5.3	5.0	4.6

coming out of the condenser with a centigrade thermometer, and referring to the accompanying tables, which give the weight of condenser water corresponding to unit weight of water evaporated from the juice. And this number multiplied by the percentage evaporation, calculated from the formula

Per cent. evaporation (by weight) =

$$\frac{\text{Brix of Evaporated Juice} - \text{Brix of Thin Juice}}{\text{Brix of Evaporated Juice}}$$

gives the weight of condenser water corresponding to unit weight of juice entering evaporator.<sup>1</sup> This

<sup>1</sup> For convenience this may be taken to be equal to the weight of diffusion juice or mixed juice, which is usually known.

result, multiplied by the weight of mixed juice or diffusion juice for twenty-four hours, gives the weight of condenser water per day, from which the weight of sugar in the condenser water for one day can be calculated directly, by multiplying by its polarization as found above. For example, with a triple effect, suppose we have:

Temperature of water going into condenser.....	20° C.
Temperature of water coming out of condenser...	42° C.
Brix of clarified or thin juice.....	14.7
Brix of syrup or thick juice.....	62.0
Weight of mixed juice or diffusion juice in one day.	500 tons

Using these figures, we find from the table that there are 10.1 pounds of condenser water for each



pound of water evaporated from the juice. The evaporation calculated from the formula is 76.3 per cent. We therefore have  $10.1 \times 0.763 = 7.7$  pounds of condenser for each pound of juice. And since the diffusion, or mixed juice weighs 1,000,000 pounds, the condenser water will weigh  $1,000,000 \times 7.7 = 7,700,000$  pounds and contain  $7,700,000 \times 0.00017 = 1309$  pounds of sugar per day.

The tables were calculated from the formulas<sup>1</sup>

$$(I) W_a = \frac{w(c - t_b)}{t_b - t_a}, \quad (II) W_b = (W_a + I)E,$$

in which

$W_a$  = weight of injection water going into condenser;

$W_b$  = weight of water coming out of condenser;

$w$  = weight of vapor from last body;

$c$  = total heat of unit weight of vapor from last body, in calories;

$t_a$  = temperature in °C. of injection water going into condenser;

$t_b$  = temperature in °C. of water coming out of condenser;

$E$  = average per cent. evaporation in last body.

In calculating the tables the factor  $c$  is taken equal to 621.3, which is the total heat of unit weight of steam under a vacuum of 25 inches. The corresponding factors at other pressures are:

Inches.	Calories.
22	625.9
23	624.4
24	623.1
26	619.5
27	616.8
28	613.4

For the factor  $E$  the following numbers were used in the calculations:

Single effect.....	1.000
Double effect.....	0.534
Triple effect.....	0.370
Quadruple effect.....	0.284
Quintuple effect.....	0.235

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## AN ACCURATE METHOD FOR CALORIMETRIC DETERMINATIONS ON ANTHRACITE COAL.

By OSCAR W. PALMENBERG.

Received July 9, 1910.

As I have been called upon during the past year to make a considerable number of calorimetric determinations on anthracite coals and using an Atwater-Mahler calorimeter in all my work, it was necessary to find some convenient, reliable and accurate method to burn these coals, as it is a well-known fact that anthracite coals do not burn readily in the oxygen bomb calorimeter. No matter how finely the coal is pulverized, it is very seldom that a complete combustion can be obtained, and although I have been able to make duplicate tests which agreed very

<sup>1</sup> For a discussion of formula I see E. Hausbrand, "Evaporating, Condensing and Cooling Apparatus," p. 212.

well, in many cases, the calorimetric determinations were low and it would be necessary to filter the bomb washings and burn the residue in a Gooch crucible and from this loss in weight correct for the unburned coal. Such a method is not alone inaccurate, but requires too much time where many tests a day are to be run. The combustion in the bomb became less complete the greater the ash content. It occurred to me that the coal would have to be mixed with some material that would assist combustion and burn up this small amount of coal which was invariably left in the bomb washings. I first tried to make a pellet by mixing pulverized cane sugar with the coal and succeeded in getting rather concordant results this way from a mixture of 1 gram of sugar and 0.5 gram coal, but this method had its fault and was not very satisfactory.

Next I tried to enclose the coal (about 0.5 gram) in a gelatine capsule, but never succeeded in obtaining complete combustion; at least not with very high ash coals; and besides, gelatine being so hygroscopic, errors would occur in the weighings.

The only satisfactory and simple method I have found which gives absolutely complete combustion is to weigh out 0.2 gram of semi-bituminous coal of about 14300 to 14500 B. t. u. in a shallow nickel capsule (1 in. x 3/8 in.) which fits in the supports in the bomb conveniently, and after spreading this soft coal evenly over the bottom of the capsule, weigh on top of this 0.8 gram of the anthracite coal, pulverized to pass a 100 mesh. A little more or less in weight of either coal does not affect the result, so long as the quantity taken has been accurately weighed. This weighing can be done in a very short time and there is nothing more to do but to have the wire touch the coal, whereupon the combustion will be complete and tests can be made as accurately as on any soft coal.

It is only necessary to know the calorific content and the amount of sulphur in the kindler (soft coal) burned, whereupon the correction for these is applied very conveniently. I keep a four-ounce bottle of stock coal which has been passed through a 60-mesh sieve and thoroughly mixed and from this draw a small specimen tube full, which tube full will last for about 50 determinations.

My stock bottle of coal was put up June 4, 1909, and calorimetric tests were made from time to time to make sure of the corrections and the following results were obtained:

Test on coal as burned.	Dry basis.	Date of test.
7942 Calories	14338 B. t. u.	June 4, 1909
7952 Calories	14349 B. t. u.	June 18, 1909
7954 Calories	14359 B. t. u.	Aug. 3, 1909
7936 Calories	14332 B. t. u.	Feb. 5, 1910
7920 Calories	14300 B. t. u.	Feb. 16, 1910
7899 Calories	14260 B. t. u.	Mar. 26, 1910
7915 Calories	14283 B. t. u.	Apr. 20, 1910
7927 Calories (used 1/2 gram coal)	14297 B. t. u.	Apr. 26, 1910
7923 Calories (used 1 gram coal)	14298 B. t. u.	Apr. 26, 1910

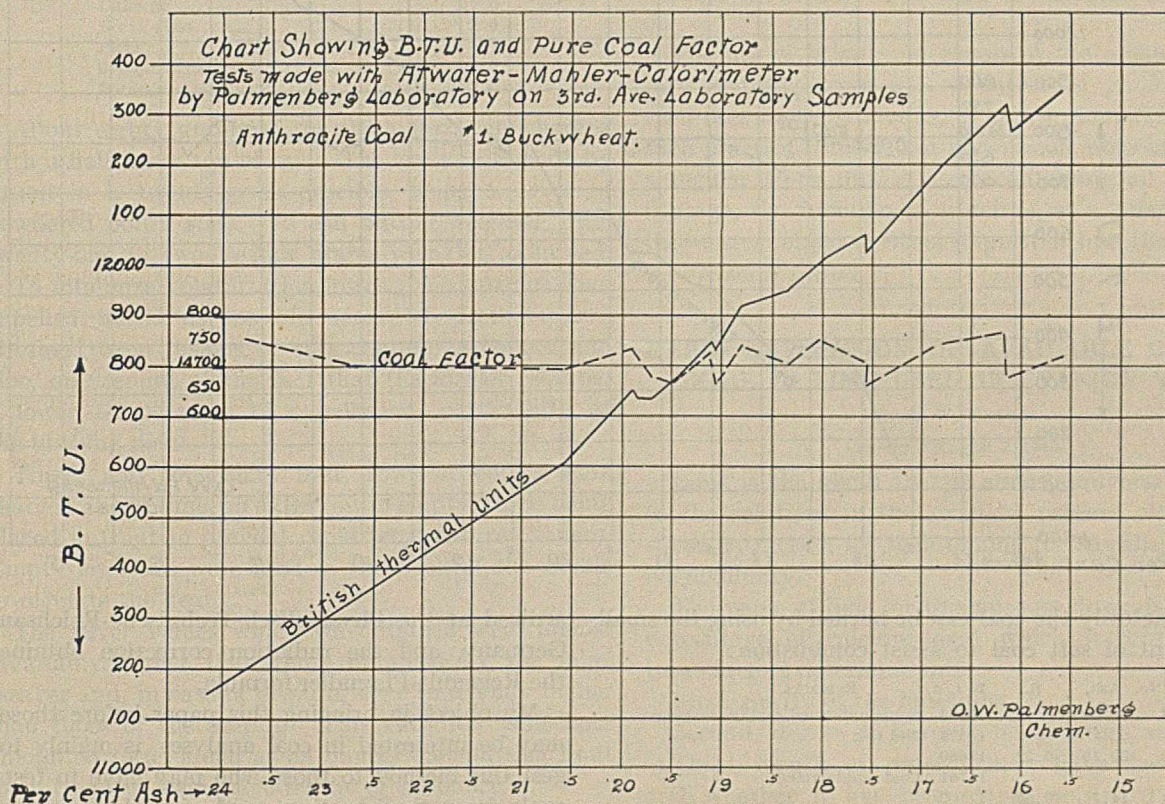


There seems to be a gradual decrease in the thermal content of the coal, due probably to oxidation, but on the whole very little change has taken place and not enough to make any appreciable error in the determination when running the anthracite. The variations in the above results are probably due, in part, to the fact that the moisture determination was not made with each new calorific determination. Thus the first four B. t. u. figures were calculated from the calorific test, using the moisture content of the first test and as the coal lost in moisture the calculation would be too high. This accounts for the apparent increase in the thermal value. On the other five tests the moisture was determined in every case.

$$H = \frac{\text{B. t. u.} - \text{Sulphur} \times 4050}{1 - (\text{Ash} + \text{S})}$$

for dry coal, and gives a very good line on the accuracy of the work where many tests are run on a coal coming from the same region. The factor in this case averages 14690, and from this it is plainly seen that the results are consistent. These two charts are practically identical, and one line would cover the other if placed over one another.

There are a few irregularities, but nothing out of the ordinary general run of work. In this case all the results were taken as obtained, not being picked with the intention of giving a symmetrical chart, and in this way the consistency of the work and



When using the coal for combustion with the anthracite, the calorific content of the coal as drawn from the tube only need be known. To show how reliable calorific tests run by this method are, when burning an anthracite in a calorimeter of the Berthelot-Mahler type, I arranged all my results on two sets of tests made on the same coal delivered to different places of consumption, in consecutive order from the lowest to the highest ash content and placed these B. t. u. figures corresponding to the ash in each sample on cross-section paper. The accompanying charts bring out this fact very plainly. The lower line represents the calorimetric test and the upper line the "Pure Coal" factor. This pure coal factor is obtained by Lord and Haas' formula,

method can be proven. The results were all taken from the general run of tests, and time did not permit to run tests over unless there was some striking error. The coal on which all these determinations were made was No. 1 buckwheat size and came from the Lehigh locality; it was shipped in 800-ton lighters to the consumer and the samples were taken automatically from the weighing hopper.

After titrating the bomb washings and acidifying, boiling and filtering, the residual ash from the solution was dried on the paper, brushed on to a watch glass and examined under the microscope, and in very few cases was there any unburned coal to be found, and if present the amount was too small to have any appreciable effect upon the test.

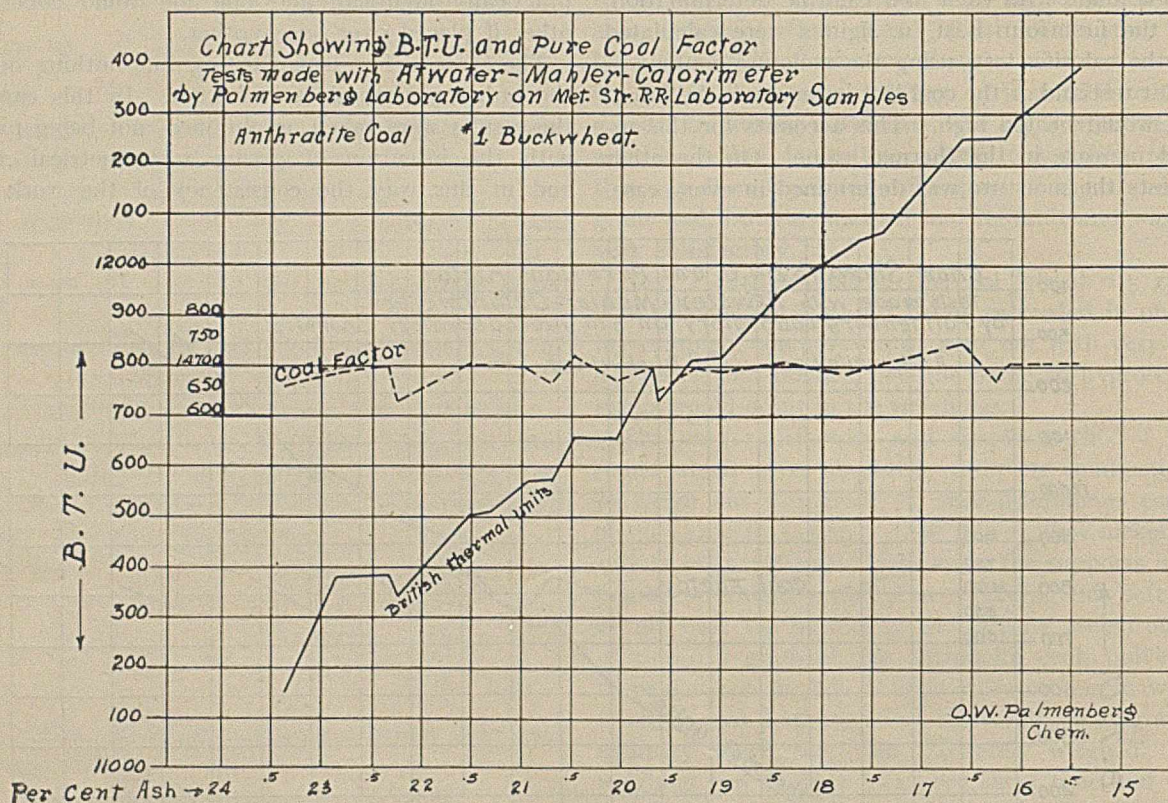


The sulphur tests were all made from the bomb washings.

Following are a few duplicate tests run on different samples of anthracite coal. On some a second test was made because the first appeared suspicious and on others a duplicate was run to test the reliability of the method. These results show very plainly how

of particular interest, as they show how close results will run when all conditions are alike.

All tests were run in the Atwater-Mahler bomb calorimeter platinum-lined, and the hydrothermal equivalent was determined with cane sugar as obtained from the Bureau of Standards, Washington, D. C., using a Fuess thermometer which was stand-



satisfactorily the coal can be burned by using the small amount of soft coal to assist combustion:

Rep. No.	Ash.	S.	B. t. u.	Remarks.
328	26.58	0.68	10494	Some unburned coal in washings
			10523	2nd test
330	20.19	0.72	11680	
			11761	Coal repulverized
333	14.36	0.60	12502	
			12460	2nd test
438	14.29	0.54	12506	
			12554	2nd test
440	25.20	0.44	10890	
			10895	2nd test
442	20.26	0.70	11626	
			11601	2nd test
444	23.62	0.43	11085	
			11088	2nd test
462	15.22	0.67	12506	
			12533	2nd test
399	21.19	0.49	11465	P
	21.26	0.49	11439	J
400	21.67	0.49	11430	P
			11432	J
494	8.99	0.60	13655	P
			13636	P
			13645	J

Mean = 13645

On samples Nos. 399, 400 and 494 the tests were made by another chemist, using this method, and also by myself, as indicated by the letter P for mine and J for the other operator. These last three tests are

ardized at the Physikalisch-Technische-Reichsanstalt, Germany, and the radiation correction obtained by the Regnault-Pfaundler formula.

My object in bringing this paper before those who may be interested in coal analyses is mainly to suggest this method to those who may have to test hard coals from time to time, and give them an entirely reliable method so that determinations can be obtained when made by different operators, which will agree within reason, as I have too often heard of the direct combustion test being run and correct determinations claimed by the operator. In my experience I have never succeeded in burning anthracite coals completely without the aid of a kindler in an oxygen bomb calorimeter.

#### THE EFFECT OF VARYING AMOUNTS OF LITHARGE IN THE FIRE ASSAY FOR SILVER.

By KENNETH WILLIAMS.

Received August 1, 1910.

The effect of varying amounts of litharge in the fluxes used in the fire-assay of ores for silver was



brought to my attention during several months of work as assayer at the Tintic Smelter.

During that time we had considerable trouble in checking with the mine assayer in our assays for silver.

The ores on which varying results were obtained were oxidized lead ores, containing from forty to sixty per cent. of silica, about five per cent. of iron and two per cent. of lime, from fifteen to forty per cent. of lead, from twenty to eighty ounces of silver, never more than two per cent. of zinc, about one per cent. of sulphur, and only a trace of copper.

The regular (stock) flux used on these ores was made up as follows:

	Lbs.
Litharge.....	12.0
Borax glass.....	2.0
Flour.....	1.1
Sodium bicarbonate.....	12.0
Potassium carbonate.....	12.0

About eighty grams of the above flux were charged with a half-assay ton of ore. The assay was made by fusion in a twenty-gram crucible, using a cover of powdered borax glass. A lead button weighing from twenty-one to twenty-four grams was thus obtained.

To minimize volatilization losses these buttons were cupelled, as far as possible, separately from buttons obtained from fusions of ores containing copper and zinc, on account of the fact that the former required a lower temperature of cupellation, particularly at thelicking point.

Three, and sometimes four, rows of cupels were charged at one time, an extra row of empty cupels being placed in front to prevent rapid temperature changes. Empty crucibles were used in the back of the muffle to regulate the heat.

The silver values which we obtained were almost invariably higher than those obtained by the mine assayer and, in case of dispute, very commonly higher than those of the umpire. This variation sometimes amounted to as much as an ounce, the splitting limit for settlement being five-tenths of an ounce.

The lower results obtained by the second and third parties I attribute to two causes:

- (1) Higher temperature of cupellation.
- (2) Excessive amounts of litharge in the fluxes.

With regard to temperature of cupellation, I understand that it is rather common practice in some assay offices, in running control and umpire work, to cupel in single rows. In so doing the cupels often show much heavier "feathers" on one side than on the other—evidence that the temperature was higher than necessary.

Moreover, if buttons of the character described be cupelled with buttons containing copper, the latter will require a higher cupelling temperature, involving a correspondingly greater volatilization loss in the former.

With regard to excessive use of litharge, I made up two other fluxes, similar to our own stock flux, but containing, in the first case (A) about fifty grams of litharge, and in the second case (B) seventy-five grams of litharge per half-assay ton charge.

Three ores were assayed using these different fluxes, care being taken to cupel buttons from different fluxes in the same muffle, so that the temperature of cupellation should be as nearly as possible the same in each case.

The average of results obtained were as follows:

	Regular flux.	Flux A.	Flux B.
Ore No. 1.....	51.05	50.86	50.62
Ore No. 2.....	42.36	42.20	42.02
Ore No. 3.....	27.82	27.70	27.55

The figures given are ounces of silver per ton of ore.

The use of litharge in excess of the amount necessary to bring down a lead button of proper size on such ores as the above is, of course, unnecessary, but such practice I find is not uncommon, especially where a regular stock flux is used, containing an excess of litharge, the function of which is, as a "cleanser," to throw any copper or other impurities into the slag.

UNIVERSITY OF UTAH.

### RAPID ESTIMATION OF AVAILABLE CALCIUM OXIDE IN LIME USED IN CYANIDE WORK.

By L. W. BAHNEY.

Received August 2, 1910.

Lime is the alkali that is almost universally added to the solutions in the cyanide process of gold and silver extraction for maintaining the so-called protective alkalinity.

The value of lime for this purpose depends upon the percentage of calcium oxid which it contains, and three factors determine this.

First, purity of the limestone.

Second, degree of burning temperature and time.

Third, length of time it may have been stored and whether it has been damp or dry during this period of storage.

These three factors combine to make the quality of lime which is bought in the open market rather uncertain.

While lime bought in the United States from a reliable firm, which thoroughly burns a pure limestone and delivers at once to the consumer from the kilns, may be of a fairly high and uniform composition, yet in Mexico and Central America, where it is purchased from many small producers who often have a poor grade of limestone to start with, and whose kilns are small and crude and who use as little fuel as possible, the quality of the lime is quite variable.

The determination of calcium by the gravimetric method, with the necessity of also determining the proportion of carbon dioxide, silica and iron, requires



too much time, and is usually out of the question for the isolated plant without the necessary apparatus and the services of a skilled chemist.

The calculating of all the calcium found to calcium oxid, although sometimes done, is manifestly very inaccurate.

Several methods<sup>1</sup> of titrating by means of a standard acid have been described and no doubt give good results, which are sufficiently accurate for a technical method, and the objections to these are that they involve the preparation of a standard solution of some acid, usually  $N/10$  HCl, which cannot be weighed out but must be standardized with some other standard solution. The idea suggested itself of using the solution which is employed in determining the alkalinity of the cyanide solutions.

Solutions of the following acids have been used by different operators for this purpose: sulphuric, nitric, hydrochloric and oxalic. Oxalic acid is perhaps the best for this purpose because a standard solution can be prepared by weighing the solid acid and dissolving it in water.

While this method of standardization is open to the objection that the hydration of the acid may vary somewhat, yet it yields a solution sufficiently accurate for technical work, and so far as I am aware, the use of oxalic acid for this purpose has not been suggested.

Pure calcium oxid was prepared by selecting pure white crystals of calcite, grinding fine in an agate mortar and igniting in a platinum crucible over a strong blast until constant weight resulted.

This oxid was cooled in a desiccator, then ground in an agate mortar to pass 200 mesh, and the calcium oxid determined gravimetrically; the result was 99.98 per cent. against a theoretical 100 per cent.

This calcium oxid was then used as a standard throughout the succeeding tests.

Similar weighed portions were titrated with decinormal HCl and oxalic acid, using phenolphthalein as indicator, and where 44.2 cc. of HCl were required, 44.6 cc. oxalic acid completed the reaction. The solution of the oxalic acid used in the subsequent experiments was made by dissolving 14.6068 grams in enough distilled water to make a liter. The first experiments were made upon small amounts of 140 mg., to which was added 100 cc. of water before titration, the idea being to have just enough lime present to be theoretically soluble in that amount of water.

As this is a rather small amount to handle and the method given in Treadwell and Hall,<sup>2</sup> of weighing out 14.000 grams, making 1000 cc. of emulsion, removing 100 cc., again diluting to 1000 cc., and removing 100 cc. for titration, did not give results which

checked upon low-grade limes and is open to the objection of extra manipulation, larger amounts were tried and were introduced directly into the flask in which the determination was to be carried out.

The weight of lime to be taken was calculated so that each cubic centimeter of oxalic acid solution should represent 1 per cent. of calcium oxid:

$$\begin{array}{rcl} \text{Lime} & : & \text{Lime} & : & \text{Oxalic} & : & \text{Oxalic} \\ 56.09 & & x & & 126.048 & & 1.46068 \\ & & & & & & x \text{ equals } 650 \end{array}$$

This weight in milligrams (650) was then used in all the tests, and the following table will show results which are satisfactory for a technical method.

The titrations were made in the cold by introducing 650 mg. of the sample into a 300 cc. Erlenmeyer flask containing 50 cc. of distilled water, using phenolphthalein as indicator.

Calcium carbonate. Per cent. present.	Calcium oxide.	Calcium oxide. Per cent. found.
95	5	5.2
90	10	10.3
85	15	15.3
80	20	20.5
75	25	25.0
70	30	30.2
65	35	35.0
60	40	40.0
55	45	45.0
50	50	49.8
45	55	54.5
40	60	59.9
35	65	64.8
30	70	69.6
25	75	74.5
20	80	79.2
15	85	84.8
10	90	90.0
5	95	94.7
0	100	100.0

This series indicates that calcium oxid, in the presence of calcium carbonate, can be determined by this method within a fair degree of accuracy.

Silica, which is present in most limes, does not interfere.

Magnesia, which is also present in most limes, in greater or lesser amount, is very slightly soluble in water and shows a faint reaction with the indicator, but it is of no value as an alkali in cyanide work and should not be shown in a determination of the available alkali in lime to be used for this purpose.

Fortunately the point where the alkalinity due to CaO stops is readily recognized after a little practice, for the color is a vivid pink while that of MgO is faint, and the color in the titration of MgO disappears with the addition of only one- or two-tenths of a cubic centimeter of the oxalic acid solution, and the color returns very slowly and feebly while that of lime is rapid and sharp.

This is illustrated by the fact that a titration of pure CaO required only 5 minutes while the same amount of MgO required three and one-half hours.

In order to test the method in the presence of magnesia, two samples of limestone containing magnesia were ground to 200 mesh and titrated (after ignition

<sup>1</sup> Sutton's "Volumetric Analysis." "Quantitative Analysis," Vol. II. Treadwell and Hall. *Trans. Met. Soc. S. Africa.*

<sup>2</sup> Vol. II, p. 453.



in platinum crucibles to constant weight). The CaO was determined by the gravimetric method in each sample, there was no silica present and no more than a trace of iron, with these results:

	CaO present by gravimetric method. Per cent.	Calcium oxid found by the oxalic acid method. Per cent.
Sample No. 1.....	57.6	57.6
Sample No. 2.....	50.4	51.0

This indicates that the magnesia does not interfere. Its presence and approximate amount can be judged by the behavior of the titration and quite accurately by continuing the titration if one can spare the time needed.

Iron oxid is sometimes present in impure limes in considerable amount and it obscures or masks the color of the indicator, but if the precipitate be allowed to subside the titration may be carried out to within 1 per cent. of the correct result.

If it is desirable to determine the amount of carbonate present in an imperfectly burned lime it may be carried on as follows:

After grinding the sample to pass 200 mesh weigh out 650 mg. and make the titration in the usual manner; call this result 1-available calcium oxid.

Now ignite some of the finely ground sample in a muffle or over a blast and make another determination, using 650 mg., calling this result No. 2.

Subtract 1 from 2 and divide by 1.78.

*Method of Procedure.*—The sample must be ground to pass through a 200-mesh screen. Into a 300 cc. Erlenmeyer flask place 50 cc. of distilled water, then add the 650 mg. of the finely ground sample, stopper the flask and shake vigorously for 10 seconds, add two drops of solution of phenolphthalein and then run in the standard solution of oxalic acid until the pink color is discharged, then place in the stopper and shake again.

When the color returns, if it is lime, it will be a bright vivid pink, and the addition of perhaps one-half cc. will be necessary to discharge this color, but if the flask is again shaken and the color returns slowly and is a faint, weak pink this is the end point for the lime.

At all time during the running in of the oxalic acid solution the flask should be shaken as violently as possible, care being taken not to allow it to splash out, so that calcium oxid will pass into solution. In nearly every instance of titrating a high-grade lime the pink color remained vivid nearly to the finish, which shows that the CaO is rapidly soluble. If a completed titration is allowed to stand for from 15 minutes to half an hour the pink color will return and show as brightly as in the beginning.

The reading of the burette is in per cent. of calcium oxid. The solutions necessary are:

Oxalic acid, 14.6068 grams of pure crystals dissolved in enough water to make a liter.

Phenolphthalein 0.5 gram dissolved in a mixture of 50 cc. alcohol and 50 cc. water.

STANFORD UNIV.,  
CALIFORNIA.

[CONTRIBUTION NO. 10 FROM THE RESEARCH LABORATORY OF APPLIED CHEMISTRY OF THE MASSACHUSETTS INSTITUTE OF TECHNOLOGY.]

## DIFFERENTIAL CONDENSATION IN DISTILLATION AND RECTIFICATION OF BINARY LIQUID MIXTURES.

By ALCAN HIRSCH.

The object of this paper is to show the important part which condensation plays in distillation and rectification of binary liquid mixtures, and how the efficiency of operation of commercial plants may be increased by proper partial condensation, especially in the case of alcohol-water. The design of total condensers is well understood, and standard textbooks such as *Verdampfen Kondensieren und Kühlen*, by E. Hausbrand, contain sufficient data on cooling surfaces, radiation, heat transfers, etc., for their correct design and construction. But these same methods of design are usually employed in the case of partial condensers—in fact in commercial practice the same piece of apparatus is often used interchangeably as a partial or total condenser simply by regulating the flow of cooling water. It will be shown that in the case of the design of partial condensers several additional factors enter which have not heretofore been taken into account, and that in the great majority of cases the yield obtained from commercial apparatus, both partial condensers and rectifying columns, may be materially increased by proper condensation without increasing the coal consumption under the boilers.

If a mixture of two miscible liquids of different boiling points be boiled, the composition of the boiling liquid being kept constant, the vapor evolved will be richer in the more volatile constituent than the liquid, unless the latter be of the constant boiling composition, in which case the vapor is of the same composition as the liquid. The curves showing the composition of the liquid and the composition of the vapor in equilibrium with this liquid have been determined for many different mixtures, but for water-ethyl alcohol mixtures with which the experimental part of this paper is concerned, there are three (3) principal curves, as shown in Fig. 1; the Grönig, the Rachewsky, and the Sorel. The Grönig is the original data, but Sorel<sup>1</sup> claims that the composition of the vapor in the upper part of Grönig's curve is greater than the true value, due to partial condensation of the vapor in the upper part of the distilling flask, and Sorel has accordingly applied

<sup>1</sup> *Compt. rend.*, 116, 693.



a correction to the Grönig data. The Rachewsky data seems to be a compromise between the Grönig and the Sorel. E. Hausbrand<sup>1</sup> states that the Sorel data is the most reliable, and that the condensation in practice, where there is always more or less priming, would be much closer to the Sorel curve than the Grönig. The Sorel data were used for all computations in this article where compositions of vapor-liquid are involved, for while they are not absolutely correct, yet they are the best at hand. It is desirable that the equilibrium liquid-vapor composition be known exactly<sup>2</sup> and that the necessary correction for priming be made for commercial cases.

In order that a vapor may be enriched in respect to the more volatile component, two general methods

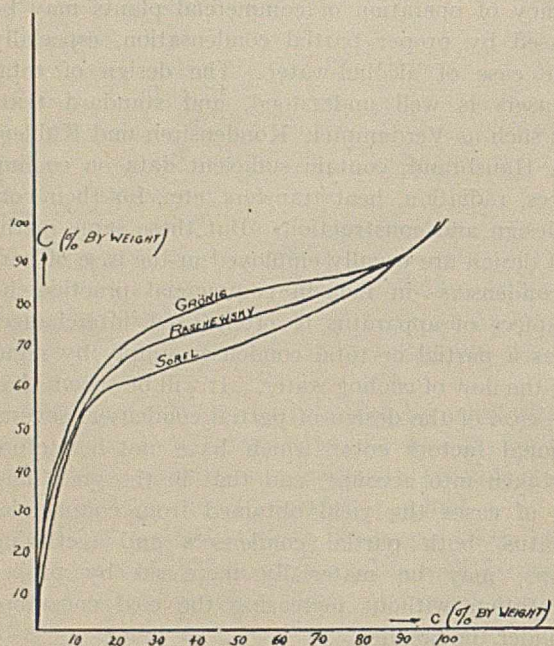


Fig. 1.

of procedure have long been in use. First, there may be induced a partial condensation in which a large portion of less volatile component and a smaller portion of the more volatile component may be deposited in liquid form, depending upon the removal of the latent heat by an external cooling medium, for example running water in a cooling coil. Second, the vapor may be enriched in respect to the more volatile constituent by rectification. This operation consists of an exchange of heat between vapor and liquid mixture only, the condensed vapor delivering its latent heat to the liquid, from which is vaporized in time a new vapor richer in the more volatile component. In order that the last operation may be

<sup>1</sup> E. Hausbrand, *Zeit. f. Chem. Apparut.*, 2, 585 (1907).

<sup>2</sup> A method for calculating exactly the composition of equilibrium vapor-liquid from thermodynamic data and from some of the laws of physical chemistry has been devised to check Sorel's curve. The results of this work will be presented in a later paper.

continuous each plate must have an appropriate intake and overflow of liquid from the plate above and to the plate below, respectively, the top plate being fed with the condensate from a partial or total condenser.

The nomenclature used in this article is as follows:

The composition of both vapor and liquid is expressed in per cent. by weight of the more volatile component—in the case of alcohol-water, by per cent. alcohol by weight. The composition of the vapor is expressed by  $C$ , with subscripts to denote the composition of different vapors such as  $C_1, C_2$ , etc. The composition of the liquid is expressed by  $c$ , with subscripts to denote the composition of different liquids, such as  $c_1, c_2$ , etc. Furthermore, the compositions of equilibrium vapor and liquid are denoted by the same subscripts, as  $C_1, c_1; C_2, c_2$ ; the values to be found from Sorel's curve for each specific case. For weight of vapor two letters are used:  $G$  with proper subscripts such as  $G_1$  and  $G_2$  for the theoretical discussion, and  $W$  with proper subscripts, such as  $W_1$  and  $W_2$ , for actual experimental determinations. For instance, suppose that we wish to enrich a vapor of composition  $C_1$  to a vapor of composition  $C_2$  by partial condensation. By actual experiment a partial condensation is effected, and from a weight  $W_1$  of a vapor of composition  $C_1$  we have remaining a weight  $W_2$  of a vapor of composition  $C_2$ . The ratio  $W_2/W_1$  is called the yield. We can compute by use of the equations shown below the theoretical maximum yield that can be obtained in any given case. A weight  $G_1$  of vapor of composition  $C_1$  will give theoretically a maximum weight  $G_2$  of vapor of composition  $C_2$ .  $G_2/G_1$  is the theoretical maximum yield. Then  $W_2/W_1$ , the actual yield divided by  $G_2/G_1$ , the theoretical yield gives  $W_2G_1/W_1G_2$ , which, multiplied by 100, gives the percentage efficiency.

Since the total heat contained in the weight  $G_1$  of the vapor of composition  $C_1$  remains the same, however, the quantity  $G_2$  of the composition  $C_2$  varies, the percentage efficiency which here represents weight of product may be also taken as the thermal efficiency.

In a paper by Dr. W. K. Lewis,<sup>1</sup> of this laboratory, on "The Theory of Fractional Distillation," it was shown mathematically that the largest possible yield in enriching a vapor from a composition  $C_1$  to a composition  $C_2$  by partial condensation can be obtained when the conditions of a certain differential equation<sup>2</sup> are fulfilled, and such a condensation was accordingly

<sup>1</sup> THIS JOURNAL, 1, No. 8, 1909.

<sup>2</sup> If a weight  $G$  of vapor of composition  $C$  enter a partial condenser; and a weight  $dG$  is condensed, then,

$$\begin{aligned}
 C.G. &= (G-dG)(C-dC) + c.dG \\
 C.G. &= C.G - G.dC - C.dG + dG.dC + c.dG \\
 dG(C-c) &= -G.dC \\
 dC/G &= -dC/C-c \\
 \ln G_2/G_1 &= -\int_{C_1}^{C_2} dC/C-c
 \end{aligned}$$



called a *differential* one. It was also shown that a differential condensation may be considered an infinite series of infinitesimal simple condensations, and is more efficient than a finite series of simple condensations between the same limits of composi-

tion change. The maximum theoretical yield may be computed for any given case graphically from the  $(C_1/C-c)$  curve shown in Fig. 2, by planimetry the area under the curve between the limits of composition change, equating the value to the logarithmic

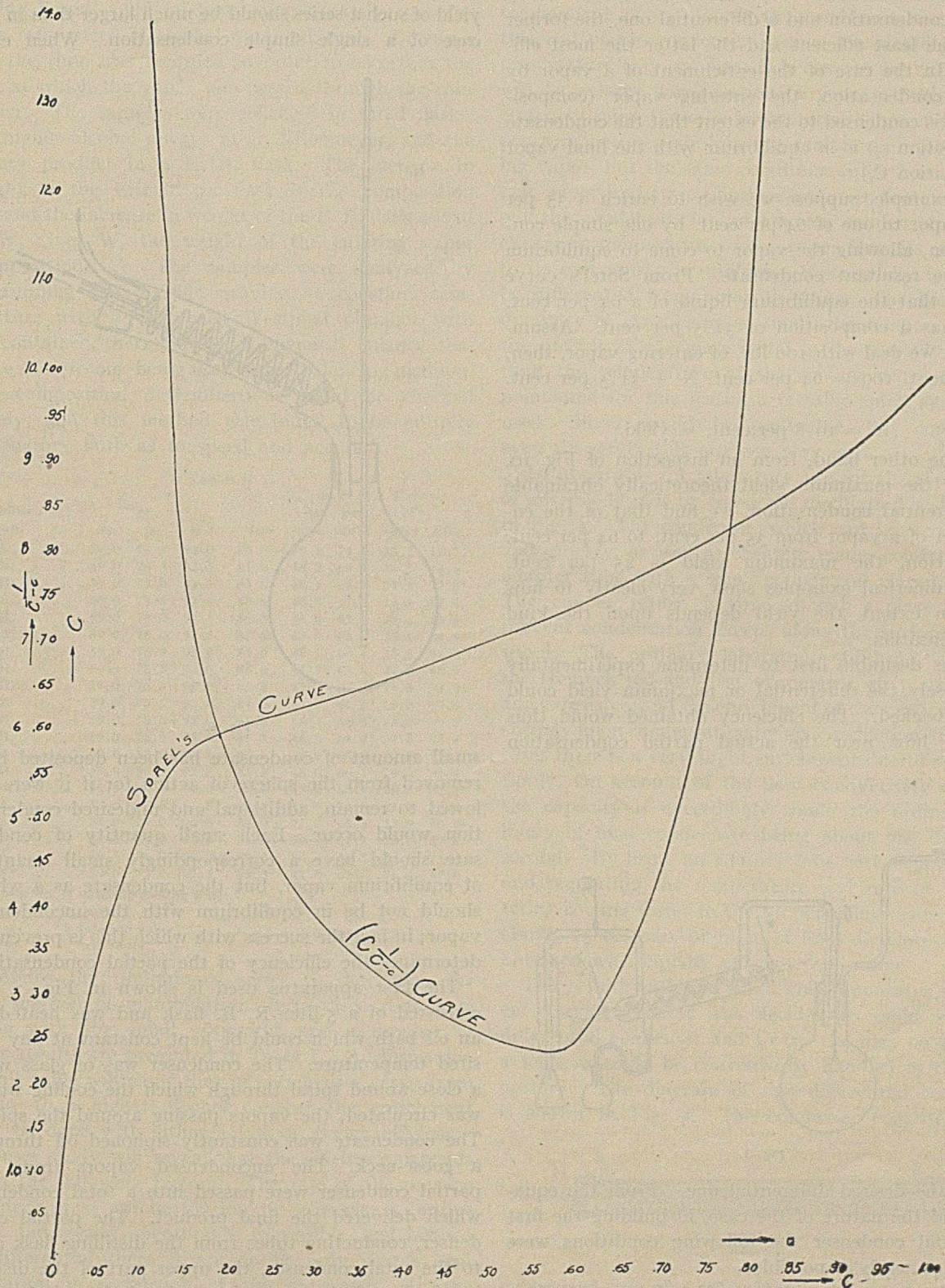


Fig. 2.



equation, and solving. The curves of maximum theoretical yield have been thus computed and are plotted in Fig. 10, so that by reference to these curves the maximum yield may be read off for any given practical case of composition change.

The two extremes of partial condensation are a simple condensation and a differential one, the former being the least efficient and the latter the most efficient. In the case of the enrichment of a vapor by simple condensation, the entering vapor (composition  $C_1$ ) is condensed to the extent that the condensate (composition  $c_2$ ) is in equilibrium with the final vapor (composition  $C_2$ ).

For example, suppose we wish to enrich a 35 per cent. vapor to one of 64 per cent. by one simple condensation, allowing the vapor to come to equilibrium with the resultant condensate. From Sorel's curve we find that the equilibrium liquid of a 64 per cent. vapor has a composition of 31.5 per cent. Assuming that we deal with 100 lbs. of entering vapor, then, 35 per cent.  $100 = 64$  per cent.  $X + 31.5$  per cent.  $(100 - X)$ .

$$X = 10.8 \text{ per cent.} = \text{yield.}$$

On the other hand, from an inspection of Fig. 10, showing the maximum yield theoretically obtainable by differential condensation, we find that in the enrichment of a vapor from 35 per cent. to 64 per cent. composition, the maximum yield is 45 per cent. These numerical examples show very clearly to how great an extent the yield depends upon the kind of condensation.

It was desirable first to determine experimentally how closely the differential or maximum yield could be approached. The efficiency obtained would thus indicate how near the actual partial condensation

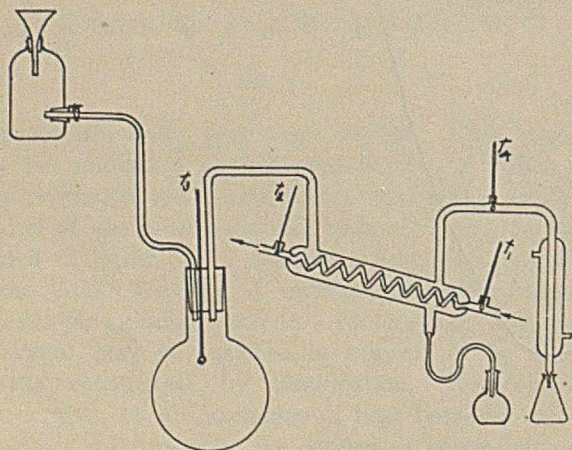


Fig. 3.

was to the desired differential one. From the equations and the nature of the case, in building the first differential condenser the following conditions were fulfilled as nearly as possible:

(1) The vapor should be uniformly condensed, a

very small amount at a time, and the process should be continuous.

(2) The condensate should be removed as fast as formed.

By fulfilling these conditions, there should be a number of very small simple condensations, and the yield of such a series should be much larger than in the case of a single simple condensation. When each

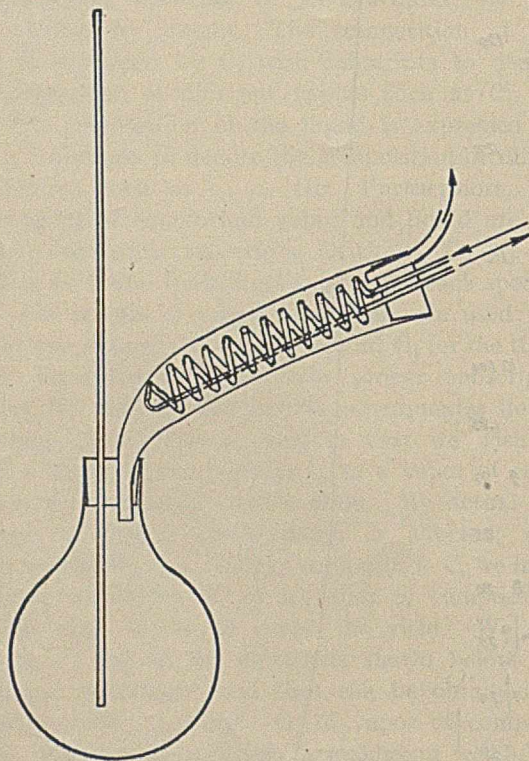


Fig. 4.

small amount of condensate has been deposited it is removed from the sphere of action for if it were allowed to remain, additional and undesired condensation would occur. Each small quantity of condensate should have a correspondingly small quantity of equilibrium vapor, but the condensate as a whole should not be in equilibrium with the uncondensed vapor; in fact the success with which this is prevented determines the efficiency of the partial condensation.

The first apparatus used is shown in Fig. 3. It consisted of a 5-liter R. B. flask and was heated in an oil bath which could be kept constant at any desired temperature. The condenser was of glass with a close-wound spiral through which the cooling water was circulated, the vapors passing around the spiral. The condensate was constantly siphoned off through a goose-neck. The uncondensed vapors from the partial condenser were passed into a total condenser which delivered the final product. The partial condenser, conducting tubes from the distilling flask and to the total condenser, the upper part of the distilling flask and the projecting ends of the spiral, were



all thoroughly lagged with asbestos-magnesia insulating material. No recorded run was made before the apparatus was well warmed up, so that there was no appreciable condensation due to heat conduction from without during a run. The composition of the boiling liquid could be kept approximately constant by a return-feed of alcohol-water mixture. During a run the time was recorded in order to ascertain the rate at which the vapor was passed through the condenser. The samples were collected in tared flasks, the higher alcohol always in an Erlenmeyer, and the watery product in a F. B. flask. The increase in weight of the Erlenmeyer flask is  $W_2$  (composition  $C_2$ ), and the increase in weight of the F. B. flask added to  $W_2$  gives  $W_1$  the weight of the entering vapor (composition  $C_1$ ). The samples were analyzed by determining the specific gravity at constant temperature with a calibrated Westphal plumbet with self-contained thermometer, a chemical balance sensitive to 1/10 mg. being used as the balancing medium. The composition determinations could be checked closely, and this method was found to be entirely satisfactory both as to speed and accuracy.

TABLE I.

Number of run.	$t_1$	$t_2$	$t_3$	$t_4$	$C_1$ in per cent.	$C_2$ in per cent.	Rate of vapor.	Eff.
29 B.....	59.0	76.5	92.0	80.0+	49.5	74.5	13.2	17.7
30 B.....	59.0	76.5	92.5	81.0	48.2	69.3	16.9	47.5
31 B.....	59.0	77.0	92.3	85.0	48.1	68.7	21.5	77.8
32 B.....	59.0	77.5	92.5	85.5	46.8	67.3	22.5	77.4
33 B.....	59.0	78.0	92.5	86.5	45.0	63.8	24.6	83.5
34 B.....	59.0	76.0	92.5	86.0	42.9	63.7	24.1	84.5
35 B.....	59.0	76.0	92.0	85.0	47.1	66.4	24.8	84.8
36 B.....	59.0	76.5	92.0	86.0	45.3	63.6	27.5	88.9
37 B.....	59.0	76.0	93.0	82.8	41.7	67.9	14.5	33.8
38 B.....	59.0	76.5	93.0	84.0	41.0	64.3	15.8	40.6
39 B.....	59.0	76.0+92.0	85.6	48.4	66.6	28.0	90.8	
40 B.....	59.0	76.5	93.0	87.5	46.9	61.8	37.8	95.0
41 B.....	59.0	83.0	92.5	85.0	43.4	66.8	22.7	77.5
42 B.....	59.0	85.0	92.5	85.5	46.0	66.1	24.8	91.4
43 B.....	59.0	85.0	92.0	85.5	47.4	65.5	29.5	92.2
25 A.....	52.0	69.0	91.5	82.0	49.0	71.5	21.4	35.0
8 A.....	57.0	79.0	96.0	85.0+	25.0	65.5	12.9	50.0
10 A.....	64.0	84.0	94.0+	89.0	39.4	57.3	15.0	87.0
11 A.....	59.5	84.0	94.0	89.0	36.4	58.5	13.8	71.6
22 A.....	59.0	77.0	92.5	85.0	47.0	65.5	26.4	87.0

$t_1$  = temp. of entering cooling water.

$t_2$  = " " exit " "

$t_3$  = " " entering vapor.

$t_4$  = " " exit vapor.

Besides the preliminary series to determine the best method of manipulation, forty-three runs were made with the small apparatus and a number of these results are presented in Table I. An inspection of this table will show that in enriching a vapor from a low to a higher alcohol content the enrichment may be done with either a low or a high efficiency. A closer study will reveal that the efficiency depends upon the following factors: The rate of the passage of vapor must not be too low or otherwise a continuous and steady operation is impossible, and instead of differential there is apt to be only simple condensation in a portion of the apparatus. In the small laboratory apparatus used the minimum rate

for satisfactory operation proved to be about 25 cc./min. The temperatures of both the entering and the leaving cooling water is perhaps the most important factor in differential condensation. Suppose that a vapor entered the differential condenser at a temperature of 92° C. If the condensing surface at this end of the condenser is at a considerably lower temperature (say 20° less) it is easy to see that there will be a large condensation of vapor at this point. Not only must there be no material "jump" in temperature between the cooling surface and the entering vapor, but the same conditions must hold for the exit vapor and its cooling surface. In fact throughout the condenser, for an efficient condensation, the change in temperature of the vapor from its entrance to its exit must be uniform—theoretically it should be differential from point to point. The heat conductivity of glass being so small, the temperature on the outside of the glass spiral is considerably higher than the temperature of the cooling water at that point, and for this reason a metallic spiral should be used. Silver would be preferable, but copper or even tin serves this purpose very well.

An apparatus for laboratory use for the separation of miscible liquids by fractional distillation is sketched in Fig. 4. The condenser, which can be very easily and quickly, as well as cheaply, made, consists of an adapter tube with a small copper coil placed within. The copper return is covered with a glass tube to prevent condensation except along the surface of the spiral. The ordinary laboratory still-head, such as the Hempel, rod-and-disc, evaporator, etc., are fragile and expensive, and the enrichment of the vapor depends entirely upon condensation by air conduction (in some cases there is a very slight enrichment due to rectification). On account of the poor conductivity of glass the capacity is exceedingly small, the ordinary delivery of final condensate being about one drop per second. By using an adapter tube with copper spiral and regulating the temperature and rate of the entering cooling water to the proper values as explained above, 25 cc./min. or more of final distillate may be obtained with equally efficient separation.

The results with the small glass apparatus were so encouraging that it was decided to build a larger differential condenser and to try the idea on as large a scale as could be conveniently handled in this laboratory. The diagram of this differential condenser is shown in Fig. 5. The condenser consisted of a



Fig. 5.

galvanized iron pipe, 3" inside diameter and 4 ft. long, with reducing caps, 3"-1", at the ends. The spiral



consisted of 20 feet of copper tubing 1/4" o. d. No. 20 gauge, wound to fit inside the galvanized pipe. The condenser and all communicating tubes and pipes were heavily lagged with steam pipe insulation. In the first experiments the cooling water was circulated by gravity with a head of about 8 1/2 feet, the water being heated by live steam in an elevated barrel of thirty gallons capacity. Even with suction the circulation was not as fast as was desired in some experiments, and so an iron tank of 60 gallons capacity was used as a reservoir, in which the water was heated by steam to the correct temperature, and by means of compressed air was circulated at the desired rate. In the later experiments an electrically driven gear pump was used and proved very satisfactory. A comparatively small amount of water could be used over and over, and the temperature was regulated simply by slightly cooling the exit water by radiation. By inserting resistance in the armature circuit of the motor, any desired rate could be had and maintained constant.

The still for supplying the vapors was of copper, 20 liter capacity with the bottom steam-jacketed. The still and entire steam system from calorimeter to exhaust was thoroughly insulated. However, when steam was used as a heating medium it was found impossible to maintain a constant rate of vapor even with a needle-valve adjustment and direct atmospheric exhaust, owing to the low quality of the steam and variations in the line pressure.

Electrical heating was finally resorted to and proved entirely satisfactory. An inexpensive heater of simple construction, which gave excellent service, consisted of ten arc-carbons, one-fourth inch diameter and about eleven inches long, mounted in two wooden rings (preferably white pine or cypress) o. d. 7 1/2", i. d. 4". The carbons were connected in series by cast lead lugs, and the bottom was of wood which insulated the heater from the still, and was weighted with lead to keep it firmly seated. 25 amp. at 100 V. through this heater delivered over 75 cc./min. from 15 liters of a low alcohol.

With the single coil differential condenser 76 recorded runs were made and a number of different series are presented in Tables II and III. The amount of cooling water used was not measured in many of the early experiments, owing to the difficulty of accurately measuring it with the circulation system used at that time (gravity and suction). But in later experiments this was done and is discussed in detail further on.

Curves A and B shows the influence of temperature of the entering cooling water on the results obtained, and emphasize the effect of this temperature on the efficiency.

The great importance of having the temperature of the entering cooling water high enough was shown

TABLE II.

Number of run.							Rate <sup>1</sup> of		Eff.
	t <sub>1</sub> .	t <sub>2</sub> .	t <sub>3</sub> .	t <sub>4</sub> .	C <sub>1</sub> .	C <sub>2</sub> .	vapor.		
10 C.....	63.0	89.0	93.0	88.5	53.4	62.4	92.0	73.7	
12 C.....	60.0	91.0	93.0	88.0	47.1	60.0	114.5	51.8	
13 C.....	55.0	90.0	93.0-	90.0+	41.8	51.0	200.0	65.4	
14 C.....	72.5	90.0	94.0	90.5	39.5	52.0	90.0	74.5	
15 C.....	58.0	90.0	94.0+	89.0	38.0	58.0	86.5	26.0	
16 C.....	59.5	91.5	95.0+	88.5	37.0	58.0	90.0	13.8	
18 C.....	69.0	92.0	95.5	89.5	34.0	50.2	46.0	50.7	
19 C.....	61.0	91.5	95.5	91.5	34.0	49.0	77.5	46.0	
20 C.....	74.0	90.0	95.0	86.0	36.8	66.8	50.0	73.3	
21 C.....	76.0	91.0	95.0	90.0-	32.7	55.4	55.4	79.5	
22 C.....	74.0	93.0	95.5	90.0+	33.0	51.0	55.7	52.0	
23 C.....	75.0	91.5	95.5	89.5	37.0	55.4	53.0	75.5	
24 C.....	75.0	92.0	95.5	90.0+	30.6	44.0	75.0	75.0	
25 C.....	72.0	90.5	94.5	90.0	36.4	56.0	70.8	65.6	
26 C.....	78.5	91.0	94.5	89.5	36.1	56.4	62.9	82.0	
27 C.....	77.0	90.0	94.0	90.0	40.5	55.7	68.9	89.0	
28 C.....	78.5	90.0	94.0+	89.5+	39.9	56.4	53.2	91.0	
29 C.....	81.0	90.5	94.5	89.0	36.4	56.4	53.5	88.0	
30 C.....	78.5	90.5	94.0+	89.0+	35.0	57.7	52.0	82.8	
31 C.....	78.5	90.5	94.0-	90.0+	36.7	47.5	77.0	81.5	
32 C.....	79.0	89.5	93.5	89.0	40.9	57.2	69.0	82.0	
33 C.....	80.0	88.0	93.5	86.0	41.9	67.0	41.4	83.6	
34 C.....	78.5	88.0	93.5	87.5	39.6	64.8	36.2	85.4	
35 C.....	78.0-	89.0	93.5	87.5	41.7	62.8	57.8	71.0	
36 C.....	80.0	90.5	94.0+	91.5	36.3	49.7	69.8	90.0	
37 C.....	82.0	90.0-	94.0+	89.0-	37.9	60.5	35.6	85.5	
38 C.....	79.5	90.5	94.0+	91.0	37.6	50.4	48.7	86.1	
39 C.....	80.0	90.0	94.0-	87.5	38.7	64.0	40.6	82.7	
40 C.....	78.5	90.5	94.0+	90.0+	36.1	53.4	59.4	81.7	
41 C.....	78.5	90.5	94.0+	90.0+	34.9	56.3	35.8	79.0	
42 C.....	80.5	90.0	94.0-	90.5	38.1	54.2	67.0	82.7	
43 C.....	81.0	90.0	94.0+	90.5	36.5	54.7	40.4	81.6	
44 C.....	81.5	84.5	89.0-	83.5	58.2	74.3	27.8	100.0	
45 C.....	81.0	84.0	89.0	84.0-	57.3	73.9	32.5	100.0	
46 C.....	79.0	86.0	94.0	82.0+	37.5	73.4	61.5	86.0	
49 C.....	80.8	86.0	93.0	84.0-	45.5	70.1	53.5	92.8	

TABLE III.

Number of run.							Rate <sup>2</sup> of			Eff.
	t <sub>1</sub> .	t <sub>2</sub> .	t <sub>3</sub> .	t <sub>4</sub> .	C <sub>1</sub> .	C <sub>2</sub> .	vapor.	C. W.		
1 E...	74.5	79.0	81.3	79.0	76.0	83.3	50.6	2125	73.6	
2 E...	76.3	78.3	81.8	78.5	76.6	83.5	32.7	2000	57.7	
3 E...	76.4	78.4	81.5	78.0	75.7	83.6	24.4	1600	58.8	
4 E...	76.6	78.1	81.9	78.0	75.2	83.3	23.5	2280	37.1	
5 E...	75.6	79.0	81.5	79.2	76.8	81.4	32.4	1350	73.8	
6 E...	76.0	78.0	81.9	78.0	75.6	85.6	18.1	1275	57.0	
7 E...	76.5	78.1	82.0	78.0	77.0	84.5	19.7	1206	81.9	
8 E...	75.0	78.3	82.3	77.3	75.7	81.7	19.7	1000	38.9	
9 E...	75.0	78.6	82.8	78.0	73.7	84.2	46.6	2740	45.5	
10 E...	77.8	78.0-	82.0	78.0+	79.0	85.8	23.4	3000	100.0	
11 E...	77.6	78.0	82.2	78.9	76.4	82.8	20.0	2980	62.2	
12 E...	77.8	78.0	81.8	79.0	77.3	83.4	17.0	2740	81.4	
13 E...	78.0	79.0	80.0	...	78.8	83.4	15.1	1830	87.6	
14 E...	77.5	78.0	81.4	78.0	71.7	82.5	15.2	2680	96.7	
15 E...	77.5	78.0	82.0	78.5	74.1	83.7	17.8	2625	100.0	
16 E...	77.5	78.0	82.0	79.0	76.5	82.8	17.5	2525	95.4	
17 E...	77.5	77.5	81.2	77.5	79.2	84.3	16.6	2675	90.0	
18 E...	77.5	77.5	81.2	78.5	79.4	83.3	19.2	2480	89.2	
19 E...	77.5	77.5	81.1	77.5	80.4	85.5	15.1	2200	65.0	
20 E...	77.9	78.0	81.0	78.5	80.0	84.4	12.6	2370	100.0	
21 E...	77.5	77.5	81.2	78.0	79.9	84.2	13.2	2525	90.8	
22 E...	77.0	77.0	80.3	78.0	79.9	84.3	10.5	2425	78.0	
23 E...	77.1	77.1	80.1	77.0	80.6	84.4	10.0	2375	85.6	
24 E...	76.9	77.0	80.2	78.0	79.6	84.3	10.5	2640	68.0	
25 E...	76.6	77.0	80.2	77.5	80.8	85.1	12.6	2725	70.0	
26 E...	76.4	77.0	80.1	76.5	80.0	85.6	10.9	1100	50.5	
27 E...	76.6	77.0-	79.9	77.8	81.8	85.5	19.2	1130	96.6	
28 E...	76.7	77.0	80.0	77.6	82.0	85.9	23.0	2630	100.0	
29 E...	76.5	77.0	79.8	75.0	81.2	85.6	15.0	1540	56.2	
30 E...	76.5	77.0	80.0	78.0	83.0	86.0	22.2	1260	100.0	
31 E...	76.5	77.0	80.0	78.0	80.9	86.3	20.5	1720	86.0	
32 E...	76.5	77.0	79.9	78.0	82.4	85.9	26.1	1175	48.5	
33 E...	76.5	77.5	80.1	78.0	81.9	85.9	23.1	1816	90.8	
34 E...	76.5	77.5	80.1	78.0	81.7	85.8	26.8	1725	98.5	
35 E...	76.0	77.0	80.1	76.7	80.9	89.1	18.3	1566	67.0	
36 E...	76.0	77.5	80.2	78.5	81.5	85.9	21.6	1320	83.1	
37 E...	76.0	77.5	80.3	78.0	81.8	85.4	21.4	1018	95.8	

<sup>1</sup> Rate of vapor in cc. per minute.

<sup>2</sup> Rate of vapors and cooling water given in grams per minute.



recently by a case in commercial practice where cold water had previously been used in a partial condenser. When the temperature of the entering cooling water was raised and the amount properly regulated, the yield was double the best that had heretofore been obtained using cold water, and yet the enrichment of the vapors was the same in both cases.

The area of the cooling surface of the coil in the differential condenser was not as large as was desired, and it was feared that some of the vapors could probably pass through the space inside the coil at a high velocity without being properly enriched. Moreover, when the rate of vapor passage was rapid the air pressure required to supply the cooling water was so high that the rate of water circulation could not be kept constant. For these reasons a double coil was substituted for the single coil and was built as follows: A copper coil of the same length, diameter, and gauge as the original one was wound to fit within the first coil and the two coils were brazed together at each end. The second coil was wound left-handed with respect to the first, in order to hold it rigidly in place. The cooling water had two parallel paths of equal resistance to traverse, and thus with the same pressure heretofore used, twice the amount of water could be circulated. Baffle plates were placed within the inner coil to insure a thorough mixing up of vapors, and to prevent any vapor passing through the center of the condenser. The results obtained with this differential condenser are given in Table IV.:

TABLE IV.

Number of run.	$t_1$ .	$t_2$ .	$t_3$ .	$t_4$ .	$C_1$ .	$C_2$ .	Rate <sup>1</sup> of vapor.	Eff
1 F.....	77.3	81.5	88.0	81.3	57.4	77.2	58.5	100
2 F.....	78.5	81.5	88.0	81.8	57.7	75.9	55.0	100
3 F.....	79.2	82.3	88.5	82.5	58.3	74.4	52.9	100
4 F.....	79.0	83.0	91.5	81.0	47.5	77.9	34.4	100

The proper amount of cooling water to use in any differential condensation was determined in the following manner:

Suppose that a weight of vapor  $W_1$ , composition  $C_1$ , enters the differential condenser, is partially condensed, the uncondensed vapor having the weight  $W_2$ , composition  $C_2$ , and the condensate having the weight  $W_3$ , composition  $c_3$ .

We know that

$$W_1 = W_2 + W_3$$

and

$$C_1 W_1 = C_2 W_2 + c_3 W_3.$$

$L_1$  = total heat in vapor, composition  $C_1$ .

$L_2$  = total heat in vapor, composition  $C_2$ .

$H_3$  = heat in liquid, composition  $c_3$ .

$K$  = weight of cooling water.

The total heat must be equal to the sum of its components, and therefore for any case,

$$L_1 W_1 = L_2 W_2 + H_3 W_3 + K(t_2 - t_1).$$

The values for  $L_1$  and  $H_3$  are given in Hausbrand's article.<sup>1</sup> For any specific differential condensation, we have seen that the temperature  $t_1$  and  $t_2$  are fixed,  $t_2$  being slightly below the temperature of the entering vapor and  $t_1$  slightly below that of the exit vapor.

From the thermal equation given above it is seen that a certain definite quantity of heat,  $K(t_2 - t_1)$  per unit of time must be removed by the cooling water, and as the temperature change ( $t_2 - t_1$ ) is fixed, the amount of cooling water  $K$  must be a certain quantity, and can be calculated for each specific case.

In order to check this method the data from a very efficient run was used, and the calculation made as given below:

Rate = 44.5 grams = 0.098 lb. vapor per minute.

$C_1$  = 45 per cent.

$C_2$  = 74 per cent.

Rate cooling water = 5.5 lbs. per min.

$t_2 - t_1 = 9^\circ \text{F}$ .

Maximum yield = 40 per cent.

$c_3 = 25.7$  per cent.

From tables (*l. c.*):

$L_1 = 822$  B. t. u.

$L_2 = 628$  B. t. u.

$H_3 = 119$  B. t. u.

Then,

$$0.098 \times 822 = (0.40 \times 0.098)628 + (0.60 \times 0.098)119 + (5.5 \times 9).$$

$$80.5 \text{ B. t. u.} = 24.6 + 7.0 + 49.5 \text{ B. t. u.} = 81.1$$

In some of the cases given in Table III the thermal equations will not check as closely as in the example given above, but these discrepancies are due to the fact that in series E the temperature differences of entering and exit cooling waters were very small and the temperature could not be read exactly because the thermometers were placed in oil wells. In the numerical example given above the temperature difference was large and therefore the percentage of error in determining  $t_2 - t_1$  was small.

In designing and building a differential condenser for commercial use the following method should be followed: The capacity of the condenser is known and the change in temperature of the cooling water is also approximately known for any change of composition of the vapors from  $C_1$  to  $C_2$ . It is known that the heat transfer must be made in a certain manner to obtain the maximum yield. From tables on the heat conductivity of copper the surface is calculated, and allowing for fairly close-coiling, and several coils being placed one within the other, the size of copper tubing to use is determined approximately. The size of tubes should be chosen of sufficient diameter to conduct easily the amount of cooling water necessary (see tables of flow of liquids through tubes). The weight of cooling water can be calculated as

<sup>1</sup> Rate of vapor in grams per minute.

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



shown above and if the apparatus is operated properly a high efficiency is assured.

A series of experiments were made to determine if the performance of the differential condenser is as efficient at high condensations as it is at low, and the results of this work are given in Table III. The apparatus is more difficult to regulate at the high concentrations, as each corresponding  $c$  and  $C$  lie closer together as the constant boiling mixture is approached and consequently the temperatures of the entering and exit cooling water must be delicately adjusted. In some cases a difference of a few tenths of a degree in the temperature of the entering cooling water materially affected the efficiency in that particular run. Fluctuations in temperature, rate of cooling water or vapor that would not be noticed at low concentrations were troublesome at high. An apparatus in which a composition change of 40 per cent.

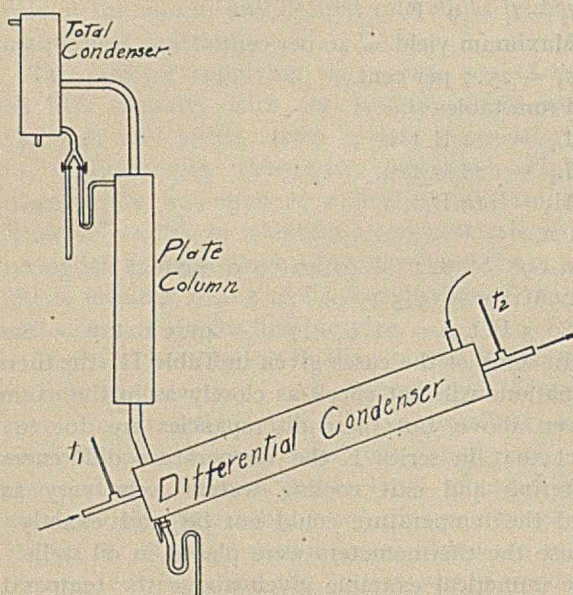


Fig. 6.

can be easily effected at low concentrations may only be large enough to give a composition change of 10 per cent. or less at high concentrations. So if a 190° proof alcohol is desired, the size of the differential condenser may be several times as large as that necessary if only 145° proof alcohol is required, if the passage of the vapor be rapid. Except for the susceptibility of the apparatus to slight changes of temperature and rate, and the increased size of apparatus necessary if the output is large, it is not especially difficult to obtain a high alcohol at a high efficiency by differential condensation. With the single coil apparatus a 186° proof alcohol at approximately 100 per cent. efficiency was obtained.

A differential condenser of medium size (compared with the size of the ordinary dealcoholating and rectifying columns) and large capacity, which will

enrich the vapors from about 25 per cent. to 75 per cent., can be built and run at a high efficiency, but for higher alcohols the differential condenser cannot be so conveniently used for reasons given above. As the high proof of the rectified alcohol is of first importance to many distillers, it was desired to be able to produce as high a proof as was wanted at a high efficiency of operation.

An efficiency test on one of the very latest types of French distilling apparatus was made by the writer, and the thermal efficiency of the rectifying column alone was computed as 40 per cent. of the theoretical maximum, which is high for the average column. The composition of liquid and vapor in different parts of the apparatus was as follows:

Composition of final distillate, 193° (96.5 per cent. by vol.).	
Composition of vapors entering bottom plate, $C_0 = 20.6$ per cent.	
Composition of vapors entering second plate, $C_1 = 58.5$ per cent.	
Composition of vapors entering third plate, $C_2 = 68.0$ per cent.	
Composition of liquid on the bottom plate, $c_1 = 14.2$ per cent.	
Composition of liquid on the second plate, $c_2 = 43.2$ per cent.	

This test showed that the composition change on the bottom plates was very rapid, and as the change from successive plate to plate is done by simple rectification, the yield in this part of the apparatus was poor. The column tested had eighty-eight plates, of which the greater number were used for rectification of high vapors.

The ordinary type of rectifier column can be more efficiently operated if the apparatus is well insulated and if the composition change from plate to plate is small. Ordinarily, this latter would mean that an apparatus twice the size of the one in use for the same composition change and the same capacity would be required, and on account of cost of installation this is prohibitive. Many designers have tried to build apparatus having large capacity and a small number of plates, and the operation depended upon the rapid enrichment of the vapors in the bottom plate. Although the first cost is reduced, it is impossible to obtain a high efficiency with such an apparatus because the total rectification is a series of simple rectifications of large composition change, and the yield is therefore low. In case it is desired to obtain high proof, the last step in concentration can most successfully be accomplished by using a plate column. Above this a total condenser, returning the calculated amount of overflow from the same to the top plate, will usually be found most satisfactory, requiring much less attention at this high proof and yielding results almost as economical as a differential type. This is due to the fact that the composition of vapor and liquid at these high proofs lie so close together. A differential condenser is not suitable for the last step in concentration for high alcohols but can be most successfully used during the earlier steps of rectification.

Fig. 6 is a sketch of a plate column of eight plates



delivering into a total condenser with return-feed to the top plate, the rectifying column being placed above a differential condenser. The proper return-feed from the total condenser is calculated as follows:

It has been shown<sup>1</sup> that a rectifying column is working at its maximum efficiency when the composition of the overflow from the bottom plate is equal to that of the equilibrium liquid of the vapor entering the bottom plate. If the composition of the entering vapor is  $C_o$ , that of the rectified vapor  $C_c$ , then for  $W_o$  pounds of entering vapor the maximum yield of final product  $W_c$  is given by the equation

$$\frac{W_c}{W_o} = \frac{C_o - c_o}{C_c - c_o}$$

The total weight of alcohol mixture of composition  $C_c$  that can be obtained from the weight of entering

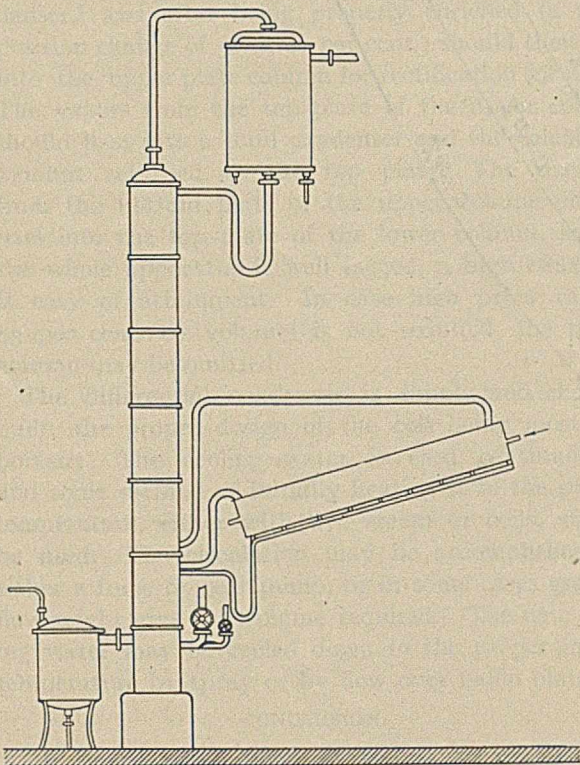


Fig. 7.

vapor  $W_o$ , composition  $C_o$  is calculated, and after subtracting the maximum yield  $W_c$  the remainder gives the amount of distillate of composition  $C_c$  that should be returned to the top plate of the plate column.

The results of a test with the apparatus of Fig. 6 are as follows:

Composition of vapors entering diff. cond. = 45.1 per cent.  
 Rate of vapors entering diff. cond. = 44.5 gram./min.  
 Composition of vapors leaving diff. cond. = 74.0 per cent.  
 Composition of final product from plate column = 85.3 per cent. (by vol. 90 per cent.).

The double coil differential condenser was opera-

Lewis, *Loc. cit.*

ted under conditions which previous tests had shown allowed a very high efficiency to be obtained.

Forty and five-tenths per cent. of the vapors condensed in the total condenser were returned to the

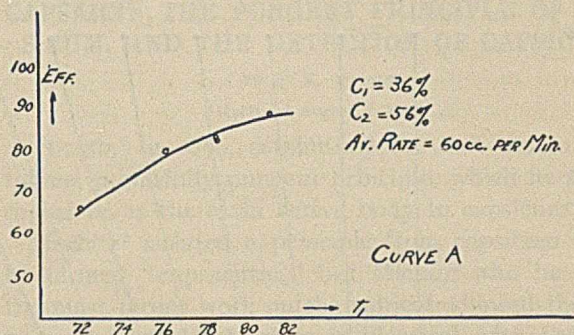


Fig. 8.

top plate. The theoretical amount to be returned for a composition change from 74 per cent. to 85.3 per cent. was 38.8 per cent., calculated from the formula:

$$\text{Max. yield (for plate columns)} = \frac{C_o - c_o}{C_c - c_o}$$

The actual yield of final product was 22.86 grams in  $2\frac{1}{2}$  min.; 111.35 grams of 45.1 per cent. vapor entered the condenser, and the maximum yield (40 per cent. of the weight of entering vapor) = 44.56 grams of 74 per cent. vapor entered the plate column, and was enriched to 85.3 per cent. in that space of time ( $2\frac{1}{2}$  min.).

The maximum yield of the final product was:

$$W_c = W_o \frac{C_o - c_o}{C_c - c_o} = 44.56 \frac{0.74 - 0.59}{0.853 - 0.59} = 44.56 \times 0.57 = 25.4 \text{ gm.}$$

The yield obtained was 22.86 grams.

$$\text{Efficiency of total operation} = \frac{22.86}{25.7} = 90.2 \text{ per cent.}$$

The apparatus was run in this manner for several consecutive days for about 10 hours daily, and the performance was perfectly steady and constant at all times. The plate column used had only eight plates,

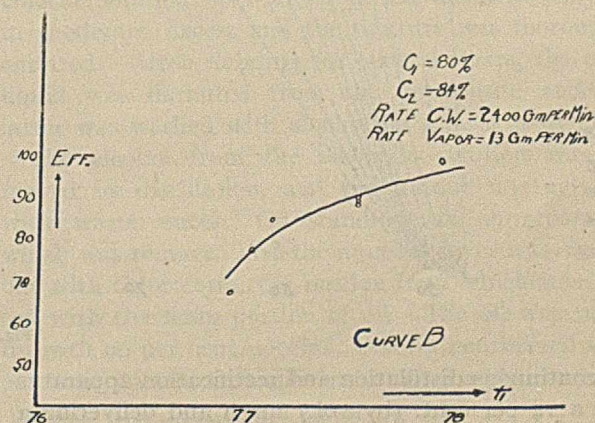


Fig. 9.



but if enough plates are used, and this method carefully followed, an alcohol approaching the composition of the constant boiling mixture can be obtained. The apparatus as run in the above test corresponded

apparatus being 90 per cent. of the theoretical maximum.

It was shown<sup>1</sup> that a differential condenser placed above a plate column and returning the differential

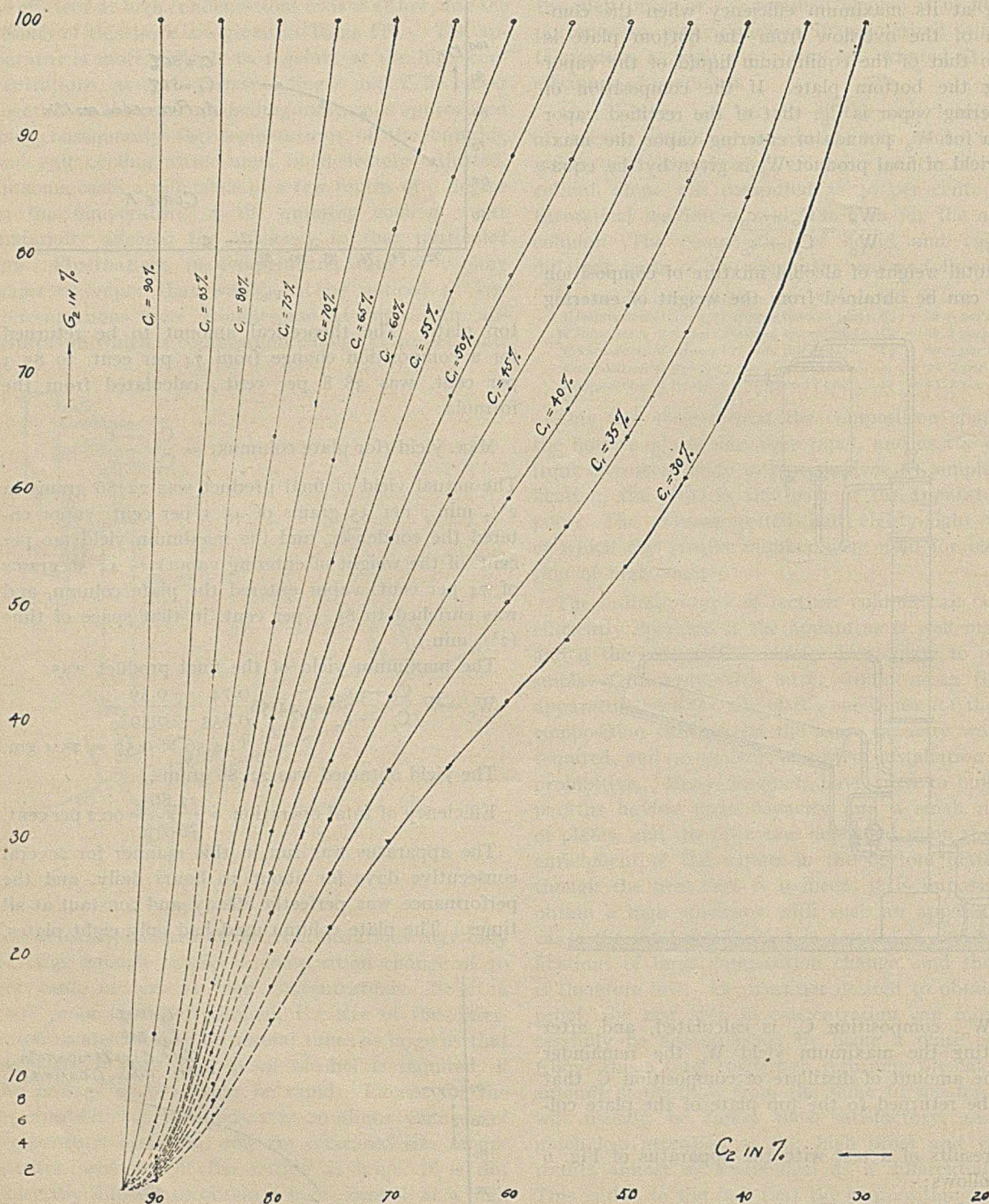


Fig. 10.

to a continuous distillation and rectification apparatus using a 14 per cent. (by vol.) mash and delivering a 90 per cent. (by vol.) alcohol, the performance of the

condensate to the top plate reduces the number of plates necessary to a minimum and enables the

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



operation of the plate column to be made at a high efficiency. The condensate from a differential condenser should never be returned directly to the still, but should always be passed through a short plate column which removes the maximum amount of the volatile component from the condensate, and the overflow from the bottom plates is of approximately the same composition as the liquid in the still—the condition of maximum efficiency.

Fig. 7 shows the arrangement which should be used to increase the efficiency of commercial rectifying columns. The rectifying column is made into two columns, the lower one being rather short, by placing a solid metal sheet between two successive plates. The lower column should contain fifteen or twenty plates,<sup>1</sup> and the vapors from the top plate of this short column should pass into the differential condenser,<sup>2</sup> and after being properly enriched (a composition change of 50 or 60 per cent.) should then pass into the upper plate column for rectification for proof. The vapors from the top plate of the upper column should pass into a total condenser and the calculated amount returned to the top plate. The overflow from the bottom plate of the upper column should pass into the top plate of the lower column, and if the whole apparatus is well lagged, a high efficiency is easy of attainment. In case high proof (above 85 per cent. by volume) is not required, the upper column may be omitted.

The differential condenser is simply and cheaply built, the proper design of the coil being most important. The cooling water is used continuously, and some method of initially heating it to the proper temperature, either with live steam or coils, should be used. The circulation may be accomplished by either a force or gear pump, or in some cases gravity flow might give the volume required. The exit cooling water may be cooled down to the proper intake temperature by spray or by flow over baffle plates.

#### CONCLUSION.

An apparatus has been developed for partial condensation of mixed vapors, to be used in conjunction with a plate rectifying column below it, and if necessary with a further column above it, which will:

(1) Materially increase the thermal efficiency and capacity of commercial types of rectifying apparatus, and

(2) Decrease the initial cost of the same by decreasing the length of the plate towers used.

Extensive experimental results have been submitted to prove the accuracy of the deductions and conclusions, and to furnish data for the proper design

<sup>1</sup> The correct methods of design for such a column can be found in Lewis' article.

<sup>2</sup> It will be noticed that in Fig. 7 the differential condenser is tipped so that the condensate flows towards the entering vapor. This is for the purpose of preventing any depletion whatever of the vapor in volatile component, although the condensate is removed as fast as formed from the sphere of action by tubes shown in the sketch.

of such apparatus; the methods and calculations for such designs have been outlined.

## CAPSAICIN, THE PUNGENT PRINCIPLE OF CAPSICUM, AND THE DETECTION OF CAPSICUM.

By E. K. NELSON.

Received August 1, 1910.

Thresh,<sup>1</sup> in 1876, established the fact that a crystalline, powerfully pungent principle, which he named capsaicin, is the main active body in capsicum.

Morbitz<sup>2</sup> isolated a principle from capsicum which he termed "capsacutin," but Micko,<sup>3</sup> who has done the most recent work on the subject, showed that the body isolated by Morbitz is identical with the capsaicin of Thresh, being probably an impure form of the latter body.

Micko developed a process for the isolation of capsaicin and proved it to be the pungent principle of pepper (*capsicum fastigiatum*), but that *capsicum fastigiatum* contains about twenty times as much capsaicin as does *capsicum annum*.<sup>4</sup>

In view of the fact that the detection of capsicum is desirable in those cases where it is used to fortify pungent beverages, such as ginger ale, it seemed desirable to isolate some of the pure principle for the purpose of studying some of its properties.

The method of Micko was employed, and while extremely tedious and difficult, it is apparently the only way at present known whereby the body can be satisfactorily separated from the fatty and resinous bodies with which it is associated.

Fifteen hundred grams of ground African cayenne were completely exhausted with ether. The ether was distilled off and the residue, a red, very pungent oil, was dissolved in 1 liter of 90 per cent. alcohol, made slightly alkaline with alcoholic potash, and well agitated. A portion of the oil remained undissolved. This was separated, agitated with 90 per cent. alcohol, and the alcoholic solutions, which contained the pungent principle, were united. Barium chlorid solution was added to the alcoholic solution in moderate excess and the mixture was thoroughly agitated. After standing for several hours, the clear liquid was decanted from the precipitate and the latter was washed with alcohol on a glass wool filter.

The alcohol from the alcoholic solution was removed by distillation, and the residue was agitated with warm water. On standing, an oil separated, which was removed and the aqueous layer was shaken out with chloroform, the residue from which was united with the main portion of oil. The oil was taken up with 90 per cent. alcohol, exactly neutralized with alcoholic potash, and sufficient alcohol added to bring

<sup>1</sup> *Phar. Jour. and Trans.*, 3, 7, 21, 259, 473.

<sup>2</sup> *Phar. Zeitsch. f. Russland*, 1897, 369.

<sup>3</sup> *Zeits. f. Unters. d. N. u. Genussm.*, 1898, 818.

<sup>4</sup> *Ibid.*, 1899, 411.



the volume to two liters. Alcoholic solution of silver nitrate was then added, while stirring, in moderate excess. A brownish red, very voluminous precipitate was thrown down which was filtered off and washed with alcohol until the pungent principle was completely removed.

From the filtrate silver nitrate was removed by adding sodium chlorid in aqueous solution, and filtering. The alcohol was distilled from the filtrate, the residue mixed with water and the oil separated. This oily material was then dissolved with an excess of five per cent. potassium hydrate solution and carbon dioxid passed into the solution until it was completely saturated with that gas.

This caused the separation of an oily layer which was removed by shaking out with ether. The residue from the ether was put into an eight-liter flask, mixed with 100 cc. of five per cent. potassium hydrate, diluted with three liters of water, heated to 60°, and allowed to stand overnight at room temperature. The alkaline, slightly colored, clear solution was siphoned from the oily, insoluble material left adhering to the walls of the flask.

The above process was repeated until pungent bodies were completely removed from the residue. The alkaline, clear solutions thus obtained were united in a large flask, heated to 60° and barium chlorid solution, in sufficient excess, was added, while stirring.

On standing, the precipitate settled out and the clear, supernatant liquor was removed. The precipitate was repeatedly treated with dilute potassium hydrate solution and subsequent precipitation with barium chloride until it was free from pungent bodies. The clear, filtered solutions from the barium precipitate were acidified (whereupon they became milky), and shaken out with chloroform.

The chloroform was evaporated and after standing for some days the residue began to crystallize in rosettes and set finally to a solid, beautifully crystalline mass. The crystalline material had a brownish yellow color and was contaminated with some oily material. It was spread on a porous plate and the oil and much of the color absorbed. The crude crystals were dissolved in carbon bisulphid, a gummy residue remaining undissolved.

The carbon bisulphid was evaporated in a flask and the residue boiled out with petroleum ether under a reflux condenser, decanting the petroleum ether extracts into beakers.

Upon cooling, pearly leaflets separated from the petroleum ether solutions.

The body thus obtained was perfectly pure capsaicin, in the form of pearly white leaflets.

All residues and liquors were carefully examined for other bodies of a pungent nature with negative results. Hence, it is concluded that capsaicin is the only pungent principle of capsicum. An alkaloidal

substance, capsin, possessing a conin-like odor, is present in minute amount, but it is not pungent.

From 1500 grams of selected African pods (*capsicum fastigiatum*), 2.13 grams of pure, crystalline capsaicin were obtained, representing 0.14 per cent. of the original material. The crystals melt at 64.5° (Micko's capsaicin melted at 64°). The properties of the body agree well with those described by Micko.

Capsaicin is not an alkaloidal body but possesses some of the properties of the phenol group. Micko has shown that it contains a methoxy group and a hydroxyl group and from analysis and molecular weight determinations ascribes to it the formula  $C_{18}H_{28}NO_3$ . The enormous pungency of capsaicin can be appreciated only by those who have been brought intimately in contact with it. One drop of a solution containing 1 part in 100,000 causes a persistent burning on the tongue. A drop of a solution of 1 part to a million imparts a perceptible warmth.

Now one drop of a solution of one part per million contains at the most 1/20,000 of a milligram of capsaicin, an amount not only unweighable, but extremely difficult of detection even by the most delicate of chemical reagents. The only test based on chemical reaction indicating capsaicin on record, to the writer's knowledge, is that described by Micko,<sup>1</sup> and is as follows: An alcoholic solution of capsaicin acidulated with hydrochloric acid is treated with an excess of platinic chlorid; no precipitate is formed. On spontaneous evaporation of this mixture, however, a characteristic vanilla-like odor is developed after some time.

To test the delicacy of this reaction, weighed amounts of capsaicin in alcoholic solution were treated with platinic chlorid and hydrochloric acid, as directed, with the following results:

- 4/10 mg. gave no odor.
- 2 mg. gave a slight odor which could not be described as the odor of vanilla.
- 4 mg. gave a slight vanilla odor.
- 10 mg. gave a distinct vanilla odor.

Thus it seems that this method, depending on the sense of smell, is entirely valueless for the detection of small amounts of capsicum. Careful search was made for a microchemical reaction which could be utilized for the detection of small amounts of capsaicin, but none was found.

The following test indicates the presence of capsaicin: On treating a small amount of capsaicin with a dilute neutral solution of ferric chloride and a little alcohol, an evanescent greenish blue color is given. This reaction is neither characteristic nor delicate, and is therefore of no value for analytical purposes.

When capsaicin is dissolved in sulphuric acid and a particle of sugar added, a beautiful violet color de-

<sup>1</sup> *Zeits. f. Unters. d. Nahr. u. Genussm.*, 1898, 818.



velops on standing, a reaction, however, which is afforded by other substances.

The detection of small amounts of capsicum therefore appears to be restricted to the physiological action of the capsaicin.

Garnett and Grier<sup>1</sup> were the first to describe a method for the detection of capsicum in ginger preparations, based on this reaction. The pungent body of ginger (gingerol) is destroyed by them by heating on the water bath with alcohol potassium hydrate.

The pungent substance of ginger is not, however, completely destroyed by this method, as can readily be observed by the very perceptible burning taste left after treating tincture of ginger by their method. Furthermore, the residue obtained according to this method is quite considerable, as ether is used for the extraction, and it dissolves the oleo-resins of ginger, thus diluting the pungent body in the residue and rendering the test less delicate.

A method is here proposed which departs from the Garnett and Grier method in important particulars and which is described as follows:

Ten cc. of a tincture, or the ether extract from 100 cc. of a beverage (from which any alcohol present has been evaporated previous to extraction with ether) are treated in a porcelain dish with 10 cc. double normal alcoholic potassium hydroxide and evaporated on the steam bath.

About  $\frac{1}{10}$  grain powdered manganese dioxid is added, 5-10 cc. water, and the heating on the bath continued for about 20 minutes, or until volatile oils are expelled. Cool, acidify with dilute sulphuric acid and extract at once with petroleum ether. Evaporate the petroleum ether in a small crucible, keeping the residue within as small an area as possible and heating on the steam bath for about five minutes. The residue will be very small and in some cases scarcely visible, and it will contain all the capsaicin from capsicum, mixed with a minimum amount of extractive matter. Apply the tongue to the residue, being careful to keep the material on the tip of the tongue. If capsicum is present the characteristic burning sensation will soon be felt.

If but a minute amount of capsicum is present, it may take a few minutes for the reaction to develop. In some cases it may be necessary to apply the entire residue to the tongue.

From the nature of the substance it is desirable to allow an interval of time between the several testings. Failure to follow this course may result in confusion.

The following data will show the delicacy of the test where capsicum in small amount is mixed with ginger:

Tr. ginger	U. S. P.	10 cc.	} No capsicum found.
Tr. capsicum	U. S. P.	none	
Tr. ginger	U. S. P.	10 cc.	} Capsicum detected by placing entire residue on tongue.
Tr. capsicum	U. S. P.	1/100 cc.	
Tr. ginger	U. S. P.	10 cc.	} Capsicum taste very pronounced.
Tr. capsicum	U. S. P.	1/10 cc.	
Tr. ginger	U. S. P.	10 cc.	} Capsicum taste, extremely hot.
Tr. capsicum	U. S. P.	5/10 cc.	

BUREAU OF CHEMISTRY,  
WASHINGTON, D. C.

## ASAFETIDA.

By W. A. PEARSON.

Received August 1, 1910.

During the past few years so much analytical data has been presented on this subject that I hesitate to increase the voluminous records. However, after the unpleasant and tedious work has been accomplished it affords some relief to give others what little benefit may be derived from it.

When the Federal law first took effect there was a marked and prompt improvement in the quality of consignments of asafetida, but after a few inferior lots had failed to be deported the European merchants were quick to declare that the highest grades could not be obtained in sufficient quantities. Possibly the ultimate motive for this attitude was to influence the Revision Committee of the Pharmacopeia to adopt lower standards.

*The Great Variation Due to Sampling of Gum Asafetida.*—Every one is acquainted with the difficulty of obtaining representative samples of gum asafetida. An actual illustration may well be given.

A sample consisting of several lumps was pounded in an iron mortar till fairly uniform and the ash amounted to 24.9 per cent., the alcohol-soluble 50.86 per cent. The same case was later resampled and the average of four results obtained indicated 54.5 per cent. of ash and only 23.2 per cent. of alcohol-soluble material, almost the reverse of the results formerly obtained.

Samples from other cases in the same lot contained 27.6 per cent. alcohol-soluble, 50.9 per cent. ash, 27.5 per cent. alcohol-soluble and 52.9 per cent. of ash.

*Collection of Samples.*—An attempt was made to get representative samples by taking portions of several lumps from each case. If the total sample was much over one pound, half of each piece sent for examination was taken. The samples were always pounded in an iron mortar until the material could be mixed with the hands and was of fairly uniform consistency. The degree of variation obtained with this material by two chemists can be seen from the following table:

<sup>1</sup> *Pharm. Jour. and Trans.* (3), 12, 721.



## PER CENT. OF ASH.

## Sample No. 1.

	Per cent.	Chemist.
Determination No. 1.....	58.8	H. M. S.
Determination No. 2.....	51.1	H. M. S.
Determination No. 3.....	55.4	W. A. P.
Determination No. 4.....	52.5	W. A. P.
Average.....	54.5	

## Sample No. 2.

Determination No. 1.....	52.5	H. M. S.
Determination No. 2.....	51.1	H. M. S.
Determination No. 3.....	52.0	W. A. P.
Determination No. 4.....	47.8	W. A. P.
Average.....	50.9	

## Sample No. 3.

Determination No. 1.....	53.3	H. M. S.
Determination No. 2.....	54.4	H. M. S.
Determination No. 3.....	54.7	W. A. P.
Determination No. 4.....	49.4	W. A. P.
Average.....	52.9	

## AMOUNT OF ALCOHOL-SOLUBLE MATERIAL.

## Sample No. 1.

	Per cent.	Chemist.
Determination No. 1.....	20.3	H. M. S.
Determination No. 2.....	24.2	H. M. S.
Determination No. 3.....	25.6	W. A. P.
Determination No. 4.....	22.8	W. A. P.
Average.....	23.2	

## Sample No. 2.

Determination No. 1.....	28.0	H. M. S.
Determination No. 2.....	29.0	H. M. S.
Determination No. 3.....	27.3	W. A. P.
Determination No. 4.....	26.1	W. A. P.
Average.....	27.6	

## Sample No. 3.

Determination No. 1.....	29.4	H. M. S.
Determination No. 2.....	27.5	H. M. S.
Determination No. 3.....	26.1	W. A. P.
Determination No. 4.....	26.0	W. A. P.
Average.....	27.3	

*Method of Determining Per Cent. of Alcohol-soluble Material.*—The amount of alcohol-soluble material was determined by shaking about ten grams of the sample with 150 cc. of alcohol in a mechanical shaker for several hours. The insoluble material was collected on tared filter paper and well washed with an excess of alcohol before drying to a constant weight at 100° C. It has been suggested that this residue be taken after weighing for the determination of ash, but several obvious reasons make this method objectionable.

The results given above were obtained in the usual routine examinations and show the extent of the variations that may be expected.

*Powdered Asafetida.*—Powdered asafetida theoretically should conform to the same standards as the gum asafetida, but every one who has attempted to powder it knows that it is not convenient to reach these standards on account of the moisture and volatile matter that is present. If it is dried before powdering, considerable of the volatile material is lost.

The following records given by Mr. F. P. Sher show the loss of asafetida during drying and powdering:

Lot No. 1, 2032 lbs.	Returned after powdering, 1630 lbs.	Loss, 402 lbs.
Lot No. 2, 1055 lbs.	Returned after powdering, 844 lbs.	Loss, 211 lbs.
Lot No. 3, 515 lbs.	Returned after powdering, 448 lbs.	Loss, 267 lbs.
Lot No. 4, 936 lbs.	Returned after powdering, 697 lbs.	Loss, 239 lbs.
Total, 4538 lbs.		3619 lbs. 1119 lbs.

The average loss in drying is 24.6 per cent., and this loss is almost entirely the volatile and alcohol-soluble portion, which loss indirectly increases the ash and alcohol-insoluble material in the finished product.

For example, if 100 lbs. of asafetida containing 50 per cent. insoluble material and containing 15 per cent. of ash, be powdered and lose 20 per cent. in powdering, the resulting 80 lbs. of powder will still contain 50 lbs. of insoluble material, or 60.2 per cent., and it will still contain 15 lbs. of ash, or 18.7 per cent.

It can be easily seen that powdered asafetida contains considerably more ash and considerably less soluble matter. The analytical results on powdered samples will not vary nearly as much as on the samples of gum, because of its comparative uniformity.

The following data illustrates the variation obtained in determining the per cent. insoluble in alcohol of the powdered asafetida:

## INSOLUBLE IN ALCOHOL.

## Lot No. 1.

	Per cent.
Determination No. 1.....	59.00
Determination No. 2.....	58.84
Determination No. 3.....	59.26
Determination No. 4.....	58.88
Average.....	58.99

## Lot No. 2.

Determination No. 1.....	51.40
Determination No. 2.....	52.92
Average.....	52.16

## Lot No. 3.

Determination No. 1.....	83.08
Determination No. 2.....	82.52
Average.....	82.80

*Ash Determinations.*—It is frequently noticed in making ash determinations that lower results are obtained by using a blast lamp and also if the length of time ash is heated is increased. It is not wise to take a very small sample, particularly of the gum for ash determination, because the presence or absence of a single small stone will make the results widely divergent. On the other hand, a large amount of ash requires a great amount of heat and must be heated a long time before it becomes perfectly white.

To ascertain the amount of variation due to method of heating, the following ash determinations were made upon three samples of powdered asafetida:

	Sample No. 1. Per cent.	Sample No. 2. Per cent.	Sample No. 3. Per cent.
Determination No. 1....	36.11	29.25	46.3
Determination No. 2....	32.08	28.20	45.1

The first determinations were made by heating



about ten grams in a platinum dish over a Bunsen burner for five hours. The second determinations were made by heating about ten grams in a nickel crucible for fifteen minutes with a blast lamp.

The above data are presented not only to show the variations due to sampling and assaying, but also to give the retail pharmacist some reason for the lack of uniformity of his tincture of asafetida. Perhaps the present method of valuation is far from ideal, but certainly some definite standard for the amount of soluble constituents in the tincture should be insisted upon.

Acknowledgment are due Mr. H. M. Sechler for considerable of the data here presented.

ANALYTICAL DEPARTMENT,  
SMITH, KLINE & FRENCH CO.

## ADDRESSES.

### CHEMISTRY IN THE BUREAU OF STANDARDS.

By W. F. HILLEBRAND, Chief Chemist.

Received September 7, 1910.

I have felt impelled to speak on the subject "Chemistry in the Bureau of Standards," for the reason, mainly, that since my connection with that bureau little published evidence of chemical activity has been offered. The impression might arise and spread that proper advantage is not taken of the opportunities provided. It is my wish to forestall such an opinion. That the public closely interested in chemistry has a right to be informed on the subject goes without saying, for the bureau is a federal institution where work is expected to be directed chiefly toward providing the public with authentic information on a variety of subjects. This information must be of value for the most part to the industries, otherwise the bureau fails in its chief function. From its inception the bureau has endeavored to meet the most insistent demands upon its reservoir of physical knowledge, and at an increasing rate as facilities were given it for so doing. That it has been in the main eminently successful in this respect, is sufficiently attested by the support it has at all times received from Congress and by the marked growth it has experienced and is experiencing, a growth that would be impossible without strong backing, that is, the approval of an influential section of the community both industrial and educational, manifested through its representatives in the national capitol.

The growth of the chemical side of the bureau has been slow, but it is now increasing rapidly at a rate which bids fair to call for a special building within a couple of years. At the start it was not clear, apparently, along what lines the chemical work of the bureau might best develop and I was myself in doubt for some time. The viewpoint of my predecessor, Dr. Noyes, that research without direct practical bearing should not be elbowed out of view by the demands of the industries, is one that should need no defense before a gathering of this character. It is a view that is held by the director of the bureau as well as by myself. Nevertheless, during the interval between the withdrawal of Dr. Noyes from the bureau and my own transfer to it, the work of the small chemical force was wholly along practical lines. The demands of one and another of the government bureaus and departments for help in the preparation of specifications for materials in which they were vitally interested had become insistent. Dr. Stokes, the associate chemist in charge, was deeply impressed with the importance of the numerous problems presented and devoted himself with ardor and great ability to their solution.

He had mapped out extensive lines of investigation and had initiated several different researches of a most difficult nature. Unfortunately, his withdrawal from the bureau has hindered the prosecution of some of this work for lack of a directing spirit with full comprehension of the subject in its different bearings. At the same time it was felt, in view of the obscurity in which some of the questions were involved and the length of time needed for their study and the uncertainty of a successful issue, that our chief attention should be centered for the present on other lines of work where success was practically assured and the immediate value to the community was plainly evident.

The most important of these problems was a great extension of one already initiated under Dr. Noyes, namely, the providing of carefully analyzed materials by which chemists can check the accuracy of their analytical methods and employers can control the work of their employees. The usefulness of these materials is not confined to those employed in the industries but extends equally to educational institutions. To you, being chemists, it is hardly necessary to offer any arguments in support of the use of such standardized materials. The subject is treated briefly, but with sufficient fullness, in a publication which has been issued only very recently as a circular of information regarding the samples that have been provided thus far by the bureau or are in immediate preparation, some thirty-seven in all. The number will be very largely added to, for there is an ever increasing demand in this direction, and I feel that the bureau can do no greater service to chemists of the industries at present than by catering to this demand. Not only will benefit result from the use of the samples themselves but also from the experience that the bureau chemists will gain, and have already gained, in their close study of the methods employed in analyzing them. To this feature I will revert later.

The list of samples in stock comprises three pig irons, 5 Bessemer steels, 5 basic open-hearth steels, 6 acid open-hearth steels, and 1 vanadium steel, to which will be added, as soon as analysis can be made, a nickel steel, a chrome-vanadium steel, and a chrome-tungsten steel. Of ores there are an argillaceous limestone, suitable for the manufacture of Portland cement, a zinc ore, and 3 iron ores from the Lake Superior region. It is impossible to find any single ore from this region that can advantageously be used as a standard for all the constituents that are ordinarily determined in an iron ore. Hence, the multiplication, one being used for iron, phosphorus and silica, the second for alumina, lime and magnesia, and the third for manganese. A titaniferous iron ore from New Jersey is in process of analysis, and it is proposed to provide later one typical ore from the Alabama region. Possibly typical ores of lead and zinc from the Rocky Mountain region will follow.

It is to be said here, however, that the selection of ores that can be used advantageously as standards for analysis offers far greater difficulty than that of alloys, for the reason that they are almost always so subject to changes in moisture content from day to day or at different altitudes, as to detract very much from their value. If their sensitiveness to hygrometric variations is at all pronounced it is almost hopeless to expect that all chemists will be able to reduce them to a fixed water content, no matter how precisely formulated may be the directions for drying. The moisture content may vary somewhat without detriment so far as the minor constituents are concerned, but not so for those present in large amounts.

We have also a pure sugar for use as a standard in calorimetric and saccharimetric work, and studies are in progress looking to the applicability of other standards for the measurement of heat of combustion, such as benzoic, salicylic, phthalic and hippuric acids, naphthalene, anthracene and camphor. This work is done in co-operation with the heat division of the bureau.



The American Brass Foundrymen's Association has enlisted our aid in the preparation of standard brasses and bronzes, and a beginning has been made with two brasses, which are in preparation.

We have undertaken to aid the fertilizer interests by carefully analyzing one or more typical phosphate rocks and by testing at the same time the relative values of the different methods in common use, especially with respect to the influence on them of such interfering constituents as are common to materials of this class—pyrite, fluorine and organic matter.

I could easily devote an hour to the problems encountered in the selection and preparation of the samples themselves and several more to those connected with their analysis. An enormous volume of correspondence on these points has accumulated. We endeavor to keep in close touch with prominent manufacturers, and especially with prominent chemists, soliciting their advice and criticism. Almost without exception our efforts have met with hearty approval and support.

It may be well to introduce here some remarks relative to the plan we have hitherto followed in obtaining the composition of our samples. It is a plan of co-operation, in which leading works chemists and commercial chemists are invited to participate. The almost inevitable result is failure of satisfactory agreement in the reported results. Ordinarily we have given opportunity to those whose results differed markedly from the mean to revise their work without indicating to them the direction or magnitude of the deviation. This course has often resulted in much loss of time and necessitated much correspondence. For certain cases, and eventually perhaps for all, the bureau may decide to depend solely on its own chemists to establish the composition of a given material, but for some time to come this plan will be employed to a limited extent only. The bureau must first afford to its analysts sufficient time to become masters in their particular fields to an extent that will command general confidence. Until this condition is fulfilled it is far better policy to share the responsibility with experts outside of the bureau. The certificates which the bureau issues with its analyzed samples represent, therefore, the best efforts of a considerable number of analysts who are supposed to be especially expert in the analysis of the given material, and the mean undoubtedly gives in most cases a close approximation to the correct composition. The bureau does not, however, undertake to vouch for their correctness. The results by different analysts and by different methods are reported in such a way that the person using them can see at a glance what variations are to be expected in commercial work done by supposed experts and under conditions that are presumably the best that are to be encountered in commercial laboratories.

In certain cases, such as pig iron and the ordinary steels, the methods commonly used and the precautions observed are of the highest known order. The commercial results on the average are, therefore, to be accorded fully as great weight as those obtained in the bureau. It is not always so with other materials, when the character of the reagents or the apparatus play a more important part, and when the complexity of composition, as in ores, introduces special difficulties in the way of effecting complete separation of the constituents and their accurate determination. In special cases the bureau regards its own values as closer to the truth than the mean of any number of outside determinations. This is true, for instance, of the alumina and magnesia in one of the Lake Superior iron ores and even in the value for iron itself in another of them.

One without experience can form little idea of the difficulties and delays that have been encountered, from the initial selection of one of our samples through the machining, grinding, mixing, analyzing, and assembling of data to its final issuing. It took us nearly a year to get the bars to replace one of our

steels of which the supply gave out. The steel company that undertook to fill the order had to make five castings before a product of the right composition was obtained. Once delivered to the bureau, it takes from two to three weeks to reduce the sample of steel or iron to fine chips. These sometimes have to be ground finer in a special machine and in any case must be most thoroughly mixed. The successful mixing of 300 or 400 pounds of such a material requires a special mechanical mixer and no one of those on the market is entirely satisfactory. The one we have used we modified materially and are now having one made that works on a different principle entirely.

We have experimented somewhat with a view to securing homogeneous samples of relatively low melting alloys and metals in a state of sufficient division, without the use of a lathe, and have some hopes of eventual success.

Much time has to be expended by us in analyzing these materials, particularly the ores, brasses and special steels, for which the commercial methods are either more or less inadequate or have not yet received the extended study and testing that has been given to those for irons and plain steels. It is to be borne in mind that we must not be content with a single determination or with a few determinations by a single method, but that our problem is to ascertain just as closely as is possible the true composition of these complex materials. Literally months of time may thus be consumed in the study of a single sample of a new kind. The knowledge thus gained, however, shortens by far the time required for later analyses of similar or related samples. In acquiring this knowledge and in testing the various commercial methods against each other, we have already made useful observations that will be of value to others as well as to ourselves. We shall not hasten to publish our observations, but wait until we have had full opportunity to verify them repeatedly. Much information on certain of our own methods and in less degree on those used by others is to be found in our recently issued circular—"Methods of Analysis for Iron and Manganese Ores."

In addition to the above, we have taken the preliminary steps looking toward the issuance by the bureau of a sodium oxalate for the standardization of solutions to be used not only in oxidimetry, but in acidimetry as well. Our reasons for making choice of sodium oxalate I need hardly go into now, save to say that for oxidimetry it seems to be the most satisfactory material that can be had in considerable quantity. When pure (and the different makes on the market are not to be taken at their face value, but must be tested by each buyer) the article is stable and eminently satisfactory as a standard. The use of so-called piano wire as a basis for standardizing permanganate and bichromate solutions cannot be too strongly condemned. Electrolytic iron is difficult to prepare and it too is not as pure as could be wished. The chief difficulty before us is to secure from manufacturers an article of sufficient purity in the large amounts called for. If found, it will be tested by us and issued under our seal. Should success attend our efforts in this case, we may go farther.

Having thus dwelt at some length on that feature of our work that is likely to appeal most strongly to chemists as a whole, I will pass over others more briefly.

Various bureaus of the government have called on the Standards Bureau for assistance. These requests range from simple requests for tests of materials to those that involve more or less extended research. Six months were required for a research on pyroxylin plastics with reference to their acceptance or rejection as freight on American passenger vessels. The Treasury Department wants information to enable it to draw specifications for its various record inks and to improve their quality. The Government Printing Office needs similar information in regard to its papers and inks. In fact the Standards Bureau has for



some years done the greater part of the physical and chemical testing of papers for that great printing establishment, and frequently analyzes the metals and alloys that enter into the composition of type metals or find direct use in printing. This work requires not only the time of one chemist for the routine work but of another for research connected with paper.

The following list embodies tests that were made by us in one year: Papers, writing and printing inks, mucilage, lubricating oils, fuel oil, linseed oil, turpentine and white lead, paints, gelatine compound, boiler compounds, soaps, rubber hose and valves, silks, brines, electroplating solutions and scrapings from anode plates, flooring compositions, silicate brick, glasses, gypsum, boiler tubes, steels (other than those in our list of analyzed samples), graphite, tinfoil, solder, monotype metal, antimonial lead, jute bagging, rasped pine sawdust cover for paper rolls, condenser tubes and water, guide rails. Many of these called for far more than routine testing.

In consequence of recent legislative action by Congress, it is probable that a vast deal of work in connection with the testing of structural materials will devolve upon the bureau. Its proper attention will require large additions to the chemical force and necessitate the erection in the near future of a building specially devoted to chemistry.

As is generally known, the units in terms of which practically all electrical measurements are expressed, are the International Units defined in terms of concrete standards, and not the absolute units of the C. G. S. system. The latter have been displaced, for the present at least, on account of the higher accuracy attainable with the former by the aid of carefully drawn specifications.

To secure the highest attainable accuracy, the chemist must be called upon to study the methods of purification or preparation of the materials employed, to develop special methods of analysis, qualitative as well as quantitative, and incidentally to investigate various problems such as arise in connection with every research.

The London International Electrical Conference of 1908 adopted the international ohm and ampere, defined in terms of the mercury column and the voltameter respectively, as the two fundamental units. The Weston normal cell, which must, however, be employed to fix the results of voltameter work, was adopted as a secondary unit.

Owing to the difficulty of reaching agreement in specifications, this question and others were assigned to an International Scientific Committee of 15 under the auspices of which a meeting of representatives of the National laboratories of Germany, England and France was recently held at the Bureau of Standards. The object was to compare the results obtained with the types of voltameters used in the various laboratories and to compare standard cells representing the procedures adopted in those institutions as well as to investigate sources of variation in the results, in order to pave the way for the adoption of uniform specifications guaranteeing the highest accuracy of reproduction.

The results, though incomplete, are to be regarded as most satisfactory, but cannot be discussed here. The services of a chemist were needed during all the work on the voltameter and for many months in advance of the coming of the foreign delegates. To his skill was due in large measure the unraveling of some most troublesome and obscure points in the action of different types of coulometers and of the solutions employed in them.

The main chemical questions that have a bearing on the results of voltameter measurements are the following:

1. The preparation of pure silver nitrate.
2. Methods of testing the purity of silver nitrate
3. The influence of impurities in the electrolyte.

4. The contamination of the electrolyte by the septum employed or by the electrolysis effected.

The chemical side of the reproducibility and constancy of the Weston cell as a standard of electromotive force has been under consideration at the Bureau of Standards for some time with the result that an accuracy of reproduction of two or three parts in 100,000 is now attainable. (Parenthetically it may be said that a like degree of accuracy is striven for in the reproducibility of conditions and results by the voltameter, that is, of at least two or three parts in 100,000 as a mean of several determinations.) The work with the Weston cell has embraced the investigation of equilibrium conditions in the cell, the methods of washing and the hydrolysis of the depolarizer, mercurous sulphate, the preparation and purification of the materials mercury, cadmium, mercurous sulphate and cadmium sulphate, and the effect of added impurities.

It is hoped that the ultimate result of the international comparison of cells and voltameters and the exchange of the various materials will result in the adoption of uniform specifications for these materials. The bureau has made the proposal that when this shall have been effected the various national laboratories shall undertake to furnish such materials ready for use to investigators and others.

The need of a new and authoritative table of densities of alcohol-water solutions has long been apparent. Fundamental for this is an exact determination of the density of absolute alcohol. The data heretofore chiefly relied on are those of Mendeléef and the Squibbs, but these differ, hence the desirability of a new determination, which necessitates the prior preparation of pure alcohol, a most difficult problem. How this has been done is described in a paper by Mr. E. C. McKelvy, which I shall have the pleasure of presenting in abbreviated form at this meeting.

An investigation is in progress having in view the unification and simplification of methods of gas testing in this country. An important point in connection with this relates to the permissible limit for hydrogen sulphide in illuminating gas, and the accurate and quick determination of the amount present. In co-operation with the proper physical division a chemist is at work on this problem.

Another has worked in co-operation with the heat division of the bureau in the preparation of the pure materials for calorimetric comparisons, of which I have already made mention.

This latter research is closely related to another, namely, that of determining the heats of combustion of methane and ethane, ethylene and acetylene, carbon monoxide and hydrogen, which are needed as fundamental constants for a study of the calorimetry of illuminating gases. The above-named substances must be prepared in a state of great purity and for their combustion large quantities of very pure oxygen are needed. One chemist in co-operation with our very efficient glassblower has devised a generator for oxygen and hydrogen consisting of nine cells. The electrodes are of wrought iron and are separated by a glass bell-jar. The electrolyte is caustic soda of 20 per cent. strength. The cells are electrolytically in series and the current used about 20 amperes. With this current the yield of oxygen is about 47 liters per hour. The gas is passed through a platinized quartz tube two meters long, then through two towers filled with solid potassium hydroxide, then through a soda-lime tower, and finally through a calcium chloride tower. The gas is then compressed into cylinders at 100 atmospheres pressure by a special mercury piston pump. It is expected that the bureau will, in future, draw its supply of oxygen and hydrogen from this source.

A comprehensive co-operative study of underground electrolysis and corrosion is to be started this year and for this the services of two chemists will probably be needed.

Finally, a long investigation, still unfinished, is the deter-



mination of the atomic weight ratio between bromine and hydrogen. This research forms a logical extension of that already done at the bureau by Drs. W. A. Noyes and H. C. P. Weber on the ratio between chlorine and hydrogen, a work that was crowned some time ago by the award of the Nichols medal.

The force of chemists now employed numbered 13 on June 30th, which number will be increased from 4 to 6 this summer, and probably by a number of others. At present the quarters are uncomfortably crowded, but before the summer is over additional room will be provided to accommodate 17 or 18 in all.

There is a weekly journal meeting for the chemists, also certain courses of lectures and laboratory work to aid those who desire to take advanced university degrees. This latter work is done out of office hours and is accepted by some universities.

In the foregoing I have endeavored to outline in the briefest possible manner the character of the chemical work done at the bureau. You will see that it is most varied and that administration and correspondence must necessarily make such large demands on my own time as to leave little for direct experimentation. The most that I can hope to do must be at odd moments, but it is my hope and wish to be able to exercise direct supervision over the more exact analytical work, so far as this falls in line with my personal acquaintance.

The bureau will, before long I hope, do its full share in the way of publication to forward the science of chemistry both practically and theoretically. But the bureau is yet young and so are most of its chemists. Hence active publication from the start is not to be expected, for unusual care must be taken lest work of doubtful value shall emanate from an institution which should be what its name implies, a Bureau of Standards. It is too much to hope that this will never happen, but my aim will be to keep it at a minimum.

## NOTES AND CORRESPONDENCE.

### NOTE ON THE VOLATILITY OF COCAIN.

When cocain residues were dried in a covered dish heated over the steam bath or at 100° C. in a drying oven, it was noted that a solid film soon collected on the cover. This sublimate was frost-like in appearance, insoluble in water, and on further examination was found to consist of cocain. The fact that this body is volatile at 100° is important in connection with analytical work, if the analyst expects to heat a residue after the solvent has been evaporated, and a caution to this effect would not be out of place in the pharmacopeial directions for assaying coca leaves.

Two cocain residues were tested by heating first for seven hours over a steam bath and then for seven hours in an oven at 100° C., the weights being 0.0131 gram and 0.0121 gram, the dish in the case of the first sample being open to the air and the second covered with a watch glass. At the end of the first stage of the experiment they weighed respectively 0.0108 gram and 0.0104 gram and at the end of the second stage 0.0083 gram and 0.0089 gram. There seemed to be practically no difference in the proportional loss whether the dish were covered or not.

A cocain residue heated several hours in a drying oven at temperatures of 60°, 80° and 90° suffered no loss in weight, and when heated to 98° there was only a slight loss though there was considerable sublimation upward on the walls of the dish.

It was evident from the observations that the last portions of the solvent should be evaporated as rapidly as possible from a cocain residue, using a blast; and that the residual material should be dried cautiously, not over 90° and for safety should be brought to a constant weight by drying over sulphuric acid.

H. C. FULLER.

### NOTE ON DR. BACKE'S INVESTIGATION OF MALTOL AND ISOMALTOL.

*Editor Journal of Industrial and Engineering Chemistry:*

Since the publication of my short paper on "A Source of Error in the Examination of Foods for Salicylic Acid," which was sent you September, 1909, and appeared in this Journal for January, 1910, it has come to my notice that a communication relating to the same source of error was published by Dr. Arnold Backe in *Annales des falsifications* for November, 1909, and Dr. Backe has recently informed me by letter that the experiments upon which his communication was based were made in July of last year. Since my own experiments were made the month following, I beg space to acknowledge the priority of Dr. Backe's work on this point over my own, and also to call attention to his more recent papers,<sup>1</sup> in which he discusses the isolation and properties of the substances to which the interfering reaction is due.

H. C. SHERMAN.

### ACCURACY IN SAMPLING.

*Editor Journal of Industrial and Engineering Chemistry:*

On the appearance of Mr. Bailey's paper on "Accuracy in Sampling Coal"<sup>2</sup> I gave it considerable study. By the time I had arrived at my conclusions in regard to it, interest, judging by cessation of correspondence, had waned. I therefore hesitated to publish my views. The recrudescence of Mr. Bailey's paper in that of Mr. F. B. Porter,<sup>3</sup> however, seems to warrant calling attention to some errors in the former. Mr. Bailey began his paper by comparing some results obtained in the A. D. Little Laboratory with the results tabulated on pp. 281-284, Professional Paper No. 48, U. S. Geological Survey. This tabulation led Mr. Bailey into an error which may best be explained by a concrete instance. 4833 lbs. of an Iowa coal were sampled and analyzed. This lot showed 20.70 per cent. ash, 10986 lbs. from the same carload showed 16.04 per cent. ash, 12000 lbs. showed an ash of 14.46 per cent. Of course, the proper way to obtain an average analysis is to multiply each analysis by the weight of the coal which it represents, add the products and divide by the total weight of coal. Calculated in this manner the coal in question showed 16.17 per cent. ash. The carload from which these portions were taken was sampled as a whole and showed 15.22 per cent. ash. The best value available for this coal is an average of the two figures, 15.69 per cent., and the error due to sampling is 0.47 per cent. In the averaging of the Geological Survey the figures were averaged, as much weight being allowed to the analysis representing 4833 lbs. as to the analysis representing the carload. Calculated in this way the average was 16.60 per cent., and the difference between that figure and 20.70 per cent., or 4.10 per cent., Mr. Bailey considered error.

Recalculating this tabulation the maximum error is 1.16 per cent. and only 5 analyses out of 34 show an error greater than 0.7 per cent. It is probable that these higher errors would disappear if we could trace all the coal involved, but as it stands the maximum error is much less than that of the commercial laboratory as might reasonably be expected from the favorable conditions under which the government chemists worked. Whereas Mr. Bailey thought the government error was more than twice as large as the other.

Mr. Bailey closes his comparison of these two sets of figures with the following statement:

"As this data follows the probability curve very closely, the formula for probable and possible error can be safely applied in determining the reliability of different methods of sampling."

Even if Mr. Bailey had substantiated his formulae by correct figures there would be good reason to be suspicious of them on a

<sup>1</sup> *Compt. rend.*, 150, 540 and 151, 78.

<sup>2</sup> *THIS JOURNAL*, 1, 161 (1909).

<sup>3</sup> *Ibid.*, 2, 148 (1910).



*priori* grounds, for, as Nernst and Schoenflies put it, "we can with certainty regard  $\Delta x$  (the error) as a small amount, otherwise the whole measurement would be illusory" or as another writer puts it "there are no large errors;" that is, the method of least squares is not applicable where errors are large. The errors in Mr. Bailey's large table of analyses run up to almost 40 per cent. of the total ash. These are large errors and consequently the so-called probable error does not comply with the definition, which is "in any series of errors the probable error has such a value that the number of errors greater than it is the same as the number less than it. Or, it is an even wager that an error taken at random will be greater or less than the probable error." Of 193 analyses 111 are greater than Mr. Bailey's calculated probable error and 82 less. The formula for possible error is, except for the constant, the same as that for probable error and must likewise be rejected.

At the time of the appearance of the paper I suggested to Mr. Bailey, in private correspondence, that 1300 experiments were insufficient to demonstrate the theory of probabilities. Mr. Bailey replied that the smoothness of the curve obtained, was good evidence that the experiments were sufficient. In the paper Mr. Bailey describes the curve as being like a parabola, but the points determined do not fit into the equation for the parabola and calculation shows that the curve is, in fact, a broken line. Furthermore, the figures obtained prove themselves inadequate. Varying numbers of pieces of whitened coal were sent over the riffle sampler. The pieces found on the right side of the machine were counted. Each set was sent a few hundred times. When four pieces were used the maximum number found was four; the minimum none. When eight were used the maximum number was eight, the minimum one; when sixteen were used the maximum was twelve the minimum three. Judging from the experiments with eight pieces it might be supposed that the machine was untrue and threw to the right but the next experiment showed that this was not the case. The experiments were obviously inadequate.

A few simple experiments will give an idea of the difficulty of proving the theory of probabilities.

A coin was tossed 50 times and came up heads 26 times, tails 24 times, and at no time were the heads and tails equal.

A die was thrown 96 times, the faces came up as follows:

Face.....	1	2	3	4	5	6
Times.....	13	18	12	9	26	18

I have at my disposal a record of 9000 poker hands actually dealt.

Theory demands that 50.12 per cent. of the hands should have been worthless, whereas 51.42 per cent. was the figure actually obtained.

A consideration of these figures and of the greater complexity of the riffle sampler problems make it evident how 1300 experiments must be.

In conclusion I wish to protest most vigorously against the phrase "size-weight per cent."

The only excuse for the coinage of a new word or term is the birth of a new idea.

There is absolutely no idea intended to be conveyed by this phrase which is not entirely covered by the one word size. Size must, of course, be expressed in regard to something else and Mr. Bailey had a perfect right to express the size of his largest pieces in percentages of his sample before dividing, and with this word of explanation the cumbersome phrase might have been avoided together with the numerous explanations which it required.

Mr. Porter says that there is a prevalent idea that the larger the sample and the smaller the pieces the better. This is more than a prevalent idea, it is a fact. Both gentlemen have done well to emphasize this fact; but that they have shown any

relationship which can be expressed in a mathematical equation, can not be maintained.

LOWELL, MASS.

FRED. C. WELD.

EXPLOSIBILITY OF COAL DUST.

The recently created Federal Bureau of Mines has just issued a bulletin on the explosibility of coal dust, with chapters by J. C. W. Frazer, Axel Larsen, Frank Haas and Carl Scholz. The bulletin was prepared by the Technologic Branch of the United States Geological Survey, which is now a part of the Bureau of Mines, and therefore will be known as Geological Survey Bulletin 425, but will be distributed by the Bureau of Mines. The author of the bulletin, George S. Rice, chief mining engineer of the Bureau, goes fully into one of the most serious and most perplexing problems that the coal mines have had to contend with in the last few years. He traces the growth in the belief in the explosibility of coal dust, summarizes the experiments and mine investigations that have established this belief and gives the present status of preventive measures.

In the introduction, Mr. Rice says: "Only within comparatively few years has the dry dust of bituminous and lignitic coal been generally recognized as an explosive agent more insidious, threatening and deadly to the miner than firedamp. Firedamp carries its own flag of warning—the 'cap' in the safety lamp—but coal dust, though visible, does not attract attention until present in large quantities. Firedamp is of local occurrence and except in notable and very exceptional cases is controllable by careful manipulation of the ventilating currents. If by mischance a body of firedamp is ignited in a mine, the force of the explosion is terrific, but the effect is localized unless dry coal dust is present, or unless (as it very rarely happens) an explosible mixture of methane gas and air extends through large areas of the mine. In a dry mine dust accumulates everywhere, and the blast from the ignition and combustion of bituminous dust may traverse miles of rooms and entire and wreck structures at the entrance of the mine. The comparative potential destructiveness of gas and of bituminous dust is strikingly shown by the history of the Pennsylvania anthracite mines. These mines not infrequently have large inflows of gas, and the resulting mixtures of gas and air have sometimes been ignited, yet no such wide sweeping explosions have taken place, despite the presence of dry anthracite dust, as have happened in excellently ventilated bituminous mines."

Mr. Rice reviews the experiments into the explosibility of coal dust in foreign countries and dwells at considerable length upon the attitude taken in France by the engineers, who, until the great disaster at Courrieres in 1906, which cost 1,000 lives, did not believe that coal dust would explode without the presence of firedamp. Since that terrible disaster a testing station has been established in France and now the French engineers are fully convinced of the dangers of coal dust.

"The coal dust question in this country," continues Mr. Rice, "can not be said to have awakened wide-spread interest among mining men until the terrible disasters of December, 1907, which resulted in the death of 648 men. In response to a demand by those interested in coal mining throughout the country, Congress, in 1908, made an appropriation for the investigation of mine explosions. The United States Geological Survey was charged with the investigation. A testing station was at once decided upon and was established at Pittsburg, Pa.

"While it is probable that for several years the leading mining men in the country have believed in the explosibility of coal dust without the presence of firedamp, yet until the public demonstrations were given at the testing station at Pittsburg, during 1908-'09, and reports were received of similar tests made abroad, a large proportion disbelieved. These tests were so convincing to those who saw them, and such general pub-



licity has been given to them, that it is now exceptional to find a mining man who does not accept the evidence of the explosibility of coal dust. The question of the day no longer is 'Will coal dust explode?' but 'What is the best method of preventing coal dust explosions?'

The following are some of the tentative conclusions of Mr. Rice on the dust problem:

"That coal dust will explode under some circumstances, both in the presence of firedamp and without it, is now generally accepted by mining men. The writer fully agrees with this and takes the following views of the explosibility of dust and the conditions necessary for explosion.

"Experiments at Pittsburg indicate that under ordinary conditions the dust must be from coal having at least about 10 per cent. of volatile combustible matter, though in certain foreign experiments, it is claimed explosions were obtained with charcoal dust.

"Dusts with higher percentages of volatile combustible matter are more sensitive, ash, moisture contents, and size being constant. This view is based partly on the preliminary experiments at Pittsburg and on the results of experiments of M. Taffanel and other foreign investigators. Where there is a large amount of dry coal dust, judging from the Pittsburg experiments, a humid atmosphere has little effect on ignition of dust or propagation of an explosion. A long continuance of the humid conditions renders the coal dust moist and inert, but the presence of moisture in the air at the moment of explosion is not sufficient to prevent an explosion; that is, not enough moisture is carried by the mine air to reduce materially the temperature of the flame. Fully saturated vapor at 65° F., an ordinary mine temperature in this country, weighs 6.78 grains per cubic foot (15.5 grams per cubic meter). Coal dust suspended in such a saturated atmosphere in a cloud of moderate density weighs, say, 200 grams per cubic meter. At the figures given the weight of vapor is but 7.8 per cent. of the weight of dust. The Pittsburg experiments with wetted dust showed that several times this percentage of moisture in the dust, in addition to a nearly saturated atmosphere, was required to prevent propagation.

"Probably with a low dust density, the relative humidity of the air would be an important factor in tending to prevent the initiation of an explosion. However, the great purpose of artificially humidifying mine air is that it may serve as a vehicle for carrying water to the dust."

Mr. Rice concludes by reviewing the various remedies that are offered for the coal dust problem, giving the good and bad points of each.

This bulletin may be obtained by addressing the director of the Bureau of Mines, Washington, D. C.

## BOOK REVIEWS AND NOTICES.

**Chemische Technologie der Neuzeit.** Lieferung, 1, 2, 3. Edited by Dr. Otto Dammer with the coöperation of 49 experts. Stuttgart: Ferdinand Enke, 1910.

The volumes on chemical technology which are being published under the editorship of Dr. Dammer, to complete and bring down to date his large five-volume work issued 1895-1898, are excellent. Three volumes will complete the work which has the title "Chemische Technologie der Neuzeit." A few sins of omission may be charged against the chapters which have thus far appeared, but these are easily excused in consideration of the difficulties in the way of securing authentic, accurate detailed information concerning modern chemical industries and processes. This work represents by far the best attempt which has yet been made to give the student and reader accurate information in regard to the recent developments in the field of

applied chemistry. In connection with the five volumes of Dammer's "Handbuch der Chemischen Technologie" they constitute an invaluable aid to the student of industrial chemistry. The subjects thus far treated are: water, liquid air, ozone, hydrogen, sulfur, selenium, carbon bisulfide, carbon tetrachloride, sulfur chloride, thiosulfates, hydrosulfites, sulfurous acid, sulfites, sulfuric acid, Stassfurt salts, salt, soda, sodium sulfate, hydrochloric acid, chlorine, calcium chloride, potash, saltpeter, nitric acid, air nitrates, ammonia, iodine, fluorine, carbon dioxide, cyanides, borates, phosphorus, the alkali earth salts, aluminium salts, artificial precious stones, rare earths and glow-lights, electric glow-lights, peroxides, carbides, silicides and explosives.

**Van Nostrand's Chemical Annual.** By JOHN C. OLSEN. Second issue, 1909. Cloth, 12mo. pp. vi + 580. New York: D. Van Nostrand Co., 1909. Price, \$2.50 net.

In the second issue of this well-known book of tables the pages have been increased from 496 to 580—those devoted to chemical and other tables from 415 to 465; the rest of the increase is in the pages devoted to the review of chemical literature. The frontispiece of the book is a portrait of Mendeléeff and on a flyleaf there is given a sketch of his life. The tables have been used by a large number of chemists since the first issue was published and have been found accurate. The table for sulfuric acid by Ferguson and Talbot adopted by the Manufacturing Chemists' Association of the United States is included. If any suggestion were to be made it would be for a more compact form with thinner paper and flexible cover for the next issue of the annual.

**Fuel and Refractory Materials.** By A. HUMBOLDT SEXTON. New and Revised Edition. 12mo. pp. x + 364. New York: D. Van Nostrand Co., 1910. Price, \$2.50 net.

The changes in the present edition are chiefly in the chapters on gas producers, pyrometry and coke ovens. In describing modern plant and apparatus it has been found necessary in order to save space and also because of lack of interest in the older forms to omit many of these. The chapters are: 1, Combustion; 2, Heating Power of Fuels; 3, Fuels, Wood, Peat, Coal; 4, Solid Prepared Fuels; 5, Coal Washing; 6, Liquid Fuels; 7, Gaseous Fuels; 8, Recovery of By-Products; 9, Furnaces for Metallurgical Purposes; 10, Furnace Operation; 11, Pyrometry; 12, Calorimetry; 13, Utilization of Fuel; 14, Testing Fuels; 15, Refractory Materials, Bricks, Crucibles. As the table of contents indicates, practically all of the book is devoted to fuels and furnaces and a small part only to refractories. There are 104 well selected illustrations representative for the most part of English design and practice. Under calorimetry Parr's calorimeter is omitted. In general an excellent textbook brought nearly down to date.

The Bureau of Standards has issued two circulars of the greatest interest to chemists. Circular No. 25 is entitled "Standard Analyzed Samples—General Information." This bulletin is published for the general information of those interested in standard analyzed samples; it is divided into the following parts: Introduction, Function of Standard Analyzed Samples, History of Bureau of Standards' Samples, The Choice of Standard Materials, Preparation of Materials, Uses of Standard Samples, General Considerations, Standard Analyzed Samples issued or in preparation, Summary of Analyses, Ordering and Shipping. Under the head of "Uses of Standard Samples" the circular states: "The principal uses of the bureau's analyzed samples appear to be as follows: First: In the settling and avoiding of disputes between buyers and sellers. Such disputes very frequently arise through the selection of inappropriate or faulty methods of analysis by one of the chemists, and the analysis of a standard material usually brings these to light



before the case comes to court. Second: In standardizing volumetric solutions; for example, the determination of sulphur and manganese in iron and steel; iron ores are used for establishing the iron standard for permanganate or bichromate solutions, from which the standards for most other titrations are calculated. Third: As material for trying out new methods. The use in this connection is obvious. Fourth: As practice material for new and inexperienced chemists. They have quite an extended use in universities in this connection." A list of thirty-seven samples already issued or in preparation is given together with prices.

Circular No. 26 is entitled "Analyzed Iron and Manganese Ores—Methods of Analysis." The following are the contents of this circular: I, Introduction; Mixing, Drying, Reagents, Apparatus. II, Methods of Analysis at the Bureau of Standards; Silica, Phosphorus, Aluminum, Titanium, Standardization of Permanganate Solutions, Iron, Available Oxygen, Manganese, Lime, Magnesia. III, Methods used by other analysts; Commercial chemists, Works chemists, Mine chemists.

## SCIENTIFIC AND INDUSTRIAL SOCIETIES.

### DIVISION OF PHARMACEUTICAL CHEMISTS, AMERICAN CHEMICAL SOCIETY.

San Francisco Meeting, July 16, 1910.

In the absence of the Chairman the Secretary requested Dr. H. E. Barnard, of Indianapolis, to preside over the meeting. The customary reading of the minutes was followed by the report of the treasurer and Committee on Quantitative Methods.

The following papers were read and discussed:

Albert Schneider, "The Relationship of the Microscopical and Chemical Analyses of Vegetable Drugs, Foods and Spices."

W. A. Pearson, "Asafetida."

B. L. Murray, "Electrolytic Determination of Mercury in the Mercury Salts of the Pharmacopoeia."

E. K. Nelson, "Capsaicin, the Pungent Principle of Capsicum."

E. K. Nelson, "The Detection of Capsicum."

H. C. Fuller, "Note on the Volatility of Cocain."

H. C. Fuller, "Separation and Determination of Cocain and Strychnin and of Atropin and Strychnin when they Occur Together."

Dr. F. Klein, "New Tests."

G. H. P. Lichthardt, "Identification of Caramel."

L. A. Brown, "Determination of Acidity in Hydrogen Peroxide."

Following the ideas brought out by Prof. Schneider in his paper a motion was passed as follows: Moved that the chairman and executive committee, after consulting with the Division of Agricultural and Food Chemistry, consider the question of appointing a committee on micro-analysis, with power to appoint such a committee if advisable. The idea of this was to attract micro-analyst to the ranks of this division.

B. L. MURRAY.

### DIVISION OF AGRICULTURAL AND FOOD CHEMISTRY OF THE AMERICAN CHEMICAL SOCIETY.

San Francisco Meeting, July 15 and 16, 1910.

The session on Friday morning, July 15th, was devoted to an inspection of the Laboratories at the University of California, Berkeley, Cal. In the absence of the Secretary the reading of the minutes was omitted and E. F. Ladd was appointed to act as Secretary, *pro tem*.

The meeting on Saturday was held in the Green Room of St. Francis Hotel, San Francisco, and was called together by G. E. Colby. The following papers were read:

C. A. Browne, "The Saccharimeter Scale and the Means for its Verification."

C. A. Browne, "The Normal Weight of Dextrose."

H. H. Bunzell, "The Role of Hydrogen Peroxide in the Oxidation of Pyrogallol by the System  $H_2O_2$ -Peroxidase."

H. E. Barnard, "The Influence of the Ingestion of Spices upon the Excretion of Hippuric Acid."

H. W. Wiley and J. A. LeClerc, "Environmental Work on Wheat."

J. A. LeClerc and J. F. Breazeale, "Translocation of Plant Foods and the Elaboration of Plant Material during the Germination of Wheat."

P. L. Hibbard, "Time Factors in Determination of Nitrogen, and Other Observations on the Kjeldahl Method."

G. W. Shaw, "Effect of Environment on the Composition of Wheat."

J. A. LeClerc and B. R. Jacobs, "High Protein Bread."

Dr. E. W. Hilgard, "Unification of Chemical Soil Analysis."

The following papers were read by title only:

G. O. Higley, "The Relation of Carbon Dioxid Excretion to Body Weight."

G. O. Higley, "The Carbon Dioxid Excretion as Modified by Barometric Changes."

R. E. Swain, "The Physiological Action of Thallium Salts as Shown by the Nitrogenous Metabolism."

R. E. Swain, "The Utilization of Starch Introduced Directly into the Circulation."

E. M. Chace and A. R. Albright, "The Detection of Lemon Oil in Orange Oil."

C. E. Bradley, "The Reactions of Lime and Gypsum on Some Oregon Soils."

R. S. Hiltner, "Citral in Lemon Oils and Extracts."

J. B. Rather and G. S. Fraps, "The Composition and Digestibility of the Ether Extract of Cowpea Hay."

F. C. Cook, "The Destruction of Invertase Solutions in the Absence of a Preservative."

F. C. Cook, "The Destruction of Invertase Solutions by Shaking and by the Electric Current."

Edward Bartow and B. H. Harrison, "Determination of Ammonia Nitrogen in Water in the Presence of Hydrogen Sulfide."

Edward Bartow and C. E. Millar, "Extent and Composition of the Incrustation on Filter Sands."

Chas. B. Bennett, "A Note on the Hypoxanthine of Meat."

Alfred N. Cook, "The Relative Toxicity of Substances Found in Foods."

M. E. Jaffa, "Résumé of the Work of the California State Food and Drug Laboratory."

Louis H. Jackson, "Testing for Saccharin."

Waldemar Koch, "Quantitative Chemical Analysis of Animal Tissue: V. Estimation of Chlorine."

M. M. MacLean, "A Convenient Drying Oven."

J. B. Rather and G. S. Fraps, "The Constituents of the Wax of Candelilla, or Mexican Wax Plant."

P. A. Yoder, "A Polariscopic Method for the Estimation of Malic Acid and Its Application in Cane and Maple Products."

P. A. Yoder, "Notes on the Acid Constituents of Sugar Cane Juice and on Methods for Their Determination."

C. L. Alsberg and O. F. Black, "Biochemical and Toxicological Studies upon a Number of Species of Penicillium."

Charles L. Parsons, Secretary of the American Chemical Society, spoke on The Methods of Securing Funds for Promoting the Work of the Division. A motion was made by H. H. Bunzell to make an annual assessment of fifty cents on all members of the Division of Agricultural and Food Chemistry for the purpose of defraying the expenses of this division.

There were present at the meeting Saturday morning about forty members.

E. F. LADD.



## AMERICAN INSTITUTE OF CHEMICAL ENGINEERS.

REPORT OF COMMITTEE ON CHEMICAL ENGINEERING EDUCATION.<sup>1</sup>F. W. FRERICHS, *Chairman.*

WM. M. BOOTH.

F. G. WIECHMANN.

GEO. D. ROSENGARTEN.

A. A. L. VELLON.

Circular letter No. 1 to the members of the Committee on Chemical Engineering Education of the A. I. C. E.

3828 WESTMINSTER PLACE, ST. LOUIS, MO.,  
FEBRUARY 4, 1910.

DEAR SIR: The Committee on Chemical Engineering Education was created at the Pittsburg meeting December, 1908. It was the result of a general feeling which prevailed among the members of the A. I. C. E., that the course of education generally pursued by prospective chemists at the present time does not produce men who are prepared in the best possible way for practical work in the manufacturing establishments of the United States.

As chemistry is a science which is most highly developed in Europe, and especially in Germany, it is natural that the methods of education of chemists in the United States, especially in post-graduate work, have been to a large extent copied from the methods adopted in that Continent. This is more the case, since a large percentage of the older teachers of chemistry have been educated abroad. The highly developed chemical industries such as the coal-tar industries, the manufacture of artificial perfumes and pharmaceutical preparations, require chemists of the highest theoretical education in organic chemistry, who, at the same time are satisfied to do actual work for long hours and for small salaries.

This condition can obtain in Europe, since there is an abundance of chemists trained for that kind of work and anxious to make a living. This very fact enables European manufacturers to export these goods to the United States, as long as the American chemist is not sufficiently protected by tariff laws. On the other hand, many European firms support large research laboratories for the systematic development of new processes and new articles. They can do so, since the cost of experimenting and the salaries of chemists who can do research work, are comparatively low. If patents have been obtained in the United States, the goods are made abroad and imported, and the American chemist is barred from the manufacture of this class of articles.

As long as our tariff and our patent laws are not improved, there would not seem to be any hope that such goods can be made successful in the United States.

These conditions have caused the enormous growth of organic chemical manufacturers in Europe, and since a very large percentage of all the chemists find employment in this class of works, the courses at European universities seem to be shaped to suit their special requirements. For the student such a course appears to be highly acceptable, since it greatly favors organic chemistry, which branch permits of brilliant speculation, and is, therefore, most fascinating to the beginner. Besides, it coincides largely with the course which is most approved for the education of teachers of chemistry, and therefore prepares for professorships and for a large branch of a specific chemical industry at the same time.

For the universities it may be no small consideration that the students working on their theses for the degree are welcome assistants for the scientific work carried on in their laboratories.

As an excuse for this, the remark is frequently heard that every student owes it to science to devote one or two years to theoretical work, even if he does not reap direct benefit from it.

It seems natural that the scientific work, to which the individual laboratories devote their efforts, more frequently is guided by the personal liking of the professor in charge than

by the earnest desire to fit the student most effectively for his chosen profession. Now the personal liking of the professors in charge would go, most probably, in the direction of organic chemistry, in which they can most easily become conspicuous, and therefore it would seem that at European universities the interests of all chemical industries not needing a specific knowledge of organic chemistry, are sacrificed to the interests of the specific organic manufacturers, which have become of so vital importance to the economic system of European countries.

But from those branches of manufacture the American chemist is largely barred, and for this reason his course of education ought to be different from that of European chemists in order to suit the requirements of his own country.

It is evident that the American student is in the same position as the European chemist entering the great variety of chemical industries other than coal-tar products, artificial perfumes, and pharmaceutical preparations. It might be well to recognize that many experienced manufacturers abroad do not consider the courses offered at European universities to be the most desirable for this class of chemists. This feeling apparently has been recognized by the authorities and has found its expression in the inauguration of the degree of Engineer Chemist at polytechnic schools in Germany.

Under these conditions, it would be of great benefit if the peculiar requirements could be pointed out, which would best fit the prospective chemist to enter practical life in the United States. An expression of opinion coming from the A. I. C. E., which embraces a large body of practical men, would seem to have a special value, and in order to crystallize this opinion into a definite form, the Committee on Chemical Engineering Education has been appointed.

It is thought that the best course for the work of this committee would be:

- 1st. To formulate the opinion of this committee in regard to the most suitable course of education for a "chemical engineer."
- 2nd. To submit the opinion of the committee to the entire membership for discussion by letter.
- 3rd. To submit the result of this discussion to a general meeting of the A. I. C. E. for final discussion and action.

If in this way the opinion of the A. I. C. E. has been formulated, it may be submitted to the institutions of learning in the United States, and the educators in these institutions might be induced to lay out courses which are likely to produce the desired results with the least outlay of time and money on the part of the prospective chemical engineers.

In order to establish the opinion of the committee on the most effective course of education, it seems necessary first to define the meaning of the term "chemical engineer."

The constitution of the A. I. C. E. in Article III, Section 1, defines the qualifications for active membership and from these the intended meaning of the term "chemical engineer" may be inferred.

In Art. III, Sec. 1, Paragraph 1, our constitution provides for such candidates who have not had the opportunity of an early education, but who, as self-made men, have acquired by persistent work such ability in handling chemical problems as rightly entitles them to recognition as chemical engineers. The A. I. C. E. in this paragraph pledges itself to recognize the work of a true genius, independently from the way in which it has been accomplished.

The same seems to apply to candidates accepted under Paragraph 2, who have had only the opportunity to acquire the degree of B.A.

Art. III, Sec. 1, Paragraph 3, apparently indicates the course of education which is thought to produce the average "chemical engineer." But even here the definition is somewhat vague and evidently induced by the feeling that a satisfactory understanding of the term "chemical engineer" has not been established,

<sup>1</sup> From August, 1910, *Bull. Am. Inst. Chem. Eng.*



A civil engineer who has specialized in water and sewage purification may be a very desirable member of the Institute, and so may be the mechanical engineer who has specialized in power plant economics, and the architect who made sanitary installations a specialty. But with these branches of engineering the study of chemistry is only of secondary consideration, and for this reason a course of education would preferably be devised in every specific instance.

Somewhat different is the study of electrical engineering, since electricity is so closely allied with chemical manufacture that electrochemistry has become a study by itself.

It would seem to be the education of the prospective chemist who is to hold the degree of B.S. in chemistry which comes under discussion by this committee, since it prepares the student for the science of chemical engineering in its more specific sense. In order to simplify the discussion I propose to confine, at the present, the work of the committee to the education of chemical engineers in this sense, leaving it to the pleasure of the committee to discuss the other branches at a later time.

In order to have a basis for discussion, I introduce the courses of a typical American university, and shall ask a number of definite questions in order to learn the position of the members of this committee in relation to the course of "chemical engineering" offered by this university.

SYNOPSIS OF THE COURSE IN CHEMICAL ENGINEERING.

FRESHMAN YEAR.

First Term.

Subject	Hours per Week.	
	Rec.	Lab., etc.
English Composition.....	3	
German Grammar, or } French	3	
Trigonometry.....	5	(9 weeks)
Algebra.....	5	(9 weeks)
Chemistry (General Descriptive).....	2	3
Physics.....	1	3
Drawing, Freehand.....		4
Lettering.....		2
	Total, 14	12

Second Term.

English.....	3	
German, or } French	3	
Analytical Geometry.....	5	
Physics.....	1	3
Chemistry (General Descriptive).....	2	3
Drawing.....		6
	Total, 14	12

SOPHOMORE YEAR.

First Term.

German Grammar, or } Reading in Modern French	3	
Differential Calculus.....	4	
Descriptive Geometry.....	3	
Sound, Heat, Optics.....	4	
Qualitative Analysis.....		6
Machine Drawing.....		3
	Total, 14	12

Second Term.

Quantitative Analysis (Gravimetric and Volumetric).....	1	5
Surveying.....	2	5
Integral Calculus.....	3	
Statics.....	3	
Optics.....	3	3
Assaying and Stoichiometry.....	1	3
	Total, 13	16

JUNIOR YEAR.

First Term.

Subject.	Hours per week.	
	Rec.	Lab., etc.
Organic Chemistry.....	3	
Laboratory Organic Chemistry.....		6
Assaying and Stoichiometry.....	1	3
Quantitative Analysis (Commercial and Industrial Products).....		6
Mineralogy.....	2	3
Electrical Machinery.....	3	
Mechanics.....	3	
	Total, 12	18

Second Term.

Organic Chemistry.....	3	
Laboratory Organic Chemistry.....		6
Advanced Quantitative Analysis (Commercial and Industrial Products).....		6
Electrical Machinery.....	3	
Mineralogy.....	2	3
Dynamics.....	3	
Electrical Laboratory.....		3
	Total, 11	18

SENIOR YEAR.

First Term.

Advanced Quantitative Analysis (Food, Water, and Gas).....		6
Physical Chemistry.....	1	3
Industrial Engineering.....	2	
Economic Geology.....	2	3
Engineering Materials.....	3	
Economics.....	2	
Bacteriology.....		6
English.....	1	
	Total, 11	18

Second Term.

Advanced Quantitative Analysis (Food, Water, and Gas).....		6
Seminar.....	1	
Industrial and Engineering Chemistry.....	2	
Research.....		4
Physical Chemistry.....	1	3
Geodesy.....	1	
Economic Geology.....	2	3
Hydraulics.....	3	
Economics.....	2	
English.....	1	
	Total, 13	16

In the School of Engineering of this university a regular high school education is required from entering students.

The degree of B.S. is conferred on the satisfactory completion of the four years' work.

The degree of Ph.D. is open, after not less than three years of residence and study, to all who have received the degree of B.S.

By glancing over the full engineering courses it will be found that they are arranged on very broad lines, so that specialization would not seem necessary before the Junior year. At this time the student of chemistry would specialize, obtaining at the end of his four years' course the degree of B.S. in chemistry, being at this time equally well prepared for post-graduate work in pure chemistry and in chemical engineering.

If the graduate from the B.S. course desires to complete his studies in pure chemistry, or if he desires to prepare himself better for practical work, he may take a post-graduate course of three years' study and obtain the degree of Ph.D.

Having explained existing conditions as I see them, I now take the liberty of asking the following questions:

1st. Is, in your opinion, a chemist having completed a four-year course as offered at this university and possessing the degree of B.S. satisfactorily equipped to enter practical work?

2nd. If question 1 is answered in the negative, can, in your



opinion, a four-year course be arranged in such a way that a chemist after its completion is satisfactorily equipped to take up practical work?

3rd. If questions 1 and 2 are answered in the negative, can, in your opinion, a four-year course supplemented by one year post-graduate work taken at the same university equip a chemist to be efficient for practical work?

4th. Are the post-graduate courses as offered at American universities, in your opinion, the most effective courses which can be designed for the education of chemical engineers?

5th. If question 4 is answered in the negative, what changes are, in your opinion, necessary to make the offered courses effective?

6th. Are the courses offered at European universities and polytechnic schools, in your opinion, the most effective courses for producing chemical engineers, fit for work in American places of manufacture?

7th. Have you any specific suggestions for improving the courses, which are offered at present by American universities?

Respectfully submitted,

F. W. FRERICHS,  
Chairman.

APRIL 15, 1910.

DR. F. W. FRERICHS, *Chairman*, Committee on Chemical Engineering Education of the A. I. C. E., St. Louis, Mo.

MY DEAR DOCTOR FRERICHS:

I will give briefly my views upon the subject of chemical engineering education in answer to your circular letter of February 4th.

The differences observable in the development and practice of chemistry in Europe and the United States are very well set forth in that letter, and I agree with all your suggestions upon that subject. I would add one further point of difference which, of course, springs from the same cause, and that is the larger scale of operations which we find always manifested in this country by great tonnage production. This will continue to grow as a marked feature of our work. When one understands that the genius of America is most often applied to increase of production rather than to refinement of products, it would seem to me to be a corollary from this that our students should be well trained in mechanics to meet the demand which the world seems to put upon us to supply staples. The type that we should in the future choose to imitate would be rather that of the metallurgical engineer.

In answer to your questions, I would make the following statements:

1. No.
2. Only in case the preliminary examination for entrance on the B.S. course is more severe than the present high-school graduation test.
3. Such a course as last named, with one year post-graduate course, would be adequate to properly equip a chemist.
4. Not at all.
5. More devotion to problems truly combining theory and practice.
6. No.
7. I may state my views in general as follows:

For a course in chemical engineering, the best efforts should be spent in the beginning, either in the high school or in the first year of the B.S. course. Advanced work should have been done in literature, philosophy, the nature of matter and substances, logic and mathematics and advanced algebra. The beginning of real scientific work should include most of what we have to-day in such schools as the university whose course you have quoted and might be in four years as follows:

1st year. Physics, drawing, chemistry, metallurgy, geometry.

2nd year. Analysis, applied electricity, surveying, calculus.

3rd year. Advanced metallurgy, mechanics, organic chemistry.

4th year. Construction, theory, and design. Materials, accounting, organic chemistry, physical chemistry, electrical chemistry.

For a doctor's degree, two years' research in metallurgy, process work, design, practical execution or economic studies.

In general, I should say, that if we could try to grasp the genius of European regard for deep study and combine it with the American genius for adaptation to any conditions, no matter how embarrassing, we would have produced a result combining the greatest strength and promise for the future.

Yours sincerely,

CHAS. F. MCKENNA.

MARCH 3, 1910.

DR. F. W. FRERICHS, *Mfg. Chemist*,  
3828 Westminster Place,  
St. Louis, Mo.

DEAR SIR:—Your favor of February 5, 1910, duly received.

In accordance with your request, I herewith submit my answers to your questions, and my suggestions in the matter.

In reply to your questions:

1. No.
2. No.
3. Yes.
4. I am not sufficiently acquainted with the different post-graduate courses now offered at the American universities to form an opinion.
5. My answer to this question is implied by my answer to No. 4.

6. As far as my limited knowledge as to the courses offered at the European universities and polytechnic schools permits me to frame a reply to this question, I would state that in my opinion, the Polytechnic High School at Charlottenburg (Berlin, Germany) offers the best at the present time. The degree granted upon completion of this course is *Docktor-Ingenieur*.

7. My reply to this is as follows:

I would suggest that there be demanded for entrance to the university a thorough knowledge of German grammar and French grammar, and that the ability to read easy works in both languages, at sight, be required. This would permit the reading at sight in the first year of technical scientific text-books in German and in French.

I would further advise that German be thus taught in the first term of the first year and French in the second term of the first year, or *vice versa*, as might prove most convenient to the departments in charge.

In the second year I would also assign one term to the study of German and one term to the study of French, continuing and progressing along the lines followed in the first year, and in addition thereto, conferences on technical subjects in chemistry and physics, in these two languages.

I would also suggest adding to the first term's work, in the second year, the study of qualitative analysis and stoichiometry. In the second term of the second year I would consider it to be an advantage to omit the study of statics and to replace the same by instruction in chemical philosophy, and furthermore to complete the study of assaying in the second year.

In the third year, first term, I would replace the study of assaying, which would now have been achieved, by the study of statics and mechanics, *i. e.*, mechanics too, if this could be arranged. If not, I would have mechanics taught in the second term of the third year.

In the fourth year, first term, I would substitute for the study of bacteriology, the course of botany 5 for botany 7, and botany 7 in the second term. In the second term of the fourth year, I would omit the study of geodesy, which I do not consider necessary for students in chemical engineering.



I have not been able to give sufficient thought to enable me to outline completely the courses of study which should be included in the post-graduate year for chemical engineers, but proceeding upon the belief that chemical engineering is essentially the adaptation of laboratory processes to factory conditions, I believe that these courses should include the study of theoretical and practical electrochemistry, the designing and laying out of factories and the study by each student of some one industrial process along the lines in which he might be especially interested; for instance, in the manufacture of steel or of some other metallurgical process, in the manufacture and dyeing of textile fabrics, in the refining of oils, in the refining of sugar, in practical ceramics and in the manufacture of pharmaceutical products, etc., etc.

Of course, you will understand that these are merely suggestions, and can as yet be given only in a tentative way. That the proposition before us is a large one and will require a great deal of patient study, of course, I thoroughly appreciate.

With sincere regards,

Faithfully,

F. G. WIECHMANN.

ST. LOUIS, MO., JUNE 18, 1910.

DR. F. W. FRERICHS,

3828 Westminster Place, St. Louis, Mo.

DEAR SIR: I shall try to comply with your request, to express my opinion on the subject placed before the Committee on Chemical Engineering Education by the A. I. C. E.

I have carefully read your very interesting circular letter of February 4th, and will try to answer the questions therein put forth as well as it is possible for me, knowing but very little about the conditions now existing in American institutions of learning.

You ask in your letter to take as a basis for the discussion the course of chemical engineering submitted with your letter. I must confess that after having gone over the program of the subjects given in this course, I thought that a rather unfavorable example of chemical education in America had been chosen, having reasons to believe that the standard of chemical education of several other American universities and technical schools is by far higher than that at the university cited. On perusing the synopsis of chemical courses of this institution, I was confronted by the striking fact that the whole of the chemical education is in the hands of only three teachers, and most likely no one of the three has had any practical experience in any branch of chemical industry.

Considering the great diversity of the different branches of chemistry, it seems unlikely to me, to say the least, that effective teaching in all of these branches could be expected by such a small staff of teachers. In order to offer the opportunity to the student for thorough instruction in each one of the many courses, the combination of which constitutes a complete chemical engineering course, it seems evident that the teacher in each case should specialize in his branch. This is true of course for every line of study, but in our case more particularly so, especially for the instruction in chemical technology or industrial chemistry, where not only specialization on the part of the teacher is paramount, but where it is necessary also that his knowledge be based on practical experience.

Question No. 1.

Answer. No.

Question No. 2.

Answer. Yes and No. Four years should be a sufficient time for the education of a chemist, to make him fit to enter an industrial establishment, but all of these four years must be available for courses in chemistry and those other branches of knowledge necessary for the prospective chemical engineer. A sound general education must precede such a four years' course. This does not seem to be the practice followed in institutions

such as the one whose course is quoted, where a large part of the four years are devoted to topics of general educational character. In this connection, the question naturally arises, At what age should the special training of the student for chemical engineering begin? I do not feel qualified to make recommendations in this direction, not being sufficiently acquainted with the conditions in American grammar and high schools, but I venture to say that it can hardly be expected that a young man's general education could be rounded off before the age of 19 to 20 years. At this age, however, sufficient knowledge can have been acquired by the average student to make him fit to take up special studies.

It goes without saying that during these years of special studies opportunity must be given the student to follow courses of his own choice, not necessarily embodied in the program of the chemical engineering course. If this is done, the danger of unilateral professionalism can be satisfactorily met.

Question No. 3.

Answer. If a four years' course is made effective, as outlined in my answer to question 2, post-graduate work should not be considered an absolute necessity, although it can but be beneficial. If, however, a four years' course comprises, besides chemistry, instruction in general educational topics, a post-graduate course of at least one or two years becomes a necessity.

Agreements should exist between institutions of the same grade, which should enable the student to follow a post-graduate course in the institution which offers the best opportunities for the particular line of work which he has chosen.

Question No. 4.

Answer. Not being acquainted with the character of post-graduate courses offered at American institutions, I do not feel qualified to express an opinion on this question.

Question No. 5.

Answer. Conditional for effective post-graduate courses is a thorough preparation for such work in the form of a course of chemical engineering which in itself should equip the average student with the necessary knowledge to enter chemical industry.

Question No. 6.

Answer. In answering this question, I can be guided by the example of the university chosen for comparison, and in this case I consider chemists from European polytechnic institutions by far better equipped even for American manufacturing plants than chemists having followed a course as such given at this university, even if they had taken one year's post-graduate work. However, I would not like to generalize and include among the chemists with European education those who come from all European universities, for the reason that in a great many of these latter institutions very little weight is given to the technical part of the chemist's education. Still, I believe that the average chemist with the European university education will be more efficient for research work in connection with chemical manufacturing than his brother from the American university quoted.

Question No. 7.

Answer. I do not feel qualified to pass criticism on the courses offered by the different American universities. I take the liberty, however, to submit a course which, in my opinion, would answer the purpose of turning out chemists ready to enter the practical field as chemical engineers. This plan is taken from the course prescribed for chemical engineers at the Swiss Polytechnic School in Zurich, and comprises 3½ years. It should, in my opinion, be increased to four years, thus providing more time for some of the important courses which suffer by the shortness of a 3½ year term. It must be remembered however, that this course presupposes a thorough general education.



	Hours per Week.
<i>First Year—Winter Semester:</i>	
Differential and Integral Calculus.....	7
General and Inorganic Chemistry.....	7
Analytical Chemistry.....	8
Analytical Chemical Laboratory.....	16
Mineralogy.....	5
<i>Summer Semester:</i>	
Physics.....	5
Organic Chemistry I (Aliphatic).....	7
Analytical Chemical Laboratory.....	16
Applied Mechanics and Mechanical Engineering.....	7
<i>Options:</i>	
Petrography.....	3
Applied Calculus.....	4
<i>Second Year—Winter Semester:</i>	
Organic Chemistry II (Aromatic).....	3
Industrial Chemistry (Water, Salt, Acid).....	5
Fuel and Theory of Heat.....	3
Industrial Chemical Laboratory (Inorganic).....	16
Physics.....	5
Physical Laboratory.....	4
Applied Mechanics and Mechanical Engineering.....	7
<i>Options:</i>	
General Geology.....	4
Botany.....	2
Identification of Minerals.....	3
Microscopy I.....	4
<i>Summer Semester:</i>	
Organic Chemistry II (Aromatic).....	3
Industrial Chemical Laboratory (Organic).....	16
Physical Chemistry I.....	3
Industrial Chemistry (Alkalies, Chlorine, Phosphate).....	4
Technical Analysis.....	5
Engineering Laboratory.....	4
<i>Options:</i>	
Bacteriology.....	2
Botany II.....	2
Microscopy II.....	4
<i>Third Year—</i>	
Industrial Chemistry (Organic), Bleaching, Dyeing, Dyes.....	5
Metallurgy.....	3
Physical Chemistry II.....	3
Physical Chemistry Laboratory.....	4
Analytical Chemical Laboratory or Industrial Chemical Laboratory.....	16
Gas Analysis with Laboratory.....	1
Machines and Apparatus I.....	7
Industrial Hygiene.....	2
Electrochemistry.....	2
<i>Options:</i>	
Food Hygiene.....	1
Water Supplies.....	1
Bacteriological Laboratory, Elementary.....	4
Bacteriological Laboratory, Advanced.....	daily
Industrial Chemistry, Glass, Ceramics, Cement.....	3
Industrial Chemistry, principally Organic Dyestuffs.....	4
Industrial Chemistry, Organic Fats and Illuminants.....	3
Machines and Apparatus II.....	3
Analytical Chemical Laboratory or Industrial Chemical Laboratory.....	16
Explosives.....	1
Carbohydrates and Fermentation.....	2
Physical Chemistry, Advanced.....	2
Assaying.....	2
Hygiene of Heating, Ventilating and Lighting.....	2
Hygiene of Removal of Offal.....	2
Bacteriological Laboratory, Elementary.....	4
Bacteriological Laboratory, Advanced.....	daily
Human Anatomy and Physiology.....	2
Organic Electrochemistry.....	2
<i>Fourth Year—</i>	
Chemical Laboratory, Advanced.....	daily
Political Economy.....	2
<i>Options:</i>	
Food Analysis with Laboratory.....	4
Paper Manufacture.....	2
Spinning and Weaving.....	2
Business Law.....	4
Patents.....	1

I am fully aware of the fact that the few suggestions offered in the foregoing are a very superficial attempt toward the final solution of the great problem now before the institute. I am convinced, however, that a continued effort on the part of the members of the institute will eventually bring about the desired results of better efficiency of future generations of American chemical engineers.

Respectfully submitted,

L. VEILLON.

DR. F. W. FRERICHS,

DEAR SIR: Replying to your letter of inquiry, I would make the following suggestions:

Place at the head of the industrial chemical department a man with practical experience in chemical technology.

By correspondence and coöperation with the principal chemical concerns and the judgment and experience of educators evolve a course in industrial chemistry. This should include a course in mathematics to and including a working knowledge of the calculus, two years each of French and German, one year of mechanical drawing with special attention to chemical machinery.

English throughout the course; *equal attention to physics and chemistry* throughout the course; one year of mineralogy and one year of economic geology.

If possible the student should be given practical work in a large chemical establishment after the Sophomore year.

The elaboration of these ideas would include the number of hours to be devoted to each feature of chemistry, physics, etc.

I do not believe in *thesis* work for the undergraduate student.

Very truly yours,

W. M. BOOTH.

From this correspondence and from oral discussions with members of the committee, it would seem that in their opinion even a successful four years' course at one of our good universities following a regular high school education is not sufficient to equip a student of chemistry satisfactorily to enter practical work.

As to the remedy, the opinions are divided, since it is proposed to require either a more advanced education in chemistry in the high schools or else post-graduate work after completion of the regular college course.

It seems to be the consensus of opinion that the post-graduate courses as offered now at American and many European universities are open to improvement if it is desired to fit post-graduate students for efficient research work in the chemical industries of the United States. In offering the subject for discussion in this meeting, it would seem to be of importance to bear in mind that the field of chemistry at the present day comprises specific knowledge in an almost infinite number of directions, and that in order to be successful it would seem to be imperative for a chemist to specialize and to concentrate his efforts on a small but well-defined part of the almost endless field of chemical research. This would not exclude the prospective manufacturing chemist from having a good general knowledge of the elements which form the basis of chemical engineering. On the contrary, he should have a good general knowledge of inorganic and organic chemistry and of physics and he should have perfectly assimilated these subjects so that it comes easy for him to think in terms of these sciences.

By making a large number of preparations he should have familiarized himself with the properties of the elements and their compounds, and should have a good knowledge of the methods of separating chemical compounds from mixtures and of the production of pure chemicals. Particularly should he be an expert in chemical analysis, even to that extent that he could criticize existing methods, devise new ones to fit new conditions, and he should be able to control the reliability of methods and to ascertain their limit of accuracy.

The graduate student should have a fair knowledge in ad-



vanced mathematics and its application, and be well trained in the drawing of machinery and structures.

He should have had manual training and be able to handle tools of the more important trades. His knowledge in languages should be such that he can read with ease professional publications in French and German languages and express himself clearly in English.

These studies, if pursued properly, would seem to fill an entire four years' course, and before they have been assimilated advanced work would seem to be of little benefit.

A chemist thus prepared may go into practice, but he leaves it to chance whether his progress will be rapid or slow. There may be instances where an intelligent manufacturer will lead him to apply his faculties to promising problems, but in most cases the first years will be wasted in futile efforts and costly experiments.

Quicker, certainly, and less costly experience in applying accumulated knowledge could be obtained by post-graduate work under the direction of good teachers at a well-equipped institution. But these institutions must be fitted to suit the particular requirements of American chemical industries, and the courses must be directed by teachers who are entirely at home among domestic manufacturing methods.

It has been pointed out by one member of your committee that the University of Zurich, Switzerland, has been particularly favored by having had for a long period of years two teachers, *viz.*, Guckum and Lunge, the former a successful manufacturer of coal-tar preparations, the other an expert in the soda industries, both of whom taught at the same time and were constantly in close contact with their respective industries.

Unfortunately it would seem impossible to duplicate these conditions in the United States. Expression has been given to the thought that no university could tempt a successful manufacturer in the United States to take up teaching, even if he had the ability to do so, and the average professor of industrial chemistry would hardly have the opportunity to obtain a full insight into the actual conditions of the chemical industries. What is published in hand-books of industrial chemistry to a large extent is hearsay and antiquated, and it is only natural that manufacturers are loath to divulge their experience for publication. The consequence is that the collecting and reporting professor of industrial chemistry is often teaching methods and processes long since abandoned and replaced by better ones.

What we must have for post-graduate work are producing teachers of industrial chemistry who, like Lunge, work ahead and do not follow the progress of the technical industries. Men of this kind will have information to offer to the manufacturers and in turn will obtain information upon which they are able to base the progress of their work.

To the places where such men teach, post-graduate students will flock and will be willing assistants for the work of their laboratories.

Some of the studies which might be taken up to advantage in a post-graduate course for chemical engineers are the following:

1. Exercises in making chemical preparations quantitatively—that means the making of chemical preparations, observing weights of raw materials used, time of reaction, concentration, pressures and temperatures most favorable for the production of chemical compounds, and the yield obtained. These exercises should be carried on in great variety and with great exactness, since they form the basis of most research work in chemical engineering.

2. Study of materials of construction for chemical apparatus, including metals, alloys, stoneware, cement, rubber, protal, wood, and others. These materials should be studied, not only

with reference to strain and workability, but also with reference to action of various chemicals upon the materials.

3. Study of construction of machinery and apparatus. This should include a study of the limitation of materials in their application for making apparatus and also the preservation of apparatus from destructive influences.

4. Measuring apparatus and methods of measuring.

5. Study of the generation of steam: types of boilers and boiler furnaces, boiler feed water, purification of same, fuels, methods of making tests for efficiency, firing with coal, oil or gas, calculation of cost.

6. Water supply.

7. Study of generation of power, steam engine, water wheels, electric motors, transmission of power.

8. Construction of furnaces, pyrometry, materials for furnaces, construction, repairing same, construction of chimneys, fuels for furnaces, efficiency, calculation of cost.

9. Study of evaporation of liquids and solutions under high pressure, under atmospheric pressure and *in vacuo*, concentrating solutions, calculation of cost.

10. Construction of drying plants, theory of drying, drying *in vacuo*, collecting evaporated solvents.

11. Theory of distillation and construction of stills, rectification, dephlegmation condensation.

12. Theory of saturation, extraction, and absorption.

13. Study of evaporation of solids, resublimation.

14. Theory of crystallization, crystallization in motion and *in vacuo*.

15. Methods of treating solids with liquids.

Methods of treating solids with gases.

Methods of treating liquids with gases.

16. Methods for purifying solids, liquids, or gases.

17. Grinding and disintegration machinery, separating and mixing machinery.

18. Handling of solids in large quantities.

Handling of liquids in large quantities.

Handling of gases in large quantities.

19. Buildings and structures for chemical plants.

20. Packages and materials for same.

21. Handling and disposition of labor and prevention of accidents.

Liability insurance.

Accident insurance.

Regulation of wages.

22. Sampling and testing chemicals.

23. Controlling the manufacture by logs.

Factory bookkeeping and calculations.

24. Patent law, tariff law, corporation law, contracting.

25. Discussion of special industries.

It is true, many practical problems *cannot* be carried out in a laboratory on a small scale, and therefore *cannot* be an object of study in a post-graduate course. But a very great number of processes *can* be worked out in the laboratory and it would seem that in connection with these the theoretical part of industrial chemistry could be worked out in a satisfactory manner. In order to do this there must be at least one teacher of industrial chemistry and at least one institute which will carry out the work.

Our friend, Dr. Baekeland, said recently in his address at Pittsburg, that the principal object in a research laboratory is the research chemist himself. I think he is right. But it is believed that it will be a long time before the universities without the assistance of practical manufacturers can produce such a man. In my opinion, the A. I. C. E. could be of great assistance in bringing this about. If a university of high standing would undertake to establish a post-graduate course for chemical engineering, the A. I. C. E. might offer the assistance of a committee from among its members, to act in a consulting capacity



to the faculty and to help in this manner, to originate an efficient course. But it would seem necessary that the professor of industrial chemistry to whom the work of post-graduate students is entrusted devote his entire time to this work and be not burdened with work in undergraduate departments. He should have a well-equipped laboratory and a staff of the best assistants at his disposal, since the field of research is large and the detail work, particularly in the beginning, would seem overwhelming. As to the duration of such a course, it is thought that two years are sufficient if only well-prepared students are entered. Upon the termination, an appropriate degree might be conferred which no doubt would be highly treasured by the possessor.

COMMITTEE ON CHEMICAL ENGINEERING EDUCATION.

F. W. FRERICHS, *Chairman*.

PRESIDENT MCKENNA:

This painstaking and valuable report will be printed in a Bulletin in anticipation of the regular Transactions. The members of the institute will be asked to send to the Secretary their opinions on this subject. These will be edited, by the Committee on Chemical Engineering Education, and will, together with this report, form the basis of a progress report at least at the annual meeting. If there is anything of importance that members wish to put forward at this time—some important view—it would be well, of course, for us to have it.

SECRETARY OLSEN:

I feel in the first place that the institute is undertaking a very important work. I am ready to admit that the conditions in American colleges and American universities are in many cases deplorable. I think there is no question of it. There are certain influences at work at present to improve and change those conditions, and I am glad to see this institute entering the field. There are too many colleges in the country poorly equipped and with very little endowment. Why, in one State they passed a law that a college should not grant degrees unless they had an endowment. The law as at first proposed was that that endowment should be—half a million? No, \$25,000 was enough to make a college: After the legislators had discussed the matter, they decided that it was too high, and brought it down to \$5000. As Dr. Butrick has said, any preacher can give his note for \$5000, and establish a college and give degrees. I say that public opinion should stamp out such institutions, and place American education on as good a basis as American industries generally are, and root out a lot of those poorly equipped institutions which are in the field giving degrees.

After having said that, and saying that we ought to educate public sentiment for thorough, sound, and broad education (and we can begin a lot lower than Dr. Frerichs has suggested and do good work), I want to say a few words with reference to the typical courses which have been given. You will have noticed the great difference between the three propositions. The last one, giving the course at Zurich, included a long list of technical subjects covering all branches of applied chemistry. That was in Europe. The other suggestions are much simpler. Our practice differs from that in European countries in education, as well as in industry. When American capitalists have built a railroad through the country, they have in many cases thrown the ties down on the bare ground, spiked down the rails and started running trains. That's cheap construction. After they have constructed it in that way they begin to gather in the earnings. Then they proceed to raise the track here, place a bridge there, straighten the track and put in ballast, and to improve the equipment, and after a few years, using the earnings, the road will be in pretty fair condition. In Europe how do they do it? Their capitalization per mile is very heavy, because they build a railroad complete in the beginning. Now the education given in that course at Zurich was designed to be complete right at the start; it is supposed to turn out a man who

knows all about vacuum drying right from the school. We do not do that in America. How do we do? In America we say, give a man a broad, general education, give him the principles of his science, drill him until he knows the broad fundamental principles, and then, if he goes into Dr. Frerichs' laboratory, he will soon learn how to distil ammonia. We trust him to pick things up as he goes along, and go upon the plan of not giving specialties in college. I received my technical training at the Johns Hopkins University. They said: "We will not teach qualitative analysis very thoroughly; we will teach you the properties of chromium until you know chromium—know it so that you could find it under any and all conditions." So we in America have gone at the problem of education from that standpoint. Give the students a broad general training in the principles of the sciences—theory, if you choose to call it so. We have said that if he has the principles of chemistry and engineering, if he has his mathematics, his German, and his French, so that he can read the literature, he will get the applications. Now the question is, Is that the right principle to start out on? I would like an expression of opinion of this institute as to whether that is the way to proceed.

MR. BAKER:

I do not want to throw cold water on any steps which may be taken to turn out industrial chemists, but I do not think it is possible to turn out a full-fledged industrial chemist. The conditions in the factory are entirely different, if not diametrically opposite to those in the laboratory. In the laboratory the work is done under ideal conditions, and the least important factor is economy. In the industrial plant the most important factor is the economical one, because an industrial plant is run to make money and not to demonstrate a chemical theory. Another important factor is that of the physical conditions he must contend with, and does not meet in the laboratory—the "cussedness of inanimate things"—I do not know a better way in which to express it. Some one has aptly said that to gain an education is to cultivate an attitude of mind, which I take to mean, to learn how to look at things. The best chemist is not the man who works all hours of the day working out a problem given in a book, nor a man who burns the midnight oil, but the man who looks into things—the man of an investigative turn of mind—the one whom you would term a crank—one with the spirit of the boy who cut the head of the drum to find out what made the noise.

PRESIDENT MCKENNA:

The institution named in the committee's report was taken only by chance, and, if we were to take the statements made and opinions expressed upon the course in that institution too literally, it would appear as if it were very unfairly treated. That is not the meaning, I feel sure, of such an inquiry. It was referred to only as a typical institution of medium grade.

AMERICAN ELECTROCHEMICAL SOCIETY.

Chicago Meeting, October 13 to 15, 1910.

Headquarters Congress Hotel.

WEDNESDAY, OCTOBER 12TH.

6 P.M. Registration.

THURSDAY, OCTOBER 13TH.

9 A.M. Meeting of Directors.

10-12 A.M. Reading of papers at Florentine Room, Congress Hotel. Thursday afternoon, four alternative excursions.

(1) Fisk St. Power House of the Commonwealth, Edison Co. (2) Western Electric Co. Works. (3) Automatic Telephone Co. Works, Market St. Transformer and Storage Battery Station of Commonwealth Edison Co., and the Demonstration Installation of the Hoskins Mfg. Co. (4) The Union Stock Yards and the Plants of Swift and Company, Armour and Company and Morris and Company.



## FRIDAY, OCTOBER 14TH.

9-12 A.M. Reading of papers at Florentine Room, Congress Hotel. Friday afternoon, three alternative excursions.

(1) Illinois Steel Co., South Chicago Works. (2) Gary Steel Works. (3) Sears, Roebuck and Co., with a complimentary luncheon for visitors.

Friday evening, dinner at Congress Hotel.

## SATURDAY, OCTOBER 15TH.

9-12 A.M. Reading of papers at Kent Chemical Laboratory, University of Chicago, followed by inspection of laboratories and museums and a luncheon at the University Commons. Afternoon, two alternative excursions.

(1) The Lockport Power House and the Drainage Canal. (2) The Field Columbian Museum.

For the entertainment of visiting ladies an automobile trip to Hull House, The Juvenile Court, Mark White Square and the Stock Yards, with a luncheon at Hull House is planned for Thursday. On Friday morning visits will be made to Marshall Field's Retail Store and The Art Institute. Sears, Roebuck and Co. will entertain both ladies and gentlemen at luncheon and later show visitors through their establishment. Ladies are especially invited to participate in the luncheon at the University on Saturday. In the afternoon they may visit either the Field Museum or the Drainage Canal and Lockport Power House.

General Committee—Herbert N. McCoy, *Chairman*; Arba B. Marvin, *Secretary*. Entertainment—Harry McCormack, *Chairman*. Excursions—F. T. Snyder, *Chairman*. Press—H. T. Matthew, *Chairman*. Ladies—Mrs. W. D. Richardson, *Chairman*.

## PAPERS.

## THURSDAY, OCTOBER 13TH.

10.00 a. m. Reading and Discussion of Papers, Florentine Room, Congress Hotel: "Method for Determining Thermal Conductivity," Carl Hering.

"The Electric Furnace and Its Application," Marcus Ruthenburg.

"Electrolytic Method of Producing Metals," John W. Beckman.

"Reduction of Tin Dross in the Electric Furnace," R. S. Wile.

"Notes on Tribo-Luminescence," W. S. Andrews.

"The Proximate Constituents of the Chemical Elements with Reference to Radioactivity and Electrons," Gustavus D. Hinrichs.

## FRIDAY, OCTOBER 14TH.

9.00 a. m. Reading and Discussion of Papers, Florentine Room, Congress Hotel: "Electrochemical Preparation of Calcium," Prof. A. R. Johnson.

"Efficiency in the Electrolytic Preparation of Calcium," Francis C. Frary, Henry R. Bicknell and Carl A. Tronson.

"Electrochemistry of Polonium and Other Radioactive Elements," Prof. A. R. Johnson.

"The Present Status of Ozone," R. D. Small.

"The Evolution of the Lead Storage Battery," Pedro G. Salom.

"A New Secondary Cell," W. S. Block.

"The Self Discharge of Plante and Faure Storage Batteries," Prof. O. W. Brown and W. G. Bowers.

"Corrosion of Metallic Food Containers—Causes and Prevention," Dr. Edward Gudeman.

"Electrolytic Corrosion of Various Metallic Anodes in a Liquid Ammonia Solution, Ammonium Trinitride," A. W. Browne and E. Holmes and J. S. King, Jr.

"Electrolysis of Solutions of Ammonium Trinitride and of Potassium Amide in Liquid Ammonia, A. W. Browne and M. E. Holmes.

"On the Electrolysis of Some Cyanides," H. E. Patten and W. R. Mott.

## SATURDAY, OCTOBER 15TH.

9.00 a. m. Reading and Discussion of Papers, Room 20, Kent Chemical Laboratory, University of Chicago.

"Application of Electrostatic Separation to Ore Dressing," F. S. MacGregor.

"The Isolation of an Ion and a Precision Measurement of Its Charge," Prof. Robert A. Milliken.

"Phenomenon of Crystallization through Membranes," Dr. J. H. Walton, Jr.

"On the Relative Basicity of the Metals as Shown by their Power to Replace One Another in Chemical Compounds," Dr. L. Kahlenberg.

"On the Effect of Water in Causing Chemical Reactions," Dr. David Klein.

"The Physical and Magnetic Hardness of Some Electrolytic Iron Alloys," James Aston.

## RECENT INVENTIONS.

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

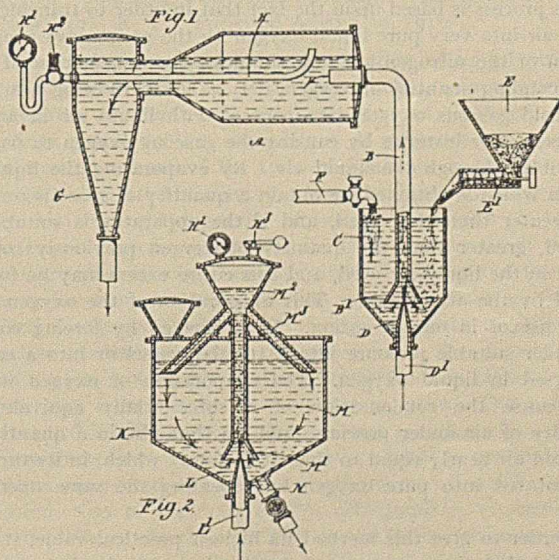
Only a few patents which are deemed of greatest importance are abstracted in this department.

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

966,389. Wet Process for the Treatment of Ores. H. T. DURANT, *et al.*, patented August 2, 1910.

This invention relates to wet processes for the treatment of metalliferous ores or compounds particularly of zinc, and more particularly to processes in which ores containing zinc oxid are treated with a solution of sulfurous acid so as to obtain a solution of bisulfite of zinc.

Hitherto it has been proposed to effect the agitation of the ore or compound with water or other liquid in an apparatus comprising a tank having a pipe open at both ends and arranged vertically therein, and means for introducing at the foot of the pipe a lifting medium such as compressed air for raising the liquid contents of the tank up through the aforesaid pipe thereby promoting a circulation of the said contents. Apparatus of the general type in which a jet or injector is used



to cause circulation of a mixture of ore and liquid will be hereinafter referred to as an injector agitator.



According to this invention an injector agitator is supplied with a medium which does not merely promote circulation of the contents of the apparatus but has a chemical effect upon the said contents. The aforesaid medium may be in the form of a soluble gas, in which case it will become absorbed by the liquid in the agitating apparatus, or it may be already in solution when introduced. For example, when treating zinc ores suspended in water with sulfur dioxide, the sulfur dioxide derived from the roasting of the sulfid ore, or otherwise, may be introduced directly at the foot of the vertical pipe in the injector agitator containing the roasted ore in suspension in water; or the gas may be first absorbed by water and the dilute sulfuric acid solution so obtained may be injected under pressure up the aforesaid pipe. In the former case the gas will soon become absorbed by the water forming dilute sulfuric acid and in either case the dilute acid solution will exert a chemical action on the roasted ore in the known manner.

Another example of the application of this invention to the zinc bisulfite process already referred to resides in the use of steam or hot gases in an injector agitator for the conversion of soluble bisulfite of zinc into the insoluble monosulfite. The steam or hot gas may be introduced at the foot of the pipe in an injector agitator and a reduced pressure may be maintained on the surface of the bisulfite of zinc solution, the result being the heating and agitation of the contents of the apparatus and the conversion of the bisulfite of zinc into the monosulfite with evolution of sulfur dioxide. Zinc or other ores may be similarly treated with other appropriate chemically active gases, either free or in solution. Ores containing zinc or copper may be, for example, treated in the manner hereinbefore described with ammonia either in the hydrated or gaseous form. The soluble gas or gases or the solution thereof may be used in substitution for the mere lifting medium hitherto employed in the injector agitator in addition thereto.

The accompanying illustration shows the apparatus in which the process is carried out.

967,105. Process for the Separation of Gaseous Mixtures into their Elements. GEORGES CLAUDE, *et al.* Patented Aug. 9, 1910

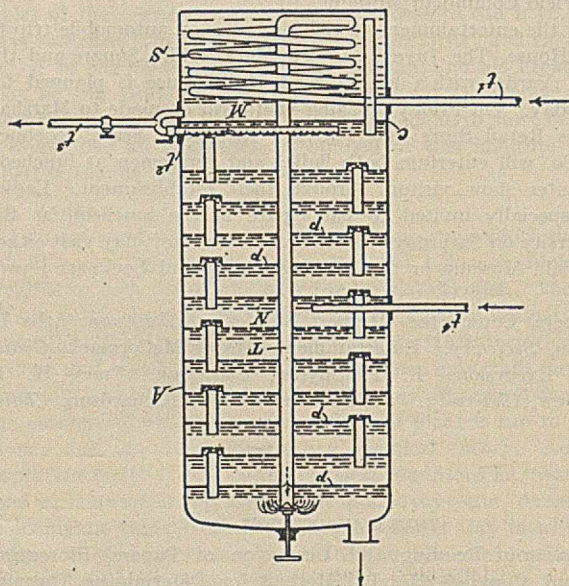
This is a process for the separation of oxygen in any desired degree of purity from atmospheric air by means of liquefaction and evaporation.

This process is based upon the fact that in order to transform liquid air into very pure liquid oxygen by the progressive evaporation of the nitrogen and the simultaneous liquefaction of a corresponding quantity of oxygen, it is sufficient to bring nearly pure cold gaseous oxygen into contact with liquid air in any manner, as for instance by causing the gaseous oxygen to pass in bubbles through the liquid air. By evaporating the liquid oxygen which is obtained we obtain a quantity of gaseous oxygen greater than that used, and if the apparatus is suitably devised, greater than the quantity of oxygen previously contained in the liquid air used, and this entire excess may be furnished by the atmosphere. This evaporation of the oxygen is made use of in our invention. It is effected by forcing cold air under suitable pressure into a tubular vessel or into a coil immersed in liquid oxygen. The evaporation of oxygen will thus cause the condensation of a substantially equivalent quantity of air under pressure, and we thus obtain a quantity of liquid air nearly equal to that at the start which, in its turn, is separated into pure oxygen by repeating the same operations.

In order to give this method its highest practical value, it is essential that the oxygen gas be utilized as completely as possible; the simple passage in bubbles through the liquid mass would yield poor results, for it is evident that the gas at its outcome from the liquid holding a large quantity of oxygen

and the loss would be greater than the gain. It is therefore of importance that this passage of the oxygen gas through the liquid air be carried out in a particular manner, for instance, by supplying the liquid air to be converted into oxygen at the upper part of a column provided with baffle plates or other suitable device and by supplying the gaseous oxygen at the lower end of the column, so that the gas travels through the successive chambers and issues from the column at the point of supply of the liquid air.

It is well known that if the liquid air at its formation contains, like the atmospheric air, about 21 per cent. of oxygen, the gas at its issue contains only about 6 or 7 per cent. of oxygen. If it be necessary to retain these quantities, it is possible to provide the column with an extension, at the end of which



there is fed liquid air containing less than 21 per cent. of oxygen. The losses in cold in the column can be compensated for either by the introduction of liquid air obtained from an auxiliary source or by the expansion of the air to be liquefied, compressed for this object.

The figure shows an apparatus in which the process is carried out.

967,996. Method of Extracting or Eliminating Sulfur, Phosphorus, and Other Impurities from Coal Ore, Etc. LELAND L. SUMMERS. Patented August 23, 1910.

This invention relates to a method of eliminating those impurities from coal, ore, etc., which are detrimental and troublesome in the manufacture of iron and steel, and the object is to provide a simple and efficient method of removing such impurities from the raw material used. Thus, according to this method, in the case of coal to be coked, the sulfur and phosphorus are removed from the coal before the same is charged into the coking appliances, and in the case of ore, the impurities are removed previous to charging the ore into the blast furnace or other refining apparatus.

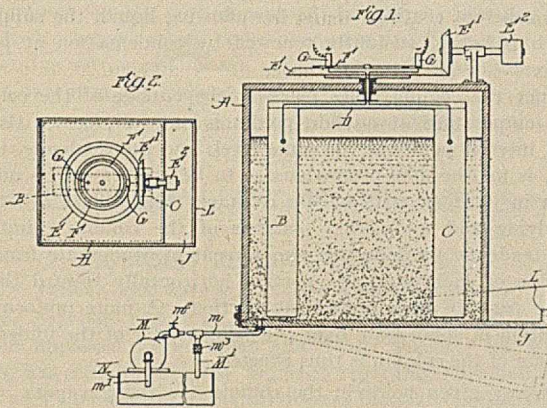
In general terms, the invention consists in providing a method by which the sulfur and phosphorus is chemically acted upon and its form of occurrence changed so that it may be removed as a different chemical compound.

The method is based essentially upon the fact that upon the passage of an electric current in a suitable electrolyte, electrolytic decomposition will result. According to this method as practiced, the coal or ore to be electrolyzed is preferably in a finely subdivided form and is submerged in the electrolyte,



so that upon revolving the electrodes *B* and *C* a continual agitation of the substance is in contact with the products of electrolysis.

In the use of the apparatus illustrated in the practice of the process, the rotation of the electrodes *B* and *C* in addition to agitating the electrolyte, will cause a portion of the material



treated to first be subjected to the acid and oxidizing reaction and immediately afterward to be subjected to the alkaline and reducing action. Thus, the bisulfid and monosulfid of iron in the vicinity of the anode will be violently reacted upon, the bisulfid being oxidized and the monosulfid being decomposed as before mentioned. Immediately after this action, the same particles will be subjected to the action of the sodium hydroxid liberated by the cathode, this being caused by the rotation of the electrodes so that the cathode and anode are changing places. The rate of rotation is determined by the strength of the reaction desired.

968,954. Metallurgical Furnace. JOHN E. JONES. Patented August 30, 1910.

This invention relates particularly to furnaces for treating or sintering ores, flue dust, and other comminuted materials, preliminary to further treatment in a blast furnace or other reduction apparatus.

In processes of obtaining metals such as iron, zinc, copper and the like now in common use, large quantities of dust and material rich in metal, but too fine to be smelted, are obtained. Also various metals occur in the form, or ores which are so fine as to require sintering or nodulizing prior to being reduced.

This invention has for its object the production of a furnace for the treatment of such materials whereby ores can be de-

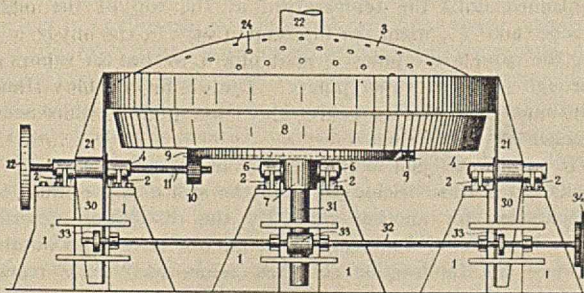


Fig. 1

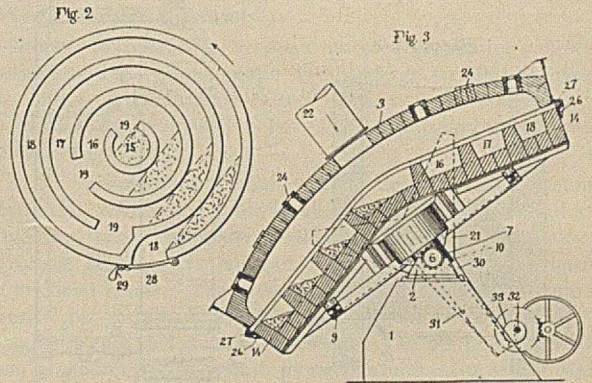
sulfurized, and also formed into nodules by partial fusion, and thereby rendered capable of economic treatment without undue loss in the blast furnace.

According to this invention, the patentee has provided a

furnace wherein the finely divided material to be treated is fed into the center of a rotary inclined hearth provided with a tortuous path, such as a spiral, but more preferably with communicating grooves surrounding each other, the material dropping from one groove into the adjacent groove by gravity, as the hearth rotates, and in conjunction therewith, a relatively stationary hood is provided whereby to form a heating chamber enclosing the hearth. In conjunction therewith means are provided for applying heat to the interior of the furnace, and preferably, the heating means are so disposed as to direct a flame directly on the material undergoing treatment.

The invention further comprises a furnace of the general character above described wherein the hearth will be mechanically driven, and also mounted in such manner that the inclination of the hearth can be adjusted as may be desired without interfering with the arrangements for heating the interior, or with the driving means.

In the specific embodiment of the invention herein shown, the grooved hearth is made in the general form of a cone, that is to say, the grooves or channels containing the material being disposed in different plane, and the hearth itself being mounted on trunnions whose axis coincides with the axis of the driving



shaft, whereby permitting the hearth, together with the table, to be swung on the trunnions to any desired angle and still maintain the driving connection.

968,760. Obtaining Petroleum Products. HERMAN FRASCH, of New York, N. Y. Patented August 30, 1910.

This invention relates to the production of useful products from a certain kind of petroleum which is characterized as hereinafter set forth and is herein termed Coalinga oil, or Coalinga petroleum for convenience of reference. It is found in the neighborhood of the town of Coalinga, in the county of Fresno, and State of California. Ordinarily, on distilling petroleum and testing the gravity of successive samples of the distillate as it runs from the tail pipe of the condenser, it will be found that the distillate becomes gradually denser and less volatile as the temperature in the still rises. In other words, the boiling points and densities of the hydrocarbons of petroleum usually increase together; and the greater the density of a hydrocarbon from a given crude petroleum, the higher boiling point may it ordinarily be expected to have. The patentee claims to have discovered in the Coalinga petroleum an extraordinary exception to this general rule; that is to say, he claims to have discovered in Coalinga petroleum an oil which, on distillation, may at intervals exhibit retrogressions of density in the distillate, while the volatility of the latter continues to decrease. Coalinga oil, therefore, contains hydrocarbons which are specifically heavier than others of higher boiling point in admixture therewith.

The still *a* is supported and heated in any suitable way. The



column *b* is shown as made in sections, and has a perforated or reticulated floor plate, *c*, at the bottom of each section, which floor plate supports the finely divided solid material *d*, say, two feet of gravel for each section. The gravel in the two lower sections may be such as to pass through a sieve of two meshes to a linear inch and to be arrested by one of three meshes to the linear inch; that in the upper sections may be a little smaller, say, gravel which will pass through the last-mentioned sieve and be arrested by one of four meshes to the linear inch. Either size of gravel could be used in all the sections, but it is considered better to have the gravel of the lower sections a little larger on account of the larger volume of liquid of condensation therein.

Below each floor plate, *c*, is a collar, *e*, for directing the down-flowing liquid of condensation from the walls of the column toward the axis thereof.

The column *b* is covered with a coating, *f*, of an inch and a half of asbestos cement.

The vapor pipe *g* leads from the top of the column *b* to the condenser, of known or suitable construction. Branch pipes, *h*, with valves, *i*, therein are shown connecting with the space

as customary. As the mixed vapors rise through the tall column of finely divided solid material, they become partially condensed to a liquid, which immediately commences to descend toward the still *a*, covering the pieces, *d*, of solid material in its passage. The cooling of the air, modified by the asbestos jacket *f*, gives a gradual fall of temperature in the column from the bottom upward, and keeps the solid pieces effectively covered with coherent coatings of the downflowing liquid, the supply of which is being constantly renewed by condensation at points above.

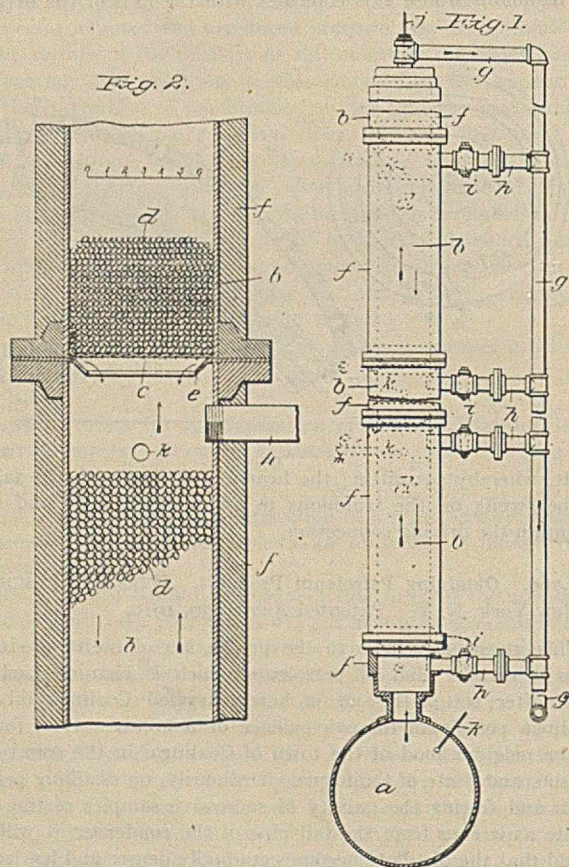
Since the cooling takes place at the outside of the column, the temperature at outside points is naturally lower than it is at interior points at the same level; and the tendency, therefore, is for the flow of condensate to be greater at the outside than in interior parts of the column. The collars *e* between overlying and underlying portions of the column counteract this tendency by deflecting the current of condensate from the outside of each overlying portion horizontally toward the interior parts of each underlying portion. A more uniform distribution of condensate over the solid matter of the underlying portion of the column is thus effected.

At any given point in the column the rising vapors, which consist of a mixture of hydrocarbons of different boiling points, are meeting and coming into intimate contact with the descending liquid, which consists likewise of a mixture of hydrocarbons of different boiling points. Of these liquid hydrocarbons, some are of lower and some of higher boiling points than a part at least of the vaporized hydrocarbons, which meet them in any particular point. There are consequently repeated fractional condensations from the vapors, and fractional re-evaporations from the liquid of condensation, until the well rectified vapors escape by the vapor pipe *g*.

The fire under the still *a* may be so regulated as to distil off about 2 per cent. per hour of the charge placed in the still *a* at the beginning of the run. As distillation proceeds, the temperature in the still *a* and column *b* rises; the density of the distillate at the tail pipe of the condenser for the most part increases and the volatility of said distillate steadily diminishes, but at certain periods said density gradually diminishes while said volatility continues to decrease. Such a retrogression in density of distillate takes place before the thermometer *j* reaches 260° F.; and when this temperature is reached, the distillate receiver may be changed, so that distillate next collects in the new receptacle.

The distillation being continued, the density of distillate gradually increases with the rise of the distilling temperature until another retrogression of such density takes place which occurs before the temperature at the top of the column reaches 320° F. When this temperature is attained the distillate receiver may be again changed. The distillation may then be continued until the temperature at the top of the column reaches 400° F., when the top section may be cut out by opening the valve *i* in topmost branch pipe *h*, so that the vapors pass through it to the vapor pipe *g*. The distillation may then be continued until the temperature at the top of the third section reaches 485° F.

It could be continued longer, or stopped earlier. The residuum is then discharged from the still *a* and column *b*, in preparation for another run. By the distillation described, three fractions of Coalinga petroleum will have been obtained, namely (1) the fraction collected before 260° F. is reached; (2) the fraction collected before 260° F. and 320° F.; and (3) the fraction collected between 320° F. and, say, 485° F., more or less. The first contains the benzene and toluene of the crude oil, the second the xylene and some of the mesitylene, and the third the rest of the mesitylene and the naphthalene. This separation is not expected to be perfect, however. In making it as described, the hydrocarbons which come over near



below each column section, for enabling the vapors to pass to the condenser direct from the still *a* or after passing through any one or more of the column sections as may be desired.

A thermometer, *j*, is placed in the top of the column *b* to indicate the temperature of the vapors entering the vapor pipe *g*; and there may be thermometers at the points *k*.

This distilling and rectifying apparatus being at hand, the still is supplied with the crude Coalinga petroleum; and the temperature of the still and contents are raised to the distilling point. After a time the vapors pass over by the pipe *g* to the condenser, where they are condensed to a liquid, which runs out of the tail pipe into the customary box, which is so arranged that the flow can be observed and samples taken for testing,



the beginnings of the respective retrogressions would not be collected apart from those which come over near the endings thereof.

Each of the so obtained fractions is distilled by itself with rectification in the column *b*, or with other rectification able to effect the aforesaid retrogressions of density of distillate contemporaneously with continued decrease of volatility; and the hydrocarbons which come over near the beginnings of the respective retrogressions are collected apart from those which come over near the endings thereof.

The inventor is Herman Frasch, of New York, N. Y.

## INDUSTRIAL AND TRADE NOTES.

### GERMAN RESINITE COMPOUND.

(From Consul Thomas H. Norton, Chemnitz, Germany.)

The previous report on the new German material, resinite, which appeared in Consular and Trade Reports for May 7, 1910, has elicited so many inquiries regarding its manufacture and uses that the following additional facts are presented:

The essential feature in the technical method now used to produce resinite is the condensation of phenol and formaldehyde. By condensation is understood the union of two or more molecules of organic compounds, with elimination of one or more molecules of water. The operation is, in this last respect, comparable to that which occurs when an acid and a metallic hydroxid react to form a salt and water is liberated. In most organic compounds condensation is effected by bringing them in contact with other substances which have an affinity for water and thus favor the reaction.

The condensing agents used by the inventors of resinite are certain mineral salts, such as sodium sulphite, ammonium sulphite, tertiary sodium phosphate, sodium acetate, and a few others. All of these salts are neutral or exert a feeble alkaline reaction. The use of these simple agents constitutes the principal peculiarity of the patented process, as contrasted with the employment of acids or alkalis, methods which have been practised for many years in effecting synthetic condensations similar to those giving rise to resinite. The inventors claim that by the use of the milder, less energetic agents the ensuing reaction is under more perfect control than when acids are employed, and that it is possible to isolate certain valuable intermediary products through the ease with which the operation can be interrupted at any point. The condensing agents may properly be considered as catalytic substances.

In actual manufacture phenol (carbolic acid) is mixed with an aqueous solution of formaldehyde (40 per cent.) and heated, after the addition of one of the salts mentioned above. The product is a yellowish, mobile liquid, which is termed resinite-A. It is admirably adapted for impregnating wood, paper, and other porous materials, and thereby rendering them waterproof.

If resinite-A be freed from the water present by distillation, a residual product is secured, termed resinite-B. The properties of this substance vary, according to the rapidity of the distillation and the prevailing temperature. Normally it is hard under ordinary conditions, but becomes plastic as the temperature rises. If it be now heated for a long time at 80° C., and then the temperature be raised to 200° C., resinite, properly so called, is the result—a perfectly solid, infusible, insoluble substance, exceedingly resistant against both acids and alkalines. Ordinarily exceedingly resistant against both acids and alkalines. Ordinarily it is of a ruby-red or purple tint. The color is yellow if ammonium salts have been used as condensing agents. It is transparent, or translucent, highly lustrous, and shows a conchoidal fracture. The ease with which it can be colored through

the application of anilin dyes to the surface, or throughout the entire mass, renders it of value for a multitude of technical purposes. It is used for the heads of hat pins, for chandeliers, for decorating signs, etc. It can be ground, turned, polished, etc., but as it is somewhat brittle, its use in this form is subject to certain limitations.

Resinite can be modified during the process of formation by the addition of a variety of substances, such as talcum, infusorial earth, starch, etc. The resultant product is no longer transparent, but it has acquired the important property of elasticity, and can now be easily worked.

For certain purposes, for example, in forming buttons from resinite, the long-continued heating, for at least two hours, as involved in the process as outlined above, is practically out of the question. In such cases treatment with hydrochloric acid is employed. If resinite containing 20–30 per cent. starch be warmed with dilute hydrochloric acid, a viscous product is obtained which can be poured into molds, rolled into sheets, etc. The acid employed for this "tempering" is easily removed by washing with a dilute soda solution. The solidified mass is no longer brittle, but soft and easily worked upon the lathe or with various tools. When poured into molds very clear, sharply defined results are obtained.

Numerous difficulties were encountered in coloring masses of resinite, as the various anilin dyes undergo striking changes in the course of the final operation. It is now possible, however, to secure resinite in any color except white and gradations of white.

In its various modifications resinite shows valuable, technical properties, resembling those of glass or celluloid in some forms, while in others it approaches ebonite, horn, vegetable ivory, or Japanese lacquer. It has but little in common with natural or artificial resins. The inventors propose the general name of "resit" for resinite-B and similar compounds and "resol" for resinite-A and analogous substances. (The address of the German manufacturer of resinite is on file in the Bureau of Manufactures.)

It is worthy of note that an American invention closely corresponds with resinite in many of its properties and applications. It is of wide use in the United States as an insulating material in electric installations and for other purposes. One of the largest chemical companies in Germany has recently begun manufacturing under its patents for the European trade. This substance is called "bakelite." The essential features of the American invention consist in the use of bases as condensation agents and in the combined action of heat and high pressure to effect the final change into the hard, insoluble modification. There would appear to be a certain measure of conflict between the claims of the two patentees.

The exact chemical composition of these materials has not yet been established on account of the chemical inertness of the substances. The analytic results harmonize with the empirical formula  $C_{43}H_{38}O_7$ .

### GERMAN POTASH PRICES.

THE SYNDICATE FIXES QUOTATIONS TO DOMESTIC CONSUMERS.

Consul Talbot J. Albert writes from Brunswick that negotiations have been in progress for some time between the new potash syndicate and the associations belonging to the organized German agriculture as to the prices for raw and fertilizing salts to be furnished for the use of the domestic agriculture.

An agreement has been reached and the following are the prices per double centner of 220 pounds for raw salts, based upon the maximum prices established by the potassium law just enacted (mark = 23.8 cents):



	Raw salt.	Now.	Formerly.
Karnallit:		Marks.	Marks.
9 per cent. potassium.....		0.765	} 0.90
10 per cent. potassium.....		0.85	
11 per cent. potassium.....		0.935	
Hartsalz and sylvanit:			
12 per cent. potassium.....		1.20	} 425
13 per cent. potassium.....		1.30	
14 per cent. potassium.....		1.40	
15 per cent. potassium.....		1.50	

Formerly, when sylvanit contained more than 13 per cent. potassium, there was an addition of 10 pfennigs (2.3 cents) for each half per cent. potassium, unless the customer ordered expressly lower—that is to say, normal per cent. goods. In the case of hard salts formerly an excess of 12.4 per cent. potassium was not taken into consideration in the reckoning. The syndicate now seeks, in consequence of the reduction of prices for normal marks which has to be made under the law, to equalize prices partly by imposing on orders for salts having a larger percentage potassium than the normal an additional sum to that formerly charged. No just objection can be made against this method by consumers, as it enables the reasonable employment of the better works.

#### PRICES OF FERTILIZING SALTS.

The prices of manure salts, which also show greater differentiation, are as follows, per 220 pounds: Containing 20 per cent.  $K_2O$ , 2.80 marks; 21 per cent., 2.94 marks; 22 per cent., 3.08 marks; 30 per cent., 4.35 marks; 31 per cent., 4.495 marks; 32 per cent., 4.64 marks; 40 per cent., 6.20 marks; 41 per cent., 6.355 marks; 42 per cent., 6.51 marks.

There is an additional charge to all prices of 0.04 mark for transport from the works to the shipping station; for mixing with 2½ per cent. turf mull the price is increased 10 pfennigs. All prices are understood to be for 100 kilos (220 pounds), with 2½ per cent. discount at the works. For 100-kilo sacks for karnallit, kainit-hartsalz, and sylvanit there is an additional charge of 40 pfennigs per sack and for manure salt sacks 47 pfennigs.

#### REBATES—EXPORT PRICES NOT FIXED.

The question of rebates has been regulated in the following way: In case of the annual purchase of 10–500 double centners of kali (potassium salts) the discount for karnallit containing 9–11 per cent. potassium is 3 marks; for kainit-hartsalz, and karnallit containing 12–15 per cent. potassium, 5 marks; for manure salts containing 20–22 per cent. potassium, 8 marks; containing 30–32 per cent., 12 marks; and 40–42 per cent., 15 marks. In case of the annual purchase of 501–2,000 double centners the rebate rates have the following sequence, namely, 4, 7, 10, 14, and 19 marks, and in case of the purchase of more than 2,000 double centners of salts the sequence is 6, 9, 12, 17, and 23 marks.

In order to secure the second grade of rebates on 501 double centners of salts there must be an annual purchase of about 40 carloads of kainit-hartsalz or about 12–13 carloads of manure salts containing 40 per cent. of potassium, or about 30 carloads of kainit and 3–4 carloads of manure salts containing 40 per cent. of potassium, equivalent to 100 double centners. What is specially to be noted is that by these rebates no commission or allowance of any kind, directly or indirectly, is to be made to the consumer.

As has been said, these prices are for the German agriculturists. No prices have as yet been published for the American importers. The difference which arose some time since between the old syndicate and the mines outside of the same who had contracts with American companies have not yet been settled. There are continuous negotiations between the new syndicate and the outside parties, but no result has been reached. The foregoing prices will at least be valuable to show whether any

discrimination is made in the future against the American farmer.

#### WATER PURIFIED BY OZONE.

(From Consul William Dulany Hunter, Nice, France.)

As the water used here for drinking and other purposes was considered to be unwholesome, a new method of sterilization has been adopted. There is an electric power plant worked by water turbines, the force being the water which is afterwards sterilized. The two dynamos are run separately, so that there can be no stoppage while one of them is being cleaned or repaired. The force produced is 110 volts, 500 periods per second, which is transformed into an alternating current with a potential power of 17,000 volts. This high-power current is conducted to a so-called ozone battery. Each of the five batteries composing a system consists of three vertical copper plates 2 feet square and 1½ inches thick, with a space of 8 inches between each; in each of these spaces there is a pair of glass sheets, between which the electric sparks decompose the air, which is forced through them by a sucking machine. The decomposed air consists of ozone and azotic acid. The azotic acid is retained and the pure ozone made available by passing the decomposed air through a vertical vitrified pipe containing charcoal dust and pieces of cement. The water, under the pressure of a fall of 12 feet, flows through a system of earthen tubes about 1 inch in diameter and having 33 holes each; these tubes pass through a space filled with ozone, which the water absorbs.

After this first process of sterilization the water flows into a tank in which there is a wire netting supporting about 3 feet of pebbles; these pebbles divide the water so that it falls like a heavy rain to the bottom of the tank and a strong ozone current, coming from the sides of the tank, is absorbed by the falling water. The ozone is then extracted by having the water fall on stone steps. Medical authorities declare that after having undergone this process water is absolutely germ-free and that it is impossible to produce any kind of germ culture in it.

Two ozone plants were built in 1909; one of them gives an output of 39.61 gallons per second, and the other 79.22 gallons. Each of these plants is composed of two separate systems, so that there can be no possibility of a shortage of water. A new plant is under construction which is to be large enough to supply all the towns and cities between Nice and Mentone, a distance of 24 miles. New water-works are also to be constructed in the near future at both Cannes and Grasse.

#### WORLD METAL PRODUCTION.

(From Consul-General Richard Guenther, of Frankfurt.)

From the annual reports of three German joint stock metallurgical companies, with head offices here, the following translation has been made of certain statistics on metal production and consumption:

The world production of copper in 1909: aggregated 844,100 tons (metric ton = 2,204.6 pounds), which exceeds by about 100,000 tons that of the year before. The United States had the largest share, 498,200 tons, an increase of about 16 per cent. over the output in 1908. The world consumption of copper in 1909 reached 782,800 tons, exceeding that of the preceding year by 85,100 tons. The giant share in the consumption fell to the United States, and amounted to 318,900 tons (against 208,800 tons in 1908); Germany consumed 179,100 tons; England, 109,100 tons; France, 73,100 tons.

The world production of lead last year aggregated 1,052,500 tons, divided thus: Europe, 505,800 tons; United States, 339,700 tons; Mexico, 118,000 tons; Australia, 77,200 tons. The United States consumed 365,200 tons of lead; Germany, 213,200 tons; Great Britain, 199,500 tons; France, 110,400 tons; Russia, 38,300 tons.



Of tin, 108,300 tons were produced in 1909, of which 61,500 tons came from the Straits Settlements ports; 35,600 tons from Bolivia; Germany produced (mainly from Bolivian ores) 8,990 tons. The consumption of tin was distributed: United States, 42,800 tons; England, 17,500 tons; Germany, 17,100 tons; France, 8,750 tons; and Belgium, 1,300 tons.

The zinc production in 1909 was 783,200 tons, of which the United States contributed 240,446 tons; Germany, 220,100 tons; and Belgium, 167,100 tons.

In 1909 the world produced 16,100 tons of nickel, the chief contributors being United States, 9,000 tons; Germany, 3,100 and England, 2,800 tons.

Of aluminium, 24,200 tons were produced and 30,800 tons consumed throughout the world, while the production of quicksilver amounted to 3,200 tons, of which Great Britain took 1,445 and Germany 723 tons.

**POTATO STARCH IN GERMANY.**

(By Vice-Consul-General Frederic W. Cauldwell, of Berlin.)

Potatoes are the principal source of the starch manufactured in Germany, and that potato starch, known as "kartoffelmehl," enters extensively into German export trade.

The potatoes are first thoroughly washed and then mashed between heavy rolling cylinders, on the surface of which there are grooves and teeth to tear up the potato cells. The starch is washed out of this ground-up pulp by means of running water. The resulting so-called "starch milk" is first drained through wire or milk screens or through perforated copper plates to remove the cell pulp, after which it is allowed to flow directly into cement settling vats or caves, or it may first be led over small, gently sloping troughs or drains, upon which the purest of the starch then settles.

The starch from the settling vats and from the settling drains is refined by being rinsed in stirring vats, and the impurities and cell substances are skimmed or dipped off. The water is then removed by special centrifugal hydro-extractors and the resulting so-called "green starch" is generally worked up into dextrin and sugar. For producing the starch of commerce it must be further dried in specially constructed drying chambers. The potato pulp, or cell stuff, left over is subjected to a renewal of the refining process, or it may be dried and fed to stock.

(A detailed report on potato-desiccating machinery, by Consul Thomas H. Norton, of Chemnitz, appeared in *Daily Consular and Trade Reports* of February 19, 1910.)

**IMPORTATION OF TUNGSTEN FROM THE PLAUEN DISTRICT.**

According to Consul Carl Bailey Hurst, tungsten powder, heretofore rarely sent from the Plauen district, was shipped to the United States in 1909 to the value of \$34,996. This metal, produced from wolfram ore found in that district, has been used in Germany in a process for hardening steel. It promises to become an important item of export as it is more extensively taken up in the American steel works.

**OFFICIAL REGULATIONS AND RULINGS.**

**NOTICE OF JUDGMENT NO. 497, FOOD AND DRUGS ACT.**

IN THE CIRCUIT COURT OF THE UNITED STATES  
SOUTHERN DISTRICT OF IOWA  
CENTRAL DIVISION.

SHAWNEE MILLING COMPANY, *Complainant*,

*vs.*

MARCELLUS L. TEMPLE, United States District Attorney, and FRANK B. CLARK, United States Marshal, *Respondents*.

No. 2490.  
Equity.

THE UPDIKE MILLING COMPANY, a corporation, *Complainant*,

*vs.*

MARCELLUS L. TEMPLE, as United States District Attorney for the Southern District of Iowa, FRANK B. CLARK as United States Marshal for the Southern District of Iowa, and A. BROWN, full first name unknown, as Food & Drug Inspector of the United States Department of Agriculture, *Respondents*.

No. 2492.  
Equity.

**SUITS TO RESTRAIN SEIZURES, UNDER SECTION 10 OF THE ACT, OF COMPLAINANTS' FLOUR BLEACHED BY THE ALSOP PROCESS.**

On or about December 14, 1909, the Shawnee Milling Co., a corporation, of Topeka, Kans., filed in the United States Circuit Court for the Southern District of Iowa a bill in equity naming as defendants thereto Marcellus L. Temple and Frank B. Clark, United States Attorney and United States Marshal, respectively, for said district, alleging that said defendants were about to proceed without warrant of law and to the detriment of complainants' rights to make seizures of flour bleached by the Alsop process, under Section 10 of the Food and Drugs Act, and further alleging that said act was unconstitutional, and praying that said defendants be enjoined from proceeding under said act and from making seizures of complainants' flour bleached as aforesaid pending the determination of this case.

Subsequent to the filing of the above bill the Updike Milling Co., a corporation, of Nebraska, filed in said court a similar bill against the same defendants and A. Brown, a food and drug inspector of the United States Department of Agriculture, containing substantially the same allegations and prayers. The material allegations of both bills of complaint appear more fully in the opinion of the court hereinafter set out.

To both bills of complaint the defendants filed a demurrer alleging as grounds therefor that complainants' bill failed to any cause which would entitle them to the relief sought in said bills.

On April 26, 27, and 28, 1910, the cases came on for hearing and the questions of law raised by the bills of complaint and the demurrers thereto were fully argued to the court. On May 10, 1910, after full consideration, the court dismissed complainants' bills, delivering the following opinion:

OPINION.

SMITH MCPHERSON, Judge.

Each of these two cases is by a bill in equity, practically the same. One of the complainants, Updike Milling Company, is a corporation under the laws of Nebraska, there engaged in the business of manufacturing wheat into flour both for domestic use, and for shipments into Iowa and other states for sale and consumption. The other complainant, Shawnee Milling Company, is a corporation under the laws of Kansas, there engaged in a like business, sales and shipments.

The defendants are the United States Attorney and Marshal for this District, and the relief sought is to enjoin the respondent officers from having issued, or serving process for seizing complainants' flour in interstate shipments under the National Pure Food statute of June 30, 1906.

The allegations are that complainants' flour is whitened and aged by a process, and that the same is not harmful, but is more nutritious, wholesome and attractive for making bread. It is not alleged in the bill of complaint in terms that the flour is bleached by the Alsop process as covered by certain English and American patents as set forth by the Circuit Court of Appeals for this circuit in the case of Naylor *vs.* Alsop Process Company (168 Fed. Rep., 911), but all the arguments, both by briefs and orally, were on that state of facts. Counsel for the



United States have appeared for the defendants, thereby in effect making the cases controversies between the United States Government on the one side, and western flour mill owners on the other, who bleach their flour by the agency of nitrogen peroxide under the Alsop Patent process.

A literal reading of the bills of complaint will show that they are fairly subject to the criticism, that the allegations as to the aging, whitening and improving the flour are largely by the use of adjectives and adverbs, instead of reciting just what is done; how the flour is aged; how whitened; how made more nutritious; why not harmful; and why better by the use of some agency not named nor described. But this criticism need not be elaborated. The cases are now for determination on demurrers to the bills of complaint, and sufficient allegations appear to cover the rulings now to be made.

A bill in equity in which the writ of injunction can issue to enjoin the enforcement of a criminal or penal statute is allowable only when:

1. Such statute is unconstitutional or otherwise invalid;
2. In an attempt to enforce such invalid statute, rights of property are invaded and trampled on; or,
3. The often repeated attempts to enforce such invalid statute creates a multiplicity of actions which are of themselves oppressive.

The important and recent case of *Ex parte Young* (209 U. S. 123) illustrates this, in which case it was held that a bill in equity would confer jurisdiction because of the oppressive penalties if an effort should be made to protect the rights of property. In *City of Hutchinson vs. Beckham* (118 Fed. Rep., 399), the Circuit Court of Appeals for this district held that an injunction should issue against the prosecution of cases under an invalid ordinance requiring an illegal license, which would be followed by many criminal prosecutions. In *Dobbins vs. Los Angeles* (195 U. S., 223, 241), the holding was clearly and tersely stated:

"It is well settled that where property rights will be destroyed lawful interference by criminal proceedings under a void law or ordinance may be reached and controlled by a decree of a court of equity."

But if property rights are not invaded, then a court of equity ordinarily will not interfere, because the defense as to the invalidity of the statute can be urged in the criminal or penal action or special proceeding. Thus, in the case of *In re Sawyer* (124 U. S., 200), it was held that proceedings for the ouster of a city officer could not be enjoined for the alleged invalidity of the law under which the proceedings were being conducted. And of like holdings are the cases of *Hardrader vs. Wadley* (172 U. S., 148), and *Fitts vs. McGhee* (172 U. S., 516).

And if the proceedings for seizure are to be regarded as civil, then section 723, R. S., will prohibit the filing of a bill in equity to enjoin the enforcement of a valid statute.

In the one case now before the court, the bill of complaint recites that several seizures of flour were made in this judicial district, and after a number of efforts by the complainant to have the cases submitted to the court with or without a jury for a hearing on the merits, the Government dismissed the cases, after the flour thus seized had deteriorated in quality and value.

In the cases now before the court as property rights are involved, bills in equity will be entertained, provided the statute under which the Government claims the rights to proceed is not a valid one. Herein is the question in the case; that is to say, Is the pure food statute of June 30, 1906, a valid enactment? Did Congress have the power to enact it? Is it within the commerce clause of the Constitution, or is it a mere police regulation erroneously garbed and cloaked as a regulation of commerce?

Good, sound wheat of the best variety, properly and timely harvested, put through the "sweat" in the stack, well ground and bolted, makes nutritious, wholesome, and white flour.

This fact is so generally known that courts will take judicial notice of the fact.

It is said that flour made from new and poorer wheat, not "sweated," and made by the process covered by the English patent of Andrews, or the American patent of Alsop as illustrated in the patent decision hereinbefore referred to (168 Fed. Rep., 911), will also be equally white. This is quite likely true. But is it equally pure, equally nutritious, or is it adulterated and poisoned?

This court in these cases is not to decide those questions. Nitrogen peroxide under the Andrews patent is produced by combining nitric acid with a metallic compound. Under the Alsop patent it is produced by subjecting atmospheric air to a flaming electric arc. It is claimed by some that nitrogen peroxide is the agent for bleaching flour under both patents, while others claim that it is the ozone that does the effective work, while the nitrogen peroxide is a by-product when the ozone is thereby created.

Whatever the truth is as to what does the bleaching, it is both claimed, and denied, by chemists who ought to be able to agree, that the flour is poisoned by such process. But it is known that after the air is thus subjected to continuous flaming electrical discharges, that the result and gas is conveyed by means of pipes to a compartment and there is commingled with the flour agitated or in a cloud, and thus subjected to said treatment it becomes dry and white. The result of it all is that new wheat and of an inferior quality is converted into flour with the appearance of flour from a better wheat that has been aged by time.

The Government contends that flour thus bleached is flour in the language of the statute "whereby inferiority is concealed," and that "it contains added poisonous ingredient which may render such article (flour) injurious to health." The patentees and the millers deny this.

Here is a question for determination by a jury, or by the court if a jury is waived, and not to be determined in this case if the statute is valid.

Several of the states within the past few years have enacted pure food statutes. Congress June 30, 1906, enacted the statute in question. All these statutes were enacted to cure evils well nigh intolerable that had grown up during this age of greed and avarice and commercialism that has made money-getting the prime object of life with so many. The evils were such that much of the foods we ate, whether meats of any kind, including fish, and poultry, or fruits in all forms, and breadstuffs, were so adulterated and "loaded" or "doctored" as to deceive the consumer. And the same was true of flavors and condiments. The evil as to confectionery and flavors and extracts was as great. Still greater was the evil as to drugs and medicines. In fact the evils were everywhere present, as to food and medicine, and other things. And to eliminate some of these evils and to enable the purchasers to receive what they ordered and paid for, many states passed statutes aimed at those frauds. But it was soon found that the states in some instances were disposed to condone as to some articles of local manufacture, and in many other instances the states were powerless to work out a remedy. Thereupon Congress, acting upon the theory that the evil was of national concern, enacted the statute in question. The debates in Congress show that the measure was earnestly fought as being one of paternalism, and a police regulation with which the states only could act.

The Secretary of Agriculture, Mr. Wilson, performed his duty both in letter and spirit when he submitted the question as to flour bleached by nitrogen peroxide to the Board of Food and Drug Inspection. And the board, the Secretary concurring, after a hearing given to all parties in interest, found that such flour is in contravention of the statute. Such finding is not binding as against the parties thus bleaching flour. But it is



conclusive as against all criticism for making the seizures and bring the question before the courts for determination.

Congress is given the power to provide for the general welfare of the United States. But without doubt if this legislation is sustained, it is because of that provision of the Constitution that provides that Congress shall have the power to regulate commerce among the several states. That provision is the life of the nation, and to adopt which was the great concern of the convention of 1787. Important as it is, it is ever before the courts. It gives great comfort to all who believe in one common country, and yet is antagonized oftener than any other provision of the Constitution, by those whose shield of defense is articles 9 and 10 of the amendments, as to the reserved power of the States.

No one claims that Congress can be the sole judge of its powers. All thoughtful persons concede that any court having jurisdiction in the first instance must pass upon the question of the powers of Congress, and that it is for the Supreme Court in the end to finally set the matters at rest. But so careful have our Congresses and Presidents been, that for the first hundred years of our Government, the Supreme Court found it necessary to hold that Congress had exceeded its powers in only twenty instances. (See Appendix to Volume 131, U. S. Reports, p. cccxxv.) And of those twenty statutes thus held void, not one related to commerce. Since then, the Supreme Court has held three Congressional enactments void. One was a statute making a judgment of convicting conclusive evidence against a party in another case. (*Kirby vs. U. S. Farmers Loan Co.*, 157 U. S., 429, and 158 U. S., 601.) The other, and only one from the organization of our Government to date as to commerce, is that of the employers' liability statute, enacted under the claim that the commerce clause would sustain it. (*The Employers' Liability Cases*, 207 U. S., 463.) If other enactments of Congress have been held void by the Supreme Court such cases have been overlooked, and it is believed there are none other. There are almost innumerable decisions touching the power of the states with reference to commerce. It would be to no purpose to discuss many of these authorities. And it would be needless waste of energy to discuss the many decisions relating to the use of the mails, for the obvious reason that a distinct clause of the Constitution empowers Congress to control our postal system, and there is not the slightest difference whether the mails thus carried are state or interstate.

Neither the court nor the parties are aided by a review of those matters. It must be and is conceded that police regulations alone are for the State, and not for Congress to deal with.

But it does not follow that if the subject matter to be regulated is one of commerce, that it is for the state alone to deal with, because such subject matter is also one that pertains to the morals, health, or good order of the community.

Thus when the question arose as to the inspection of meats for food, legislatures claiming that they alone could determine when and to what extent police regulations should be carried, the Supreme Court decided that such inspection also impinged upon the rights of commerce and were therefore void. (*Minnesota vs. Barber*, 136 U. S., 313; *Brimmer vs. Rebman*, 138 U. S., 78.)

It will serve no purpose to discuss the principle upheld in *Wilson vs. Blackbird Creek Company* (2 Peters, 245), that the State can regulate certain interstate commerce of a local character, if Congress had not acted, nor of that other principle upheld by Congress that the State can legislate with reference to liability of a party when doing an interstate business when Congress has not acted. (*Sherlock vs. Alling*, 93 U. S., 99.) The complete answer to those suggestions is that in the matter now before the court, Congress has acted. The question now for consideration is not as to the power of the State relating to commerce, as held in *Smith vs. Alabama* (124 U. S., 463), up-

holding a state statute requiring a locomotive engineer even though operating an interstate train to submit to tests for color blindness.

The question here is as to the power of Congress over articles of interstate commerce, even though such articles in the end become subject to state statutes. No one doubts but that wheat and flour, as well as all articles of food, are subjects of commerce, and when carried over and across state lines, are subject to be regulated by Congress. And it is no answer to say, that when adulterated, or wrongly labeled, because in the end they will fall under a state statute, that they when being shipped can not be covered by a congressional enactment. The liquor cases illustrate this. Because of all the subjects of commerce there is no one thing more peculiarly and distinctly and appropriately subject to regulation by the State even to the extent of prohibition than are intoxicating liquors. And yet Congress legislates with reference to liquors. The Wilson Act of 1890 provided that when liquors arrived in a State they should be subject to State laws. This statute was upheld in the case *In re Rahrer* (140 U. S., 545), thereby modifying the practical effect of the holding in *Leisy vs. Hardin* (135 U. S., 100), that the State could not interfere by legislation as to liquors shipped interstate as long as the liquors were in the original packages, while in *Rhodes vs. Iowa* (170 U. S., 412), it was held that the liquors must be in fact and actually delivered to the purchaser before the State laws became effective as to such interstate shipment. No one should doubt but that legislation by Congress can control the interstate subject of commerce for a time at least, and then the State by a police regulation can control.

If liquors do not sufficiently illustrate the question, lottery tickets will. The Louisiana Lottery was conducted by men of high repute and much renown. But it became a national scandal. It was struck at by denying it the use of the mails. The legislature of the State gave it encouragement; even its life. But Congress provided in addition that it should be a crime to carry lottery tickets from one State to another by means other than through the mails. Can any person doubt but that the Louisiana Lottery was or could have been made subject to the laws of Louisiana? And yet this congressional enactment was upheld in the Lottery Case (188 U. S., 321). But little need be said of that case. It was argued by counsel of great eminence. It was argued upon two separate occasions. It received the fullest consideration by the Supreme Court. Apparently no other case that was ever before that court received more attention and fuller consideration. Counsel for complainants herein concede all these things. And the only answer that has been made, or that can be made to that case, is in the statement that the case was decided by a divided court, four justices dissenting. It may be, or it may not be, that that weakens the case as an authority. It is barely possible that later on, that court changing as to its personnel, the decision may be overruled. But such reasoning is a mere speculation. On the other hand the fact that the court was so divided emphasizes the fact that the court gave great consideration to the question. But be these things as they may, it is not for this court to usurp the prerogative by blindly declining to follow that decision. That decision stands, and as long as it stands, it is the law of the country, and this court not only must, but does cheerfully observe it in all its phases.

Much more could be said. Cases commencing with *Gibbons vs. Ogden*, and then to date, could be reviewed. The question could be illustrated in many ways. But all that would be to no purpose; it would be academic.

Congress has enacted a safety appliance law for the preservation of life and limb.

Congress has enacted the anti-trust statute to prevent immorality in contracts and business affairs.



Congress has enacted the live stock sanitation act to prevent cruelty to animals.

Congress has enacted the cattle contagious disease act to more effectively suppress and prevent the spread of contagious and infectious diseases of live stock.

Congress has enacted a statute to enable the Secretary of Agriculture to establish and maintain quarantine districts.

Congress has enacted the meat inspection act.

Congress has enacted the employers' liability act.

Congress has enacted the obscene literature act.

Congress has enacted the lottery statute above referred to.

Congress has enacted (but a year ago) statutes prohibiting the sending of liquors by interstate shipment with the privilege of the vendor to have the liquors delivered c. o. d., and to prohibit shipments of liquors except when the name and address of the consignee and the quantity and kind of liquor is plainly labeled on the package.

The statutes, police regulations in many respects, are alike in principle to the act of June 30, 1906, under consideration. Can it be possible they are all void?

This statute by its title, and by its every provision plainly shows that it is with reference to commerce, and that it is not with reference to local police regulations.

It is also contended that so much of Section 7 of the statute as relates to food is void because no standard has been fixed.

That argument is made because drugs are fixed by a standard recognized by the United States Pharmacopoeia or National Formulary, and as to confectionery a standard is fixed by declaring what confectionery *shall not* contain. Whereas as to foods no standard has been fixed. It is a fact most obvious that no standard could be fixed other than was done by Congress. The one provision as to food is, that it shall not be mixed so as to reduce or lower or injuriously affect its quality or strength. Another provision is that some substance shall not be substituted wholly or in part for the article. Another provision is that no valuable constituent of the article shall be abstracted. Another provision is that it shall not be mixed, colored, powdered, coated, or stained in a manner whereby damage or inferiority is concealed. Another provision is that poisonous or other deleterious ingredients shall not be added. Still another provision is that filthy, decomposed, or putrid substance shall not be added. And so on more in detail than herein enumerated. These provisions present questions of fact as to every alleged contraband article. This objection is without merit.

This case was argued upon both sides with most signal ability, displaying much learning, and was argued at great length. The case has received from this court the fullest consideration, and the conclusions are that these bills in equity cannot be maintained, and therefore will be dismissed.

DES MOINES, IOWA, May 10, 1910.

JAMES WILSON,  
*Secretary of Agriculture.*

WASHINGTON, D. C., June 25, 1910.

#### NOTICE OF JUDGMENT NO. 498, FOOD AND DRUGS ACT.

In the case of the United States ex rel. Alsop Process Company, petitioner, vs. James Wilson, Secretary of Agriculture, a mandamus proceeding in the Supreme Court of the District of Columbia to restrain the Secretary of Agriculture from publishing and circulating Food Inspection Decision No. 100 of the United States Department of Agriculture relative to flour bleached by the Alsop Process, and to compel cancellation of said decision.

On or about January 23, 1909, the Alsop Process Company filed in the Supreme Court of the District of Columbia a petition for a writ of mandamus directed to the Secretary of Agriculture alleging in substance that relator is a corporation engaged in the

business of manufacturing and selling machinery and apparatus used by millers for the bleaching of flour by so-called Alsop Process (giving a description of said process), and further, that the Secretary of Agriculture caused hearings to be held to determine whether flour bleached by the Alsop Process was adulterated within the provisions of the Food and Drugs Act of June 30, 1906, and after hearing the evidence for and against flour thus bleached, decided that, in his judgment, flour so bleached was adulterated within the meaning of the aforesaid act, and that the Secretary of Agriculture, without warrant or color of law, published and caused to be published the said decision designated as Food Inspection Decision No. 100, which publicly condemned as adulterated within the meaning of the Food and Drugs Act flour bleached by relator's process to the great damage of its business. The petition prayed that the Secretary of Agriculture be commanded to revoke and cancel and annul said decision and not to deliver or circulate additional copies thereof.

Upon the filing of the petition the court issued a rule directed to the Secretary of Agriculture as respondent requiring him to show cause by a certain date therein named why the prayer of said petition should not be granted.

Respondent duly answered said petition and to this answer the relator filed a demurrer. The case came on for hearing upon the questions raised by the above-mentioned pleadings and the court overruled relator's demurrer. The following is the opinion of the court delivered by Mr. Justice Stafford:

IN THE SUPREME COURT OF THE DISTRICT OF COLUMBIA.

UNITED STATES OF AMERICA EX REL. ALSOP PROCESS COMPANY, <i>Petitioner</i> ,	} At Law No. 51348.
JAMES WILSON, SECRETARY OF AGRICULTURE, <i>Respondent.</i>	

OPINION OF THE COURT.

By STAFFORD, J.

This is a petition for a writ of mandamus. A rule to show cause was issued which the respondent has answered and to this answer the petitioner has demurred. The case was heard upon the demurrer and would have been disposed of at the time had it not been that the court understood that the parties desired that an opinion should be filed dealing fully with all the points involved. The case has been left undisposed of in the hope that opportunity would be found to prepare such an opinion, but the pressure of other duties having thus far prevented, and no likelihood appearing that the same can be done within the next few days, it is thought best to dispose of the case without answering categorically the numerous points made in the brief of the petitioner. After all what the case amounts to is this. The Secretary of Agriculture has made up his mind that bleached flour is obnoxious to the provisions of the pure food act and has made that opinion public, announcing at the time that after six months, during which time the manufacturers and dealers will have an opportunity to adjust themselves to the situation, he will call upon the respective district attorneys to proceed against violators of the law. The petitioner claims to be the owner of a patent on the bleaching process and to be injured by the announcement of this opinion and intention. He is not the owner of any flour; he merely owns the patent and makes and sells the machinery. He says that the Secretary did not proceed according to the provisions of the pure food law in making up his mind; that he had no right to tell the public what opinion he had formed, nor what course he intended to pursue; that if he is going to recommend prosecutions at all he is bound to do so at once and not wait six months. He therefore asks this court, by the great writ of mandamus, to command the Secretary to vacate his decision, to take back what he has said, and hereafter to proceed strictly according to the law. The mere statement of the proposition seems to furnish its own



answer and to render an elaborate opinion unnecessary. This court cannot change the fact that the Secretary entertains this opinion, nor the fact that he intends to call on the district attorneys to test the case in the courts. It cannot command him not to make his opinion and intention known and if it could it would be useless for he has already made it known, and the petitioner itself is making the fact still more widely known by this proceeding. The merits of the real question, namely, whether flour subjected to the bleaching process may be sold without violating the pure food law, is one that will ultimately be determined by the courts. In the meantime the Secretary is not violating any law in having an opinion and in telling the public what it is.

The demurrer is overruled.

WENDELL P. STAFFORD,  
*Justice.*

The said Alsop Process Company stood upon its demurrer and prosecuted an appeal from the aforesaid judgment to the Court of Appeals for the District of Columbia. The case was then heard by said court on appeal and the judgment of the lower court was affirmed. The following opinion by Mr. Justice Robb was rendered by the appellate court:

UNITED STATES OF AMERICA EX RELATION ALSOP PROCESS COMPANY, <i>Appellant</i> , <i>vs.</i> JAMES WILSON, SECRETARY OF AGRICULTURE.	}	No. 2021.
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This is an appeal from the Supreme Court of the District overruling the demurrer of the relator to an answer of the defendant, appellee, here to a rule to show cause why a mandamus should not be issued against him.

In its petition the relator states that it is a corporation of the State of Missouri engaged in the manufacture of flour bleaching machinery, which is sold throughout the United States and elsewhere and is extensively used by millers for bleaching flour. The process for which this machinery is designated is known as the "Alsop Process" and is covered by patent which is owned by the relator. The bleaching of flour by this process is accomplished by the passage of pure air through a flaming discharge of electricity and the application of the resultant gaseous medium to the freshly milled flour as the latter passes through an agitator. The flour thus treated, the relator states, has no substance mixed and packed with it so as to reduce or lower or injuriously affect its quality or strength, is not deprived of any valuable substance, nor has it been mixed, colored, or treated in any manner whereby inferiority is concealed, and contains no deleterious ingredient or other element injurious to health. The relator further states that prior to November 18, 1908, the Secretary of Agriculture inserted, or caused to be inserted, in certain milling journals and other periodicals throughout the country a notice to the effect that a hearing would be held on the subject of bleached flour at the Department of Agriculture on November 18, 1908, at which time the relator says it was present by a duly authorized officer and by an attorney, and that the hearing was also attended by many millers from various parts of the country; that this hearing was continued five days, and testimony for and against said process was introduced; that the attorney for the relator conducted the case for the millers favoring the bleaching process; that the relator's manager gave extended testimony at this hearing; that the entire proceedings were transcribed by a stenographer and made accessible to the public generally. This hearing, the relator avers, was without color of authority of law. The petition further states that on the 10th of December, 1908, the said Secretary of Agriculture unlawfully, arbitrarily, and oppressively, and without color or right of law issued, the following bulletin:

## FOOD INSPECTION DECISION 100.

*Bleached Flour.*

"Flour bleached with nitrogen peroxide, as affected by the Food and Drugs Act of June 30, 1906, has been made the subject of a careful investigation extending over several months.

"A public hearing on this subject was held by the Secretary of Agriculture and the Board of Food and Drug Inspection, beginning November 18, 1908, and continuing five days. At this hearing those who favored the bleaching process and those who opposed it were given equal opportunities to be heard.

"It is my opinion, based upon all the testimony given at the hearing, upon the reports of those who have investigated the subject, upon the literature, and upon the unanimous opinion of the Board of Food and Drug Inspection, that flour bleached by nitrogen peroxide is an adulterated product under the Food and Drugs Act of June 30, 1906; that the character of the adulteration is such that no statement upon the label will bring bleached flour within the law; and that such flour cannot legally be made or sold in the District of Columbia or in the Territories; or be transported or sold in interstate commerce; or be transported or sold in foreign commerce except under that portion of Section 2 of the law which reads:

"\* \* \* Provided that no article shall be deemed misbranded or adulterated within the provisions of this act when intended for export to any foreign country and prepared or packed according to the specifications or directions of the foreign purchaser when no substance is used in the preparation or packing thereof in conflict with the laws of the foreign country to which said article is intended to be shipped; \* \* \*

"In view of the extent of the bleaching process and of the immense quantity of bleached flour now on hand or in process of manufacture, no prosecutions will be recommended by this Department for manufacture and sale thereof in the District of Columbia or the Territories or for transportation or sale in interstate or foreign commerce, for a period of six months from the date hereof.

JAMES WILSON,  
*Secretary of Agriculture.*

WASHINGTON, D. C., December 9, 1908."

The promulgation and circulation of this bulletin, the relator states, has worked irreparable harm and injury to it, and in effect deprived it of its property without due process of law "in that since the issuance and promulgation of said unlawful decision aforesaid by the respondent herein, and by reason thereof your petitioner has been unable to sell its patented process and apparatus aforesaid, the prospective purchasers of said patented process and apparatus aforesaid, refusing to buy and install the same for fear that they or their customers will, upon the recommendation of the Secretary of Agriculture, be prosecuted for manufacturing or selling an adulterated food product in violation of the provisions of said Food and Drugs Act, June 30, 1906." The petition closes with a prayer that the writ of mandamus issue to compel the Secretary of Agriculture to withhold recommendation of prosecutions against manufacturers of and dealers in flour bleached by said Alsop Process; to revoke, cancel and annul said decision of said Secretary, and not to deliver or circulate additional copies thereof, and that the Secretary of Agriculture be commanded to proceed relative to the subject of bleached flour in strict conformity with said Food and Drugs Act and the regulations of the Department promulgated thereunder.

A rule to show cause was issued. In the answer filed by the Secretary he states "that it does not appear by the said petition that the said relator has any right, title, or interest in the matters affected by the judgment and action of your respondent referred to in the said petition, and is not a party to nor legally interested in the proceedings in which said judgment and action of your



respondent have been made." He admits the relator owns the patent known as the "Alsop Process" for bleaching flour, but claims that its patented rights are wholly collateral to the right of said Secretary of Agriculture to decide whether flour bleached by the use of nitrogen peroxide is deleterious and adulterated within the meaning of said Food and Drugs Act; that the patenting of said process confers no right on relator and gives it no status to compel the respondent to change or revoke his decision that flour so bleached is adulterated. The answer denies that the effect of flour by the use of said process is as stated in the petition; on the contrary, the answer states "that the flour which is bleached is reduced and lowered in its quality and strength; that the said flour is so artificially colored as to conceal inferiority, and that it contains a poisonous and deleterious ingredient which has been added, and that the said flour is deleterious and injurious to health." The respondent in his answer further says "that the bleaching of the said flour is affected by nitrogen peroxide, and that the resultant product is deleterious and is adulterated within the meaning of the aforesaid Food and Drugs Act approved June 30, 1906"; that for many months prior to November 18, 1908, the respondent had made an exhaustive inquiry into the character, composition and purity of bleached flour and had caused the matter to be investigated exhaustively by the Bureau of Chemistry of his Department, and "that from all the evidence adduced it was conclusively established that flour bleached with nitrogen peroxide was adulterated within the meaning of said Food and Drugs Act;" that in the exercise of abundant caution, however, the Secretary decided to renew the investigation and to consider the matter more fully before finally deciding under the authority of said Act whether said bleached flour was adulterated; that accordingly he issued a notice for said public hearing; that this hearing was entirely advisory; and that the millers and manufacturers and others who attended did so voluntarily. The result of this hearing, the Secretary says, was to put him in possession of further and additional evidence relative to the subject; that this hearing was authorized both impliedly by the provisions in said Food and Drugs Act and expressly by the provisions of the Agricultural Appropriation Act of Congress of May 23, 1908; that after due consideration he decided that flour bleached by the use of nitrogen peroxide is adulterated within the meaning of said Food and Drugs Act and forbidden by the terms of said Act, and that he thereupon announced and published said decision of December 10, 1908; that this decision in no wise mentioned or in any way relates to the relator, and that, therefore, it has no status to seek any relief or redress in connection therewith. The Secretary in his answer denies the averments of the petition that his action was without right or color of law, denies the jurisdiction of the court to grant the writ, and states that he "passed no judgment upon the machinery of the relator, and has no jurisdiction over the same, nor concern therewith. The said relator is not an owner of bleached flour nor a manufacturer of the same. The judgment of the said respondent has to do only with the bleached flour, the product itself, and has no jurisdiction over or concern in one of the kinds of process by which the said product may be secured. And respondent submits that the claims of the said relator are wholly collateral, and that its petition fails to show any legal damage."

To this answer a demurrer was filed, which was overruled, and, relator choosing to stand upon its demurrer, final judgment was entered, and this appeal taken.

The first question to be disposed of is whether the interest of the relator in the subject matter involved is of such a nature as to entitle it to maintain this proceeding. The decision of

the Secretary of Agriculture, which is here sought to be challenged, is to the effect that flour bleached by nitrogen peroxide is an adulterated product under said Food and Drugs Act. Neither the relator nor its process is mentioned in this decision. The relator is neither the owner nor the manufacturer of bleached flour. Its sole excuse for attempting to stay the hand of the Secretary is that since the promulgation of this decision by the Secretary it has been unable to sell its patented process and apparatus owing to the fear of prospective purchasers that upon the recommendation of the Secretary they will be prosecuted for manufacturing or selling an adulterated food product.

While it is true that there is a distinction between cases where the extraordinary aid of mandamus is invoked merely for the purpose of enforcing or protecting a private right and cases where the purpose of the application is the enforcement of a purely public right, the people at large being the real party in interest (High on Extraordinary Remedies, Parg. 430; 26 CYC 404 and cases there cited), it has never been held, at least to our knowledge, that such an indirect and collateral interest as is here shown will sustain a petition for the writ.

The relator as a corporate entity has no interest in the enforcement of duties owing by the Secretary to the public. It seeks to arrest the operations of an Executive Department of the Government solely because the indirect effect of the promulgation of an opinion by the head of the Department has been to cause millers to cease purchasing relator's machinery. In all the cases relied upon by relator mandamus was granted to secure to the relator's rights which they were entitled personally to enjoy. Measured by this test, it is apparent that the relator has no such interest in the subject matter of this controversy as to entitle it to the writ. Being neither an owner nor a manufacturer of bleached flour, its legal rights were not involved or invaded by the action of the Secretary. It is a mere volunteer in this proceeding and as such is without standing.

There is some analogy between a suit in equity for the abatement of a public nuisance and the present case. Yet it is well settled that such a suit will not be sustained unless the complainant shows special, direct, and material damages; *Georgetown v. Alexandria Canal Co.*, 12 Peters, 91; *Irwin v. Dixon et al.*, 9 How. 9; *State of Penna. v. Wheeling Bridge Co., et al.*, 13 How. 518; *Miss. & Mo. R. R. Co. v. Ward*, 2 Black, 485. In the case last cited it was said: "A bill in equity to abate a public nuisance, filed by one who has sustained special damages, has succeeded to the former mode in England of an information in chancery, prosecuted on behalf of the Crown, to abate or enjoin the nuisance as a preventive remedy. The private party sues rather as a public prosecutor than on his own account; and unless he shows that he has sustained, and is still sustaining, individual damages, he cannot be heard."

The rule permitting private parties, whose rights are directly jeopardized, to maintain mandamus to compel a public duty is a salutary one, but it should not be enlarged to such an extent as to permit interference with the operations of the Government by those whose rights are only remotely and indirectly affected.

Having determined that the relator's interest in the subject matter involved is too remote to entitle it to institute this proceeding, it becomes unnecessary to consider any other question.

The order is, therefore, affirmed, with costs.

CHAS. H. ROBB,  
*Associate Justice.*

AFFIRMED.

JAMES WILSON,  
*Secretary of Agriculture.*

WASHINGTON, D. C., June 25, 1910.





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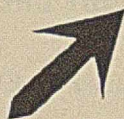
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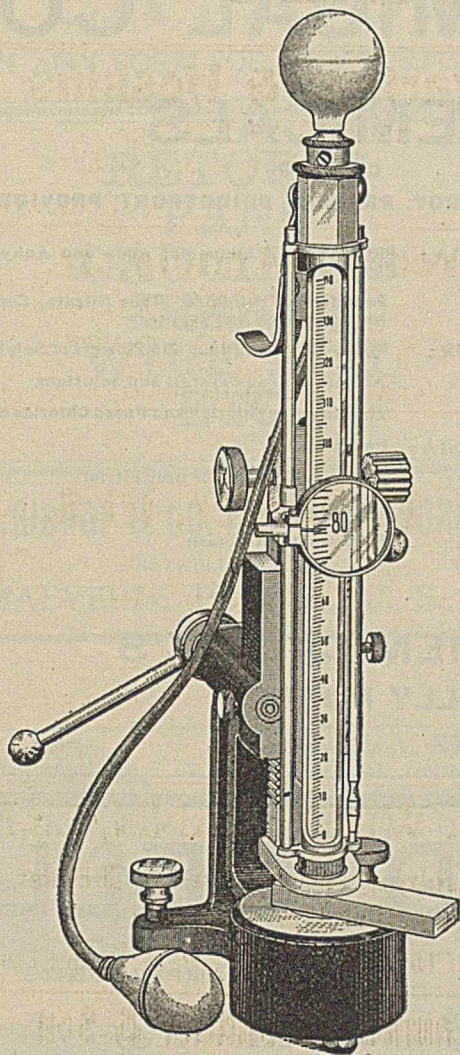
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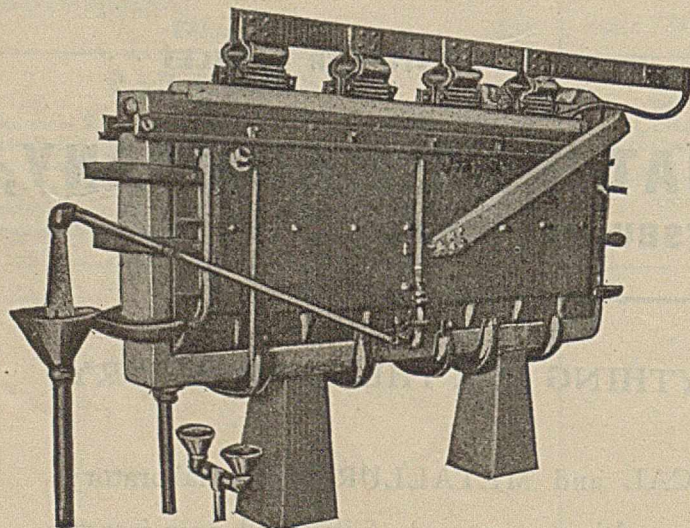
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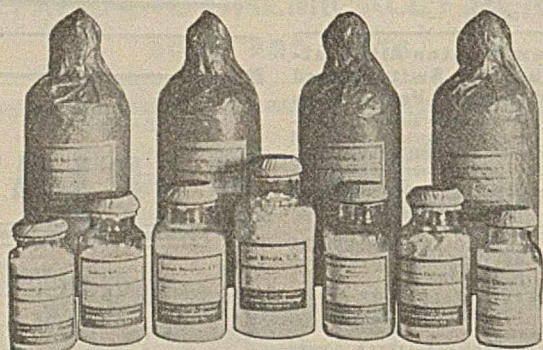
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