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EDITORIALS.

THE WORKS LABORATORY.

On a summer journey the writer arrived at a well built paper mill situated on a river in a town of some 12,000 people. The river had been diverted from the rapids through which it once ran and was dammed to furnish power with a head of 20 feet. Electrical power was brought from another power plant situated on a rapids 8 miles below. The mill was of considerable capacity and its sulphite towers prominent landmarks for some distance. Inquiry was made for the chemical laboratory. The first three men asked had never heard of it. A foreman said it was in the sulphite department. Further inquiry developed the fact that it was up three flights of stairs in the tower. Up five flights the cooker was found standing near the digesters. Yes he knew the chemist but he was out to lunch—would be back soon. The laboratory proved to be a small room the size of a closet on the stair landing, and had been passed unidentified. It was within a few feet of one of the digesters, was extremely warm and badly ventilated. Apparatus was all but lacking. Standard solutions were bought in Chicago and diluted for use. One man had scarce room to turn around in this laboratory, two could not possibly have worked in it. An ordinary business man's desk

could not have been placed in it if it were otherwise empty. The cooker explained that the chemist was not a "real chemist" but one who had "just picked it up." It seems he had learned the art from somebody who himself had learned it from a chemist. "We do not do much testing here" said the cooker. "I do some of it myself. Now there's a mill 100 miles from here where they test everything. They test the coal, and the sulphur and the rock and the water, besides the liquors. But we only test the liquors. We get along all right." The chemist did not return. It was time to go. Eight miles down the river the odor of sulphur dioxide was easily detected.

What is true of this paper mill is true of thousands of manufacturing plants throughout this country with this difference: some plants have no chemist at all and no testing apparatus. What is true of this country is true of all countries to a greater or lesser extent. Even in Germany there are many plants chemically uncontrolled, working empirically, by formula. It should not be difficult to demonstrate that in nearly all cases a chemical laboratory pays and pays well. This point has been learned these many years by all progressive superintendents and managers. The difficulty seems to lie in the question: Can a given plant be operated without a laboratory? The cost of a laboratory (although small) shows conspicuously on the books. The profits made possible by a laboratory are lost among other figures unless a special search is made for them. Under these circumstances men of little insight will continue to operate their plants without proper scientific control, until by the competition of better managed plants they are forced to adopt modern methods or go out of business.

In reassuring contrast to the plant with inadequate laboratory and equipment or with none at all stand such establishments as the General Chemical Company, the Solvay Process Company, the Illinois Steel Company, the Galena Signal Oil Company, the Chicago Packing Houses, Parke Davis and Company, the Du Pont de Nemours Powder Company and many others, with their excellent and adequate laboratory equipment and staff. These are all large works and the question may fairly be asked, is not the size of the plant the determining factor in deciding whether a chemical laboratory is a profitable investment? It would not be safe to conclude that the large plant or corporation could afford the expense of a laboratory

while the small one could not. Possibly if a census were now taken the data would show that the investment and expenses for laboratory in the majority of large manufacturing plants were greater per unit of capitalization than in the small ones. Again there are other ways of meeting the issue. If the expense of a chemical laboratory should appear too great, at least a superintendent with thorough chemical and technical training could be obtained. Again the commercial laboratory doing business on a yearly contract basis could be consulted. The intelligent observer of today is again and again forced to conclude that there is now no excuse for a manufacturing plant to operate without adequate scientific and technical control.

ORIGINAL PAPERS.

THE NEUTRALIZATION AND FILTRATION OF SMELTER SMOKE.

By W. C. EBAUGH.

Received August 1, 1910.

In the *Journal of the American Chemical Society* for July, 1907, there appeared a series of articles dealing with various phases of the smelter smoke problem, and presenting the matter from points of view that were quite different from one another.

In one of the articles special stress was laid upon the method of measuring the velocities of gases and determining the quantities of solids sent out of a stack during a given time. The results were startling to one who had had no previous experience in work of that kind, and probably awoke a new train of thought in the minds of all concerned with the problems of economic smoke handling and treatment.

A second article pointed out the damage that could be done by even small quantities of sulphur dioxide in the atmosphere, if present a large amount of the time, and cited experiences in many fields to bear out the statements made. The effects of arsenic escaping with the smoke and of copper bearing water issuing from a metallurgical plant also received attention.

The third article dealt with a comparison of the action of gaseous and solid emanations from smelters, and tried to show that although the damage done to crops by sulphur dioxide in certain climatic conditions might be enormous, nevertheless in the dry atmosphere of the intermountain west perhaps the importance of the gases in producing damage might have been overestimated, and the importance of the solid emanations might have been underestimated. The rôle of the solid emanations in injuring stock had already been acknowledged, but its importance in causing injury to vegetation had not been given the attention that it merited.

As the result of litigation long drawn out, in 1906 four smelters in the Salt Lake valley were enjoined from operating, the court decreeing that in order to

be considered harmless a smelter should treat ores that in their natural state contained not to exceed 10 per cent. of sulphur. To comply with such a condition was a commercial impossibility, and a petition that the smelters be allowed to mix ores in such a way that the average sulphur content should not exceed 10 per cent. was presented to the court, but the petition was denied. On appeal the circuit court affirmed the decision of the district court in all material points. Two smelters then went out of commission. A third entered into an agreement with the plaintiffs to filter all the solids from its blast-furnace gases and to send its roaster gases through some 1800 feet of Monier flue and a cooling and expansion chamber of specified construction, and a modification of the injunction was therefore permitted by the court in the case of this party to the suit. The fourth smelter went at the matter somewhat differently, and as a result of many months of experimenting evolved a system for neutralizing the gases from its devices other than blast furnaces in such a way as to permit their filtration. When their plans had been perfected the case was reopened before the district court, and upon the showing made permission was granted to try the new scheme. In other words the injunction was suspended, but not removed.

More or less full accounts of the modifications used have been published in the local press as well as in the technical periodicals, our own JOURNAL OF INDUSTRIAL AND ENGINEERING CHEMISTRY having an article upon the subject in its issue for October, 1909. A brief résumé of that article may not be out of place. It was shown that the gases coming from lead blast furnaces and from zinc plants had been filtered successfully for many years, but that roaster and other gases were not amenable to such treatment. The reasons for the latter statement are (1) that the temperature of gases coming from roasters is so high as to make the cooling of the gases before filtration impracticable or at least unprofitable; (2) that such gases contain more sulphur trioxide than do those from lead and zinc furnaces, and therefore corrode the bags very rapidly; and (3) that finely divided carbon and fume coming from roasters of the ordinary types seem to result in frequent cases of spontaneous combustion in the bag house, with consequent loss of efficiency and greatly increased cost of operation. Messrs. Sprague and Rhoades found that it was possible to prolong the life of a bag indefinitely if the gases led into it were first neutralized with zinc oxide. Experiments upon a larger scale were then conducted, and as a result they proposed to neutralize all the gases coming from a lead plant, roasters as well as blast furnaces, and filter the cooled mixture through bags. The plant was remodeled accordingly, and has now been in use for about two years. The success that has attended its operation under the new plan of operation

has given great satisfaction to all concerned. Looking at the plant one would think that it was out of commission, or at least running only a battery of ordinary steam boilers, for the only stacks that seem to be in use are those on the power house. Prior to the changes the vegetation in the neighborhood of the plant was always more or less injured by products thrown off by the smelter, but repeated examinations of these same districts during 1909 and 1910 have failed to disclose any damage that could be detected by the casual observer. The latter part of the summer of 1909 was unusually wet, and the spring and summer thus far this year have been unusually dry and hot, but the surrounding country has never looked better, according to impartial observers, than during these two seasons.

After the lead plant had been in operation for some months the court granted permission to start the copper plant under similar restrictions, but the company has not taken advantage of this decree. Failure to do so is to be attributed more to economic than to technical or metallurgical causes.

That the improved state of affairs is really due to the changed methods of operation rather than to climatic or other conditions is shown by the fact that other plants working on a similar scale to the one herein referred to, but without such efficient filtration for all gases, have not been surrounded by an unharmed area. Since sulphur dioxide and other flue gases are thrown off into the air in all cases, and only the solids are retained by filtration, it seems that there is some connection between the solids emitted and the damage inflicted upon vegetation, thus affording proof for the conclusions offered in one of the papers printed three years ago.

At best the results given in this paper can be taken only as a "report of progress." Theoretically and practically the escape of sulphur dioxide into the air constitutes an economic waste in the broad sense of the term, and were it possible to convert this gas into sulphuric acid and find a market where it could be utilized directly or indirectly at even a small margin of profit, a new and better method of disposing of this smelter waste might be introduced. At present, however, such a plan appears to be impracticable. Figures taken from the United States Census for 1900 and from various trade reports formed the basis for a calculation made several years ago, that showed that the smelters of Utah alone would be able to make more sulphuric acid than was consumed in the whole United States. If to this enormous output be added that obtainable from Montana, Arizona, California, Washington and other smelting states, the magnitude of the undertaking could be appreciated. The matter of freight rates from smelting districts to localities in which there is manufacturing on a large scale must also be taken into account. It would seem

that for the present at least filtration of smelter smoke offers a partial solution of the problem, but that a better means of avoiding damage may be evolved in the future. Most of us who are still in our youth can remember how the rivers near gas works were polluted by tar and other refuse, how certain industrial plants used to belch out great clouds of black smoke twenty-four hours of the day, and how slaughter-houses treated as waste much of the material that they now turn into profit. Is it too much to hope that changed conditions as exemplified in the gas works, power houses and abattoirs just mentioned may be tokens of what may occur to smelters in the not very distant future?

UNIVERSITY OF UTAH,
SALT LAKE CITY.

EFFLORESCENCE ON BRICK WORK.

By F. GREAVES-WALKER.

Received August 3, 1910.

Wherever brick are used for building purposes, brick walls will be noted which are covered or partially covered with a film of scum. This scum is generally white but may be yellow or green.

It appears at its worst on red brick walls, but may be seen more or less plainly on brick of any color.

When the walls are new it appears as white blotches at various places. Later it may come out all over the building, giving the brick work a faded or dirty appearance. In a few years a red brick building so affected will take on a faded pink appearance which will stay with it forever.

The scumming is always worst near the eaves and downspouts, and under the window-sills and water table. This is caused by leakage of rainwater between the roof and trough and from the downspouts, and through the catching of the water by the sills and water table which consequently soaks into the brick beneath.

The scum is simply a film of soluble salts carried from the interior of the brick to the surface by the water which they absorb, and left there after it evaporates.

This scum is generally composed of sulphates, although it may be composed of any other soluble salt. Chlorides are found in seacoast clays and alkalis in clays of the Western arid region.

In the clays of this country, the scum causing salts, in order of their abundance are:

- CaSO₄ calcium sulphate.
- MgSO₄ magnesium sulphate.
- K₂SO₄ potassium sulphate.
- Na₂SO₄ sodium sulphate.
- FeSO₄ ferrous sulphate.
- Al₂(SO₄)₃ aluminum sulphate.

Often all of these occur in a single clay, especially if it has been weathered and subjected to the attack of decomposing pyritiferous minerals.

All of the above salts are very soluble except calcium sulphate, which is soluble 1 : 400. But this is great enough to completely ruin face brick in which it occurs, as the surface is small in comparison with the weight. This particular salt, in fact, is probably responsible for half the ruined walls in this country.

The sulphates of soda, potash, and magnesia will often cause scum when present in the most minute quantities. As little as 0.01 per cent. of any one of these salts has been known to ruin face brick.

There are four ways in which salts are incorporated into the brick:

- (1) Soluble salts in the clays or other materials used.
- (2) Soluble salts in the water used for tempering the clay.
- (3) Soluble salts formed in kilns by oxidation of minerals in clay, or by reaction of sulphurous kiln gases on clay.
- (4) Soluble salts introduced into brick after burning, principally from mortar.

A great majority of clays contain soluble salts, some large quantities, others but a trace. Surface clays are more likely to have a high content than the older deposits. Trouble is encountered in all parts of the world from this source.

Probably 75 per cent. of the scumming comes from this cause.

Often materials are mixed with clays that carry soluble salts into them. Examples of this are the brick made around London, England, in which ashes are used to reduce shrinkage, and brick made along the Hudson in which coal slack is used to aid in burning. In both of these cases the soluble salts in coal add to, if they do not entirely produce, the scum.

In clays that vitrify scumming can be overcome by burning them to a vitrified body, in which case the salts form silicates and become harmless.

The water used for mixing and tempering mud brick often contains soluble salts. This, of course, produces the same effect as if the clays contained them. Many good clays are injured in this way.

The formation of soluble sulphates in the kiln is a very common trouble. It is often encountered with clays that contain practically no troublesome ingredients before being fired.

In many cases there is really no excuse for trouble at this stage. It is simply a case of ignorance on the part of the brickmaker or poorly designed kilns and stacks.

When a kiln is set and the heating up process started, the ware contains considerable moisture. If good judgment is used and the fires are kept at such a stage that only so much of the moisture is being driven off as the sluggish draft of a cool stack will carry off, trouble is not likely to occur; but if the moisture is driven off more rapidly than the stack can handle it, it is precipitated on the ware again, when it absorbs

sulphur from the gases and forms sulphuric acid. This acid attacks the carbonates and other less soluble salts and forms soluble sulphates.

There is no doubt that considerable trouble is caused by the mortar in which the brick are laid. Poorly mixed mortar is dissolved by the rain water and the salts carried into the brick, only to be brought to the surface later. It is therefore always important to take great care with the mortar.

Methods of Overcoming Scum.—Many methods are employed to overcome or prevent scum. It is possible to do this in nearly every case, but is often impossible on account of the low selling price of the ware. Where facing brick or other wares are made by the wet process or the ware is vitrified there is practically no excuse for trouble.

The preliminary treatment is the principal method of overcoming or preventing scum. This is done by treating the clay or treating the water used.

Treating the Clay.—Clays that contain soluble salts can be washed before using and the salts leached out. This may be done by treating piles of clay, after mining, with water and then allowing them to dry. This method is only possible where high-priced ware is made. Clays are often allowed to stand in piles for from six months to a year, in which case the rain and snow do the same thing.

In many clays the above methods will only cause the trouble. This would be the case where a clay did not contain soluble salts originally, but upon exposure to the weather insoluble salts were converted into soluble salts. This would be the case with pyritiferous clays.

Chemical treatment is very often resorted to. This is accomplished in clays made up by the wet process, by adding, to the water used, such chemicals as will precipitate the soluble salts and form harmless by-products, such as BaSO_4 . Barium salts are the favorites with which to accomplish this reaction, but others can be used. Barium chloride and carbonate are most commonly used. When these barium salts come in contact with soluble sulphates, barium sulphate is formed, which is practically insoluble in water.

In case soluble sodium compounds are present, the addition of the above-mentioned barium salts would form either Na_2CO_3 or NaCl , both of which are soluble. However, they are so soluble that they would be readily washed from the surface of the ware by the rain, as soon as they formed.

According to Gerlach, a clay containing 0.1 per cent. CaSO_4 , which is 0.4 gram per pound, would require 0.6 gram of BaCO_3 to perform the reaction. However, 6 or 7 grams should be added to every pound of clay, for safety. This would mean practically 100 pounds per 1000 brick. If BaCl_2 is used, but 26 pounds are required per 1000 brick. However when

BaCl₂ is used, close to the theoretical amount must be added, for if added in excess it will form a scum itself.

This method cannot, of course, be used in the dry press process, where no water, or at least very little, is added, as the barium or other salts must be used in solution to do the required work.

Treating the Water.—When it is necessary to use a water that contains injurious soluble salts, it is simply necessary to precipitate them before adding the water to the clay.

Many good clays are being ruined by bad water, when it needs but a few hours of a chemist's time to rectify the trouble.

Preventing Scum by Correct Drying.—Scumming is often caused by incorrect drying of the ware. If ware containing soluble salts is placed in a drier and allowed to sweat slowly, the chances are all in favor of scummed ware, whereas, if the water is carried away from the surface of the ware as fast as it appears this will not often be the case.

It is a remarkable physical fact, not yet fully explained, that where a clay contains soluble salts, they are not deposited on the surface at all, or at least to a less degree, where the drying is steady and rapid, than where it is slow and discontinuous. It seems that rapid drying causes the water to come through the pore channels of the clay as through a filter and a sort of osmosis is set up, by which the saline matters are retained inside the mass of the clay, while the water escapes as vapor from the outside. But where the drying is slow the salts get through this barrier and arrive on the surface plentifully.

It is therefore only necessary that brickmakers, in order to avoid trouble from this source, build good driers and instal in them hygrometers by which their operation can be controlled.

Many other methods are used to prevent this trouble. Some of them, however, have but temporary effects.

One of these is to paint the surface of the scummed ware with oil, which for the time being covers up the scum. This is really practicing a fraud, for while the ware does not show scum when put into the building it shows up as soon as the oil evaporates.

Another method is to paint one surface of the unburned brick with a heavy oil or tar. This prevents evaporation from that surface, the salts being carried to the other surfaces. The oil or tar is burned off in the kiln, leaving a clean face. This, too, is only temporary unless the brick is vitrified, as otherwise the salts will be drawn to the clean surface after a few soakings.

SALT LAKE CITY, UTAH.

THE ELECTROLYTIC DETERMINATION OF ZINC IN ORES.

By GEO. KEMMERER.

Received July 30, 1910.

The investigation was undertaken to ascertain if

the zinc content of an ore could not be accurately and quickly determined electrolytically.

The original plan was to separate the zinc from the other constituents of the ore, as zinc sulphide, by a method similar to the "Modified Waring;" this sulphide was then to be dissolved in a little acid, the hydrogen sulphide boiled out, made alkaline with sodium hydroxide and electrolyzed.

Numerous experiments with an ore of known zinc content, using an electrolyte containing from 7–10 grams of sodium hydroxide, gave high non-concordant results.

About this time E. B. Spear, E. E. Wells and B. Dyer¹ showed that several methods gave high results and Spear² stated that the high results were due to zinc oxide or hydroxide deposited with the zinc and suggests that a strong alkaline electrolyte might prevent this, but proved that it is theoretically impossible to deposit all the zinc from such an electrolyte. Harrison Hale³ determined the zinc content of a pure blend, using an electrolyte containing 15 grams of sodium hydroxide. His results were not high and the average of a large number of determinations was very close to the gravimetric results.

Experimental.—The electrolytic cell used consisted of a cylindrical nickel gauge cathode which fitted loosely into a 150 cc. Jena beaker, and a revolving propeller-shaped anode.

The cathode surface was about 160 sq. cm. while the anode was rotated about 600 times per minute.

Weighed pieces of C. P. zinc were dissolved with nitric acid in a 180 cc. flask, 2 cc. of sulphuric acid added and evaporated to dense fumes of sulphur trioxide. After cooling, the contents of the flask were rinsed into a 150 cc. beaker, diluted to 100 cc. and stick sodium or potassium hydroxide added. This heated the solution nearly to boiling and it was electrolyzed at once.

When the deposition was complete the cathode was removed without opening the switch and plunged into a large beaker of water. It was then dried with absolute alcohol, and ether, which had been distilled over sodium, heated to 70–100°, cooled and weighed at once.

The gauze electrodes cool quickly and it is important to weigh at once for on standing the deposits gain in weight.

	Weight of deposit.		Weight after standing in desiccator.	Gain.
1.....	0.2501	Over night.....	0.2505	0.0004
2.....	0.2500	Two hours.....	0.2504	0.0004
3.....	0.2517	One hour.....	0.2521	0.0004

After the electrolysis the solution remaining was always tested for zinc by acidifying with dilute sulphuric acid, cooling to 60° and adding 50 cc. of 10 per cent. solution of potassium ferrocyanide. This test

¹ *J. Am. Chem. Soc.*, **32**, 530.

² *Ibid.*, **32**, 533.

³ Thesis, U. of P., 1908.

as shown by E. B. Spear, E. E. Wells and B. Dyer¹ was found to produce a turbidity with 0.5 mg. of zinc in 200 cc. of solution.

After weighing the deposits, the zinc was dissolved from the nickel cathode in 2-3 per cent. sulphuric acid. By this treatment the cathode loss was kept below 0.5 mg., but it gradually darkened due to a trace of lead in the zinc. After ten to twenty determinations the black deposit was removed by pouring a little nitric acid over the electrode.

With an electrolyte containing from 7-10 grams of sodium hydroxide the results ran as much as 0.9 per cent. high and were not concordant.

When the alkali was increased to 20-25 grams the following results were obtained:

	A.	V.	Time.	Zinc taken.	Zinc found.	Error.
1.....	6	4.4	15	0.1762	0.1759	-0.0003
2.....	6	4.2	15	0.2099	0.2019	0.0000
3.....	5	3.8	15	0.3180	0.3184	+0.0004
4.....	5.2	3.8	15	0.3740	0.3743	+0.0003
5.....	5	4.0	15	0.2412	0.2410	-0.0002
6.....	5	4.0	10	0.2502	0.2501	-0.0001
7.....	5	4.2	14	0.2502	0.2500	-0.0002
8.....	5	4.2	15	0.2502	0.2496	-0.0006
9.....	6	4.4	20	0.2149	0.2147	-0.0002
10.....	6	4.4	20	0.2149	0.2144	-0.0005

The deposits were all a beautiful bluish white and in every case the remaining solution showed no zinc present by the ferrocyanide test.

These results seem to justify the application of the method to ore analysis, so a simple carbonate ore was chosen, the zinc content of which had been determined several times before this work was started. It contained 42.35 per cent. zinc, 9 per cent. iron, no lead. It was treated by the modified Waring method given in Low's "Technical Methods of Ore Analysis" as follows:

"After decomposing the weighed sample by acids alone, or aided by fusion, as the case may require, all the zinc is to be brought into solution as sulphate. If nitric acid was used in the decomposition, all traces of it must be expelled by evaporation with hydrochloric and sulphuric acids successively, or by two evaporations with sulphuric acid, finally to abundant evolution of SO₃ fumes. Dissolve the mass in 25-40 cc. of water and add sufficient sulphuric acid to bring the free acid in the solution up to 10 or 15 per cent. Introduce a piece of heavy sheet aluminum and boil 10 minutes, or to complete reduction. Filter and wash through a filter containing a piece of aluminum into a beaker containing a stirring-rod or strip of the same metal, cool, add a drop of methyl orange, and neutralize carefully with sodium bicarbonate to a light straw color. Add, dropwise, dilute formic acid (20 per cent. strength) until the pink color is just restored, then 5 drops more. (Dilute hydrochloric acid, 1 part strong acid to 5 parts water, may be substituted for formic acid when ammonium thiocyanate is to be introduced.) Dilute to about 100 cc. for each 0.1 gram of

zinc possibly present, add, if much iron is present, 2-4 grams ammonium thiocyanate, remove the strip of aluminum, heat nearly to boiling, and saturate with hydrogen sulphide. Allow the pure white zinc sulphide to subside for a few minutes, then filter and wash with hot water."

The zinc sulphide was carefully washed with hot water and dissolved on the filter in a little hot dilute hydrochloric acid. To this solution in the 400 cc. beaker in which the sulphide was originally precipitated 2 cc. of sulphuric acid were added and the whole allowed to evaporate until most of the sulphuric acid was driven off. The zinc sulphate was then rinsed into a 150 cc. beaker diluted to 100 cc., 25 grams of sodium hydroxide added and electrolyzed.

The nickel gauze cathode previously described was used and a current of 6 amperes at a pressure of 4.4 v. was allowed to flow for 15 minutes, while the anode revolved about 600 times per minute.

The results were concordant, but always about 0.3 per cent. high.

Several of these deposits were dissolved, the iron determined colorimetrically and found to be equal to 0.0014-0.0016 gram of iron. When using a 1/2 gram sample of ore this would equal 0.3 per cent.

The sodium hydrate was carefully tested and did not contain more than a trace of iron, so that this large amount must have been carried down with the zinc sulphide.

After numerous experiments it was determined that if 4-5 drops of concentrated hydrochloric acid in place of 5 drops of 1-6, were added to the neutral solution before precipitating the zinc sulphide, that in the presence of ammonium sulphocyanide all the zinc was precipitated as sulphide free from iron.

With this modification the ore containing 42.35 per cent. of zinc, gave the following electrolytic results:

Weight of ore.	A.	V.	Time, min.	Zinc found.	%.
0.5000	5	3	15	0.2116	42.32
0.5007	6	4.2	15	0.2114	42.22
0.4995	6	4.2	15	0.2107	42.18

Average, 42.24

The next ore was a standard sent out by the New Jersey Zinc Company. The results by various chemists ran as follows: 35.5, 35.57, 35.5, 35.2, 35.5, 35.67, 35.87, 35.7; average, 35.56.

ELECTROLYTIC METHOD.

Wt. ore.	A.	V.	Time, min.	Zinc found.	%.
0.5002	6	4.2	15	0.1792	35.83
0.5003	6	4.2	15	0.1783	35.64
0.5002	6	4.2	15	0.1794	35.86
0.5009	6	4.2	15	0.1790	35.73
0.5007	6	4.2	15	0.1790	35.77

Average, 35.77

Each of these deposits was tested for iron and not more than a trace was found.

¹ J. Am. Chem. Soc., 32, 530-3;

All the results obtained for this ore included those corrected for iron are as follows:

35.73, 35.74, 35.74, 35.57, 35.67, 35.83, 35.64, 35.86, 35.77; average, 35.73.

CONCLUSIONS.

This work has proven that zinc can be accurately determined electrolytically; that the results are not high if the electrolyte contains from 20-25 grams of sodium hydroxide and with a current of $N. D._{100} = 3.1$ A, all the zinc can be deposited; that this method which is shorter than the usual gravimetric procedure gives results fully as accurate.

NEW MEXICO SCHOOL OF MINES,
SOCORRO, N. M.

THE DETERMINATION OF FREE ACID IN HYDROGEN PEROXIDE SOLUTIONS.

By LINWOOD A. BROWN.
Received July 25, 1910.

Sometime ago in the examination of drug samples, I had occasion to analyze practically all of the different makes of hydrogen peroxide to be found on the Kentucky market, and one of the determinations to be made in the analysis of this preparation is the determination of the free acid present.

The U. S. Pharmacopœia, which is recognized by both Federal and State Food and Drugs Acts, as the standard for drugs, defines this preparation as being:

"A slightly acid, aqueous solution of hydrogen dioxide, which should contain, when freshly prepared, about 3 per cent. by weight of absolute hydrogen dioxide, corresponding to about 10 volumes of available oxygen."

Among other tests for purity for this preparation, it states that if to 25 cc. of the solution, 5 cc. of tenth normal potassium hydroxide V. S. be added, and the mixture be evaporated to about 10 cc. and 3 drops of phenolphthalein T. S. be added, not less than 2.5 cc. of tenth normal sulphuric acid should be required to destroy the red color of the solution after continued boiling (limit of free acids).

This does not appear at first glance, to be a difficult determination, but after a few trials, we found that it did not yield correct results, and upon examination it developed surprising possibilities for trouble.

Acids occur in hydrogen peroxide solutions for two reasons: first, as an aid in preservation; second, in order to remove the barium from which the hydrogen peroxide is prepared. Sulphuric, hydrochloric, phosphoric, oxalic, benzoic acids, etc., are the acids likely to be present.

Acetanilid is used in a large number of hydrogen peroxide preparations on the market, as an additional preservative.

The official method for determining the free acid was soon found to be unreliable, giving high results, so in order to locate the source of trouble, the author

prepared a 3 per cent. hydrogen peroxide solution, using perhydrol, a neutral 30 per cent. H_2O_2 solution. This perhydrol was tested for sulphuric, phosphoric, hydrochloric, oxalic acids, etc., and was found to be entirely free from such acids.

The 3 per cent. aqueous solution of hydrogen peroxide thus prepared was used as a basis for preparing the following solutions containing known amounts of sulphuric, hydrochloric, phosphoric, and benzoic acids.

Solution "A."—This solution was made by adding sufficient sulphuric acid to the 3 per cent. solution of H_2O_2 so that 25 cc. of the solution thus prepared were exactly equivalent to 2.5 cc. of $N/10$ H_2SO_4 , which is the limit of acid allowed in the U. S. P. article.

Solution "B."—This was prepared in the same way as solution "A", using hydrochloric acid. 25 cc. of this solution were equivalent to 2.5 cc. $N/10$ HCl.

Solution "C."—Prepared as in solution "A," 25 cc. of which were equivalent to 2.5 cc. $N/10$ H_3PO_4 .

Solution "D."—25 cc. of solution were equivalent to 2.5 cc. $N/10$ benzoic acid. This solution was prepared as a type of the organic acids likely to be met with in some preparations of hydrogen peroxide found on the market.

Solution "E."—This solution was prepared by dissolving 0.040 gram acetanilid, m. p. 114° C. (cor.), in a sufficient amount of the hydrogen peroxide solution to make 100 cc. corresponding to $3/16$ grain acetanilid per fl. oz., the strength ordinarily used in preserving peroxide solutions.

Solution "E" was prepared to determine what effect the presence of acetanilid would have on the methods used. The solutions thus prepared were analyzed by the following methods:

First Method.—The U. S. P. method, which was carried out as follows: 25 cc. of the solutions representing 2.5 cc. $N/10$ acid were treated with 5 cc. $N/10$ KOH and evaporated in porcelain to about 10 cc., and three drops of phenolphthalein added and the excess of alkali titrated back, using tenth normal sulphuric acid, and boiling.

Second Method.—A modification of the U. S. P. method was used as follows: 25 cc. of the solution equivalent to 2.5 cc. tenth normal acid were treated with 5 cc. tenth normal potassium hydroxide and evaporated on a water bath in a large platinum dish, kept covered with a watch glass, to avoid loss by the rapid disengagement of gas until effervescence ceased and all H_2O_2 was decomposed. Phenolphthalein was then added and the excess of alkali titrated back, using tenth normal sulphuric acid and boiling off CO_2 after each addition of acid.

Third Method.—25 cc. of the solution, equivalent to 2.5 cc. tenth normal acid, were titrated in the cold, with tenth normal potassium hydroxide, using phenolphthalein as indicator.

By the first method used (U. S. P. method) the result of a large number of determinations ran uniformly high, but erratic, due possibly to one or more of the following factors:

- (a) Absorbed CO_2 from the air.
- (b) Oxidation of the ethyl alcohol in the phenolphthalein indicator by the undecomposed H_2O_2 .
- (c) Oxidation of phenolphthalein to phthalic acid or anhydride.
- (d) Acetic acid liberated from acetanilid by the saponification.
- (e) Any undecomposed H_2O_2 acting as an acid.

In the second method we have taken advantage of the well-known property that some metals, such as gold, platinum, etc., have of decomposing hydrogen peroxide solutions catalytically, by substituting a platinum dish for the porcelain with the result that *all* the hydrogen peroxide is decomposed. Then by adding the indicator, and titrating the excess of alkali, boiling off any absorbed CO_2 after each addition of acid, we eliminate all errors with the exception of saponifying action of the alkali on the acetanilid, and get results that are theoretical, varying in no case more than 0.05 cc. tenth normal acid, from the theoretical amount added, which is well within the limit of error.

By the direct titration method (third method) we find the results just a trifle higher than by the modified U. S. P. method, due possibly to a small amount of absorbed CO_2 in the solution, but more likely to the acidic nature of the H_2O_2 itself.

The results by this method were sufficiently accurate for all practical purposes, with the advantage of greater ease and simplicity. The results are not affected by the presence of acetanilid as in the U. S. P. and the modified U. S. P. methods.

To determine the amount of undecomposed H_2O_2 existing after the evaporation in porcelain, as in the U. S. P. method, we took several of the samples and determined the strength of the H_2O_2 by diluting the liquid remaining after the evaporation to 250 cc. and determining the amount of H_2O_2 by titration with $N/10$ potassium permanganate.

If calculated back to the original sample of 25 cc. used, the amount of absolute H_2O_2 found varied between 1.97 per cent. and 2.21 per cent., showing that very little decomposition takes place when the method is carried out in porcelain.

The H_2O_2 thus reaches a concentration, if the liquid be evaporated to 10 cc. each time, of 4.4–5.5 per cent. absolute H_2O_2 , which is quite sufficient to exert a considerable disturbing influence on the method.

In the modified U. S. P. method no undecomposed H_2O_2 could be detected after evaporating or heating in the platinum dish.

Conclusion.—The U. S. P. method as given in the U. S. P. for free acid does not yield trustworthy re-

sults, and it is necessary to decompose all the H_2O_2 by heating in platinum.

In both the U. S. P. and the modified U. S. P. methods, acetanilid seriously interferes, acting the part of free acid.

Direct titration in the cold appears to be the most satisfactory method as it gives results that are sufficiently accurate for all practical purposes, is not affected by the presence of acetanilid, and has the advantage of greater ease, rapidity, and simplicity.

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SEPARATION AND DETERMINATION OF COCAIN AND STRYCHNIN, AND ATROPIN AND STRYCHNIN WHEN THEY OCCUR TOGETHER.

By H. C. FULLER.

Received July 25, 1910.

The alkaloids of the coca leaf and of *nux vomica* and of belladonna and *nux vomica* occur together as the active ingredients of certain classes of medicinal products, and it becomes necessary at times to determine how much of each is actually present. The groups follow each other so closely in their solubilities that no separation based on this property is possible, and one is obliged to employ other means.

It is a well-known fact that the coca bases are readily hydrolyzed by acids and by alcoholic potash and a method for the determination of cocain in a mixture of its allied alkaloids based on this phenomenon has been recommended by Garsed.¹ This chemist carries out the hydrolysis in alcoholic potash by heating for half an hour over the steam bath using a reflux condenser. It has been determined that the *nux vomica* alkaloids under these conditions are not changed and hence a separation can be effected depending on this difference in behavior.

A liquid drug product containing both cocain and strychnin is first evaporated cautiously to drive off any alcohol if present and then quickly cooled to prevent the decomposition of the cocain. If the material is a pill, tablet or powder, it is ground up and digested with alcohol containing a little ammonia, the solvent filtered and the treatment repeated twice more, the combined alcoholic solution being evaporated cautiously and the residue taken up with dilute sulphuric acid and filtered if necessary. To the solution obtained by either of these methods, ammonia water is added in excess and the mixture shaken out three or four times with Prolius' mixture, reserving each portion of the solvent and finally discarding the alkaline aqueous liquid. The solvent solution is then filtered, the filter paper washed with more of the solvent and the filtrate evaporated over the steam bath using a fan, care being taken to prevent the residue from

¹ *Pharm. Jour.*, 1903, p. 784.

being heated excessively. The resulting product is now dissolved in about 15 cc. of dilute sulphuric acid and the acid solution shaken out five times with chloroform, if caffeine is known to be present; if not twice is sufficient. All the chloroform shakings are finally combined, washed once with water and then the chloroform discarded and the wash water added to the acid liquid. Ammonia is then added in excess and the solution shaken out three times with chloroform, the solvent solutions are combined, washed once with water, and the chloroform run through cotton inserted in the stem of the separator, into a tared dish; two small portions of chloroform are then run through the cotton into the dish and the whole evaporated cautiously using a fan, and the residue dried at 80 for a short time and then cooled in a desiccator and weighed. Cocain being volatile the drying must be at a low temperature. As the final portions of the chloroform evaporate, the dish should be rotated and removed from the steam to avoid spattering.

The weight thus obtained will give the combined cocain and strychnin, or if the drug extract was present the combined coca bases with strychnin and brucin. The mixture is then dissolved in $N/5$ alcoholic potash, using 15 cc. for each decigram of cocain supposed to be present, the solution poured into a digestion flask arranged with an air-tight stopper, the dish washed out with 10 cc. alcohol and finally with 10-15 cc. water. The flask is then stoppered and placed over the steam bath for one hour. At the end of that time the flask is opened, the contents poured into an evaporating dish, washing out the flask with alcohol and water, and evaporated until all the alcohol has been driven off. The residue is then transferred to a separator, using water and chloroform, and the alkaline liquid shaken out three times with chloroform, collecting the solvent in another separator. The combined chloroform extracts are then shaken three times with 10 cc. portions of dilute sulphuric acid, the acid being reserved and the chloroform finally discarded. Ammonia is now added until in excess, and on cooling if much strychnin is present it will come out as a fine crystalline precipitate. The solution is shaken out three times with chloroform, the chloroform extract collected in another separator, washed with water, run through cotton into a tared dish and the solvent evaporated. The dried residue will be found to consist of strychnin or strychnin and brucin.

This same general plan may be followed in separating atropin from the nux vomica bases. The tropeins are readily hydrolyzed and the strychnine and brucin can then be shaken out and finally purified according to the above method.

VOLUMETRIC DETERMINATION OF POTASSIUM BY THE COBALTI-NITRITE METHOD.

By O. M. SHEDD.

Received June 30, 1910.

The writer, in making some determinations on the fertilizer samples sent this year by the referee on potash of the Association of Official Agricultural Chemists, obtained such interesting results in using the cobalti-nitrite method as modified by Drushel¹ for the determination of potassium that it was decided to publish a summary of them, since they throw much light upon the best conditions for applying this method.

The accuracy of this method has been considerably discussed of late and it has been considered of sufficient importance to have been recommended to the above-named association for coöperative work by the referee on potash and also by the referee on soils.

Drushel in his original article had published some results obtained with this method on fertilizers and recommended it for this purpose, and his method, with a slight modification by Bowser,² is the one proposed for study this year. On the other hand, the writer³ has proposed it in connection with the J. L. Smith method for the determination of total potassium in soils and has obtained very good results in this manner. Later on, further work⁴ by the writer and others was presented to the association at its annual meeting last year with the result that this method has been taken up by the referee on soils. Incidentally, it might be of interest to mention that the writer has obtained very good results in using this method on the soils sent this year by the referee on soils for coöperative work.

In the method, as proposed for soils, a change was made in the outline as given by Drushel which, while it may seem of small importance, is very essential in the application of the method. Drushel had recommended that the solution (apparently not restricting the volume) at the proper point was to be slightly acidified with acetic acid and a liberal excess of the sodium cobalti-nitrite reagent added and evaporated to a pasty condition on the water bath. On the other hand, the writer in his article had recommended that the volume should be concentrated to about 5-10 cc. before adding the nitrite reagent. That this is the best condition under which to work will be shown later, and it is not necessary to discuss it here, except in a general way. The nitrite reagent being unstable at room temperature and especially so on heating, the method that completely precipitates the potassium when the reagent is added with the minimum amount of heating, of course, is the one to be preferred. Again, when the dilution is too large, the nitrite reagent is all decomposed on heating before the

¹ *Chem. News*, 97, 124 (1908).

² *This Journal*, 1, 791.

³ *Ibid.*, 1, 302.

⁴ *A. O. A. C. Proceedings*, 1909.

potassium salt is precipitated and consequently low results will be obtained. Finally, the potassium salt itself is acted on to some extent by the acetic acid and water present, and while it may precipitate at ordinary temperature, on heating the precipitate will disappear and the solution become pink. Therefore, if at the end of the evaporation there is not an excess of the nitrite reagent present, low results will be obtained.

To illustrate the above points, a few qualitative tests might be given. The potassium salt was made by precipitating C. P. KCl solution cold with an excess of the reagent and washed with cold water by decantation. A portion suspended in water and heated in the boiling water bath dissolved slowly, giving a clear pink solution after about two hours' heating. This solution evaporated on the water bath in a dish left a blue residue. On taking up with cold water acidified with acetic acid, a notable quantity of the yellow potassium salt remained, perhaps about half of the original quantity, and the solution was pink. On repeating the evaporation and re-solution a second and a third time, only traces of the yellow salt remained.

Another portion was suspended in water and divided equally between two test tubes. To one, a few drops of acetic acid were added and the two heated side by side in the water bath. The potassium salt disappeared much quicker in the one containing the acid than in the pure water. This experiment was repeated with the same result.

Another portion was allowed to stand at room temperature in contact with 2 or 3 cc. of water, shaking occasionally. After several days, the water became distinctly pink, showing decomposition of the yellow compound.

Another portion was let stand at room temperature in about 50 cc. of water, with occasional shaking. At the end of a month, all but a trace of the yellow compound had disappeared. On evaporating part of the clear liquid at room temperature over sulphuric acid, there remained a yellow residue, apparently consisting mostly of the potassium double salt, but mixed with pink, needle-shaped crystals, having the appearance of a cobalt salt.

A little of the reagent diluted with 10 or 15 times its volume of water and heated on the water bath soon lost its brown color and became pink, showing complete decomposition of the reagent. An equal portion treated in the same manner, except that several drops of acetic acid were added, underwent the change more quickly. The greater the dilution, the quicker this change takes place.

Upon evaporating the reagent to dryness after diluting with water, as in the above experiment, the mass is distinctly purple in color, and upon adding water there remains an insoluble greenish residue containing cobalt while the solution is pink. The char-

acteristic color of the reagent has entirely disappeared which indicates complete decomposition.

To prove that this greenish substance will have the same effect as the potassium salt, if it should be present, the following tests were made: two 10 cc. portions of the reagent were diluted to 15 times their volume and then evaporated until the contents of one was pasty on cooling. The other was further evaporated until the contents were just dry and firm on cooling. They were then taken up with water, filtered, and treated with permanganate and oxalic acid in the same manner as the potassium salt in the method. The following results were obtained:

Contents on cooling.	N/10 KMnO ₄ required in titration.
Pasty.....	1.74 cc.
Dry.....	3.08 cc.

The above results show that if these same amounts of this substance were present in two determinations worked like the above on 0.10 gram aliquots of a potassium salt, the results would be too high to the extent of 1.50 per cent. and 2.64 per cent. K₂O in the respective determinations.

These tests show that too great a dilution should be avoided, because it decomposes the reagent, and also continued heating in the evaporation should be avoided, because in the final stage of evaporation, the last traces of acetic acid may be expelled and as soon as this takes place, the greenish substance is formed more rapidly than it would be if there still remained some free acid.

To show that the above holds true, two 5 cc. portions of the reagent were diluted to 15 times their volume and evaporated until the volume of one was about 5 cc. and liquid on cooling. The other was evaporated until pasty on cooling. When water was added to both, there was a small amount of the greenish residue in the first, but considerably more in the other.

This source of error could be avoided in the work, if a few drops of acetic acid were added to the solution before filtering, as this readily dissolves this substance and would leave the potassium salt practically pure.

The above experiments show that both the cobalt-nitrite reagent and the yellow potassium compound are decomposed by heating with water and that the change is hastened in the presence of acetic acid.

The samples sent by the referee on potash were three in number, namely, commercial muriate, kainit and a complete mixed fertilizer. The directions called for a study of the cobalt-nitrite method in comparison with the official platinum method. The aliquots recommended for the volumetric method were 0.10 gram for the muriate, 0.5 gram for the kainit and 1 gram for the mixed fertilizer. These aliquots were used in all of the determinations. The directions also called for the volume of the solution to be about

25 cc., when 1 cc. glacial acetic acid and 10 cc. recently prepared nitrite reagent were to be added and the evaporation made.

The referee's directions were followed in the determinations below, the aliquots for the two methods being taken from the same solution. In the volumetric method, the solutions were heated until the contents were just dry and firm on cooling.

OFFICIAL METHOD.			
	Grams K ₂ O found.	Percentage K ₂ O found.	
Muriate.....	0.26025	52.05	
	0.25980	51.96	
Average.....	0.26005	52.01	
Kainit.....	0.06380	12.76	
	0.06435	12.87	
Average.....	0.06410	12.82	
Fertilizer.....	0.04660	4.66	
	0.04660	4.66	
COBALTI-NITRITE METHOD.			
Muriate.....	0.04928	49.28	
Kainit.....	0.05930	11.86	
Fertilizer.....	not determined	...	

It was apparent that the referee's directions had a tendency to give low results, probably due to the volume being too large when the nitrite reagent was added. To see if an increase of the amount of reagent added at that volume would recover the potassium present, the following determinations were made on the muriate.

	Grams K ₂ O found.	Percentage K ₂ O found.
10 cc. nitrite added.....	0.04928	49.28
15 cc. nitrite added.....	0.05022	50.22
20 cc. nitrite added.....	0.05125	51.25

The following determinations were made by evaporating the solution to about 5 cc. before adding the acetic acid and nitrite reagent. The reagent was added in various amounts and dishes heated until residues were of the consistency as noted on cooling.

MURIATE.			
Nitrite.	Contents on cooling.	Grams K ₂ O found.	Percentage K ₂ O found.
10 cc.....	dry	0.05159	51.59
10 cc.....	dry	0.05151	51.51
15 cc.....	dry	0.05223	52.23
15 cc.....	dry	0.05155	51.55
15 cc.....	dry	0.05169	51.69
15 cc.....	very dry	0.05142	51.42
15 cc.....	pasty	0.05142	51.42
15 cc.....	thick paste	0.05159	51.59
15 cc.....	very thin paste	0.05134	51.34
25 cc.....	dry	0.05095	50.95
25 cc.....	pasty	0.05108	51.08
KAINIT.			
15 cc.....	dry	0.06280	12.56
15 cc.....	dry	0.06360	12.72
15 cc.....	very dry	0.06230	12.46
15 cc.....	very thin paste	0.06315	12.63
25 cc.....	dry	0.06355	12.71
25 cc.....	pasty	0.06345	12.69

C. P. K₂SO₄—THEORY, 54.05 PER CENT. K₂O.

Nitrite.	Contents on cooling.	Grams K ₂ O found.	Percentage K ₂ O found.
15 cc.....	dry	0.05382	53.82
15 cc.....	dry	0.05347	53.47
15 cc.....	dry	0.05458	54.58
15 cc.....	dry	0.05453	54.53
15 cc.....	pasty	0.05390	53.90
15 cc.....	pasty	0.05399	53.99
15 cc.....	pasty	0.05399	53.99

MIXED FERTILIZER.			
Nitrite.	Contents on cooling.	Grams K ₂ O found	Percentage K ₂ O found.
25 cc.....	pasty	0.04680	4.68
25 cc.....	pasty	0.04630	4.63

In following the directions of the referee in the above work, the dishes were heated until the residues were syrupy while hot and just dry and firm on cooling. According to Drushel, this is the best plan to follow, for it thus avoids errors in the continued heating of the residues which will vitiate the results. This was done regardless of the time consumed which was noticed varied in similar determinations and very often with different salts.

After some correspondence with the referee, who stated that the method as outlined by him generally gave satisfactory results in his hands, the writer made some further determinations on the samples and also on C. P. salts. In this work, the referee's directions were followed and also the modification as stated above, whereby the solution was concentrated to about 5 cc. before adding the nitrite reagent. The time consumed in the evaporation after adding the nitrite reagent and the consistency of the contents on cooling after the evaporation was made were also noted. The evaporations were made in casseroles of 750 cc. capacity, except in the case of the kainit, when 400 cc. casseroles were used.

REFEREE'S DIRECTIONS.

MURIATE.			
Time.	Contents on cooling.	Grams K ₂ O found.	Percentage K ₂ O found.
45 min.....	thin syrup	0.05187	51.87
45 min.....	thin syrup	0.05226	52.26
45 min.....	thin syrup	0.05179	51.79
45 min.....	thin syrup	0.05145	51.45
			Average, 51.84
40 min.....	thin paste	0.05145	51.45
40 min.....	thin paste	0.05136	51.36
45 min.....	thin paste	0.05104	51.04
45 min.....	thin paste	0.05159	51.59
			Average, 51.36
1 hr.....	dry	0.05119	51.19
1 hr.....	dry	0.05110	51.10
			Average, 51.15
KAINIT.			
50 min.....	pasty	0.06300	12.60
1 hr.....	pasty	0.06345	12.69
1 hr.....	pasty	0.06285	12.57
			Average, 12.62
1 1/2 hr.....	dry	0.06275	12.55

C. P. KCl—THEORY, 63.17 PER CENT. K₂O.

Time.	Contents	Grams K ₂ O found.	Percentage K ₂ O found.
20 min.....	liquid about 15 cc. vol.	0.05093	50.93
45 min.....	thin syrup	0.06108	61.08
45 min.....	thin syrup	0.06358	63.58
45 min.....	thin syrup	0.06283	62.83
1 1/8 hrs.....	pasty	0.06342	63.42
1 1/8 hrs.....	pasty	0.06325	63.25

The determinations below were made by having the volume about 5 cc. when the nitrite reagent was added and 15 cc. of this were used in each case.

C. P. KCl.			
Time.	Contents on cooling.	Grams K_2O found.	Percentage K_2O found.
20 min.	liquid	0.06383	63.83
30 min.	syrup	0.06366	63.66
50 min.	pasty	0.06383	63.83
C. P. K_2SO_4 .			
20 min.	liquid	0.05401	54.01
30 min.	thin paste	0.05435	54.35
50 min.	thick paste	0.05467	54.67

No reason can be easily assigned why the higher results were obtained on the samples when worked by the referee's directions than those given before, unless it is that the time given to the evaporation is a factor to be considered. In this connection, it might be of interest to note that in the low results obtained before, while following the referee's directions, and in some of the higher results obtained at the same time when the volume was reduced to about 5 cc. before adding the reagent, the same stock of reagent was used. Evidently, these variations were not due to the reagent, but rather to the different volumes of the solution on adding the same. In support of this, reference might be made to the more uniform results which have been obtained where this change has been made in the referee's directions.

From the above work, one must recognize that the evaporation is a point that is to be watched in the working of the method and, in fact, an important point which needs further attention before definite conclusions can be drawn.

To determine what other factors influence the results, first the effect of precipitating in larger volumes with the reagent; second, using old nitrite reagent and finally using more or less acetic acid was tried. Also, the plan was tried of adding the nitrite in two portions at different times in the evaporation in order to more completely precipitate the potassium by maintaining the excess of fresh reagent in the solution.

Effect of Volume.—10 cc. of new nitrite reagent were added to 0.10 gram of the sample in the volume indicated, which included the volume of the reagent added. The evaporations were then made in the usual manner. In the case of KCl, the residue after evaporation consisted mostly of the greenish substance already mentioned and only a small amount of the potassium salt.

C. P. KCl.		
Volume including reagent.	Grams K_2O found.	Percentage K_2O found.
200 cc.	0.00377	3.77
A. O. A. C. MURIATE.		
100 cc.	0.03533	35.33

To illustrate further what effect the reducing of the volume of the solution before adding the reagent has on the results obtained, those determinations made on the A. O. A. C. muriate according to the referee's directions and also where the volume was further

concentrated to 5 cc. before adding the reagent are given for comparison.

Volume of solution on adding reagent.	Grams K_2O found.	Percentage K_2O found.
25 cc.	0.04928	49.28
25 cc.	0.05022	50.22
25 cc.	0.05125	51.25
25 cc.	0.05187	51.87
25 cc.	0.05226	52.26
25 cc.	0.05179	51.79
25 cc.	0.05145	51.45
25 cc.	0.05145	51.45
25 cc.	0.05136	51.36
25 cc.	0.05104	51.04
25 cc.	0.05159	51.59
25 cc.	0.05119	51.19
25 cc.	0.05110	51.10
Average,		51.22
5 cc.	0.05159	51.59
5 cc.	0.05151	51.51
5 cc.	0.05223	52.23
5 cc.	0.05155	51.55
5 cc.	0.05169	51.69
5 cc.	0.05142	51.42
5 cc.	0.05142	51.42
5 cc.	0.05159	51.59
5 cc.	0.05134	51.34
5 cc.	0.05095	50.95
5 cc.	0.05108	51.08
Average,		51.49

The plan was also tried of adding the nitrite reagent to the dry residue of the salt after evaporation, but on account of the potassium salt being precipitated so finely divided, that it was impossible to filter it, the determination was discarded. This was repeated with the same result.

Effect of Old Reagent.—The reagent used here had been made for about eight months and kept in the dark. The referee's directions were followed in the work, except in the last two determinations on the muriate. In these, the volume of the solution was about 5 cc. on adding the reagent.

A. O. A. C. MURIATE.			
Reagent used.	Contents on cooling.	Grams K_2O found.	Percentage K_2O found.
10 cc.	dry	0.03531	35.31
10 cc.	dry	0.03655	36.55
20 cc.	dry	0.05050	50.50
20 cc.	dry	0.05076	50.76
KAINIT.			
20 cc.	dry	0.06085	12.17
20 cc.	dry	0.06095	12.19

In connection with the above work, the following experiments were tried to see if it was possible to determine colorimetrically how much the old reagent had decomposed on standing. Two cc. each of the new and of the old reagents were diluted to 50 cc. in Nessler tubes, and, by comparing the two, it was found that, judging from the color, two-fifths of the old reagent had decomposed.

When the color test was made on the old reagent in the above, the solution had the characteristic brown color of the reagent. After the test was made, the tubes were left standing in the light for about six

hours, and during that time about one hour in the sunlight. At the end of the time, the solution in the tube containing the old reagent had changed to pink, showing complete decomposition, while the new reagent solution was still faint brown, but much lighter in color than before, showing a decided decomposition had taken place. After standing about $1\frac{1}{2}$ hours longer in the sunlight, this solution also became pink in color.

Evidently, the reagent must be kept in the dark to prevent decomposition.

Effect of More or Less Acetic Acid.—The plan followed was to evaporate the solution to about 5 cc. volume, 15 cc. new reagent added and the amounts of acetic acid stated. The contents of the dishes were evaporated until the volume was about 5 cc., when they were taken from the bath. This was done in order to eliminate any error caused by further evaporation.

A. O. A. C. MURIATE.			
Acetic acid.	Contents on cooling.	Grams K_2O found.	Percentage K_2O found.
None.....	thin paste	0.05142	51.42
1 cc.....	thin paste	0.05134	51.34
5 cc.....	pasty	0.05246	52.46

A. O. A. C. KAINIT.			
		Grams K_2O found.	Percentage K_2O found.
None.....	thin paste	0.06330	12.66
1 cc.....	thin paste	0.06315	12.63
5 cc.....	pasty	0.06450	12.90

Addition of the Reagent in Two Portions.—The determinations below were made according to the referee's directions, that is, by adding 10 cc. new reagent to the volume at 25 cc. The solution was then evaporated to about 5 cc. volume and 5 cc. more of reagent were added. The evaporation was continued until the contents were dry on cooling.

Muriate.....	50.09 per cent. K_2O
Kainit.....	12.41 per cent. K_2O

In the following experiments, an attempt was made to estimate the potassium by adding the salts which compose the nitrite reagent in separate solutions at the time the evaporation was to be made. In other words, it simply consisted of preparing the reagent in the presence of the potassium salt to be estimated. If found to work, it was thought that this would improve the method, because it would be possible in this way to regulate the volume of the solution, which is important, and at the same time avoid the use of the prepared reagent which decomposes slowly on standing.

For the work 220 grams $NaNO_2$ were dissolved in H_2O and made to 500 cc. and designated the sodium nitrite solution. 113 grams of cobalt acetate were dissolved in H_2O and 100 cc. 50 per cent. acetic acid added (one-half of the amount prescribed) and the solution made to 500 cc. and designated as the acetate solution.

A. O. A. C. Muriate.—The solution containing 0.10 gram salt was evaporated to dryness and taken up

with 5 cc. of the sodium nitrite solution and then 5 cc. of the acetate solution were added, but no acetic acid, and the whole evaporated until the contents were pasty on cooling. Result, 50.59 per cent. K_2O .

Another trial was made in same manner, except the acetate solution was added first and then the sodium nitrite. Result, 51.53 per cent. K_2O .

C. P. K_2SO_4 .—The same method as followed in the second determination above was then tried on a solution containing 0.10 gram of the salt, and the evaporation continued until the contents were dry on cooling. Result, 52.43 per cent. K_2O .

Some further determinations were made on 0.10 gram aliquots of C. P. K_2SO_4 and C. P. KCl by adding the acetate to the dry residue after evaporation and increasing the amount of acetic acid present to that prescribed in the regular method. In this work, 1.5 cc. of glacial acetic acid and 7.5 cc. each of cobalt acetate and sodium nitrite solutions were added and the evaporations continued until the contents were pasty on cooling. The results obtained were:

C. P. K_2SO_4 —	55.10 per cent. and 54.84 per cent. K_2O .
C. P. KCl —	63.39 per cent. and 63.52 per cent. K_2O .

The plan of adding the acetate solution first seems to be the most promising one, and further work along this line may prove that this is a safe plan to follow, although as yet there has not been sufficient work done to draw definite conclusions. It is hardly necessary to add here that for the above work, it is better to have chemicals free from potassium, or a blank is all the more essential for work of this kind.

The blank used in all the other work had remained constant and never varied from 0.20–0.23 cc. $N/10$ $KMnO_4$ solution but the blank here contained too much potassium from the chemicals used, it not being removed as it is in the ordinary nitrite reagent. As no purer chemicals were at hand, this work was discontinued, but it merits further investigation.

According to Bowser,¹ the cobalti-nitrite method for qualitative tests for potassium is much more satisfactory when an equal volume of strong alcohol is added to the solution to be tested. In this manner, he claims that very small amounts of potassium are precipitated in much less time than from an aqueous solution.

From his work, the writer was led into a series of experiments to see if the above could be used for quantitative purposes. If such would prove to be the case, then the evaporation in the method would be eliminated, which would be a great improvement in the work.

C. P. K_2SO_4 .—The plan followed at first was to evaporate the solution containing 0.10 gram of the salt to dryness. To the residue was added 5 cc. of the acetate solution and then 5 cc. of the sodium

¹ *Jour. Am. Chem. Soc.*, 32, 78; *Chem. News*, 101, 100.

nitrite solution already described. 10 cc. of alcohol were then added and allowed to stand at ordinary temperature for the time indicated.

Time.	Percentage K ₂ O found.
15 minutes.....	52.20
60 minutes.....	51.99

The next plan tried was to evaporate the solution to about 5 cc. volume and then add 10 cc. of the ordinary nitrite reagent and 15 cc. alcohol. In the first two determinations below, 0.1 cc. glacial acetic acid was added and in the third, no acetic acid. The solution stood at room temperature for the time indicated.

Time.	Percentage K ₂ O found.
Filtered immediately.....	50.78
5 minutes.....	50.80
15 minutes.....	45.85

In the next experiments, the addition of the alcohol was omitted and the work pursued along a different line. It had been found in working on the A. O. A. C. samples that the addition of more acetic acid than the directions called for had a tendency to give higher results in the regular method.

The plan was then followed of evaporating the solution containing 0.10 gram aliquots to about 5 cc. volume, cooling, and adding 10 cc. of nitrite reagent and the amounts of glacial acetic acid indicated. The solution was then allowed to stand for 30 minutes at room temperature.

A. O. A. C. MURIATE.

Acetic acid added.	Percentage K ₂ O found.
1 cc.....	48.98
5 cc.....	50.38
15 cc.....	Could not filter

Evidently here, as in the regular method, an increase of acetic acid seems to give a higher result, but whether this increase is due to more potassium precipitated or to other causes is not easy to determine.

It was thought that perhaps the increase of acetates present in the solution influenced the precipitation of the potassium by the nitrite reagent and to prove this, if possible, the following experiments were made on C. P. KCl. Two 0.10 gram aliquots of the salt were taken and to one, after evaporating to dryness, was added 10 cc. of a saturated solution of sodium acetate and 10 cc. nitrite reagent. To the other aliquot, after evaporating to about 5 cc. volume, was added 10 cc. reagent and then 10 grams of sodium acetate. Both were allowed to stand 1 hour at room temperature and gave respectively 55.02 per cent. K₂O and 48.34 per cent. K₂O.

To determine if it was possible to completely precipitate the potassium at room temperature by this method, if the volume of the solution was reduced, the following experiment was tried on C. P. KCl.

Two 0.1 gram aliquots were evaporated to about 5 cc. volume, 1 cc. glacial acetic acid and 10 cc. nitrite reagent added and the whole put in a desiccator over H₂SO₄ and the air exhausted as much as possible by means of the suction pump. It was then allowed to stand about 24 hours and at the end of that time, the solution had evaporated and the residue was nearly pasty. Result, 65.74 per cent. and 67.52 per cent. K₂O.

In every determination that has been made without heating the solution, it has been impossible to wash the potassium sodium cobalti-nitrite with water until the filtrate was colorless. While the washings did not contain any of the precipitate in suspension, they had a yellow color which seemed to indicate that either the salt itself was dissolving, or, which is more probably the case, it was contaminated with some substance which made it difficult to wash clean. While it is easy to understand how low results are obtained while working in this procedure, the high results can only be explained on the supposition above, that the potassium salt contains some other compound that has been formed which is difficult to wash out and reacts with permanganate.

From his experience with the Drushel modification of the cobalti-nitrite method, the writer believes that it is very accurate if properly handled, but an inexperienced worker not knowing its weak points may not have this opinion with his first use of it. Briefly stated, the outline of the method which has given the best and most uniform results in this work is as follows:

The solution containing the potassium salt, after the preliminary work has been done to get it at this stage, is evaporated in a 500 cc. casserole to a small volume of about 5 cc., slightly acidified with acetic acid and 15 cc. fresh nitrite reagent added. The larger amount of nitrite reagent makes possible a better filtration and a good excess of reagent after the evaporation is made. The solution is evaporated on the water bath for about 45-60 minutes, or until the contents become a thick syrup while hot and pasty on cooling. Continued heating is to be avoided, and this is important to obtain good results. After the filtration is made and the casserole washed, the Gooch crucible and contents can be put in the same casserole and treated with permanganate, as prescribed in the method.

In conclusion, the writer desires to express his sincere thanks to Dr. A. M. Peter, of this Station, for the valuable advice he has rendered during the progress of the work.

A COMPARISON OF BOLLING'S, HARDING'S AND DREHSCHMIDT'S METHODS FOR DETERMINING SULPHUR IN ILLUMINATING GAS.

By E. P. HARDING AND CARL TAYLOR.

Received June 8, 1910.

Randolph Bolling, chief chemist for the Nova Scotia Steel & Coal Co., Sidney Mines, N. S., in suggesting the advisability of ascertaining the total sulphur in suction producer gas, used a wet oxidation method, in which he assumed that all of the sulphur in the sulphur compounds is oxidized to sulphur dioxide. Bolling states that Bunte's method¹ could be used in analyzing illuminating gas which has been more or less purified but that, with unpurified producer gas which contains sulphur in other than simple combinations like hydrogen sulphide and carbon bisulphide, the method would fail to give all of the sulphur. He also states that Drehschmidt's method² gives accurate results by completely oxidizing all of the sulphur, but objects to the method on account of the complexity of the apparatus used and the difficulty of keeping the gas burning continuously in the Drehschmidt burner.

The principle of Bolling's method is the oxidation of the sulphur in the sulphur compounds present to sulphuric acid by means of a saturated solution of bromine in hydrochloric acid, and a gravimetric determination of the sulphur by precipitating and weighing it as barium sulphate. The apparatus consisted of a five liter aspirator bottle, a thermometer, barometer and two 500 cc. capacity absorption bottles.

The method in detail is as follows: The aspirator is filled with water and connected with the absorption bottles which are connected to the producer gas main by means of a short length of pipe packed with loose asbestos fiber. The water is allowed to flow slowly from the aspirator, thereby drawing gas through the bromine solutions. The temperature and pressure are taken during the process. The contents of the absorption bottles are transferred to a 750 cc. beaker and the bromine completely removed from the bottles by washing with distilled water. Two grams of sodium carbonate are then added to the solution which is heated to boiling to drive off the excess of acid and water. The sulphur is precipitated as barium sulphate by adding to the diluted hot solution a 10 per cent. solution of barium chloride, and boiling vigorously for thirty minutes. The barium sulphate is then dried, ignited and weighed and the sulphur determined by the following formula,

$$S = 200.0 \ p \ 0.013748 \times \frac{750.8}{283} \times \frac{273 \ t}{B-f} = \\ p \ 729.47 \times \frac{273+t}{B-f},$$

in which t is temperature of the gas, f the tension of

water vapor at the temperature t , B the barometric pressure, p the weight of the barium sulphate and S the amount of sulphur in 100 cu. m. of the gas.

The writers of this paper believed that purified coal gas contains more complex sulphur compounds than suction producer gas made from hard coal in a continuous automatic feed producer. They believed Bunte's method to be more efficient for determining the total sulphur in such producer gas than in purified coal gas. And they doubted the efficiency of Bolling's method as applied to purified coal gas.

This doubt led to determinations of the total sulphur in Minneapolis illuminating gas by both Bolling's method and Harding's modification of Drehschmidt's method and to a comparison of the efficiency of the two methods.

Harding's method and the apparatus used were those described by him in the *Journal of the American Chemical Society*; 28, 537, with the exception that only 0.5 cc. of bromine was used instead of four. Drehschmidt's original method is given in *Chem.-Ztg.*, 2, 1382 (1887). The principle of the method as modified is the combustion of the gas in a specially constructed glass burner within the body of a large retort, in the presence of bromine vapors, the aspiration of the products of combustion with some bromine vapors through a 5 per cent. solution of potassium carbonate, and the precipitation of sulphur as barium sulphate.

The apparatus with the exception of the burner is of the simplest construction and can be set up at once in any working laboratory. This burner eliminates one of Bolling's objections to the original Drehschmidt method.

The Bolling process was slightly modified so that it was possible to run under the same conditions as in the Harding method. Four cylinders of 150 cc. capacity were used, in each of which was placed 120 cc. of bromine water and 15 cc. of concentrated hydrochloric acid saturated with bromine. This gives the gas a longer column of solution to bubble through. The gas was aspirated through by means of a suction pump, after first passing through a wet meter and governor. Gas was drawn through till the first three solutions were nearly decolorized, leaving the fourth only slightly affected. This required about three-fourths of a cubic foot of gas.

In the Bolling method as described only five liters of gas were used. As the Minneapolis illuminating gas is low in sulphur content, and as it was found that in using this amount, a milligram difference in the weight of barium sulphate obtained made a 2 per cent. error it was believed that better results could be obtained by using larger amounts of gas and more absorbent.

In each method the gas was passed through the same wet meter which had been used for several

¹ Bunte, *Journal für Gasbelsuchung*, 31, 895 (1888).

² *Chem.-Ztg.*, 2, 1362 (1887).

years for measuring the illuminating gas for analysis, and through which gas had been passed several days to insure complete saturation of the water.

The amounts of gas used were reduced to standard conditions of temperature and pressure by the following formula:

$$V = V \frac{b - c}{760 (1 \times 0.003635 T)}$$

The results obtained were as follows:

Date.	Temp.	Press., mm.	Obs. vol.
Jan. 20, 1909.....	20°	748	3/4 cu. ft.
Jan. 30.....	22	745	3/4 cu. ft.
Feb. 9.....	20	741	3/4 cu. ft.
Feb. 18.....	21	743	1/2 cu. ft.
Feb. 27.....	21	744	3/4 cu. ft.
Mar. 2.....	23	744	3/4 cu. ft.

Grains sulphur per 100 cu. ft.

Harding.	Bolling.	Ratio.
9.0360	5.2884	1 : 1.709
10.2302	5.6843	1 : 1.801
7.9555	5.2824	1 : 1.506
8.9620	5.4467	1 : 1.647
8.3041	5.5886	1 : 1.485
9.3067	5.7635	1 : 1.615

Two tests were made, using the original Bolling method to observe if the oxidation was complete in the modified process with the following result:

Date.	Temp.	Press., mm.	Obs. vol.	Ratio.
Feb. 10, 1909.....	24°	745	3/4 cu. ft.	1 : 1.371
Feb. 10.....	24	745	1/6 cu. ft.	
Feb. 11.....	22	743	3/4 cu. ft.	1 : 1.264
Feb. 11.....	22	743	1/6 cu. ft.	

Grams sulphur per 100 cu. ft.		Ratio.
Harding.....	10.5978 }	1 : 1.371
Bolling.....	7.7329 }	
Harding.....	11.0443 }	1 : 1.264
Bolling.....	8.7352 }	

These results as compared to the above are within the limits of experimental error showing complete oxidation.

The sulphur content of the sodium carbonate and bromine was not determined but, as much more of each was used in the Bolling method than in the Harding method, if sulphur was present as impurities it would be in favor of the Bolling process.

The same wet meter was used in each process and the same amount of gas used in each determination so that the error of absorption of any sulphur compounds was eliminated.

The solution from the Bolling process gave considerable trouble in concentrating and precipitating the barium sulphate. It was difficult to remove the last trace of bromine and the large amount of bromine present produced bumping difficult to control. It was necessary to evaporate nearly to dryness and dilute the residue very much and it was often necessary to reconcentrate and redilute in order to precipitate the barium sulphate. Carrying the evaporation too far decomposed some of the organic bromide compounds, liberating bromine.

During the process the bromine vapors attacked the rubber connections and tubing, and bromine products were deposited to such an extent in the suction pump as to impair its efficiency.

From these observations it appears that Bolling's process is insufficient for determining the total sulphur in a gas of such composition as Minneapolis illuminating gas.

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THE NITROGEN AND HUMUS PROBLEM IN DRY-LAND FARMING.

By ROBERT STEWART.

Received July 22, 1910.

The effect of cultivation and the growth of crops upon the nitrogen and humus content of soils has been studied by various investigators, both in America and Europe. In general the results of the various investigators indicate that cropping and cultivation is very destructive to the organic matter and the nitrogen of the surface soil.

Berthelot,¹ in 1886, reported that the carbon and nitrogen content of calcareous clayey soil, originally very deficient, was gradually increasing owing to the action of diatoms.

Deherain² found that the soils from plots which had not been manured has lost over 50 per cent. of their carbon. The light soil of Grignon lost nitrogen and the amount lost greatly exceeded the amount removed by the crops. The loss of carbon was due to the oxidation of the organic matter, while the greater loss of nitrogen was due to drainage.

Snyder³ has found that in the humid section in grain-cultivated soils there is a rapid decline of the vegetable and animal matter, and a loss of the element nitrogen. According to Snyder the nitrogen was lost not only by the removal of the nitrogen in the crop but that drainage and denitrification played a very important part in the loss of nitrogen. Snyder also found that summer fallowing⁴ was more destructive to the nitrogen and humus content of the soil than was the continuous cropping to wheat. When summer fallowing was practiced, six times as much nitrogen was lost by other processes as was removed by the plant as food.

Ladd⁵ found that long-continued cropping of land with wheat by the fallow process was very destructive to the nitrogen content of the soil.

Alway,⁶ in 1909, reported that fields in Nebraska which had been long under cultivation showed a great loss of nitrogen, humus and unhumified organic

¹ *Compt. rend.*, 102, 951 (1886).

² *Ibid.*, 109, 781 (1889).

³ *Minn. Exp. Sta. Bulls.*, 30, 41, 53, 65, 70 and 94.

⁴ *Bull.* 70, *Minnesota Exp. Sta.*, p. 260.

⁵ *Bull.* 24, *North Dakota Exp. Sta.*

⁶ *Bull.* 111, *Nebraska Agr. Exp. Sta.*, p. 10.

matter. He says: "The greatest losses of organic matter, nitrogen and humus have been caused either by the washing or by the blowing away of the surface soil."

Alway and Vail,¹ in 1909, reported the accumulation of nitrogen in a prairie soil due to the deposition in rifts of organic matter by the wind.

Alway and Trumbull, in April, 1910, reported on the loss of nitrogen in cropped land when compared with adjacent virgin land. They found that the loss of nitrogen, humus and organic matter in twenty-five years was about one-third of the amounts originally present in the prairie soil. In certain cases they found that the content of nitrogen, humus and organic matter in the long-cultivated soil was slightly higher than in the virgin soil, which they attribute to the difficulty of obtaining samples of virgin soil which correctly represented the original composition of the cultivated field. The observed loss of nitrogen, humus and organic matter in the cultivated soil is due to two causes: oxidation of the organic matter and erosion by the wind.

At the same time Bradley² reported a study made of the virgin and cropped soils of Eastern Oregon. He found that while the nitrogen content of the soils which had been cropped for about twenty-five years was about the same as that of the adjacent virgin land the carbon content had decreased to a marked extent.

Headden,³ in February, 1910, reported the accumulation of nitrogen in the form of nitrates in some Colorado soils. This accumulation of nitrogen he believed to be due to the fixation of free nitrogen of the atmosphere by microorganisms.

EXPERIMENTAL PART.

In view of the known action of continuous cropping and summer fallowing upon the nitrogen and humus of humid soils, a knowledge of the effect of dry-farming upon the nitrogen and humus of dry-land soils became of exceedingly great importance. With this idea in view the author, in the summer of 1907, had a survey made of the dry-farming area in Cache Valley, the oldest dry-farming district in the State of Utah. Some of the farms of this district have been under cultivation for forty-five years and yield as good crops apparently as they ever did. The important principle of dry-farming is summer fallowing for the purpose of conserving the moisture of the soil. According to all the teachings of the humid agricultural districts as noted above, the practice of this principle, while it conserves the moisture of the soil, is also ideal for the destruction of the organic matter of the soil with the resulting loss of nitrogen.

The survey was started with the hope of learning something regarding the effect of crop production and

summer fallowing upon the humus, nitrogen and nitric nitrogen of the soil. The results obtained were so opposite to the teachings of the humid agriculturists and so significant that the author felt that they should be confirmed by further work before publication. The publication of the recent articles along this same line by other investigators has led the author to believe that these results would be of interest to others working along similar lines. More extended studies of the soils of the dry-farming sections of the State are being made at present, but the results now available are reported.

The soils studied are derived from the near-by mountain ranges, which are composed largely of limestone and quartzite. Cache Valley was at one time Cache Bay of old Lake Bonneville. The erosion of the near-by mountain ranges loaded the mountain streams with finely divided particles of limestone and quartzite, and when the running waters of the streams met the quiet waters of Cache Bay the streams deposited their load. It is of this material that the soils of the dry-farming area of Cache Valley is composed.

The plan of the experiment called for a careful examination of the individual farms of the section and careful sampling in several places of the cropped land and also of the adjacent virgin soil. The soil was sent to the chemical laboratory and the nitric nitrogen determined immediately by the phenoldisulphuric acid method. The moisture was also determined in order to convert results for nitric nitrogen to the dry basis. The samples were then air-dried and preserved for determination of total nitrogen and humus. The total nitrogen was later determined by the Kjeldahl method and the humus determined by the method of the Official Agricultural Chemists.

While the data obtained are insufficient from which to draw sweeping conclusions, there are certain facts which are brought out by the study. The cropping of the dry land to alfalfa has a tendency to cause a decrease of the humus content of the soil and in a majority of cases the nitrogen content also. The cropping of land to wheat, either continuously or by the summer fallow method, has a tendency either to not effect or else slightly to increase the nitrogen and humus content of the surface foot of the soil when compared with that of the adjacent virgin soil.

In Table 1 will be found the results obtained by taking an average of all the determinations made on the wheat, virgin and alfalfa land studies.

TABLE 1.—SUMMARY OF RESULTS.

Results for Total Nitrogen and Humus Reported as Per Cent. of Dry Soil.

Crop.	Depth of sample.	No. of analysis.	Total nitrogen.	Nitric nitrogen.	Humus.
Wheat.....	0-12	27	0.2055	0.80	2.67
Virgin.....	0-12	22	0.1984	1.04	2.45
Alfalfa.....	0-12	11	0.2009	1.74	2.27

Results for Nitric Nitrogen Reported as Parts Per Million of Dry Soil.

Wheat.....	12-24	24	0.1466	0.76	2.45
Virgin.....	12-24	19	0.1823	1.38	1.99
Alfalfa.....	12-24	11	0.1604	0.60	1.73

¹ THIS JOURNAL, 1, 74 (1909).

² *Ibid.*, 2, 139.

³ *Bull.* 155, *Colo. Agric. Exp. Sta.*, Feb., 1910.

The nitrogen content of the first foot of the virgin soil is lower than that of either the alfalfa or wheat land. The humus content of the wheat land is slightly higher, while that of the alfalfa land is lower. The nitric nitrogen content varies from 0.80 part per million in case of the wheat land to 1.74 parts in the alfalfa land.

In the second foot the nitrogen of the virgin soil is higher than either the alfalfa or wheat land. The humus of the wheat land is higher than that of the virgin while that of the alfalfa is lower. All three of the constituents decrease with the depth of the samples.

In studying the results given for an explanation of the phenomenon noted there are several explanations which suggest themselves.

In the first place the assumption is made that the composition of the virgin soil now correctly represents the composition of the cultivated soil before the beginning of cultivation. The impossibility of obtaining a sample of virgin soil which correctly approximates the original composition of the soil is obvious. The uniformity of the results obtained on so many samples over such a large district would seem, however, to indicate that this would not explain all of the phenomenon noted.

In the second place a part of the increase in nitrogen in the first foot may be accounted for by causes similar to those which cause the "rise of the alkali" in arid regions. This, however, is hardly probable and if it has any influence at all it would be very slight.

Again it is possible that azotobacter or other microorganisms which fix the free nitrogen of the atmosphere are in relatively great abundance in our arid soil and fix the free nitrogen of the atmosphere to a relatively great extent. This explanation seems to be the favorite explanation of all observed phenomenon which does not otherwise find a ready explanation from other causes. If such is the case, however, we have not evidence of it and it is somewhat ungenerous to throw the burden of proof upon the shoulders of the bacteriologist. This is a line, however, along which more work should be done with arid soils.

The last and more probable explanation which suggests itself is the following: the method of successful dry-farming for conservation of the limited moisture supply and the demands of the plants for water cause their roots to penetrate to a far greater depth than similar plants in a humid climate. The feeding range of the plant for plant food is thus increased. The plant probably gets a greater supply of its nitrogen from below the first foot than a similar plant would in a humid climate. The wheat also is harvested by a header and practically all of the straw containing nitrogen derived from below the first foot is all added to the surface foot. This greatly increases the nitrogen and humus content at the expense of the lower foot

section. This explanation appears to be probable. It is important to observe in this connection that the nitric nitrogen is somewhat low in all cases, indicating that all of the increase is due to nitrogen in the organic form.

The author wishes to acknowledge his indebtedness to Mr. F. D. Farrell, who made the survey and who made the nitric nitrogen determinations; to Mr. W. L. Walker, who made the nitrogen determinations; and to Mr. F. S. Harris, who made the humus determinations.

CONCLUSIONS.

1. The cropping of dry-farming land in the district studied to wheat either by the summer fallowing method or by continuous cropping does not decrease the nitrogen or humus of the surface foot of the soil.
2. The second foot of grain-cropped land contains less nitrogen and humus than does the second foot of the adjacent virgin soil.
3. The cropping of dry-farm land to alfalfa causes a decrease of the humus and in a majority of cases the nitrogen over that of the adjacent virgin soil.
4. The observed phenomenon in case of the grain-cropped land is probably due to the addition of nitrogen to the surface foot from lower depth by the addition of straw which also increases the humus content.
5. This work indicates that in a study of the nitrogen and humus problems in dry-farming, attention must be paid to greater depth of soil than the traditional ploughed surface.

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NOTES AND CORRESPONDENCE.

A USE FOR THE GOOCH IN PAINT ANALYSIS.

Recently the writer had occasion to analyze a green pole paint falling under the following specifications: Vehicle, pure linseed oil, 60-7 per cent; benzine, not over 2 per cent.; pigment, 33-40 per cent., made up as follows: graphite, 50 per cent.; gypsum, 35 per cent.; and a mixture of Prussian blue and chrome yellow, 15 per cent.

On attempting to separate vehicle from pigment, much difficulty was experienced owing to the exceeding fineness of the pigment. On extracting with ether in a Soxhlet thimble, as done by Holley and Ladd, the pigment repeatedly passed through the thimble in spite of all precautions. On centrifuging as done by the same authors, there was practically no separation after whirling for three hours at a high rate of speed.

Finally extraction with a succession of solvents, as done by Walker in *Bull.* 109, Bureau of Chemistry, was tried. Here we met with some measure of success, but still the separation was anything but complete, and it occurred to the writer to try the use of a gooch.

A gooch was carefully prepared, using a heavy bed of very fine asbestos; and through this crucible the successive extractions (as per *Bull.* 109) were decanted, using a fairly strong suction. In this manner we obtained a very satisfactory separation where other standard methods had failed.

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SOME NEW TESTS.

No. 1. *Differentiation of Anhydrous Acetic Acid from Glacial Acetic Acid.*—To the solution to be tested is added a small crystal of selenious acid (SeO₂) or the sodium salt. The latter is in this test preferable.

If anhydrous acetic acid is present a red precipitate of amorphous selenium will be noticed after boiling.

Glacial acetic acid will not react and after boiling will give absolutely clear solution.

No. 2. *Differentiation of High Per cent. Ethyl Alcohol from Methyl Alcohol.*—After distilling over a few cc. of the suspected mixture to concentrate alcohol if not present as such, add one or two drops of selenic acid, H₂SeO₄, with trace of silver halogen salt (best silver bromide). Ethyl alcohol will give a white amorphous precipitate. The methyl alcohol, common wood spirit, will not give this reaction. In exceptional cases it will give crystalline precipitate, leaving solution clear. Water will dissolve the milky precipitate of the ethyl alcohol. This test can also be used for esters.

No. 3. *Differentiation of Different Alkaloids, Especially Cantharidin.*—The extracted alkaloid is dissolved in concentrated sulfuric acid, 94-5 per cent., and a trace of sodium selenite (Na₂SeO₃) added to the solution. After heating, a purple reaction will be noticed, very characteristic, see table of color reaction of different alkaloids with same reagent.

TABLE OF COLOR REACTION OF DIFFERENT ALKALOIDS.

	H ₂ SO ₄ 96%.
Digitalin ¹ . . .	Only slight coloration.
Strychnin ¹ . . .	Only slight coloration.
Brucin ¹	Rose coloration (pink).
Aconitin ¹	Only slight coloration.
Atropin ¹	Only slight coloration.
Morphin ²	Green coloration in cold.
Codein ²	Light brown coloration.
Caffein ³	No coloration.
Chinin ⁴	No coloration in cold; in heat, slight coloration.
Spartin ³	No coloration.
Veratrin ⁴	Cherry-red coloration.
Cantharidin ⁵ . . .	No coloration.

No. 4. *Test to Detect Small Amounts of Benzine in Turpentine where the Official Test did not React or Satisfy the Definite Amount Present.*—A five or ten per cent. copper sulphate solution is used (3-4 cc. for reaction):

Benzin 1 to 2 cc.	Turpentine 1 to 2 cc.
Copper sulphate, 10 per cent. sol. 3 to 4 cc., gives clear solution; to this is added a small crystal of <i>potass. iodide</i> : dissolves with purple color, when shaken turns yellowish brown after some time.	Copper sulphate, 10 per cent. sol. 3 to 4 cc.; solution is not clear; to this is added a small crystal of <i>potass. iodide</i> : purple red, which gradually fades when shaken and when pure turpentine, green coloration results.

When "rosin" besides benzine and turpentine is present, the smell can distinctly be defined; solution is of a brownish color. The amount of benzine present will make solution more or less clear.

No. 5. *Nitric Acid Test.*—The acid must be concentrated, 40 per cent., or if diluted, neutralized and evaporated to dryness, in form of a nitrate.

Reagent.—To two or three cc. of fuming sulfuric acid is added a trace of "powdered tellurium," an eosin red color is produced. To this fuming solution one to two cc. of 95 per cent. H₂SO₄ are added. One cc. each in two test tubes is heated until decolorized. To one is added the nitrate and to the other a drop

¹ With Na₂SeO₃, brown coloration.

² With Na₂SeO₃, after heating, black precipitate.

³ With Na₂SeO₃, no coloration.

⁴ With Na₂SeO₃, purple coloration.

⁵ The cantharidin as cryst. alcohol, dissolved in concentrated 96% H₂SO₄ with Na₂SeO₃, gives slight purple coloration in the cold. By heating, the coloration increases and gradually turns into a dark coloration. This can be changed by adding alcohol which again turns up the darker purple coloration. Cocain same as morphin.

or more of the reagent. Then both tubes are slightly heated and to the tube containing the nitrate a drop or more of the reagent is added and immediately decolorized, whereas the one *without acid or nitrate* is still colored *eosin red*. On this principle I have founded or based a quantitative test of nitric acid by titration taking a unit of 0, 1 tellurium dissolved in 10 cc. fuming acid H₂SO₄.SO₃ and 10 cc. sulfuric acid, 95 per cent., added, will equal to or decolorize 0, 66 nitric acid of 66 per cent. No indicator is necessary.

Test No. 6. Detection of Copal Amber.—This test might not be useful, but is of the most tedious character and no book or literature will assist in this most complicated test.

The sample of the suspected mixture is finely powdered and about 4 cc. acetic ether added with 0.5 or less *cobalt nitrate* dissolved; then *acetic acid anhydrous* is added 2 cc. and about 1-2 cc. chloroform and heated. The copal mixture will dissolve, the amber mixture will not dissolve and will granulate. These two solutions added each separate to high per cent. ethyl alcohol, the copal solution will give silky color with precipitate of rosin. The amber solution will not alter the methyl alcohol solution, leaving a clear solution.

If both are together it is best to add water to the methyl alcohol solution, whereas the amber solution after shaking is still clear and the copal, if any or in small quantities, will give *milky* or *opalescent* solution.

DR. FRED. KLEIN.

IDENTIFICATION TEST FOR CAMEL.

The use of caramel as a coloring agent in pharmaceutical preparations, flavoring extracts, liquors and vinegar is so universal and the tests for identification of this substance having proven unsatisfactory in many instances, I submit the following reagent or solution which has been in use in our laboratory for a year with success on flavoring extracts and liquors, and, while its employment for detecting caramel in vinegar has been very limited it has given good results when put to such use.

The Solution.—

	Grams.
Tannic acid	1.00
Sulphuric acid (sp. gr. 1.84)	0.75
Water, q. s., ad	50.00

Dissolve the tannic acid in about 30 cc. of water, add the sulphuric acid (the precipitate first forming will dissolve) and then the rest of the water; let stand 24 hours. Lastly, filter. This solution keeps well.

The method of using for vanilla extracts is simply to add 5 cc. to 5 cc. of the extract, heat gently until the precipitate at first formed is almost all dissolved, then, on standing for 12 hours or overnight, a light or dark brownish substance (according to the amount of caramel present) will have been precipitated. True vanilla extractive gives a very slight precipitate of a different character than that of caramel.

When whiskey, brandy or other spirituous liquors are to be examined the greater part of the alcohol is evaporated, water added and tested the same as vanilla extract.

Much time is gained by this method when working upon many samples at one time as any number of test tubes can be prepared and allowed to stand overnight, the precipitate being observed the next morning.

G. H. P. LICHTHARDT.

SACRAMENTO CITY BOARD OF HEALTH.

TO DETERMINE FUSEL OIL IN DISTILLED LIQUORS.

The tedious and unsatisfactory methods now in use for the determination of fusel oil in distilled liquors may be replaced by the much more rapid and accurate method of Savelle with a few modifications.

To 25 cc. sample add 5 cc. of a normal alkali solution and digest for one hour on the water bath under a reflux condenser to saponify the esters, then connect with a suitable distilling apparatus and distil 25 cc.; add 5 cc. of water and distil an additional 5 cc.

To this distillate add 0.2 gram of *m*-phenylenediamine hydrochloride and boil under a reflux condenser for one hour to remove the aldehydes, after which distil, collecting 25 cc.; add 5 cc. of water and distil an additional 5 cc., bringing the distillate up to the original volume.

To this distillate add slowly an equal volume of C. P. H_2SO_4 conc., heat nearly to boiling, and while still hot add 5 to 10 drops of a 1 : 1000 solution of furfural; a pink or red color will develop in the presence of the higher alcohols, which may be compared with a standard solution of amyl alcohol treated in the same manner.

By this method less than 0.01 per cent. of fusel oil can be determined and 1 part in 20,000 detected.

H. P. BASSETT.

DELAWARE EXPERIMENT STATION, NEWARK.

BOOK REVIEWS AND NOTICES.

Metallography. C. H. DESCH, Graham Young Lecturer in Metallurgical Chemistry, Glasgow University. Crown 8vo. 9s. Longmans, Green & Co.

The following are the principal headings by which the book is divided into 18 chapters: Introduction, The Diagram of Thermal Equilibrium; Solid Solutions or Mixed Crystals; Ternary or More Complex Systems; Metals which are only Partially Miscible in the Liquid State; Practical Pyrometry and Thermal Analysis; The Preparation of Microsections; The Microscopical Examination of Prepared Sections; The Crystallization of Metals and Alloys; Undercooling and the Metastable State; Diffusion in the Solid State; The Physical Properties of Alloys; Density, Thermal Expansibility, Hardness, Electrical Conductivity, Thermo-electric Power, Magnetic Properties; Electromotive Force and Corrosion; The Construction of the Equilibrium Diagram; Molecular Condition of Metals in Alloys and the Nature of Inter-metallic Compounds; The Plastic Deformation of Metals and Alloys; The Metallography of Iron and Steel; The Metallography of Industrial Alloys.

Metallography is defined as a study of the internal structure of metals and alloys and of its relation to their composition and to their physical and mechanical properties. The book, which is one of the text-books of physical chemistry edited by Sir William Ramsay, follows this line and summarizes our present knowledge in a very clear and logical way.

Taking the diagram of thermal equilibrium and the freezing point curve for a beginning, the different type cases are developed step by step. The Phase rule is given in brief and its application to alloys is shown, while the section on the ternary systems is well written. The chapter on the crystallization of metals and alloys is very clear indeed and the illustrations well chosen.

The later part of the book, dealing with the Metallography of Iron and Steel, gives us a good summary of our present knowledge and includes Upton's diagram. An appendix shows in tabular form the systems of which the equilibrium diagrams have been published, with the probable formulae of intermetallic compounds formed.

Mr. Desch has written an excellent book in a convincing way which will be welcomed alike by the student and the practical man. A possible criticism is that the section on practical pyrometry and thermal analysis is too condensed for the general reader, but the references given will help the student over any difficulty. In conclusion, the author is to be congratulated

in setting forth so important a subject as metallography in so instructive and so interesting a manner.

W. CAMPBELL.

Transactions of American Institute of Chemical Engineers. Vol. 2, 1909. Cloth, large 8vo. pp. 308. New York: Published by the Institute. For sale by D. Van Nostrand Co.

The second volume of the transactions of the American Institute of Chemical Engineers contains a number of valuable and readable articles, in addition to the official transactions, the constitution and a list of officers, committees and members. The papers are nineteen in number and are written by well-known chemical engineers, among whom are the names of Prof. Chas. E. Munroe, Samuel P. Sadtler, Wm. M. Booth, A. C. Langmuir and Edw. R. Taylor. The Institute has grown so that it now consists of about 130 members. The volume will find a wider circle of readers than its own members, as it will be found of interest to many chemists and engineers. Of the papers the following are selected to show the general character of the volume:

Chemical Industries of America, Chas. E. Munroe; Conservation and the Chemical Engineer, Samuel P. Sadtler; Efficiency Limits in the Power-Gas Producer, Prof. Wm. D. Ennis; Heat Efficiency of Smokeless Combustion and Heat-Absorbing Capacity of Boilers, A. Bement; Creosote Oil from Water Gas Tar, Samuel P. Sadtler; A Method of Clay Control for Manufacture of Cement, John G. Dean; The Utilization of Waste India Rubber, Stephen P. Sharples; Commercial Extraction of Grease and Oils, Wm. M. Booth; Glycerine Refining in Multiple Effect Stills, F. J. Wood; Electric Furnace for the Smelting of Iron Ore, Edward R. Taylor.

Allen's Commercial Organic Analysis. Vol. II. Oils and Fats.

Edited by HENRY LEFFMANN and W. A. DAVIS, with the co-operation of C. AINSWORTH MITCHELL, LEONARD ARCHBUTT, C. REVIS, E. R. BOLTON, C. A. KLEIN, W. ROBERTSON, JOHN ADDYMAN GARDNER and AUGUSTUS H. GILL. Large 8vo. pp. 520. Philadelphia: P. Blakiston's Son & Co. 1910. Price, \$5.00 net.

Following the new plan in the present edition of Allen's widely known "Commercial Organic Analysis," the editors have produced what is to a large extent a new work. The best distinctive feature of the previous editions, namely, the general descriptive matter, is prominent in the fourth edition. It is larger than the third edition by 133 pages, but this numerical difference scarcely covers the extension of matter. C. Ainsworth Mitchell has contributed the chapter on General Properties and Analytical Methods; Leonard Archbutt, Special Characters and Methods; Butter Fat is treated by Cecil Revis and E. R. Bolton; Lard, by C. Ainsworth Mitchell; Linseed Oil, by C. A. Klein; Higher Fatty Acids, by W. Robinson; Soap, by Henry Leffmann; Glycerol, by W. A. Davis; Cholesterols, by John A. Gardner; and Wool-Fat and Cloth Oils, by Augustus H. Gill. The book is essentially a descriptive and analytical work. It enters into technology only to the extent necessary to make the analytical control methods intelligible. More concise than the works of Lewkowitsch, Hefter, Benedikt Ulzer and Ubbelohde, it is generally accurate, painstaking and well up-to-date.

Theorie der Gewinnung und Trennung der Aetherischen Oele durch Distillation. By DR. C. VON RECHENBERG. Large 8vo. pp. v + 490. Miltitz: Schimmel & Co.

The present work is an excellent addition to the literature of the volatile oils. Previously the student was limited in the study of the subject to the treatment of moderate length only in Gildmeister & Hoffmann's "Volatile Oils." The first chapter takes up the practice of distillation, beginning with a general

description of the processes and apparatus as applied to the distillation of volatile oils. Next in order in the same chapter is a brief account of the occurrence of volatile oils in plants, illustrated with histological cuts of the oil-bearing parts of plants. Then follows a description of the preparation of plants for distillation, together with an account of the losses which may occur and the general methods of distillation. The details of present-day practice and the forms of stills used are given. The second chapter takes up Dalton's law of diffusion; the third, distillation of mixtures of insoluble fluids; the fourth, distillation under increased and diminished pressure; the fifth, distillation with superheated steam; sixth, physico-chemical phenomena in plant distillation. Thus the second, third, fourth, fifth and sixth chapters are largely theoretical. In the seventh chapter a description is given of the practice in various parts of the world, of distilling essential oils on the small scale. This chapter is of a descriptive nature, is well illustrated and of general interest. The eighth chapter considers molecular association and dissociation and their influence on vaporization. The ninth chapter takes up boiling points and vapor tensions of substances, and includes a table of the boiling points under various pressures of many organic substances, a number of which occur in the volatile oils. This chapter concludes with an excellent bibliography of the works used in the preparation of the table. The tenth chapter considers solution in general; the eleventh, distillation of mixtures of substances of limited solubility; the twelfth, distillation of homogeneous mixtures with minor boiling temperatures; the thirteenth, distillation of homogeneous mixtures without constant boiling point; the fourteenth, distillation of homogeneous mixtures with major boiling temperatures. It is stated by the author that the work will form a part of the second edition of Gildmeister & Hoffmann's "Die ätherischen Oele," and pages are numbered from 259 to 751 as though reprinted from another work. It appears that the work could be somewhat better arranged, by bringing together all of the theoretical parts in one place, and the practical portions in another. Such an arrangement would make it somewhat more systematic. In general, it is to be commended as an excellent addition to the literature.

Technologie der Fette und Oele. By GUSTAV HERTER, with co-operation of O. HELLER, G. LUTZ, FELIX KASSLER, and others. Vol. III. Large 8vo. pp. xii + 1022. Well illustrated. Berlin: Julius Springer. 1910. Price, paper 32 Marks; bound 35 Marks.

With the third volume of this valuable work out of the press, chemists in the oil and fat industries may look for its speedy completion. The two preceding volumes gave promise that the work as a whole would be in all probability the most comprehensive reference work on the subject of fats and oils industries which has yet been published, and the third volume reaches the high standard set by the first two volumes. The arrangement of the subject matter is as follows: Edible Oils and Fats; Plant and Animal Lubricating Oils and Fats; Polymerized Oils; Oxidized Oils; Sulphurized Oils; Iodized, Brominized, and Nitrated Fats; Textile Oils; Stearic Acid Manufacture; Candle Manufacture. Prof. Max Bottler, Wurzburg, has contributed the chapter on burning oils and Dr. Herbig, of Chemnitz, the chapter on textile oils. While all portions of the volume are commendable, those divisions devoted to edible oils and fats, stearic acid and candle manufacture are especially good for their excellent arrangement and classification and comprehensive treatment. If any criticism can be brought against the chapter on edible oils and fats, it would be the failure to give due prominence to the ever-increasing use of tropical vegetable oils and fats in the manufacture of oleomargarine. In the chapter on stearic acid manufacture, following an excellent general and historical introduction,

the methods of saponification by means of the autoclave, sulphuric acid, Twitchell's process, and the enzymes are given proper consideration. The treatment of waste fats and glycerine is reserved for Volume IV. It is needless to add that, inasmuch as the work issues from the press of Julius Springer, the typography, paper and make-up are the best.

The Polytechnic Engineer. Vol. X. Large 8vo. pp. 144. Brooklyn: Published by the Undergraduates of Polytechnic Institute of Brooklyn. 1910.

Volume X of the *Polytechnic Engineer* contains four excellent articles of special interest to chemists and chemical engineers. They have the following titles and authors: The Investigation of Congo Blue and Preparation of Congo White, by Irving W. Fay, A. H. Tag and M. G. Hawkins; Some Experiments on the Case-Hardening of Steel by Gases, John C. Olson, John R. Briery, and John S. Weiffenback; Initial Temperatures at which Oxides of Metals Give up Oxygen to Reducing Gases, by Irving W. Fay, Albert F. Seeker, Frederick H. Lane and Geo. E. Ferguson; A Liquid which will Dissolve Cotton, by Irving W. Fay and Chas. H. Lewis. The volume as a whole is commendable and is well printed.

Engineering Chemistry. Fourth Edition. By THOMAS B. STILLMAN. 8vo. pp. ix + 744. Easton, Pa.: Chemical Pub. Co. 1910. Price, \$5.00.

The three previous editions of Dr. Stillman's work have proved of the greatest usefulness to chemists in works laboratories. The reason for this is that they have contained a large amount of specific information concerning the physical and chemical examination of gas, water, materials of construction and engineering materials not readily accessible elsewhere. The work is so widely known and has been so favorably received in the past that special comment would be superfluous. In the present edition the subject of pyrometry is handled by Edw. A. Uehling and W. H. Bristol. "Foundry Chemistry" is treated by Herbert E. Fields, "Acetylene" by R. E. Bruckner. The official methods of sampling iron ore as adopted by the U. S. Steel Corporation are included. Many other additions have been made in the present edition in bringing it down to date. It is a useful book for all works laboratories and colleges.

The North Carolina Geological and Economy Survey has issued Bulletin No. 18, *Bibliography of North Carolina Geology, Mineralogy and Geography*, with list of maps, by FRANCIS BAKER LANEY and KATHERINE HILL WOOD; and Bulletin 21, *Gold Hill Mining District of North Carolina*, by FRANCIS BAKER LANEY.

SCIENTIFIC AND INDUSTRIAL SOCIETIES.

ANNOUNCEMENT OF THE TWENTY-SEVENTH ANNUAL CONVENTION, ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, NOVEMBER 10-12, 1909.

The twenty-seventh convention of the Association of Official Agricultural Chemists will be held in Washington, D. C., at the Raleigh Hotel, corner of Twelfth Street and Pennsylvania Avenue, opening on November 10th, at 9 o'clock.

The hotel rates will be as follows, European plan:

Room for one person.....	\$2.00
Double room for two persons.....	3.00
Room and bath for one person.....	3.50
Double room and bath for two persons.....	5.00

As the hotel will provide a large hall on the top floor of the building for the convention, as well as other facilities for the meeting, it is urged that the members make the Raleigh their headquarters, if possible, and reserve their rooms a few days in advance.

ORDER OF BUSINESS.

Thursday, November 10th.

Morning session: Phosphoric acid; Nitrogen; Potash; Soils; Inorganic plant constituents.

Afternoon session: Appointment of committees (resolutions, etc.); Insecticides; Water; Committee A on recommendations of referee; Report of special committees—(amendments to constitution; appropriation; availability of phosphoric acid; compilation of by-laws; food standards; unification of terms; standardization of alcohol tables; testing of chemical reagents; unification of methods of analysis of fats and oils).

Friday, November 11th.

Morning session: Food adulteration (reports to be called for in order given in list of associate referees).

President's address (special order for 12 o'clock).

Afternoon session: Food adulteration continued. Separation of nitrogenous bodies (meat proteids; milk and cheese proteids; vegetable proteids). Committee C on recommendations of referees.

Saturday, November 12th.

Morning session: Dairy products. Foods and feeding stuffs. Sugar (chemical methods and molasses methods). Committee B on recommendations of referees. Committees (resolutions, constitution, etc.). Tannin.

Afternoon session: Drugs and medicinal plants.

Referees Please Note.—Recommendations in triplicate, together with an abstract of the report, should be in the hands of the respective chairmen of committees A, B, and C, by October 17th at the latest, if action is to be taken at the meeting and the necessary approval of the Committee on Revision as a whole obtained (see pages 168 and 205 of Bulletin 132, Bureau of Chemistry, or Circular 52, pages 19 and 32).

H. W. WILEY,
Chief, Bureau of Chemistry,
Secretary, Association of Official
Agricultural Chemists.

WASHINGTON, D. C., August 13, 1910.

AMERICAN ELECTROCHEMICAL SOCIETY.

The Eighteenth General Meeting of the American Electrochemical Society will be held in Chicago, October 13th to 15th, by invitation of the newly formed Chicago Section. The program will include excursions to the Illinois Steel Co.'s plant at South Chicago, the Indiana Steel Co.'s plant at Gary, the U. S. Smelting, Refining and Mining Co.'s plant at Grasselli, the packing plants, etc. Dr. H. N. McCoy, of the University of Chicago, is chairman of the Committee on Arrangements.

EINLADUNG ZUR 26. JAHRESVERSAMMLUNG DES
DEUTSCH-AMERIKANISCHEN TECHNIKER-VER-
BANDES. ABZUHALTEN VOM 1. BIS 5. SEP-
TEMBER D. J. IN NEWARK, N. J.
HAUPTQUARTIER: L. ACHEL-
STETTER, 842 BROAD ST.

FESTORDNUNG.

DONNERSTAG, DEN 1 SEPTEMBER.

Vormittags 10 Uhr: Sitzung der Delegaten im Hauptquartier.

Nachmittags 8 Uhr: Empfang und Begrüssung der Gäste im Hauptquartier auf Einladung des T. V. Newark.

FREITAG, DEN 2 SEPTEMBER.

Vormittags 9 Uhr: Zusammenkunft im Hauptquartier. Von da Abfahrt mit der Strassenbahn (Orange-Linie) nach

Edison's Laboratorien, West Orange, N. J. Von New York nehme man die Erie-Bahn bis West Orange, ab "Chambers Street Ferry," punkt 9 Uhr morgens. Vom Bahnhof West Orange etwa 8 Minuten zu Fuss nach den Laboratorien.

Vormittags 10-Uhr-30: Beginn der Besichtigung der Werke.
Nachmittags 1 Uhr: Gemeinschaftliches Gabelfrühstück im Hauptquartier.

Nachmittags 2-Uhr-30: Abfahrt vom Hauptquartier mit der Strassenbahn (Turnpike- oder Kearny-Linie) nach Henry R. Worthington Hydraulic Works, Harrison, N. J.

Nachmittags 3 Uhr: Beginn der Besichtigung der Werke.

Nachmittags 8 Uhr: Festkommers im Hauptquartier.

Tagesplan für die Damen.

Zum Vormittags-Ausflug sind die Damen freundlichst eingeladen.

Nachmittags 3 Uhr: Abfahrt vom Hauptquartier mit der Strassenbahn nach Krügers "Greisenheim." Dort Preiskegeln und

Nachmittags 6 Uhr: Gemeinsames Abendessen auf Einladung der Damen des T. V. Newark.

Hierauf Besuch des Sommertheaters im "Olympic Park".

SAMSTAG, DEN 3 SEPTEMBER.

Familienausflug.

Vormittags 10 Uhr: Zusammenkunft im Hauptquartier.

Vormittags 10 Uhr 30: Abfahrt in Sonderwagen der Strassenbahn (Orange-Linie bis Endpunkt), Spaziergang über "Eagle Rock" nach "Crystal Lake Park Hotel" (30 Minuten zu Fuss, auch Omnibusverbindung).

Nachmittags 1 Uhr: Gemeinschaftliches Mittagessen.

Danach Unterhaltungen im Freien: Gesellschaftsspiele, Bootfahren u. s. w. Photographische Aufnahme aller Festteilnehmer.

Bei ungünstiger Witterung steht ein grosser Tanzsaal zur Verfügung.

SONNTAG, DEN 4 SEPTEMBER.

Vormittags 10 Uhr: Allgemeiner Technikertag im Hauptquartier Berichte der Delegaten und Jahresberichte. Nachbar Fröhschoppen.

Nachmittags 1 Uhr: Gemeinschaftliches Gabelfrühstück.

Nachmittags 3 Uhr: Vortrag von Herrn Dr. L. H. Friedburg, Professor am "College of the City of New York," Ehrenmitglied des T. V. Brooklyn.

"Radioaktivität und Beständigkeit der Elemente mit Seitenlicht auf Transmutation und Alchemie."

Nachmittags 8 Uhr: Festessen und Ball im Hauptquartier.

MONTAG, DEN 5 SEPTEMBER.

(Labor Day.)

Vormittags 11 Uhr: Katerfrühstück und Abschied.

Nachmittags: Unoffizieller Ausflug nach Coney Island nach Verabredung.

Absteigequartier: Continental Hotel, 454 Broad St., Newark, N. J.

Der Fest-ausschuss:

Z. Freund, Obmann.	H. Hoppe, Schriftführer.
Th. Koch, Rechnungsführer.	
B. A. von Bergen, Kurt Ledig,	Phil. Volz,
Wilh. Heck,	L. F. Mergott,
	S. I. Oesterreicher.

Der Ausschuss der Damenvereinigung:

Frau E. F. Harder,	Frl. E. Koch,	Frau Th. Teimer,
Frau Wilh. Heck,	Frau S. I. Oesterreicher,	Frau A. Wack,
Frau Th. Koch,	Frau J. H. Sebald,	Frau E. W. Zeh,
	Frl. H. Sebald.	

Verein.	Delegaten:	Stellvertreter.
Baltimore,	Emil Eiselt,	Heinrich Turk.
Brooklyn,	Hermann Dann,	August Wittel.
Chicago,	A. Sonderhof,	
Newark,	Georg Sulzer,	B. A. von Bergen.
New York,	Wm. Mueser,	H. J. Schäfer.
Philadelphia,	O. Güssefeldt,	C. D. Rinald.
Pittsburg,	H. Schütte,	A. C. Sprecht.
Rochester,	O. Kallenbach,	Arno Greiner.
Washington,	Dr. H. C. P. Weber,	Dr. S. O. Graser.

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.

963,174. Process for Making Concentrated Sulfuric Acid. OTTO PROLSS, of Kansas City, Missouri. July 5, 1910.

This is a process in which sulfuric acid of a strength of 66° is produced by independently denitrating and concentrating different bodies of liquor, using sulfurous acid as gas in this connection, and thereafter bringing together the hot gases from the concentrating means, and the cooler gases from the denitrating means, and subjecting them to the action of the weak sulfuric acid.

In carrying out the process there are used three independent and separate chambers, which may be within one or more structures or buildings, but which, as shown in the accompanying illustration, are in three towers, A¹, A² and B, each of which may be a Glover tower of ordinary construction.

With the lower end of each of the towers A¹, A² communicates a pipe or flue E, from which hot sulfurous acid gas is conducted to the tower from the burners, or roasting furnaces, etc., and from the top of each tower A¹, A² a pipe or flue V extends laterally to the tower B. From the tower B a pipe or flue, 4, extends to an exhauster, F, of any suitable character to convey the gases to the chambers; and suitable coolers, C¹, C², C³, are arranged to receive the liquor from the bottom of each of the towers.

The tanks T¹, T² and T³, are suitably connected with the towers A¹, A², and B respectively, by means of suitable dis-

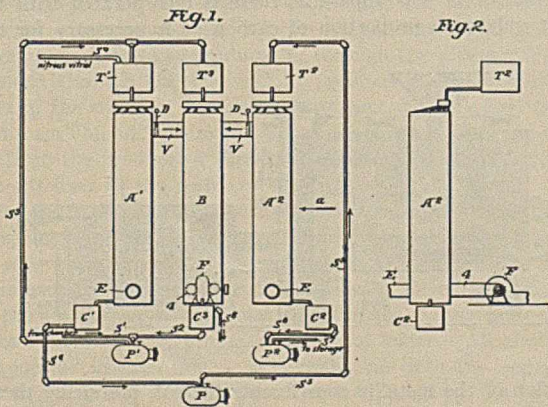
tributing devices. The liquor in C³ contains nitrogen oxides in solution. The weak acid from tank T³ passes down the tower B and is delivered into the cooler C³, an overflow pipe, S³, being provided which may lead to a storage tank or the chambers. A pipe, S³, supplies the tank T¹ with nitrous vitriol from the Gay-Lussac tower. The liquor from tank T¹ passes down the tower A¹, collects in the cooler C¹, and is delivered by means of a pipe, S¹, to the pump P, which delivers the liquor by means of a discharge pipe, S², to the tank T². The liquor from the tank T² passes down the tower A², discharges into the cooler C², which delivers by a pipe, S⁶, to a pump, P², which is connected to a discharge pipe, S⁷, leading to the storage tank.

The hot gases from the tower A² pass to tower B and the cooler gases from the tower A¹ also pass to the tower B, suitable dampers, D, D, regulating the flow of these gases, and as these are mixed together in the tower B, they are subjected to the action of weak sulfuric acid, which is discharged into the tower B from the tank T³ supplied from the lead chambers and from tank C³ through S³ as described and properly distributed in the tower.

In operating the apparatus both towers are supplied through the flues E with the sulfurous acid gas, and the tower A¹ is fed with a mixture of chamber acid and nitrous vitriol, as usual, with the result that the liquor at the bottom of the tower and carried to the cooler C¹ is of a strength of from 60° to 62° Bé. This liquor is then elevated to the top of the tower A² by the pump P, the apparatus being so regulated that a concentration will be effected in the tower A² and will in all cases produce a liquor exceeding 60° Bé., and in order to obtain this liquor free from nitrogen oxides, no niter in any shape must be admitted to the tower A² so that the denitrating is effected wholly in the other tower. Owing to the high concentration of the acid in the tower A² the gases issuing therefrom through the pipe V are very hot and must be cooled down before they enter the lead chambers.

The gases issuing from the denitrating tower, of moderate temperature and laden with nitrogen compounds, and those issuing from the concentrating tower, and free from nitrogen compounds and of high temperature, are brought together in the intermediate tower, the function of which is to unite and thoroughly mix these gases and reduce the temperature of the gases from the concentrating tower so that the mixed gases are at a temperature which will not be injurious in the further operations. To this end these mixed gases are subjected in the intermediate tower to the action of the cool, weak sulfuric acid, the operation being so regulated that the gases will issue from the intermediate tower at such temperature as is demanded by mechanical and chemical considerations. In other words, my process splits the available burner gas into two parts: one part is used to denitrate the nitrous vitriol and concentrate the weak sulfuric acid formed in the chambers, and the other part of the burner gases is used to effect a further concentration of the 60-62° sulfuric acid thus produced. The waste gases resulting from the two parallel operations are then brought together, mixed and cooled in a subsequently intermediate tower before being admitted to a series of chambers and Gay-Lussac towers.

The liquor from the intermediate tower is suitably cooled in the cooler C³ and constitutes part of the body of liquor which is operated upon in denitrating and concentrating in the tower A¹. The liquor from the tank C² is carried by a pump, P², to a suitable storage reservoir. By means of this process sulfuric acid containing as high as 96° (H₂SO₄) has, it is claimed, been obtained without any greater amount of fuel, labor or supervising than is ordinarily required in producing acid of much lower strength, and by means of properly constructed flues and dust chambers the acid may be had of the proper light color, or freedom from color.

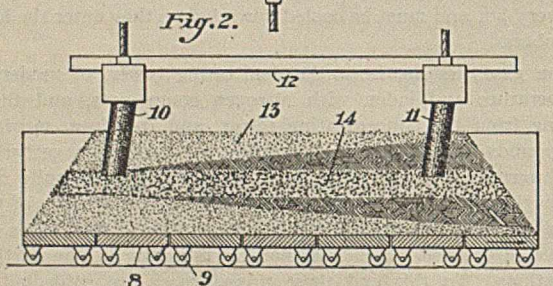
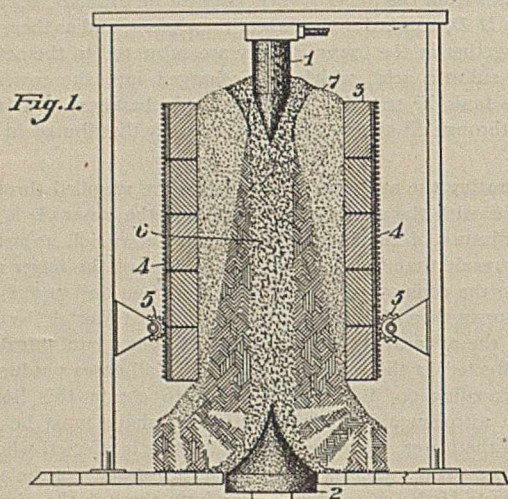


tributing devices. A pump, P¹, supplied by a pipe, S¹, with chamber acid from the chambers, and by a pipe, S², from the cooler C³, delivers the liquor through the discharge pipe S³, and branch pipes into the tanks T¹ and T³. It will be noted

965,142. Method of Making Silicon Carbide. FRANK J. TONE, Niagara Falls, New York. July 19, 1910.

This invention relates to the production of silicon carbide, and is designed to reduce the number of furnaces necessary for a certain output and increase the output of single furnaces.

Under the present practice, a stationary electric furnace is employed consisting of a long rectangular receptacle with stationary electrodes at opposite ends, the charge of coke and sand being placed around a resistance core embedded therein. The current passes through the resistance core which is in contact with the electrodes in its end portion. The furnace is operated for a certain period varying from twenty-four to



thirty-six hours, after which the current is cut off, the furnace cooled down and the contents removed. It is then recharged and the operation repeated. The period of cooling and recharging occupies several days, and consequently, a series of furnaces is necessary in order to keep one furnace in operation at all times. The output of a furnace is therefore comparatively small.

This invention is designed to afford a continuous process in which there is no cooling down or cessation of operation, the current being continuously supplied as are also the charge and core. The finished products are also removed in a substantially continuous manner.

The accompanying illustration shows a furnace of vertical type in which the process may be carried out. The side walls of the furnace are formed of transversely divided refractory sections, 3, secured to or having rack sections, 4, engaging with pinions, 3, 5, which may be operated to give a continuous or intermittent downward movement to the wall sections. The wall sections may be guided within any suitable supports or guides to hold the charge and core in place and are added at the top and removed at the bottom in the downward movement.

The resistance conductor consists of granular core material, 6, which is supplied at and around the upper electrode, and is

surrounded by the charge mixture of carbon and silicious material, 7, which is also supplied at the top. As the containing walls move downward, they carry with them the charge and resistance core. The rate of motion is so regulated that when the charge shall have reached the lower electrode, it will have been converted into silicon carbide, the zone of carbide which is formed being of such diameter as will represent an economical rate of production. The lower electrode is preferably pointed or conical in shape, so that the core material and finished product will be given an outward movement, and fed into a position where it may be readily removed from the furnace.

964,268. Apparatus for Smelting Ores Yielding a Volatile Metal. WOOLSEY MCA. JOHNSON, of Hartford, Conn. July 12, 1910.

This apparatus comprises an electric smelting furnace, a reducer for the volatile reaction products therefrom, and a condenser.

The furnace is so constructed as to facilitate the production and removal of the fluid slag, and the reducer comprises a chamber for containing a body of coke, coal or other carbonaceous material, provided with electrodes in contact with said body.

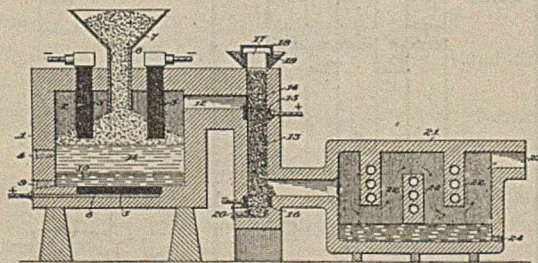
In the accompanying illustration the furnace is shown as comprising a smelting chamber, 2, formed of or interiorly lined with a suitable refractory material, 3, 3, representing carbon electrodes supported in the upper part of the furnace and terminating above the level of a slag tap, 4. The electrodes 3, 3 are of the same polarity, the opposing electrode 5 being embedded in the hearth of the furnace, in electrical contact with a conductive plate or bar, 6. 7 is a hopper for the introduction of the charge 8, and 9 is a tap for the removal of non-volatile metals or matte, 10.

The volatile reaction products pass through the outlet 12 and thence through a column, 13, of coke or coal contained in a chamber, 14, and adapted to be heated by an electric current passing between upper and lower ring electrodes, 15, 16, set in the walls of the reducing chamber. The reducing chamber is preferably connected electrically in series with the furnace.

From the reducing chamber 14 the vapors, free from carbon dioxide, aqueous vapor and other compounds capable of oxidizing zinc, pass to a suitable condenser, 21, shown as provided with water-cooled partial partitions, 22, constructed to direct the vapors in a tortuous path.

23 is the outlet for non-condensing gases, and 24 the metal tap.

In the use of this apparatus there is incorporated with the charge only such proportion of carbon as is necessary for the



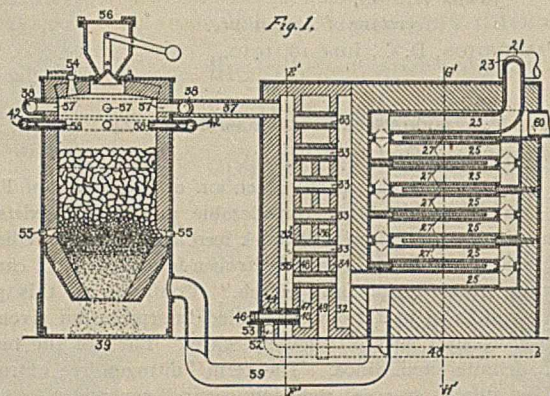
reduction of the metallic constituents thereof, producing thereby an easily fusible slag which may be tapped off as desired; any lead, copper or iron accumulates in metallic state beneath the slag and is withdrawn as desired, and fresh portions of the charge may be admitted continuously or at intervals.

This invention consigned to the Continuous Zinc Furnace Company, of Hartford, Connecticut.

964,901. Process of Treating Combustible Gas. HENRY L. DOHERTY, of New York, N. Y. July 19, 1910.

This invention consists of a method of treating combustible gases (such as blast-furnace gases and what is known as Mond gas) containing an appreciable amount of carbon dioxide by preheating the gas to a temperature sufficient to supply the heat required for the dissociation of the carbon dioxide—or to a temperature as near to such temperature as is practicable—and then passing the preheated gas through the fuel bed of a down-draft producer, whereby the contained carbon dioxide is reduced to carbon monoxide with the production of a gas nearly free from carbon dioxide and which is particularly suitable for use in gas engine and high temperature work.

The object of the invention is to furnish a process for carrying out such treatment of the gas without the addition thereto of a



large volume of inert nitrogen such as is unavoidably introduced into the gas in the method of carrying out such treatment heretofore known.

The method of operation is as follows:

Air enters the bottom flues of the recuperator 4 through the dampers 1, 2 and 3 which regulate the volume and distribution of the air passing into the recuperator. Passing through the air flues of 4 the air comes into contact with a layer of water which is maintained in the necessary number of flues to insure proper saturation of the air current. The hot gas from the producer passes through the gas flues enveloping the air flues its direction of flow being parallel to, but opposite in direction to the air current. The greater part of the sensible heat which the gas carries out of the producer is thus returned to the latter in the heated air and water vapor. The hot air-water vapor current after leaving the recuperator is introduced into the lower part of the gas producer 6, through the pipe 5. Passing upward through the fuel bed in producer 6, the oxygen and part of the water of the air current react with the fuel therein and form "Mond" gas in the ordinary manner. This gas passes off from the producer 6 through the pipe 7 and thence through the gas flues of the recuperator 4.

In order to maintain as high a temperature differential, as possible, between the air current and gas current it is desirable to secure the evaporation of the water as low down in the recuperator as possible. For this reason, I introduce the water to the air flues at such a point that the air passing through the flues is loaded with proper quantity of water. All the water evaporated in each flue is, preferably, run into the uppermost water pan in use and the pans below supplied by the overflow from this. Dams in the flues having water connections insure the maintenance of a shallow layer of water in the flues and thus form water pans of the bottom of the sections. If the amount of evaporation is lower than is required, water is admitted to a section higher up in the recuperator. The cooled

Mond gas leaving the recuperator then passes to the ordinary scrubbing towers 17, 18 and 19, where the ammonia is removed from the gas. These towers do not necessarily differ from the ones at present in use. From 19 the gas is withdrawn by the exhauster 20 and forced through the pipe 21 to the recuperator 22. This recuperator is in reality two separate recuperators, one for air and the other for gas, built, side by side, within the same shell. The section shown in Fig. 1 is through one of the gas flues, but the construction is identical on the air side.

The cold Mond gas and air passing through the recuperator go next to their respective superheaters, 30 and 31. These are built within the same shell but are so arranged that there is no communication between the gas and air sides.

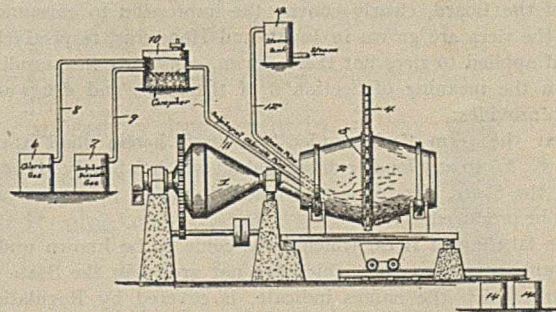
The process is defined in the inventor's patent as consisting in treating a combustible gas containing carbon dioxide which consists in removing from said gas substantially all of its condensable water-vapor, superheating the major portion of said gas in a suitable superheater, and passing the superheated gas downward through a deep bed of fuel in a suitable enclosed chamber, said high temperature to reduce the carbon dioxide of said gas by adding to said gas, previous to passing the same through said fuel bed, the minimum quantity of oxygen that will maintain said fuel bed at the desired temperature.

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

965,714. Process for Extracting or Separating Precious Values from Ores. JAMES S. ISLAND, Toronto, Ontario, Canada. July 26, 1910.

This invention relates to a process of extracting the precious values from ores, by reducing the mineral ores to a soluble state or salts, by subjecting the ore to a mixture of sulfuric and hydrochloric acids.

The ore, if coarse, is crushed by a suitable crusher (not shown) and then pulverized by a pulverizer. The ore is pulverized to a grade, that will pass through a hundred mesh screen, then deposited in a converter or separator, where it is subjected to the



sulfuric and hydrochloric acids. These sulfuric and hydrochloric acids are produced by passing a mixture of chlorine and sulfur dioxide gases over an area of camphor, then together with the steam, is projected into the converter separator, thereby producing sulfuric and hydrochloric acids.

When the sulfuric and hydrochloric acids come in contact with the ore in the converter or receptacle, it reduces the ore and the metal therein, into a soluble salt.

The process is defined in the patent as consisting in separating the metal values from ores, by reducing or converting the metal values into soluble salts, by subjecting the ores to a chemical fluid, composed of chlorine, sulfur dioxide and camphor, then combining this fluid with steam, thereby producing sulfuric and hydrochloric acids.

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

OFFICIAL REGULATIONS AND RULINGS.

FOOD INSPECTION DECISION NO. 122.

The Labeling of Port and Sherry Wines Produced in the United States.

A hearing was held on March 21, 1910, before the Secretary of Agriculture and the Board of Food and Drug Inspection on the labeling of wines produced in California, which for many years have been known as "California Port" and "California Sherry," respectively.

It is the view of the Department that the terms "Port" and "Sherry" without qualification are properly applied only to the products from Portugal and Spain, respectively, but it is held that domestic ports and sheries are not misbranded if the terms "Port" or "Sherry," as the case may be, are qualified by the name of the State where the wine is produced.

F. L. DUNLAP,
GEO. P. McCABE,

Board of Food and Drug Inspection.

Approved:

JAMES WILSON,

Secretary of Agriculture.

WASHINGTON, D. C., May 31, 1910.

FOOD INSPECTION DECISION NO. 123.

Labeling of Rices.

Inquiries have been received as to what is the proper branding under the food and drugs act of certain varieties of rice which have come to be known under geographic names. It is well known among the trade that there are current in commerce in the United States varieties of rice grown in Japan and varieties of rice grown within the United States from seed originating in Japan, which are marked and sold as "Japan Rice," irrespective of origin, and that a variety of rice grown in Mexico is imported as "Honduras Rice." The names "Japan Rice" and "Honduras Rice," used without qualification, in the opinion of the Board, clearly convey the impression to consumers that the rices are grown in Japan and Honduras, respectively, and if applied to rices not there grown, constitute misbranding within the meaning of Section 8 of the foods and drugs act, which provides:

That the term "misbranded" as used herein shall apply * * * to any food or drug product which is falsely branded as to the State, Territory, or country in which it is manufactured or produced.

The labeling of rices, which have come to be known under geographical names, and which are not grown in the State or country which the names indicate, is covered by Regulation 19, paragraph (c), reading as follows:

The use of a geographical name in connection with a food or drug product will not be deemed a misbranding when by reason of long usage it has come to represent a generic term and is used to indicate a style, type, or brand; but in all such cases the State or Territory where any such article is manufactured or produced shall be stated upon the principal label.

To meet the requirements of this regulation rices grown within the United States, labeled "Japan Rice," should have also plainly stated on the label "Grown in the United States;" rices grown in Mexico or Louisiana, for example, labeled "Honduras Rice," should have also stated plainly on the label "Grown in Mexico," or "Grown in Louisiana," as the case may be.

There are also on the market varieties of rice labeled "Carolina White" and "Carolina Gold," which are grown in North and South Carolina, and also in any other States from Carolina seed. The Board is of the opinion that the names "Carolina White" and "Carolina Gold" by long usage have come to mean

particular varieties of rice rather than rice grown in North or South Carolina, and such rices will not be held to be misbranded if plainly labeled "Carolina White" or "Carolina Gold," as the case may be, whether qualified or not, as growers or packers may see fit, by a statement of the name of the locality where the rice is actually grown. On the other hand, if it is desired to designate rices grown from Carolina seed in States other than North and South Carolina as "Carolina Rice," there should also be plainly stated on the label the name of the locality where the rice is actually grown, as, for example, "Carolina Rice, Grown in Arkansas."

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

Board of Food and Drug Inspection.

Approved:

JAMES WILSON,

Secretary of Agriculture.

WASHINGTON, D. C., June 16, 1910.

FOOD INSPECTION DECISION NO. 124.

Labeling of Stock Feed.

It has been brought to the attention of the Board of Food and Drug Inspection that considerable uncertainty exists in the minds of manufacturers of stock feed as to what ingredients are included within the terms "nitrogen-free extract," "carbohydrates," and "sugar and starch." Confusion in this particular results in part from the varied interpretation given to the feeding stuff laws of different States. Each of the terms has a definite significance. The term "nitrogen-free extract" includes starch, sucrose, reducing sugars, pentosans, organic acids, coloring matter, and certain other ingredients in small quantities, and the amount of nitrogen-free extract present in a stock feed is determined by subtracting the sum of the moisture, crude fiber, protein, fat, and ash content from 100 per cent. Stock feed will not be held to be misbranded on account of statements on labels of the "nitrogen-free extract" content if analysis shows that the amount obtained by this method is correctly declared.

The term "carbohydrates" includes most of the specified ingredients which make up the nitrogen-free extract, plus crude fiber, but does not include organic acids and coloring matter. The amount of ingredients included in nitrogen-free extract which are not carbohydrates is so small in stock feeds that they may be disregarded in stating the amount of carbohydrates, and stock feeds will not be held to be misbranded on account of statements on labels of the proportion of carbohydrates if analysis shows that the percentage of carbohydrates declared equals the percentage of nitrogen-free extract obtained as indicated, plus the percentage of crude fiber.

Sugar and starch are carbohydrates and are included in determining the amount of carbohydrates present in stock feed. The term "starch and sugar," however, is properly applied only to the actual starch, sucrose, and reducing sugars contained therein, and stock feed will not be held to be misbranded on account of statements on labels of the percentage of starch and sugar, as such, if the percentage stated is the correct percentage of the amount of the starch, sucrose, and reducing sugars actually present.

This decision will go into effect January 1, 1911.

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

Board of Food and Drug Inspection.

Approved:

JAMES WILSON,

Secretary of Agriculture.

WASHINGTON, D. C., June 28, 1910.



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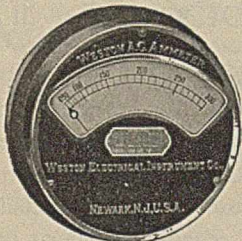
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