

NOTES.

The Qualitative Detection of Sulphur Dioxide in Linseed Oil, Etc.—A rapid and sensitive method for detecting sulphur dioxide in various oils, by means of starch iodate paper, has been used by the writer, with success for a period of several years. Starch iodate paper is made by immersing filter paper in the following solution, and drying slowly.

100 cc. of boiling distilled water, is added to two grams of starch, care being taken to prevent lumps, then 0.2 gram of potassium iodate, which has been dissolved in 5 cc. of cold distilled water is added to the starch solution.

On testing a suspected sample of linseed oil, or other oils, which are of a high boiling point, the following is the most satisfactory method of procedure.

10 cc. of the oil is put into a test tube, about 150×18 mm. and a strip of the iodate paper, which has been slightly moistened half its length, is suspended in the mouth of the test tube. The oil is gradually heated until near the boiling point, when, if sulphur dioxide is present, it will appear instantly, by the characteristic blue reaction, at the junction where the paper has been moistened.

In the case of low boiling oils, such as washed petroleum, and coal tar oils, etc., it is most suitable to distill 100 cc. in a glass distilling bulb, using a short Liebig condenser, and placing the moistened starch iodate paper at the end of the condensing tube. The distillation should not be carried on too rapidly.

In conclusion, the writer would state that very good results were obtained in determining the presence of very slight traces of sulphur dioxide, which could not be practically determined by any other method.

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Report of the Committee on Standard Methods for the Analysis of Iron.—

At the annual convention of the Association in 1905, this committee reported a method for the determination of silicon in iron, and last year added methods for determining total carbon and sulphur. The committee now adds methods for determining graphitic carbon, manganese and phosphorus, thus including all the determinations usually made on iron in which occasion for difference between the buyer and seller is apt to arise. This report will therefore include all the methods decided upon.

We would also like to call attention to the following quotation from the report of 1905, which indicates the intentions of these methods.

"In recommending the above method, it was recognized that it is almost an impossibility to get chemists to use a standard method in their daily work. Hence the above method, as recommended, is intended primarily as a check method in case of dispute between laboratories, or as between buyer and seller.

¹ From Transactions of the American Foundrymen's Association, Philadelphia Convention May 20-24, 1907.

"Hence a method accurate in every point was sought, shortness being sacrificed to some extent to insure accuracy or against the chance of error by a careless operator. Little in the above is left to the judgment of the chemist.

"It will be further recognized that in the purchase and sale of pig iron or castings under specification, that standard methods are essential in order to allow the parties of both parts to make their determinations with the assurance that, on the score of method, they are on the same footing."

We wish also to emphasize the ideas involved in the selection of members of this committee—that is to have representatives from commercial laboratories as well as works chemists, and to have members from different sections of the country. The success in carrying out these ideas will be seen by the following list of members :

Andrew A. Blair, Booth, Garrett & Blair, Philadelphia.

H. E. Diller, chemist, Western Electric Co., Chicago.

H. E. Field, metallurgical engineer, Mackintosh, Hemphill & Co., Pittsburg.

R. F. Flinterman, chief chemist, McCormick division, International Harvester Co., Chicago.

Allan P. Ford, metallurgist, Eaton, Cole & Burnham Co., Bridgeport, Conn.

J. O. Handy, chief chemist, Pittsburg Testing Laboratory, Pittsburg.

J. R. Harris, chief chemist, Tennessee Coal, Iron & Railroad Co., Birmingham, Ala.

H. C. Loudenbeck, chief chemist, Westinghouse Air Brake Co., Pittsburg.

R. S. MacPherran, chief chemist, Allis-Chalmers Co., Milwaukee.

W. G. Scott, chief chemist, J. I. Case Threshing Machine Co., Racine, Wis.

Henry Souther, Henry Souther Engineering Co., Hartford, Conn.

Prof. Thomas B. Stillman, Stevens Institute of Technology, Hoboken, N. J.

Determination of Silicon.—“Weigh one gram of sample, add 30 cc. nitric acid (1.13 sp. gr.); then 5 cc. sulphuric acid (conc.). Evaporate on hot plate until all fumes are driven off. Take up in water and boil until all ferrous sulphate is dissolved. Filter on an ashless filter, with or without suction pump, using a cone. Wash once with hot water, once with hydrochloric acid, and three or four times with hot water. Ignite, weigh, and evaporate with a few drops of sulphuric acid and 4 or 5 cc. of hydrofluoric acid. Ignite slowly and weigh. Multiply the difference in weight by 0.4702, which equals the per cent. of silicon.”

Determination of Sulphur.—Dissolve slowly a three gram sample of drillings in concentrated nitric acid in a platinum dish covered with an inverted watch glass. After the iron is completely dissolved, add two grams of potassium nitrate, evaporate to dryness and ignite over an alcohol lamp at red heat. Add 50 cc. of a one per cent. solution of sodium carbonate, boil for a few minutes, filter, using a little paper pulp in the filter if desired, and wash with a hot one per cent. sodium carbonate solution. Acidify the filtrate with hydrochloric acid, evaporate to dryness, take up with 50 cc. of water and two cc. of concentrated hydrochloric

acid, filter, wash and after diluting the filtrate to about 100 cc. boil and precipitate with barium chloride. Filter, wash well with hot water, ignite and weigh as barium sulphate, which contains 13.733 per cent. of sulphur.

Determination of Phosphorus.—Dissolve two grams samples in 50 cc. nitric acid (sp. gr. 1.13), add 10 cc. hydrochloric acid and evaporate to dryness. In case the sample contains a fairly high percentage of phosphorus it is better to use half the above quantities. Bake until free from acid, redissolving in 25 to 30 cc. of concentrated hydrochloric acid, dilute to about 60 cc., filter and wash. Evaporate to about 25 cc., add 20 cc. concentrated nitric acid, evaporate until a film begins to form, add 30 cc. of nitric acid (sp. gr. 1.20) and again evaporate until a film begins to form. Dilute to about 150 cc. with hot water and allow it to cool. When the solution is between 70° and 80° C. add 50 cc. of molybdate solution. Agitate the solution a few minutes, then filter on a tared Gooch crucible having a paper disc at the bottom. Wash three times with a three per cent. nitric acid solution and twice with alcohol. Dry at 100°-105° to constant weight. The weight multiplied by 0.0163 equals the per cent. of phosphorus in a one gram sample.

To make the molybdate solution add 100 grams molybdic acid to 250 cc. water, and to this add 150 cc. ammonia, then stir until all is dissolved and add 65 cc. nitric acid (1.42 sp. gr.). Make another solution by adding 400 cc. concentrated nitric acid to 1100 cc. water, and when the solutions are cool, pour the first slowly into the second with constant stirring and add a couple of drops of ammonium phosphate.

Determination of Manganese.—Dissolve one and one-tenth grams of drillings in 25 cc. nitric acid (1.13 sp. gr.) filter into an Erlenmeyer flask and wash with 30 cc. of the same acid. Then cool and add about one-half gram of sodium bismuthate until a permanent pink color forms. Heat until the color has disappeared, with or without the precipitation of manganese dioxide, and then add either sulphurous acid or a solution of ferrous sulphate until the solution is clear. Heat until all nitrous oxide fumes have been driven off, cool to about 15°; add an excess of sodium bismuthate—about one gram—and agitate for two or three minutes. Add 50 cc. water containing 30 cc. nitric acid to the liter, filter on an asbestos filter into an Erlenmeyer flask, and wash with 50 to 100 cc. of the nitric acid solution. Run in an excess of ferrous sulphate and titrate back with potassium permanganate solution of equal strength. Each cc. of N/10 ferrous sulphate used is equal to 0.10 per cent. of manganese.

Determination of Total Carbon.—This determination requires considerable apparatus; so in view of putting as many obstacles out of the way of its general adoption in cases of dispute your committee has left optional several points which were felt to bring no chance of error into the method.

The train shall consist of a pre-heating furnace, containing copper oxide (Option No. 1) followed by caustic potash (1.20 sp. gr.), then calcium chloride, following which shall be the combustion furnace in which either a porcelain or platinum tube may be used (Option No. 2). The tube shall contain four or five inches of copper oxide between plugs of platinum gauze, the plug to the rear of the tube to be at about the point where the tube extends from the furnace. A roll of silver foil

about two inches long shall be placed in the tube after the last plug of platinum gauze. The train after the combustion tube shall be anhydrous cupric sulphate, anhydrous cuprous chloride, calcium chloride and the absorption bulb of potassium hydroxide (sp. gr. 1.27) with prolong filled with calcium chloride. A calcium chloride tube attached to the aspirator bottle shall be connected to the prolong.

In this method a single potash bulb shall be used. A second bulb as sometimes used for a counterpoise being more liable to introduce error than to correct error in weight of the bulb in use, due to change of temperature or moisture in the atmosphere.

The operation shall be as follows: To one dram of well mixed drillings add 100 cc. of potassium chloride solution and 7.5 cc. of hydrochloric acid (conc.). As soon as dissolved as shown by the disappearance of all copper, filter on previously washed and ignited asbestos. Wash thoroughly the beaker in which the solution was made with 20 cc. of dilute hydrochloric acid (1 to 1) pour this on the filter and wash the carbon out of the beaker by means of a wash bottle containing dilute hydrochloric acid (1 to 1) and then wash with warm water until all the acid is washed out of the filter. Dry the carbon at a temperature between 95° and 100°.

Before using the apparatus a blank shall be run and if the bulb does not gain in weight more than 0.5 milligram, put the dried filter into the ignition tube and heat the preheating furnace and the part of the combustion furnace containing the copper oxide. After this is heated start the aspiration of oxygen or air at the rate of three bubbles per second, to show in the potash bulb. Continue slowly heating the combustion tube by turning on two burners at a time, and continue the combustion for 30 minutes, if air is used; 20 minutes, if oxygen is used. (The Shimer crucible is to be heated with a blast lamp for the same length of time).

When the ignition is finished, turn off the gas supply gradually so as to allow the combustion tube to cool off slowly and then shut off the oxygen supply and aspirate with air for ten minutes. Detach the potash bulb and prolong, close the ends with rubber caps and allow it to stand for five minutes, then weigh. The increase in weight multiplied by 0.27273 equals the percentage of carbon.

The potassium copper chloride shall be made by dissolving one pound of the salt in one liter of water and filtering through an asbestos filter.

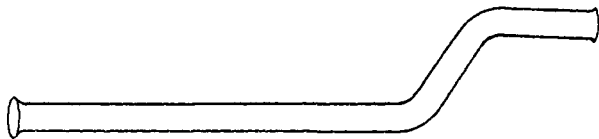
Option No. 1.—While a pre-heater is greatly to be desired, as only a small percentage of laboratories at present use them it was decided not to make the use of one essential to this method; subtraction of the weight of the blank to a great extent eliminating any error which might arise from not using a pre-heater.

Option No. 2.—The Shimer and similar crucibles are largely used as combustion furnaces and for this reason it was decided to make optional the use of either the tube furnace or one of the standard crucibles. In case the crucible is used it shall be followed by a copper tube 3-16 inch inside diameter and ten inches long, with its ends cooled by water jackets. In the center of the tube shall be placed a disk of platinum gauze, and for three or four inches in the side towards the crucible shall be silver foil and for the same distance on the other side shall be copper oxide. The ends shall be plugged with glass wool, and the tube heated with a fish tail burner before the aspiration of air is started.

Graphite.—Dissolve one gram sample in 35 cc. nitric acid (1.13 sp. gr.) filter on asbestos, wash with hot water, then potassium hydroxide (1.1 sp. gr.) and finally with hot water. The graphite is then ignited as specified in the determination of total carbon.

H. E. DILLER, Secretary.

Analysis of Fuming Sulphuric Acid.—The following method of analysis for fuming sulphuric acid has given excellent results for both speed and accuracy. Put about 15 cc. distilled water into a small Erlenmeyer flask, and then introduce a piece of 6 mm. glass tubing with a narrow constriction as shown in the cut. Allow a few drops of water to



trickle down the glass tube. Now weigh the sample in a Lunge pipette, and introduce the sample through the glass tube. The acid runs down through the tube and is absorbed by the water without spattering. Some fumes remain in the tube. By allowing a few drops of water to trickle down the tube, these fumes are completely absorbed. The analysis of one acid was checked within 0.05 per cent. ten times in succession by this method.

T. J. BREWSTER.

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NEW BOOKS

RADIOAKTIVE UMWANDLUNGEN. BY E. RUTHERFORD. Translated by M. Levin. (Die Wissenschaft, No. 21). Braunschweig: F. Vieweg and Sohn. 1907. IX. 285 pp. Unbound. Mark 8.00; Bound. Mark 8.60.

The Silliman lectures, at Yale University, for the year 1905, were delivered by Prof. Rutherford. These lectures were published, in English, under the title, *Radioactive Transformations* (New York: Charles Scribner's Sons, 1906). The present work is a translation, into German, by Dr. Max Levin. It is less comprehensive than the author's book, *Radioactivity*, the second edition of which appeared in 1905. The new work deals especially with the marvelous spontaneous transformations which are taking place continuously in radioactive matter.

After an historical introduction, in which a general resumé of the development of radioactivity is given, the transformations of the element thorium are discussed. With clear and convincing argument, the author presents the facts which lead to the conclusion that thorium is very slowly decomposing. The first product of the decomposition is radiothorium; this, in turn, yields another radioactive product, thorium X., etc. The successive changes which have so far been established are as follows: Thorium \rightarrow Radiothorium \rightarrow Th X \rightarrow Th Emanation \rightarrow Th A \rightarrow