carrying out the Blank and Finkenbeiner method has been adopted by the authors. It has been tested on a number of samples and has been found to always lead to safe, accurate results with the expenditure of a minimum amount of time and attention.

Method.—Measure out 50 cc. of normal sodium hydroxide in a small Erlenmeyer flask (say of 500 cc. capacity) and add 50 cc. of pure 3 per cent. hydrogen peroxide. Now add from a pipette 3 cc. of the formaldehyde solution under examination (the specific gravity of which has been previously determined), allowing the point of the pipette to almost reach the liquid in the flask. Place a funnel in the neck of the flask and put on the steam-bath for five minutes, shaking occasionally during this time. Remove from the steam-bath, wash the funnel with distilled water, cool the flask down to about room temperature and titrate the excess of sodium hydroxide with normal acid, using litmus as indicator. This cooling of the flask before titration with acid was found necessary so as to get a sharp end-reaction with the litmus. From the volume of formaldehyde used and the specific gravity, the percentage by weight of formaldehyde can be determined.

INNER CRUCIBLE METHOD FOR DETERMINING SULPHUR AND HALOGENS IN ORGANIC SUBSTANCES.

By S. S. Sadtler.

Received July 21, 1905.

To all workers in organic chemistry who have to make determinations of sulphur and halogens in research or commercial work, the use of the Carius furnace is almost always found troublesome. It has always been so to the writer, and after many plans to avoid its use, he devised the way to be here described.

The general idea does not seem to be a new one, as Dr. Edgar F. Smith said in discussion upon an unpublished paper read by the writer before the Philadelphia Section of this Society, that he had used an inverted inner crucible with caustic lime as a reagent to determine chlorine in organic substances when a student. More recently Shimer[1] described an inner crucible method for determining carbon in steel, etc. The writer tried inverting small platinum and porcelain crucibles in large platinum crucibles, but with sulphur compounds containing volatile constituents the oil

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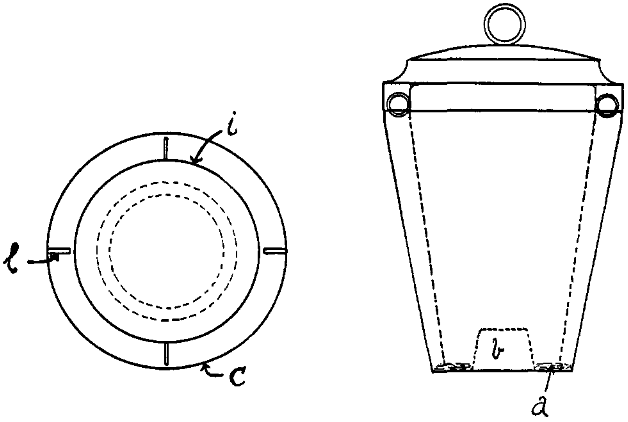
[1] *Chem. Eng.*, November, 1904.

not only began to distil before the absorbing layer of the reagent became sufficiently heated to take up the sulphur, but also came off too fast.

Successful results even with fairly volatile oils containing sulphur were finally obtained by modifying the inner crucible so as to meet the requirements. It was made at first of sections of glass combustion tubing, the idea being to keep the charge to be analyzed as far as possible from the point of application of the heat, and to have a relatively poor conductor of heat, which was readily obtained with glass. The charge was put in the rounded end, which had been drawn out to seal it, and packed with Eschka mixture. This made a fairly satisfactory apparatus for, while the absorbing mixture became red hot, the oil was only gradually vaporized. It was found, however, that the glass was acted upon by the alkaline mixture and sulphur seemed to be taken up at times in an adhering mass in one determination and given off in another. An ordinary 20-gram platinum crucible was used for the outer crucible. The inner crucible of glass, however, was not considered practicable and a cylinder of platiunm closed at the upper end was tried, and it was found that the Eschka mixture used expanded with heat and lifted the cylinder. The following apparatus was, therefore, tried after other intermediate attempts, and found to be satisfactory.



a Layer of pure asbestos.
b Indentation in outer crucible.
c Outer crucible.
l Ring-shaped lugs.
i Inner crucible.

Dimensions.
Large crucible—2 inches high (for oils, etc., low in sulphur).
Small crucible—1½ inches high (for oils, etc., containing more than 0.5 per cent. sulphur).

PLATINUM APPARATUS.

The outer crucible is made with straight sides like an inverted truncated cone with a tightly fitting lid. In the bottom is a cylindrically shaped indentation so as to extend the highly heated zone into the inner crucible.

The inner crucible was made with sides very nearly parallel to the outer crucible when inverted within it. Small rings of platinum were soldered to the wide, closed end, so as to keep it centered with respect to the outer one.

The open end is made as thin as possible so as to minimize the tendency to conduct heat. The edge fits about midway between the walls of the central indentation and the outer crucible.

PROCEDURE.

The method of procedure varies somewhat with the material for analysis. With solids careful insulation of the two crucibles is not necessary. With liquids, especially those containing sulphur, careful insulation is requisite, and with very volatile ones, such as carbon disulphide, it is necessary to fill them into small capillary bulbs such as are used for sealed tube combustions.

An important difficulty in the way of getting accurate results with this method was the puffing of the Eschka mixture, and the author found that the chemically pure light magnesia he was using contained 12 per cent. of water, and when this was driven off, and carefully dried sodium carbonate was used, that lifting the crucible and puffing ceased. The mixture was made up of equal weights of dried magnesia and sodium carbonate instead of 2 to 1.

The substance is first weighed into the inner crucible, which is placed open end up on the balance pan. It is found desirable to take the minimum weights which will give final precipitates suitable for weighing. Thus about 1 gram of sulphur compound containing 1 per cent. sulphur is taken.

An amount of a halogen compound containing about 1/20 gram of actual halogen, depending somewhat upon the one in question, and about the same amount of phosphorus-containing substance.

Enough mixture is then put in to absorb the substance, when it is a liquid, and then filled nearly to the top with moderate tamping. Plain ignited magnesia is then put in level with the top, so as to keep the soda away from the platinum, as the platinum on the bottom of the crucible is attacked by the hydrate of soda which is formed at the high temperatures or some compound with reducing gases.

Freshly ignited, white asbestos is then put around the raised portion of the bottom of the outer crucible to keep the two crucibles from being in actual contact. It is then lowered over the inner crucible and then both crucibles inverted. A layer of magnesia mixture about one-fourth inch deep is then put in between the crucibles. The outer crucible is then put through a hole in a piece of thin but firm asbestos board so that very little, if any, of the sides can be exposed to the direct action of the flame.

A very small pointed flame of a Bunsen burner is now used so that the flame is chiefly in the indentation on the bottom. If gases do not come off from the crucible in three to five minutes with this flame, the heat is increased, and when no more odor is noticeable the crucible is placed in a piece of asbestos having a larger hole, so that half of the crucible may become red hot and is kept so for ten minutes. The flame is then withdrawn, and when cool the inner crucible is carefully raised and tapped, so that its contents are dropped into the outer one, when the carbon is burned out with a shield of asbestos to protect the contents from the sulphur gases of the flame in the case of sulphur determinations.

For sulphur determinations the contents of the crucibles are washed into a beaker. Bromine is added to oxidize sulphites, etc., the solution is filtered, acidified and precipitated with barium chloride as in the case of determinations of sulphur in coal.

With halogen compounds the contents are washed into a beaker, and dissolved with pure nitric acid, the asbestos filtered off and the halogen precipitated by means of silver nitrate. The chief precaution the author has found necessary is to bring all the contents of the charge to red heat to break up any oxygen compounds of chlorine.

In the case of phosphorus the writer is undecided as to whether there is any real necessity in using this method, but as metaphosphates give off phosphine gas when heated, it seems probable that this method would be found useful. The entire contents of both crucibles must be dissolved in nitric acid and filtered, and then molybdate solution added in the usual way. On dissolving the yellow precipitate, a white crystalline precipitate ($Mg_3(PO_4)_2$) may be found, which must be redissolved in a little nitric acid and added to the ammoniacal solution to which magnesia mixture is to be added.

This method, so far as the writer has been able to investigate it, applies to all organic combinations of sulphur, the halogens and phosphorus, but the amount taken must be limited to take only a moderate final weight of barium sulphate or silver halide, such as 0.1000 to 0.2500 gram.

RESULTS OF ANALYSES.

Sample.	Carius.	Inner cone crucible.
1. Texas crude oil	1.70	1.709
2. Texas gas oil	0.40	0.39-0.41
3. Ohio crude oil	0.99	1.03-1.05
4. Ohio gas oil	0.36	0.32-0.32
5. Kansas crude oil	0.62	0.57-0.59

The writer has made a number of analyses of organic combinations of sulphur, chlorine and bromine, but owing to the incomplete character of the results at this time, he prefers to reserve the work of investigating the use of the crucible for halogen compounds for a later publication.

The author wishes to express his indebtedness to J. Bishop & Company for kindness in making four different sets of crucibles for this work.

LABORATORY OF SAMUEL P. SADTLER AND SON,
PHILADELPHIA, PA.

A SIMPLE COLORIMETER FOR GENERAL USE, WITH REMARKS ON COLORIMETRIC METHODS AND APPARATUS.[1]

BY OSWALD SCHREINER.

Received June 22, 1905.

THE USE of colorimetric methods for the estimation of small amounts of materials has been on the increase in the past few years, not only in technical analysis but also in other lines of investigation. The applications of colorimetry to the determination of ammonia, nitric and nitrous acids, has been of long standing and these tests are familiar to every chemist. They have been used by the sanitary chemist for some time, and to these has lately been added the colorimetric determination of phosphates for detecting polluted waters. The mineralogical chemist has likewise recognized in the colorimetric tests for certain metallic elements the best, and in some cases the only, method for deter-

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[1] Published by permission of the Secretary of Agriculture.