NOTES AND CORRESPONDENCE.

DETERMINATION OF TIN AND ANTIMONY IN SOFT SOLDER.

The following details for the rapid volumetric determination of tin and antimony in soft solder are adapted from A. H. Low's method.¹

Antimony.—On a counterpoised watch glass weight exactly 2 grams of filings, which should be fine enough to pass a 30-mesh sieve. With a quill brush transfer the filings through a stemless funnel into a 300 cc. Jena Erlenmeyer flask. Add 5 grams of KHSO_4 crystals and 10 cc. of sulphuric acid, specific gravity 1.8. By means of cork-lined tongs or test tube holder manipulate the flask over a bare Bunsen flame until most of the free acid is expelled and no sulphur remains on the walls of the flask nor in the liquid. Do not attempt to take to dryness. Place the hot flask on a piece of asbestos. The tin is now all a stannic and the antimony all an antimonous salt. When the flask cools sufficiently add 25 cc. of cold water and 5 cc.² of hydrochloric acid, specific gravity 1.2. Manipulate over a free flame for half a minute to complete the solution of the tin and antimony salts and to expel any sulphuric dioxide. Cool the flask under running water. Add 100 cc. of cold water and titrate rapidly with N/20 potassium permanganate. From the volume of the latter required to give the first pink color calculate the per cent. of antimony. Dry to constant weight some highest purity sodium oxalate, made according to Sorensen, and use this to standardize the N/20 potassium permanganate solution.

Tin.—Weigh exactly 0.2 gram of the filings and transfer as before to a 300 cc. Jena Erlenmeyer flask. Add 5 cc. of 15 per cent. sodium carbonate solution. Add 20 cc. of hot water. Add 25 cc. of hydrochloric acid, 1.2 specific gravity. Add one drop of 5 per cent. antimony chloride solution from a dropping bottle. This solution should be strongly acid with HCl. Close the flask with a 1-hole rubber stopper carrying a capillary U-tube of 1 mm. bore. The short arm of the U-tube should just reach through the stopper while the long arm should almost reach the surface on which the flask stands. Place the flask on a hot plate where it will boil very slowly but not suck air back through the tube. The solder will dissolve in about 15 minutes, leaving a small black precipitate of antimony. As soon as this occurs and without interrupting the slow boiling bring a test tube of 15 per cent. sodium carbonate solution under the U-tube. Carry to the sink and cool the flask under running water, allowing the carbonate solution to suck back into it. When cold add 5 cc. of 15 per cent. sodium carbonate solution and 5 cc. of cold, fresh starch liquor and titrate immediately with N/20 iodine solution. From the volume of the latter required to give the first deep blue color calculate the per cent. of tin. Standardize the N/20 iodine solution by titrating in the same way 0.1 gram portions of filings made from a stick of Kahlbaum's highest purity tin.

¹ *J. Am. Chem. Soc.*, 29, 66.

² P. H. Walker and H. A. Whitman also use less hydrochloric acid than A. H. Low, *This Journal*, 1, 519.

This method can be used to advantage for soft solder containing 0 to 2 per cent. of antimony, 30 to 60 per cent. of tin, 40 to 70 per cent. of lead and not more than traces of any other metals. Duplicate determinations of tin and antimony can be made in one hour.

J. H. GOODWIN.

THE USE OF ALUNDUM CRUCIBLES IN FATTY ACID DETERMINATIONS.

Recently the writer has had some opportunities of making separations and determining fatty acids from soaps and soap powders.

These separations were, at first, made by the usual methods, as taking up with beeswax, or upon a balanced filter.

The porous alundum crucibles, manufactured by the Norton Co., were then used as a Gooch crucible, by placing the crucible into a Gooch funnel, using a filtering bottle and a pump for suction.

The method used was by acidifying the soap solution, as usual, with an acid, allowing it to stand for some time, to give the fatty acids enough time to thoroughly coagulate. The solution was then run through this crucible, which was weighed, and the acids remaining in the crucible washed once or twice with cold water. It was then dried and weighed.

The writer finds this method to be very rapid and accurate. When the determinations are completed the fatty acids can be burned out very readily, leaving the crucible as clean as before filtering, and may be used again for a considerable number of determinations.

As the crucible is not acted upon, or contaminating the solutions with any foreign matter, but leaving it in a perfectly clear state, the filtrate passing through may be used for other determinations if so desired.

As this method is found to be more rapid and fully as accurate as the two former methods, also owing to the fact that the crucible can be used for numerous determinations, the writer can say that the use of these crucibles excel any methods, for this separation.

H. O. ANDERSON.

DISSOLVING SILICON ALLOYS.

Editor Journal of Industrial and Engineering Chemistry:

In working on some silicon alloys recently I found they may be readily decomposed by a mixture of strong hydrofluoric with hydrochloric or sulphuric acid.

The alloys so far tried include samples of 50 per cent. ferro-silicon and one of silico-spiegel (21 per cent. Si, 39 per cent. Mn and 42 per cent. Cr). The sample is placed in a large platinum crucible, conc. hydrofluoric acid added and then hydrochloric or sulphuric as desired. The solution starts at once and is complete in a few minutes at a low heat. The sample does not even have to be finely powdered for I have dissolved ordinary crushed ferro-silicon in pieces as large as 10 mesh.

For determination of iron or manganese this method is much more rapid and far simpler than either of the fusion methods.

These facts do not appear to be widely known and are not mentioned in several of the best known books on metallurgical analyses which I have consulted. The ordinary method is to fuse with sodium peroxide in a nickel crucible or with sodium carbonate and potassium nitrate in a platinum crucible. Both these methods have well-known objections which need not be discussed here.

After doing this work in looking up methods for analyses I found that the method of decomposing silicon alloys had been used by at least one concern making ferro-silicon.

But in view of the fact that this method has not been widely published I thought this note might be of interest to the readers of the Journal.

GEORGE C. DAVIS.

THE DETERMINATION OF FREE SULPHUR IN CONDIMENTAL FEEDS, STOCK TONICS AND CONDITIONERS.

In the examination of condimental feeds, stock and poultry tonics, etc., under the recently enacted Condimental Feed Law of North Carolina¹ it was found to be a very important point to know exactly how much free sulphur the various preparations contained. Accordingly, a considerable amount of time was spent in trying various methods for the determination of free sulphur, and as none of them proved entirely satisfactory for the material in hand a method was finally worked out which gave satisfactory results and answered all the requirements for this class of work.

The condimental feeds,² conditioners, etc., to which this method was applied usually consist of a base material, such as wheat bran, middlings, oil meal or cotton-seed meal to which has been added various condiments and drugs. The most common vegetable drugs are pepper, fenugreek, ginger, turmeric, gentian, and licorice. The most common mineral drugs are salt, Glauber's salt, Epsom salts, calcium carbonate, sodium bicarbonate, saltpeter, sulphur, oxide of iron and black antimony. From this it will be seen that a method for determining free sulphur in these preparations must eliminate both the combined sulphur in the base material and the vegetable drugs and the combined sulphur in the mineral drugs in the form of sulphates and sulphides. These difficulties are best overcome by dissolving out the free sulphur by carbon bisulphide, which was used in the method as finally worked out. Quite a number of methods were tried applying them to the original sample and also to the sulphur after it had been extracted with carbon bisulphide. The accuracy of the methods was tested by applying them to a wheat feed to which 10 per cent. flowers of sulphur had been added, and after the present method had been worked out further tests were made with the various methods using the results as checks.

The first method tried was the Pozzi-Escot³ method.

¹ Public Laws of North Carolina, 1909.

² A complete report of this work will be published in a bulletin of this Department at a later date.

³ This method, published in *Rev. gen. chim. Appl.*, April 7, 1904, was used by J. P. Street, New Jersey Expt. Sta. *Bull.* No. 184, and the general procedure was very kindly furnished the author in a personal communication.

This method depends on the oxidation of the sulphur by chromic acid. Unsatisfactory results were obtained when the method was tried on flowers of sulphur and a composite sample so that it was not tested further.

The Avery¹ method for determining sulphur in insecticides was tried out fully. This method consists, briefly, in putting the sulphur in solution in sodium hydroxide solution (100 grams in 100 cc. water), then oxidizing with hydrogen peroxide and precipitating the sulphates with barium chloride. When applied to the composite sample and to two stock tonics of varying composition this method gave results too low to permit its being used. When applied to the sulphur extracted from these samples by carbon bisulphide the results were nearer correct, but were still too low. In this case the low results were attributed to the error introduced by the frothing of the solution and not to incomplete oxidation.

Oxidation with bromine was then tried in a somewhat similar manner to Eschka's² method for determining sulphur in coal. The sulphur was first taken up with sodium hydroxide solution and then oxidized by treating with saturated bromine water and the sulphates precipitated with barium chloride. This gave low results both on the original sample and the sulphur extracted from the samples.

Oxidation with chlorine gas, according to the method of Rivot, Beudant and Daguin,³ when applied to the original samples gave high results. Further tests showed that the high results were caused by the oxidation of part of the sulphur of the base material.

The method as finally worked out is as follows: One gram of the sample is weighed into a side neck tube

(see figure), 50 cc. carbon bisulphide added, the tube stoppered tightly, placed in a shaking machine and shaken for five hours. The tube is then taken out of the machine and allowed to stand overnight, thus giving a perfectly clear solution. 20 cc. of the solution = 0.4 gram of the sample are then drawn off, by means of the side neck, into a flask, the carbon bisulphide evaporated off and the flask dried in a water oven. The sulphur is then taken up with sodium hydroxide solution as in the Avery method, the solution diluted and the sulphur oxidized by passing in a stream

of chlorine, the solution made acid and heated to expel chlorine, filtered and the sulphates precipitated with barium chloride. On

¹ Bureau of Chemistry, *Bull.* 90 and *Bull.* 107 (revised).

² *Z. anal. Chem.*, 13, 344. Fresenius "Quant. Anal.," Vol. II, revised, 1908.

³ Fresenius "Quant. Anal.," Vol. I, revised, 1908.