

X.—*A Colorimetric Method for the Determination of Small Percentages of Iron in Copper Alloys.*

By ARNOLD WILLIAM GREGORY, B.Sc. (Lond.).

It is often a matter of considerable importance to be able to determine accurately the amount of iron in copper alloys, on account of the effect of this element on their physical properties. The gravimetric methods are such that only by working with very large quantities of material can accurate results be obtained. Moreover, there is always a danger of iron being introduced into the solution of the alloy by the addition of large quantities of reagents which may contain a trace of

that element, and from external sources during the lengthy method of procedure.

The following method has been found to give extremely accurate results ; it is simple in operation, and very rapid in execution. It is based upon the colour reaction given by salicylic acid and ferric chloride. The violet coloration produced when salicylic acid is added to ferric chloride, although affording a delicate test for iron under properly chosen conditions, cannot be relied on for the quantitative determination of that element, since the colour is destroyed in the presence of mineral acids and also by excess of alkalis.

If, however, an excess of a solution of sodium acetate be added to a ferric salt, and then a solution of salicylic acid in acetic acid, a deep red colour is produced. Under these conditions, the depth of colour is proportional to the amount of iron present, and this method may be used for the estimation of small quantities of iron.

In the case of copper alloys, the blue or green colour of the solution entirely masks the red colour produced by the iron. This difficulty is overcome by the addition of a weak solution of potassium cyanide in quantity sufficient for the formation of the colourless, complex cyanide of copper and potassium. The red colour is unchanged by this treatment. The exact method of procedure is as follows : 0.2 gram of the alloy is dissolved in a minimum quantity of strong nitric acid. If a precipitate is produced, due to tin or antimony, the liquid is diluted slightly and filtered. Lead, if present, must be removed as sulphate.

To this solution, 20 c.c. of a concentrated solution of sodium acetate are added, and 10 c.c. of a 2 per cent. solution of salicylic acid in glacial acetic acid. A 3 per cent. solution of potassium cyanide is now added gradually until the green colour of the solution has disappeared, and the precipitate of copper cyanide is re-dissolved. The solution, which is red if iron is present, is now made up to a definite volume (depending on the intensity of the colour), and a measured amount is transferred to a Nessler comparison tube.

Into a similar tube, 20 c.c. of sodium acetate solution and 10 c.c. of the salicylic acid solution are placed, and diluted to approximately the same volume as the solution of the alloy. A standard solution of ferric chloride is added drop by drop, with constant stirring, until the colour produced is similar in intensity in the two tubes. From the amount of the standard solution used, the percentage of iron may be calculated.

By this method, it is possible to detect as little as 0.00002 gram of iron in the presence of 0.2 gram of copper.

A strong solution of potassium cyanide must on no account be used, as this gives a coloured solution with pure copper salts, especially on warming.

This test cannot be satisfactorily employed in the case of alloys containing considerable percentages of bismuth. Zinc and antimony, however, may be present without any appreciable error being introduced.

#### EXPERIMENTAL.

A solution of pure copper sulphate was made, such that 1 c.c. was equivalent to 0.02 gram of copper. Ten c.c. of this solution and 3 c.c. of concentrated nitric acid were placed in each of seven beakers, and to all but the first of these varying amounts of a standard solution of ferric chloride were added. The tests were then carried out as described above, the solutions in each case being made up to 100 c.c. The results obtained are given in the following table. The second column shows the number of c.c. of ferric chloride solution added to the copper sulphate; the third gives the percentage of iron that each quantity represents; the fourth gives the number of c.c. of ferric chloride required to produce the same colour as that obtained in each of the test experiments, and the fifth shows the percentage of iron calculated from the volume of ferric chloride added:

Number.	C.c. of $\text{FeCl}_3$ used.	Per cent. Fe.	C.c. of $\text{FeCl}_3$ required.	Per cent. Fe
1.	nil	nil	nil	nil
2.	1	0.01	1.1	0.011
3.	2	0.02	2.1	0.021
4.	5	0.05	5.2	0.052
5.	10	0.10	10.4	0.104
6.	15	0.15	15.7	0.157
7.	20	0.20	21.0	0.210

APPLEBY IRON WORKS,  
FRODINGHAM.

---