RAPID METHODS FOR THE CHEMICAL ANALYSIS OF SPECIAL STEELS, STEEL-MAKING, ALLOYS AND GRAPHITE

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RAPID METHODS

FOR THE

CHEMICAL ANALYSIS

OF

SPECIAL STEELS, STEEL-MAKING ALLOYS, AND GRAPHITE.

BY

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PREFACE.

In offering this little volume the author desires to call attention to the portions of it that he has worked out in his own way and that are, as far as he is aware, new features. (1) A qualitative test for titanium in the *presence* of *vanadium*. (2) The annealed test for chromium in steel. (3) The test for annealing in steel. (4) The pouring of the indicator into the solution when titrating for vanadium and chromium in steel, in the presence of either or both elements. (5) The determination of small amounts of copper and nickel in steel and ferro-vanadium by first separating the copper and nickel from the bulk of the iron and vanadium by means of potassium ferricyanide. (6) The EXACT determination of phosphorus in ferro-vanadium, demonstrating that as little as one-eighth of the actual phosphorus may be obtained by the ordinary processes. (7) The application of the new heating wire to a combustion tube. (8) The modified method for higher per cents of nickel. (9) The determination of silicon carbide in old plumbago crucibles and ITS EXISTENCE THEREIN. (10) The automatic laboratory still. (11) The simple laboratory method for making clay combustion boats. (12) The method for annealing Hadfield's steel. (13) The author's method for the rapid volumetric determination of manganese in the presence of iron, calcium and magnesium, by means of potassium-ferricyanide. (14) The new form of potash absorption and weighing apparatus for carbon dioxide. (15) The new form of combustion train.

The test for annealing in steel was first suggested to

the writer about ten or twelve years ago by Dr. E. S. Johnson. The author has since studied it in its application to all kinds of alloy steels.

It is the author's hope that, at least, some of the information contained in this book may prove as helpful to its readers as it has to him.

PITTSBURG, PA., July 1, 1908.

LIST OF PAPERS.

ANALYSIS OF SPECIAL STEELS, STEEL-MAKING ALLOYS, AND GRAPHITE.

CHAPTER I.

QUALITATIVE TESTS FOR CHROMIUM, TUNGSTEN, NICKEL, AND MOLYBDENUM.

DISSOLVE 0.200 gram of the sample with 5 c.c. 1 : 3 sulphuric acid in 152.4 (6 inches) by 16 mm. test tube. Also 0.200 gram of a plain carbon steel in the same way. Place the two tests in boiling water for a half hour.

The plain carbon steel will be free from black sediment and practically water white as to color. If the unknown contains as little as 0.2 or 0.3 per cent of chromium it will look distinctly greener than the known steel. Nickel also produces this effect, but the color is not so marked.

If the steel has 0.100 to 0.3 per cent of tungsten a black insoluble residue will be found in the bottom of the tube. This black sediment forms also with similar amounts of molybdenum and phosphorus. But on addition of 1 c.c. of 1.20 nitric acid to such a solution the black entirely disappears if due to the presence of the two last named elements. The black precipitate, if caused by a small quantity of tungsten, on addition of the nitric acid, changes to a yellow one. If the amount of the latter is small it is better to put the test tube back on the water bath and permit the tungstic acid to settle for two hours, when it can be seen plainly as a yellow spiral thread rising

up through the solution by giving the test tube a rotary motion. The black residue of phosphide can be recognized by filtering it out and dropping 1 : 1 hydrochloric acid on it, when the characteristic odor of phosphine is obtained. Or it can be dissolved off the filter with 1.20 nitric acid and the filtrate precipitated with molybdate solution after boiling it with a slight excess of potassium permanganate. Finish as in plain steel to get the yellow precipitate. If steels are quite high in silicon, the silicic acid and the carbon, together, produce black flakes that float about. They turn to white ones on being heated with 1.20 nitric acid on a water bath for an hour or two.

The annealed test for chromium is given under "Annealing of Steel." See page 202.

Molybdenum.

A further qualitative test for the latter element is as follows: Dissolve 0.500 gram of sample in 25 c.c. of 1 : 1 hydrochloric acid. Boil till action ceases, using a 254 by 25.4 mm. (10 by 1 inch) tube. Heat further with 2.5 grams of potassium chlorate until a clear solution has been obtained or the residue, if any, is a bright yellow. Add an equal volume of water. Filter without washing. Dissolve 10 grams of potassium hydroxide in 10 c.c. of water. Add this to the filtered solution. Boil for five minutes. Filter. Do not wash. Pour 8 c.c. of this filtrate into a 254 by 25.4 mm. tube. Add conc. hydrochloric acid until crystals form. Dilute with water to 30 c.c. Add a few grains of granulated tin. Heat to the *first* indication of boiling; remove from heat immediately and cool. Add to the cold solution 2 c.c. of potassium sulphocyanate. A light brownish red indicates 0.2 or 0.3 per cent of molybdenum. A distinct red indicates 1 to 2 per cent and a deep red higher amounts of molybdenum. This is a fine test,