International Standard 4947

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Steel and cast iron — Determination of vanadium content — Potentiometric titration method

Aciers et fontes — Dosage du vanadium — Méthode par titrage potentiométrique

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Foreword

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Steel and cast iron — Determination of vanadium content — Potentiometric titration method

1 Scope and field of application

This International Standard specifies a potentiometric titration method for the determination of vanadium in steel and cast iron.

The method is applicable to vanadium contents between 0,04 and 2 % (m/m).

2 Reference

ISO 377, *Wrought steel — Selection and preparation of samples and test pieces.*

3 Principle

Dissolution of a test portion with appropriate acids. Addition of hydrofluoric acid to keep tungsten in solution.

Oxidation of chromium and vanadium by potassium peroxydisulfate. Partial oxidation of chromium.

While checking the potential of the solution,

— reduction of chromium(VI) and vanadium(V) by ammonium iron(II) sulfate;

— oxidation of vanadium by slight excess of potassium permanganate; reduction of the excess permanganate by sodium nitrite, and reduction of the excess sodium nitrite by sulfamic acid.

Potentiometric titration of vanadium by ammonium iron(II) sulfate standard solution.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity, free from reducing or oxidizing activity.

4.1 Potassium peroxydisulfate ($K_2S_2O_8$).

4.2 Hydrochloric acid, ϱ about 1,19 g/ml.

4.3 Nitric acid, ϱ about 1,40 g/ml.

4.4 Hydrofluoric acid, ϱ about 1,15 g/ml.

4.5 Sulfuric acid, ϱ about 1,84 g/ml, diluted 1 + 4.

4.6 Sulfuric acid, ϱ about 1,84 g/ml, diluted 1 + 50.

4.7 Orthophosphoric acid, ϱ about 1,70 g/ml.

4.8 Ammonium iron(II) sulfate [$Fe(NH_4)_2(SO_4)_2{\cdot}6H_2O$], solution in sulfuric acid medium.

Dissolve 40 g of ammonium iron(II) sulfate hexahydrate in approximately 500 ml of water, add 20 ml of sulfuric acid, ϱ about 1,84 g/ml, cool, make up the volume to 1 000 ml and mix.

4.9 Potassium permanganate, 5 g/l solution.

4.10 Sodium nitrite, 3 g/l solution.

4.11 Sulfamic acid (NH_2SO_3H), 100 g/l solution.

This solution is stable for only one week.

4.12 Potassium dichromate, standard reference solution.

Weigh, to the nearest 0,001 g, approximately 1 g of potassium dichromate (the highest purity grade) previously dried at 150 °C until a constant mass is obtained on cooling in the desiccator. Introduce the weighed mass into a 250 ml beaker, dissolve it in 20 ml of water and add 160 ml of sulfuric acid (4.5). Transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, cool, dilute to the the mark with water and mix.

4.13 Ammonium iron(II) sulfate [$Fe(NH_4)_2(SO_4)_2{\cdot}6H_2O$], standard solution.

1 ml of this solution corresponds to approximately 1,275 mg of vanadium.

4.13.1 Preparation of the solution

Dissolve 10 g of ammonium iron(II) sulfate hexahydrate in approximately 500 ml of water, add 25 ml of sulfuric acid, ϱ about 1,84 g/ml, make up the volume to 1 000 ml and mix.