International Standard 4939

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Steel and cast iron — Determination of nickel content — Dimethylglyoxime spectrophotometric method

Aciers et fontes — Dosage du nickel — Méthode spectrophotométrique à la diméthylglyoxime

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4939 was prepared by Technical Committee ISO/TC 17, *Steel.*

Steel and cast iron — Determination of nickel content — Dimethylglyoxime spectrophotometric method

1 Scope and field of application

This International Standard specifies a dimethylglyoxime spectrophotometric method for the determination of nickel in steel and cast iron.

The method is applicable to nickel contents between 0,10 and 4 % (*m/m*). Cobalt, copper and manganese may cause interferences. (See the note to 7.3.2.)

2 Reference

ISO/R 377, *Selection and preparation of samples and test pieces for wrought steel.*

3 Principle

Dissolution of a test portion in hydrochloric, nitric and perchloric acids.

Formation of a coloured complex of nickel(III) with dimethylglyoxime in ammoniacal solution containing iodine and potassium iodide.

Spectrophotometric measurement at a wavelength of about 535 nm.

4 Reagents

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

4.1 Acid mixture.

Mix 2 volumes of hydrochloric acid, ϱ approximately 1,19 g/ml, 1 volume of nitric acid, ϱ approximately 1,40 g/ml, and 2 volumes of water.

4.2 Perchloric acid, ϱ approximately 1,54 g/ml.

NOTE — Perchloric acid, ϱ approximately 1,67 g/ml, may also be used. 100 ml of perchloric acid, ϱ approximately 1,54 g/ml is equivalent to 79 ml of perchloric acid, ϱ approximately 1,67 g/ml.

4.3 Ammonium citrate solution.

Dissolve 250 g of citric acid monohydrate ($C_6H_8O_7 \cdot H_2O$) in 250 ml of ammonia solution, ϱ approximately 0,91 g/ml, cool, dilute to 1 litre and mix.

4.4 Iodine solution.

Dissolve 25 g of potassium iodide and 12,7 g of iodine in the minimum volume of water. Dilute to 1 litre and mix.

4.5 Dimethylglyoxime solution.

Dissolve 1 g of dimethylglyoxime in 500 ml of ammonia solution, ϱ approximately 0,91 g/ml, dilute to 1 litre and mix.

4.6 Ammonia solution ϱ approximately 0,91 g/ml, diluted 1 + 1.

4.7 Nickel standard solution, corresponding to 0,5 g of Ni per litre.

Weigh, to the nearest 0,000 1 g, 0,500 0 g of high purity nickel and dissolve in 20 ml of nitric acid, ϱ approximately 1,40 g/ml, diluted 2 + 3. Boil to remove fumes and cool. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,5 mg of Ni.

5 Apparatus

Ordinary laboratory apparatus and

Spectrophotometer.

6 Sampling

Carry out sampling in accordance with ISO/R 377 or appropriate national standards for cast iron.