

International Standard



7525

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Nickel — Determination of sulfur content — Methylene blue molecular absorption spectrometric method after generation of hydrogen sulfide

Nickel — Dosage du soufre — Méthode spectrométrique par absorption moléculaire au bleu de méthylène après distillation d'acide sulfhydrique

First edition — 1985-12-01

UDC 669.24 : 543.422 : 543.845

Ref. No. ISO 7525-1985 (E)

Descriptors: nickel, chemical analysis, determination of content, sulfur, spectrochemical analysis.

Foreword

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International Standard ISO 7525 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

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Nickel — Determination of sulfur content — Methylene blue molecular absorption spectrometric method after generation of hydrogen sulfide

1 Scope and field of application

This International Standard specifies a molecular absorption spectrometric method after generation of hydrogen sulfide for the determination of the sulfur content of refined nickel in the range 0,000 1 to 0,002 % (*m/m*).

2 Reference

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests*.

3 Principle

Dissolution of a test portion in a nitric acid-chloric acid mixture and oxidation of total sulfur to sulfate ion. Removal of nitrates by evaporation with hydrochloric and formic acids. Reduction of sulfate to hydrogen sulfide by hydroiodic and hypophosphorous acids in an argon atmosphere.

Absorption of the hydrogen sulfide by a zinc ammine complex solution. Conversion of absorbed sulfide sulfur to methylene blue and measurement of absorbance of the solution at 665 nm. Determination of the mass of sulfur from a calibration graph.

4 Reagents and materials

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

All acids used in the dissolution of the test portion and the nitrate removal step shall be purified by distillation to eliminate sulfur-containing species, such as sulfur dioxide and sulfuric acid. Impurity levels of 1 mg of sulfur per litre are not sufficiently low. To keep the blank test sulfur content low, hydrochloric acid, in particular, should contain less than 0,05 mg of sulfur per litre.

4.1 Hydrochloric acid, $\rho_{20} = 1,18$ g/ml, diluted 1 + 4.

4.2 Purified hydrochloric acid, $\rho_{20} = 1,098$ g/ml.

Distil hydrochloric acid ($\rho_{20} = 1,18$ g/ml) diluted 3 + 2 with water using the apparatus shown in figure 1 and discard the first 10 % (*V/V*) of the distillate. Collect the remaining distillate (except for the last few millilitres) in a thoroughly cleaned glass bottle.

4.3 Purified formic acid, $\rho_{20} = 1,20$ g/ml.

Distil formic acid using the apparatus shown in figure 1 and discard the first 10 % (*V/V*) of the distillate. Collect the remaining distillate (except for the last few millilitres) in a thoroughly cleaned glass bottle.

4.4 Nitric acid-chloric acid mixture.

4.4.1 Distil nitric acid ($\rho_{20} = 1,41$ g/ml) using the apparatus shown in figure 1 and discard the first 10 % (*V/V*) of the distillate. Collect the remaining distillate (except for the last few millilitres) in a thoroughly cleaned brown glass bottle.

4.4.2 Dissolve 3 g of potassium chlorate (KClO_3) in 30 ml of water and add 100 ml of purified nitric acid (4.4.1). Prepare fresh as needed.

4.5 Zinc acetate, absorbing solution.

Dissolve 5 g of zinc acetate dihydrate [$\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$] and 70 g of ammonium chloride (NH_4Cl) in about 350 ml of water. Add 7,5 g of sodium hydroxide (NaOH), stir to dissolve, and dilute to 500 ml with water.

Store in a glass bottle.

4.6 Reducing mixture

Transfer 420 ml of hydroiodic acid [$\rho_{20} = 1,7$ g/ml, 55 % (*m/m*) HI minimum], 80 ml of hypophosphorous acid [$\rho_{20} = 1,21$ g/ml, 50 % (*m/m*) H_3PO_2 minimum] and 70 g of sodium iodide to the purifying apparatus (see figure 2). Attach the hydrogen sulfide trap containing 50 ml of the absorbing solution (4.5). Purge with argon at a flow rate of 200 to 300 ml/min for 10 min to expel air from the system. Switch on