International Standard 7527

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Nickel, ferronickel and nickel alloys — Determination of sulfur content — Iodimetric titration method after induction furnace combustion

Nickel, ferro-nickel et alliages de nickel — Dosage du soufre — Méthode par titrage iodométrique après combustion dans un four à induction

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Foreword

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International Standard ISO 7527 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

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Nickel, ferronickel and nickel alloys — Determination of sulfur content — Iodimetric titration method after induction furnace combustion

1 Scope and field of application

This International Standard specifies a titrimetric method after combustion for the determination of the sulfur content of nickel and ferronickel in the range 0,001 to 0,3 % (m/m), and of nickel alloys in the range 0,002 to 0,1 % (m/m). Examples of compositions are given in the annex.

2 References

ISO 385/1, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648, *Laboratory glassware — One-mark pipettes.*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks.*

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

ISO 7525, *Nickel — Determination of sulfur content — Methylene blue molecular absorption spectrometric method after generation of hydrogen sulfide.*

3 Principle

Combustion of a test portion in a flow of oxygen at a high temperature in a high frequency induction furnace in the presence of fluxes and accelerators.

Absorption of the sulfur dioxide formed in an acidified starch-iodide solution and continuous titration with potassium iodate standard volumetric solution.

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.

4.1 Oxygen (O_2), 99,5 % (m/m) minimum.

4.2 Ascarite or **soda lime**, 0,7 to 1,2 mm (14 to 22 mesh).

4.3 Magnesium perchlorate [$Mg(ClO_4)_2$], 0,7 to 1,2 mm (14 to 22 mesh).

4.4 Glass-wool.

4.5 Crucibles and lids.

4.5.1 Ceramic crucibles shall be of precise dimensions so that the sample is positioned correctly in the induction coil of the furnace (see 9.1).

4.5.2 Pre-ignite the crucibles in air or oxygen in a furnace for not less than 1 h at 1 100 °C and store in a desiccator or closed container. A resistance furnace with a combustion tube through which a flow of oxygen passes may be used. Crucible lids, used to help retain the solid oxidation products in the hot zone, are pre-ignited in a similar manner.

4.6 Fluxes: Low sulfur tin, copper plus tin, copper or vanadium pentoxide (see 9.2).

4.7 Accelerators: Low sulfur copper, iron, tungsten or nickel (see 9.2).

4.8 Nickel, low sulfur of known value [< 0,001 % (m/m)].

4.9 Standard reference steel, containing 0,1 to 0,2 % (m/m) sulfur.

4.10 Hydrochloric acid, ϱ_{20} = 1,19 g/ml, diluted 1 + 99.

4.11 Starch-iodide, solution.

Transfer 9 g of soluble starch to a 50 ml beaker, add 5 to 10 ml of water and stir until a smooth paste is obtained. Pour the mixture slowly into 500 ml of boiling water. Cool, add 15 g of potassium iodide and stir until it is dissolved. Dilute to 1 litre with water and mix.