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

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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INTERNATIONAL STANDARD ISO 7527-1985 (E)

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.