International Standard



INTERNATIONAL ORGANIZATION FOR STANDARDIZATION® MEX CHAPOCHAR OPPAHUSALUR TO CTAHDAPTUSALUN® ORGANISATION INTERNATIONALE DE NORMALISATION

Nickel, ferronickel and nickel alloys — Determination of sulfur content — Infra-red absorption method after induction furnace combustion

Nickel, ferro-nickel et alliages de nickel — Dosage du soufre — Méthode par absorption dans l'infrarouge après combustion dans un four à induction

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Descriptors: nickel, nickel alloys, ferronickel, chemical analysis, determination of content, sulfur.

Foreword

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

International Standard ISO 7526 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 — Infra-red absorption method after induction furnace combustion

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1 Scope and field of application

This International Standard specifies an infra-red absorption 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 0,001 to 0,1 % (m/m). Examples of compositions are given in annex A.

NOTE — It may be possible to apply this method in the range $0,000 \ 2$ to $0,001 \ \% \ (m/m)$. However, there were insufficient laboratory test data to support the inclusion of this lower level in the scope.

2 References

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.

Measurement of the sulfur dioxide formed using an infra-red analyser and an integration procedure.

4 Reagents and materials

- **4.1** Oxygen (O₂), 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 1100 $^{\circ}$ 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 nicket (see 9.2).

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

4.9 Standard reference steels, containing 0,1 to 0,2 % (*m*/*m*) suffur.

5 Apparatus 2

The apparatus required for combustion in a high frequency induction furnace and the subsequent infra-red absorption measurement of the evolved surful dioxide may be obtained commercially from a number or manufacturers. Follow the manufacturer's instructions for the operation of the equipment. A pressure regulator is required to control the oxygen pressure to the furnace according to the manufacturer's specification (usually 28 kN/m²). Features of commercial equipment are given in annex B.

6 Sampling and samples

6.1 Sampling and preparation of the laboratory sample shall be carried out by normal agreed procedures or, in case of dispute, by the relevant International Standard.