

INTERNATIONAL STANDARD

**ISO
7528**

First edition
1989-10-15

Nickel alloys — Determination of iron content — Titrimetric method with potassium dichromate

Alliages de nickel — Dosage du fer — Méthode titrimétrique avec le dichromate de potassium



Reference number
ISO 7528 : 1989 (E)



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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 7528 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

Annex A of this International Standard is for information only.

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International Organization for Standardization

Case postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Nickel alloys — Determination of iron content — Titrimetric method with potassium dichromate

1 Scope

This International Standard specifies a titrimetric method for the determination of the iron content of nickel alloys in the range 1 % (m/m) to 50 % (m/m). This method is applicable only to alloys containing less than 0,2 % (m/m) of vanadium. Typical compositions of some nickel alloys are given in annex A.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1 : 1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648 : 1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042 : 1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 5725 : 1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests*.

3 Principle

Dissolution of a test portion in a nitric/hydrochloric acid mixture and oxidation of chromium to chromium(VI) with perchloric acid.

Precipitation of iron with ammonium hydroxide and dissolution of the precipitate in hydrochloric acid.

Reprecipitation and dissolution of the iron.

Reduction of the iron(III) with a slight excess of tin(II) chloride and oxidation of the excess with mercury(II) chloride.

Addition of a sulfuric/phosphoric acid mixture and titration of the iron(II) with potassium dichromate using diphenylamine sulfonate as the indicator.

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 Ammonium hydroxide, $\rho_{20} = 0,89$ g/ml.

4.2 Ammonium hydroxide, $\rho_{20} = 0,89$ g/ml, diluted 1+2.

4.3 Hydrochloric acid, $\rho_{20} = 1,18$ g/ml.

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

4.5 Hydrochloric acid, $\rho_{20} = 1,18$ g/ml, diluted 1+50.

4.6 Perchloric acid (HClO_4), $\rho_{20} = 1,66$ g/ml.

4.7 Ammonia, wash solution.

Cautiously add 20 ml of ammonium hydroxide ($\rho_{20} = 0,89$ g/ml) to 1 litre of warm water. Prepare this solution as required.

4.8 Tin(II) chloride, solution.

Dissolve 5 g of tin(II) chloride dihydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in 10 ml of hydrochloric acid ($\rho_{20} = 1,18$ g/ml). Heat to boiling and dilute to 100 ml. Prepare this solution just before use.

NOTE — It is possible to store this solution in a brown glass bottle with the addition of granular or spongy tin metal.

4.9 Mercury(II) chloride, saturated solution.

WARNING — Mercury(II) chloride is very poisonous. Appropriate precautions shall therefore be taken in handling this chemical.

Add 60 g to 100 g of mercury(II) chloride (HgCl_2) to 400 ml of hot water, shake, and cool to ambient temperature. More water may be added from time to time as long as crystals remain undissolved.