
**Nickel alloys — Determination of
chromium content — Potentiometric
titration method with ammonium
iron(II) sulfate**

*Alliages de nickel — Détermination du chrome — Méthode par
titrage potentiométrique avec du sulfate de fer(II) et d'ammonium*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html. (standards.iteh.ai)

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

This second edition cancels and replaces the first edition (ISO 7529:1989), which has been technically revised with the following changes:

- the scope has been modified;
- [Table 1](#) has been modified.

Nickel alloys — Determination of chromium content — Potentiometric titration method with ammonium iron(II) sulfate

1 Scope

This document specifies a potentiometric titration method for the determination of chromium content in nickel alloys which do not contain insoluble carbides and which have a vanadium content less than a mass fraction of 0,2 %. The method is applicable to chromium contents between a mass fraction of 5 % to a mass fraction of 22 %.

Vanadium, which can be present as an impurity in the alloy, will give a positive bias interference. However, at a level of a mass fraction of 0,2 %, this bias is equivalent to a mass fraction of 0,068 % chromium, which is about half the reproducibility of the method.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

4 Principle

Dissolution of a test portion in a nitric/hydrochloric acids mixture, and evaporation to fumes of sulfuric acid.

Dissolution of the salts in water and oxidation of chromium to chromium(VI), with ammonium peroxydisulfate using silver nitrate as a catalyst.

Removal of excess peroxydisulfate by boiling, and reduction of manganese(VII) with hydrochloric acid.

Titration of chromium(VI) with ammonium iron(II) sulfate using potentiometric end-point detection.

5 Reagents

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

- 5.1 **Hydrochloric acid**, $\rho_{20} = 1,19$ g/ml.
- 5.2 **Hydrochloric acid**, $\rho_{20} = 1,19$ g/ml, diluted 1 + 3.
- 5.3 **Nitric acid**, $\rho_{20} = 1,41$ g/ml.
- 5.4 **Sulfuric acid**, $\rho_{20} = 1,84$ g/ml, diluted 1 + 1.
- 5.5 **Silver nitrate** (AgNO_3), 15 g/l solution.
- 5.6 **Ammonium peroxydisulfate** $[(\text{NH}_4)_2\text{S}_2\text{O}_8]$.
- 5.7 **Nitric/hydrochloric acids**, mixture.

WARNING — This acid mixture is highly corrosive and unstable. Noxious chlorine gas is liberated on standing. It shall be prepared and used in a fume hood and shall not be kept in a closed container.

Carefully mix 25 ml of nitric acid (5.3) and 75 ml of hydrochloric acid (5.1).

This mixture is not stable and shall be prepared just before use.

- 5.8 **Potassium dichromate**, standard solution, $c(1/6 \text{K}_2\text{Cr}_2\text{O}_7) = 0,100$ mol/l.

Dissolve exactly 4,903 g of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$, 99,95 % minimum purity), previously dried at 105 °C for 1 h, in 500 ml of water.

Transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

- 5.9 **Ammonium iron(II) sulfate**, standard solution, $c[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2] = 0,1$ mol/l.

5.9.1 Preparation

Dissolve 40 g of ammonium iron(II) sulfate hexahydrate $[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$ in 400 ml of water. Add slowly, with constant stirring, 100 ml of sulfuric acid (5.4).

Cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

5.9.2 Standardization

Add, using a burette, 40,0 ml of the potassium dichromate solution (5.8) to 200 ml of water in a 400 ml tall-form beaker. Add 10 ml of sulfuric acid (5.4), 5 ml of the silver nitrate solution (5.5) and 5 ml of hydrochloric acid (5.2). Titrate this solution potentiometrically with the ammonium iron(II) sulfate solution (5.9), as specified in 8.2.

The real concentration, c , of the ammonium iron(II) sulfate solution (5.9), expressed in moles of iron per litre, is given by [Formula \(1\)](#):

$$\frac{V_1}{V_2} \times 0,1 \tag{1}$$

where

V_1 is the volume, in millimetres, of the potassium dichromate solution (5.8) taken for the standardization (= 40,0 ml);

V_2 is the volume, in millimetres, of ammonium iron(II) sulfate solution (5.9), used for the titration.

6 Apparatus

Ordinary laboratory apparatus and the following.

6.1 Potentiometric titration apparatus.

6.1.1 Indicator electrode, of bright platinum, which shall be kept in a clean, highly polished condition. It shall be cleaned by dipping in nitric acid (5.3) or aqua regia and rinsed with water prior to use.

6.1.2 Reference electrode, of silver/silver chloride or calomel.

The manufacturer's instructions on the care and maintenance of these electrodes shall be followed.

6.1.3 Titration assembly, consisting of a 400 ml beaker, two 50 ml burettes complying with the requirements of ISO 385, class A, and a magnetic stirrer.

6.1.4 High impedance electric voltmeter.

Normally, a pH-meter can be used as a voltmeter. Commercial automatic titrators or potentiographs have an advantage over manual systems in that the titration curve is plotted and the end-point can be evaluated by interpolation of the curve rather than by calculation from the first or second derivative (see 10.2).

6.2 Volumetric glassware.

All volumetric glassware shall be class A in accordance with ISO 385, ISO 648 or ISO 1042, as appropriate.

7 Sampling and sample preparation

Sampling and preparation of the laboratory sample shall be carried out by normal agreed procedures or, in case of dispute, by appropriate national standards.

The laboratory sample normally is in the form of millings or drillings and no further preparation of the sample is necessary.

If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling process, it shall be cleaned with high purity acetone and then dried in air.

If the laboratory sample contains particles or pieces of widely varying sizes, the test sample should be obtained by riffing.

8 Procedure

8.1 Preparation of the test solution

8.1.1 Weigh a test portion containing between 20 mg and 80 mg of chromium according to [Table 1](#) and transfer to a 400 ml or 600 ml tall-form beaker.

Table 1 — Mass of sample to be taken

Expected chromium content % in mass fraction	Mass of sample g	Weighing accuracy g
5 to 8	0,5 to 1,0	0,001
8 to 15	0,3 to 0,5	0,000 5
15 to 22	0,2 to 0,3	0,000 2

8.1.2 Add 20 ml of the nitric/hydrochloric acids mixture ([5.7](#)) and heat until the sample is dissolved. If the alloy resists dissolution, add hydrochloric acid ([5.1](#)) in 1 ml increments and continue heating to dissolve the sample. For some alloys, an acid mixture containing 30 ml of hydrochloric acid ([5.1](#)) and 2 ml of nitric acid ([5.3](#)) is more effective.

If the laboratory sample consists of large pieces, a 1 g test portion is taken for chromium contents higher than a mass fraction of 10 %. The solution is transferred to a 100 ml one-mark volumetric flask and aliquots of 50 ml for a mass fraction of 10 % to a mass fraction of 15 % chromium or 25 ml for a mass fraction of 15 % to a mass fraction of 22 % chromium should be taken, using a pipette, and processed as in [8.1.3](#).

8.1.3 Add 20 ml of sulfuric acid ([5.4](#)) and evaporate to fumes. Cool slightly, add 100 ml of hot water and boil until residues are dissolved.

8.1.4 Dilute the solution to 200 ml, add a piece of porous porcelain and heat to boiling. Add 5 ml of the silver nitrate solution ([5.5](#)) and 5 g of ammonium peroxydisulfate ([5.6](#)). Boil gently for 15 min to oxidize the chromium completely to chromium(VI). This is indicated by a pink colour which persists for about 10 min if the manganese content of the sample is significant. For samples with a very high chromium content associated with a low manganese content, add a few drops of a potassium permanganate solution (10 g/l) to supply this indication of complete oxidation.

8.1.5 Add 5 ml of hydrochloric acid ([5.2](#)) and continue boiling for a further 5 min, when all the pink permanganate colour should disappear. If the permanganate colour persists after boiling for the further 5 min, add 5 ml more of hydrochloric acid ([5.2](#)) and boil again for 5 min. Repeat, if necessary, until the permanganate colour is completely removed.

8.1.6 Cool the solution to room temperature and titrate potentiometrically with the ammonium iron(II) sulfate solution ([5.9](#)) as specified in [8.2](#).

8.2 Determination

Place the beaker containing the test solution on the stirrer of the titration apparatus ([6.1](#)). Insert the indicator electrode ([6.1.1](#)) and reference electrode ([6.1.2](#)) and connect to the electronic voltmeter ([6.1.4](#)). Switch on the stirrer and titrate with the ammonium iron(II) solution ([5.9](#)). Add the titrant quite rapidly until the end-point is approached. Continue the titration by adding 0,1 ml or one drop increments and record the burette and potential readings when equilibrium is reached after each incremental addition. Continue the titration through the end-point. Determine the end-point by interpolation or from the titration curve (see [10.2](#)).

8.3 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all reagents as used for the determination.

8.4 Number of determination

Carry out the determination at least in duplicate.

9 Expression of results

9.1 Method of calculation

The chromium content, expressed as a percentage by mass, is given by [Formula \(2\)](#):

$$\frac{(V_3 - V_0) \times c \times 0,01733}{m} \times 100 \quad (2)$$

where

- V_0 is the volume, in millimetres, of the ammonium iron(II) sulfate solution ([5.9](#)) used for the blank test;
- V_3 is the volume, in millimetres, of the ammonium iron(II) sulfate solution ([5.9](#)) used for the determination;
- c is the real concentration, expressed in moles of $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2$ per litre, of the ammonium iron(II) sulfate solution ([5.9](#));
- 0,017 33 is the mass, in grams, of chromium corresponding to 1,00 ml of ammonium iron(II) sulfate solution, $c[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2] = 1,000 \text{ mol/l}$;
- m is the mass, in grams, of the test portion.

9.2 Precision

9.2.1 Interlaboratory tests

Fourteen laboratories in six countries participated in the interlaboratory testing of methods for the analysis of nickel alloys. Of these, 10 reported a complete set of results for this document. Six samples having the nominal composition given in [Table 2](#) were analysed in duplicate on different days.

Table 2 — Nominal composition of test samples (% in mass fraction)

Sample label	Al	Co	Cr	Cu	Fe	Mn	Ni	Si	Ti
825	0,2	0,07	21	1,6	30	0,7	Remainder	0,4	1,1
902	0,4	0,05	5	0,04	48	0,4	Remainder	0,35	2,5
3920	0,15	2	19	0,1	3	0,3	Remainder	0,6	2,3
3927	0,1	1	20	0,05	44	0,4	Remainder	0,8	0,6
7013	1,5	17	20	0,2	0,2	0,05	Remainder	0,7	2,4
7049	1	0,01	15	0,15	7	0,8	Remainder	0,3	2,3