
International Standard



6351

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Nickel — Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents — Flame atomic absorption spectrometric method

Nickel — Dosage de l'argent, du bismuth, du cadmium, du cobalt, du cuivre, du fer, du manganèse, du plomb et du zinc — Méthode par spectrométrie d'absorption atomique dans la flamme

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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.

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 6351 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.

Nickel — Determination of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents — Flame atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead and zinc contents of refined, wrought and cast nickel within the ranges specified in table 1. This method is applicable to the independent determination of any one or more of the elements listed without including all the elements specified in the standard solutions.

Table 1 — Concentration ranges of elements to be determined

Element	Concentration range, [% (m/m)]*	
	Procedure A	Procedure B
Ag	0,000 2 to 0,01	—
Bi	0,001 0 to 0,01	—
Cd	0,000 2 to 0,002 5	—
Co	0,001 0 to 0,01	0,01 to 1,00
Cu	0,000 2 to 0,01	0,01 to 1,00
Fe	0,002 5 to 0,01	0,01 to 0,15
Mn	0,000 5 to 0,01	0,01 to 0,20
Pb	0,000 5 to 0,01	—
Zn	0,000 2 to 0,002 5	0,00 1 to 0,01 5

* For specific compositions, see ISO 6283.

The lower level for iron can be extended to less than 0,002 5 % (m/m) provided nickel containing less than 0,000 1 % (m/m) iron is used for preparation of standard solutions; see 4.1.

The upper limit for the determination of cobalt and copper can be raised to 2 % (m/m) by a minor modification to the method; see 11.1.

For potential interferences, see clause 10.

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*.

3 Principle

Dissolution of a test portion in nitric acid diluted (1+1), evaporation of excess acid and dilution of the solution to a known volume.

Aspiration of the solution into the air-acetylene flame of an atomic absorption spectrometer.

Measurement of the absorption of the resonance line energy from the spectrum of each element and comparison with that of calibration solutions of the same element in a matched nickel matrix.

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 Nickel powder, high purity containing less than 0,000 5 % (m/m) iron and less than 0,000 1 % (m/m) each of silver, bismuth, cadmium, cobalt, copper, manganese, lead and zinc.

4.2 Nitric acid (HNO₃) $\rho_{20} = 1,41$ g/ml, diluted 1+1.

The same batch of nitric acid shall be used throughout the procedure.

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The same batch of nitric acid shall be used throughout the procedure.

4.4 Mixed analyte standard solutions.

4.4.1 Metal stock standard solutions, corresponding to 1,000 g of Ag, Bi, Cd, Co, Cu, Fe, Mn, Pb, or Zn per litre.

Prepare separately for each metal of interest.

Weigh to the nearest 0,001 g, 1,00 g of silver, bismuth, cadmium, cobalt, copper, iron, manganese, lead, or zinc [at least 99,9 % (*m/m*) pure], transfer to a 600 ml beaker and dissolve in 40 ml of nitric acid (4.2). Heat to complete dissolution, boil gently to expel oxides of nitrogen, cool and transfer to a 1 000 ml one-mark volumetric flask containing 160 ml of nitric acid (4.2). Make up to the mark with water and mix.

Store in polyethylene bottles, except for silver which is stored in glass.

4.4.2 Mixed analyte standard solution A, corresponding to 20 mg each of Ag, Bi, Cd, Co, Cu, Fe, Mn, Pb per litre and 10 mg of Zn per litre.

Pipette 20,0 ml each of the silver, bismuth, cadmium, cobalt, copper, iron, manganese and lead stock standard solutions (4.4.1) and 10 ml of the zinc stock standard solution (4.4.1) into a 1 000 ml one-mark volumetric flask containing 160 ml of nitric acid (4.2). Make up to the mark with water and mix.

Store in a glass bottle.

4.4.3 Mixed analyte standard solution B, corresponding to 100 mg each of Co, Cu, Fe, Mn per litre and 10 mg of Zn per litre.

Pipette 50,0 ml each of the cobalt, copper, iron and manganese stock standard solutions (4.4.1) and 5,0 ml of the zinc stock standard solution (4.4.1) into a 500 ml one-mark volumetric flask. Make up to the mark with water and mix.

Store in a polyethylene bottle.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Atomic absorption spectrometer.

5.1.1 The atomic absorption spectrometer used in this method shall meet the instrument performance parameters given in annex A.

5.1.2 The instrument shall be equipped with a burner head capable of accepting a solution containing 25 g of Ni as nitrate per litre and suitable for an air-acetylene flame.

5.1.3 The instrument should be capable of using single element hollow cathode or electrodeless discharge lamps operated at currents recommended by the instrument manufacturer.

5.2 Burette, of capacity 50 ml graduated in divisions of 0,1 ml in accordance with ISO 385/1, class A.

5.3 Pipettes, of capacities 5; 20; 25; 50; and 100 ml, in accordance with ISO 648, class A.

5.4 Volumetric flasks, of capacities 200; 250; 500; and 1000 ml, in accordance with ISO 1042, class A.

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.

6.2 The laboratory sample normally is in the form of a powder, granules, millings or drillings and no further preparation of the sample is necessary.

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

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

7 Procedure A

This procedure is applicable to 0,000 5 % (*m/m*) to 0,01 % (*m/m*) of silver, bismuth, cadmium, cobalt, copper, iron, manganese and lead, and 0,000 5 % to 0,005 % (*m/m*) zinc.

7.1 Preparation of test solution

Weigh, to the nearest 0,01 g, 4,9 to 5,1 g of the laboratory sample and transfer to a clean unetched 600 ml beaker. Add sufficient water to cover the test portion and dissolve by adding 60 ml of nitric acid (4.2) in small portions. Heat to complete dissolution, boil gently to expel oxides of nitrogen and evaporate to a viscous syrup. Redissolve the salts by adding 20 ml of nitric acid (4.2) and 100 ml of water. Heat to complete dissolution, cool and filter, if necessary, through glass-wool washed with nitric acid (4.2) or a cellulose filter into a 200 ml one-mark volumetric flask. Wash the filter with water. Make up to the mark with water and mix.

If sample inhomogeneity is suspected, a larger test portion, 10 to 50 g, may be taken for analysis. However, an aliquot portion to correspond to a 5 g test portion shall be taken from such a solution and processed in accordance with the procedure given. See 11.2.

7.2 Blank test

The zero member of the set of calibration solutions A (7.3) serves as the blank test since the same batch of nitric acid is used for dissolution of both the nickel reference and test samples.

If it is impossible to use the same batch of nitric acid a second blank test shall be prepared using the same nickel powder (4.1). This blank is then compared with the zero member and an appropriate correction made.

7.3 Preparation of the set of calibration solutions A

7.3.1 This set corresponds to 0; 0,2; 0,5; 1,0; 1,5; 2,0; and 2,5 mg each of Ag, Bi, Cd, Co, Cu, Fe, Mn and Pb per litre and 0; 0,1; 0,25; 0,5; 0,75; 1,0; and 1,25 mg of Zn per litre (see table 2). All the solutions contain a matrix of 25 g of Ni per litre.