
**Nickel alloys — Determination of
molybdenum — Inductively coupled
plasma atomic emission spectrometric
method**

*Alliages de nickel — Détermination du molybdène — Méthode par
spectrométrie d'émission atomique avec source à plasma induit par
haute fréquence*

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ISO 11435:2005

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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.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 11435 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*, Subcommittee SC 3, *Analysis of nickel, ferronickel and nickel alloys*.

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Nickel alloys — Determination of molybdenum — Inductively coupled plasma atomic emission spectrometric method

1 Scope

This International Standard specifies an inductively coupled plasma atomic emission spectrometric method for the determination of the mass fraction of molybdenum between 0,05 % and 20 % in nickel alloys.

2 Normative references

The following referenced documents are indispensable for the application 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 648:1977, *Laboratory glassware — One-mark pipettes*

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

3 Principle

Dissolution of a test portion in a mixture of hydrochloric, nitric and phosphoric acid and fuming with a mixture of phosphoric and perchloric acids. Addition of hydrofluoric acid and, if desired, of an internal reference element and dilution of the solution to known volume. Nebulization of the solution into an inductively coupled plasma atomic emission spectrometer and measurement of the intensity of the emitted light from molybdenum, and eventually from the internal reference element, simultaneously.

Examples of the analytical lines for molybdenum are given in Table 1.

Table 1 — Examples of analytical lines together with interfering elements

Element	Analytical line nm	Interferences
Molybdenum	202,03	Ta
	281,61	Al, Hf

The method uses a calibration based on a very close matrix-matching of the calibration solutions to the sample and bracketing of the contents between 0,75 and 1,25 of the approximate concentration of molybdenum in the sample to be analysed. The concentration of all elements in the sample has, therefore, to be approximately known. If the concentrations are not known, the sample has to be analysed by some semi-quantitative method. The advantage with this procedure is that all possible interferences from the matrix will be automatically compensated, which will result in high accuracy. This is most important for spectral interferences, which can be severe in very highly alloyed metals. All possible interferences shall be kept at a minimum level. Therefore, it is essential that the spectrometer used meets the performance criteria specified in the method for the selected analytical lines.

Two lines have been carefully investigated (see Annex B). The strongest possible interferences are given in Table 1. If other lines are used, they shall be carefully checked so that interferences are not higher than the values given in Annex B. The analytical line for the internal standard should be selected carefully. It is recommended to use Scandium 363.07 nm. This line is interference-free for the elements and concentrations generally found in nickel alloys.

NOTE The use of an internal standard is not essential since no relevant differences between laboratories operating with or without internal standards were found.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognised analytical grade and only grade 2 water as specified in ISO 3696:1987.

4.1 Hydrofluoric acid, HF, 40 % (mass fraction), $\rho = 1,14$ g/ml, or 50 % (mass fraction), $\rho = 1,17$ g/ml.

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes producing severe skin burns which are slow to heal. In the case of contact with skin, wash well with water, apply a topical gel containing 2,5 % (mass fraction) calcium gluconate, and seek immediate medical treatment.

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4.2 Hydrochloric acid, HCl, $\rho = 1,19$ g/ml.

4.3 Nitric acid, HNO₃, $\rho = 1,40$ g/ml.

4.4 Phosphoric acid, H₃PO₄, $\rho = 1,70$ g/ml.

4.5 Perchloric acid, HClO₄, 60 % (mass fraction), $\rho = 1,54$ g/ml or 70 %, $\rho = 1,67$ g/ml.

4.6 Internal standard solution, 100 mg/l.

Choose a suitable element to be added as internal reference and prepare a 100 mg/l solution.

4.7 Molybdenum standard solution, 10 g/l.

Weigh, to the nearest 0,000 5 g, 1 g of high-purity molybdenum [min 99,95 % (mass fraction)], place it in a 250 ml beaker and dissolve in a mixture of 10 ml hydrochloric acid (4.2) and 10 ml nitric acid (4.3).

Cool and transfer to a calibrated 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

NOTE 1 ml of this solution contains 10 mg of molybdenum.

4.8 Molybdenum standard solution, 1 g/l.

Using a calibrated pipette (or burette), transfer 10 ml of the molybdenum standard solution (4.7) into a calibrated 100 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.2). Dilute to the mark with water and mix.

NOTE 1 ml of this solution contains 1 mg of molybdenum.

4.9 Molybdenum standard solution, 100 mg/l.

Using a calibrated pipette (or burette), transfer 10 ml of the molybdenum standard solution (4.8) into a calibrated 100 ml one-mark volumetric flask. Add 10 ml of hydrochloric acid (4.2). Dilute to the mark with water and mix.

NOTE 1 ml of this solution contains 0,1 mg of molybdenum

4.10 Standard solutions of interfering elements.

Prepare standard solutions for each element whose mass fraction is higher than 1 % of that contained in the test sample. Use pure metal or chemical substances with molybdenum contents less than 10 µg/g.

5 Apparatus

All volumetric glassware shall be class A and calibrated in accordance with ISO 648:1977 or ISO 1042:1998, as appropriate.

Ordinary laboratory apparatus and

5.1 Polytetrafluoroethylene (PTFE) beakers

5.2 Polypropylene volumetric flasks, of capacity 100 ml, calibrated according to ISO 1042.

5.3 Atomic emission (AES)

The spectrometer shall be equipped with an inductively coupled plasma (ICP) and a nebulization system resistant to hydrofluoric acid. The ICP-AES used will be satisfactory if, after optimising according to 7.3, it meets the following performance criteria.

The spectrometer can be either the simultaneous or the sequential type. If a sequential spectrometer can be equipped with an extra arrangement for simultaneous measurement of the internal standard line, it can be used with the internal reference technique. If the sequential spectrometer is not equipped with this arrangement, an internal reference cannot be used and an alternative technique without an internal standard should be applied.

5.3.1 Practical resolution of the sequential spectrometer

Calculate the bandwidth (full width at half maximum), according to A.2, for the analytical line used including the line for internal reference. The bandwidth shall be less than 0,030 nm.

5.3.2 Short-term stability

Calculate the standard deviation of ten measurements of the absolute intensity or intensity ratio of the emitted light of the most concentrated calibration solution for molybdenum according to A.3. The relative standard deviation shall not exceed 0,4 %.

5.3.3 Background equivalent concentration

Calculate the background equivalent concentration (BEC) according to A.4, for the analytical line using a solution containing only the analyte element. The maximum values of BEC obtained should be 0,4 mg/l.

6 Sampling and sample preparation

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 is normally in the form of millings or drillings and no further mechanical preparation is necessary.

6.3 The laboratory sample shall be cleaned by washing with pure acetone and drying in air.

6.4 If brazed alloy tools are used in the preparation of the laboratory sample then the sample shall be further cleaned by pickling in 15 % (mass fraction) nitric acid for a few minutes. It shall then be washed several times with distilled water, followed by washing in acetone and drying in air.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,000 5 g, 0,25 g of the test sample.

7.2 Preparation of test solution T_{Mo}

7.2.1 Place the test portion in a 200 ml Pyrex Erlenmeyer flask.

NOTE A PTFE or PFA beaker with a graphite base and a cover can also be used. In this case, the solution does not need to be transferred to another PTFE beaker after fuming (7.2.2).

7.2.2 Add 30 ml of HCl (4.2), 3 ml of HNO₃ (4.3) and 2,5 ml of H₃PO₄ (4.4). Let the dissolution begin at room temperature. If necessary, heat to complete dissolution. Add 7,5 ml of HClO₄ (4.5) and heat until the perchloric acid starts to fume. Continue to fume for two to three minutes (the white smoke shall be on the top of the Erlenmeyer).

In the case where a complete dissolution is difficult to obtain, 5 ml of HF (4.1) may be added together with the HCl (4.2), HNO₃ (4.3) and H₃PO₄ (4.4). In this case, a PTFE or PFA beaker should be used.

7.2.3 Cool the solution and add 10 ml of water to dissolve the salts. Some residues can remain undissolved. Transfer the solution and possible undissolved residues quantitatively to a 100 ml PTFE beaker. Add 2 ml of HF (4.1). Heat gently for 20 min until the residues dissolve completely.

7.2.4 Cool the solution to room temperature and transfer the solution quantitatively to a 100 ml volumetric polypropylene flask (5.2). If an internal standard is used, add an adapted quantity with a calibrated pipette (or burette), for example 10 ml of the internal standard solution (4.6).

7.2.5 Dilute to the mark with water and mix.

7.3 Optimisation of spectrometer

7.3.1 Start the ICP-AES and let it run for at least 30 min before taking any measurements.

7.3.2 Optimise the instrument according to the manufacturer's instructions.

7.3.3 Prepare the software to measure the intensity, mean value and relative standard deviation of the analytical lines.

7.3.4 If an internal standard is used, prepare the software to calculate the ratio between analyte intensity and internal standard intensity. The intensity of the internal standard shall be measured simultaneously with the analyte intensity.

7.3.5 Check the instrument performance requirements given in 5.3.1 to 5.3.3.

7.4 Pre-determination of the test solution

Prepare a calibration solution K_{20} , corresponding to a molybdenum concentration of 20 % (mass fraction) and matrix-matched to the test sample solution as follows.

7.4.1 Using a calibrated pipette (or burette), add 5 ml of the molybdenum standard solution (4.7) to a 100 ml volumetric polypropylene flask (5.2) marked K_{20} .

7.4.2 In this volumetric flask K_{20} , add volumes of standard solutions (4.10) necessary to match the sample matrix to be tested, for each element whose concentration is above 1 %.

The matrix should be matched to the nearest percent.

7.4.3 Add 2,5 ml of H_3PO_4 (4.4), 7,5 ml of $HClO_4$ (4.5) and 10 ml of the internal standard solution (4.6). Dilute with water and mix.

7.4.4 Also prepare a blank calibration solution, K_0 , prepared in the same way as the calibration solution K_{20} omitting molybdenum.

7.4.5 Measure the absolute intensities I_0 and I_{20} for the solutions K_0 and K_{20} .

7.4.6 Measure the absolute intensity $I_{T_{Mo}}$ for the test solution T_{Mo} .

7.4.7 Calculate the approximate concentration of molybdenum, $K_{T_{Mo}}$, in % (mass fraction), in the test solution by means of the following formula:

$$K_{T_n} = \frac{I_{T_{Mo}} (K_{20} - K_0)}{I_{20} - I_0}$$

7.5 Preparation of calibration solutions for bracketing, $K_{l,Mo}$ and $K_{h,Mo}$

For each test solution T_{Mo} prepare two matrix-matched calibration solutions, $K_{l,Mo}$ and $K_{h,Mo}$ with molybdenum concentrations in $K_{l,Mo}$ slightly below and in $K_{h,Mo}$ slightly above the concentration in the unknown test solution as follows.

7.5.1 Using calibrated pipettes (or burette), add molybdenum standard solution (4.8) or (4.9) to one 200 ml Pyrex Erlenmeyer marked $K_{l,Mo}$ so that the molybdenum content $K_{l,Mo}$, in %, is approximately $K_{T_{Mo}} \times 0,75 < K_{l,Mo} < K_{T_{Mo}} \times 0,95$. If you use a pipette, select $K_{l,Mo}$ in such a way as to take a volume easily.

7.5.2 Using calibrated pipettes (or burettes), add molybdenum standard solution (4.8) or (4.9) to one 200 ml Pyrex Erlenmeyer marked $K_{h,Mo}$ so that the molybdenum content $K_{h,Mo}$ in %, is approximately $K_{T_{Mo}} \times 1,05 < K_{h,Mo} < K_{T_{Mo}} \times 1,25$. If you use a pipette, select $K_{h,Mo}$ in such a way as to take a volume easily.

7.5.3 Add to the calibration solutions $K_{l,Mo}$ and $K_{h,Mo}$ all matrix elements whose mass fractions are above 1 % in the test sample, using the appropriate amount of standard solutions 4.10 to match the equivalent matrix concentration to the nearest %.

7.5.4 Proceed as directed in 7.2.2 to 7.2.5.