

praktijkrichtlijn

NPR-ISO/TS 13899-3 (en)

Steel - Determination of Mo, Nb and W contents in alloyed steel - Inductively coupled plasma atomic emission spectrometric method - Part 3: Determination of W content (ISO/TS 13899-3:2005, IDT)

maart 2005
ICS 77.080.20

Als Nederlandse praktijkrichtlijn is aanvaard:

- ISO/TS 13899-3:2005, IDT

Normcommissie 342 093 "Chemische analyse van metalen"

Apart from exceptions provided by the law, nothing from this publication may be duplicated and/or published by means of photocopy, microfilm, storage in computer files or otherwise, which also applies to full or partial processing, without the written consent of the Netherlands Standardization Institute.

The Netherlands Standardization Institute shall, with the exclusion of any other beneficiary, collect payments owed by third parties for duplication and/or act in and out of law, where this authority is not transferred or falls by right to the Reproduction Rights Foundation.

Auteursrecht voorbehouden. Behoudens uitzondering door de wet gesteld mag zonder schriftelijke toestemming van het Nederlands Normalisatie-instituut niets uit deze uitgave worden veeleenvoudigd en/of openbaar gemaakt door middel van fotokopie, microfilm, opslag in computerbestanden of anderszins, hetgeen ook van toepassing is op gehele of gedeeltelijke bewerking.

Het Nederlands Normalisatie-instituut is met uitsluiting van ieder ander gerechtigd de door derden verschuldigde vergoedingen voor veeleenvoudiging te innen en/of daartoe in en buiten rechte op te treden, voor zover deze bevoegdheid niet is overgedragen c.q. rechtens toekomt aan de Stichting Reprorecht.

Although the utmost care has been taken with this publication, errors and omissions cannot be entirely excluded. The Netherlands Standardization Institute and/or the members of the committees therefore accept no liability, not even for direct or indirect damage, occurring due to or in relation with the application of publications issued by the Netherlands Standardization Institute.

Hoewel bij deze uitgave de uiterste zorg is nagestreefd, kunnen fouten en onvolledigheden niet geheel worden uitgesloten. Het Nederlands Normalisatie-instituut en/of de leden van de commissies aanvaarden derhalve geen enkele aansprakelijkheid, ook niet voor directe of indirecte schade, ontstaan door of verband houdend met toepassing van door het Nederlands Normalisatie-instituut gepubliceerde uitgaven.

**Steel — Determination of Mo, Nb and W
contents in alloyed steel — Inductively
coupled plasma atomic emission
spectrometric method —**

**Part 3:
Determination of W content**

*Aciers — Dosage du Mo, du Nb et du W dans les aciers alliés —
Méthode par spectrométrie d'émission atomique avec plasma induit par
haute fréquence —
Partie 3: Dosage du W*



PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

Copyright
Preview

© ISO 2005

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword.....	iv
1 Scope.....	1
2 Normative references	1
3 Principle.....	1
4 Reagents.....	2
5 Apparatus.....	3
6 Sampling and samples	4
7 Procedure.....	4
7.1 Preparation of test solution, T_n	4
7.2 Preparation for spectrometric measurements.....	5
7.3 Pre-analysis of the test solution.....	5
7.4 Preparation of calibration solutions for bracketing, K_{Ln} and K_{Hn}	6
7.5 Analysis of test solutions	6
8 Expression of results.....	6
8.1 Method of calculation	6
8.2 Precision	6
9 Test report	7
Annex A (normative) Procedure for the determination of instrumental criteria.....	8
Annex B (informative) Additional information on the international collaborative trial	10
Annex C (informative) Graphical representation of precision data.....	12

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

- an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;
- an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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/TS 13899-3 was prepared by Technical Committee ISO/TC 17, Steel, Subcommittee SC 1, *Methods of determination of chemical composition*.

ISO/TS 13899 consists of the following parts, under the general title *Steel — Determination of Mo, Nb and W contents in alloyed steel — Inductively coupled plasma atomic emission spectrometric method*:

- *Part 1: Determination of Mo content*
- *Part 2: Determination of Nb content*
- *Part 3: Determination of W content*

Steel — Determination of Mo, Nb and W contents in alloyed steel — Inductively coupled plasma atomic emission spectrometric method —

Part 3: Determination of W content

1 Scope

This Technical Specification specifies a method for the determination of tungsten content in steel by means of inductively coupled plasma atomic emission spectrometry.

This method is applicable to tungsten contents between 0,1 % and 20 % (mass fraction).

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*

ISO 14284:1996, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

3 Principle

The sample is dissolved in a hydrochloric, nitric and hydrofluoric acid mixture and fumed with a phosphoric and perchloric acid mixture. Hydrofluoric acid and an internal standard element (if used) are added and the solution is diluted to known volume. The solution is filtered and nebulized into an inductively coupled plasma (ICP) spectrometer and the intensity of the emitted light from the element is measured simultaneously with the light emitted from the internal standard element.

The method uses a calibration based on close matrix matching of the calibration solutions to the sample and close bracketing of the tungsten content around the approximate concentration of tungsten in the sample to be analysed. This compensates for matrix interferences and provides high accuracy, even in highly alloyed steels where spectral interferences can be severe. Nonetheless, all interferences shall be kept to a minimum and it is therefore essential that the spectrometer used meets the performance criteria specified in the method for the selected analytical lines.

In order to accurately matrix match, it is necessary to know the content of all elements in the sample (to the nearest %). To this end, it may be necessary to carry out a preliminary analysis of the sample by a semi-quantitative method.

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.

4.1 Hydrofluoric acid, HF, 40% (mass fraction), ρ approximately 1,14 g/ml

4.2 Hydrochloric acid, HCl, ρ approximately 1,19 g/ml

4.3 Nitric acid, HNO₃, ρ approximately 1,40 g/ml

4.4 Phosphoric acid, H₃PO₄, ρ approximately 1,70 g/ml, diluted 1 + 1

4.5 Perchloric acid, HClO₄, ρ approximately 1,54 g/ml, diluted 1 + 1

4.6 Fuming acid mixture: mix 100 ml of the phosphoric acid (4.4) and 300 ml of the perchloric acid (4.5)

4.7 Internal standard solution, 1 000 mg/l

Choose a suitable element to be added as internal standard and prepare a 1 000 mg/l solution. The internal standard chosen

- shall be pure,
- shall not be present in the sample, and
- shall not interfere with analytical wavelengths, nor should the internal standard element wavelength be interfered with by elements in the test solution.

The internal standard shall be soluble in the acids used and it shall not cause precipitation. Moreover, the excitation conditions of the analytical line and the internal standard element line should match.

4.8 Tungsten stock standard solution, 1 000 mg/l

Weigh, to the nearest 0,001 g, 0,5 g of high purity tungsten [minimum 99,95 % (mass fraction)] and dissolve in a mixture of 10 ml hydrofluoric acid (4.1) and 20 ml nitric acid (4.3). Cool and transfer the solution quantitatively to a calibrated 500 ml one-mark plastic volumetric flask. Dilute to the mark with water and mix. 1 ml of this solution contains 1 mg of W.

It is not permitted to use pre-prepared tungsten standard solutions supplied by second parties.

4.9 Tungsten standard solution, 100 mg/l

Transfer, with a calibrated micropipette, 25 ml of the tungsten stock standard solution (4.8) into a calibrated 250 ml one-mark plastic volumetric flask. Add 5 ml of hydrofluoric acid (4.1) and 10 ml of nitric acid (4.3). Dilute to the mark with water and mix. 1 ml of this solution contains 0,1 mg of W.

NOTE If calibrated micropipettes of suitable volume are not available it would be more advantageous to use more concentrated standard solutions. These solutions must, however, be prepared for each series of analysis, in order to avoid possible precipitation.

4.10 Standard solutions of interfering and matrix elements

Prepare standard solutions for each element of which more than 1 % (mass fraction) is contained in the test sample. Use pure elements or oxides with tungsten contents less than 10 µg/g. Commercial certified standard solutions can also be used if the tungsten content is less than the value specified above.

NOTE If a large amount of an element is to be added (e.g. iron), it might be more advantageous to use the pure metal and weigh the correct amount (see 7.3 and 7.4). In this case, use the dissolution procedure described in 7.1.2.

5 Apparatus

The plastic pipettes and flasks to be used shall be calibrated in accordance with ISO 648 or ISO 1042, as appropriate.

Ordinary laboratory apparatus and

5.1 Atomic emission spectrometer, equipped with an inductively coupled plasma (ICP) and a nebulization system resistant to hydrofluoric acid.

When a polytetrafluoroethylene (PTFE) nebulizer is used, it is recommended that a surface active agent should be added to improve wetting in the nebulizer and spray chamber. Modern nebulizers are, however, often manufactured of plastic materials with better wetting-characteristics than PTFE and can therefore (as is the case with sapphire nebulizers) be used without a surface active agent.

The ICP-ES spectrometer used will be satisfactory if, after optimising according to 7.2.1 to 7.2.4, it meets the performance criteria given in 5.1.2 to 5.1.4.

The spectrometer can be either the simultaneous or the sequential type. A sequential type can be used either with or without an internal standard. However, if a sequential spectrometer is to be used with an internal standard, it shall be fitted with an arrangement which allows for the simultaneous measurement of the internal standard line.

5.1.1 Analytical lines

This standard does not specify any particular emission line. It is mandatory that each laboratory carefully investigates the line/lines available on its own equipment to find the most suitable one regarding sensitivity and freedom from interferences.

In Table 1, however, a suggestion is given together with possible interferences.

The line for the internal standard element should be selected according to 4.7.

Table 1 — Example of analytical line and lines for internal standard element

Element	Wavelength nm	Possible interferences
W	207,91	Cr, V, Mo
Sr (as internal standard)	407,77	
Sc (as internal standard)	363,07	
Cd (as internal standard)	214,44	

5.1.2 Minimum practical resolution of the spectrometer

Calculate the bandwidth, according to Clause A.1, for the wavelength used including the line for internal standard. The bandwidth shall be less than 0,030 nm.

5.1.3 Minimum short-term precision

Calculate the short-term precision according to Clause A.2. The relative standard deviation shall not exceed 0,5 % of the mean absolute or ratioed intensities for concentrations 100 to 1 000 times the LOD (5.1.4) mg/l. For concentrations 10 to 100 times the LOD the RSD shall not exceed 5 %.

5.1.4 Limit of Detection (LOD) and Limit of Quantification (LOQ)

Calculate the LOD and LOQ, according to Clause A.3, for the analytical line used. The values shall be below the values in Table 2.

Table 2 — Limit of Detection (LOD) and Limit of Quantification (LOQ)

Element	LOD mg/l	LOQ mg/l
W	1	5

5.2 Polytetrafluoroethylene (PTFE) beakers

5.3 100 ml polypropylene volumetric flasks

6 Sampling and samples

Sampling and preparation of the laboratory sample shall be carried out in accordance with ISO 14284 or appropriate national standards for steel.

7 Procedure

7.1 Preparation of test solution, T_n

7.1.1 Weigh, to the nearest 0,000 5 g, a test portion of the laboratory sample in accordance with Table 3, and transfer it to a PTFE beaker.

Table 3 — Test portion

Expected content % (mass fraction)	Test portion g
0,1 to 5	0,5
5 to 20	0,1

7.1.2 Add 10 ml of HCl (4.2), 2 ml of HNO₃ (4.3) and 5 ml of HF (4.1). Heat to complete dissolution. Make sure that all deposits that may have formed on the walls of the beaker are washed down using a glass stick with a rubber head. Add 20 ml of fuming acid mixture (4.6) and heat until the perchloric acid starts to fume. Continue to fume for 2 to 3 min (the white smoke must be on the top of the PTFE beaker).

Bestelformulier

NEN

Stuur naar:

NEN Standards Products & Services
t.a.v. afdeling Klantenservice
Antwoordnummer 10214
2600 WB Delft

NEN Standards Products & Services

Postbus 5059
2600 GB Delft

Vlinderweg 6
2623 AX Delft

T (015) 2 690 390
F (015) 2 690 271

www.nen.nl/normshop

Ja, ik bestel

___ ex. NPR-ISO/TS 13899-3:2005 en Steel - Determination of Mo, Nb and W contents in alloyed steel - Inductively coupled plasma atomic emission spectrometric method - Part 3: Determination of W content € 48.19

Wilt u deze norm in PDF-formaat? Deze bestelt u eenvoudig via www.nen.nl/normshop

Gratis e-mailnieuwsbrieven

Wilt u op de hoogte blijven van de laatste ontwikkelingen op het gebied van normen, normalisatie en regelgeving? Neem dan een gratis abonnement op een van onze e-mailnieuwsbrieven. www.nen.nl/nieuwsbrieven

Retourneren

Fax: (015) 2 690 271
E-mail: klantenservice@nen.nl
Post: NEN Standards Products & Services,
t.a.v. afdeling Klantenservice
Antwoordnummer 10214,
2600 WB Delft
(geen postzegel nodig).

Gegevens

Bedrijf / Instelling _____

T.a.v. _____ O M O V

E-mail _____

Klantnummer NEN _____

Uw ordernummer _____ BTW nummer _____

Postbus / Adres _____

Postcode _____ Plaats _____

Telefoon _____ Fax _____

Factuuradres (indien dit afwijkt van bovenstaand adres)

Postbus / Adres _____

Postcode _____ Plaats _____

Datum _____ Handtekening _____

Voorwaarden

- De prijzen zijn geldig tot 31 december 2016, tenzij anders aangegeven.
- Alle prijzen zijn excl. btw, verzend- en handelingskosten en onder voorbehoud bij o.m. ISO- en IEC-normen.
- Bestelt u via de normshop een pdf, dan betaalt u geen handeling en verzendkosten.
- Meer informatie: telefoon (015) 2 690 391, dagelijks van 8.30 tot 17.00 uur.
- Wijzigingen en typfouten in teksten en prijsinformatie voorbehouden.
- U kunt onze algemene voorwaarden terugvinden op: www.nen.nl/leveringsvoorwaarden.