INTERNATIONAL STANDARD

ISO 13898-2

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Steel and iron — Determination of nickel, copper and cobalt contents — Inductively coupled plasma atomic emission spectrometric method —

Part 2:
Determination of nickel content

Aciers et fontes — Dosage du nickel, du cuivre et du cobalt — Méthode par spectrométrie d'émission atomique avec plasma induit par haute fréquence —

Partie 2: Dosage du nickel

Reference number
ISO 13898-2:1997(E)

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.

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.

International Standard ISO 13898-2 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition.*

ISO 13898 consists of the following parts, under the general title *Steel and iron — Determination of nickel, copper and cobalt contents — Inductively coupled plasma atomic emission spectrometric method.*

- *Part 1: General requirements and sample dissolution*
- *Part 2: Determination of nickel content*
- *Part 3: Determination of copper content*
- *Part 4: Determination of cobalt content*

Annexes A and B of this part of ISO 13898 are for information only.

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Steel and iron — Determination of nickel, copper and cobalt contents — Inductively coupled plasma atomic emission spectrometric method —

Part 2:
Determination of nickel content

1 Scope

This part of ISO 13898 specifies an inductively coupled plasma atomic emission spectrometric method for the determination of nickel content in unalloyed steel and unalloyed iron.

The method is applicable to nickel contents between 0,001 % (*m/m*) and 0,30 % (*m/m*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 13898. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 13898 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 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: 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 13898-1:1997, *Steel and iron - Determination of nickel, cobalt and copper contents - Inductively coupled plasma atomic emission spectrometric method - Part 1: General requirements and sample dissolution.*

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

3 Principle

The principle is described in clause 3 of ISO 13898-1:1997.

4 Reagents

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

In addition to the reagents given in clause 4 of ISO 13898-1:1997, the following are required.

4.1 Nickel standard solution.

4.1.1 Stock solution, corresponding to 1,0 g of nickel per litre.

Weigh, to the nearest 0,1 mg, 1,000 g of nickel metal [purity >99,99 % (*m/m*)] and transfer to a 200 ml beaker. Add 50 ml of nitric acid (4.3 of ISO 13898-1:1997) cover with a watch-glass and heat to dissolve gently. Cool to ambient temperature , quantitatively transfer to a 1 000 ml one-mark volumetric flask and dilute to the mark with water and mix.

1 ml of this stock solution contains 1,0 mg Ni.

4.1.2 Standard solution A, corresponding to 0,100 g of nickel per litre.

Transfer 20,0 ml of the nickel stock solution (**4.1.1**) to a 200 ml one-mark volumetric flask, dilute to the mark with water and mix.

Prepare this standard solution immediately before use.

1 ml of this standard solution contains 0,10 mg Ni.

4.1.3 Standard solution B, corresponding to 0,020 g of nickel per litre.

Transfer 10,0 ml of the nickel stock solution (**4.1.1**) to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

If the calibration graph is found to be non-linear, additional calibration series may be used.

Prepare this standard solution immediately before use.

1 ml of this standard solution contains 0,020 mg Ni.

5 Apparatus

The apparatus required is given in clause 5 of ISO 13898-1:1997.

6 Sampling

Carry out sampling in accordance with ISO 14284.

7 Procedure

7.1 Test portion

Weigh, to the nearest 1 mg, about 1,00 g of the test sample.

7.2 Blank test (corresponding to the zero member)

Proceed as directed in 7.2 of ISO 13898-1:1997.

7.3 Determination

7.3.1 Preparation of the test solution

Proceed as directed in 7.3.1 of ISO 13898-1:1997.

7.3.2 Preparation of the calibration solutions

Transfer to a series of six 200 ml beakers 1,00 g ± 0,001 g of the pure iron (**4.1** of ISO 13898-1:1997). Add to each beaker 10 ml of nitric acid (**4.3** of ISO 13898-1:1997), cover the beaker with a watch-glass and heat gently until the end of effervescence. Add 10 ml of hydrochloric acid (**4.2** of ISO 13898-1:1997) and continue the heating until complete dissolution occurs.

Cool to ambient temperature and transfer the solution to six 200 ml one-mark volumetric flasks, rinsing the beakers with the minimum quantity of water.

Using a pipette or burette, add to the volumetric flasks the volume of nickel standard solution A (**4.1.2**) indicated in table 1. If the calibration graph is found to be non-linear, additional calibration series may be used (e.g. tables 2 and 3). If the internal standard technique is used, using a pipette, add 2 ml of the scandium internal standard solution (**4.4** of ISO 13898-1:1997) or 10 ml of the yttrium internal standard solution (**4.5** of ISO 13898-1:1997). Dilute to the mark with water and mix.

7.4 Spectrometric measurements

7.4.1 Optimization of the instrument

Proceed as directed in 7.4.1 of ISO 13898-1:1997.

7.4.2 Measurements of the emitted intensities

Proceed as directed in 7.4.2 of ISO 13898-1:1997.

7.4.3 Preparation of the calibration graph

Proceed as directed in 7.4.3 of ISO 13898-1:1997.

Table 1— Nickel contents between 0,001 % (*m/m*) and 0,30 % (*m/m*)

Volume of nickel standard solution A (4.1.2) ml	Nickel concentration µg/ml	Corresponding nickel content in the test portion % (*m/m*)
0[1]	0	0
5,0	2,50	0,050
10,0	5,00	0,100
15,0	7,50	0,150
20,0	10,00	0,200
30,0	15,00	0,300

[1] Zero member.

Table 2 — Example for nickel contents up to 0,010 % (*m*/*m*)

Volume of nickel standard solution B (4.1.3) ml	Nickel concentration µg/ml	Corresponding nickel content in the test portion % (*m*/*m*)
0[1]	0	0
0,5	0,050	0,001 0
1,0	0,100	0,002 0
2,0	0,200	0,004 0
3,0	0,300	0,006 0
5,0	0,500	0,010 0

1) Zero member.

Table 3 — Example for nickel contents between 0,010 % (*m*/*m*) and 0,060 % (*m*/*m*)

Volume of nickel standard solution B (4.1.3) ml	Nickel concentration µg/ml	Corresponding nickel content in the test portion % (*m*/*m*)
0[1]	0	0
5,0	0,50	0,010
10,0	1,00	0,020
20,0	2,00	0,040
30,0	3,00	0,060
50,0	5,00	0,100

1) Zero member.

8 Expression of results

8.1 Method of calculation

Proceed as directed in 8.1 of ISO 13898-1:1997.

8.2 Precision

A planned trial of this method was carried out by 26 laboratories in 12 countries at 11 levels of nickel, each laboratory making three determinations of nickel content at each level (see notes 1 and 2).

The test samples used are listed in table A.1.

The results obtained were treated statistically in accordance with ISO 5725-1, ISO 5725-2 and ISO 5725-3, using the data obtained from the samples containing 11 levels of nickel within the application range.

The data obtained showed a logarithmic relationship between nickel content and repeatability limit (*r*) and reproducibility limits (*R* and *R*w) of the test results (see note 3), as summarized in table 4. The graphical representation of the data is given in annex B.

NOTES

1 Two of the three determinations were carried out under repeatability conditions as defined in ISO 5725-1, i.e. one operator, same apparatus, identical operating conditions, same calibration, and a minimum period of time.

2 The third determination was carried out at a different time (on a different day) by the same operator as in note 1 using the same apparatus with a new calibration.

3 From the results obtained on day 1 the repeatability limit (*r*) and reproducibility limit (*R*) were calculated using the procedure specified in ISO 5725-2. From the first result obtained on day 1 and the result obtained on day 2, the within-laboratory reproducibility limit (*R*w) was calculated using the procedure given in ISO 5725-3.

Table 4

Nickel content	**Repeatability limit**	**Reproducibility limits**	
% (*m*/*m*)	*r*	*R*	*R*w
0,001	0,000 39	0,000 76	0,000 55
0,002	0,000 46	0,000 95	0,000 67
0,005	0,000 56	0,001 3	0,000 87
0,010	0,000 66	0,001 6	0,001 0
0,020	0,000 77	0,002 0	0,001 3
0,050	0,000 95	0,002 8	0,001 6
0,050	0,001 3	0,003 7	0,002 0
0,100	0,002 6	0,007 4	0,003 7
0,200	0,005 1	0,015	0,007 0
0,300	0,007 5	0,023	0,010

9 Test report

Proceed as directed in clause 9 of ISO 13898-1:1997.

Annex A

(informative)

Additional information on the international cooperative tests

The repeatability and reproducibility data in table 4 were derived from the results of international analytical trials carried out in 1993 on nine steel samples and two iron samples in 12 countries involving 26 laboratories.

The results of the trials were reported in document ISO/TC 17/SC 1 N 1024, March 1994. The graphical presentation of the precision data is given in annex B.

The test samples used are listed in table A.1.

Table A.1

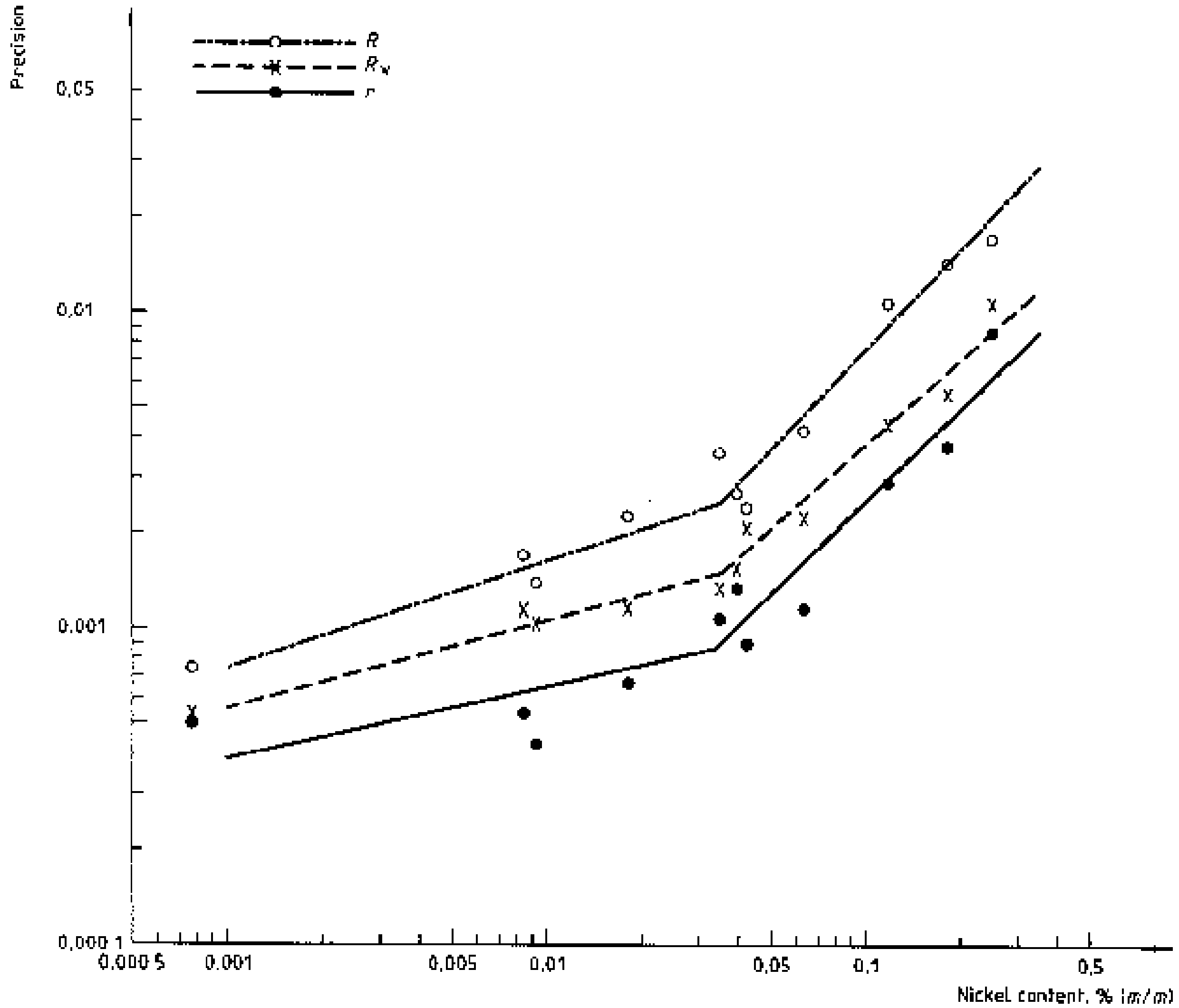
Sample	**Nickel content** % (*m/m*)			**Precision data**		
	Certified	**Found**		**Repeatability limit**	**Reproducibility limits**	
		$\overline{w}_{Ni,1}$	$\overline{w}_{Ni,2}$	r	R	R_W
JSS 003-3 (Unalloyed steel)	0,000 8	0,000 76	0,000 76	0,000 49	0,000 75	0,000 53
NR 1C (Unalloyed steel)	0,009 0	0,009 0	0,008 9	0,000 43	0,001 4	0,001 0
NR 21 (Unalloyed steel)	0,035	0,034 6	0,034 5	0,001 1	0,003 6	0,001 3
NBS 15h (Unalloyed steel)	0,017	0,017 6	0,017 6	0,000 69	0,002 3	0,001 1
NBS 16f (Unalloyed steel)	0,008	0,008 3	0,008 2	0,000 54	0,001 7	0,001 1
BAS 087-1 (Unalloyed steel)	0,118	0,119	0,119	0,002 9	0,010 8	0,004 3
BCS 452 (Unalloyed steel)	0,19	0,191	0,191	0,004 0	0,014 6	0,005 6
IRSID 081-1 (Unalloyed steel)	0,042	0,042 2	0,042 2	0,000 90	0,002 4	0,002 1
IRSID 010-1 (Unalloyed steel)	0,259	0,264	0,264	0,008 9	0,017 5	0,010 7
EURO 488-1 (Pig iron)	0,065 1	0,065 5	0,065 4	0,001 2	0,004 3	0,002 3
EURO 487-1 (Pig iron)	0,040	0,040 5	0,040 4	0,001 4	0,002 7	0,001 5

$\overline{w}_{Ni,1}$: general mean within a day

$\overline{w}_{Ni,2}$: general mean between days

Annex B
(informative)

Graphical representation of precision data



Nickel contents up to 0,03 %(*m*/*m*)

$\lg r = 0{,}224\ 6 \lg \overline{w}_{\mathrm{Ni},1} - 2{,}731\ 8$

$\lg R = 0{,}331\ 3 \lg \overline{w}_{\mathrm{Ni},1} - 2{,}127\ 4$

$\lg R_W = 0{,}279\ 1 \lg \overline{w}_{\mathrm{Ni},2} - 2{,}419\ 0$

Nickel contents between 0,03 %(*m*/*m*) and 0,3 %(*m*/*m*)

$\lg r = 0{,}994\ 1 \lg \overline{w}_{\mathrm{Ni},1} - 1{,}599\ 2$

$\lg R = 0{,}988\ 8 \lg \overline{w}_{\mathrm{Ni},1} - 1{,}144\ 1$

$\lg R_W = 0{,}920\ 8 \lg \overline{w}_{\mathrm{Ni},2} - 1{,}510\ 3$

where

$\overline{w}_{\mathrm{Ni},1}$ is the average nickel content, expressed as a percentage by mass, obtained within a day;

$\overline{w}_{\mathrm{Ni},2}$ is the average nickel content, expressed as a percentage by mass, obtained between days.

Figure B.1 - Logarithmic relationship between nickel content ($\overline{w}_{\mathrm{Ni}}$) and repeatability limit (*r*) or reproducibility limits (*R* and *Rw*)

ICS 77.080.01

Descriptors: iron and steel products, unalloyed steels, unalloyed cast iron, chemical analysis, determination of content, nickel, spectrometric method, atomic emission spectrometric method.

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