INTERNATIONAL STANDARD

ISO 13898-4

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

Part 4:
Determination of cobalt 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 4: Dosage du cobalt

Reference number
ISO 13898-4:1997(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 13898-3 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.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

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

Part 4:
Determination of cobalt content

1 Scope

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

The method is applicable to cobalt contents between 0,001 % (*m/m*) and 0,10 % (*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 5725-2:1994, *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 Cobalt, standard solution.

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

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

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

4.1.2 Standard solution, corresponding to 0,020 g of cobalt per litre.

Transfer 10,0 ml of the cobalt 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, an additional calibration series may be used.

Prepare this standard solution immediately before use.

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

5 Apparatus

The apparatus required is given in clause 5 of ISO 13898-1:—[1].

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

Introduce into 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 cobalt standard solution (**4.1.2**) indicated in table 1. If the calibration graph is found to be non-linear, an additional calibration series may be used (e.g. table 2). If the internal standard technique is used, using a pipette, add 2 ml of the scandium internal standard solution (**4.4** of ISO 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.

Table 1 — Cobalt contents between 0,001 % (*m/m*) and 0,10 % (*m/m*)

Volume of cobalt standard solution (4.1.2) ml	Cobalt concentration µg/ml	Corresponding cobalt 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.		

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

Volume of cobalt standard solution (4.1.2) ml	Cobalt concentration µg/ml	Corresponding cobalt 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.		

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.

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 eight levels of cobalt, each laboratory making three determinations of cobalt content at each level (see notes).

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 eight levels of cobalt within the application range.

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

Table 3

Cobalt content	Repeatability limit	Reproducibility limits	
% (m/m)	r	R	R_W
0,001	0,000 18	0,000 51	0,000 30
0,002	0,000 25	0,000 70	0,000 40
0,005	0,000 37	0,001 05	0,000 57
0,010	0,000 50	0,001 4	0,000 75
0,020	0,000 68	0,001 9	0,000 98
0,050	0,001 02	0,002 9	0,001 4
0,100	0,001 4	0,004 0	0,001 8

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.

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 3 were derived from the results of international analytical trials carried out in 1993 on seven steel samples and one iron sample 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

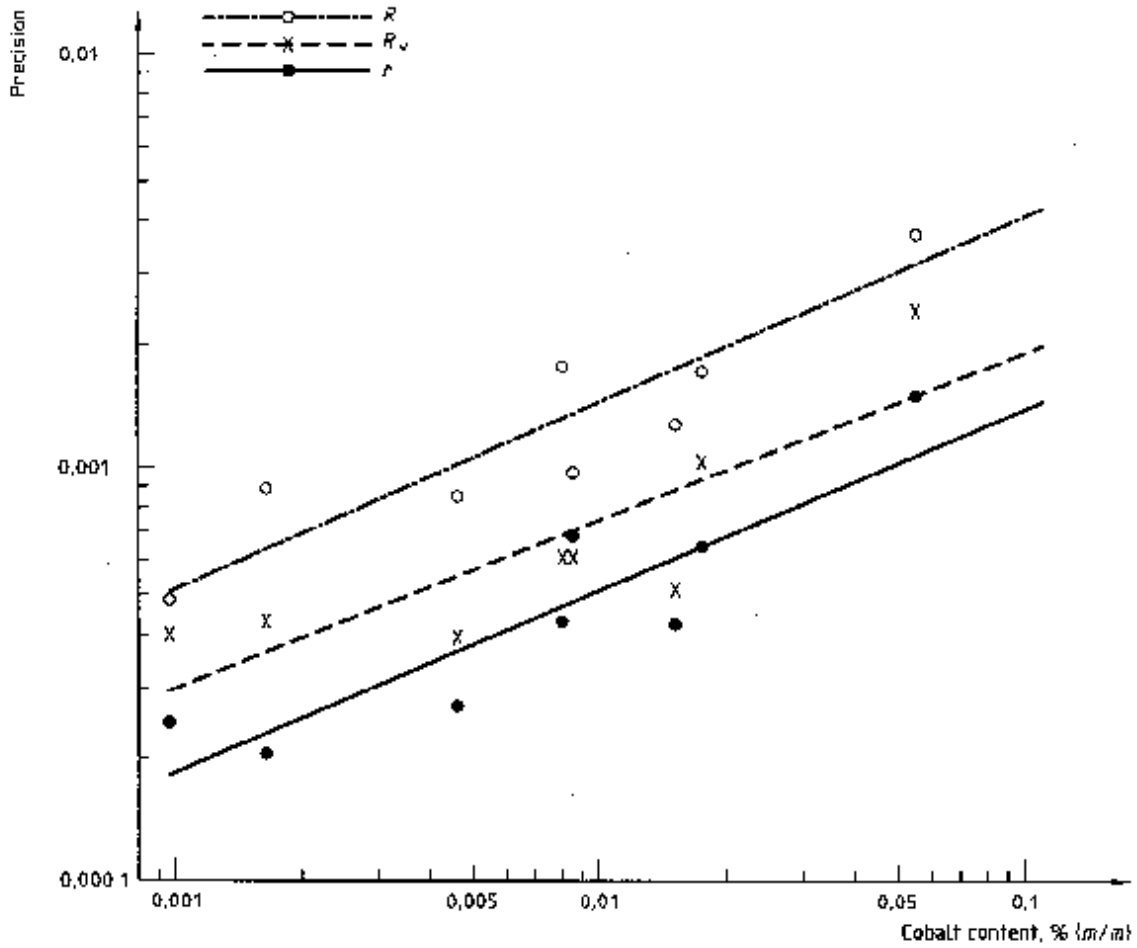
Sample	Cobalt content % (*m/m*)			Precision data		
	Certified	Found		Repeatability limit	Reproducibility limits	
		$\overline{w}_{Co,1}$	$\overline{w}_{Co,2}$	r	R	R_W
JSS 003-3 (unalloyed steel)	0,001 0	0,000 97	0,000 98	0,000 24	0,000 48	0,000 39
NR 1C (unalloyed steel)	0,004 6	0,004 7	0,004 7	0,000 26	0,000 84	0,000 38
NR 21 (unalloyed steel)	0,009	0,008 0	0,008 0	0,000 43	0,001 7	0,000 60
NBS 16f (unalloyed steel)	0,003	0,003 6	0,003 6	0,000 34	0,000 87	0,000 49
BAS 087-1 (unalloyed steel)	0,015	0,014 9	0,014 8	0,000 42	0,001 3	0,000 93
BCS 456-1 (unalloyed steel)	0,052	0,054	0,054	0,001 5	0,003 6	0,002 3
IRSID 081-1 (unalloyed steel)	0,017	0,017 5	0,017 4	0,000 64	0,001 7	0,001 01
EURO 487-1 (pig iron)	0,008 8	0,008 5	0,008 5	0,000 66	0,009 4	0,000 60

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

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

Annex B
(informative)

Graphical representation of precision data



$$\lg r = 0{,}439\ 5 \lg \overline{w}_{Co,1} - 2{,}421\ 1$$

$$\lg R = 0{,}445\ 9 \lg \overline{w}_{Co,1} - 1{,}953\ 8$$

$$\lg R_W = 0{,}392\ 8 \lg \overline{w}_{Co,2} - 2{,}340\ 9$$

where

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

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

Figure B.1 — Logarithmic relationship between cobalt content ($\overline{w}_{Co}$) and repeatability limit (r) or reproducibility limits (R and R_W)

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

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