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Low alloyed steel — Determination of C, Si, Mn, P, S, Cr, Ni, Al, Ti and Cu - Glow discharge optical emission spectrometry (routine method)

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National foreword

This British Standard is the UK implementation of ISO 19272:2015.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Aciers faiblement alliés — Détermination de C, Si, Mn, P, S, Cr, Ni, Al, Ti et Cu — Spectrométrie d'émission optique à décharge luminescente (méthode de routine)

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Foreword

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 17, *Steel*, Subcommittee SC 1, *Method of determination of chemical composition*.

Low alloyed steel — Determination of C, Si, Mn, P, S, Cr, Ni, Al, Ti and Cu - Glow discharge optical emission spectrometry (routine method)

1 Scope

This International Standard specifies a glow discharge optical emission spectrometric (GD-OES) method for determination of the mass fraction Carbon, Silicon, Manganese, Phosphorus, Sulfur, Chromium, Nickel, Aluminium, Titanium and Copper in low alloyed steels.

The content ranges to which the method is applicable are shown in Table 1.

Table 1 — Content ranges

Element	Content ranges mass fraction %
C	0,060 to 0,35
Si	0,14 to 1,50
Mn	0,090 to 0,70
P	0,010to 0,070
S	0,005 to 0,050
Cr	0,008 to 0,65
Ni	0,050 to 0,50
Al	0,006 to 0,90
Ti	0,014 to 0,13
Cu	0,005 to 1,00

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable to its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 5725-2, *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, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

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

ISO 14707, *Surface chemical analysis — Glow discharge optical emission spectrometry (GD-OES) — Introduction to use*

3 Principle

The method used involves the following steps:

a) a sample with a flat and smooth surface is used as the cathode of a direct current or radio frequency glow discharge device;

b) cathodic sputtering of the sample surface, atomization of the sputtered atoms and ions from the sample surface; excitation of these atoms and ions in the plasma formed in the glow discharge device;

c) spectrometric measurement of the intensity of the emitted light from the ions or the atoms of the elements to be determined and, optionally, the emitted light from iron at 371,994 nm or 271,441 nm or another appropriate wavelength (if internal standard is used);

d) conversion of the measured signals to the contents through calibration curves established by measuring certified reference materials.

4 Apparatus

4.1 Glow discharge optical emission spectrometer

4.1.1 General

An optical emission spectrometer system consists of a Grimm type or similar glow discharge source (direct current or radio frequency powered) and a simultaneous optical spectrometer as described in ISO 14707, with suitable spectral lines for the elements to be determined. A sequential optical system may also be used alone or combined with simultaneous channels.

A 2 mm to 8 mm range of the inner diameter of the anode of the glow discharge is recommended. A cooling device is also recommended, but not strictly required for implementation of the method. The anode-cathode gap is normally around 0,1 mm to 0,3 mm; otherwise, follow the instrument manufacturer's instructions.

4.1.2 Short term stability

Carry out at least 10 measurements of the emission intensity of an element having a content around the corresponding upper limit specified in this International Standard on an appropriate sample. Allow the discharge to stabilize for at least 50 s before each measurement. The data acquisition time should be in the range of 5 s to 30 s. Each measurement shall be carried out on a newly polished surface of the sample. Calculate the corresponding standard deviation and mean. The relative standard deviation (RSD) should not exceed 5 % for contents less than 0,5 %, otherwise, the RSD should not exceed 3 %.

4.1.3 Detection limit

Detection limits may be determined by either the SNR method or SBR-CVB method (see A.1 and A.2).

4.1.4 Data acquisition and processing system

The data acquisition and processing system is conducted by a computer, equipped with software suitable for controlling the instrument parameters and running the test programs.

4.1.5 Others

For other basic requirements of the instrument, use ISO 14707.

5 Sampling

Carry out sampling in accordance with ISO 14284 or appropriate national standards for steel.

6 Procedure

6.1 Sample preparation

Samples shall be homogeneous, flat and free of porosity. The surface of the sample shall be prepared by using suitable methods to ensure cleanliness and flatness. Surface preparation may be achieved by using abrasive-wheel or milling machine. All the reference materials and samples shall be prepared under the same conditions and their size should be suitable for the glow discharge source used.

6.2 Selection of spectral lines

For each element to be determined, there are a number of spectral lines that can be used. Suitable lines shall be selected on the basis of several factors, including the spectral range of the spectrometer used, element concentration, sensitivity of the spectral lines and spectral interference from other elements present in the samples. Table 2 contains some suggestions concerning suitable spectral lines. With these lines, no obvious inter-element interferences are observed in low alloyed steels. Spectral lines other than those listed may be used, if they have favourable characteristics, but special attention needs to be paid to the influence of co-existing elements and corrections might be required.

Table 2 — Suggested wavelengths for the analysis of low alloy steels

Element	Wavelength nm
Fe	371,994
	271,441
C	165,700
	156,143
Si	288,158
	251,611
Mn	403,449
	257,610
P	177,497
	178,287
S	180,731
Cr	425,433
Ni	349,296
	341,477
Al	396,152
Ti	337,279
	365,350
Cu	219,228
	327,396

6.3 Optimization of the instrument

6.3.1 General

Follow the manufacturer's instructions or other suitable documented procedures to start and set the GD-OES and let it stabilize. Check, or adjust if necessary, the entrance slit in accordance with the manufacturer's instructions to ensure it is correctly positioned so that the intensity is measured at the peak of the spectral line in order to obtain the best signal to background ratio (make reference to ISO 14707 for further information). Carry out at least three preliminary discharges on a generic sample before any measurement procedures.

NOTE Analyte lines are centred by adjusting the entrance slit whilst carrying out discharges on a reference material. It is advisable to profile the instrument on a daily basis for a newly installed instrument during the first week of operation and then periodically over time. Profiling is carried out initially on all lines or at least on those included in each analytical program or by following the recommendations of the instrument manufacturer.

Follow manufacturer's recommendation or other suitable documented procedures to select or adjust all instrumental parameters: evacuation time, argon purging time [pumpdown cycles], glow discharge spectrometer opening mode [startup time], flush time [cleaning time], sputtering time, etc.

6.3.2 Setting the discharge parameters of a direct current (DC) source

DC glow discharge spectrometers usually have three parameters (current, voltage and pressure) to be controlled. Any two of these three parameters may be fixed to constant values by varying the third one. The user may adopt the following procedures, or any other mode recommended by the manufacturers.

6.3.2.1 Constant applied voltage and current

The controlled parameters are applied voltage and current. Set the power supply of the glow discharge source to constant voltage and constant current operation. Firstly, set the voltage and current to typical values recommended by the manufacturer or reliable values. If the recommended values are not available or not suitable for the task, following steps could be pursued to determine the optimum parameters.

Set the high voltage of the detectors as described in 6.3.4.

Step one: set an initial value or the value recommended by the manufacturer for current and voltage (initial discharge condition). For DC glow discharge, electrical current is normally in the range of 5 mA to 10 mA for a 2 mm to 2,5 mm anode, 15 mA to 45 mA for a 4 mm anode and 40 mA to 100 mA for a 7 mm to 8 mm anode. Voltage is usually in the range of 600 V to 1250 V. Determine the intensities corresponding to an appropriate sample under the initial discharge condition. Fix one parameter (current or voltage), and change the other one gradually. Measure the intensities at least seven times at each setting. Do this at the entire parameter range allowed by the instrument.

Step two: fix the parameter which changed in step one and gradually change the other parameter. Repeat the operation as described in step one.

Step three: calculate the CVs of the measurements for each setting. Investigate the influence of the discharge parameters on the intensities and CVs and fix the optimum instrumental condition.

6.3.2.2 Constant applied current and pressure

The controlled parameters are applied current and pressure. Set the power supply of the glow discharge source to constant current and constant pressure operation. Firstly, set the current and pressure to typical values recommended by the manufacturer or values suitably documented. If the recommended values are not available or not suitable for the task, the following steps could be pursued to determine the optimum parameters.

Set the high voltage of the detectors as described in 6.3.4.

Step one: set an initial value or the value recommended by the manufacturer for current and pressure (initial discharge condition). For DC glow discharge, electrical current is normally in the range of 5 mA to 10 mA for a 2 mm to 2,5 mm anode, 15 mA to 45 mA for a 4 mm anode and 40 mA to 100 mA for a 7 mm to 8 mm anode. Set the initial pressure to a value to obtain a voltage in the range of 600 V to 1250 V or set the initial pressure to a value which is in the middle of the allowed range of the instrument. Determine the intensities corresponding to an appropriate sample under the initial discharge condition. Fix one parameter (current or pressure) and change another one gradually. Measure the intensities at least seven times at each setting. Do this at the entire parameter range allowed by the instrument.

Step two: fix the parameter which changed in step one and gradually change the other parameter. Repeat the operation as described in step one.

Step three: calculate the CVs of the measurements for each setting. Investigate the influence of the discharge parameters on the intensities and CVs and fix the optimum instrumental condition.

Use a similar procedure to optimize the analytical parameters for other operation modes.

6.3.3 Setting the discharge parameters of radiofrequency (RF) source

At the time of publication of this International Standard, most RF sources are operated with constant applied power and constant pressure. Other modes also exist such as:

- constant applied power and constant voltage;
- constant voltage and constant pressure;
- constant power and constant argon flow, etc.

All RF operational modes are allowed in this International Standard, provided they meet the requirements described in 4.1.2 and 4.1.3 and ensure that constant excitation conditions in calibration and analysis can be kept for optimum accuracy.

6.3.3.1 Constant applied power and pressure

The controlled parameters are applied power and pressure. Set the power supply of the glow discharge source to constant power and constant pressure. First set the power and pressure to typical values recommended by the manufacturer or values suitably documented. If the recommended values are not available or not suitable for the task, following steps could be pursued to determine the optimum parameters.

Set the high voltage of the detectors as described in 6.3.4.

Step one: set an initial discharge condition which may be the value recommended by the manufacturer or a value in the middle of the parameter range allowed by the instrument for power and pressure. Determine the intensities corresponding to an appropriate sample under the initial discharge condition. Fix one parameter (power or pressure) and change the other one gradually. Measure the intensities at least seven times at each setting. Do this at the entire parameter range allowed by the instrument.

Step two: fix the parameter which changed in step one and gradually change the other parameter. Repeat the operation as described in step one.

Step three: calculate the CVs of the measurements for each setting. Investigate the influence of the discharge parameters on the intensities and CVs and fix the optimum instrumental condition.

6.3.3.2 Constant power and voltage

The controlled parameters are applied power and voltage. Set the power supply of the glow discharge source to constant power and constant voltage. First set the power and voltage to typical values recommended by the manufacturer or values suitably documented. If the recommended values are not available or not suitable for the task, following steps could be pursued to determine the optimum parameters.

Set the high voltage of the detectors as described in 6.3.4.

Step one: set an initial discharge condition which may be the value recommended by the manufacturer or a value in the middle of the parameter range allowed by the instrument for power and voltage. Determine the intensities corresponding to an appropriate sample under the initial discharge condition. Fix one parameter (power or voltage) and change the other one gradually. Measure the intensities at least seven times at each setting. Do this at the entire parameter range allowed by the instrument.

Step two: fix the parameter which changed in step one and gradually change the other parameter. Repeat the operation as described in step one.

Step three: calculate the CVs of the measurements for each setting. Investigate the influence of the discharge parameters on the intensities and CVs and fix the optimum instrumental condition.

Use a similar procedure to optimize the analytical parameters for other operation modes.

6.3.4 Optimization of the detection system

Photomultiplier power input shall be selected as a function of the type of detector and the content range of each element to be determined. Operate the source and observe the output signals from the detector of the elements concerned, adjust the high voltage of the detectors in such a way that sufficient sensitivity at the lowest analyte concentrations is ensured, without overflowing the detectors at the highest analyte concentrations.

6.3.5 Pre-burning time and integration time

After a glow discharge is initiated, it needs some time to reach stabilization. This stabilization time is dependent on the conditions of the instrument and sample surface. Measurement of the signals can only be done after stabilization. This requirement is met by setting a suitable pre-burning time. A pre-burning time reasonably set may also clean the sample surface to eliminate a possible contamination caused by sample preparation.

Select pre-burning time according to the manufacturer's recommendation. If the recommended value is neither suitable nor available, select it as described below:

Excite the sample continuously, record the diagram of intensity vs sputtering time. Select a sputtering time, at which the intensity became sufficiently stable, as pre-burning time.

Select integration time according to the manufacturer's recommendation. If the recommended value is neither suitable nor available, select it as described below:

Set a series of integration times, normally in the range of 5 s to 50 s. Measure the intensities corresponding to the elements to be determined at least seven times at each integration setting. Check the influence of the integration times on intensities and CVs. Integration time shall be long enough to ensure CVs required in 4.1.2.

6.3.6 Validation of the instrumental parameters

The discharge parameters and detector high voltages selected shall be used for the analytical program. Using a suitable sample, carry out some preliminary tests and ensure that the key instrumental parameters are appropriate to meet the criteria described in 4.1.2 and 4.1.3.

6.4 Calibration

A series of certified reference materials (CRM) (at least five for each element), which have the same or at least very similar matrix and metallurgical structure as the samples to be analysed is used to prepare the calibration curves. The content range of the CRM used shall cover that of all the samples to be analysed within each specific analytical program. Adjust the source parameters as described in 6.3. Measure each CRM at least three times and every time on a new area of the sample surface. The mean intensity is correlated to the corresponding certified content and a regression is calculated by the least squares method. In order to minimize possible signal fluctuations, it is advisable to use the internal standard method.

6.5 Checking of the trueness of the method

In order to check the trueness of the method, select a series of certified reference materials, necessarily independent from that used for the calibration of the spectrometer and analyse it. The composition of the certified reference materials selected shall cover the full range of the calibration. The results obtained for each certified reference material shall be situated in the interval “Certified Value ± 2σ”, where σ is standard deviation of certified value.

If necessary, in order to fulfil this trueness requirement for all the certified reference materials analysed, carry out adjustments of the calibration curves (inter element corrections and/or modification of the mathematical function defining each curve). If the trueness criteria cannot be reached, re-prepare the calibration curve or re-select the parameters.

6.6 Drift correction

Before starting a series of determinations by using an already recorded program, it’s necessary to check the drift condition of the instrument. The procedure for drift checking is the same as described in 6.5. Drift correction is not necessary if the results are within specified accuracy; otherwise, the drift correction shall be carried out.

Whenever the drift correction is performed, an accuracy control needs to be carried out as described in 6.5.

6.7 Analysis of the samples

Each sample shall be analysed at least three times. Use the mean intensity to derive the content of each element from the corresponding calibration curve.

In the case where the internal standard method is employed, use the intensity ratio between each analyte and the internal standard element.

The content of each element to be determined shall be within the range of the calibration curves.

7 Expression of results

7.1 Method of calculation

Using the intensities (or intensity ratios) obtained, the content of each element measured, expressed as mass fraction, is derived from the calibration curves established in 6.4.

7.2 Precision

A planned precision trial of this method was carried out by 11 laboratories, at 19 content levels for Carbon, Silicon and Manganese, 21 for Phosphorus and Chromium, 22 for Sulfur, 18 for Nickel, 15 for Aluminium and Titanium and 23 for Copper. Each laboratory made three determinations for each element at each level.

NOTE 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.

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

The composition of the test samples used is listed in Table B.1.

The results obtained were treated statistically in accordance with ISO 5725-1, ISO 5725–2, and ISO 5725–3.

The relationship between element contents and repeatability, r, and reproducibility (R and R_W) of the test results are summarized in Tables 3 to 12. The graphical representations of the data are shown in Annex C.

NOTE 3 From the two values obtained on day one and the value obtained on day two, the repeatability limit, r, and reproducibility limits (R and R_W) were calculated using the procedure specified in ISO 5725-3.

NOTE 4 For several elements and samples (levels of content), r and R_W are mostly at the same level whilst the corresponding R is much higher. It would be caused by “laboratory effect”.

Table 3 — Carbon — Repeatability limit and reproducibility limits

Carbon content mass fraction %	**Repeatability limit** r mass fraction %	**Reproducibility limits**	
		R_W mass fraction %	R mass fraction %
m	$\lg r = 1{,}016 \lg m - 1{,}310$	$\lg R_W = 0{,}607 \lg m - 1{,}457$	$\lg R = 0{,}719 \lg m - 1{,}107$
0,060	0,0028	0,0064	0,0103
0,10	0,0047	0,0087	0,0149
0,20	0,0096	0,0132	0,0246
0,35	0,0169	0,0185	0,0368

Table 4 — Silicon — Repeatability limit and reproducibility limits

Silicon content mass fraction %	**Repeatability limit** r mass fraction %	**Reproducibility limits**	
		R_W mass fraction %	R mass fraction %
m	$\lg r = 1{,}304 \lg m - 1{,}553$	$\lg R_W = 1{,}118 \lg m - 1{,}346$	$\lg R = 0{,}904 \lg m - 1{,}081$
0,14	0,0022	0,0050	0,0141
0,20	0,0034	0,0075	0,0194
0,50	0,0113	0,0208	0,0444
1,00	0,0280	0,0451	0,0830
1,50	0,0475	0,0710	0,1196

Table 5 — Manganese — Repeatability limit and reproducibility limits

Manganese content mass fraction %	**Repeatability limit** r mass fraction %	**Reproducibility limits**	
		R_W mass fraction %	R mass fraction %
m	$\lg r = 1{,}146 \lg m - 1{,}466$	$\lg R_W = 1{,}119 \lg m - 1{,}163$	$\lg R = 1{,}313 \lg m - 0{,}781$
0,09	0,0022	0,0046	0,0070
0,10	0,0024	0,0052	0,0081

Table 5 *(continued)*

Manganese content mass fraction %	**Repeatability limit** r mass fraction %	**Reproducibility limits**	
		R_W mass fraction %	R mass fraction %
0,20	0,0054	0,0113	0,0200
0,50	0,0155	0,0316	0,0666
0,70	0,0227	0,0461	0,1037

Table 6 — Phosphorus — Repeatability limit and reproducibility limits

Phosphorus content mass fraction %	**Repeatability limit** r mass fraction %	**Reproducibility limits**	
		R_W mass fraction %	R mass fraction %
m	$\lg r = 0{,}933 \lg m - 1{,}359$	$\lg R_\mathrm{W} = 1{,}034 \lg m - 0{,}985$	$\lg R = 0{,}796 \lg m - 1{,}001$
0,010	0,0006	0,0009	0,0026
0,020	0,0011	0,0018	0,0044
0,050	0,0027	0,0049	0,0092
0,070	0,0037	0,0066	0,0120

Table 7 — Sulfur — Repeatability limit and reproducibility limits

Sulfur content mass fraction %	**Repeatability limit** r mass fraction %	**Reproducibility limits**	
		R_W mass fraction %	R mass fraction %
m	$\lg r = 0{,}679 \lg m - 1{,}666$	$\lg R_\mathrm{W} = 0{,}638 \lg m - 1{,}561$	$\lg R = 0{,}559 \lg m - 1{,}440$
0,005	0,0006	0,0009	0,0019
0,010	0,0009	0,0015	0,0028
0,020	0,0015	0,0023	0,0041
0,050	0,0028	0,0041	0,0068

Table 8 — Chromium —Repeatability limit and reproducibility limits

Chromium content mass fraction %	**Repeatability limit** r mass fraction %	**Reproducibility limits**	
		R_W mass fraction %	R mass fraction %
m	$\lg r = 1{,}066 \lg m - 1{,}257$	$\lg R_\mathrm{W} = 0{,}740 \lg m - 1{,}380$	$\lg R = 0{,}790 \lg m - 0{,}992$
0,008	0,0003	0,0012	0,0023

Table 8 *(continued)*

Chromium content mass fraction %	**Repeatability limit** r mass fraction %	**Reproducibility limits** R_W mass fraction %	 R mass fraction %
0,010	0,0004	0,0014	0,0027
0,020	0,0009	0,0023	0,0046
0,050	0,0023	0,0045	0,0096
0,10	0,0048	0,0076	0,0166
0,20	0,0100	0,0127	0,0286
0,50	0,0264	0,0249	0,0590
0,60	0,0321	0,0286	0,0681

Table 9 — Nickel — Repeatability limit and reproducibility limits

Nickel content mass fraction %	**Repeatability limit** r mass fraction %	**Reproducibility limits** R_W mass fraction %	 R mass fraction %
m	$\lg r = 1{,}012 \lg m - 1{,}474$	$\lg R_W = 0{,}867 \lg m - 1{,}433$	$\lg R = 0{,}903 \lg m - 0{,}888$
0,050	0,0016	0,0027	0,0087
0,10	0,0033	0,0050	0,0161
0,20	0,0066	0,0091	0,0303
0,50	0,0167	0,0202	0,0692

Table 10 — Aluminium — Repeatability limit and reproducibility limits

Aluminium content mass fraction %	**Repeatability limit** r mass fraction %	**Reproducibility limits** R_W mass fraction %	 R mass fraction %
m	$\lg r = 0{,}759 \lg m - 1{,}588$	$\lg R_W = 0{,}838 \lg m - 1{,}279$	$\lg R = 0{,}646 \lg m - 1{,}109$
0,0060	0,0005	0,0007	0,0030
0,010	0,0008	0,0011	0,0042
0,020	0,0013	0,0020	0,0065
0,050	0,0027	0,0043	0,0117
0,10	0,0045	0,0076	0,0184
0,20	0,0076	0,0137	0,0287
0,50	0,0153	0,0294	0,0519
0,90	0,0238	0,0482	0,0759

Table 11 — Titanium — Repeatability limit and reproducibility limits

Titanium content mass fraction %	**Repeatability limit** r mass fraction %	**Reproducibility limits**	
		R_W mass fraction %	R mass fraction %
m	$\lg r = 0{,}536 \lg m - 2{,}192$	$\lg R_W = 0{,}536 \lg m - 1{,}970$	$\lg R = 0{,}878 \lg m - 1{,}272$
0,014	0,0006	0,0010	0,0013
0,020	0,0008	0,0012	0,0017
0,050	0,0013	0,0020	0,0039
0,10	0,0019	0,0029	0,0071
0,13	0,0022	0,0034	0,0089

Table 12 — Copper — Repeatability limit and reproducibility limits

Copper content mass fraction %	**Repeatability limit** r mass fraction %	**Reproducibility limits**	
		R_W mass fraction %	R mass fraction %
m	$\lg r = 0{,}717 \lg m - 1{,}920$	$\lg R_W = 0{,}708 \lg m - 1{,}634$	$\lg R = 0{,}760 \lg m - 1{,}056$
0,005	0,0003	0,0005	0,0016
0,010	0,0004	0,0009	0,0026
0,020	0,0007	0,0015	0,0045
0,050	0,0014	0,0028	0,0090
0,10	0,0023	0,0046	0,0152
0,20	0,0038	0,0074	0,0259
0,50	0,0073	0,0142	0,0519
1,00	0,0120	0,0232	0,0879

8 Test report

The test report shall include the following information:

a) all information necessary for identification of the sample, the laboratory and the date of analysis or of the test report;

b) the method used by reference to this International Standard, i.e. ISO 19272;

c) the results and the units in which they have been expressed;

d) any unusual features noted during the determination;

e) any operation not specified in this International Standard, or any optional operation which might have influenced the results.

Annex A
(informative)

Detection limit

A.1 General

The following two methods, which are reference to ISO 16962, are recommended to determine detection limits.

A.2 Signal-to-noise ratio (SNR) method

In order to evaluate the detection limit for a given analyte, the following steps should be performed:

Step one: Select a sample to be used as a blank. The sample composition should be similar to the samples to be analysed in terms of composition of the matrix and shall contain less than 1 μg/g of the analyte.

Step two: Perform 10 replicate burns on the blank. For each burn, acquire the emission intensity at the analytical wavelength for 10 s. These are the background emission intensity measurements. The glow discharge conditions used should be the same as those that will be used in the analysis of the samples. For each measurement, the blank should be pre-burned at these conditions for a sufficient period of time to achieve stable signals prior to the quantification of the emission intensity. Use an unsputtered area of the surface of the blank for each individual burn.

Step three: Compute the detection limit, L_{D}, expressed as a mass fraction using Formula (A.1):

$$L_D = \frac{3 \times S}{m_{\mathrm{SLOPE}}} \quad \text{(A.1)}$$

where

L_{D} is the detection limit, expressed as a mass fraction;

S is the standard deviation of the 10 background intensity measurements performed in step 2;

m_{SLOPE} is the slope of the calibration curve expressed as the ratio of intensity to mass fraction.

If the detection limit calculated is unacceptable, then the test should be repeated. If the second value calculated is also unacceptable, then the cause should be investigated and corrected prior to analysing samples.

A.3 Signal-to-background ratio - Coefficient of variation of the background (SBR – CVB) method

The second method, which does not require a blank, is often called the SBR-RSDB (Signal-to-background ratio –Coefficient of variation of the background) method. The method is performed as follows:

Step one: Select a sample having a matrix composition similar to that of the samples to be analysed, and for which the mass fraction of the analyte is greater than 0,1 % and well-known. If an analytical transition that is prone to self-absorption is to be used, then the mass fraction of the analyte should not exceed 1 %.

Step two: Perform three replicate burns on the chosen sample. For each burn, integrate the emission intensity at the analytical wavelength for 10 s. The glow discharge conditions used should be similar to those that will be used in the analysis of the samples. For each measurement, the sample should be pre-burned at these conditions for a sufficient period of time to achieve stable signals prior to the quantification of the emission intensity. Use an unsputtered area of the surface of the sample for each individual burn. Average the three replicate emission intensities.

Step three: Select a peak-free region of the emission spectrum within 0,2 nm of the analytical peak. Perform 10 replicate burns on the chosen sample. For each burn, integrate the intensity at the peak-free region for 10 s. These are the measurements of the background intensity. The glow discharge conditions and pre-burn should be the same as those used in step b). An unsputtered area of the surface of the sample should be used once more for each individual burn. Compute the mean and relative standard deviation of the 10 replicate measurements.

Step four: Calculate the detection limit, L_D, using Formula (A.2):

$$L_\mathrm{D} = \frac{3 \times \left(m_\mathrm{ANALYTE} \times C_{V,r,B} \; / \; 100\right)}{\left(S - B\right) / B} \tag{A.2}$$

where

L_D is the detection limit;

m_ANALYTE is the mass fraction of the analyte in the sample;

$C_{V,r,\mathrm{B}}$ is the coefficient of variation of the background from step 3, expressed as a percentage;

B is the mean background intensity from step 3;

S is the mean peak intensity from step 2.

If the detection limit calculated is unacceptable, then the test should be repeated. If the second value calculated is also unacceptable, then the cause should be investigated and corrected prior to analysing sample.

Annex B
(informative)

Additional information on international interlaboratory test

The composition of the test samples is listed in Table B.1. Detailed results obtained in international interlaboratory tests are shown in Tables B.2 to B.11.

Tables B.2 and B.11 show the results of the international trial carried out in five countries involving 11 laboratories in 2011.

The precision data are presented in graphical form in Annex C.

Table B.1 a — Composition of the test samples (mass fraction, %)

ID	58–1	58–2	58–3	58–4	58–5	58–6	58–7	58–8	58–9	58–10	58–11	58–12
No. of CRM	NIST	GBW 01390	GBW(E) 010139	GSBH40 068–93–4	GBW 01386	YSBS 23301–2	GBW 01391	YSBS 13108	GBW(E) 010141	GBW01 396	YSBS 16203–2006	YSBS 11273A-2001
C	0,0067	0,032	1,09	0,35	0,063	0,71	0,036	0,59	1,18	0,076	0,092	0,99
Si	0,008	3,38	1,12	0,41	1,49	0,90	2,47	0,25	0,48	0,97	0,36	0,576
Mn	0,0057	2,08	0,32	0,67	1,42	2,53	1,75	0,73	1,30	0,095	0,43	0,99
P	0,0025	0,018	0,054	0,036	0,062	0,0061	0,061	0,0098	0,068	0,027	0,101	0,017
S	0,0055	0,0087	0,0043	0,052	0,017	0,017	0,011	0,0257	0,0009	0,010	0,0136	0,0061
Cr	0,0072	0,63	2,12	1,04	0,22	3,08	0,49	0,014	0,16	1,89	0,42	1,47
Ni	0,041	0,060	2,06	1,94	0,37	0,13	0,16	0,008	2,17	0,99	0,18	0,053
Al	0,0007	0,0067	0,059	0,27	0,47	0,42	1,18		0,81	0,858		
Ti	0,0006	0,037	0,26	0,24	0,032	0,050	0,043		1,06	0,26		
Cu	0,0058	0,35	0,095	1,00	0,25	0,24	0,278	0,015	0,044	0,22	0,29	0,15
Co	0,0070			0,20						0,16		
Mo	0,0050		0,43	0,12		2,00			0,47	1,09		0,029
Nb			0,24	0,31		0,0050				0,21		
V	0,0006		0,43	0,20		0,48			0,05	0,24		
W			0,0080	0,6		0,65			0,053	2,076		
As	0,0002			0,0075				0,0019		0,0025		
B	0,0001	0,022	0,0019	0,010	0,013	0,0083	0,021		0,017	0,011		
Bi										0,0014		
Ca										0,0015		
N		0,005			0,0062	0,038	0,0042					
Pb	0,00002									0,0021		
Sb		0,0011			0,0012		0,0008			0,0007		
Sn		0,105		0,011	0,033		0,072			0,0016		
Zr		0,0035		0,039	0,0084	0,021	0,023			0,045		

Table B.1 b — Composition of the test samples (mass fraction, %)

ID	58–13	58–14	58–15	58–16	58–17	58–18	58–19	58–20	58–21	58–22	58–23
No. of CRM	YSBS 13307	GBW 01388	YSBS110 78-2003	YSBS 15006-98 1	YSBS 11279-2000	YSBS112 81-2000	GBW 01397	YSBS 23205 7-90-18/2	GBW 01398	YSBS 29395(B) B1	YSBS 29395(B) B2
C	0,14	0,12	0,0018	0,012	0,80	0,24	0,93	1,00	0,50	0,87	0,10
Si	0,45	0,16	0,0038	0,017	0,88	0,29	1,10	0,13	2,14	1,00	0,15
Mn	0,62	0,26	0,0057	0,034	0,30	0,72	1,20	2,17	0,80	0,099	2,01
P	0,134	0,040	0,0031	0,0019	0,02	0,014	0,011	0,0049	0,029	0,0028	0,067
S	0,022	0,038	0,0016	0,020	0,031	0,015	0,044	0,0044	0,021	0,078	0,0029
Cr	0,85	0,090	0,032	0,016	2,75	1,05	3,23	0,054	0,974	2,48	0,077
Ni		0,84	0,066	0,030	0,14	1,42	0,50	0,064	1,97	0,087	2,53
Al		1,47	0,0010	0,050	0,20	0,067	0,11	0,24	0,027	0,016	0,917
Ti		0,014			0,13	0,0019	0,32	0,41	0,082	0,51	0,081
Cu	0,39	0,157	0,022	0,021	0,055	0,15	0,36	0,367	0,30	0,044	0,86
Co			0,0041				0,50	0,036	0,24	0,060	0,51
Mo	0,38		0,019		0,98	0,16	0,29	0,078	0,83	0,043	0,49
Nb							0,25	0,027	0,12	0,41	0,023
V	0,19				0,14	0,013	0,061	0,38	0,47	0,035	0,42
W					0,24	0,057	0,81	1,80	1,56	2,38	0,075
As			0,0021		0,010	0,0078	0,007		0,0072		
B		0,009			0,006	0,0016	0,015		0,0047	0,0020	
Bi							0,013				
Ca									0,0011		
N		0,0025		0,015							
Pb							0,0023		0,017		
Sb		0,0004	0,0003		0,0019	0,0011	0,0023		0,0061		
Sn		0,017	0,0019		0,0058	0,058	0,011		0,018		
Zr		0,014					0,011	0,038	0,051		

Table B.2 — Results obtained in international interlaboratory test for carbon

Sample name	**Carbon contents** mass fraction %		**Repeatability limit** r mass fraction %	**Reproducibility limits**	
	Certified	Found		R_w mass fraction %	R mass fraction %
GBW 01386	0,063	0,062	0,0059	0,0099	0,0126
GBW01396	0,076	0,078	0,0028	0,0071	0,0139
YSBS 16203-2006	0,092	0,090	0,0033	0,0075	0,0140
YSBS 29395(B)B2	0,10	0,10	0,0061	0,0087	0,0115
GBW 01388	0,12	0,12	0,0031	0,0057	0,0228
YSBS 13307	0,14	0,13	0,0051	0,0099	0,0113
YSBS11281-2000	0,24	0,23	0,0110	0,0148	0,0235
GSBH40068-93-4	0,35	0,34	0,0220	0,0220	0,0460

Table B.3 — Results obtained in international interlaboratory test for silicon

Sample name	**Silicon contents** mass fraction %		**Repeatability limit** r mass fraction %	**Reproducibility limits**	
	Certified	Found		R_w mass fraction %	R mass fraction %
YSBS 23205 7-90-18/2	0,13	0,14	0,0025	0,0076	0,0120
YSBS 29395(B)B2	0,15	0,16	0,0054	0,0079	0,0166
GBW 01388	0,16	0,16	0,0051	0,0065	0,0184
YSBS 13108	0,25	0,22	0,0028	0,0102	0,0278
YSBS11281-2000	0,29	0,29	0,0025	0,0091	0,0262
YSBS 16203-2006	0,36	0,35	0,0068	0,0143	0,0362
GSBH40068-93-4	0,41	0,43	0,0109	0,0166	0,0420
YSBS 13307	0,45	0,44	0,0083	0,0148	0,0325
GBW(E)010141	0,48	0,41	0,0102	0,0105	0,0200
YSBS 11273A-2001	0,576	0,575	0,0178	0,0202	0,0555
YSBS 11279-2000	0,88	0,88	0,0230	0,0779	0,0779
YSBS 23301-2	0,90	0,93	0,0120	0,0376	0,0767
GBW01396	0,97	1,00	0,0484	0,0484	0,0980
YSBS 29395(B)B1	1,00	1,06	0,0247	0,0556	0,1000
GBW 01397	1,10	1,13	0,0572	0,0615	0,0989
GBW(E)010139	1,12	1,13	0,0246	0,0305	0,0757

Table B.4 — Results obtained in international interlaboratory test for manganese

Sample name	**Manganese contents** mass fraction %		**Repeatability limit** r mass fraction %	**Reproducibility limits**	
	Certified	Found		R_w mass fraction %	R mass fraction %
GBW01396	0,095	0,092	0,0059	0,0084	0,0117
YSBS 29395(B)B1	0,099	0,098	0,0019	0,0048	0,0058
GBW 01388	0,26	0,26	0,0133	0,0217	0,0363
YSBS 11279-2000	0,30	0,30	0,0077	0,0210	0,0451
GBW(E)010139	0,32	0,33	0,0073	0,0094	0,0399
YSBS 16203-2006	0,43	0,43	0,0190	0,0384	0,0631
YSBS 13307	0,62	0,64	0,0100	0,0480	0,1068
GSBH40068-93-4	0,67	0,68	0,0308	0,0357	0,0564

Table B.5 — Results obtained in international interlaboratory test for phosphorus

Sample name	**Phosphorus contents** mass fraction %		**Repeatability limit** r mass fraction %	**Reproducibility limits**	
	Certified	Found		R_w mass fraction %	R mass fraction %
GBW 01397	0,011	0,013	0,0005	0,0007	0,0041
YSBS11281-2000	0,014	0,014	0,0010	0,0011	0,0029
YSBS11273A-2001	0,017	0,015	0,0012	0,0025	0,0045
GBW 01390	0,018	0,018	0,0010	0,0011	0,0020
YSBS 11279-2000	0,020	0,019	0,0011	0,0042	0,0067
GBW01396	0,027	0,030	0,0010	0,0024	0,0061
GBW 01398	0,029	0,032	0,0023	0,0024	0,0057
GSBH40068-93-4	0,036	0,041	0,0018	0,0033	0,0107
GBW 01388	0,040	0,037	0,0024	0,0024	0,0033
GBW(E)010139	0,054	0,054	0,0029	0,0037	0,0121
GBW 01391	0,061	0,063	0,0034	0,0074	0,0137
GBW 01386	0,062	0,062	0,0070	0,0070	0,0118
GBW(E) 010141	0,068	0,065	0,0018	0,0055	0,0139
YSBS 29395(B)B2	0,067	0,067	0,0034	0,0086	0,0086

Table B.6 — Results obtained in international interlaboratory test for sulfur

Sample name	Sulfur contents mass fraction %		Repeatability limit r mass fraction %	Reproducibility limits	
	Certified	Found		R_w mass fraction %	R mass fraction %
GBW(E)010139	0,0043	0,0049	0,0003	0,0012	0,0024
YSBS 23205 7-90-18/2	0,0044	0,0049	0,0002	0,0003	0,0020
NIST	0,0055	0,0049	0,0009	0,0009	0,0019
YSBS 11273A-2001	0,0061	0,0060	0,0007	0,0011	0,0014
GBW 01390	0,0087	0,0085	0,0020	0,0020	0,0027
GBW01396	0,010	0,010	0,0015	0,0015	0,0034
GBW 01391	0,011	0,010	0,0018	0,0018	0,0027
YSBS 16203-2006	0,0136	0,0116	0,0008	0,0019	0,0028
YSBS11281-2000	0,015	0,013	0,0011	0,0018	0,0026
GBW 01386	0,017	0,016	0,0021	0,0030	0,0037
YSBS 23301-2	0,017	0,015	0,0011	0,0016	0,0026
YSBS 15006-98 1	0,020	0,016	0,0025	0,0030	0,0044
GBW 01398	0,021	0,022	0,0012	0,0014	0,0047
YSBS 13307	0,022	0,018	0,0010	0,0030	0,0040
YSBS 13108	0,0257	0,0217	0,0009	0,0017	0,0050
YSBS 11279-2000	0,031	0,030	0,0020	0,0042	0,0042
GBW 01388	0,038	0,034	0,0033	0,0034	0,0070
GBW 01397	0,044	0,046	0,0014	0,0028	0,0044
GSBH40068–93–4	0,052	0,054	0,0032	0,0033	0,0091

Table B.7 — Results obtained in international interlaboratory test for chromium

Sample name	**Chromium contents** mass fraction %		**Repeatability limit** r mass fraction %	**Reproducibility limits**	
	Certified	Found		R_w mass fraction %	R mass fraction %
NIST	0,0072	0,0079	0,0005	0,0028	0,0028
YSBS 13108	0,014	0,015	0,0004	0,0014	0,0019
YSBS 15006-98 1	0,016	0,017	0,0009	0,0032	0,0041
YSBS11078–2003	0,032	0,045	0,0032	0,0034	0,0132
YSBS 23205 7-90-18/2	0,054	0,049	0,0014	0,0014	0,0086
YSBS 29395(B)B2	0,077	0,076	0,0026	0,0033	0,0177
GBW 01388	0,090	0,091	0,0048	0,0074	0,0170
GBW(E)010141	0,16	0,17	0,0053	0,0166	0,0276
GBW 01386	0,22	0,22	0,0083	0,0091	0,0217
YSBS 16203-2006	0,42	0,41	0,0305	0,0305	0,0516
GBW 01391	0,49	0,50	0,0334	0,0334	0,0730
GBW 01390	0,63	0,64	0,0395	0,0395	0,0560

Table B.8 — Results obtained in international interlaboratory test for nickel

Sample name	**Nickel contents** mass fraction %		**Repeatability limit** r mass fraction %	**Reproducibility limits**	
	Certified	Found		R_w mass fraction %	R mass fraction %
YSBS11273A-2001	0,053	0,053	0,0025	0,0025	0,0043
GBW 01390	0,060	0,059	0,0014	0,0038	0,0130
YSBS 23205 7–90–18/2	0,064	0,075	0,0011	0,0013	0,0174
YSBS11078–2003	0,066	0,062	0,0033	0,0051	0,0090
YSBS 29395(B)B1	0,087	0,100	0,0024	0,0083	0,0203
YSBS 23301–2	0,13	0,14	0,0087	0,0087	0,0275
YSBS 11279–2000	0,14	0,14	0,0031	0,0034	0,0195
GBW 01391	0,16	0,16	0,0036	0,0091	0,0298
YSBS 16203–2006	0,18	0,18	0,0116	0,0116	0,0272
GBW 01386	0,37	0,36	0,0155	0,0228	0,0541
GBW 01397	0,50	0,53	0,0124	0,0133	0,0536

Table B.9 — Results obtained in international interlaboratory test for aluminium

Sample name	**Aluminium contents** mass fraction %		**Repeatability limit** r mass fraction %	**Reproducibility limits**	
	Certified	Found		R_w mass fraction %	R mass fraction %
GBW 01390	0,0067	0,0059	0,0005	0,0008	0,0024
YSBS 29395(B)B1	0,016	0,018	0,0009	0,0015	0,0063
GBW 01398	0,027	0,028	0,0016	0,0029	0,0090
YSBS 15006–98 1	0,050	0,055	0,0044	0,0061	0,0126
GBW(E)010139	0,059	0,063	0,0033	0,0041	0,0121
YSBS11281–2000	0,067	0,067	0,0023	0,0052	0,0139
GBW 01397	0,11	0,11	0,0059	0,0061	0,0183
YSBS 11279–2000	0,20	0,20	0,0055	0,0132	0,0297
YSBS 23205 7–90–18/2	0,24	0,23	0,0093	0,0167	0,0194
GSBH40068–93–4	0,27	0,28	0,0168	0,0168	0,0410
YSBS 23301–2	0,42	0,44	0,0240	0,0386	0,0726
GBW 01386	0,47	0,46	0,0162	0,0272	0,0353
GBW(E)010141	0,81	0,80	0,0127	0,0325	0,0450
GBW01396	0,858	0,886	0,0135	0,0556	0,1053
YSBS 29395(B)B2	0,917	0,907	0,0280	0,0480	0,0672

Table B.10 — Results obtained in international interlaboratory test for titanium

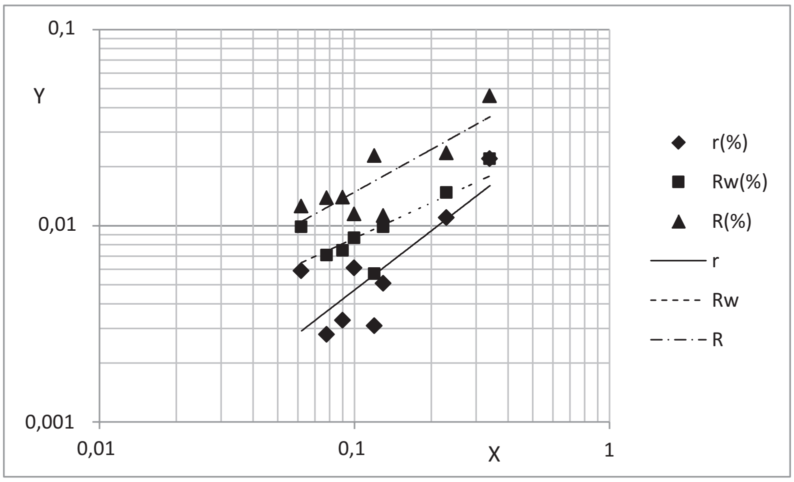
Sample name	**Titanium contents** mass fraction %		**Repeatability limit** r mass fraction %	**Reproducibility limits**	
	Certified	Found		R_w mass fraction %	R mass fraction %
GBW 01388	0,014	0,014	0,0019	0,0034	0,0043
GBW 01386	0,032	0,033	0,0032	0,0034	0,0055
GBW 01390	0,037	0,039	0,0031	0,0058	0,0060
GBW 01391	0,043	0,046	0,0030	0,0046	0,0104
YSBS 23301–2	0,050	0,049	0,0046	0,0050	0,0153
YSBS 29395(B)B2	0,081	0,079	0,0028	0,0074	0,0138
GBW 01398	0,082	0,083	0,0039	0,0055	0,0232
YSBS 11279–2000	0,13	0,13	0,0094	0,0134	0,0228

Table B.11 — Results obtained in international interlaboratory test for copper

Sample name	**Copper contents** mass fraction %		**Repeatability limit** r mass fraction %	**Reproducibility limits**	
	Certified	Found		R_w mass fraction %	R mass fraction %
NIST	0,0058	0,0058	0,0004	0,0004	0,0005
YSBS 13108	0,015	0,014	0,0003	0,0021	0,0021
YSBS 15006–98 1	0,021	0,021	0,0011	0,0013	0,0073
YSBS11078–2003	0,022	0,023	0,0015	0,0015	0,0096
GBW(E)010141	0,044	0,047	0,0005	0,0020	0,0095
YSBS 29395(B)B1	0,044	0,050	0,0014	0,0038	0,0276
YSBS 11279–2000	0,055	0,059	0,0026	0,0035	0,0208
GBW(E)010139	0,095	0,096	0,0018	0,0054	0,0162
YSBS 11273A-2001	0,15	0,15	0,0018	0,0088	0,0193
YSBS11281–2000	0,15	0,16	0,0051	0,0070	0,0146
GBW 01388	0,157	0,160	0,0028	0,0068	0,0189
GBW01396	0,22	0,22	0,0032	0,0137	0,0397
YSBS 23301–2	0,24	0,25	0,0055	0,0077	0,0363
GBW 01386	0,25	0,25	0,0064	0,0094	0,0258
GBW 01391	0,278	0,283	0,0064	0,0064	0,0260
YSBS 16203–2006	0,29	0,29	0,0020	0,0020	0,0267
GBW 01398	0,30	0,31	0,0083	0,0164	0,0379
GBW 01390	0,35	0,36	0,0063	0,0098	0,0570
GBW 01397	0,36	0,38	0,0044	0,0130	0,0397
YSBS 23205 7–90–18/2	0,367	0,390	0,0053	0,0069	0,0296
YSBS 13307	0,39	0,39	0,0066	0,0142	0,0309
YSBS 29395(B)B2	0,86	0,87	0,0102	0,0281	0,0593
GSBH40068–93–4	1,00	1,00	0,0191	0,0299	0,0920

Annex C
(informative)

Graphical representation of precision data



Key

X carbon content, mass fraction (%)

Y precision, mass fraction (%)

Figure C.1 — Logarithmic relationship between carbon content (*m*) and repeatability limit (*r*) or reproducibility limits (R_W and *R*)

$$\lg r = 1{,}016 \lg m - 1{,}310 \qquad R = 0{,}8209$$

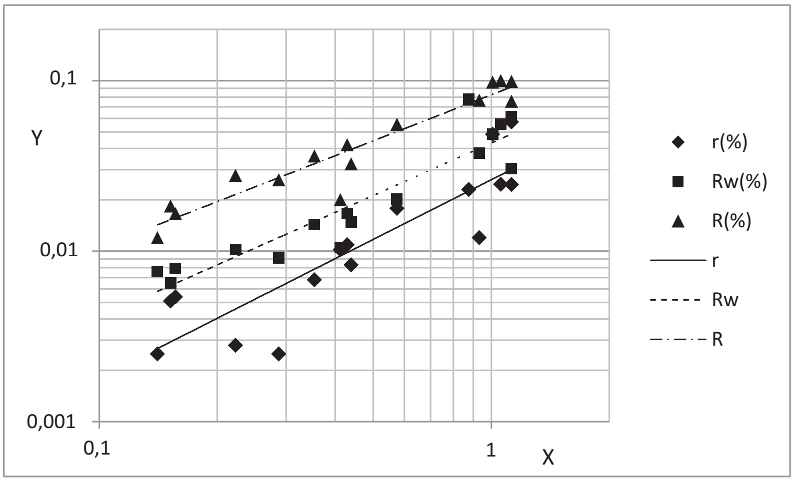
$$\lg R_W = 0{,}607 \lg m - 1{,}457 \qquad R = 0{,}7947$$

$$\lg R = 0{,}719 \lg m - 1{,}107 \qquad R = 0{,}8355$$

where

m is the general mean, expressed as a mass fraction %;

R is the correlation coefficient.



Key

X silicon content, mass fraction %

Y precision, mass fraction %

Figure C.2 — Logarithmic relationship between silicon content (m) and repeatability limit (r) or reproducibility limits (R_W and R)

$\lg r = 1{,}304 \lg m - 1{,}553$ $R = 0{,}8930$

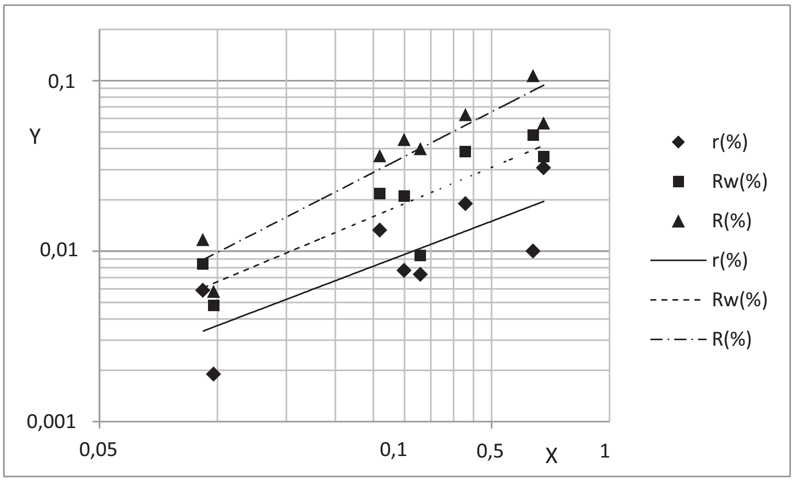
$\lg R_W = 1{,}118 \lg m - 1{,}346$ $R = 0{,}9275$

$\lg R = 0{,}904 \lg m - 1{,}081$ $R = 0{,}9463$

where

m is the general mean, expressed as a mass fraction %;

R is the correlation coefficient.



Key

X manganese content, mass fraction %

Y Precision, mass fraction %

Figure C.3 — Logarithmic relationship between manganese content (m) and repeatability limit (r) or reproducibility limits (R_W and R)

$\lg r = 1{,}146 \lg m - 1{,}466 \qquad R = 0{,}5455$

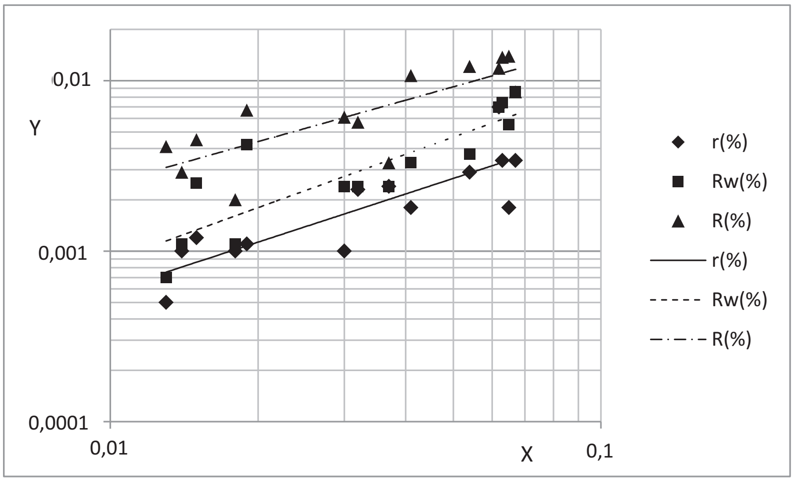
$\lg R_W = 1{,}119 \lg m - 1{,}163 \qquad R = 0{,}7749$

$\lg R = 1{,}313 \lg m - 0{,}781 \qquad R = 0{,}7579$

where

m is the general mean, expressed as a mass fraction %;

R is the correlation coefficient.



Key

X phosphorus content, mass fraction %

Y Precision, mass fraction %

Figure C.4 — Logarithmic relationship between phosphorus content (*m*) and repeatability limit (*r*) or reproducibility limits (R_W and *R*)

$$\lg r = 0{,}933 \lg m - 1{,}359 \qquad R = 0{,}8499$$

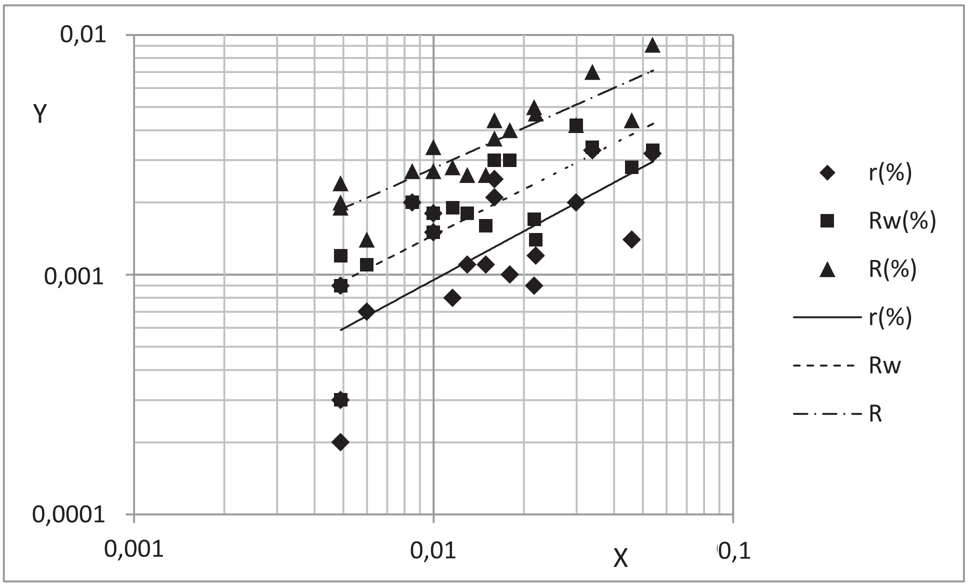
$$\lg R_W = 1{,}034 \lg m - 0{,}985 \qquad R = 0{,}8502$$

$$\lg R = 0{,}796 \lg m - 1{,}001 \qquad R = 0{,}7958$$

where

m is the general mean, expressed as a mass fraction %;

R is the correlation coefficient.



Key

X sulfur content, mass fraction %

Y Precision, mass fraction %

Figure C.5 — Logarithmic relationship between sulfur content (*m*) and repeatability limit (*r*) or reproducibility limits (R_w and *R*)

$\lg r = 0{,}679 \lg m - 1{,}666$ $R = 0{,}6845$

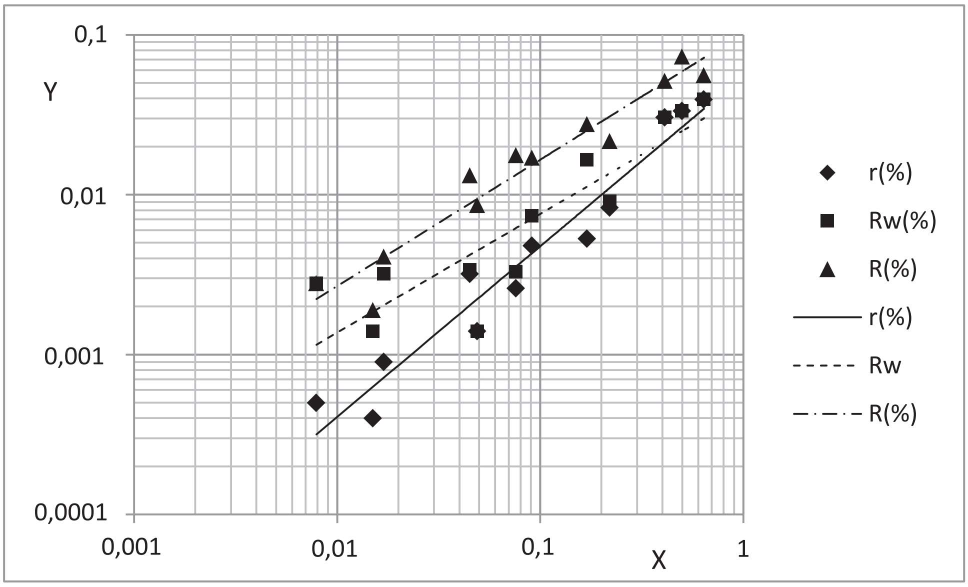
$\lg R_\mathrm{w} = 0{,}638 \lg m - 1{,}561$ $R = 0{,}7624$

$\lg R = 0{,}559 \lg m - 1{,}440$ $R = 0{,}8895$

where

m is the general mean, expressed as a mass fraction %;

R is the correlation coefficient.



Key

X chromium content, mass fraction %

Y Precision, mass fraction %

Figure C.6 — Logarithmic relationship between chromium content, *m*, and repeatability limit, *r*, or reproducibility limits, R_W and *R*

$\lg r = 1{,}066 \lg m - 1{,}257$ $R = 0{,}9734$

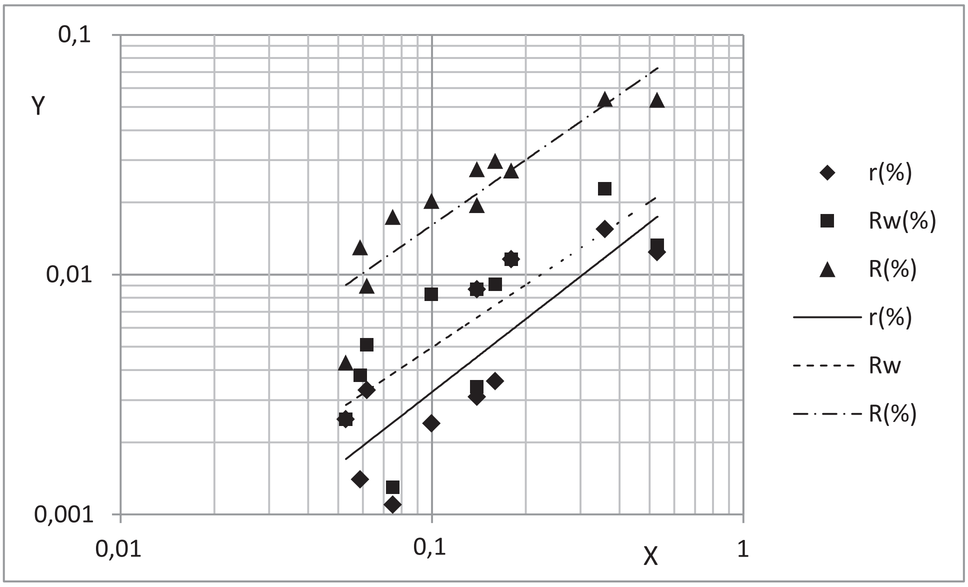
$\lg R_W = 0{,}740 \lg m - 1{,}380$ $R = 0{,}8862$

$\lg R = 0{,}790 \lg m - 0{,}992$ $R = 0{,}9676$

where

m is the general mean, expressed as a mass fraction %;

R is the correlation coefficient.



Key

X nickel content, mass fraction %

Y Precision, mass fraction %

Figure C.7 — Logarithmic relationship between nickel content (m) and repeatability limit (*r*) or reproducibility limits (R_W and *R*)

$\lg r = 1{,}012 \lg m - 1{,}474$ $R = 0{,}8287$

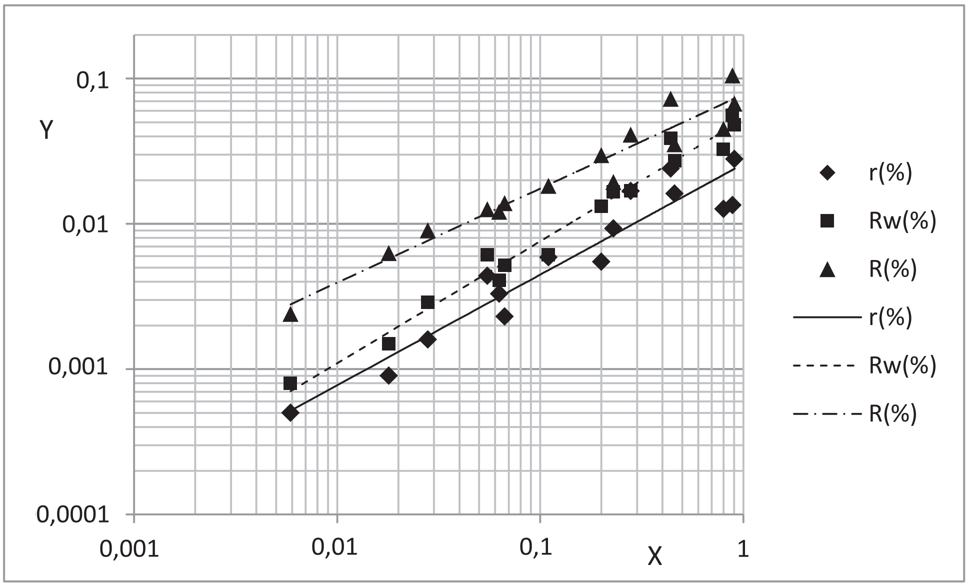
$\lg R_\mathrm{W} = 0{,}867 \lg m - 1{,}433$ $R = 0{,}7728$

$\lg R = 0{,}903 \lg m - 0{,}888$ $R = 0{,}9005$

where

m is the general mean, expressed as a mass fraction %;

R is the correlation coefficient.



Key

X aluminium content, mass fraction %

Y Precision, mass fraction %

Figure C.8 — Logarithmic relationship between aluminium content (*m*) and repeatability limit (*r*) or reproducibility limits (R_W and *R*)

$\lg r = 0{,}759 \lg m - 1{,}588$ $R = 0{,}9549$

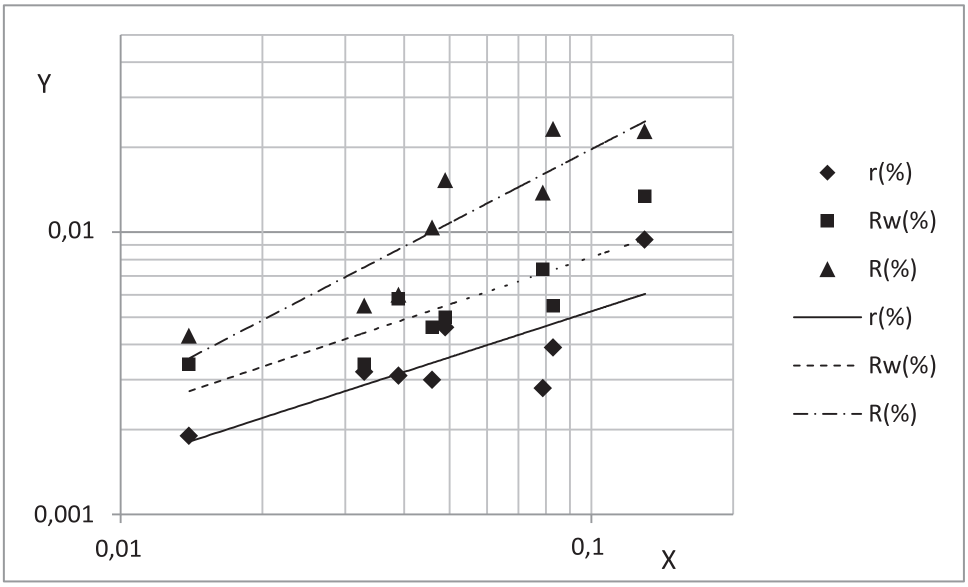
$\lg R_W = 0{,}838 \lg m - 1{,}279$ $R = 0{,}9888$

$\lg R = 0{,}646 \lg m - 1{,}109$ $R = 0{,}9677$

where

m is the general mean, expressed as a mass fraction %;

R is the correlation coefficient.



Key

X Titanium content, mass fraction %

Y Precision, mass fraction %

Figure C.9 — Logarithmic relationship between titanium content (*m*) and repeatability limit (*r*) or reproducibility limits (R_W and *R*)

$\lg r = 0{,}536 \lg m - 2{,}192$ $R = 0{,}7862$

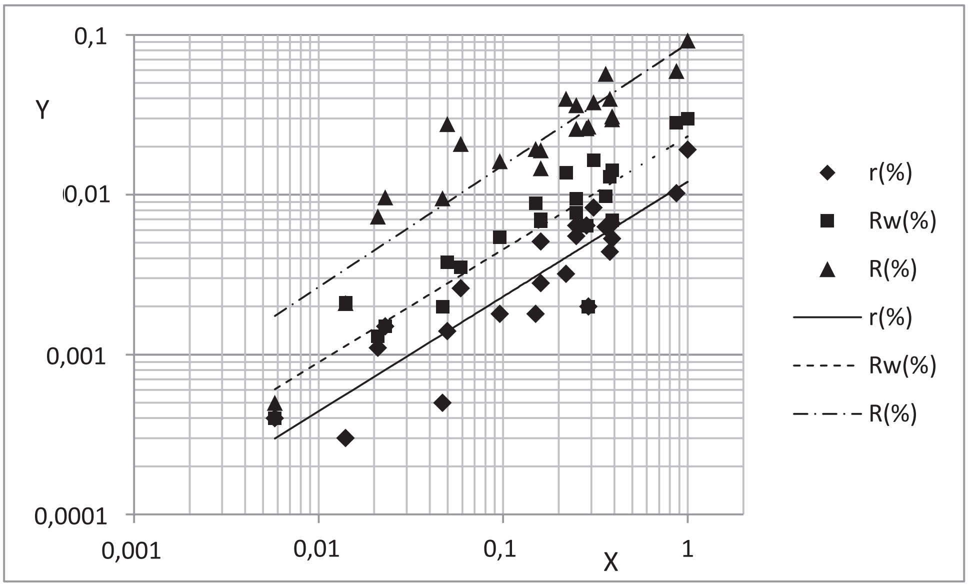
$\lg R_W = 0{,}536 \lg m - 1{,}970$ $R = 0{,}8520$

$\lg R = 0{,}878 \lg m - 1{,}272$ $R = 0{,}9076$

where

m is the general mean, expressed as a mass fraction %;

R is the correlation coefficient.



Key

X Copper content, mass fraction %

Y Precision, mass fraction %

Figure C.10 — Logarithmic relationship between copper content (*m*) and repeatability limit (*r*) or reproducibility limits (R_W and *R*)

$\lg r = 0{,}717 \lg m - 1{,}920$ $R = 0{,}9026$

$\lg R_W = 0{,}708 \lg m - 1{,}634$ $R = 0{,}8991$

$\lg R = 0{,}760 \lg m - 1{,}056$ $R = 0{,}9011$

where

m is the general mean, expressed as a mass fraction %;

R is the correlation coefficient.

Bibliography

[1] ISO 16962, *Surface chemical analysis — Analysis of zinc- and/or aluminium-based metallic coatings by glow-discharge optical-emission spectrometry*

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