INTERNATIONAL STANDARD

ISO 9683-2

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Iron ores — Determination of vanadium —

Part 2:
Flame atomic absorption spectrometric methods

Minerais de fer — Dosage du vanadium —

Partie 2: Méthodes par spectrométrie d'absorption atomique dans la flamme

Reference number
ISO 9683-2:2009(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

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 9683-2 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

ISO 9683 consists of the following parts, under the general title *Iron ores — Determination of vanadium*:

- *Part 1: BPHA spectrophotometric method*
- *Part 2: Flame atomic absorption spectrometric methods*

INTERNATIONAL STANDARD **ISO 9683-2:2009(E)**

Iron ores — Determination of vanadium —

Part 2:
Flame atomic absorption spectrometric methods

WARNING — This part of ISO 9683 may involve hazardous materials, operations and equipment. This part of ISO 9683 does not purport to address all of the safety issues associated with its use. It is the responsibility of the user to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 9683 specifies two flame atomic absorption spectrometric methods for the determination of the mass fraction of vanadium in iron ores.

Method 1 is applicable to mass fractions of vanadium between 0,004 % and 0,06 %, and Method 2 is applicable to mass fractions of vanadium between 0,06 % and 0,5 %, in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

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, *Laboratory glassware — Single-volume pipettes*

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

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis*

3 Principle

3.1 Dissolution

The test portion is decomposed by digestion with hydrochloric acid in a polytetrafluoroethylene (PTFE) beaker, hydrofluoric and nitric acids are added and the solution is evaporated to dryness. Hydrochloric and boric acids are added and the solution is again evaporated to dryness. The salts are dissolved in hydrochloric and nitric acids (Method 1), or hydrochloric acid (Method 2), and the solution is filtered. The residue is ignited and fused with sodium carbonate and the cooled melt is dissolved in the test solution.

3.2 Determination

3.2.1 Extraction (Method 1 only)

The solution is oxidized with cerium(IV) solution, and phosphoric acid and sodium tungstate solution are added. The vanadium complex is extracted with a 1 + 1 mixture of 1-pentanol and 4-methyl-2-pentanone. The solvent phase is treated with water and then ascorbic acid solution to return the vanadium to an aqueous phase.

3.2.2 Measurement (Methods 1 and 2)

Aluminium solution is added to the test solution, which is diluted to volume. The solution is aspirated into a dinitrogen oxide/acetylene flame in an atomic absorption spectrometer and the absorbance is measured at a wavelength of approximately 318,5 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

4.1 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml.

4.2 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 1.

4.3 Hydrofluoric acid, ρ 1,13 g/ml, 40 % (by mass) or 1,19 g/ml, 48 % (by mass).

4.4 Nitric acid, ρ 1,4 g/ml.

4.5 Nitric acid, ρ 1,4 g/ml, diluted 1 + 1. (Method 1 only.)

4.6 Phosphoric acid, ρ 1,7 g/ml, diluted 1 + 2. (Method 1 only.)

4.7 Sodium carbonate (Na_2CO_3), anhydrous powder.

4.8 Boric acid (H_3BO_3).

4.9 Di-ammonium cerium(IV) nitrate [$(NH_4)_2Ce(NO_3)_6$], 20 g/l solution. (Method 1 only.)

Dissolve 2,0 g of di-ammonium cerium(IV) nitrate in a mixture of 15 ml of nitric acid (4.5) and 85 ml of water.

4.10 Sodium tungstate ($Na_2WO_4 \cdot 2H_2O$), 165 g/l solution. (Method 1 only.)

Dissolve 16,5 g of sodium tungstate dihydrate in approximately 70 ml of water, dilute to 100 ml and mix.

4.11 Ascorbic acid ($C_6H_8O_6$), 10 g/l solution. (Method 1 only.)

Prepare freshly for each series of tests.

4.12 1-Pentanol (*n*-amyl alcohol, 1-pentan-1-ol). (Method 1 only.)

4.13 4-Methyl-2-pentanone (methyl isobutyl ketone, MIBK). (Method 1 only.)

4.14 Mixed solvent. (Method 1 only.)

Prepare a 1 + 1 volume ratio mixture of 1-pentanol (4.12) and MIBK (4.13).

4.15 Aluminium chloride ($AlCl_3 \cdot 6H_2O$), 220 g/l solution.

Dissolve 220 g of aluminium chloride hexahydrate in water, add 50 ml of hydrochloric acid (4.1), dilute to 1 000 ml with water and mix.

4.16 Iron oxide, high purity (containing less than 0,002 % V).

4.17 Iron background solution. (Method 2 only.)

Dissolve 90 g of high-purity iron oxide (4.16) in 750 ml of hydrochloric acid (4.1) while heating. Cool and carefully add a solution of 30 g of sodium carbonate (4.7) in 200 ml of water. Dilute with water to a final volume of approximately 1 000 ml and mix.

NOTE The use of an equivalent quantity of metallic iron (of similarly low vanadium content), in conjunction with a suitable oxidant, is permissible.

4.18 Vanadium standard solution, 1 mg V/ml.

Dry ammonium monovanadate (NH_4VO_3) in an air oven at 100 ºC for 1 h and cool to room temperature. Dissolve 2,296 g of the dried product in approximately 600 ml of water, transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

4.19 Diluted vanadium standard solution, 0,2 mg V/ml. (Method 1 only.)

Transfer 20,0 ml of vanadium standard solution (4.18) to a 100 ml one-mark volumetric flask, dilute to volume with water and mix.

4.20 Vanadium calibration solutions. (Method 1 only.)

Transfer 1,8 g of high-purity iron oxide (4.16) to each of five 100 ml PTFE beakers (5.1). Proceed according to 7.5.1, incorporating the operation in 7.5.2 to dissolve the evaporated salts.

Transfer 0 ml; 1,00 ml; 2,00 ml: 4,00 ml and 6,00 ml of the diluted vanadium standard solution (4.19), respectively, to the beakers. Omit the residue-processing steps and add 10 ml of nitric acid (4.5) and 25 ml of water. Evaporate to 50 ml and proceed as specified in 7.5.3.

4.21 Vanadium calibration solutions. (Method 2 only.)

Transfer 0 ml; 1,00 ml; 2,00 ml; 4,00 ml and 6,00 ml of vanadium standard solution (4.18), respectively, to five 100 ml one-mark volumetric flasks. Add 10 ml of iron background solution (4.17), 4,0 ml of aluminium chloride solution (4.15) and 0,5 g of boric acid (4.8) to each flask. Dilute to volume with water and mix.

5 Apparatus

Ordinary laboratory apparatus, including one-mark pipettes and one mark-volumetric flasks complying with the specifications of ISO 648 and ISO 1042, respectively (unless otherwise indicated), and the following.

5.1 Polytetrafluoroethylene (PTFE) beakers, of capacity 100 ml, preferably low form.

5.2 Platinum crucibles, of minimum capacity 20 ml.

5.3 Muffle furnace.

5.4 Separating funnels, 100 ml (preferably graduated).

5.5 Atomic absorption spectrometer, equipped with a dinitrogen oxide/acetylene burner.

The atomic absorption spectrometer used in this method is satisfactory if it meets the following criteria.

a) **Minimum sensitivity**: the absorbance of the most concentrated vanadium calibration solution (4.21) is at least 0,25 (see Note 2).

b) **Graph linearity**: the slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) is not less than 0,7 of the value of the slope for the bottom 20 % of the concentration range determined in the same way.

c) **Minimum stability**: the standard deviation of the absorbance of the most concentrated calibration solution and that of the zero calibration solution, each being calculated from a sufficient number of repetitive measurements, are less than 1,5 % and 0,5 %, respectively, of the mean value of the absorbance of the most concentrated solution.

The use of a strip-chart recorder and/or digital read-out device is recommended to evaluate criteria a), b) and c) and for all subsequent measurements.

NOTE 1 Instrument parameters:

V-hollow cathode lamp, mA	10
Wavelength, nm	318,5
Dinitrogen oxide flow rate, l/min	9,5
Acetylene flow rate, l/min	3,8

In systems where the values shown above for gas flow rates do not apply, the given ratio of the flow rates (5:2) can still be a useful guideline.

The slit width should be sufficiently narrow to substantially reject the stronger emitting but weaker absorbing line at 318,4 nm.

NOTE 2 To check the instrument criteria when only Method 1 is involved, prepare the highest calibration solution specified for Method 2, omitting the iron background solution.

6 Sampling and samples

6.1 Laboratory sample

For analysis, use a laboratory sample of minus 100 µm particle size which has been taken and prepared in accordance with ISO 3082. In the case of ores having significant contents of combined water or oxidizable compounds, use a particle size of minus 160 µm.

NOTE Guidance on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

6.2 Preparation of predried test samples

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a way that it is representative of the whole contents of the container. Dry the test sample at 105 °C ± 2 °C, as specified in ISO 7764. (This is the predried test sample.)

7 Procedure

7.1 Number of determinations

Carry out the analysis at least in duplicate in accordance with Annex A, independently, on one predried test sample.

NOTE The expression “independently” means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure is carried out either by the same operator at a different time or by a different operator including, in both cases, appropriate recalibration.

7.2 Safety precautions

WARNING — Follow the manufacturer's instructions for igniting and extinguishing the dinitrogen oxide/acetylene flame to avoid possible explosion hazards. Wear tinted safety glasses whenever the burner is in operation.

7.3 Test portion

Taking several increments, weigh, to the nearest 0,000 5 g, approximately 2 g (Method 1) or 1 g (Method 2) of the predried test sample obtained in accordance with 6.2.

The test portion should be taken and weighed quickly, in order to avoid reabsorption of moisture.

7.4 Blank test and check test

In each run, one blank test and one analysis of a certified reference material of the same type of ore shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions. A predried test sample of the certified reference material shall be prepared as specified in 6.2.

The certified reference material should be of the same type as the sample to be analysed and the properties of the two materials should be sufficiently similar to ensure that, in both cases, no significant changes in the analytical procedure will become necessary.

When the analysis is carried out on several samples at the same time, the blank value may be represented by one test, provided that the procedure is the same and the reagents are from the same reagent bottles.

When the analysis is carried out on several samples of the same type of ore at the same time, the analytical value of one certified reference material may be used.

7.5 Determination Method 1 (mass fraction of vanadium between 0,004 % and 0,06 %)

7.5.1 Decomposition of the test portion

Transfer the test portion (7.3) into a 100 ml PTFE beaker (5.1) and add 25 ml of hydrochloric acid (4.1). Cover and mix, and then digest just at the boiling point for 1 h. Add 0,25 ml of nitric acid (4.4), mix and digest for a further 10 min. Add 10 ml of hydrofluoric acid (4.3) and evaporate to dryness. Add 10 ml of hydrochloric acid (4.1) and 0,5 g of boric acid (4.8), and repeat the evaporation to dryness.

7.5.2 Dissolution of salts and residue treatment

Add 2 ml of hydrochloric acid (4.2), 4 ml of nitric acid (4.5) and 10 ml of water to the PTFE beaker. Add a stirring rod and heat to dissolve the salts. Filter through a close-texture filter paper into a 150 ml low-form beaker marked at the 50 ml capacity point, adding a further 10 ml of nitric acid (4.5) and 25 ml of water to the original PTFE beaker, while heating, to facilitate cleaning of the beaker and the transfer of any residue to the paper. Wash the residue with water.

Transfer the residue to a platinum crucible (5.2), dry, ash and ignite the residue at 600 °C to 700 °C, then fuse over a burner or for 30 min in a muffle furnace (5.3) at 1 000 °C in 0,3 g of sodium carbonate (4.7). Transfer the cooled crucible to the main solution using a stirring rod and leach the melt while evaporating the solution. Remove and rinse the crucible as soon as leaching is complete and continue the evaporation to a volume of 50 ml.

NOTE The leach of the fused residue in the main solution can attack the crucible if the evaporation is accidentally carried too far.

7.5.3 Extraction of vanadium

Add 1 ml of di-ammonium cerium(IV) nitrate solution (4.9) to the solution prepared in 7.5.2, and mix. Cover, heat until boiling starts, then remove from the heater and add 5 ml of phosphoric acid (4.6) and 2,5 ml of sodium tungstate solution (4.10). Replace the cover and heat to maintain gentle boiling for 10 min.

Cool, transfer to a 100 ml separating funnel (5.4) with minimum washing, and retain the beaker for subsequent use. Add 20 ml of mixed solvent (4.14), shake for 40 s and allow to stand for 1 min. Discard the lower aqueous phase, add 20 ml of water to the separating funnel and shake it for 30 s. Allow the phases to separate for 1 min and transfer the lower aqueous phase to the 150 ml beaker calibrated at the 20 ml point. Add a further 20 ml of water to the separating funnel, shake it for 30 s and combine this aqueous phase with the first. Add 10 ml of ascorbic acid solution (4.11) to the funnel containing the solvent phase and shake for 30 s. Allow the phases to separate and transfer the lower aqueous phase to the solution in the beaker. Repeat the extraction of vanadium with a second 10 ml portion of ascorbic acid solution and transfer the aqueous phase to the beaker.

The separation of phases should occur without the formation of emulsion. If emulsion does form, add 0,1 ml of nitric acid (4.5) and shake for 15 s.

NOTE Normally, the solvent phase will now be colourless, indicating that the vanadium has been effectively removed. However, with ores containing organic matter, a residual colour which is not due to vanadium can remain. This can usually be ignored, but if it is considered excessive, it can be prevented by igniting the test portion in a silica or porcelain crucible at 700 °C for 30 min prior to dissolution.

To the combined water and ascorbic acid strip solutions, add 1 ml of aluminium chloride solution (4.15) and evaporate to approximately 20 ml. Cool, transfer to a 25 ml one-mark volumetric flask, dilute to volume with water and mix.

7.5.4 Adjustment of the atomic absorption spectrometer

Set the wavelength for vanadium (318,5 nm) to obtain minimum absorbance and, following the manufacturer's instructions, fit the correct burner and light the flame. After 10 min of preheating the burner, aspirate the calibration solution of highest vanadium content (4.21, Method 2) and, following the manufacturer's instructions concerning the minimum flow rate of acetylene, adjust the gas flows and burner height to obtain maximum absorbance.

Aspirate water and the calibration solution to establish that the absorbance reading is not drifting. Set the reading for water to zero absorbance and evaluate the criteria in 5.5.

7.5.5 Atomic absorption measurements

Aspirate the calibration and test solutions in order of increasing absorption, starting with the blank test solution and the zero calibration solution.

Aspirate water between readings, reset to zero if necessary, and record the readings when stable responses are obtained. Repeat the measurements at least twice.

Calculate the average absorbance of the calibration and test solutions. Correct the absorbance values obtained for the calibration solutions by subtracting the absorbance of the zero calibration solution, and prepare a calibration graph by plotting the net absorbance values against micrograms of vanadium per millilitre. If the graph is substantially linear, subtract the absorbance obtained for the blank test from the absorbance obtained for the test solution and, using the graph, convert the net absorbance to micrograms of vanadium per millilitre.

If any curvature obtained approaches the limit specified in 5.5 b), replot the graph using uncorrected values for all solutions and establish the concentration of the zero calibration solution from the intercept of the graph on the negative side of the concentration axis. Add this value to the nominal concentration values of the calibration solutions and replot the graph to pass through the origin. Determine from the graph the concentration of vanadium, in micrograms per millilitre, in the blank test solution and test solution, respectively, and correct the concentration of the test solution with the concentration of the blank test solution.

7.6 Determination Method 2 (mass fraction of vanadium between 0,06 % and 0,5 %)

7.6.1 Decomposition of the test portion

Decompose the test portion as specified in 7.5.1.

7.6.2 Dissolution of salts and residue treatment

Add 10 ml of hydrochloric acid (4.2) and a stirring rod to the PTFE beaker and heat to dissolve the salts. Add 20 ml of water and continue heating for several minutes, if necessary, to complete the dissolution. Filter through a close-texture filter paper into a 100 ml one-mark volumetric flask and wash the residue with water. Transfer the residue to a platinum crucible (5.2), dry, ash and ignite the residue, then fuse over a burner or for 30 min in a muffle furnace (5.3) at 1 000 °C in 0,3 g of sodium carbonate (4.7). Cool the melt and treat in the covered crucible with 5 ml of water and 5 ml of hydrochloric acid (4.2). Warm to dissolve the fused salts and combine with the main test solution. Cool, add 4 ml of aluminium chloride solution (4.15), dilute to volume with water and mix.

Continue with the determination of the vanadium content as specified in 7.5.4 and 7.5.5.

8 Expression of results

8.1 Calculation of mass fraction of vanadium

The mass fraction of vanadium, w_{V}, expressed as a percentage, is calculated from Equations (1) or (2):

Method 1:

$$w_{\mathrm{V}} = \frac{2{,}5\rho_{\mathrm{V}}}{1\,000m} \tag{1}$$

Method 2:

$$w_{\mathrm{V}} = \frac{\rho_{\mathrm{V}}}{100m} \tag{2}$$

where

ρ_{V} is the concentration, in micrograms per millilitre, of vanadium in the test solution;

m is the mass, in grams, of the test portion.

8.2 General treatment of results

8.2.1 Repeatability and permissible tolerance

The precision of this analytical method is expressed by the following regression equations:

Method 1

$$R_\mathrm{d} = 0{,}003\,3X^{0{,}280\,8} \tag{3}$$

$$P = 0{,}022\,1X^{0{,}604\,1} \tag{4}$$

$$\sigma_\mathrm{d} = 0{,}001\,2X^{0{,}280\,8} \tag{5}$$

$$\sigma_\mathrm{L} = 0{,}010\,8X^{0{,}719\,1} \tag{6}$$

Method 2

$$R_\mathrm{d} = 0{,}017\,2X^{0{,}424\,4} \tag{7}$$

$$P = 0{,}077\,0X^{0{,}775\,7} \tag{8}$$

$$\sigma_\mathrm{d} = 0{,}006\,1X^{0{,}424\,4} \tag{9}$$

$$\sigma_\mathrm{L} = 0{,}027\,0X^{0{,}813\,5} \tag{10}$$

where

X is the mass fraction of vanadium, expressed as a percentage, of the predried test sample, calculated as follows:

- within-laboratory Equations (3), (5), (7) and (9): the arithmetic mean of the duplicate values;
- between-laboratories Equations (4), (6), (8) and (10): the arithmetic mean of the final results (8.2.3) of the two laboratories;

R_d is the independent duplicate limit;

P is the permissible tolerance between laboratories;

σ_d is the independent duplicate standard deviation;

σ_L is the between-laboratories standard deviation.

Additional information is given in Annexes B and C.

8.2.2 Determination of analytical result

Having computed the independent duplicate results according to Equation (1) or (2), compare them with the independent duplicate limit (R_d), using the procedure given in Annex A, and obtain the final laboratory result, μ_c (see 8.2.5).

8.2.3 Between-laboratories precision

Between-laboratories precision is used to determine the agreement between the final results reported by two laboratories. The assumption is that both laboratories followed the procedure described in 8.2.2.

Compute the following quantity:

$$\mu_{1,2} = \frac{\mu_1 + \mu_2}{2} \tag{11}$$

where

μ_1 is the final result reported by laboratory 1;

μ_2 is the final result reported by laboratory 2;

$\mu_{1,2}$ is the mean of the final results.

Substitute $\mu_{1,2}$ for X in Equation (4) or (8) and calculate P.

If $|\mu_1 - \mu_2| \leqslant P$, the final results are in agreement.

8.2.4 Check for trueness

The trueness of the analytical method shall be checked by applying it to a certified reference material (CRM) or a reference material (RM) (see the second paragraph of 7.4). Calculate the analytical result (μ_c) for the RM/CRM using the procedures in 8.1, 8.2.1, 8.2.2 and 8.2.3, and compare it with the reference or certified value, A_c. There are two possibilities:

a) $|\mu_c - A_c| \leqslant C$ in which case the difference between the reported result and the reference/certified value is statistically insignificant;

b) $|\mu_c - A_c| > C$ in which case the difference between the reported result and the reference/certified value is statistically significant;

where

μ_c is the analytical result for the CRM/RM;

A_c is the certified/reference value for the CRM/RM;

C is a value dependent on the type of CRM/RM used.

Certified reference materials used for this purpose should be prepared and certified in accordance with ISO Guide 35:2006, *Reference materials — General and statistical principles for certification*.

For a CRM/RM certified by an interlaboratory test programme, C shall be calculated as follows:

$$C = 2\sqrt{\frac{s_c^2}{N_c} + \sigma_L^2 + \frac{\sigma_d^2}{n}} \tag{12}$$

where

s_c is the standard deviation of laboratory means (each value for calculating the standard deviation is the average value in each certifying laboratory) of the CRM/RM;

N_c is the number of certifying laboratories;

n is the number of replicate determinations carried out on the CRM/RM.

For CRMs certified by only one laboratory, C shall be calculated as follows:

$$C = \sqrt{2\sigma_{\mathrm{L}}^2 + \frac{\sigma_{\mathrm{d}}^2}{n}} \qquad (13)$$

A CRM certified by only one laboratory should be avoided, unless it is known to have an unbiased certified value.

8.2.5 Calculation of final result

The final result is the arithmetic mean of the acceptable analytical values for the test sample, or as otherwise determined by the operations specified in Annex A, calculated to five decimal places and rounded off to the third decimal place as follows:

a) where the figure in the fourth decimal place is less than 5, it is discarded and the figure in the third decimal place is kept unchanged;

b) where the figure in the fourth decimal place is 5 and there is a figure other than 0 in the fifth decimal place, or when the figure in the fourth decimal place is greater than 5, the figure in the third decimal place is increased by one;

c) where the figure in the fourth decimal place is 5 and the figure 0 is in the fifth decimal place, the 5 is discarded and the figure in the third decimal place is kept unchanged if it is 0, 2, 4, 6 or 8 and is increased by one if it is 1, 3, 5, 7 or 9.

8.3 Oxide factor

The oxide factor, expressed as a percent, is given by the following equation:

$$w_{\mathrm{V_2O_5}} = 1{,}785\ 2\,w_{\mathrm{V}} \qquad (14)$$

9 Test report

The test report shall include the following information:

a) name and address of the testing laboratory;

b) date of issue of the test report;

c) a reference to this part of ISO 9683;

d) details necessary for the identification of the sample;

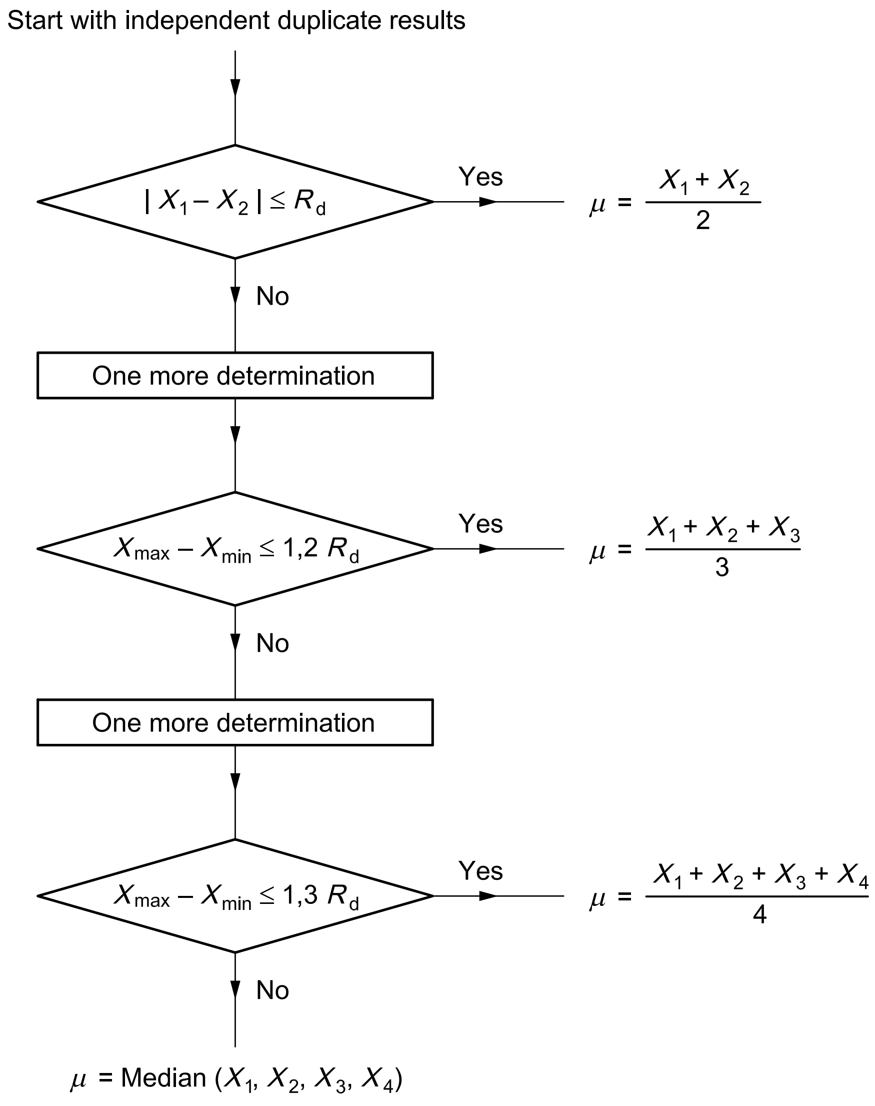
e) result of the analysis;

f) reference number of the result;

g) any characteristics noticed during the determination, and any operations not specified in this part of ISO 9683 which may have had an influence on the result, either for the test sample or for the certified reference material(s).

Annex A
(normative)

Flowsheet of the procedure for the acceptance of analytical values for test samples



R_d: as defined in 8.2.1

Annex B
(informative)

Derivation of repeatability and permissible tolerance equations

The regression equations in 8.2.1 were obtained by statistical evaluation of the results of international analytical trials carried out in 1985/86 on seven iron ore samples, involving 14 laboratories in six countries.

The graphical treatment of the precision data is given in Annex C.

The test samples used are listed in Table B.1.

Table B.1 — Mass fraction of vanadium in test samples

Sample	Mass fraction of vanadium %	Method
Whyalla pellets	0,011	1
Tuollavaara fines	0,061; 0,062	1; 2
Malmberget MAC 10	0,106	2
Philippine iron sand	0,311	2
Savage River pellets	0,527	2
Schefferville	0,004	1
IRSID MO2-1	0,035	1

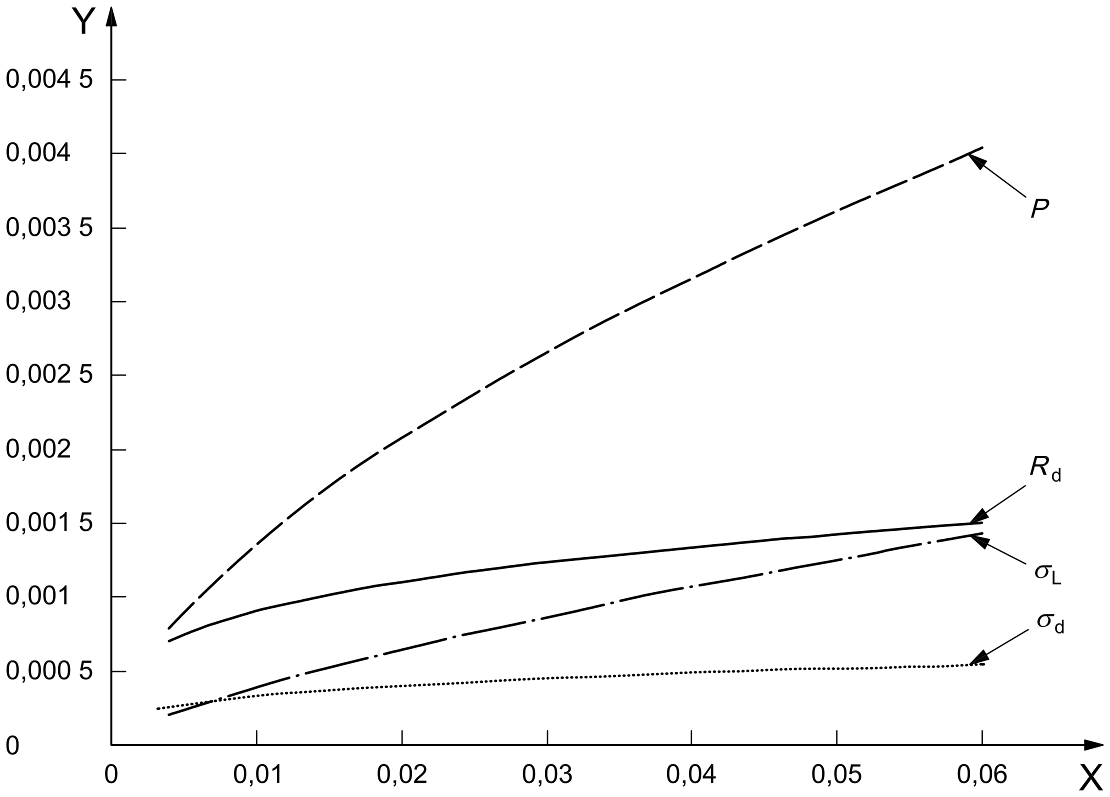
NOTE 1 A report of the international trials (Document ISO/TC 102/SC 2 N825 E, October 1986) and a statistical analysis of the results (Document ISO/TC 102 TCR 13, March 2008) are available from the Secretariat of ISO/TC 102/SC 2.

NOTE 2 The statistical analysis was performed in accordance with the principles embodied in ISO 5725 (all parts), *Accuracy (trueness and precision) of measurement methods and results*.

Annex C
(informative)

Precision data obtained by international analytical trials

NOTE Figures C.1 and C.2 are graphical representations of the equations in 8.2.1.

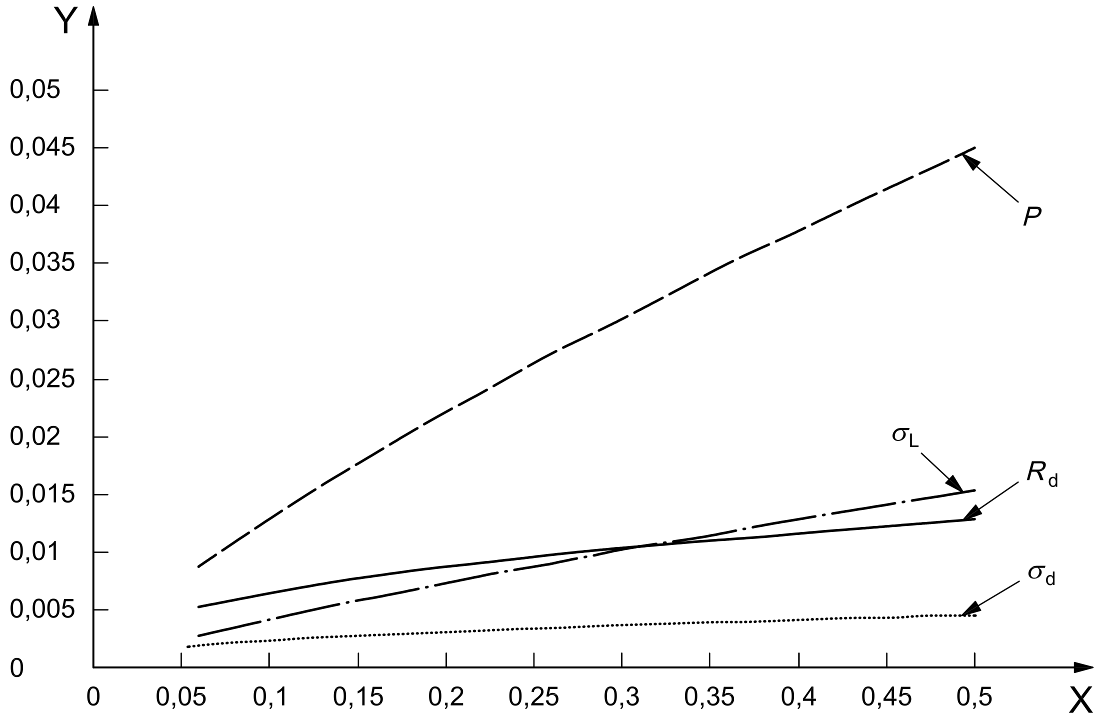


Key

X mass fraction of vanadium, %

Y precision, %

Figure C.1 — Least-squares fit of precision against X for vanadium (Method 1)



Key

X mass fraction of vanadium, %

Y precision, %

Figure C.2 — Least-squares fit of precision against *X* for vanadium (Method 2)

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