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Iron ores — Determination of nickel and/or chromium contents — Flame atomic absorption spectrometric method

Minerais de fer — Dosage du nickel et/ou du chrome — Méthode par spectrométrie d'absorption atomique dans la flamme

Reference number
ISO 9685:1991(E)

Foreword

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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 9685 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Sub-Committee SC 2, *Chemical analysis*.

Annex A forms an integral part of this International Standard. Annexes B and C are for information only.

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Iron ores — Determination of nickel and/or chromium contents — Flame atomic absorption spectrometric method

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of nickel and/or chromium contents of iron ores.

This method is applicable to nickel and/or chromium contents between 0,003 % (*m*/*m*) and 0,1 % (*m*/*m*)[1] in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard 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 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3081:1986, *Iron ores — Increment sampling — Manual method*.

ISO 3082:1987, *Iron ores — Increment sampling and sample preparation — Mechanical method*.

ISO 3083:1986, *Iron ores — Preparation of samples — Manual method*.

ISO 7764:1985, *Iron ores — Preparation of predried test samples for chemical analysis*.

3 Principle

Decomposition of the test portion by treatment with hydrochloric and nitric acids.

Removal of the major portion of iron in the filtrate by extraction with 4-methylpentan-2-one.

Ignition of the insoluble residue, removal of silicon dioxide by evaporation with hydrofluoric and sulfuric acids. Fusion of the residue with a mixture of sodium carbonate and sodium tetraborate, followed by dissolution with hydrochloric acid. Combination with the main solution.

Aspiration into the flame of an atomic absorption spectrometer using an air-acetylene burner for nickel and a dinitrogen oxide-acetylene burner for chromium.

Comparison of the absorbance values obtained with those obtained from the calibration solutions.

4 Reagents

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

4.1 Sodium carbonate (Na_2CO_3), anhydrous powder.

4.2 Sodium tetraborate ($Na_2B_4O_7$), anhydrous powder.

4.3 Lithium tetraborate ($Li_2B_4O_7$), anhydrous powder.

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

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[1] This method has not been tested on ores containing more than 0,056 % (*m*/*m*) of chromium. (See annex B.)

4.5 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 2 + 1.

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

4.7 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 2 + 100.

4.8 Nitric acid, ρ 1,4 g/ml.

4.9 Nitric acid, ρ 1,4 g/ml, diluted 1 + 1.

4.10 Hydrofluoric acid, ρ 1,13 g/ml, 40 % (m/m), or ρ 1,19 g/ml, 48 % (m/m).

4.11 Sulfuric acid, ρ 1,84 g/ml.

4.12 Sulfuric acid, ρ 1,84 g/ml, diluted 1 + 1.

4.13 4-Methylpentan-2-one, (methyl isobutyl ketone, MIBK).

4.14 Nickel standard solution A, 100 µg Ni/ml.

Dissolve 0,1 000 g of nickel metal [purity > 99,9 % (m/m)] (see note 1, 4.18) in 30 ml of nitric acid (4.9). After cooling, transfer to a 1 000 ml one-mark volumetric flask quantitatively, dilute to volume with water and mix.

4.15 Nickel standard solution B, 10 µg Ni/ml.

Transfer 100,0 ml of nickel standard solution A (4.14) to a 1 000 ml one-mark volumetric flask. Dilute to volume with water and mix.

4.16 Chromium standard solution A, 100 µg Cr/ml.

Dissolve 0,1 000 g of chromium metal [purity > 99,9 % (m/m)] (see note 1, 4.18) in 20 ml of hydrochloric acid (4.6). After cooling, transfer to a 1 000 ml one-mark volumetric flask quantitatively, dilute to volume with water and mix.

4.17 Chromium standard solution B, 10 µg Cr/ml.

Transfer 100,0 ml of chromium standard solution A (4.16) to a 1 000 ml one-mark volumetric flask. Dilute to volume with water and mix.

4.18 Nickel and chromium mixture solution.

Introduce into a 200 ml beaker, 30 ml of hydrochloric acid (4.6), 0,2 ml of sulfuric acid (4.12) and a mixture of 0,8 g of sodium carbonate (4.1) and 0,4 g of sodium tetraborate (4.2). Heat to remove carbon dioxide and cool. Using pipettes or burettes, add 10,0 ml of nickel standard solution A (4.14) and 10,0 ml of chromium standard solution A (4.16). Transfer to a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

NOTE 1 The purity of the metals stated on the certificates does not generally take into account the presence of adsorbed gases like oxygen, carbon monoxide, etc.

5 Apparatus

Ordinary laboratory apparatus, including one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 648 and ISO 1042, and

5.1 Platinum crucible, of minimum capacity 25 ml.

5.2 Muffle furnace.

5.3 Atomic absorption spectrometer, equipped with an air-acetylene and a dinitrogen oxide-acetylene burner.

The atomic absorption spectrometer used in this method will be satisfactory if it meets the following criteria:

a) Minimum sensitivity — the absorbance of the most concentrated calibration solution (see 7.5.4) is at least 0,3.

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.

NOTES

2 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.

3 Instrument parameters will vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines. Solutions were aspirated into an air-acetylene or a dinitrogen oxide-acetylene flame of a premix burner:

	Ni	Cr
Hollow-cathode lamp, mA	10	10
Wavelength, nm	232,0	357,9
Air flow rate, l/min	10	—
Acetylene flow rate, l/min	2,5	5
Dinitrogen oxide flow rate, l/min	—	≈ 10

In systems where the values shown above for gas flow rates do not apply, the ratio of the gas flow rates may still be a useful guideline.

6 Sampling and samples

6.1 Laboratory sample

For analysis, use a laboratory sample of minus 100 µm particle size which has been taken in accordance with ISO 3081 or ISO 3082 and prepared in accordance with ISO 3082 or ISO 3083. In the case of ores with significant contents of combined water or oxidizable compounds, use a particle size of minus 160 µm.

NOTES

4 A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

5 Ensure that the sample has not been pulverized in a nickel/chromium pot.

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 6 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 shall be 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 air-acetylene flame or 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 2 g, approximately 1 g of the predried test sample obtained in accordance with 6.2.

NOTE 7 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.

NOTE 8 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 used 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

7.5.1 Decomposition of the test portion

Transfer the test portion (7.3) to a 250 ml tall-form beaker. Moisten with a few millilitres of water. Add 25 ml of hydrochloric acid (4.4), cover with a watch-glass, and heat for about 1 h on a hotplate adjusted to give a temperature of 100 °C in a test beaker containing a similar volume and depth of sulfuric acid (4.11).

NOTES

9 When the amount of insoluble residue is high, continue heating on a higher temperature zone of the hotplate without boiling the solution.

10 After heating, the volume of the solution should have decreased to about 10 ml.

Add 5 ml of nitric acid (4.8) and 0,2 ml of sulfuric acid (4.12), digest for about 15 min and evaporate the solution almost to dryness.

NOTES

11 When the test sample contains a significant content of barium, the addition of sulfuric acid (4.12) should be omitted.

12 Be sure to cover with a watch-glass, in order to prevent sublimation of chlorides. During evaporation, shift the glass slightly.

Add 20 ml of hydrochloric acid (4.6), and heat to dissolve the salts. Cool, wash the watch-glass and the walls of the beaker, and filter the solution through a close-texture filter paper containing filter pulp (0,3 g to 0,4 g dry mass) into a 200 ml beaker. Carefully remove all adhering particles with a rubber-tipped glass rod or piece of moistened filter paper, and transfer to the filter, washing the paper with warm hydrochloric acid (4.7) until it is visibly free from iron, and again wash the paper with three or four portions of warm water. Reserve the filtrate and washings in the 200 ml beaker as the main solution. Transfer the filter paper and residue to a platinum crucible (5.1).

7.5.2 Removal of iron

Heat the main solution and evaporate almost to dryness. Dissolve the salts in 15 ml of hydrochloric acid (4.5) and transfer to a 200 ml separating funnel. Rinse the beaker with 20 ml of hydrochloric acid (4.5) and transfer the washings to the funnel.

Add 50 ml of 4-methylpentan-2-one (4.13) to the funnel and shake it for 1 min. Allow the two layers to separate and drain the lower aqueous layer into the original 200 ml beaker. Wash the organic layer by adding 10 ml of hydrochloric acid (4.5) to the funnel and shaking it for 30 s. Allow the two layers to separate and drain the lower aqueous layer into the 200 ml beaker to combine with the first aqueous layer. Discard the organic layer.

Heat the solution gently and expel almost all of the 4-methylpentan-2-one in the solution. Then add 5 ml of nitric acid (4.8) and evaporate to dryness. Dissolve the salts in 20 ml of hydrochloric acid (4.6).

7.5.3 Treatment of the residue

Dry and burn off the filter paper in the platinum crucible (5.1) at a low temperature (500 °C to 800 °C), and ignite the residue. Cool, moisten with 1 or 2 drops of water, and add 3 drops of sulfuric acid (4.11) and 5 ml of hydrofluoric acid (4.10). Evaporate slowly to expel silicon dioxide and continue heating to remove the sulfuric acid. Ignite at 800 °C for several minutes and then cool. Add 1,2 g of a mixture of 0,8 g of sodium carbonate (4.1) and 0,4 g of sodium tetraborate (4.2) to the residue and mix. Heat gently for several minutes, then heat at 1 000 °C for 15 min in a muffle furnace (5.2) or over a pressurized air burner for a time sufficient to produce a clear melt.

NOTE 13 Lithium tetraborate (4.3) may be used instead of sodium tetraborate (4.2). In this case, sodium tetraborate should be replaced with lithium tetraborate in the calibration solutions (7.5.4) and the flame setting solutions (7.5.5).

Allow the crucible to cool, add 10 ml of hydrochloric acid (4.6) and heat gently to dissolve the melt. Combine this solution with the main solution from 7.5.2, heat to remove carbon dioxide and cool. Transfer to a 100 ml one-mark volumetric flask quantitatively, dilute to volume with water and mix. (This is the test solution.)

7.5.4 Preparation of the calibration solutions

Introduce, into a series of six 200 ml beakers, 30 ml of hydrochloric acid (4.6), 0,2 ml of sulfuric acid (4.12) and a mixture of 0,8 g of sodium carbonate (4.1) and 0,4 g of sodium tetraborate (4.2). Heat to remove carbon dioxide and cool.

Using pipettes or burettes, add 0 ml, 1,0 ml, 3,0 ml, 5,0 ml, 7,0 ml and 10,0 ml of standard solution A (4.14 or 4.16) or standard solution B (4.15 or 4.17).

NOTE 14 The nickel and chromium calibration solutions should be made up separately.

Transfer to six 100 ml one-mark volumetric flasks respectively, dilute to volume with water and mix. These calibration solutions cover the concentration ranges 0 µg/ml to 10 µg/ml in the case of standard solution A, or 0 µg/ml to 1 µg/ml in the case of standard solution B.

7.5.5 Preparation of the flame setting solutions

7.5.5.1 Nickel solution

Use the most concentrated nickel calibration solution which was prepared in 7.5.4 using standard solution A (4.14).

7.5.5.2 Nickel and chromium mixture solution

Use the nickel and chromium mixture solution (4.18) to set the flame and to eliminate chromium interference.

7.5.6 Adjustment of the atomic absorption spectrometer

Optimize the response of the instrument as specified in 5.3. Set the wavelength of nickel (232,0 nm) and/or chromium (357,9 nm) to obtain minimum absorbance and adjust the readout to zero absorbance. Light the air-acetylene or dinitrogen oxide-acetylene flame. After 10 min of preheating the burner, aspirate water and, if necessary, readjust the readout to zero absorbance. Aspirate the calibration solution of highest nickel or chromium content (7.5.4) and adjust the fuel flow and burner position to obtain maximum absorbance.

In the case of nickel, adjust the acetylene flow to provide a fuel lean flame. Alternately aspirate the flame setting solutions (7.5.5), adjust the burner height and acetylene flow until the absorbance values differ by less than 0,004 at the maximum absorbance value.

NOTE 15 At the optimum conditions for elimination of chromium interference, a loss of sensitivity of about 10 % to 20 % is expected.

Check that the conditions for zero absorbance have been maintained and evaluate the criteria in 5.3.

Repeat the aspiration of water and the calibration solution of highest nickel or chromium content to establish that the absorbance reading is not drifting. Set the reading for water to zero absorbance.

7.5.7 Atomic absorption measurements

Aspirate the calibration solutions (7.5.4) and the test solution (7.5.3) in order of increasing absorption, starting with the zero calibration solution (7.5.4) and the blank test solution. When a stable response has been obtained for each solution, record the readings. Aspirate water between each calibration and test solution.

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 nickel or chromium 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 nickel or chromium per millilitre.

NOTE 16 If any curvature obtained approaches the limit specified in 5.3 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 concentrations of nickel or chromium, in micrograms per millilitre, in the blank test and test solutions respectively, and correct the concentration of the test solution with the concentration of the blank test.

8 Expression of results

8.1 Calculation of nickel or chromium content

The nickel or chromium content, $w_{\text{Ni or Cr}}$, is calculated as a percentage by mass, to five decimal places, from the equation:

$$w_{\text{Ni or Cr}} = \frac{\rho_{\text{Ni or Cr}} \times 100}{m \times 10^6} \times 100$$

$$= \frac{\rho_{\text{Ni or Cr}} \times 0{,}01}{m} \qquad \ldots (1)$$

where

$\rho_{\text{Ni or Cr}}$ is the concentration, in micrograms per millilitre, of nickel or chromium in the final test solution;

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

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[2]:

Nickel

$$r = 0{,}029\,1\;X + 0{,}000\,7 \qquad \ldots (2)$$

$$P = 0{,}089\,5\;X + 0{,}001\,1 \qquad \ldots (3)$$

$$\sigma_r = 0{,}010\,3\;X + 0{,}000\,2 \qquad \ldots (4)$$

$$\sigma_L = 0{,}030\,7\;X + 0{,}000\,3 \qquad \ldots (5)$$

Chromium

$$r = 0{,}049\,3\;X + 0{,}000\,5 \qquad \ldots (6)$$

$$P = 0{,}102\,2\;X + 0{,}000\,2 \qquad \ldots (7)$$

$$\sigma_r = 0{,}017\,3\;X + 0{,}000\,2 \qquad \ldots (8)$$

$$\sigma_L = 0{,}034\,4\;X \qquad \ldots (9)$$

where

X is the nickel or chromium content, expressed as a percentage by mass, of the predried test sample, calculated as follows:

- within-laboratory equation (2), (4), (6) and (8): the arithmetic mean of the duplicate values,
- between-laboratories equations (3), (5), (7) and (9): the arithmetic mean of the final results (8.2.3) of the two laboratories;

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[2] Additional information is given in annexes B and C.

r is the permissible tolerance within a laboratory (repeatability);

P is the permissible tolerance between laboratories;

σ_r is the within-laboratory standard deviation;

σ_L is the between-laboratories standard deviation.

8.2.2 Acceptance of analytical values

The result obtained for the certified reference material shall be such that the difference between this result and the certified value of the reference material is statistically insignificant. For a reference material that has been analysed by at least 10 laboratories using method(s) that are comparable in both accuracy and precision with this method, the following condition may be used to test the significance of the difference:

$$|A_c - A| \leqslant 2\sqrt{\frac{s_{\text{Lc}}^2 + \dfrac{s_{\text{Wc}}^2}{n_{\text{Wc}}}}{N_{\text{c}}} + \sigma_{\text{L}}^2 + \frac{\sigma_r^2}{n}} \qquad \ldots (10)$$

where

A_{c} is the certified value;

A is the result or the mean of results obtained for the certified reference material;

s_{Lc} is the between-laboratories standard deviation of the certifying laboratories;

s_{Wc} is the within-laboratory standard deviation of the certifying laboratories;

n_{Wc} is the average number of replicate determinations in the certifying laboratories;

N_{c} is the number of certifying laboratories;

n is the number of replicate determinations on the certified reference material (in most cases $n = 1$);

σ_{L} and σ_r are as defined in 8.2.1.

If condition (10) is satisfied, i.e. if the left-hand side is less than or equal to the right-hand side, then the difference $|A_{\text{c}} - A|$ is statistically insignificant; otherwise, it is statistically significant.

When the difference is significant, the analysis shall be repeated simultaneously with an analysis of the test sample. If the difference is again significant, the procedure shall be repeated using a different certified reference material of the same type of ore.

When the range of the two values for the test sample is outside the limit for r calculated according to equation (2) or (6), one or more additional tests shall be carried out in accordance with the flowsheet presented in annex A, simultaneously with an analysis of a certified reference material of the same type of ore.

Acceptability of the results for the test sample shall in each case be subject to the acceptability of the results for the certified reference material.

NOTE 17 The following procedure should be used when the information on the reference material certificate is incomplete:

a) if there are sufficient data to enable the between-laboratories standard deviation to be estimated, delete the expression $s_{\text{Wc}}^2/n_{\text{Wc}}$ and regard s_{Lc} as the standard deviation of the laboratory means;

b) if the certification has been made by only one laboratory or if the interlaboratory results are missing, it is advisable that this material not be used in the application of the standard. In case its use is unavoidable, use the equation

$$|A_{\text{c}} - A| \leqslant 2\sqrt{2\sigma_{\text{L}}^2 + \frac{\sigma_r^2}{n}} \qquad \ldots (11)$$

8.2.3 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) when 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) when 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) when 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 factors

$$w_{\text{NiO}}\ (\%) = 1{,}272\ 5\ w_{\text{Ni}}\ (\%)$$

$$w_{\text{Cr}_2\text{O}_3}\ (\%) = 1{,}461\ 5\ w_{\text{Cr}}\ (\%)$$

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) reference to this International Standard;

d) details necessary for the identification of the sample;

e) result of the analysis;

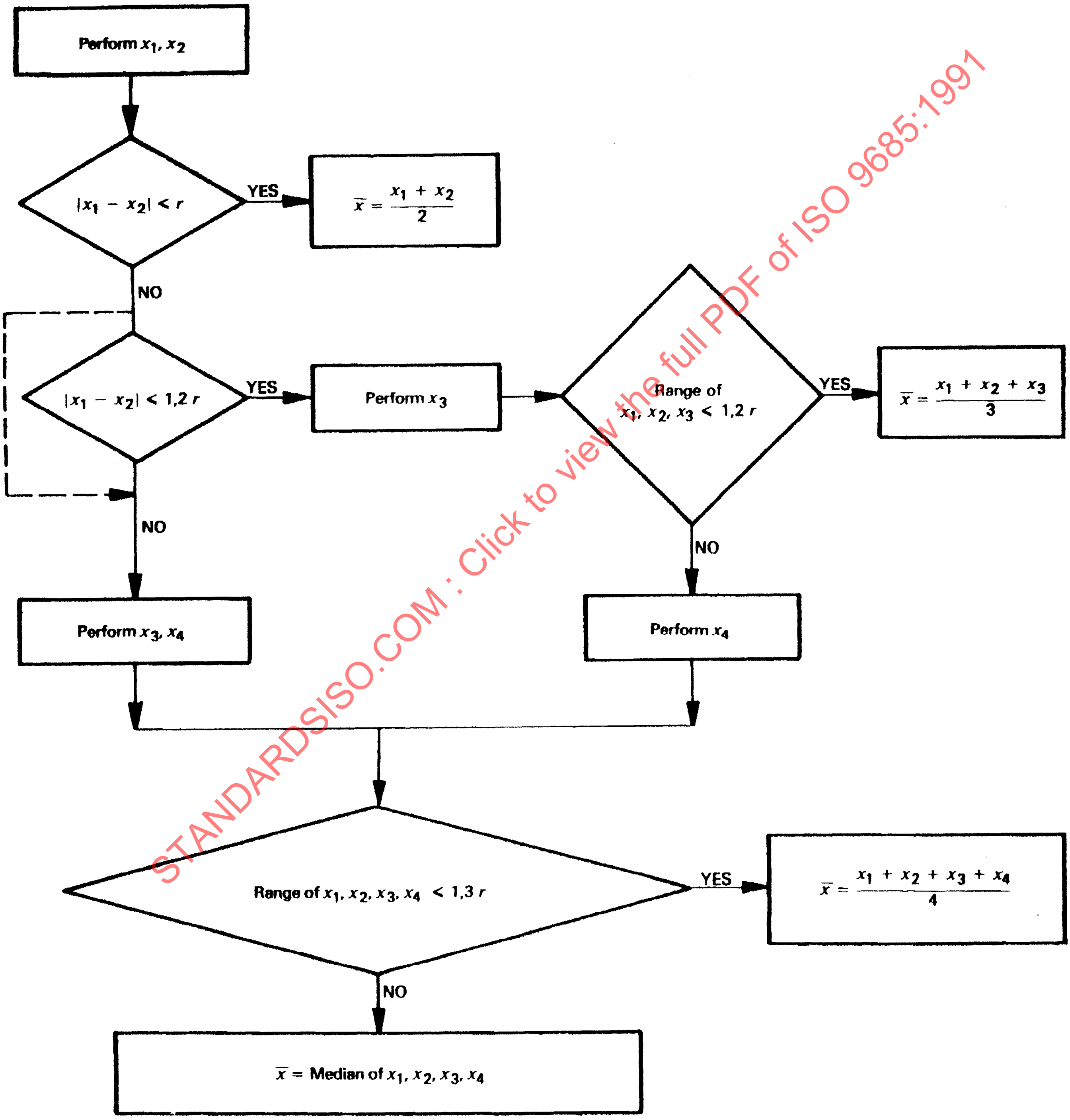
f) reference number of the result;

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

Annex A

(normative)

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



r : 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 1986 on five iron ore samples, involving 15 laboratories in six countries.

Graphical treatment of the precision data is given in annex C .

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

NOTES

18 A report of the international trials and a statistical analysis of the results (Document ISO/TC 102/SC 2 N 842 E, November 1986) is available either from the Secretariat of ISO/TC 102/SC 2 or the Secretariat of ISO/TC 102.

19 The statistical analysis was performed in accordance with the principles embodied in ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

Table B.1 — Nickel and chromium contents of test samples

Sample	Nickel content % (m/m)	Chromium content % (m/m)
Hamersley hematite	0,002	0,004
Savage River pellets	0,049	0,012
Swaziland	0,017	0,056
Sintered ore	0,011	0,016
Inco-based ore	0,084	0,019

Annex C
(informative)

Precision data obtained by international analytical trials

NOTE 20 These figures are graphical representations of the equations in 8.2.1.

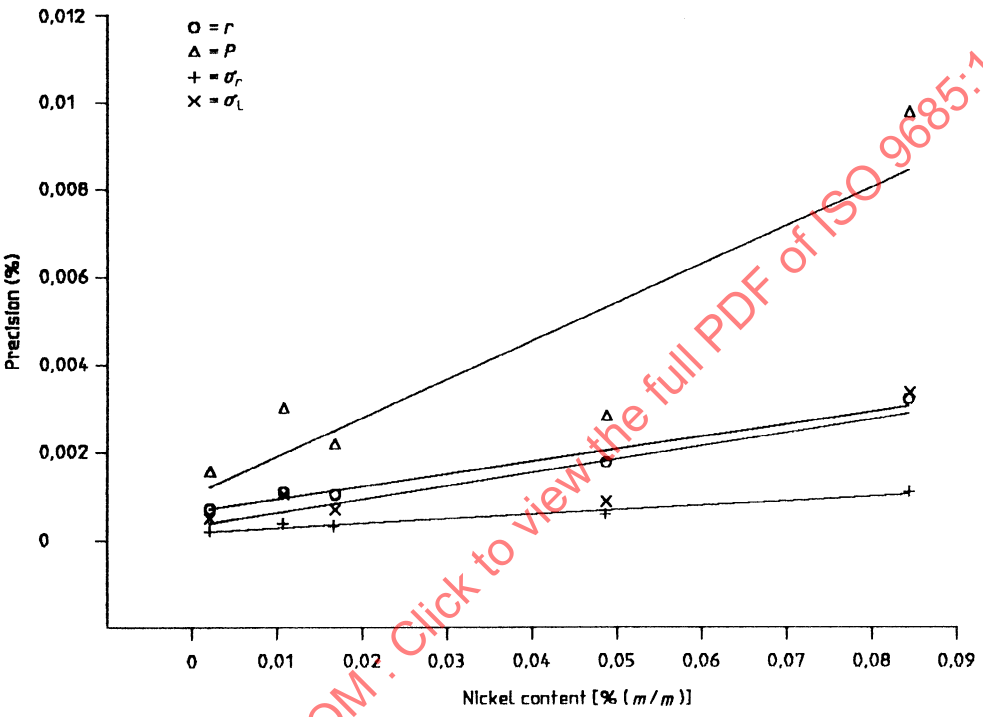


Figure C.1 — Least-squares fit of precision against X for nickel