

**Direct reduced iron —
Determination of
carbon and/or sulfur —
High-frequency
combustion method
with infrared
measurement**

ICS 73.060.10

National foreword

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The UK participation in its preparation was entrusted to Technical Committee ISE/58, Iron ores, which has the responsibility to:

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**Direct reduced iron — Determination of
carbon and/or sulfur — High-frequency
combustion method with infrared
measurement**

*Minerais de fer prééduits — Dosage du carbone et/ou du soufre —
Méthode par combustion haute fréquence et mesurage par infrarouge*



Reference number
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Foreword

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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 9686 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This second edition cancels and replaces the first edition (ISO 9686:1992), which has been technically revised. It has been updated to alter the manner in which precision data are presented.

Direct reduced iron — Determination of carbon and/or sulfur — High-frequency combustion method with infrared measurement

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

1 Scope

This International Standard specifies a method for the determination of the mass fraction of carbon and/or sulfur in direct reduced iron by infrared measurement after high-frequency combustion.

This method is applicable to mass fractions of carbon between 0,05 % and 2,5 %, and/or mass fractions of sulfur between 0,001 % and 0,05 % in direct reduced iron.

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

ISO 7550, *Laboratory glassware — Disposable micropipettes*

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

ISO 10835, *Direct reduced iron — Sampling and sample preparation — Manual methods for reduced pellets and lump ores*

3 Principle

The test portion is combusted in a refractory crucible in a flow of oxygen in the presence of an accelerator, the crucible being inserted in the combustion tube of a high-frequency (HF) furnace.

The carbon present is converted into carbon dioxide and the sulfur into sulfur dioxide. Each gas is measured by infrared absorption, with calibration using barium carbonate and potassium sulfate.

4 Reagents

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

4.1 Oxygen, minimum purity 99,5 % (m/m).

The pressure in the furnace should be controlled by a pressure regulator designed specially for the purpose and complying with the manufacturer's specification.

4.2 Magnesium perchlorate, grain size 0,7 mm to 1,2 mm.

4.3 Accelerator, tungsten (granular form) with known low mass fractions of carbon [$< 0,002\%$] and sulfur [$< 0,0005\%$].

4.4 Pure iron, or iron of known low mass fractions of carbon and sulfur, as in 4.3.

4.5 Tin capsules, of capacity 0,3 ml, diameter 5 mm, length 17 mm.

4.6 Barium carbonate (BaCO_3), fine powder.

Dry at $105\text{ }^\circ\text{C}$ for 3 h and cool in a desiccator.

4.7 Sulfur standard solutions.

Dry potassium sulfate (K_2SO_4) at $105\text{ }^\circ\text{C}$ for 1 h and cool in a desiccator.

Weigh, to the nearest 0,0002 g, the amounts of potassium sulfate specified in Table 1.

Table 1 — Sulfur standard solutions

Sulfur standard solution	Mass of K_2SO_4 g	Sulfur concentration mg/ml
SS 1	0,217 4	0,4
SS 2	0,434 8	0,8
SS 3	0,652 2	1,2
SS 4	0,869 6	1,6
SS 5	2,174 0	4,0

Transfer to five 100 ml one-mark volumetric flasks, dissolve with 50 ml of water, dilute to volume and mix.

4.8 Ascarite, used only for carbon determination.

5 Apparatus

Ordinary laboratory apparatus, including micropipettes and one-mark volumetric flasks complying with the specifications of ISO 7550 and ISO 1042 respectively, and the following.

5.1 Commercial carbon/sulfur apparatus

The apparatus required for HF combustion of the samples and the subsequent infrared absorption measurement of evolved carbon dioxide and/or sulfur dioxide may be obtained commercially from a number of manufacturers. Common features of such systems are discussed in Annex C.

The manufacturer's instruction for the operation of their equipment shall be followed.

5.2 Ceramic combustion crucibles, required for containing the sample and any additions that may be necessary for the subsequent combustion.

The crucibles shall be of precise dimensions for the system, and shall adapt to the supporting pedestal post, so that the sample in the crucible will be positioned at the correct height within the induction coil when it is in the raised position.

These crucibles shall be pre-ignited in an oxygen flow, in a furnace, for not less than 2 h at 1 350 °C (or at 1 100 °C if only sulfur is to be determined), and then stored in a desiccator or closed container before use.

For pre-ignition, a resistance furnace may be used.

5.3 Micropipette, of capacity 50 µl.

6 Sampling and samples

6.1 Laboratory sample

For analysis, use a laboratory sample of minus 160 µm particle size, which has been taken and prepared in accordance with ISO 10835.

6.2 Preparation of predried test samples

Thoroughly mix the laboratory sample using non-magnetic materials. Taking multiple increments with a non-magnetic spatula, extract a test sample in such a manner 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

WARNING — The risks related to combustion analysis are mainly hand burns when pre-igniting the ceramic crucibles, and in the subsequent combustion. Normal precautions for handling oxygen cylinders should be taken. Oxygen from the combustion process should be removed effectively from the apparatus and room, since a too high concentration of oxygen in a confined space may present a fire hazard. HF screening should be effective to avoid radiation hazards.

7.1 General operating instructions

Purify the oxygen supply using tubes packed with ascarite (4.8) and magnesium perchlorate (4.2) and maintain a quiescent flow rate of about 0,5 l/min whilst on standby.

Maintain a glass-wool filter between the furnace chamber and the analyser and change as necessary. The furnace chamber, pedestal post and filter trap should be cleaned frequently to remove oxide build-up.

The oxygen flow rate may vary from one instrument to another, but is usually about 2,0 l/min during the combustion period, according to the nature of the material. The temperature reached during the combustion stage depends on the power of the HF generator, the geometry of the furnace chamber, the induction coil and the type and quantity of sample in the crucible. This temperature may be in the order of 1 700 °C, or more.

When the mains supply is switched on, after being out of action for any length of time, allow the time recommended by the equipment manufacturer for the stabilization of each item of equipment.

After cleaning the furnace chamber, changing filters, or after the equipment has been inoperative for a period, stabilize the apparatus by burning several samples, of similar type to the samples to be analysed, prior to setting up for analysis.

Flush oxygen to the apparatus and adjust the instrument controls to give zero readings.

7.2 Test portion

Weigh 0,400 g of the sample to the nearest 0,000 1 g.

7.3 Blank test

Carry out a blank test using the same procedure and the same quantities of all reagents as for a determination (7.5), i.e.:

- 1,9 g of tungsten (4.3);
- 1,3 g of pure iron (4.4);
- 1 tin capsule (4.5).

To achieve the best accuracy possible, perform at least three blank burns. Use the mean thereof to make a zero adjustment, in accordance with the manufacturer's requirements.

7.4 Calibration

7.4.1 Crucible preparation

7.4.1.1 Analysis for carbon only

Using the outline given in Annex A, weigh, to the nearest 0,000 2 g, the amount of barium carbonate (4.6) specified in Table 2 into six combustion crucibles (5.2).

Table 2 — Carbon calibration tests

Crucible	Carbon standard	Mass of BaCO ₃ (4.6) g	C % (mass fraction)
1	CS 1	0,013 1	0,2
2	CS 2	0,032 9	0,5
3	CS 3	0,065 7	1,0
4	CS 4	0,131 4	2,0
5	CS 5	0,164 3	2,5
6	CS 5	0,164 3	2,5

7.4.1.2 Analysis for sulfur only

Using the micropipette (5.3), introduce 50 µl each of the sulfur standard solutions (4.7) into seven tin capsules in accordance with Table 3.

Table 3 — Sulfur calibration tests

Crucible	Tin capsule	Sulfur standard solutions (4.7)	S % (mass fraction)
1	1	Pure water (50 µl)	0
2	2	SS 1	0,005
3	3	SS 2	0,10
4	4	SS 3	0,015
5	5	SS 4	0,020
6	6	SS 5	0,050
7	7	SS 5	0,050

Dry the capsules and their contents slowly at 80 °C to 90 °C for 2 h, and cool in a desiccator.

Using the outline given in Annex A, prepare seven crucibles containing the tin capsules specified in Table 3.

7.4.1.3 Analysis for carbon and sulfur

Using the outline given in Annex A, prepare seven crucibles containing tin capsules (4.5), and the amounts of barium carbonate (4.6) and sulfur standard solutions (4.7) specified in Table 4.

Table 4 — Carbon and sulfur calibration tests

Crucible	Carbon (7.4.1.1) and sulfur (7.4.1.2)	C + S % (mass fraction)
1	Pure water (50 µl)	0 + 0
2	CS 1 + SS 1	0,2 + 0,005
3	CS 2 + SS 2	0,5 + 0,010
4	CS 3 + SS 3	1,0 + 0,015
5	CS 4 + SS 4	2,0 + 0,020
6	CS 5 + SS 5	2,5 + 0,050
7	CS 5 + SS 5	2,5 + 0,050

7.4.2 Combustion

With the calibration crucibles prepared as in Annex A, firstly burn crucible 7 containing the maximum content of carbon and/or sulfur, then crucible 6 for a check. Adjust the readings to the corresponding value(s).

Burn the other crucibles and note the results to check the linearity.

NOTE For all measurements, the mass compensator is set at 0,400 g.

7.5 Determination

Prepare the test in accordance with Annex A and burn as in the calibration series, in accordance with the manufacturer's instruction.

8 Expression of results

8.1 Calculation of mass fractions of carbon or sulfur

The mass fractions of carbon and sulfur are given directly by the instrument as a function of the calibration (7.4), taking into account the value of the blank test.

The total blank from all sources (oxygen, iron, tin capsules, tungsten) should not exceed 0,01 % (mass fraction) of carbon and 0,001 % (mass fraction) of sulfur.

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 equations¹⁾

Carbon

$$R_d = 0,020\ 3\ X + 0,012\ 7 \quad (1)$$

$$P = 0,020\ 8\ X + 0,024\ 4 \quad (2)$$

$$\sigma_d = 0,007\ 2\ X + 0,004\ 5 \quad (3)$$

$$\sigma_L = 0,005\ 7\ X + 0,007\ 9 \quad (4)$$

Sulfur

$$R_d = 0,047\ 9\ X + 0,001\ 0 \quad (5)$$

$$P = 0,124\ 1\ X + 0,001\ 4 \quad (6)$$

$$\sigma_d = 0,016\ 9\ X + 0,000\ 4 \quad (7)$$

$$\sigma_L = 0,044\ 3\ X + 0,000\ 4 \quad (8)$$

where

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

- within-laboratory Equations (1), (3), (5) and (7): the arithmetic means of the duplicates values,
- between-laboratories Equations (2), (4), (6) and (8): 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

1) Additional information is given in Annexes D and E.

8.2.2 Determination of analytical result

Having computed the independent duplicate results according to Equation (1), compare them with the independent duplicate limit (R_d), using the procedure given in Annex A, and obtain the final laboratory result μ (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 same procedure as described in 8.2.2.

Compute the following quantity:

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \quad (6)$$

where

μ_1 is the final result reported by laboratory 1;

μ_2 is the final result reported by laboratory 2;

μ_{12} is the mean of final results.

Substitute μ_{12} for X in Equation (3) and calculate P .

If $|\mu_1 - \mu_2| \leq P$, 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). Calculate the analytical result (μ_c) for the RM/CRM using the procedures in 8.1 and 8.2, and compare it with the reference or certified value A_c . There are two possibilities:

- $|\mu_c - A_c| \leq C$ in which case the difference between the reported result and the reference/certified value is statistically insignificant.
- $|\mu_c - A_c| > C$ 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 certified reference material;

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 certified by an interlaboratory test programme

$$C=2 \left[\sigma_L^2 + \frac{\sigma_d^2}{n} + V(A_c) \right]^{1/2}$$

where

$V(A_c)$ is the variance of the certified value A_c (= 0 for a CRM certified by only one laboratory);

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

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 of the test sample, or as otherwise determined by the operations specified in Annex B, calculated to five decimal places and rounded off to the third decimal place as follows.

- a) Where the final 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.

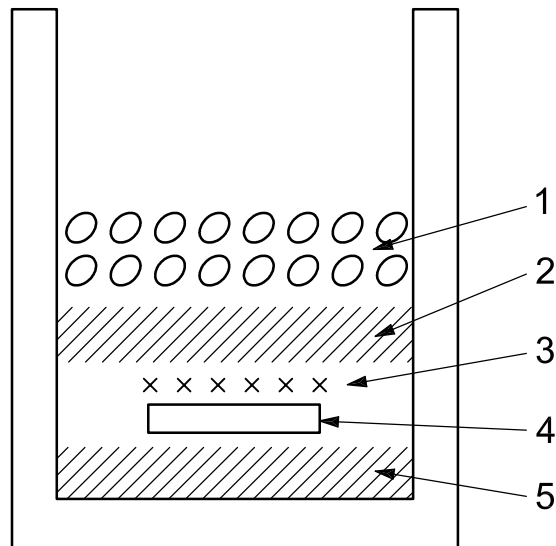
9 Test report

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 International Standard;
- d) details necessary for the identification of the sample;
- e) reference number of the result;
- f) 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 for the certified reference materials(s).

Annex A (normative)

Crucible loading sequence



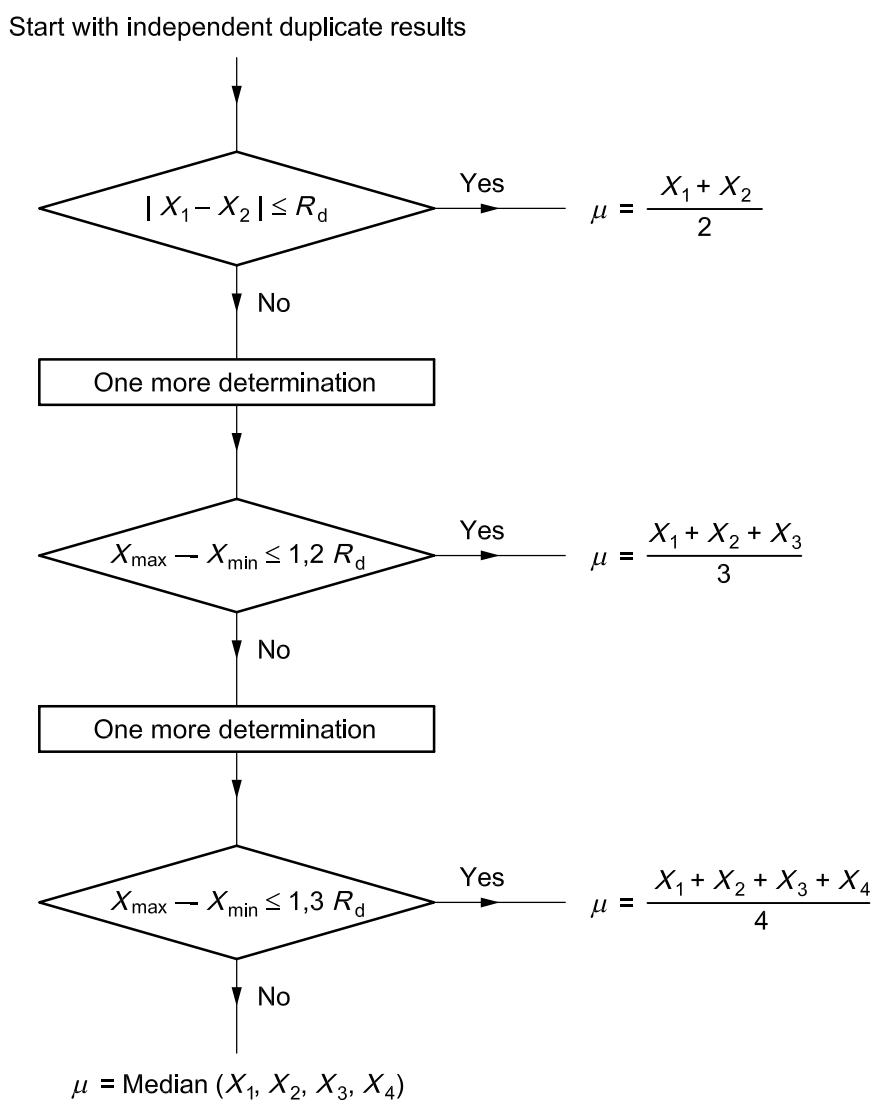
Key

- 1 tungsten (4.3): 1,9 g
- 2 pure iron (4.4): 0,4 g
- 3 test portion (7.2) or barium carbonate (4.6): 0,4 g
- 4 tin capsule (4.5)
- 5 pure iron (4.4): 0,9 g

Figure A.1

Annex B (normative)

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



Annex C (informative)

Features of commercial HF combustion/infrared sulfur analysers

C.1 Combustion

The combustion furnace has an induction coil and HF generator. The furnace chamber consists of a silica tube which fits inside the induction coil. This tube has a metal plate at the top and bottom and is sealed by O-rings. Gas inlet and outlet points are made via the metal plates, with a filter on the outlet to prevent dust particles from reaching the detection system.

The generator is usually a 1,2 kVA to 2,5 kVA unit, but the frequency used by various manufacturers may be different. Values of 2 MHz, 6 MHz, 15 MHz and 20,0 MHz have been used. The power from the generator is fed to the induction coil, which surrounds the silica furnace tube and is usually air-cooled. The crucible containing the sample and accelerator is supported on a pedestal post, which is precisely positioned when raised, so that the sample in the crucible is correctly placed within the induction coil for effective coupling when the power is applied.

Typical dimensions of combustion crucibles are as follows:

- height of crucibles: 25 mm;
- external diameter: 25 mm;
- internal diameter: 20 mm;
- wall thickness: 2,5 mm;
- thickness of base: 8 mm.

The induction-coil diameter, the number of turns and the furnace geometry determine the degree of HF coupling that can occur, and these factors are determined by the instrument manufacturer. The temperature generated depends in part on these factors, but also on the characteristics of the sample in the crucible, the sample form and the mass of materials. These factors may be varied to some extent by the operator in the light of experience.

It is essential that the oxides formed during combustion be removed by glass-wool filtration, and the filter trap should be cleaned frequently to remove oxide build-up.

C.2 Infrared gas analyser

A given volume of the gaseous products of combustion may be collected at a controlled pressure, and the mixture analysed for CO₂ and/or SO₂; or the CO₂ and/or SO₂ content in a continuous oxygen flow may be integrated during the evolution of gases from the combustion process.

The oxygen carrier-gas containing CO₂ and/or SO₂ is passed to the analyser system, consisting of an infrared cell, usually of the Luft type or equivalent (solid state), where the specific absorption of the infrared radiation is measured.

Electronic amplification of the absorption measurement is usually converted to a digital display of percentage concentration. Means are usually provided for off-setting the zero (blank compensation), adjusting the calibration and linearizing the readout.

Instruments may also be equipped with an integral automatic balance, and a system for applying sample mass correction. In modern instruments, a microprocessor is generally included.

Annex D (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 to 1988 on four direct reduced iron (DRI) samples, involving 13 laboratories in seven countries.

Graphical treatment of the precision data is given in Annex E.

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

NOTE 1 A report of the international trials and a statistical analysis of the results (Document ISO/TC 102/SC 2 929E, November 1988) is available from the Secretariat of ISO/TC 102/SC 2.

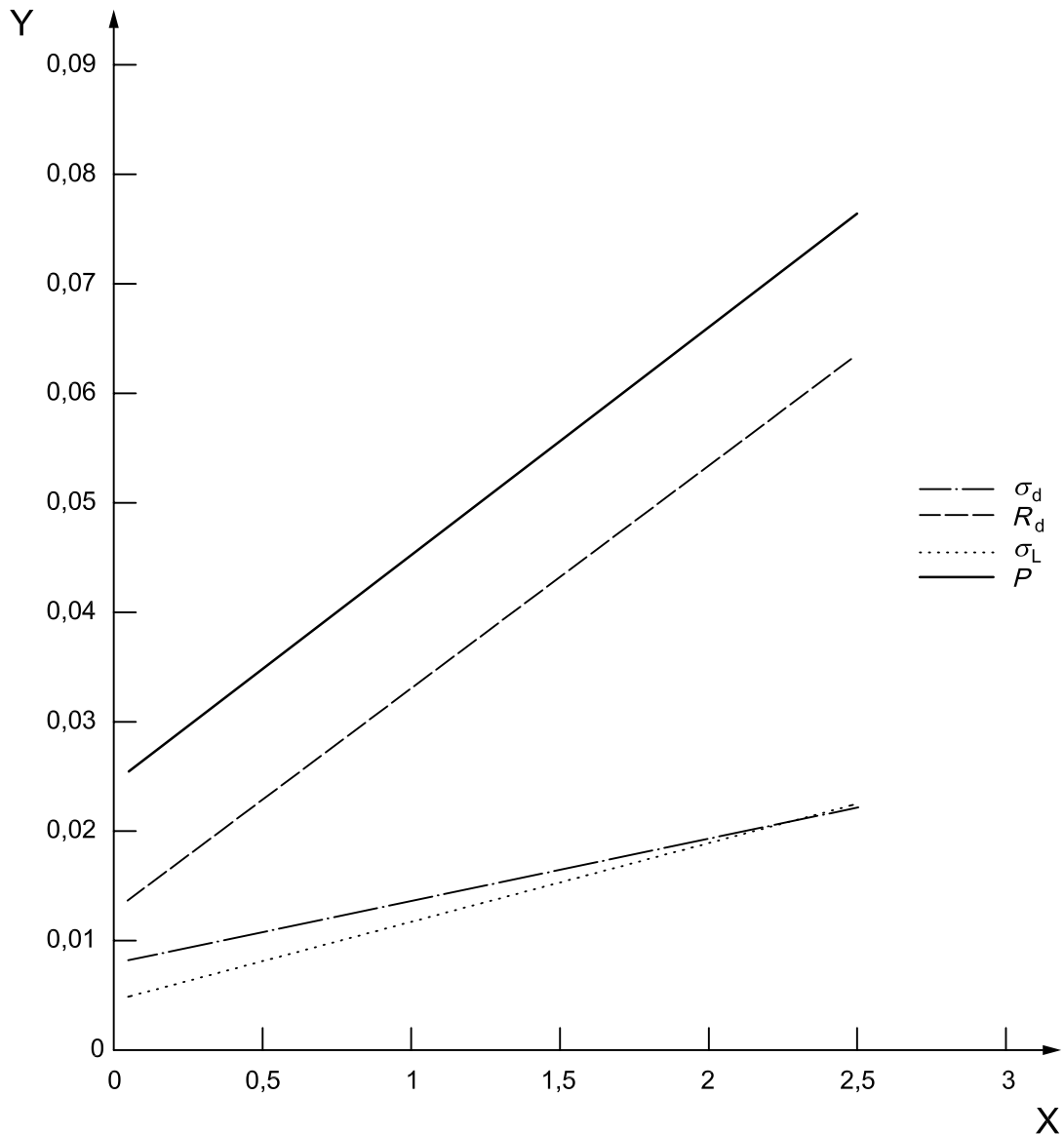
NOTE 2 The statistical analysis was performed in accordance with the principles embodied in ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*.

Table D.1 — Mass fractions of carbon and sulfur in test samples

Sample	Carbon content % (mass fraction)	Sulfur content % (mass fraction)
DRI—Gas reduced 16/85	1,838 6	0,001 8
DRI—Lab. produced 17/85	0,365 6	0,001 2
DRI—Lab. produced 18/85	0,096 9	0,001 3
DRI—Coal reduced 19/85	0,096 1	0,039 3

Annex E (informative)

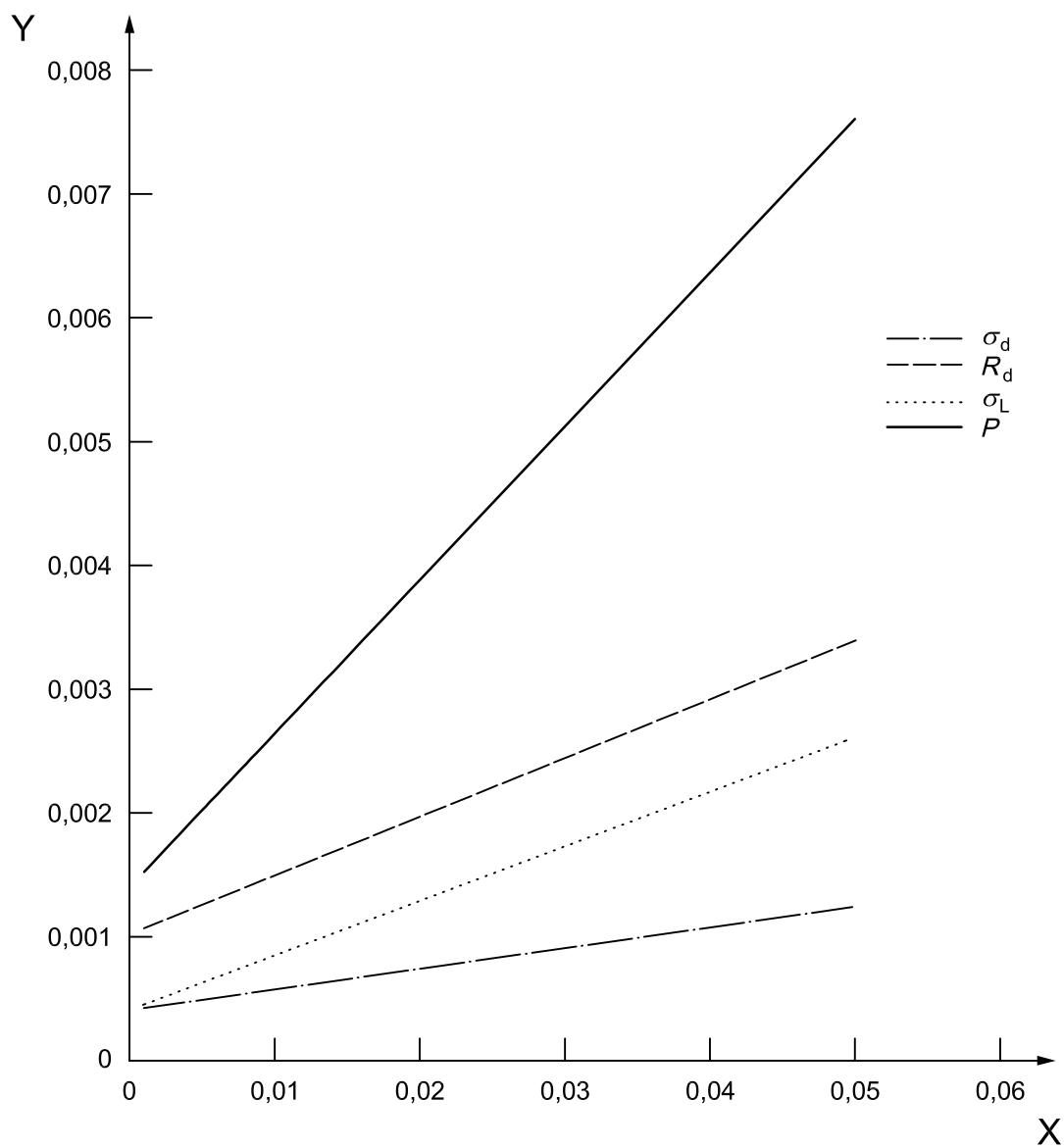
Precision data obtained by international analytical trials



Key

- Y Precision, %
X Mass fraction of carbon, %

Figure E.1 — Least-squares fit of precision against X for carbon

**Key**

Y Precision, %

X Mass fraction of sulfur, %

Figure E.2 — Least-squares fit of precision against X for sulfur

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