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Solid mineral fuels — Major and minor elements in coal ash and coke ash — Wavelength dispersive x-ray fluorescence spectrometric method

Combustibles minéraux solides — Éléments en minorité et en majorité dans les cendres de houille et de coke — Méthode spectrométrique par fluorescence aux rayons X à une longueur d'onde dispersive

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Contents

Page

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This first edition of ISO 13605 cancels and replaces ISO/TS 13605:2012, which has been technically revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Solid mineral fuels — Major and minor elements in coal ash and coke ash — Wavelength dispersive x-ray fluorescence spectrometric method

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1 Scope

This document sets out a wavelength dispersive X-ray fluorescence (XRF) procedure for the determination of silicon, aluminium, iron, calcium, magnesium, sodium, potassium, titanium, manganese, phosphorus and sulfur.

The method is applicable to coal ashes, coke ashes and boiler ashes having components within the concentration ranges specified in Table 1.

Table 1 — Ranges of application of the method

Oxide	Concentration range %
SiO_2	5 to 100
Al_2O_3	5 to 80
Fe_2O_3	0,1 to 25
CaO	0,05 to 25
MgO	0,05 to 25
Na_2O	0,05 to 5
K_2O	0,05 to 5
TiO_2	0,05 to 5
Mn_3O_4	0,005 to 5
P_2O_5[a]	0,01 to 5
SO_3	0,05 to 10

NOTE 1 Additional analytes can be included in the method, provided that appropriate validation using reference materials is carried out.

NOTE 2 The precision statistics can be determined using suitable reference materials.

NOTE 3 The method described in this document has been tested for the following additional analytes: BaO (0,01 % to 1 %); SrO (0,01 % to 1 %) and ZnO (0,005 % to 1 %).

[a] For information relating to phosphorus calculations, refer to Annex D.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1171, *Solid mineral fuels — Determination of ash*

ISO 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

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ISO 13909-4, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

ISO 13909-6, *Hard coal and coke — Mechanical sampling — Part 6: Coke — Preparation of test samples*

ISO 18283, *Hard coal and coke — Manual sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1213-2 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

The specimen on which X-ray fluorescence measurements are made is prepared by incorporating the test portion of the sample, via fusion, into a borate glass disc using a casting or press-quenching procedure. By using such a specimen, particle size effects are eliminated.

Calibration is carried out using pure chemicals and/or reference standards, and by making matrix corrections for inter-element effects.

It is expected that laboratories using this document have experience in analysing coal ashes of certain compositions and that they employ calibrations that cover these compositions.

5 Reagents

5.1 General

Unless otherwise specified, all reagents shall be of analytical grade and only distilled water or water of equivalent purity shall be used.

5.2 Flux and heavy absorber

Borate fluxes of differing compositions have been found suitable for preparing glass discs from coal/coke ash samples. These fluxes are based on mixtures of one or more of the following: lithium tetraborate, lithium metaborate and lanthanum oxide. Flux may be purchased commercially or prepared by fusion of a mixture of individual reagents.

NOTE The levels of contamination in the flux should be checked. Because levels of contamination may vary from batch to batch, the same batch of flux should be used for all specimens (synthetic standards, external standards and unknowns). When using a fresh batch of flux, reference material specimens should be prepared to determine whether adjustments to the calibration are required.

6 Apparatus

All apparatus shall be constructed from materials that are thermally stable and chemically inert under the conditions of the procedure.

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6.1 Crucible, made from a non-wetting platinum alloy.

The crucible shall have sufficient capacity to hold the flux and sample required for fusion. Normally, 15 ml crucibles are adequate for discs of 32 mm in diameter, and 25 ml crucibles for discs of 40 mm in diameter.

NOTE Either platinum/gold or platinum/gold/rhodium alloys are suitable.

Because the crucible and lid (if used) are to be used for fusion work, the normal precautions associated with the care of hot platinum ware should be observed. It is necessary, therefore, to have suitable tongs and a surface on which to rest the crucible. The hot crucible can be rested on a refractory surface, which shall be kept very clean.

Although the crucible is fabricated from an alloy that is not wetted by the glass, for the greatest precision, the crucible should be cleaned between each fusion. The use of citric acid (mass concentration of 20 %), dilute hydrochloric acid (volume fraction of 10 % to 50 %) or dilute chlorine-free nitric acid (volume fraction of 10 %) have proven to be suitable for crucible cleaning. The use of an ultrasonic bath will accelerate this process. An alternative method of cleaning is to fuse several grams of flux in the crucible, moving the melt around so as to clean the entire inner surface. The molten flux is poured from the crucible. If a droplet adheres to the crucible, this can easily be flaked off when the crucible is cold.

6.2 Mould, made from non-wetting material. For example, platinum alloy is commonly used for casting discs, and aluminium or graphite is suitable for press quenching.

In the casting technique, the bottom of the disc is the analytical surface. The mould should therefore be flat and sufficiently thick that it is not easily deformed. Casting moulds should be checked regularly for flatness and should be polished regularly, to ensure that the disc releases from the mould. Platens for press-quenching are constructed so that the depth gradually increases from the perimeter to the centre. The analytical surface for press-quenched discs is the surface which contacts the plunger head. Care should be taken not to choose the platen side for intensity measurements.

6.3 Crucible tongs, platinum-tipped or stainless steel or titanium.

NOTE Stainless steel or titanium tongs are a suitable alternative to platinum-tipped tongs.

6.4 Desiccator, containing freshly regenerated, self-indicating silica gel.

6.5 Sample holders, used for specimen presentation.

6.6 X-ray fluorescence spectrometer, any conventional wavelength dispersive (sequential, simultaneous, or combination simultaneous/sequential) vacuum path X-ray fluorescence spectrometer may be used, provided that it conforms to precision requirements at the 0,1 % (10^6 counts) precision level.

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7 Sample

7.1 Coal and coke ash prepared in the laboratory

The coal or coke sample shall be the analysis sample, prepared to a nominal top size of 212 µm. Sample preparation shall be in accordance with ISO 13909-4 for coal samples or ISO 13909-6 for coke samples or ISO 18283.

The sample shall then be ashed using the procedure specified in ISO 1171.

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7.2 Coal and coke ash

Laboratory-prepared ashes for XRF analysis should be received at the laboratory, freshly prepared If not freshly prepared coal or coke ash, reheat at 815 °C for 15 min and cool in a desiccator immediately prior to weighing for analysis.

Boiler ash received at the laboratory should be ground to a nominal top size of 63 µm and dried at 105 °C for 1 h, then stored in a desiccator over freshly regenerated, self-indicating silica gel.

8 Procedure

8.1 Number of determinations

Discs shall be prepared and analysed as single determinations and a reference material shall be prepared and analysed at the beginning and end of each batch of samples containing a maximum of 25 samples.

8.2 Test portion

The mass of the test portion shall be appropriate for the mould size and sample flux ratio chosen.

NOTE 1 Typically, sample-to-flux ratios are in the range 1:5 to 1:10.

NOTE 2 Typical total mass (sample plus flux) is 4 g for a 32 mm diameter and 7,5 g for a 40 mm diameter mould.

8.3 Check test

At least one reference material of similar composition to the samples being analysed shall be included with each batch of test samples and shall be analysed in parallel with, and under the same conditions as, the test samples. The results for the batch of samples shall be rejected if the results for the reference material fail to meet the acceptance criteria detailed in Annex A.

8.4 Calibration

Calibration shall be effected using either reference materials, synthetic standards or mixtures of reference materials and synthetic standards. Reference materials shall be pre-treated in accordance with the instructions contained on their certificates. If synthetic standards are used, stoichiometric chemicals of appropriate purity shall be used. Chemicals of 99,99 % or higher purity are recommended. Pure chemicals shall be appropriately pre-treated, immediately prior to weighing to ensure stoichiometry and freedom from moisture. Calibration discs shall be prepared in accordance with 8.5.

NOTE Reagents suitable for the preparation of synthetic calibration standards and their appropriate pre-treatment conditions are listed in Annex B.

8.5 Preparation of the fused discs

8.5.1 General

A number of different fluxes and sample-to-flux ratios have proven suitable for the preparation of fused discs that are acceptable for XRF analysis of samples of coal or coke ash. Any consistent combination of sample mass, flux mass and flux type may be used to prepare the fused discs, provided that

a) disc preparation quality conforms to the requirements of Annex C, and

b) the precision and accuracy of reference materials prepared using this sample flux combination conforms to the requirements of Annex A.

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Discs may be prepared by the casting or press-quenching technique. Both methods will produce glass discs of adequate quality. If the casting technique is used, the sample may be fused with the flux in a crucible and then poured into a separate mould; if an appropriately shaped crucible is used, the fusion may be carried out and the glass allowed to cool in the same crucible.

A conventional electric furnace, high-frequency furnace, or gas burner may be used for heating.

Disc-making machines are available commercially, and these may be used to fuse and cast the discs.

Prior to measurement, discs shall be inspected visually, paying particular attention to the analytical surface. The discs shall not contain undissolved material, and shall be whole and free from crystallization and bubbles. Defective discs shall be re-fused or discarded and substitute discs prepared.

Laboratory-prepared ashes for XRF analysis should be freshly prepared; if not, it will be necessary to reheat these to the ashing temperature and hold for 15 min at this temperature prior to preparation of the XRF disc.

Fluxes are in general hygroscopic and hence shall be dried prior to use, or a loss on fusion (LOF) determination shall be carried out and the mass of flux used corrected to an anhydrous basis. Since it is difficult to maintain flux in an anhydrous state, it is recommended that, for work requiring high accuracy, the latter practice be adopted.

It may be necessary to determine the LOF for boiler ash samples. This is done by allowing the fused material to cool (in a covered crucible) prior to pouring into the mould and reweighing the cooled crucible and fused contents. LOF is equal to the mass of the crucible and its contents prior to fusion minus the mass of the crucible and its contents after fusion. After weighing, the crucible contents is remelted and swirled (to ensure homogeneity), then cast or press-quenched in the normal manner. If loss-eliminated alphas are used, then LOF need not be determined (see Reference [1]).

NOTE Loss of sulfur can occur at the high temperatures (>1 050 °C) used in the preparation of fused discs.

8.5.2 Disc storage

As soon as possible after preparation, the discs shall be transferred to a desiccator so that adsorption of moisture and the possibility of contamination are minimized.

To avoid contamination of the analytical surface, discs shall be handled by their edges. Care shall be exercised to avoid touching the analytical surface with hands or fingers.

8.5.3 Fused disc quality

The quality of disc preparation shall be checked in accordance with Annex C, as follows:

a) prior to each new operator commencing analysis of test samples;

b) by each operator annually.

8.6 XRF measurement

8.6.1 Instrumental conditions

Instruments shall first be tested for precision in accordance with the manufacturer's instructions.

Suggested analytical lines to be used, and conditions of measurement, are given in Table 2.

Measurements shall be made under vacuum, using an appropriate counter and using specimen rotation if available. A single- or dual-anode target X-ray tube may be used. It is recommended that pulse height selection be used, particularly in the case of P Kα, where low concentrations are normally being determined.

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Measurements are made at the Kα line positions, except where there are overlap interferences at the Kα positions. Counting times shall be selected so that, for calibration discs at approximately the midpoint of the range of application of the method (see Table 1), at least the number of counts shown in Table 2 are accumulated. Using these counting times, the emitted intensity of each disc shall be recorded as its accumulated count.

The crystals listed in Table 2 are only some of those that may be used. In the case of P Kα, the Ge(111) crystal is recommended because it does not give second-order wavelengths. If, however, this crystal is unavailable and a PE crystal is used, pulse height selection shall be used and the settings shall be very carefully selected so that the possibility of interference from the second-order wavelength of Ca K$\beta_{1,5}$ is minimized.

Coarse or fine collimators can be used but, where there is the likelihood of line interference, a fine collimator shall be used, e.g. in the determination of manganese using a chromium target X-ray tube. To minimize interference due to line overlaps, a fine collimator should be used for measuring Ti Kα.

Where the crystal contributes significantly to the background, it is also advisable to use a fine collimator, e.g. magnesium and sodium determinations using the TlAP crystal.

Primary-beam filters should not be used as a method for reducing the count rate, as they will alter matrix effects. The exception to this is the determination of manganese using a chromium target X-ray tube, where a filter is required for low backgrounds.

The voltage, in kilovolts, should be that recommended by the supplier for the type of X-ray tube being used. The current, in milliamperes, used with the X-ray tube is not critical but shall be selected so that count rates do not exceed the capability of the counting circuits.

The background will generally be lower with a lower voltage, and there is little or no gain in using a voltage greater than 40 kV for the elements being determined. If magnesium is being determined with a TlAP crystal and a rhodium target X-ray tube, the background will drop considerably as the voltage is lowered.

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Table 2 — Analytical lines, crystals and recommended number of counts

Suggested line	Example of crystal	Minimum number of counts for mid-range calibration disc
Si Kα	PE	2×10^5
Al Kα	PE	2×10^5
Fe Kα	LiF (200)	4×10^5
Ca Kα	LiF (200)	10^5
Mg Kα	LSM[a] (TlAP)	10^5
Na Kα	LSM[a] (TlAP)	2×10^4
K Kα	LiF (200)	4×10^4
Ti Kα	LiF (200)	4×10^4
Mn Kα	LiF (200)	4×10^4
P Kα	Ge(111)	4×10^4
S Kα	Ge(111)	4×10^4

[a] Layer synthetic multilayer.

8.6.2 Monitor measurements

To compensate for drifts in X-ray tube output intensity, all X-ray measurements shall be made relative to a monitor specimen. Although different monitor specimens could be used for each component, it is most convenient to use a single specimen containing all the components to be measured. The requirements of the monitor specimen are that it is stable, at least for the time necessary to complete all the measurements associated with a batch of samples, and that it gives intensities for the various

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components that are comparable to, and preferably a little greater than, those of the specimens to be measured.

A suitable monitor may be prepared by fusion of a mixture of pure chemicals and flux.

The frequency of running the monitor will depend on the stability of the instrument, as determined by the use of suitable quality control samples.

9 Calculation

9.1 General

The results for the concentration of the analysed elements (see Table 1) in coal or coke ash shall be calculated from the measured X-ray intensities after correction for instrumental drift, dead time and matrix effects.

9.2 Correction of instrumental drift

The measured X-ray intensities shall be corrected for instrumental drift using Formula (1):

$$N = \frac{N_o \cdot M^*}{M} \tag{1}$$

where

N_o is the uncorrected (measured) count;

N is the count after drift correction;

M^* is the count on the monitor disc at the start of the measuring bracket;

M is the count on the monitor disc at the end of the measuring bracket.

9.3 Correction for dead time losses

The drift-corrected X-ray intensities shall be corrected for counting losses arising from the dead time of the counting equipment using Formula (2):

$$N_c = \frac{N}{T - N \cdot t} \tag{2}$$

where

N is the counts (after drift correction, using the monitor specimen) accumulated in T seconds;

T is the time, in seconds, in which counts were accumulated;

t is the dead time, in seconds;

N_c is the count rate for dead time losses, in counts per second (c/s).

NOTE Automatic dead time correction is acceptable, provided that it is correctly adjusted.

9.4 Matrix corrections

XRF intensities shall be converted to concentrations that have been appropriately corrected for interelement (matrix and spectral overlaps) effects. It is preferable that matrix correction be carried out using matrix correction (alpha) coefficients, calculated using fundamental parameters.

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Other correction procedures may be used, provided that the precision and accuracy of the method is shown to be acceptable (see Annex A).

NOTE There are commercially available computer programs that carry out matrix corrections.

10 Reporting results

The results (single determination) shall be reported to the number of decimal places and in the order indicated in Table 3.

The results will not always total 100,0 %, due to the following factors:

a) the tolerance on each of the analytical determinations and the effect of the summation of these tolerances;

b) the presence of other trace elements, e.g. boron, barium, strontium, zinc;

c) the presence of mineral forms other than oxides in the ash.

The total of results, including LOF (if required), is expected to lie within the range 98,5 % to 101,5 %.

Table 3 — Reporting of results

Oxide	Concentration range %	Report to the nearest % (mass fraction)
SiO_2	5 to 100	0,1
Al_2O_3	5 to 80	0,1
Fe_2O_3	0,1 to 5	0,02
	5 to 25	0,1
CaO	0,05 to 5	0,01
	5 to 25	0,1
MgO	0,05 to 5	0,01
	5 to 25	0,1
Na_2O	0,05 to 5	0,01
K_2O	0,05 to 5	0,1
TiO_2	0,05 to 5	0,1
Mn_3O_4	0,005 to 0,05	0,001
	0,05 to 1,0	0,01
	1,0 to 5,0	0,1
P_2O_5	0,01 to 1	0,01
	1 to 5	0,1
SO_3	0,05 to 5	0,01
	5 to 10	0,1
Total	98,5 to 101,5	0,1

11 Precision

The values of repeatability and reproducibility should not exceed those given in Table 4.

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Table 4 — Precision data

Oxide	Concentration range %	Repeatability	Reproducibility
Al_2O_3	13 to 28	0,26	1,6
BaO	0,02 to 0,60	0,01	0,286 X – 0,003
CaO	0,05 to 26,5	0,015 X + 0,030	0,088 X + 0,126
Fe_2O_3	0,6 to 21	0,017 X + 0,020	0,073 X + 0,302
K_2O	0,35 to 2,71	0,014 X + 0,008	0,049 X + 0,044
MgO	0,10 to 4,74	0,015 X + 0,015	0,25
MnO_2	0,004 to 0,21	0,01	0,307 X
Mn_3O_4	0,004 to 0,18	0,01	0,307 X
Na_2O	0,03 to 4,38	0,029 X + 0,022	0,121 X + 0,211
P_2O_5	0,07 to 1,84	0,023 X + 0,004	0,084 X + 0,032
SiO_2	27,9 to 69,9	0,47	2,46
SO_3	0,06 to 14,8	0,017 X + 0,077	0,121 X + 0,271
SrO	0,01 to 0,38	0,024 X + 0,005	0,141 X + 0,013
TiO_2	0,58 to 3,8	0,022 X + 0,001	0,066 X – 0,001

12 Test report

The test report shall include the following information:

a) complete identification of the sample tested;

b) a reference to this document, i.e. ISO 13605:2018;

c) the results of the determinations including the total of results, expressed on a dry basis as the oxide forms and in the order given in Table 3;

d) the following statement:

"The results of an ash constituent analysis do not necessarily total 100,0 %."

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Annex A
(normative)

Acceptance of analytical results for laboratory method

A.1 Acceptance of analytical values

The difference between the result obtained for the reference material and the reference value of the reference material shall be statistically insignificant at the 95 % confidence level. If the difference is significant, the analysis shall be repeated. If the difference is again significant, the procedure shall be repeated using a different reference material. If the difference is still significant, then it is likely that there is a problem with the laboratory method which must be investigated and rectified prior to the analysis of test samples. Possible problems include poor quality glass discs, incomplete fusion, sample contamination and XRF instrument faults.

The laboratory method shall only be used for the analysis of test samples if the difference between the result obtained for the reference material and the reference value of the reference material is statistically insignificant.

For a reference material that has been analysed by at least 10 laboratories using methods that are comparable in both accuracy and precision with this method, Formula (A.1) may be used to test the significance of the difference:

$$\left|A_c - A\right| \leq 2 \cdot \sqrt{\frac{S_{Lc}^2 + \frac{S_{Wc}^2}{n_{Wc}}}{N_c} + \sigma_L^2 + \frac{\sigma_r^2}{n}} \tag{A.1}$$

where

A_c is the reference value;

A is the result or the mean of results obtained for the 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 reference material (in most cases $n = 2$);

σ_r is the within-laboratory standard deviation;

σ_L is the between-laboratory standard deviation.

If the condition of the above formula is satisfied, i.e. if the left-hand side is less than or equal to the right-hand side, then the difference $\left|A_c - A\right|$ is statistically insignificant; otherwise, it is statistically significant.

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The following procedure should be used where the information on the reference material certificate is incomplete:

a) If there are insufficient data to enable the between-laboratories standard deviation to be estimated, delete the expression $\frac{S_{\mathrm{Wc}}^2}{n_{\mathrm{Wc}}}$ and regard the expression S_{Lc}^2 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 this document. If this cannot be avoided, use Formula (A.2):

$$\left|A_{\mathrm{c}} - A\right| \leq 2\sqrt{2\sigma_{\mathrm{L}}^2 + \frac{\sigma_{\mathrm{r}}^2}{n}} \tag{A.2}$$

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Annex B
(informative)

Reagents suitable for the preparation of synthetic calibration standards

Reagent	Pre-treatment conditions
SiO_2	Heat at 1 000 °C in a platinum crucible for 1 h, cool in a desiccator.
Al_2O_3	Heat at 1 250 °C in a platinum crucible for 2 h, cool in a desiccator.
Fe_2O_3	Heat at 1 000 °C in a platinum crucible for 1 h, cool in a desiccator.
$CaCO_3$	Dry at 100 °C for 1 h and cool in a desiccator.
MgO	Heat at 1 000 °C in a platinum crucible for 1 h, cool in a desiccator.
Na_2SO_4	Dry at 100 °C for 1 h and cool in a desiccator.
KH_2PO_4	Dry at 105 °C for 1 h and cool in a desiccator.
TiO_2	Heat at 1 000 °C in a platinum crucible for 1 h, cool in a desiccator.
Mn_3O_4	Prepare by heating manganese dioxide (MnO_2) at 1 000 °C for 24 h and then cool. Crush the resultant lumpy Mn_3O_4 to a fine powder. Heat at 550 °C for 1 h and cool in a desiccator.

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Annex C
(normative)

Standard deviation of glass disc preparation

C.1 General

This annex specifies a procedure for determining the precision of disc preparation.

C.2 Procedure

This procedure for determining the standard deviation of disc preparation shall be undertaken by each operator prior to commencing work on actual test discs for the first time. The procedure shall be as follows:

a) Select two coal ashes and prepare five replicate discs from each ash using the procedure specified in 8.5.

b) Measure the intensity of the Si Kα radiation for each disc, accumulating at least $4 \cdot 10^6$ counts for each disc. Repeat this measurement five times, reloading the specimens between each measurement.

c) Report the results as shown in Table C.1.

Table C.1 — Example of recording results for the evaluation of precision of disc preparation

Disc	Measurement[a]						
	1	2	3	4	5	Average	Range
1	41 502	41 485	41 511	41 498	41 492	41 498	26
2	41 505	41 501	41 497	41 513	41 537	41 510	40
3	41 470	41 455	41 459	41 409	41 453	41 449	61
4	41 526	41 545	41 558	41 517	41 565	41 542	48
5	41 500	41 532	41 517	41 494	41 508	41 510	38

[a] Measurement units can be counts, counts per second or time for a fixed number of counts.

d) Evaluate the results by calculating:

1) the average ($\overline{X}$) of the five average readings,

2) the average ($\overline{R}$) of the five range readings, and

3) the range (R) of the five average readings.

Substitute the values obtained for $\overline{X}$, $\overline{R}$ and R into Formulae (C.1) and (C.2) to obtain the standard deviation of specimen preparation (S_p) and coefficient of variation (S_r).

$$S_p = \sqrt{\left(\frac{R}{2{,}33}\right)^2 - \frac{1}{5}\left(\frac{\overline{R}}{2{,}33}\right)^2} \qquad \text{(C.1)}$$

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