
**Chemical analysis of refractory products
by X-ray fluorescence (XRF) — Fused
cast-bead method**

*Analyse chimique des matériaux réfractaires par fluorescence de
rayons X — Méthode de la perle fondue*

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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 12677 was prepared by Technical Committee ISO/TC 33, *Refractories*.

This second edition cancels and replaces the first edition (ISO 12677:2003), which has been technically revised. Although the method in this International Standard has been considerably modified editorially and in layout, the technical changes are limited. Some minor corrections have been made to certain equations. The only significant changes are a reference to a further International Standard method (being prepared) for the preparation of reduced materials for analysis by this standard, and instructions on how to add other constituents to calibrations at the end of 10.2.4, *Purity and preparation of reagents*.

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Chemical analysis of refractory products by X-ray fluorescence (XRF) — Fused cast-bead method

1 Scope

This International Standard specifies a method for the chemical analysis of refractory and technical ceramic raw materials, intermediates and products, by means of the X-ray fluorescence (XRF) fused cast-bead method. Typical materials that can be analysed by this standard are given in Clause 3. This International Standard is not applicable to non-oxide materials, such as silicon carbides or nitrides, etc. The method is applicable to a wide range of materials containing a wide range of elements.

NOTE 1 The presence of significant amounts of certain elements, such as tin, copper, zinc and chromium, can present difficulties in the fusion process. In this case, the Bibliography can be referred to.

NOTE 2 Constituents at concentrations greater than 99 % (on a dried basis) are reported by difference, provided that all likely minor constituents and any loss on ignition have been determined. These figures can also be checked by direct determination.

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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 Guide 35:2006, *Reference materials — General and statistical principles for certification*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 26845, *Chemical analysis of refractories — General requirements for wet chemical analysis, atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods*

3 Types of material

Listed below are various types of ceramic material that have been successfully analysed by this method and for which statistical data is available (see Annex I). The list is not exhaustive but serves as a guide to those using this International Standard for the first time.

- a) High alumina > 45 % Al_2O_3
- b) Alumino-silicate 7 % to 45 % Al_2O_3
- c) Silica > 93 % SiO_2
- d) Zircon
- e) Zirconia and zirconates
- f) Magnesia

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- g) Magnesia/alumina spinel (~70/30)
- h) Dolomite
- i) Limestone
- j) Magnesia/chromic oxide
- k) Chrome ore
- l) Chrome-alumina
- m) Alumina/magnesia spinel (~70/30)
- n) Zirconia-alumina-silica cast material (AZS)
- o) Calcium silicates
- p) Calcium aluminates
- q) Magnesium silicates

A list of elemental ranges and required detection limits are given in Annex A.

NOTE 1 Some of the above material types can be accommodated for common calibrations (see 10.3.4).

NOTE 2 Reduced materials, such as silicon carbide, cannot be determined directly by this International Standard and so are not listed above. Such materials require special methods both for loss on ignition and fusion into a bead prior to XRF analysis. Suitable procedures are described in ISO 21068-1, ISO 21068-2 and ISO 21068-3 and further methods are under development by the refractory standards system. Once reduced materials are suitably ignited and subsequently prepared as fused beads, this standard can be applied to the rest of the procedure.

WARNING — Failure to pretreat reduced materials, such as silicon carbide, properly not only leads to erroneous results but will also cause damage to valuable platinum alloy crucibles and dishes.

4 Principle

The powdered sample is fused with a suitable flux to destroy its mineralogical and particulate composition. The resultant melt is cast into the shape of a glass bead which is then introduced into an XRF spectrometer. The intensities of the fluorescent X-rays of the required elements in the bead are measured and the chemical composition of the sample is analysed by reference to previously determined calibration graphs or equations and applying corrections for inter-element effects. The calibration equations and inter-element corrections are established from beads produced using pure reagents and/or series reference materials (SeRMs), prepared in the same way as the samples. Certified reference materials (CRMs) may be used providing they meet all the requirements of 10.2.2 and 10.4.1.

Because of the universality of the fused cast-bead technique, various fluxes and modes of calibration are permitted, providing they have been demonstrated as being able to meet certain criteria of repeatability, sensitivity and accuracy. Provided that a laboratory's own methods conform to all the various criteria set down, they will be accepted as conforming to this International Standard.

5 Apparatus

5.1 Fusion vessels, of a non-wetted platinum alloy (Pt/Au 95 %/5 % is suitable). Lids, if used, shall be of a platinum alloy (not necessarily non-wetted).

NOTE A useful guide to the care of platinum is given in Reference [5] of the Bibliography.

5.2 Casting moulds, of a non-wetted platinum alloy (Pt/Au 95 %/5 % is suitable).

NOTE Vessels that serve both as fusion vessels and casting moulds can be used.

5.3 Heat reservoir for casting mould (optional), required in special circumstances when using moulds of small sizes, so that the mould does not cool too rapidly when removed from the furnace. A small piece of flat refractory material is suitable, e.g. a piece of sillimanite batt with dimensions 10 mm × 50 mm × 50 mm.

5.4 Air jet (optional), required to cool the mould rapidly. This may be any device whereon a narrow jet of air can be directed to the centre of the base of the casting dish. A convenient way of doing this is to use the base of a Bunsen burner without a barrel to serve as an air jet.

NOTE In most cases, it is very important to cool the melt rapidly. This is necessary to obtain a homogeneous bead and to free the melt from the dish.

A water-cooled metal plate may also be used.

5.5 Fusion apparatus, electric resistance furnaces or high-frequency induction furnaces that may be heated up to a fixed temperature of between 1 050 °C and 1 250 °C may be used.

5.6 Automatic fusion apparatus, for use in automatic bead preparation (see 9.2) where required.

5.7 Balance, capable of weighing to $\pm 0,1$ mg.

5.8 Mechanical mixer, that moves in a linear or rotary way.

NOTE Vibratory mixers cannot be used as they induce segregation.

6 Sample grinding

This International Standard shall start with a laboratory sample.

NOTE 1 Bulk sampling is not within the scope of this method but can be found in ISO 26845.

The sample shall be ground using tungsten carbide. The appropriate corrections for tungsten carbide (and its binder if necessary) shall be applied to loss on ignition and analysis figures in accordance with Annex B.

NOTE 2 It is permissible to apply the sample grinding methods cited in conventional chemical methods for the classes of materials covered. However, the tungsten carbide method is the preferred method.

The maximum particle size shall be 100 μm .

NOTE 3 The purpose of grinding is to obtain a sample sufficiently fine to be fused easily but below a set limit of introduced contamination. But for certain samples that are difficult to fuse (e.g. chrome ores), finer grinding to less than 60 μm might be necessary.

One of the following two methods shall be used to obtain the required particle size.

- a) For mechanical grinding devices, establish what grinding times are sufficient to grind the various samples to be analysed to the correct fineness and thereafter apply these minimum times for grinding. In order to establish grinding times, use the mechanical grinder to prepare typical materials analysed for progressively increasing lengths of time of ~ 2 min. Sieve each ground sample through a 100 μm sieve until a grinding time is reached where the entire sample passes through the sieve. Then use this time for that material or the longest time of any material analysed, if applied to all materials. When grinding hard materials, such as chromite, sieving shall be used, but this might induce segregation. Therefore, after sieving, the sample shall be mixed thoroughly by stirring or tumbling prior to transferring to a sample tube. Because heavier minerals can segregate on standing, it is advisable to stir the sample once more, prior to weighing out.
- b) After hand-grinding for 20 s, sieve the ground powder through a sieve of 100 μm aperture, in accordance with ISO 565. Regrind any material remaining on the sieve for a further 20 s, sieve and repeat this procedure until the whole of the sample passes through the sieve. Transfer the sample to a suitable container and mix for 1 min, using a mechanical mixer such as a vertical linear mixer.

NOTE 4 As the object of the exercise is to obtain a sample suitable for fusion, and not to test the fineness of the sample itself, method a) is generally preferred.

7 Loss on ignition (and/or drying)

Loss on ignition shall be carried out in accordance with ISO 26845.

8 Flux

8.1 Choice of flux and ratio of flux to sample

8.1.1 One of the advantages of the XRF fused cast-bead method is that a wide variety of fluxes may be chosen. For a given calibration, the same flux shall be used throughout. The conditions given in 8.1.2 to 8.1.9 shall be met for any flux and flux/sample ratio used.

NOTE Fluxes used with success in the analysis of refractory materials are given in Annex C. Prefused fluxes have the advantage of lower moisture contents.

8.1.2 Under the conditions of preparation used, the sample shall be totally dissolved by the flux and shall not come out of solution during the casting procedure.

8.1.3 The resulting bead shall be transparent and show no signs of devitrification.

8.1.4 At a reasonably high counting time (≤ 200 s), the required detection limits shall be achieved for the elements determined. Detection limits are defined as in Clause 14 and listed in Annex A.

8.1.5 At a reasonable counting time (≤ 200 s), the counts recorded for each element determined shall give the required standard of repeatability for the determination of that element (as measured according to 12.1 and defined as in G.1).

8.1.6 A heavy element absorber may be incorporated into the flux provided that:

- a) it does not reduce sensitivities so that conditions 8.1.4 and 8.1.5 are not met;
- b) the heavy element does not have a line overlap with any of the elements to be determined.

8.1.7 If volatile components are to be determined, then a flux of sufficiently low melting point, which permits a fusion temperature low enough to retain that element during fusion, shall be used.

8.1.8 For the determination of elements that alloy with platinum (e.g. lead, zinc, cobalt), the melting point shall be such as to allow fusion below the temperature at which this reaction occurs (1 050 °C).

8.1.9 The flux shall be pure with respect to the analytes determined. As the flux to sample ratio is greater than 1 (see Annex C), impurities to the flux can influence the measured result negatively. The greater the ratio of the flux to sample, the greater the influence. Therefore, the permitted levels of impurity of analyte levels in the flux shall be no more than:

$$D/(3R)$$

where

R is the ratio of flux to sample;

D is the detection limit claimed for the determination of the analyte element.

Most reagents sold by reputable manufacturers as "flux" grade quality meet this requirement but an analysis shall be obtained for each batch of flux supplied. Recheck calibrations when batches of flux are changed.

8.2 Compensations for moisture in flux

The flux contains a certain amount of moisture, which shall be compensated for in one of two ways.

- a) Calcine the entire quantity of flux required overnight at 700 °C immediately before it is used for analysis, and store it in a desiccator.
- b) Carry out duplicate losses on ignition on 1 g portions of well-mixed flux for each kilogram of flux used. Carry out the calcining at the normal fusion temperature for 10 min, or the normal fusion time, whichever is the greater [see 9.1.2 f)]. Store the flux in a tightly sealed container except when in use. The loss on ignition, expressed as a percentage by mass, w_L , is then used to calculate a flux factor, F [see Equation (1)], which is in turn used to calculate the mass of the unignited flux needed to produce the required mass of flux on the ignited basis (F times the required mass of ignited flux = required mass of unignited flux). Carry out this loss on ignition at weekly intervals or for each kilogram of flux used, whichever is the more frequent.

$$F = \frac{100}{100 - w_L} \quad (1)$$

NOTE The compensation might be unnecessary if the loss on ignition is 0,50 % or lower (prefused fluxes).

9 Fusion casting procedures

9.1 Fusion of samples and casting of beads

9.1.1 Choice of procedure

At several of the stages, a choice of procedures is given. Once a choice has been made, the procedure shall be adhered to throughout, unless a total recalibration is carried out.

9.1.2 Requirements

Before fusing the samples and casting the beads, the following requirements shall be satisfied.

- a) Duplicate or single beads may be prepared; the number used shall be stated in the test report.
- b) The total mass of sample and flux shall be chosen for the particular casting-mould type used, and this mass shall always be the same.
- c) The ratio, R , by mass of the flux to that of the sample, shall be the same for the material type analysed.
- d) The melts produced shall be visually homogeneous.
- e) There shall be no measurable loss of any component from the sample during fusion, e.g. loss by reduction or evaporation (excessive temperature).
- f) The variations of any loss of flux shall be minimized by using consistent times and temperature during fusion in the preparation of both calibration standards and samples.
- g) The sample shall not be contaminated in any way by the sample preparation by any constituent being measured on that sub-sample. This can either be established by knowing the composition of the grinding media or by measuring the amount of contamination added in grinding pure materials or materials of known composition.
- h) The beads produced shall be free from blemishes on the chosen measuring surface.
- i) If the top surface of the bead is to be used for analysis, it shall be either convex or flat and be symmetrical across any diameter.

- j) Standard glass beads of known composition shall be prepared in the same way as sample beads.
- k) If moulds become distorted in use, they shall be reshaped by pressing in a suitable former. If the bottom (flat) surface of the bead is used for analysis, the top surface of the mould shall also be kept flat and free from blemishes.
- l) Beads shall be infinitely thick for the X-ray wavelengths measured. For line parameters used in refractory analysis, infinite thickness is normally achieved.

NOTE 1 Duplicate beads are preferable to single beads. However, if all the oxides given in Annex A are determined for the relevant class of material, an analytical total will be achieved, which acts as a check on the result of analysis.

NOTE 2 Fusions at 1 200 °C will volatilize certain elements, e.g. sulfur, even when an oxidizing agent is used.

9.1.3 Conversion of the sample to bead form

The sample to be analysed may be converted into bead form in a number of ways.

- a) Calcine the sample to constant mass at $(1\,025 \pm 25)$ °C, desiccate and allow to cool to room temperature. Weigh it in the fusion dish and record the mass, m , to the nearest 0,000 1 g. Weigh the flux samples as described in 8.2.
- b) Take a sample of uncalcined flux of mass $R \cdot m \cdot F$ and mix thoroughly with the sample, where F is the flux factor determined in 8.2 b). Dry the sample to constant mass at (110 ± 10) °C. Weigh in the fusion dish and record, to the nearest 0,000 1 g, the sample mass

$$m \left(1 + \frac{w_L}{100} \% \right)$$

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where w_L is the percentage by mass of sample lost during ignition at $(1\,025 \pm 25)$ °C.

As in item a), the sample may be mixed with either calcined or uncalcined flux.

NOTE 1 For problems affecting the fusion of materials containing chromium oxide or zirconia, see 9.4.

Fuse the sample and flux together, with occasional swirling, until the sample is seen to be dissolved and the melt homogeneous.

During the initial part of the fusion process, fuse carbonate samples slowly to avoid “spurting” (ejection of sample or flux).

NOTE 2 In the case of limestone, dolomite and magnesium carbonate, it is preferable to weigh out an amount of the dried sample, corrected for loss on ignition, for fusion.

NOTE 3 The fusion temperature can be specified according to material type.

9.1.4 Manual casting of beads

9.1.4.1 General

The final part of the fusion process consists of heating the fusion vessel, the mould and the heat reservoir (if used) in a muffle furnace $(1\,200 \pm 50)$ °C for 5 min. Then cast the beads using one of the following methods.

- a) **Outside the furnace:** after 5 min at $(1\,200 \pm 50)$ °C, remove the heat reservoir (5.3) from the furnace (5.5) and place it on a horizontal surface. Immediately place the mould onto the heat reservoir. Then remove the lid from the fusion vessel and immediately pour the melt into the casting mould (5.2).

- b) **In the furnace:** after 5 min at $(1\,200 \pm 50)$ °C, remove the lid from the fusion vessel (5.1) and pour the melt into the mould (5.2) inside the furnace (5.5), ensuring that as much of the melt is transferred to the mould as possible. Remove the mould from the furnace and place it on a horizontal surface.
- c) **Combined fusion mould:** after 5 min at $(1\,200 \pm 50)$ °C, remove the fusion vessel from the furnace. If a releasing agent is not used, the melt can rise up the sides of the vessel. Therefore, careful manipulation of the vessel is required to work the melt into the mould part of the vessel. Then put the fusion vessel on a graphite brick to cool.
- d) **Mould heated over a burner:** after preparation of the melt at the fusion temperature and the time chosen for that type of material, pour the melt into the preheated mould and turn the burner off. Allow the melt to solidify and use an air jet (5.4), as described in 9.1.4.2., or a water-cooled metal plate to accelerate the cooling process.

When the top surface of the bead is used [not c)] for subsequent analysis, a rippled surface produced in the casting process can lead to erroneous results. In order to avoid this rippled effect, the melt should be poured into the mould at a point nearer to the edge of the mould than the centre. When using top surfaces, in order to maintain a uniform curvature on the top surface, it is necessary to get as much of the melt into the casting mould as possible, so as to achieve consistent bead masses.

NOTE Most refractory materials contain small amounts (as low as 0,1 %) of Cr_2O_3 , ZrO_2 , and $\alpha\text{-Al}_2\text{O}_3$ which, if the fusion is not completed at $(1\,200 \pm 50)$ °C, will cause the melt to devitrify. However, if experience shows that devitrification is not a problem, samples can be cast in furnaces at as low as $(1\,050 \pm 25)$ °C, providing calibration standards are prepared the same way.

Small amounts of lithium iodide or iodate, or ammonium iodate, may be added to the melt to assist in preventing cracking of fused beads on cooling and to aid release from the mould. Iodine does have a small line overlap with $\text{TiK}\alpha$, so if low levels of TiO_2 are to be determined, corrections may be needed. If small amounts of releasing agents are to be used, then all samples and any calibration standards prepared should include the same releasing agent added in the same quantity and at the same stage of bead preparation. Maintaining a good polish on the casting moulds should obviate the need for such agents, but there are further problems with samples containing high levels of Cr_2O_3 . It is also possible to use NH_4Br or LiBr but it should be noted that there is a $\text{Br L}\alpha$ line near the $\text{Al K}\alpha$ line. High amounts of Br may cause serious line-overlap problems when measuring low alumina concentrations and corrections may need to be applied. For low levels of Al_2O_3 , iodide or iodate is recommended. The amount of NH_4Br or LiBr added should not exceed 1 mg per gram of sample. If a chromium tube is used, the effect of bromine will be greater; therefore, the effect of bromine on aluminium should be checked before using a bromine-based releasing agent.

9.1.4.2 Cooling of beads

If no air jet is used, allow the mould to cool on a horizontal surface. If the air jet is used, transfer the mould to it when the melt has cooled from red heat. The melt may be molten or solid at this stage; if it is molten and top surfaces are to be measured, ensure that the support over the air jet is horizontal.

Hold the dish in a horizontal position above the air jet so that the air is directed onto the centre of the base of the mould. When the bead has solidified and released itself, turn off the air jet.

NOTE It might be necessary to encourage the release of the beads at this stage by gently tapping the casting mould on a solid surface.

9.2 Automatic bead preparation

Automatic bead equipment may be used as an alternative to 9.1.4, and shall be in accordance with 9.1.2 and 12.1.

9.3 Storage

Beads can deteriorate because of adverse temperature and humidity conditions. Therefore they should be stored in such a manner as to avoid hydration and contamination.

The measuring surfaces of beads shall be thoroughly cleaned before use, or possibly polished after long-term storage.

NOTE Reported sources of contamination are as follows:

- a) sulfur from vacuum oil in the spectrometer or from the laboratory atmosphere;
- b) sodium and chlorine from the atmosphere if the laboratory is near the sea;
- c) potassium from cigarette smoke;
- d) contamination from the surface of plastic bags that may be used for storing the beads.

9.4 Special problems

Samples with high chromium oxide or zirconia content can create problems during fusion. Chromium oxide is difficult to dissolve in the molten flux and zirconia also suffers from this problem to a lesser degree but can also cause devitrification on cooling, even after complete dissolution. Before establishing methods of fusion for these materials, fusion trials are required to establish a method for preparing the samples of the highest content of these oxides, likely to be encountered by the laboratory. In these trials, optimum flux, sample/flux ratio, temperatures and fusion times need to be established. Normally, different procedures will be required for chrome-bearing material, zircon and zirconia.

NOTE Even if the storage conditions in 9.3 are observed, beads containing high levels of ZrO_2 tend to absorb moisture onto the surface more than other beads. This causes increased backgrounds on light elements. The problem can be cured by drying the bead overnight at 220 °C.

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10 Calibration

10.1 Calibration standards

The calibration equations and inter-element corrections are established using beads produced with pure reagents or series reference materials (SeRMs). The SeRMs are different from certified reference materials (CRMs) which validate the calibrations using pure reagents. CRMs and SeRMs are shown in Annexes D and E, respectively. The series of CRMs meeting the requirements of 10.2.2 and 10.4.1 may be regarded as SeRMs.

10.2 Reagents and series reference materials (SeRMs)

10.2.1 Purity and preparation of reagents

The reagents used to prepare the standard beads for cations shall be pure oxides or carbonates of at least 99,95 % purity (excluding moisture or CO_2) for minor constituents and of at least 99,99 % purity for silica and alumina. For the calibration of elements such as sulfur or phosphorus which do not form stable oxides or carbonates, some guarantee of stoichiometry is required.

It is essential that the reagents be free of (or corrected for) the presence of water (and, in the case of oxides, carbon dioxide) when weighed out for fusion. Also, the reagents shall be in a known oxidation state.

The procedures listed ensure that the correct oxidation state is obtained. The reagents used for calibration shall be of high purity and, when fresh batches of reagents are purchased, they shall be compared with previous batches. Therefore, a fresh bead shall be made at the highest level of content calibrated and measured against a similar bead prepared from the previous batch of the same reagent. The intensities achieved for elements other than those in the reagent shall not differ by more than the detection limit for that element.

In order to obtain the reagents of a known stoichiometry in terms of content, they shall be treated before use as follows.

- a) Silica, alumina and magnesia: determine the loss on ignition as follows. Calcine 5 g of the material, as received, at $(1\ 200 \pm 50)$ °C and keep them at this temperature for a minimum of 30 min. Cool in a

desiccator to room temperature and reweigh. After allowing for this loss, weigh the appropriate amount of the uncalcined material to prepare the standard bead.

- b) Manganese oxide (Mn_3O_4), titanium(IV) oxide and nickel(II) oxide, chromium(III) oxide, zirconia, hafnia, ceria, yttria, lanthia and other rare earths. Calcine 5 g of the material at $(1\ 000 \pm 25)$ °C and keep them at this temperature for a minimum of 30 min. Cool in a desiccator to room temperature before use.

NOTE 1 Rare earths absorb water and carbon dioxide from the atmosphere.

- c) Iron(III) oxide, tin(IV) oxide, cobalt oxide (Co_3O_4) and lithium orthophosphate. Calcine 5 g of the material at (700 ± 25) °C and keep them at this temperature for a minimum of 30 min. Cool in a desiccator to room temperature before use.
- d) Calcium carbonate, barium and strontium carbonates, potassium and sodium carbonates, tungstic oxide, gallium oxide, lithium sulfate. Dry the material at (230 ± 20) °C before use. Cool in a desiccator to room temperature before use.

Other oxides may be added to calibrations, as long as the oxides or their compounds used for the calibration are of a sufficient purity and of guaranteed stoichiometry by heat treatment, etc. In addition, problems regarding volatility of that element/oxide in fusion are taken into account, as well as any tendency for the element to alloy with the fusion vessel during the fusion process. A list of useful references to deal with these points is given in References [5] to [9]. This list is not exhaustive and other references may also be of use. In addition, the calibrations should meet the other requirements of this International Standard.

NOTE 2 A 2 h treatment is usually sufficient for drying.

Tungsten carbide (WC) will be introduced as a contaminant if this media is used for grinding. Laboratories using tungsten carbide for sample grinding should calibrate for WO_3 in order to monitor its presence in samples, and hence correct the analysis and the loss on ignition for WC contamination (see Annex B). Unlike the wet chemical analysis procedure, X-ray fluorescence determinations are not subject to any significant cross interference due to tungsten and furthermore the contaminating tungsten may be easily monitored.

If tungsten contamination exceeds 0,5 %, corrections shall be applied. See Annex B.

10.2.2 Preparation of series reference materials (SeRMs)

SeRMs may be used for calibration instead of synthetic standards. The SeRMs shall cover the following points.

- a) SeRMs shall satisfy the requirements of ISO Guide 35:2006.
- b) SeRMs shall be formulated to provide regular intervals of concentration.
- c) The variation in concentrations of the oxides of the SeRMs shall be independent of each other.
- d) There shall be a minimum of ten SeRMs in a series.
- e) The SeRMs shall be homogeneous.
- f) Full statistics of between- and within-laboratory variation shall be provided in their certification.
- g) The chemical analyses shall be checked by a second technique [e.g. inductively coupled plasma (ICP)].