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OF CHINA**

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**Chemical analysis of refractory products by
XRF—fused cast bead method**

耐火材料 X 射线荧光光谱化学分析熔铸玻璃片法

(ISO 12677: 2003, MOD)

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Foreword

This standard is revised by reference to ISO IM77:2003 *Chemical Analysis of Refractory Products by XRF—Fused Cast Bead Method* (English version). The relevant technical differences between this standard and ISO 12677: 2003 have been identified with the single vertical line at the page margin of the involved clause. The main revised contents are as follows:

- a) Change the quoted standard to Chinese standard equivalent to ISO standard;
- b) Replace “Massive sampling does not belong to the scope of this method.” in the first paragraph of Chapter 5 with “The laboratory sample shall be collected in accordance with GB/T 10325 and GB/T 17617”.
- c) Add “high purity reagents ammonium dihydrogen phosphate and potassium dihydrogen phosphate ” to 10.2.1;
- d) Add “Note: other calibration equation can be also used” to 10.4. 2. 2;
- e) Add part of solvent and dilution ratio contents to Annex C;
- f) In Annex C, add “Note: the number behind the material is the sequence number of the listed material type in Chapter 3, for example, 1 is high aluminum $\text{Al}_2\text{O}_3 > 45\%$, 2 is alumina-silica Al_2O_3 7% ~ 45%,....., 17 is magnesium silicate” corresponding to the contents of Chapter 3 to facilitate to understand.
- g) In Annex D, add the contents of standard samples for the domestic refractory products;
- h) In Annex E, add the contents of series standard samples for the domestic refractory products;
- i) In Annex F, add “the matrix correction coefficient a can be calculated according to the software provided by the instrument supplier”;
- j) Properly modify the admissible error of ferric oxide in Annex G according to the joint test results;
- k) In Annex H, add “Note: the correction method provided by the manufacturer can be

used”;

l) In Annex I, add the application of fusion representation results for the domestic series standard samples;

m) Change the notes under the titles in 12. 9 and Annex A into the paragraphs according to our expression habit;

n) Delete the references.

In addition, editorially modify the places to be corrected in ISO 12677:2003, and add the footnotes at the corresponding places.

Annexes A, B, D, E, F, G and H of this standard are normative; Annexes C and I are informative.

This standard is put forward and controlled by National Technical Committee on Refractory Materials of Standardization Administration of China (SAC/TC 193).

The main drafting units of this standard are Baoshan Iron & Steel Co., Ltd., Sinosteel Luoyang Institute of Refractories Research Co., Ltd. and Sinosteel Refractories Co., Ltd.

The main drafter of this standard are Lu Xiaoming, Tai Li, Jin Delong, Liu Xiaoping, Liang Xianlei and Wang Fuyun.

Chemical analysis of refractory products by XRF—fused cast bead method

1 Scope

This standard specifies a method for chemical analysis of refractory products and materials, and technical ceramics composed of oxides, including the determination of oxide at levels between 0,01 % and 99 % by means of the XRF fused cast bead method.

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

2 Normative references

The articles contained in the following documents have become this standard when they are quoted herein. For the dated documents so quoted, all the modifications (excluding corrections) or revisions made thereafter shall not be applicable to this standard. For the undated documents so quoted, the latest editions shall be applicable to this standard.

GB/T 3286 Methods for chemical analysis of limestone and dolomite

GB/T 6005 Test sieves - Metal wire cloth perforated metal plate and electroformed sheet - Nominal sizes of openings (GB/T 6005-1997, eqv ISO 565: 1990)

GB/T 6900 Chemical analysis of alumina-silica refractories

GB/T 8170 Rules of rounding off for numerical values & expression and judgement of limiting values

GB/T 10325 Shaped refractory products-rule of acceptance,sampling and inspection

GB/T 15000.7-2001 Directives for the work of reference materials(7)--General requirements for the competence of reference material producers (ISO Guide 34:2000,IDT)

GB/T 17617 Refractory products--Sampling of raw materials and unshaped products

ISO/IEC Directives (1992) — Part 2: Methodology for the development of International Standards — Annex B Mention of reference materials

3 Types of material

- 1) High alumina (Al_2O_3) \geq 45%;

- 2) Alumino-silicate (Al_2O_3) 7 % to 45 %
- 3) Silica (SiO_2) ≥ 93 %
- 4) Zircon
- 5) Zirconia and zirconates
- 6) Magnesia
- 7) Magnesia/alumina spinel (~ 70/30)
- 8) Dolomite
- 9) Limestone
- 10) Magnesia/chromic oxide
- 11) Chrome ore
- 12) Chrome alumina
- 13) Alumina/magnesia spinel (~ 70/30)
- 14) Zirconia-alumina-silica cast material (AZS)
- 15) Calcium silicates
- 16) Calcium aluminates
- 17) Magnesium silicates

A list of elemental ranges and required detection limits are given in Annex A. Some of the above material types can be accommodated on to common calibrations (see 10.3.4).

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..

Because of the universality of the fused cast bead technique, various fluxes and modes of calibration are permitted providing they have been demonstrated to be able to meet certain



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