ISO 12677:2011-10 (E)

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

Contents

Page

Foreword v

1 Scope 1

2 Normative references 1

3 Types of material 1

4 Principle 2

5 Apparatus 2

6 Sample grinding 3

7 Loss on ignition (and/or drying) 4

8 Flux 4
8.1 Choice of flux and ratio of flux to sample 4
8.2 Compensations for moisture in flux 5

9 Fusion casting procedures 5
9.1 Fusion of samples and casting of beads 5
9.2 Automatic bead preparation 7
9.3 Storage 7
9.4 Special problems 8

10 Calibration 8
10.1 Calibration standards 8
10.2 Reagents and series reference materials (SeRMs) 8
10.3 Calibration using reagents 10
10.4 Calibration using SeRMs 15

11 Corrections 17
11.1 Line-overlap correction 17
11.2 Background correction 17
11.3 Drift correction 18
11.4 Calculation of results 18
11.5 Software requirements 19

12 Reproducibility and repeatability 20
12.1 Fusion tests 20
12.2 Frequency of instrument tests 20
12.3 Maximum allowance differences of sample holders 20
12.4 Sample measuring positions 21
12.5 Instrument repeatability 21
12.6 Sequential systems 21
12.7 Dead time 22
12.8 Other tests 22
12.9 Flow gas 22

13 Accuracy determined by certified reference materials 22

13.1 Validation of synthetic calibrations 22
13.2 Validation of SeRM calibrations 22
13.3 Fresh beads of the CRMs or synthetic standards used to check SeRM calibrations 22

14 Definitions of limits of detection 23

15 Test report 23

Annex A (normative) Calibration range and required detection limits 24

Annex B (normative) Corrections for tungsten carbide grinding media 28

Annex C (informative) Examples of fluxes/flux ratios 30

Annex D (normative) Examples of CRM to be used to check synthetic calibrations 32

Annex E (normative) Examples of SeRM 38

Annex F (normative) Equation for theoretical calculations 43

Annex G (normative) Certified reference materials (CRMs) 44

Annex H (normative) Method of inter-element correction used to compensate for the effects of co-existing components when using SeRM for calibration 47

Annex I (normative) Standard deviations achieved with certified reference materials 68

Bibliography 75