



Transport
Roads & Maritime
Services

Test method T1032

Chemical analysis of cements and blends
using x-ray fluorescence

NOVEMBER 2012



Revision Summary

Ed/Rev Number	Clause Number	Description of Revision	Authorisation	Date
		New Issue – David Svolos	Gavin Donald	Feb 2005
	All	Revision by David Svolos – generally revised to simplify. Sections 5 and 6 combined, Section 7 results modified to provide reporting accuracy, Appendix A containing method validation	J Friedrich	Apr 2009
Ed 3/ Rev 0	All	Reformatted RMS template	J Friedrich	November 2012

Note that Roads and Maritime Services is hereafter referred to as ‘RMS’.

The most recent revision to Test method T1032 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T1032

Chemical analysis of cements and blends using x-ray fluorescence

1. Scope

This method describes the quantitative chemical analysis of major elements (aluminium, calcium and silicon) and minor elements (iron, magnesium, manganese, potassium, sulphur and titanium) in Portland cements and cement blends using x-ray fluorescence.

Results are normally presented as the percent mass of the element as its oxide.

2. Related Documents

- (a) The preparation of XRF specimens is described in Test Method T1033: 'Preparation of Borate Glass Specimens for X-Ray Fluorescence Analysis of Cements and Blends'.
- (b) The analysis of sodium is not performed by XRF, but instead is performed as outlined in Test Method T1034: Chemical Analysis of Sodium in Cementitious Materials (by Flame Photometry)
- (c) The operation of the MiniPal 2 X-Ray Fluorescence equipment is described in this Test Method, in conjunction with Philips User Guide Manual for MiniPal PW4025 X-Ray Fluorescence Analysis (2002). The calibration of the instrument is not described in this test method, it is assumed that the instrument is already calibrated prior to using this method. The instrument calibration must be qualified using the procedure outlined in Appendix A.
- (d) Code and Practice for Protection against Ionising Radiation Emitted from X-Ray Analysis Equipment (1984), National Health and Medical Research Council, Canberra; R83/990 (4) cat. No. 8501693.
- (e) ANSTO training manual: 'Safe Use of X-Ray Devices', 2002, No. 01/08/97.

3. Apparatus

- (a) X-Ray Fluorescence (in further text: XRF) Spectrometer MiniPal PW4025
- (b) A dedicated computer capable of running the instrument control software.
- (c) Helium gas supply and regulator to deliver constant pressure output (~1 bar). Grade to be used: Ultra High Purity, 99.999%

Note: Maintenance of the XRF Spectrometer is performed by PANalytical. RTA laboratory personnel are only to clean any particulate matter on or near sample holder.

Safety Issues – XRF Spectrometer MiniPal PW4025 is fully shielded against its own radiation. Multiple safety devices (interlocks) disable the generation of x-rays at any malfunction, including lifting the lid of the device while the x-ray tube is not covered during measurement, or at any intentional or unintentional damage to the casing. Full description of safety, hazards and protection is contained in the User Guide Manual (2.2). The device complies fully with regulations (2.3 and 2.6).

4. Procedure

- (a) Prepare the specimens as described in Test Method T1033: 'Preparation of Borate Glass Specimens for X-Ray Fluorescence Analysis of Cements and Blends'.
- (b) Measure the prepared specimens as described in Section 5 (Measuring).
- (c) Retrieve the analysis results as described in Section 6 (Results Retrieval).

5. Measuring

- (a) Open Helium supply tap (valve). Check gas flow pressure on regulator.

- (b) If the green signal light adjacent to 'Open' on the spectrometer is lit, lift the spectrometer's lid.
- (c) Arrange the samples (up to twelve) in the holders by placing the measuring surface facing down. Press the samples gently with finger to assure that they are properly and securely seated in the bottom of the sample holder's cups. Record the samples' numbers in order of their positions (1–12). Close the lid.
- (d) Click the word 'Measure' on the menu bar, then select 'Measure application'. The dialog box 'measure application' containing names of applications appears. Highlight the correct application and click 'OK'.

Note: The current application for measuring a specific sample type changes with each new calibration of the system. The current valid calibration is noted prominently in the XRF Instrument room.

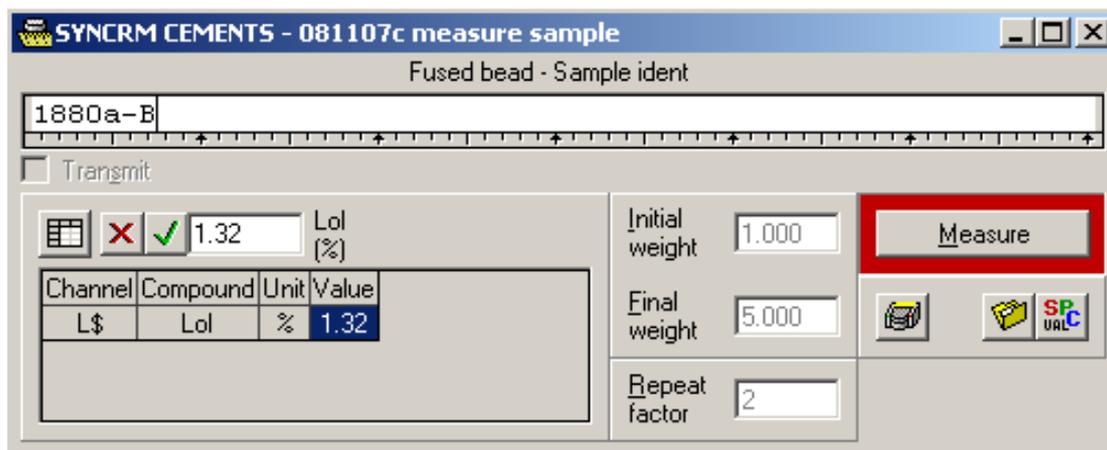


Figure 1: Measure application window

- (e) Type in the sample description. Type in the loss on ignition (LOI %) obtained before the sample preparation. Click 'Measure'.
- (f) Select the changer position when the 'Select changer position' box appears. Once the samples position is selected, measurement begins

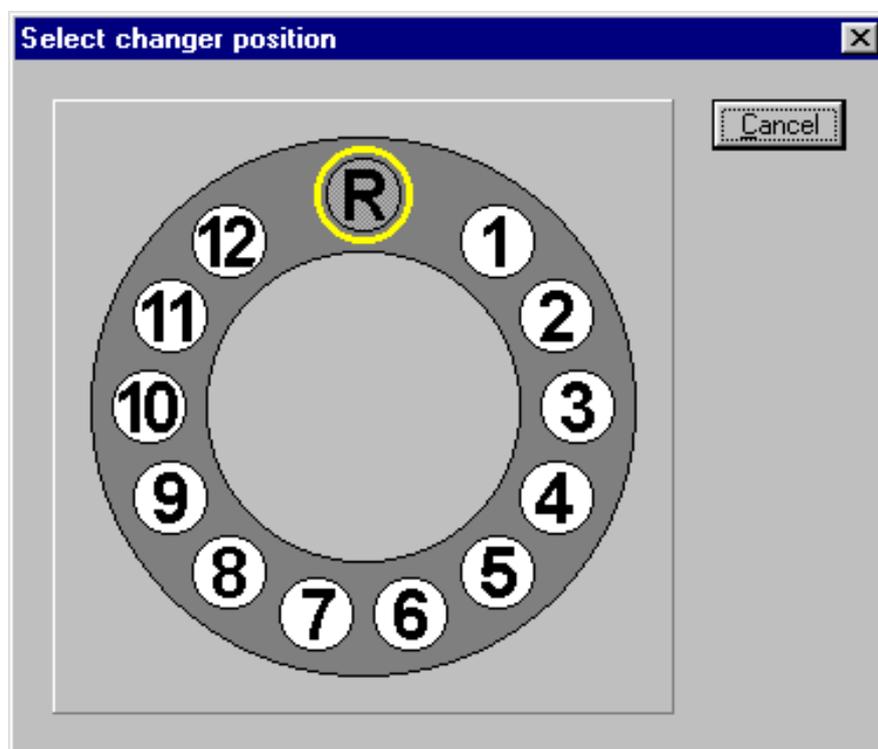


Figure 2: Changer position

- (g) Once the sample position is selected and measurement begins, the 'Measure sample' window reappears. If more samples are on the holder, repeat steps (e) and (f).
- (h) As each sample is measured, the results are automatically stored in the applications results database.
- (i) At least one appropriate certified standard reference material (SRM) shall be analysed and recorded on each day that unknown samples are analysed.
- (j) Once all measurements have been set, close the 'Measure sample' window.

6. Results Retrieval

- (a) Click the word 'Results' on the menu bar, then select 'Open results'. The dialog box 'open results' containing names of applications appears. Highlight the correct application and click 'OK'.
- (b) The 'Results' database window for the application then appears.

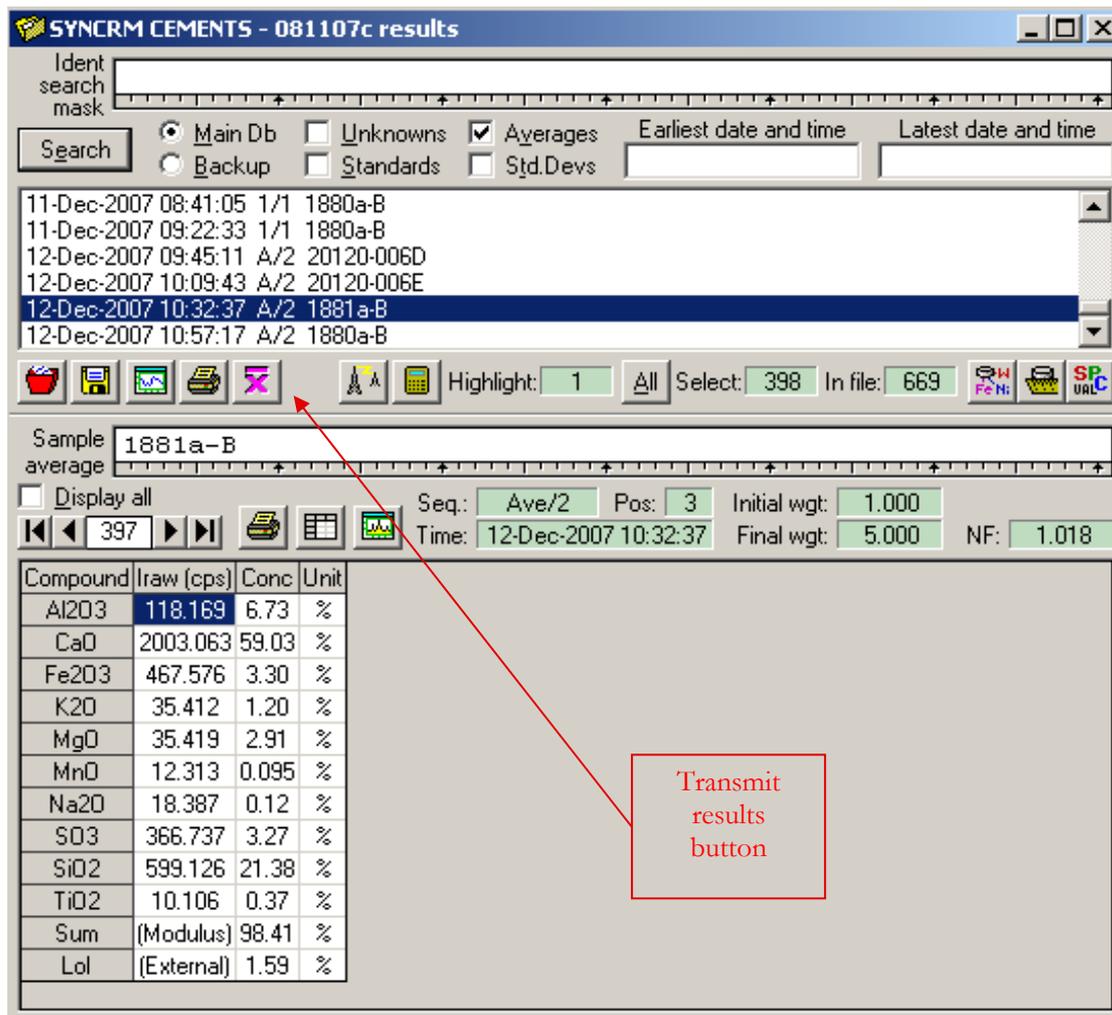


Figure 3: Application Results database window

- (c) Select the SRM that was analysed on the day of unknown sample analyses and compare the analysis with the certified values for that material. Where the results for the reference analysis differ from the certified value by more than the amount shown in Column 4 of Table A1 (Appendix A), the unknown analysis shall not be accepted.
- (d) Select the sample to be reported. Click the 'transmit results' button to export the result to file. Repeat this step for all samples to be reported
- (e) Switch to windows explorer, browse to 'D:\Data\Results\' folder, and retrieve the results file(s).

7. Reporting

Report the results of analysis to the following accuracy.

Result	Reporting Precision
Loss on Ignition (LoI)	0.1%
Al ₂ O ₃	0.1%
CaO	0.1%
Fe ₂ O ₃	0.1%
K ₂ O	0.01%
MgO	0.1%
MnO	0.01%
SO ₃	0.1%
SiO ₂	0.1%
TiO ₂	0.1%

Table 1: Precision of Reporting

8. Maintenance

The only maintenance performed by operators is cleaning the dust from the instruments and cleaning the sample area for possible pieces/specks of borate glass. Do not touch the x-ray source or detector at the far end of the sample area. A small suction pump is useful to pick up small specks.

9. Appendices

- (a) Appendix A: Calibration Qualification Procedure

Appendix A: Calibration Qualification Procedure

The chemical composition of at least four internationally accredited cement standard reference materials (SRM) shall be determined on duplicate portions using the chosen test method. The difference between duplicate values for each component and the deviation of the mean of the values from the certified value shall be compared with the figures given in Table A1. Where possible, the SRM shall be selected to cover the expected range of component percentages.

Where SRMs have been used for calibration purposes, different SRMs shall be used for qualification.

The chosen calibration validation shall be fully documented and proven as follows:

- (b) Using T1033, duplicate portions of the four SRMs shall be prepared for analysis.
- (c) Each portion shall be tested, with each duplicate portion tested on a different day.
- (d) The difference between the duplicate values for each constituent shall not exceed the maximum value given in Column 2 of Table A1 for at least three of the four SRMs and twice the maximum value for the fourth.
- (e) The arithmetic mean of duplicate determinations for each constituent shall not differ from the certified value by more than the maximum value shown in Column 3 of Table A1 for at least three of the four SRMs and twice the maximum value for the fourth.
- (f) For the purposes of qualification, all results shall be fully documented, dated and recorded to the nearest 0.01%

Result	Maximum difference between duplicates [Appendix A(c)]	Maximum difference of mean of duplicates from certified value [Appendix A(d)]	Maximum difference of a single determination from reference value [Section 6(c)]
SiO ₂	0.20	0.35	0.35
Al ₂ O ₃	0.20	0.20	0.20
Fe ₂ O ₃	0.10	0.10	0.10
CaO	0.20	0.30	0.30
MgO	0.20	0.20	0.20
SO ₃	0.10	0.15	0.15
Na ₂ O	0.03	0.05	0.05
K ₂ O	0.05	0.05	0.05

Table A1: Maximum Permissible Variations in Results