



Test method T1033

Preparation of borate glass specimens for
x-ray fluorescence analysis of cements
and blends

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. Method generally revised. Section 3,4&5 modified to include new sample preparation apparatus. Appendix A removed	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 T1033 (other than minor editorial changes) are indicated by a vertical line in the margin as shown here.

Test method T1033

Preparation of borate glass specimens for x-ray fluorescence analysis of cements and blends

1. Scope

This procedure describes the fusion of cements and cement blends with '12:22' flux containing lithium metaborate and lithium tetraborate into glass specimens (discs) for X-Ray Fluorescence (XRF) analysis of metal oxide content.

In this procedure, glass discs are prepared by pre-heating platinum casting dishes in a furnace, and then pouring the molten sample/flux mix into the dishes. The dishes are then removed from the furnace and allowed to cool.

2. Safety and Warning

- (a) The inside area of the muffle furnace heated to over 1000°C inflames any organic matter very quickly. Do not move closer to the furnace than is necessary to perform the required tasks. Always use tongs of the appropriate length to avoid exposure to excessive heat.
- (b) Always use UV protective goggles when opening the door of the heated muffle furnace. Full face mask must be worn when swirling molten samples

Caution: Frequent unprotected viewing of items in the open muffle furnace, which is at temperatures $\geq 1000^{\circ}\text{C}$, will cause extreme irritation of eyes.

- (c) When handling the molten glass in the crucible, always take care that no amount is spilled. It can cause deep burns in the skin.
- (d) Operators sensitive to cement dust and dust from cementitious substitutes, may wear the mask provided during handling of these materials.

3. Apparatus

- (a) A laboratory muffle furnace capable of maintaining temperatures in the range of 750°C – 1050°C.
- (b) A topside opening XRF sample preparation furnace, capable of holding platinum casting dishes and crucibles and maintaining fusions temperatures between 1000°C and 1200°C
- (c) Small hot plate for heating washing solution for crucibles.
- (d) Platinum-tipped tongs (300 cm) with metallic heat shield near the holding side.
- (e) Long tongs for placing crucibles into, and manipulating crucibles within, the XRF sample preparation furnace.
- (f) Long spatula for removing platinum casting dishes
- (g) Appropriate number of platinum (Pt95/Au5) crucibles for melting of specimens. (i.e one for each sample being prepared.)
- (h) Heat resistant material, capable of withstanding placement of a platinum crucible at approximately 1000°C.
- (i) UV goggles for operation with muffle furnace.
- (j) A balance accurate and readable to ± 0.0001 g.
- (k) Spatulas for weighing out samples and flux material.
- (l) Agate or tungsten carbide mortar and pestle.
- (m) One stopwatch.
- (n) A desiccator for storage of glass standards, standard chemicals and samples whilst undergoing cooling.

4. Chemicals and Consumables

- (a) X-Ray Flux, grade '12:22' for XRF fusion. (In further text: 'Flux'.)
(Eg. XRF Scientific P/L Type 12:22 Flux)

NOTE: Flux should be pre-treated by placing it in a furnace for a minimum of 2 hours at approximately 550°C in a suitable crucible. The flux should then be stored in a desiccator until use.

- (b) Ammonium Iodide tablets.
(c) Citric acid for cleaning of platinumware.
(d) Thin cotton gloves for handling finished specimens.

5. Procedure

Note: Read and understand Section 2 above before proceeding.

- (a) Preheat the muffle furnace to 950°C (750°C for Fly Ashes)
Note: Tolerance on temperature is $\pm 50^\circ\text{C}$
- (b) Preheat the XRF sample preparation furnace to 1050°C
Note: Tolerance on temperature is $\pm 50^\circ\text{C}$
- (c) Heat a known mass of the sample (3 - 4 g, weighed to an accuracy of 0.0001g) in an appropriately sized platinum crucible (6 g) at a temperature of 950°C (750°C for Fly Ashes) for 30 minutes in the muffle furnace. Cool in a desiccator and weigh to an accuracy of 0.0001g to determine loss on ignition as a percentage of the initial mass.
i.e. $\text{LoI}\% = ([\text{Initial Mass} - \text{Final Mass}] / \text{Initial Mass}) \times 100$
- (d) Grind the sample with mortar and pestle to break down the agglomerates.
- (e) Weigh out $2 \text{ g} \pm 0.0003 \text{ g}$ to an accuracy of $\pm 0.0001 \text{ g}$ in an appropriately sized platinum crucible (30 g).
- (f) Re-tare the analytical balance. Calculate the required mass of flux (4 times the mass of sample) produce a sample/flux mix of 1 part sample and 4 parts flux. Add this quantity of flux to the crucible containing the sample. The mass of the flux must be exactly 4 times the sample mass to the nearest $\pm 0.0003 \text{ g}$.
- (g) Place two platinum casting dishes into the sample preparation furnace for preheating.
- (h) Add one Ammonium Iodide tablet to each crucible, and mix the sample and flux together with a spatula. Ensure that no material is lost.
- (i) Place the mixed sample/flux crucibles into the sample preparation furnace. Start the stopwatch.
- (j) At the 5 minute mark, remove the crucibles one at a time and gently swirl them so that the molten glass becomes homogenous and the air bubbles break. Replace the crucibles into the furnace. Repeat the procedure after a further five minutes.
- (k) After another five minutes (total heating time 15 minutes) take the crucible and carefully pour its content immediately onto the platinum casting dish.
- (l) After a few moments, when the furnace temperature has returned to a value greater than 1000°C indicated, remove the casting dishes and place them onto the heat resistant material.
- (m) Transfer the platinum crucible from the heat resistant material into 10% solution of citric acid heated to just below boiling on the small hot plate. To facilitate faster cleaning, place a small magnetic stirrer bar in each crucible.
- (n) Allow glass discs to cool for approximately 10 minutes, then invert the dishes (with tongs) to release the samples.
- (o) Label each sample with its sample number, LoI% value and date by sticking a round sample label on each disc.

Note: The bottom surface of the specimens which had been in contact with the casting dish will be used for XRF determination.

- (p) Wearing a pair of cotton gloves transfer the specimens to a desiccator for temporary storage. The specimen is now ready for XRF analysis.
- (q) After the platinum crucibles have been cleaned in the citric acid, rinse the crucibles twice with distilled water. Allow to dry.
Note: Citric acid solution can be used several times.

6. Care of Platinum Equipment

6.1 Care of Platinum Crucibles:

- Do not allow platinum to come into contact with any other metal.
- Do not heat platinum with elementary sulfur, phosphorus, carbon, bromine and iodine, and any material developing carbon monoxide, like oxalic acid.
Note: The small amount of carbon developed when burning the organic materials, or small amount of carbon contained in fly ash is not dangerous in an oxidising atmosphere.
- Dry or dissolved alkali hydroxides and concentrated sulfuric acid dissolve the platinum slowly. Therefore do not use them for cleaning.
- Oxides, carbonates, borates and persulphates do not harm platinum in any condition, except pure lithium carbonate.

6.2 Cleaning Of Platinum Crucibles:

- Usually the soaking of platinum after every use in warm or boiling citric acid 10% solution will keep crucibles clean almost indefinitely. Over time platinum absorbs a lot of strontium trace element from the specimens. This can be removed by soaking them in molten sodium carbonate for 10–30 minutes and then burning on a strong mesh-Bunsen gas burner. Strontium will evaporate in several seconds with an intense purple flame. Do not mistake it with consequent weak purple coloration of absorbed lithium which can persist for a long time and is not considered necessary to remove. Most of impurities will be removed from platinum this way.
- Only place hot platinum on heat resistant material or similar dedicated refractories. Cold, clean platinum crucibles should be stored in boxes with suitably sized receptacles to protect the crucibles from mechanical damage.