

## CHEMICAL ANALYSIS OF CEMENT

1. SCOPE: This method is a modification of ASTM C-114 for the chemical testing of cement by x-ray spectroscopy.
2. APPARATUS –MATERIALS:
  - 2.1. Philips MagiX PRO Wavelength Dispersive x-ray fluorescence spectrometer.
  - 2.2. SuperQ software
  - 2.3. Philips Perl’X 3 fused bead machine
  - 2.4. Platinum dish and crucible set
  - 2.5. Lithium Bromide (LiBr): 10% solution non-wetting agent
  - 2.6. 100% Lithium Tetraborate ( $\text{Li}_2\text{B}_4\text{O}_7$ ) flux
3. PROCEDURE:
  - 3.1. Prepare porcelain crucibles by igniting at 950°C to a constant weight. Cool and store in a desiccator to avoid absorption of moisture.
  - 3.2. Weigh 1.0 gram of cement sample accurately to 0.0001 grams into a prepared porcelain crucible. Ignite sample to a constant weight in a muffle furnace at 950°C and cool in a desiccator.
  - 3.3. Calculate the loss on ignition (LOI) using the following formula:  
$$\text{LOI} = (A/B) \times 100$$
where: A = weight of sample after ignition;  
B = weight of original sample.
  - 3.4. Weigh accurately to 0.0001 grams 6.0 grams of flux directly into platinum crucible. Then weigh accurately to 0.0001 grams 0.6 grams of LOI free cement sample directly into the platinum crucible. Add 3 drops of LiBr solution. Place the platinum crucible and dish in the Perl’X 3 machine and select to run program 9\* for all types of cement. This takes approximately 15 minutes. Program run is dependent on sample type.
  - 3.5. Open the cement application and enter the sample identification and LOI information in the measure sample screen on the measure and analyze program.

- 3.6. Place sample in a 27mm steel cup. Then place in x-ray instrument and prepare to run cement application on the measure sample screen. Click on measure at the bottom of the screen. This may take a few minutes. The application chosen is dependent upon the sample type.
4. QUANTIFICATION: Program quantifies data by using a least squares program. Similar samples with known chemical make-ups are used as standards in the quantification technique. As many standards as possible are used for best quantification. The results are reported as oxides in weight percents.
5. REPORT:
  - 5.1. % LOI
  - 5.2. % Insolubles
  - 5.3. % SO<sub>3</sub>
  - 5.4. % Al<sub>2</sub>O<sub>3</sub>
  - 5.5. % Fe<sub>2</sub>O<sub>3</sub>
  - 5.6. % C<sub>3</sub>A (2.650 x %Al<sub>2</sub>O<sub>3</sub>) – (1.692 x %Fe<sub>2</sub>O<sub>3</sub>)
  - 5.7. % MgO
  - 5.8. % Na<sub>2</sub>O
  - 5.9. % K<sub>2</sub>O
  - 5.10. % CaO
  - 5.11. % SiO<sub>2</sub>
  - 5.12. % Total Alkali (Na<sub>2</sub>O + 0.658 x K<sub>2</sub>O)

\* NOTE: Program 9 includes the following: One oxidation for 2 minutes, temperature 1100°C, power of generator 77, agitation angle 25, and agitation speed 10. One fusion for 6 minutes, temperature 1100°C, power of generator 77, agitation angle 50 and agitation speed 15. Then there is a pause before casting for 10 seconds at a temperature of 1100°C. Casting lasts 2 minutes, temperature 1100°C, casting angle 123, casting speed 10 and time for solidification is 30 seconds. Lastly, there is natural cooling for 1 minute and forced air cooling for 3 minutes at a flow rate of 40. The setting of the dish height dial is 40/12 and this depends on the size of the platinum dish used.

APPROVED

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DIRECTOR  
DIVISION OF MATERIALS

DATE

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