Kentucky Method 64-254-083
Revised 1/6/0303/07/08
Supersedes 64-254-030
Dated 1/27/0001/06/03

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. Spex 8000 mixer/mill~~

   2.5~~6~~. Lithium Bromide (LiBr): 10% solution non-wetting agent

   2.6~~7~~. 100% Lithium Tetraborate ($Li_2B_4O_7$) 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:

   LOI = (A/B) x 100

   where: A = weight of sample after ignition;
   B = weight of original sample.

   ~~3.4. Place at least 1.0 gram of cement sample from LOI in the spex 8000 mixer/mill and grind until a fine powder is obtained. This takes approximately 30 seconds.~~

3.~~45~~. 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 ~~from the Spex 8000 mixer/mill~~ 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.~~6~~5. Open the cement application and e~~E~~nter ~~names of samples~~the sample identification ~~-~~and LOI information in the measure sample screen on the measure and ~~———~~analyze program.

3.~~7 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_3

5.4. % Al_2O_3

5.5. % Fe_2O_3

5.6. % C_3A (2.650 x %Al_2O_3) – (1.692 x %Fe_2O_3)

5.7. % MgO

5.8. % Na_2O

5.9. % K_2O

5.10. % CaO

5.11. % SiO_2

5.12. % Total Alkali (Na_2O + 0.658 x K_2O)

* 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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DATE 03/07/08

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~~DIVISION OF MATERIALS~~

~~DATE 1/6/03~~

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