Kentucky Method 64-253-08
Revised 03/13/08
Supersedes 64-253-03
Dated 01/07/03

CHEMICAL ANALYSIS OF FLY ASH AND MICROSILICA

1. SCOPE:

   1.1. This method is a modification of ASTM C-114, as referenced in ASTM C-311, for the chemical testing of fly ash by x-ray spectroscopy.

   1.2. This method is also used for the chemical testing of microsilica by x-ray spectroscopy.

2. APPARATUS:

   2.1. 30ml Porcelain crucible

   2.2. Muffle furnace: maintained at 750ºC

   2.3. Desiccator

   2.4. Balance: capable of accurately weighing to 0.0001g

   2.5. Drying oven: maintained at 110º C

   2.6. Philips Perl'x 3 fused bead machine

   2.7. Platinum dish and crucible set

   2.8. Lithium Bromide (LiBr): 10% solution non-wetting agent

   2.9. 67% Lithium Tetraborate (Li2B4O7) : 33% Lithium Metaborate (LiBO2) flux

   2.10. Lithium Metaborate (LiBO2) flux

   2.11. 27mm Steel sample cup and insert

   2.12. Philips MagiX PRO Wavelength Dispersive x-ray fluorescence spectrometer

   2.13. SuperQ software

3. PROCEDURE:

   3.1. Determine moisture content by weight percent.

      3.1.1. Prepare porcelain crucibles by igniting in a muffle furnace at 750°C to constant weight. Cool and store crucibles in a dessicator to avoid absorption of moisture

3.1.2 Weigh approximately 2 grams of sample into a prepared porcelain crucible. Record the sample weight to the nearest 0.0001g.

3.1.3. Dry the sample to a constant weight in an oven at 110°C.

3.1.4. Cool the sample to room temperature in a desiccator to avoid absorption of moisture.

3.1.5. Re-weigh the cooled sample. DO NOT DISCARD the sample. Record the dried sample weight to the nearest 0.0001g.

3.1.6. Calculate the moisture content of the sample in accordance with Section 4.1.

3.2. Determine loss on ignition (LOI) by weight percent.

3.2.1. Ignite sample retained from 3.1 to a constant weight in a muffle furnace at 750°C and cool in a dessicator.

3.2.2. Re-weigh the cooled sample. DO NOT DISCARD the sample. Record the ignited sample weight to the nearest 0.0001g.

3.2.3. Calculate the loss on ignition (LOI) in accordance with Section 4.2.

3.3. Prepare and analyze fly ash sample.

3.3.1. Weigh 6.0g to the nearest 0.0001g of 67% Li2B4O7:33% LiBO2 flux directly into platinum crucible.

3.3.2. Weigh 0.6g to the nearest 0.0001g of fly ash sample retained from 3.2 directly into the platinum crucible.

3.3.3. Add 3 drops of LiBr solution to the sample.

3.3.4. Place the platinum crucible and dish in the Perl'x 3 machine and select to run program 7 (Note 5.1).

3.3.5 Access SuperQ software and open the fly ash application.

3.3.6 Enter the sample identification and LOI in the measure sample screen of the measure and analyze program.

3.3.7 Place the prepared sample into a 27mm steel cup and load into the x-ray instrument.

3.3.8 Click measure at the bottom of the measure and analyze screen (Note 5.2).

3.4 Prepare and analyze microsilica sample.

3.4.1. Weigh 6.0g to the nearest 0.0001g of LiBO2 flux directly into platinum crucible.

3.4.2. Weigh 0.6g to the nearest 0.0001g of microsilica sample retained from 3.2 directly into the platinum crucible.

3.4.3. Add 3 drops of LiBr solution to the sample.

3.4.4. Place the platinum crucible and dish in the Perl'x 3 machine and select to run program 7 (Note 5.1).

3.4.5 Access SuperQ software and open the fly ash application.

3.4.6 Enter the sample identification and LOI in the measure sample screen of the measure and analyze program.

3.4.7 Place the prepared sample into a 27mm steel cup and load into the x-ray instrument.

3.4.8 Click measure at the bottom of the measure and analyze screen (Note 5.2).

4. CALCULATION:

4.1 MC = (A/B) x 100

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

4.2 LOI = (A/B) x 100

where: A = weight of sample after ignition.
B = weight of moisture free sample.

5. NOTES:

5.1 Program 7 is appropriate for all classes of fly ash samples and microsilica samples. Program 7 includes: One oxidation for 4 minutes, temperature 1100°C, generator power 77, agitation angle 30, and agitation speed 15. One fusion for 5 minutes, temperature 1100°C, generator power 77, agitation angle 60, and agitation speed 20. Then a pause before casting for 10 seconds at a temperature of 1100°C. Casting time 10 seconds, temperature 1100°C, casting angle 123, casting speed 10, and time for solidification 2 minutes. Lastly there is natural air cooling for 4 minutes and forced air cooling for 2 minutes at a flow rate of 40. The setting of the dish height dial is 12/40 and is dependent on the size of the platinum dish being used.

5.2 Program quantifies data by using a least squares program. Similar samples with known chemical analyses 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.

6. REPORT:

6.1. Report the following values for fly ash samples.

6.1.1. % Moisture Content

6.1.2. % LOI

6.1.3. % SO_3

6.1.4. % Al_2O_3

6.1.5. % Fe_2O_3

6.1.6. % R value: for class C fly ashes only. $[(CaO - 5)/ Fe_2O_3]$

6.1.7. % MgO

6.1.8. % Na_2O

6.1.9. % K_2O

6.1.10. % CaO

6.1.11. % SiO_2

6.2 Report the following values for microsilica samples.

6.2.1 % Moisture Content

6.2.2 % LOI

6.2.3 % SiO_2

APPROVED _____

DIRECTOR
DIVISION OF MATERIALS

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