

## 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 750°C
- 2.3. Desiccator
- 2.4. Balance: capable of accurately weighing to 0.0001g
- 2.5. Drying oven: maintained at 110° 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 (Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub>) : 33% Lithium Metaborate (LiBO<sub>2</sub>) flux
- 2.10. Lithium Metaborate (LiBO<sub>2</sub>) 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%  $\text{Li}_2\text{B}_4\text{O}_7$ :33%  $\text{LiBO}_2$  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  $\text{LiBO}_2$  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  $MC = (A/B) \times 100$

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

4.2  $LOI = (A/B) \times 100$

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<sub>3</sub>

6.1.4. % Al<sub>2</sub>O<sub>3</sub>

6.1.5. % Fe<sub>2</sub>O<sub>3</sub>

6.1.6. % R value: for class C fly ashes only. [(CaO – 5)/ Fe<sub>2</sub>O<sub>3</sub>]

6.1.7. % MgO

6.1.8. % Na<sub>2</sub>O

6.1.9. % K<sub>2</sub>O

6.1.10. % CaO

6.1.11. % SiO<sub>2</sub>

6.2 Report the following values for microsilica samples.

6.2.1 % Moisture Content

6.2.2 % LOI

6.2.3 % SiO<sub>2</sub>

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~~1. SCOPE: 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.~~

~~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~~

~~2.6. Lithium Bromide (LiBr): 10% solution non-wetting agent~~

~~2.7. 67% Lithium Tetraborate (Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub>) : 33% Lithium Metaborate (LiBO<sub>2</sub>) flux~~

### ~~3. PROCEDURE:~~

~~3.1. Weigh approximately 2 grams of fly ash sample into a small beaker.~~

~~3.2. Dry the sample to a constant weight in an oven at 110°C.~~

~~3.3. Calculate the moisture content using the following formula:~~

$$\text{MC} = (A/B) \times 100$$

~~where: A = weight of sample after drying;~~

~~B = weight of original sample.~~

~~3.4. Prepare porcelain crucibles by igniting at 750°C to a constant weight. Cool and store crucibles in a dessicator to avoid absorption of moisture~~

~~3.5. Weigh 1.0 gram of fly ash sample from the moisture analysis accurately to 0.0001 grams into a prepared porcelain crucible. Ignite sample to a constant weight in a muffle furnace at 750°C and cool in a dessicator.~~

~~3.6. 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 moisture free sample.~~

~~3.7. Place at least 1 gram of fly ash sample from LOI in the spex 8000 mixer/mill and grind until a fine powder is obtained and/or sample is completely uniform. This takes approximately 30 seconds.~~

~~3.8. Weigh accurately to 0.0001 grams 6.0 grams of 67:33 flux directly into platinum crucible. Then weigh accurately to 0.0001 grams 0.6 grams of fly ash 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's 3 machine and select to run program 7\* for all types of fly ash. This takes approximately 15 minutes. Program run is dependent on sample type.~~

~~3.9. Enter names of samples and LOI information in measure sample screen on the measure and analyze program.~~

~~3.10. Place sample in a 27mm steel cup. Then place in x ray instrument and prepare to run fly ash 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 makeups 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. % Moisture Content~~

~~5.2. % LOI~~

~~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. % R value: for class C fly ashes only. [(CaO - 5)/ 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>~~

~~\* NOTE: Program 7 for fly ash 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 this depends on the size of the platinum dish being used.~~

APPROVED

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DIRECTOR  
DIVISION OF MATERIALS

DATE 03/13/08

APPROVED

\_\_\_\_\_  
Director

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DIVISION OF MATERIALS

DATE 1/7/03

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