

Kentucky Method 64-224-083

Revised ~~1/6/03~~ 03/13/08

Supersedes KM 64-224-030

Dated ~~10/09/00~~ 01/16/03

CHEMICAL ANALYSIS OF LIMESTONE, FINE AGGREGATE, QUICKLIME AND HYDRATED LIME

1. SCOPE: This test method covers aggregate intended for use in various highway construction projects. Chemical analysis of aggregates are required only when specified on plans, proposals, bidding invitations, or by special provisions covering any particular material or projects. This method is a modification of ASTM C-25 for the chemical testing of limestone, fine aggregate, quicklime and hydrated lime by x-ray spectroscopy.
2. APPARATUS AND MATERIALS:
 - 2.1. Wet Chemical: All wet chemical analysis will be performed in accordance with ASTM C-25 current edition.
 - 2.1.1. 30ml Porcelain crucible
 - 2.1.2. Balance: capable of accurately weighing to 0.0001g
 - 2.1.3. Oven: maintained at 110°C
 - 2.1.3. Muffle furnace: maintained at 950°C
 - 2.1.4. Dessicator
 - 2.2. X-Ray Fluorescence:
 - 2.2.1. Philips MagiX PRO Wavelength Dispersive X-Ray Fluorescence Spectrometer
 - 2.2.2. SuperQ software
 - 2.2.3. Philips Perl'x 3 fused bead machine
 - 2.2.4. Platinum dish and crucible set
 - ~~2.2.5. Spex 8000 Mixer/Mill~~
 - 2.2.56. Lithium Bromide (LiBr) – 10% solution non-wetting agent
 - 2.2.67. ~~100~~67% Lithium Tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$):33% Lithium Metaborate (LiBO_2) -flux
 - 2.2.7. Lithium Tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$) flux

2.2.8. 27mm Steel sample cup and insert

3. PROCEDURE:

3.1. Prepare moisture free sample.

3.1.1. Prepare porcelain crucibles by igniting in a muffle furnace at 950°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.

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. DO NOT DISCARD the sample.

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

3.2.1. Weigh approximately 1 gram of original sample into a prepared porcelain crucible. Record the sample weight to the nearest 0.0001g.

3.2.2. Ignite sample to a constant weight in a muffle furnace at 950°C and cool in a dessicator.

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

3.2.4. Calculate the loss on ignition (LOI) in accordance with Section 4.1.

3.3. Prepare and analyze aggregate sample (limestone or fine aggregate).

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 aggregate sample retained from 3.1 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's 3 machine and select to run program 9 (Note 5.1).

3.3.5 Access SuperQ software and open the ledgrock 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 lime sample (hydrated lime or quicklime).

3.4.1. Weigh 6.0g to the nearest 0.0001g of $\text{Li}_2\text{B}_4\text{O}_7$ flux directly into platinum crucible.

3.4.2. Weigh 0.6g to the nearest 0.0001g of lime 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's 3 machine and select to run program 9 (Note 5.1).

3.4.5 Access SuperQ software and open the lime 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 $\text{LOI} = (\text{A/B}) \times 100$

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

5. NOTES:

5.1 Program 9 includes: One oxidation for 2 minutes, temperature 1100°C, generator power 77, agitation angle 25, and agitation speed 10. One fusion for 6 minutes, temperature 1100°C, generator power 77, agitation angle 50, and agitation speed 15. Then a pause before casting for 10 seconds at a temperature of 1100°C. Casting time 2 minutes, temperature 1100°C, casting angle 123, casting speed 10, and time for solidification 30 seconds. Lastly there is natural air 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 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.

~~3. SAMPLE: A maximum 0.95 liter (quart) sample is provided by the section requesting the analysis.~~

~~4. PROCEDURE:~~

~~4.1. Prepare porcelain crucibles by igniting at 950°C to constant weight. Cool and store crucibles in dessicator to avoid absorption of moisture.~~

~~4.2. Weigh 1.0 gram of 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.~~

4.3. Calculate the loss on ignition(LOI) using the following formula:

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

where:

A = weight of sample after ignition;

B = weight of original sample

4.4. Place at least 1.0 gram of the original sample in the spex 8000 mixer/mill and grind until fine powder is obtained and/or sample is completely uniform. This takes approximately 30 seconds.

4.5. 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 original sample from spex 8000 mixer/mill directly into the platinum crucible. Add 3 drops of LiBr solution. Place the platinum crucible and dish in the Perl'x3 machine and select to run program 9* for all types of limestone, aggregate and lime. This takes approximately 15 minutes. Program run is dependent on sample type.

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

4.7. Place sample in a 27mm steel cup. Then place in x ray instrument and prepare to run lime or ledgerock 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.

5.0 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.

6. REPORT:

6.1. Report the following values for aggregate samples.% Insolubles: for all fine aggregate materials

6.12.1. % Silica: upon request only

6.31.2. % Combined Oxides for all except "silica" sand. ($Al_2O_3 + TiO_2 + MnO + Fe_2O_3$)

6.41.3. % $CaCO_3$: for all except "silica" sand

6.51.4. % $MgCO_3$: for all except "silica" sand

6.61.5. % CaO: for slags only

6.17.6. % MgO: for slags only

6.1.7. SiO₂

6.1.8. % Ca

6.1.9. % Mg

6.1.10. % LOI

6.2. Report the following values for lime samples.

6.2.1. % CaO

6.2.2. % MgO

~~*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

DIRECTOR
DIVISION OF MATERIALS

DATE 03/13/08

APPROVED _____

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