Kentucky Method 64-222-08
Revised 05/09/08
Supersedes KM 64-222-02
Dated 12/26/02

CALCIUM CHLORIDE

1. SCOPE: This test method covers calcium chloride to be used for road conditioning purposes. Unless otherwise specified, calcium chloride may be furnished in either of two types.

   Type 1 Regular flake calcium chloride.
   Type 2 Concentrated flake, pellet, or other granular forms.

2. APPARATUS AND MATERIALS:

   2.1. Sieve sizes 3/8", No. 4 and No. 30.

   2.2. Hydrochloric Acid.

   2.3. Ammonium Hydroxide.

   2.4. Ammonium Chloride (20 gm/liter).

   2.5. Ammonium Oxalate (saturated sol.).

   2.6. Methyl Red Indicator.

3. SAMPLE: Sample in accordance with the Cabinet's Sampling Manual on Calcium and Sodium Chloride. Calcium chloride samples are received in quart metal containers. Care must be taken to keep the container covered at all times, except when portions are being removed for tests.

4. PROCEDURES:

   4.1. The sieves used in this determination are the 3/8", No. 4, No. 30 and Pan stacked in that order. Weigh 100 grams to 0.1 gm as rapidly as accuracy will permit, and pour onto the 3/8" sieve. Shake the entire stack and pan for one minute using a circular motion with frequent tapping. Weigh the fraction to 0.1 gm. Starting with the pan, and record as percent passing the No. 30 sieve. To this amount, add the fraction retained on the No. 30 sieve as the percent passing the No. 4 sieve. Repeat procedure until each fraction is weighed and recorded. Any moisture that may have formed on the sieves should be removed so that the sieve will be dry and clean for the next determination.

   4.2. Chemical Analysis:

      4.2.1. Total Calcium:

         4.2.1.1. Weigh accurately in a weighing bottle a sample of approximately 5 gm., and dissolve in distilled water. Add a drop or two of HCl to clear up solution, and dilute to 500 ml in a volumetric flask. Mix

thoroughly. Pipette a 25 ml. Aliquot, and transfer to a 400 ml beaker. Add 3 drops of methyl red, and make slightly alkaline by adding NH_4OH (1:1). Add 20 ml of saturated NH_4CL solution. Dilute to a volume of 100 to 150 ml. Heat to boiling, and carefully add 40 ml of hot $(NH_4)_2C_2O_4$ solution while stirring. Continue boiling for 3 to 5 minutes with occasional stirring to prevent bumping.

4.2.1.2. Remove from hot plate, and let settle for at least 30 minutes. Filter on No. 42 filter paper, taking care not to fill funnel more than ¾ full at any time. Wash precipitate with cold water. From this point, calcium may be determined either gravimetrically on volumetrically.

4.2.2. Gravimetric: Place paper and precipitate in a pre-weighed porcelain crucible, and char on hot plate. Ignite sample in a muffle furnace at 950 ^oC for at least one hour. Cool in dessicator, and re-weigh.

4.2.3. Volumetric: Return precipitate and filter paper to original beaker. Add 25 ml of H_2SO_4 (1:4) and macerate the filter paper with a glass rod. Dilute to 100-150 ml, and heat to about 80^o C. While still hot, titrate to a faint pink with 0.1 N $KmnO_4$.

4.2.4. Calculations:

Gravimetric = Wt. of Ash x 791.68 = $\%CaCl_2$

Volumetric:

$$\frac{mL\ of\ 0.1\ N\ KMnO_4\ x\ 11.1}{Wt.\ of\ Original\ Sample} = \%CaCl_2$$

5. CALCULATIONS: Calculations as indicated in Procedures.

6. PRECAUTIONS: Handle Calcium Oxalate precipitate in the same manner as in Agricultural Limestone Procedure.

7. REPORT: % Calcium Chloride.

APPROVED _____

DIRECTOR
DIVISION OF MATERIALS

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