

## 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 ~~9.51 mm (3/8")~~, ~~4750 µm~~ and ~~600 µm~~ (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: ~~Sampling procedure is the same as that listed in accordance with the Cabinet's Sampling Manual on Calcium for~~ and Sodium Chloride. Calcium chloride samples are received in ~~0.95 liter (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 ~~9.51 mm (3/8")~~, ~~4750 µm~~ (No. 4), ~~600 µm~~ (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 ~~9.52 mm (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 ~~600 µm~~ (No. 30) sieve. To this amount, add the fraction retained on the ~~600 µm~~ (No. 30) sieve as the percent passing the ~~4750 µm~~ (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<sub>4</sub>OH (1:1). Add 20 ml of saturated NH<sub>4</sub>CL solution. Dilute to a volume of 100 to 150 ml. Heat to boiling, and carefully add 40 ml of hot (NH<sub>4</sub>)<sub>2</sub>C<sub>2</sub>O<sub>4</sub> 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 or volumetrically.

4.2.2. Gravimetric: Place paper and precipitate in a pre-weighed porcelain crucible, and char on hot plate. ~~Burn in a bright red~~ Ignite sample in a muffle furnace at 950 °C 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<sub>2</sub>SO<sub>4</sub> (1:4) and macerate the filter paper with a glass rod. Dilute to 100-150 ml, and heat to about 80° C. While still hot, titrate to a faint pink with 0.1 N KmnO<sub>4</sub>.

4.2.4. Calculations:

Gravimetric = Wt. of Ash x 791.68 = %CaCl<sub>2</sub>

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

DATE

05/09/08

~~APPROVED~~ \_\_\_\_\_

~~Director~~

~~DIVISION OF MATERIALS~~

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