# A. Scope

For a complete list of GDTs, see the Table of Contents.

This method covers the chemical analysis of limerock and agricultural lime.

### **B.** Apparatus

- 1. Beaker 5 oz (150 ml)
- 2. Beaker 13.5 oz (400 ml) (BIT-01400)
- 3. Cylinder .85 oz (25 ml) graduated cylinder
- 4. Funnel 2.6 in (65 mm)
- 5. Hot Plate
- 6. Muffle Furnace 2400 °F (1375 °C)
- 7. Analytical Balance
- 8. Platinum Crucible 34 oz (10 ml)

# C. Sample Size and Preparation

Mix sample thoroughly. Take approximately 89 oz (25 g) portion of the thoroughly mixed sample and grind with mechanical grinder or mortar and pestle until all material passes No. 20 sieve. Store ground sample in dry container.

#### D. Procedures

- 1. Accurately weigh .032 oz (1g) of prepared sample.
- 2. Transfer weighed sampled to a 5 oz (150 ml) beaker and add .004 pts (2 ml) of 1-1 hydrochloric acid.
- 3. Digest on hot plate (short of boiling) for 15 minutes.
- 4. Remove from hot plate and dilute with .052 pts (25 ml) of hot water.
- 5. Filter through 1 No. 40 or No. 2 filter paper into .08 pts (400 ml) beaker. Wash residue on filter paper with hot water 6 to 8 times.
- 6. Transfer residue and paper to accurately weighed platinum crucible and ignite slowly in muffle furnace until paper is charred. Raised temperature to 2000 to 2200 °F (1100 ° to 1200 °C) and continue igniting at this temperature for approximately 30 minutes. Remove from furnace, cool in desiccator, weigh, and calculate percent residue.
- 7. Adjust volume of filtrate in 0.08 pts (400 ml) beaker to approximately 0.04 pts (200 ml). Place on hot plate, heat to boiling, add 2 to 3 drops of methyl red indictor, add 1-1 ammonium hydroxide, while stirring, until red color disappears. Add 1 drop excess of ammonium hydroxide, digest 1 to 2 minutes.
- 8. Remove from hot plate, let settle 5 minutes, and filter through No. 2 (11 cm) or No. 40 filter paper. Wash the precipitated iron and aluminum on filter paper 3 or 4 times with 2% hot ammonium chloride.
- 9. Transfer filter paper with the iron and aluminum precipitate to an accurately weighed platinum crucible.
- 10. Ignite slowly in muffle furnace until paper is charred. Raise temperature to approximately 1800 °F (982 °C) and ignite at this temperature for about 30 minutes. Remove the furnace, cool, weigh, and calculate percentage iron and aluminum oxide.

### E. Calculations

- 1. Percent Residue = Weight Residue x 100 Weight of Sample
- 2. Percent Iron and Aluminum Oxide = Weight of Aluminum and Iron Oxide x 100 Weight of Sample
- 3. Percent CaC03 and MgCaC03 = 100 -(Percent Residue + Percent Iron and Aluminum Oxide)

#### F. Report

Report as total carbonates.