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Lime

1. Application

This procedure covers the analysis of aglime and other liming material, and the calculation of the neutralizing index.

2. Summary of Methods

A sample of limestone is reacted with an excess of HCl. After the first reaction is complete, the H^+ remaining is titrated with NaOH. The $CaCO_3$ is calculated as the amount of acid neutralized by the sample compared with the amount neutralized by an equal weight of pure $CaCO_3$.

A dried sample of limestone is passed through a nest of 8, 20, 60, and 100-mesh sieves. The amount retained on each sieve is used to calculate the neutralizing index (NI) of the material. In Wisconsin, aglime is sold and applied on the basis of its neutralizing index. Fine lime reacts more quickly than coarse lime, so less is required to affect a given increase in pH in a three-year period. Fine lime is more costly to produce, however, and does not have as much residual activity as coarse lime.

3. Safety

Each chemical compound should be treated as a potential health hazard. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material handling data sheets should be made available to all personnel involved in the chemical analysis.

4. Interferences

There are few interferences in this procedure. Errors may arise if insufficient time is allowed for reaction of the sample in HCl or if the sample is not shaken adequately in the sieve analysis.

5. Apparatus and Materials

5.1 Erlenmeyer flasks (500 ml)
5.2 Burette (50 ml)
5.3 Shaker (Ro-Tap® Model # CL 340)
5.4 Sieves (ASTM, 8, 20, 60, 100-mesh)
5.5 Fischer filter paper, P-8 coarse, funnel (for filtering opaque samples)

6. Reagents

6.1 Standard NaOH (1.00 N): Fisher #SS266-20
6.2 1% phenolphthalein indicator solution (dissolve 1 g of phenolphthalein in 100 ml of 95% ethanol)
6.3 Standard HCl (1.00 N) Fisher #SA48-20

7. Methods

$CaCO_3$ Equivalent:

7.1 Grind lime sample to pass a 60-mesh sieve.
7.2 Weigh 1.000 g of the ground sample, and transfer to a 500 ml Erlenmeyer flask.
7.3 Add, by means of pipette, 25 ml of standard HCl solution.
7.4 Place the 500 ml flask on a hot plate, and heat just below boiling for 30 minutes ± 10 min. Do not boil. Remove from hot plate, cool to room temperature, dilute to approximately 150 ml with distilled water. Rinse condensate from sides of flask while diluting. Filter sample if opaque.
7.5 Add two drops of phenolphthalein indicator.
7.6 Titrate to a pink color with standard NaOH. Record volume to 0.1 ml. Be sure buret is at zero to begin.

Ca/Mg Analysis:

7.7 Pour titration into a 500 ml cylinder and dilute to 500 ml with deionized water.
7.8 Stir (vortex) diluted sample
7.9 Place 8 ml of diluted sample into a plastic test tube and analyze by inductively coupled plasma emission spectroscopy (ICP-OES).

Particle Size Distribution:

7.10 Weigh a separate 100 g sample of dried, but not ground aglime.
7.11 Transfer to the top of the nest of sieves (8, 20, 60, and 100-mesh)
7.12 Place the nest of sieves on the Ro-Tap shaker, and shake for 10 minutes.
7.13 Weigh the sample retained on each sieve and contained in the bottom pan of the 100, 60, and 20-mesh sieves.

8. Calculations

8.1 Calculate the $CaCO_3$ equivalent of the sample:

$$CaCO_3 = \frac{\text{mEq acid neutralized by 1.0 g of liming material}}{\text{mEq acid neutralized by1.0 g pure } CaCO_3} \times 100$$

$$= \frac{(\text{ml HCl} \times \text{N HCl}) - (\text{ml NaOH} \times \text{N NaOH})}{\text{sample weight g mEq}} \times 100$$

8.2 Calculate the neutralizing index (NI) of the liming material:

NI = [0.2(% on 20-mesh sieve) + 0.6(% on 60-mesh sieve) + 1.0(%< 60-mesh sieve)] x CaCO3 equivalent.

9. Quality Control

9.1 Standard lime sample – A 1.00 g sample of pure $CaCO_3$ is analyzed with each batch of samples to check procedural accuracy. This standard should require 5.0 ± 0.1 ml of standard NaOH to titrate to the end point.

9.2 Calcium Carbonate ($CaCO_3$) Fisher # 664-500

10. Reporting

The percent of the sample retained on each sieve and the percent finer than 60-mesh is reported, along with the $CaCO_3$ equivalent of the liming material. The neutralizing index (NI) is calculated and reported.

11. References

11.1 *Calcium Carbonate Equivalent*: See a standard quantitative analysis text for preparation of standard NaOH and HCl; e.g. Blaedel, W. J., and V. M. Meloche. 1963. Elementary Quantitative Analysis. Harper and Row, New York. Pages 355-370.

11.2 *Particle Size Distribution*: Adapted from H. L. Motto and S. W. Melsted. The Efficiency of Various Particle-Size Fractions of Limestone, 1960, Soil Sci. Soc. Am. Proc. 24: 488-490.