KAR 4-5-4

Proposed

Kansas State University Soil Testing Lab Agricultural Liming Material Testing Procedure

By Kansas State University

October 15, 2015

Moisture Content. The moisture content of ag lime materials varies widely as sampled and as delivered to customers. Current lime materials sold in Kansas vary in moisture content from a low of 1-5% moisture for "Dry Lime Products", to a high of 48 to 54% water/moisture for "Liquid Lime Products". To provide uniformity in reporting, and facilitate Kansas citizens determining the true neutralizing value of the product they are purchasing, all Ag Lime product analysis is reported on a dry basis. This requires the determination of moisture content on each sample.

1. Lime Moisture test Procedure:

    a. Label the tall, moisture sample specimen cup with Ag Lime sample ID. Weigh empty specimen cup and record weight (approx. 15 grams).

    b. Weigh 100 grams of the as received lime sample into the specimen cup. Record the weight of the sample plus the specimen cup (approx. 115 grams).

    c. Place the specimen cup with the moisture sub-sample of lime into the soil oven overnight or until the sample is completely dry. Drying is done at an oven temperature of 60° C

    d. When the moisture sub-sample is dry, weigh and record the weight of the dry sample and the specimen cup. Input weights into the lime calculation template to find the moisture percent. Calculation is made as follows:

Sample Wet = (Specimen Cup weight + Sample Wet weight) – Specimen Cup weight

Sample Dry = (Specimen Cup weight + Dry Sample weight) – Specimen Cup weight

Moisture % = (Sample Wet weight – Sample Dry weight) / Sample Wet *100

Sample Fineness. Lime particle size influences the surface area of the particle available for reaction with soil acidity, and the rate at which lime will react. Coarse particles, those > than 8 mesh in size, react very slowly and will have little effect on soil pH, while fine particles, those < 60 mesh, will react very quickly, and will react completely with soil acidity. Thus finely ground limestone will react faster, and have a greater neutralizing value than coarse ground ag lime.

In Kansas, two screen sizes, an 8 mesh sieve and a 60 mesh sieve, are used to determine three ranges of particle sizes: Those coarse particles which remain on the 8 mesh sieve with a size > 8 mesh which will not be effective at raising soil pH; those intermediate sized particles which remain on the 60 mesh sieve and have a range of particle sizes < 8 mesh but > 60 mesh and have impact on pH over a 1-3 year time frame; and those very fine particles which pass through the 60 mesh sieve with a size < 60 mesh and react very quickly to raise soil pH.

Previous protocols called for determining lime fineness from a dried sample. However, the drying process causes cementing of small particles resulting in artificially low fineness scores, and increased variability in results with some samples. To avoid this problem samples are run as as received moist samples, similar to the condition they would be applied in the field.

2. Lime Fineness test:

    a. Label the short, empty fineness specimen cup with Ag Lime sample ID. Weigh empty specimen cup and record weight (approx. 10 grams).

    b. Weigh 100 grams of the as received lime sample into the labeled specimen cup. Record the weight of the subsample plus the specimen cup (Approximately 110 grams).

    c. Pour the weighed as-received fineness sub-sample over a No. 8 and No. 60 USA Standard Testing Sieve, stacked

together with the No. 8 sieve above the No. 60. Shake vigorously until no material falls through the sieves.

d. Gently wash the lime caught on the sieves with distilled water until mostly clear water is running through the sieves.

e. Take the No. 8 sieve and wash the lime caught on top (> 8 mesh fraction) into a metal pan. Rinse until the water runs clear.

f. Take the No. 60 sieve and wash the lime caught on top (< 8 mesh but > 60 mesh intermediate sized fraction) into a metal pan. Rinse until the water runs clear.

g. Place both the pans into a 60° C oven for 2 hours, or until the sample is completely dry.

h. After 2 hours, weigh the No. 8 pan with the sample. Record the weight. Empty the pan and record the empty pan weight. Weigh the No. 60 pan with the sample. Empty the pan and record the pan weight. Input weights into the lime calculation template to find the fineness factor.

> 8-mesh sieve sample weight (g) = (Pan weight+ Sample dried weight) – Pan weight

Sample % on 8-mesh = Dry Sample weight from 8 mesh seive / Total dry sample weight (weight adjusted for moisture) * 100

< 8 mesh but > 60-mesh sample weight (g) = (Pan weight+ Sample dry weight) – Pan weight

Sample % on 60-mesh = Sample weight from 60 mesh seive / Total dry sample weight (weight adjusted for moisture) * 100

Sample through 60-mesh (< 60 mesh particle size) = Total dry sample weight – ((> 8-mesh sample weight) + (< 8 mesh but > 60-mesh sample weight))

Sample % through 60-mesh = Sample weight through 60-mesh / Total dry sample weight (weight adjusted for moisture) * 100

Fineness factor = (Sample % > 8-mesh * 0) + (Sample % < 8 mesh but > 60 mesh * 0.5) + (Sample % through 60-mesh * 1)

Calcium Carbonate Equivalent. The calcium carbonate equivalence of a limestone sample compares the acid neutralizing power of an ag lime to pure reagent grade calcium carbonate. Results are given on a percent equivalence to pure calcium carbonate. Ag limes commonly found in Kansas will vary from 60 to 105% (the low values are found in samples with a high degree of non-lime contamination such as clay or shale, while the high values generally contain dolomite, $CaMg(CO_3)_2$). The average CCE of Kansas lime is around 90 to 95%.

3. For Calcium Carbonate Equivalent:

   a. Weigh 1 gram of Calcium Carbonate (standard reagent grade) and place in plastic vial.

   b. From the dried lime sample obtained from moisture determination, scoop out approximately 5 grams of lime. Mortar and pestle until the lime sample is finely ground. Weigh 1 gram of finely ground lime sample into a plastic vial.

   c. Transfer 8 mL of Hydrochloric acid into a glass French bottle. Swirl bottle so that the HCl coats each side of the French bottle.

   d. Carefully place plastic vial of CaCO3 into French bottle, so as not to spill any.

   e. Make sure water level in gasometric apparatus is at 50 mL. Adjust water level as necessary. Attach the French bottle containing the $CaCO_3$ to the gasometric apparatus. Make sure the seal is secure and no air leaks are present. Record the mLs of water displaced.

   f. Tip French bottle so that HCl and $CaCO_3$ mix. Gently shake until the $CaCO_3$ has completely reacted with the HCl. Record the mLs of water displaced.

g. Detach the French bottle from the gasometric apparatus. Allow the water level in the graduated cylinder on the gasometric apparatus to fall back to 50 mL, allowing the instrument to reach equilibrium.

h. Repeat steps C-G with the lime sample.

i. Input displacement values into the lime calculation template to find the calcium carbonate equivalent.

Sample Displacement = Final displacement – initial displacement

Standard $CaCO_3$ Displacement = Final displacement – initial displacement

CCE = Sample Displacement / CaCO3 Displacement

Percent Effective Calcium Carbonate. The percent Effective Calcium Carbonate, (%ECC), of lime is a value calculated to allow quick comparison of the neutralizing power of ag lime products. The %ECC calculation uses the Calcium Carbonate Equivalent, CCE, an estimate of chemical purity, and the Fineness Factor, particle size effects on lime value, in the following calculation:

Percent ECC = (Fineness Factor / 100) * (CCE / 100) *100

Percent ECC values are commonly referred to as the "percent lime". Common ranges of percent ECC values range from 40 to 60%.

Effective Calcium Carbonate Equivalent. The Effective Calcium Carbonate Equivalent, ECCE, is another way to compare liming materials and to calculate lime application rates for a specific product. ECCE is calculated as follows:

ECCE in pounds ECC/ton = (%ECC) * (2000 lbs/ton)

This is a particularly valuable calculation as lime recommendations are given by most soil testing labs in the Plains region in pounds ECC per acre.