Test Procedure for

SAMPLING AND TESTING LIME

TxDOT Designation: Tex-600-J

Effective Date: February 2006

1. SCOPE

1.1 This method, divided into five parts, describes the sampling and testing of the following materials:

- hydrated lime,
- quicklime,
- commercial lime slurry, and
- carbide lime slurry.

1.2 The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.

PART I—SAMPLING LIME PRODUCTS

2. SCOPE

2.1 This part covers the sampling of lime in powdered form as:

- bulk hydrated lime discharged from tank trucks,
- bagged hydrated lime, as bagged hydrate from bag trucks being loaded, or from bagged shipments after delivery to warehouse or jobsite,
- quicklime in crushed or pebble form, discharged from tank trucks,
- commercial lime slurry, a mixture of hydrated lime solids in water, from a sampling port at the plant site or in the distributor truck, and
- carbide lime slurry, a mixture of hydrated carbide lime solids in water, from a distributor truck.

3. APPARATUS

3.1 *Bulk Hydrated Lime:*

3.1.1 *Paint brush,* 2 in. wide.

3.1.2 *Bucket,* 1 gal., with double friction type lid and bail.

3.1.3 *Top hatch sampling device,* consisting of a 9-ft. length of 1.5-in. IPS PVC 1120 plastic pipe of SDR 1.10 MPa (26,160 psi), meeting ASTM D 2241:

3.1.3.1 Fit the pipe at one end with a rubber stopper drilled with a 0.25-in. diameter hole.

3.1.3.2 Cement the stopper in place using a standard adhesive epoxy.

3.1.3.3 Spot-weld a hook to a 3 × 2-in. half-round plate of 12-gauge steel. Rivet and cement the plate with epoxy to the rubber stopper end of the pipe.

3.1.3.4 The 3-in. long metal hook has a 0.5 × 0.75 in. doubled over section, with a 0.25-in. slot.

3.1.3.5 The opening of the hook should face away from the rubber stopper.

3.1.3.6 The hook will catch the bucket bail, so the sampler can lower the bucket and pipe to the ground. This allows the sampler to alight from the truck safely.

3.1.4 *Bottom sampling tube,* consisting of two concentric plastic pipes:

3.1.4.1 The outer pipe is 126 in. long, 1.5 in. inside diameter, IPS PVC plastic pipe, fitted at one end with a tip made from a 7.5-in. length of solid aluminum round stock, 1-5/8 in. in diameter, tapered to a point along 6.5 in. of its length, inserted 1 in. into the tube, and fastened with two screws through the wall of the pipe into tapped holes on either side of the tip.

3.1.4.1.1 Round the point of the tip to a 0.5-in. diameter point for safety.

3.1.4.1.2 Use an adhesive epoxy to mold an epoxy tip in a metal, foil, or cardboard mold.

3.1.4.2 The inner pipe is a 1.25-in. IPS PVC plastic pipe, measuring 10 ft. long.

3.1.4.2.1 Slip this pipe inside the outer pipe. It will extend beyond the outer pipe at the upper end forming a handle to allow the sampler to rotate the inner tube within the outer tube.

3.1.4.2.2 Cut a 1.25 in. wide × 12 in. long sampling port through both pipes 1.5 in. from the lower, plugged end.

3.1.4.2.3 Create index marks on the outside of the outer and inner pipes at the upper end labeled “open” and “closed” to indicate the relative position of the opening in the inner pipe to that of the outer.

3.2 *Bagged Hydrated Lime:*

3.2.1 *Paint brush,* 2 in. wide.

3.2.2 *Bucket,* 1 gal., with double friction type lid and bail.

3.2.3 *Bag sampling tube,* made from 0.75-in. diameter steel electrical conduit 3 ft. long. Taper the opening at one end with a 4-in. diagonal cut.

3.3 *Quicklime:*

3.3.1 *Safety goggles.*

3.3.2 *Respirator.*

3.3.3 *Rubber gloves.*

3.3.4 *Paint brush,* 2 in. wide.

3.3.5 *Bucket,* 1 gal., with double friction type lid and bail.

3.3.6 *Device designed to hold a sample bucket between the wheel path of a bulk transport discharging quicklime*, without allowing the bucket to turn over, but permitting safe, easy removal of the container from the windrow with the sample intact. One suggested design:

3.3.6.1 From 0.75-in. plywood, cut into three squares: 18 in., 14 in., and 10 in.

3.3.6.2 Cut holes to closely fit the 1-gal. sample bucket in the centers of the smaller two plywood squares.

3.3.6.3 Center the 10-in. board on top of the 14-in. board, and fasten them together.

3.3.6.4 Center these on top of the 18-in. board, and fasten together.

3.3.6.5 To a corner of this unit, fasten an 8-ft. length of 1/8-in. diameter flexible, steel cable.

3.3.6.6 To the other end of the cable, attach a 10-in. length of steel conduit or wooden dowel as a “T” handle used to slide the filled bucket from windrowed quicklime.

3.3.7 *Plastic sample bag,* 18 × 9.5 in.—General Warehouse No. 2005000.

3.4 *Commercial Lime Slurry and Carbide Lime Slurry:*

3.4.1 *Safety goggles.*

3.4.2 *Polyethylene bottle,* 1/2 gal., large mouth (3.5-in. diameter), with a screw cap—Nalgene Company No. 2234-0020.

3.4.3 *Plastic electrical tape,* PVC 0.75 in. wide.

3.4.4 *Cloth rag or shop towel.*

4. SAMPLING PROCEDURES

4.1 *Hydrated Lime:*

4.1.1 *Bulk from Loaded Tank Trucks:*

WARNING: The Contractor pressurizes the trucks for unloading, and any attempts to open a pressurized top hatch could be fatal. Therefore, the Contractor should make the load available for sampling before pressurization with top hatches open. If the truck offered for sampling is pressurized, it is the Contractor's responsibility to bleed off the pressure and open the top hatches.

4.1.1.1 Randomly select a truck to sample. The preferred sampling method is rodding material from the truck through an open hatch before the truck is pressurized. Use the top hatch, sampling device or the bottom sampler.

4.1.1.2 The top hatch device samples the upper portion of the load through the top hatches of a bulk transport.

4.1.1.2.1 Insert the unit with the air hole in the rubber stopper open.

4.1.1.2.2 When withdrawing the tube, hold the hole shut with a thumb.

4.1.1.2.3 Place the sample in a 1-gal. bucket.

4.1.1.2.4 Collect at least a 1/2-gal. sample.

4.1.1.3 Use the bottom sampler to obtain bottom samples or to take samples at various levels within the truck.

4.1.1.3.1 Insert the device at the sampling level desired with its port closed.

4.1.1.3.2 Open the port, slide the tube back and forth, and then close the port.

4.1.1.3.3 Remove the tube.

4.1.1.3.4 To release the lime from the tube into the 1-gal. bucket, tap the device, and allow the lime to exit from the top end.

4.1.1.3.5 Collect at least a 1/2-gal. sample.

4.1.1.4 Use the paintbrush to clear the sample bucket rim of lime collected during sampling. This will ensure an effective seal and preserve the quality of the lime sample.

Note 1—Exposure to the atmosphere contaminates lime.

4.1.1.5 If unable to sample the material in the truck before unloading, then use another sampling method, provided the sample is representative and not contaminated by moisture, base, or other road material. These less desirable methods include:

- scoop samples obtained through open top hatches,
- as discharged from tank trucks,
- hose discharge, and
- dry application "catch-pan" method.

Note 2—Do not scoop samples from material applied on roadway due to likelihood of contamination.

4.1.1.6 Identify the truck on the sample ticket by seal number, name of producer, and date sampled. Never combine samples from separate trucks.

4.1.2 *Bagged from Bag Trucks:*

4.1.2.1 Sample at least six sacks to represent each truck delivery.

4.1.2.2 Sample at least four to six sacks from each inspected lot and combine the material to form a composite sample representing that lot.

4.1.2.3 Take samples from the separate lots that comprise the whole if the entire lot consigned for an individual truck is from several different warehouse lots.

4.1.2.4 Insert the bag sampling tube through the sack-loading spout and take sufficient diagonal roddings, to ensure a representative portion from each sack without significantly reducing the volume. Take care not to puncture the sack with the sampling tube. Place samples in a 1-gal. bucket.

4.1.2.5 Use the paintbrush to clear the sample bucket rim of lime collected during sampling. This will ensure an effective seal and preserve the quality of the lime sample.

Note 3—Exposure to the atmosphere contaminates lime.

4.1.3 *Bagged from Bagged Shipments:*

4.1.3.1 Use the bag sampling tube described in Section 3.1.5.3 to obtain a 1/2-gal. sample from at least six sacks of material.

4.1.3.2 Select sacks for sampling from various points in the load or shipment to collect a representative sample.

4.1.3.3 Insert the bag sampling tube through the sack-loading spout and take sufficient diagonal roddings to ensure a representative portion from each sack without materially reducing the poundage. Take care not to puncture the bottom or sides of the sack with the sampling tube. Place samples in a 1-gal. bucket.

4.1.3.4 Use the paintbrush to clear the sample bucket rim of lime collected during sampling. This will ensure an effective seal and preserve the quality of the lime sample.

Note 4—Exposure to the atmosphere contaminates lime.

4.2 *Quicklime:*

WARNING: Quicklime is extremely hazardous and capable of inflicting severe caustic burns to skin, lung damage, eye injury, and even blindness if handled improperly. Personnel handling, sampling, or testing quicklime should wear proper protective clothing, respirators, dust-proof goggles, and waterproof gloves.

4.2.1 *Discharged from Tank Trucks:*

4.2.1.1 Instruct the truck to pass over a collection device while unloading.

Caution: Quicklime generates fines in transit. Since air-blown quicklime fines are hazardous, quicklime is usually unloaded at the jobsite by gravity feed through ports at the bottom of each compartment on the truck. Most trucks are equipped with three or four such compartments, opened simultaneously to discharge the quicklime.

4.2.1.2 Collect samples from the midpoint of the unloading of the truck.

Note 5—Specifications limit the amount of fines in the sample and include sizing requirements. The sizing and gradation of the sample taken must be representative of the load. Quicklime fines tend to settle to the bottom of the compartments, and the initial discharge usually contains a higher percentage than the remainder of the load. The top of the load tends to contain the coarsest material. The center of the discharge run best represents the gradation of the entire load.

4.2.1.3 Pick up the collection device and carefully transfer the entire sample to a plastic sample bag. Close and seal the bag with tape or rubber band and place in a 1-gal. bucket for transport. Label the bucket as “caustic quicklime.”

4.2.1.4 Ship samples by motor freight only.

CAUTION: Do not ship by bus, parcel post, air, or rail. This is a hazardous material, which upon contact with water and combustibles can cause fires. For this and other safety-related reasons, the carriers listed have refused to accept the material for shipment.

4.2.2 *From Tank Trucks:*

4.2.2.1 Collect samples from the top of the trucks. Dig below the surface of the pebble quicklime at least 8 in. and scoop a sample with a 1-gal. bucket. A sample should be a minimum of 3/4 gal.

4.2.2.2 Carefully transfer the entire sample to a plastic sample bag. Close and seal the bag with tape or rubber band and place in a sample bucket for transport. Label the bucket as “caustic quicklime.”

4.2.2.3 Ship samples by motor freight only.

CAUTION: Do not ship by bus, parcel post, air, or rail. This is hazardous material, which, upon contact with water and combustibles, can cause fires. For this and other safety-related reasons, the carriers listed have refused to accept the material for shipment.

4.3 *Commercial Lime Slurry:*

4.3.1 *From the Truck:*

4.3.1.1 Draw the sample from the permanent sampling port located concentrically at the rear of the truck.

Note 6—The sampling port consists of a 0.5 in.-minimum, quick acting valve fitted to a 0.75-in. diameter pipe and outlet spout.

4.3.1.2 Open the sampling valve quickly and completely during sampling.

4.3.1.3 Half fill the plastic sample jug to permit agitation and testing.

4.3.1.4 Tightly seal the jug and tape the cap to avoid leakage during transport.

4.3.1.5 Take one sample to represent the truckload.

4.3.1.6 A Department representative must witness sampling.

Note 7—It is the sole responsibility of the truck driver to sample, cap, and seal the slurry sample upon direct request from a Department representative.

4.3.2 *From the Plant:*

4.3.2.1 Collect a sample from the sampling valve in the vertical riser from the slurry tank to the loading spout.

4.3.2.2 Half fill the plastic sample jug to permit agitation and testing.

4.3.2.3 Tightly seal the jug and tape the cap to avoid leakage during transport.

4.3.2.4 Take one sample to represent the truckload.

4.3.2.5 A Department representative must witness sampling.

4.4 *Carbide Lime Slurry:*

4.4.1 *From the Truck:*

4.4.1.1 Draw the sample from the permanent sampling port located concentrically at the rear of the truck.

Note 8—The sampling port consists of a 0.5-in. minimum, quick acting valve fitted to a 0.75-in. diameter pipe and outlet spout.

4.4.1.2 Open the sampling valve quickly and completely during sampling.

4.4.1.3 Half fill the plastic sample jug to permit agitation and testing.

4.4.1.4 Tightly seal the jug and tape the cap to avoid leakage during transport.

4.4.1.5 Take one sample to represent the truckload.

4.4.1.6 A Department representative must witness the sampling.

Note 9—It is the sole responsibility of the truck driver to sample, cap, and seal the slurry sample upon direct request from a Department representative.

PART II—TESTING HYDRATED LIME

5. SCOPE

5.1 This part discusses the method for testing high-calcium type hydrated lime. This method assumes the presence of the following constituents in the lime:

- calcium hydroxide,
- calcium oxide or water,
- calcium carbonate, and
- inert matter such as silica dioxide.

6. APPARATUS

6.1 *Electric muffle furnace,* at 2,000 ± 20°F (1093 ± 11°C).

6.2 *Platinum crucibles,* low-form, wide-bottom type, 30 cc capacity. Only substitute with ceramic crucibles if the crucibles are able to withstand temperatures of 2,000°F (1093°C).

6.3 *Tongs,* for use with muffle furnace, 20-in. length.

6.4 *Hard-surfaced temperature resistant board.*

6.5 *Oven,* capable of maintaining a temperature of 212°F (100°C).

6.6 *Vacuum desiccator.*

6.7 *Silica-gel desiccant, indicating type.*

6.8 *Analytical balance,* Class A in accordance with Tex-901-K, for rapid weighing, with a minimum weighing capacity of 100 g.

6.9 *Top loading balance,* Class G1 in accordance with Tex-901-K, for rapid weighing, with a minimum weighing capacity of 2000 g.

6.10 *Weighing boats.*

6.11 *Spatula.*

6.12 *Balance brush,* camel's hair type.

6.13 *Tall form beakers,* 400 mL, borosilicate type glass.

6.14 *Beaker,* 200 mL.

6.15 *Buret,* class A, of the following types:

- 100-mL capacity, with a 50-mL bulb at the top and a 50-mL graduated tube
- 50-mL capacity with 0.1-mL subdivisions.

6.16 *Flasks,* class A volumetric, with the following capacities:

- 250 mL
- 1000 mL.

6.17 *Graduated cylinders,* with the following capacities:

- 100 mL
- 200 mL
- 1000 mL
- 2000 mL.

6.18 *Stirrer,* magnetic type.

6.19 *Stopwatch,* to register up to 30 min.

6.20 *Plastic tubing,* 3/8-in. ID, 1/8-in. wall thickness of suitable length, fitted with a Bunsen "fan-type" burner attachment at the outlet end of the hose.

6.21 *Water pressure gauge,* 0–30 psi, 3-in. diameter face, with 0.375-in. IPS female thread fitting.

6.22 *Standard U.S. sieves,* meeting the requirements of Tex-907-K, in the following sizes:

- No. 6
- No. 30.

6.23 *pH meter,* with an accuracy of ± 0.1 pH unit or better, with automatic temperature compensation and standard combination type electrode.

6.24 *pH meter reference electrode filling solution,* KCl, 4 M saturated with AgCl.

6.25 *Sample vial,* 1-3/4-in. diameter × 3-in. high vial made of clear polystyrene with a tight fitting polyethylene cap.

6.26 *Plastic bottle,* 19 L, for storing 1.0 N sodium hydroxide.

6.27 *Glass bottle,* 19 L, for storing 1.0 N hydrochloric acid.

7. REAGENTS

7.1 Use the following reagents, which all must be American Chemical Society (ACS) reagent-grade:

- potassium hydrogen phthalate
- bromophenol blue
- ethyl alcohol
- hydrochloric acid (HCl), 36.5 to 38.0%
- phenolphthalein powder
- sodium tetraborate decahydrate
- sodium hydroxide (NaOH), 50% solution.

8. SOLUTIONS

8.1 *Sodium Hydroxide 1.0 N:*

8.1.1 *Preparation:*

8.1.1.1 Fill a 19-L plastic container 1/2–2/3 full with de-ionized water.

8.1.1.2 Add 967.6 mL of 50% sodium hydroxide solution.

Note 10—Add the sodium hydroxide solution to a sizable portion of water to dissipate the heat evolved and to avoid hazardous spattering.

8.1.1.3 Mix for 5–10 min.

8.1.1.4 Bring to 18.5 L mark on the container.

8.1.1.5 Stir for at least 6 hr., but preferably 12 hr.

8.1.1.6 Standardize with potassium hydrogen phthalate.

8.1.2 *Standardization of a 1.0 ± 0.0005 N sodium hydroxide solution:*

8.1.2.1 Transfer 50 mL of the sodium hydroxide solution into a 50-mL buret.

8.1.2.2 Weigh 7.5–8.5 g of potassium hydrogen phthalate onto a weighing boat. Record the weight to the nearest 0.0001 g.

8.1.2.3 Transfer the weighed material to a 400-mL tall-form beaker.

8.1.2.4 Add 185 mL of deionized water and mix with a magnetic stir bar. Stir the solution until all of the potassium hydrogen phthalate dissolves.

8.1.2.5 Add five drops of phenolphthalein indicator solution.

8.1.2.6 Titrate with the sodium hydroxide solution to a visual endpoint of light pink, which will persist for at least 60 sec.

8.1.2.7 Record the amount of sodium hydroxide used.

8.1.2.8 Calculate the normality. If the normality is not within the limits 1.000 ± 0.0005 N, add water or sodium hydroxide to adjust the normality.

8.1.2.9 If the sodium hydroxide solution is too:

- weak, follow "Example 1" below to calculate the amount of the 50% sodium hydroxide to add.
- strong, follow "Example 2" below to calculate the amount of deionized water to add.

8.1.2.10 Measure and add the correct amount of sodium hydroxide or deionized water. Stir the solution for at least 4 hr.

8.1.2.11 Repeat the standardization procedure. Repeat adjustments, if necessary. After all adjustments are complete, standardize the resulting solution.

8.1.3 *Calculations:*

8.1.3.1 Calculate the normality of sodium hydroxide solution:

$$\text{Normality of NaOH} = \frac{\text{Wt. of Potassium Hydrogen Phthalate in (g)}}{\text{mL of NaOH required} \times 0.204228}$$

8.1.3.2 Adjusting the sodium hydroxide solution to achieve a 1.0 N solution:

8.1.3.2.1 Example 1: If base is too weak, for example, if 18.5 L of 0.9907 N solution needs strengthening to 1.0000 N, calculate the amount of 50% sodium hydroxide to add:

- $18.500 \times 0.991 = 18.334$
- $18.500 - 18.334 = 0.166$
- $0.166 \times 52.3 = 8.68$ mL of 50% sodium hydroxide solution needed.

8.1.3.2.2 Example 2: If base is too strong, for example, if the 18.5 L of 1.021 N sodium hydroxide solution needs weakening to 1.0000 N, calculate the amount of deionized water to add:

- $18.500 \times 1.021 = 18.888$
- $18.888 - 18.500 = 0.388$ L or 388 mL of deionized water needed.

8.2 *1.0 N Hydrochloric Acid Solution:*

8.2.1 *Preparation:*

8.2.1.1 Fill a 19-L glass container 1/2–2/3 full with deionized water.

8.2.1.2 Add 1625 mL of concentrated hydrochloric acid.

Note 11—It is important to add the acid to a sizable portion of water to dissipate the heat evolved and avoid hazardous spattering of hot acid, fuming, etc.

8.2.1.3 Add sufficient water to bring the total volume to 19 L.

8.2.1.4 Stir the solution at least 4 hr., but preferably 12 hr.

8.2.1.5 Standardize against the sodium hydroxide.

8.2.2 *Standardization of a 1.0 ± 0.0005 N hydrochloric acid solution:*

8.2.2.1 Transfer 40 mL of the hydrochloric acid solution into a 400-mL tall beaker using a pipette for accuracy.

8.2.2.2 Add 140 mL of deionized water.

8.2.2.3 Add five drops of phenolphthalein indicator.

8.2.2.4 Fill a 50-mL buret with 1.0 N sodium hydroxide solution. Titrate hydrochloric acid with the sodium hydroxide solution to a visual endpoint of light pink, which will persist for at least 60 sec. Record the amount of the sodium hydroxide solution used.

8.2.2.5 Calculate the normality. If the normality is not within the limits of 1.000 ± 0.0005 N, add hydrochloric acid or water to adjust the normality.

8.2.2.6 If the hydrochloric acid solution is too:

- weak, follow “Example 1” below to calculate the amount of the concentrated hydrochloric acid to add.
- strong, follow “Example 2” below to calculate the amount of deionized water to add.

8.2.2.7 Measure and add the correct amount of hydrochloric acid or deionized water. Stir the solution for at least 2 hr.

8.2.2.8 Repeat the standardization procedure. Repeat adjustments, if necessary. After all adjustments are complete, standardize the resulting solution.

8.2.3 *Calculations:*

8.2.3.1 Calculate the normality of hydrochloric acid solution:

$$\text{Normality of HCL} = \frac{\text{mL of NaOH required} \times \text{N of NaOH used}}{\text{mL of HCL used}}$$

8.2.3.2 Adjusting the hydrochloric acid solution to achieve a 1.0 N solution:

8.2.3.2.1 Example 1: If acid solution is too weak, for example, if 19.7 L of 0.9908 N solution needs strengthening to 1.0000 N, calculate the amount of concentrated hydrochloric acid to add:

- 19.700 × 0.991 = 19.523
- 19.700 − 19.523 = 0.177
- 0.177 × 82.5 = 14.6 mL of concentrated hydrochloric acid needed.

8.2.3.2.2 Example 2: If acid solution is too strong, for example, if 19.7 L of 1.024 N solution needs weakening to 1.0000 N, calculate how much deionized water to add:

- 19.700 × 1.024 = 20.1732
- 20.173 −19.700 = 0.473 L or 473 mL of deionized water needed.

8.3 *4.0 pH Buffer (using Potassium Hydrogen Phthalate):*

8.3.1 Weigh 10.21 ± 0.05 g of potassium hydrogen phthalate into a weighing boat.

8.3.2 Add 500 mL of deionized water to a 1000-mL volumetric flask.

8.3.3 Transfer the potassium hydrogen phthalate into the volumetric flask. Add a magnetic stir bar and stir the solution until all of the potassium hydrogen phthalate dissolves.

8.3.4 Dilute the solution to 1000 mL.

8.3.5 The pH of the potassium hydrogen phthalate solution at various temperatures is shown in Table 1.

Table 1—pH of 0.05 M Potassium Hydrogen Phthalate

Temperature °F (°C)	**pH**
59 (15)	3.999
68 (20)	4.002
77 (25)	4.008
86 (30)	4.015
95 (35)	4.024
104 (40)	4.035

8.4 *9.0 pH Buffer (using Sodium Tetraborate Decahydrate):*

8.4.1 Weigh 3.81 ± 0.01 g of sodium tetraborate decahydrate into a weighing boat.

8.4.2 Add 500 mL of deionized water to a 1000-mL volumetric flask.

8.4.3 Transfer the sodium tetraborate decahydrate into the volumetric flask. Add a magnetic stir bar and stir the solution until all of the sodium tetraborate decahydrate dissolves.

8.4.4 Dilute the solution to 1000 mL.

8.4.5 The pH of borax solution at various temperatures is shown in Table 2.

Table 2—pH of 0.01 M Borax Solution

Temperature °F (°C)	pH
59 (15)	9.276
68 (20)	9.225
77 (25)	9.180
86 (30)	9.139
95 (35)	9.102
104 (40)	9.068

8.5 *Phenolphthalein Indicator:*

8.5.1 Weigh 0.5 g of powdered phenolphthalein in a 200-mL beaker.

8.5.2 Add 50 mL of ethyl alcohol and a magnetic stir bar.

8.5.3 Stir until all the phenolphthalein dissolves.

8.5.4 Add 50 mL of deionized water and stir.

8.6 *Bromophenol Blue Indicator:*

8.6.1 Weigh 0.1 g of powdered bromophenol blue into a 250-mL volumetric flask.

8.6.2 Add 7.5 mL of 0.02 N sodium hydroxide solution. Add a magnetic stir bar and stir until all of the bromophenol blue dissolves.

8.6.3 Dilute the mixture with deionized water to 250 mL.

9. SAMPLE PREPARATION

9.1 Mix the sample well for 1–2 min.

9.2 Invert and shake the bucket at least three times.

9.3 Remove the bucket lid and scoop out enough material to fill a sample vial 1/2–3/4 full.

9.4 After sample withdrawal, close the bucket and sample vial to minimize atmospheric contamination.

Note 12—Moisture in the air tends to air slake calcium oxide, converting it to calcium hydroxide. Calcium hydroxide tends to react with carbon dioxide in the air to form calcium carbonate, which degrades the quality of the sample by reducing the active lime content.

10. TESTING PROCEDURES

10.1 *Titration to pH 8.3:*

10.1.1 Weigh out on an analytical balance 2.804 ± 0.0050 g of lime from the sample vial. Reseal the vial. Record the weight of the lime sample.

10.1.2 Add 150 mL of deionized water to a 400-mL tall-form beaker.

10.1.3 Transfer the weighed sample to the 400-mL tall-form beaker.

Note 13—To minimize loss of sample, transfer material slowly.

10.1.4 Add a stirring magnet and place the beaker on a magnetic stirrer. Stir the sample mixture.

10.1.5 Calibrate the pH meter using the 4.0 and 9.0 buffer solutions.

10.1.6 Fill a 100-mL buret with 1.0 N hydrochloric acid solution.

10.1.7 Titrate the sample with the first 50 mL of the 1.0 N hydrochloric acid solution to a pH of 9.0. Titrate as rapidly as the reaction rate of the sample will allow.

Note 14—The response or reaction time of the sample dictates the rate of addition. Lime samples will vary in response to additions of titrant. An excess of titrant causes localized pooling of titrant. Carbon dioxide gas evolves from the reaction of acid with calcium carbonate at a pH of less than 5.4. In the titration to pH 8.3, rapidly add the titrant, but slow enough to keep the pH greater than 6.0 at all times. With addition of titrant, the pH will drop and then, in most cases, recover or rise to a higher value as more solids dissolve and enter into the reaction.

10.1.8 Decrease the rate of titration to a rapid drop rate until reaching a pH of between 6.0 and 9.0 that persists for at least 30 sec.

10.1.9 Use smaller increments of titrant to maintain a pH slightly less than 8.3. As the pH increases, add more titrant until a pH of 8.3 or slightly less persists for 60 sec.

Note 15—The endpoint for the titration to pH 8.3 occurs when the addition of one 2-drop increment of titrant produces a pH of 8.3, or slightly less, for exactly 60 sec. after addition.

10.1.10 Record the volume of titrant required to reach this endpoint.

10.1.11 Add an excess of hydrochloric acid to the sample. Record the total volume of acid added.

Note 16—The sample mixture contains a "measured excess" of hydrochloric acid.

10.2 *Back-Titration to pH 4.4:*

10.2.1 Fill a 50-mL buret with 1.0 N sodium hydroxide solution.

10.2.2 Titrate the sample with 1.0 N sodium hydroxide solution to a pH of 4.4 or slightly greater when observed exactly 10 sec. after a 1-drop addition of titrant.

10.2.3 Record the sodium hydroxide volume at the first occurrence of the endpoint.

10.3 *Loss on Ignition:*

10.3.1 Weigh a crucible using an analytical balance, record the weight, and tare the balance.

10.3.2 Weigh out a 2.9–3.1-g sample in the crucible. Record the weight of the sample to the nearest 0.0001 g.

10.3.3 Place the crucible containing the sample in an electric muffle furnace set at 2,000 ± 20°F (1093 ± 11°C) for a minimum of 3 hr.

10.3.4 Use tongs to remove the crucible from the muffle furnace. Place the crucible on a hard-surfaced asbestos board. Allow the crucible to cool on the board for 10–30 sec.

10.3.5 Transfer the crucible to a vacuum desiccator for cooling. Allow 20 min. for cooling.

Note 17—If running several samples simultaneously, transfer all of the samples to the desiccator and replace the lid with the vacuum stopcock open. The heated air will expand through the opening for several seconds. Then close the stopcock.

10.3.6 After the sample has cooled, remove the sample from the desiccator. Weigh the sample on an analytical balance as rapidly as possible to minimize hydration of the residue. Record the weight to the nearest 0.0001 g.

10.4 *Dry Sieve Analysis:*

10.4.1 Use the remainder of the sample in the 1-gal. sample bucket for sieve analysis. Weigh the sample on a top loading balance to the nearest 0.1 g.

10.4.2 Shake the sample through a No. 6 sieve in 200-g increments.

Note 18—Perform this operation under a hood while wearing a dust/mist mask.

10.4.3 Weigh the amount retained on the No. 6 sieve to the nearest 0.1 g, and then recombine for the wet sieve analysis.

10.5 *Wet Sieve Analysis:*

10.5.1 Use the remainder of the sample in the 1-gal. sample bucket for wet sieve analysis. Individually weigh a No. 6 and a No. 30 sieve on a top loading balance. Record the weights to the nearest 0.1 g.

10.5.2 Weigh the sieve analysis sample to the nearest 0.1 g.

10.5.3 Water wash the sample through the nested No. 6 and No. 30 sieves. Ensure that none of the sample is lost over the sides of the sieve. Use a water pressure gauge to maintain 12 ± 0.3 psi water pressure.

Note 19—Apply the wash water in a fantail spray pattern from the 3/8-in. (9.5-mm) ID plastic tubing fitted with a Bunsen "fan-type" burner attachment at the outlet.

10.5.4 Continue to wash the sample for 30 min. or until the water coming through the sieve is clear, whichever occurs first.

10.5.5 Oven-dry the residue on each sieve for 1 hr. at 212 ± 4°F (100 ± 2°C).

10.5.6 Remove the sieves from the oven. Allow the sieves to cool to room temperature. Weigh each sieve to the nearest 0.1 g.

11. CALCULATIONS

11.1 *Wet and Dry Sieve Analysis:*

11.1.1 Calculate the percent of the hydrated lime retained on each sieve. Use this calculation for both wet and dry sieve analysis. Report values to the nearest 0.1%:

$$\%\ \text{retained} = \frac{\text{residue retained}}{\text{sample wt.}} \times 100$$

Where:

% retained = weight percent of residue retained on the sieve

residue retained = weight in grams of residue retained on the sieve

sample wt. = weight in grams of the sample before being sieved.

11.2 *Total "Active" Lime Content:*

11.2.1 Limits for sample weight and titration solutions when using the following rapid calculation methods are as follows.

- Sample Wt. 2.804 ± 0.0050 g
- Acid 1.000 N ± 0.0005 N
- Base 1.000 N ± 0.0005 N.

11.2.2 Creating a hydrated lime sample worksheet:

11.2.3 Milliliters of hydrochloric acid to pH 8.3 is equivalent to the hydrate alkalinity. Record this value and the corresponding uncorrected $Ca(OH)_2$ from Table 3.

11.2.4 Total milliliters hydrochloric acid added, less the milliliters of sodium hydroxide required to obtain a pH of 4.4, is equal to the total alkalinity. Record this value and subtract hydrate alkalinity from total alkalinity to give carbonate alkalinity. From Table 4, obtain equivalent calcium carbonate and record this value.

11.2.5 Calculate the loss on ignition of the sample. Calculate the inert value by subtracting the sum of the loss on ignition and the total alkalinity from 100. Record the inert value (or an average or assumed inert value based on previous analysis of lime from a given source).

11.2.6 Total the uncorrected calcium hydroxide, calcium carbonate, and the inert value. If the total is less than 100%, the difference is free water. If the total is greater than 100%, quicklime is present. The following examples demonstrate each situation:

- Example where calcium oxide is present:

$$
\begin{aligned}
&Uncorrected\,Ca(OH)_2 = 94.8 \\
&CaCO_3 = 3.1 \\
&Assumed\,inert = 2.2 \\
&\overline{100.1}
\end{aligned}
$$

$$
\begin{aligned}
&Subtotal\,100.1 - 100 = 0.1 \\
&\%CaCO = 0.1x3.11 = 0.3
\end{aligned}
$$

$$
\begin{aligned}
&Uncorrected\,Ca(OH)_2 = 94.8 \\
&Ca(OH)_2\,Correction\,Value = -\,0.4 \\
&\overline{94.4} \\
&Analysis\,is\,shown\,as: \\
&Ca(OH)_2 = 94.4 \\
&CaCO_3 = 3.1 \\
&CaO = 0.3 \\
&Total = \overline{100.0}
\end{aligned}
$$

- Example where water is present:

$$
\begin{aligned}
&Uncorrected\ Ca(OH)_2 &&= 93.0\\
&CaCO_3 &&= 4.6\\
&Assumed\ Inert &&= 1.0\\
&\qquad\qquad Total &&= \overline{98.6}
\end{aligned}
$$

$$\% H_2O = 100 - 98.6 = 1.4$$

Analysis is shown as:

$$
\begin{aligned}
&Ca(OH)_2 &&= 93.0\\
&CaCO_3 &&= 4.6\\
&Assumed\ Inert &&= 1.0\\
&H_2O &&= 1.4\\
&Total &&= \overline{100.0}
\end{aligned}
$$

11.2.7 If free water is present, the uncorrected hydrate alkalinity will be actual hydrate alkalinity as calcium hydroxide.

Table 3—Uncorrected Calcium Hydroxide Value

mL of Acid and Hydrated Alkalinity	% Ca(OH)2
68.0	89.8
69.0	91.2
70.0	92.5
71.0	93.8
72.0	95.1
73.0	96.4
74.0	97.8
75.0	99.1
Factor = 1.32126	

11.2.8 Calcium Carbonate Value as $CaCO_3$ —Whole milliliter difference between two endpoints:

Table 4—Calcium Carbonate Value

Carbonate Alkalinity	**$CaCO_3$**
0.0	0.0
1.0	1.8
2.0	3.6
3.0	5.4
4.0	7.1
Factor = 1.78479	

11.2.9 Correction for Calcium Hydroxide Value when CaO is Present:

Table 5—Correction for Calcium

Subtotal-100%	**Correction Value**
0.1	0.4
0.2	0.8
0.3	1.2
0.4	1.6
0.5	2.1
0.6	2.5
0.7	2.9
0.8	3.3
0.9	3.7
1.0	4.1
1.1	4.5
1.2	4.9

11.2.10 Amount of Subtotal Over 100%:

- When quicklime is present: % CaO = (Subtotal - 100%) × 3.11
- When free water is present: % free water = 100% - Subtotal

Table 6—Proportional Parts

ml acid	% $Ca(OH)_2$
0.1	0.1
0.2	0.3
0.3	0.4
0.4	0.6
0.5	0.7
0.6	0.8
0.7	1.0
0.8	1.1
0.9	1.3
Factor = 1.32126	

11.2.11 Use Table 7 to obtain a corrected calcium hydroxide value by subtracting the calcium hydroxide correction value obtained from the uncorrected calcium hydroxide value.

Table 7—Proportional Parts

Fractional Carbonate Alkalinity	$CaCO_3$
0.1	0.2
0.2	0.4
0.3	0.5
0.4	0.7
0.5	0.9
0.6	1.1
0.7	1.3
0.8	1.4
0.9	1.6
Factor = 1.78479	

11.2.11.1 Use Table 7 if the subtotal (uncorrected calcium hydroxide value + calcium carbonate value + assumed inert value) is greater than 100%. This means that calcium oxide is present and not free water.

11.2.11.2 If the subtotal is less than 100%, then assume the uncorrected calcium hydroxide value is correct. Calcium hydroxide and free water are present.

12. OPTIONAL INDICATOR METHOD FOR THE TITRIMETRIC ANALYSIS

12.1 *Scope:*

12.1.1 Use this method in lieu of the prescribed pH meter method; however, for all referee tests, or in cases of dispute, the prescribed pH meter method is the official method.

12.1.2 In some cases, impurities such as aluminum compounds will prevent or hinder the appearance of the color change in the back-titration portion of this optional method. In these cases, use the prescribed pH meter method.

12.2 *Titration to Phenolphthalein Endpoint:*

12.2.1 Weigh out on an analytical balance 2.804 ± 0.0050 g of lime from the sample vial. Reseal the vial. Record the weight of the lime sample.

12.2.2 Add 150 mL of deionized water to a 400-mL tall-form beaker.

12.2.3 Transfer the sample to the beaker.

Note 20—To minimize loss of sample, transfer material slowly.

12.2.4 Add a stirring magnet and place the beaker on a magnetic stirrer. Stir the sample.

12.2.5 Add five drops of phenolphthalein indicator to the sample mixture.

12.2.6 Fill a 100-mL buret with 1.0 N hydrochloric acid.

12.2.7 Titrate to the point where the color of the solution changes temporarily from pink to clear.

12.2.8 Wait for the pink color to return.

12.2.9 Add titrant until the color change appears imminent.

12.2.10 Add acid in 4-drop increments until the pink coloration disappears for 35 sec.

12.2.11 Add acid in 2-drop increments until the endpoint occurs. Record the amount of hydrochloric acid used to reach the endpoint.

Note 21—The endpoint is the failure of the mixture to regain its pink coloration for 60 sec. after the last addition of the titrant. After reaching the endpoint, ignore the reappearance of pink color. The phenolphthalein endpoint is the equivalent to the pH 8.3 endpoint described in the official pH meter method of titration.

12.2.12 Add an excess of hydrochloric acid to the sample. Record the total volume of acid added.

Note 22—The sample mixture contains a "measured excess" of hydrochloric acid.

12.3 *Back Titration to the Bromophenol Blue Endpoint:*

12.3.1 Fill a 50-mL buret with 1.0 N sodium hydroxide.

12.3.2 Add 2.0 mL of bromophenol blue indicator to the sample mixture.

12.3.3 Titrate until the color of the mixture changes from yellow to blue with a slight tinge of purple and maintains the blue color for 10 sec., after a 1-drop addition of titrant.

12.3.4 Record the amount of sodium hydroxide used to reach the endpoint.

12.3.5 Use the same calculation method to determine active lime content as described under Section 10.

PART III—TESTING COMMERCIAL LIME SLURRY

13. SCOPE

13.1 This part discusses the method for testing high-calcium type commercial lime slurry and assumes that the principal constituents present in the lime are:

- calcium hydroxide,
- water,
- calcium carbonate, and
- inert matter such as silica dioxide.

14. APPARATUS

14.1 *Equipment,* listed under Part II, Section 6.

14.2 *Eyedropper,* large bore polyethylene.

14.3 *Weight-per-gallon cup,* stainless steel, with tare weight.

14.4 *Polyethylene bottles,* 1 pt., with screw caps.

14.5 *Glass rod.*

14.6 *Beaker,* 1000 mL.

15. REAGENTS AND SOLUTIONS

15.1 Use chemical reagents and solutions listed for the testing of bulk hydrate or powdered lime in Part III, Sections 7 and 8.

16. SAMPLE PREPARATION

16.1 *Lime Slurry Sample Preparation:*

16.1.1 Set the slurry jug in a roller and agitate for 3–4 hr.

16.1.2 Use a glass rod to determine if the lime sample is in suspension. If any lime remains caked on the bottom or sides, place the slurry jug back on the roller and agitate.

16.1.3 When the slurry is well mixed, pour a representative sample into a 1-pt. polyethylene bottle with a screw top.

Note 23—Clean the threaded portion of the bottles and the inside threads of the cap. If slurry remains on these places, the seal will not be airtight, and the slurry will dehydrate.

16.2 *Determining Bulk Density:*

16.2.1 Weigh a clean and empty weight-per-gallon cup on a top loading balance. Tare the weight of the cup.

16.2.2 Shake the 1-pt. bottle of slurry vigorously for 1 min. Rapidly pour the slurry from the 1-pt. bottle into the weight-per-gallon cup. Fill the cup to just below the top rim.

16.2.3 Tap the bottom of the filled cup against a hard surface to remove air bubbles.

16.2.4 Place the lid on the cup, gently pressing it into place. Excess slurry will flow out the small center opening in the top.

Note 24—If slurry does not flow out of the opening, additional slurry is necessary. Agitate the sample before adding more slurry.

16.2.5 Carefully wipe off all of the excess slurry on the outside of the cup and lid.

16.2.6 Place the weight-per-gallon cup on a top loader balance. Weigh the cup and its contents to the nearest 0.1 g.

Note 25—Make sure that the tared weight remains displayed on the balance just before the weight of the filled cup is measured.

16.2.7 Divide the result by ten. Report this value as the bulk density of the slurry expressed in lb./gal.

16.2.8 *Calculations:*

16.2.8.1 Calculate bulk density:

$$BD = \frac{(\text{slurry wt.})}{10}$$

Where:

BD = bulk density, (lb./gal.)

slurry wt. = weight of the slurry in the weight-per-gallon cup, (g).

16.3 *Determination of Sample Size:*

16.3.1 Use Figures 1, 2, and 3 to determine the sample size for titrimetric analysis and loss on ignition. Find the bulk density of the slurry on one of the three graphs. Scan to the right to find the area between the minimum and maximum lines. Then, scan to the bottom of the graph to find the corresponding sample weight range. This quantity will contain the equivalent of 2.9–3.1 g of dry lime.

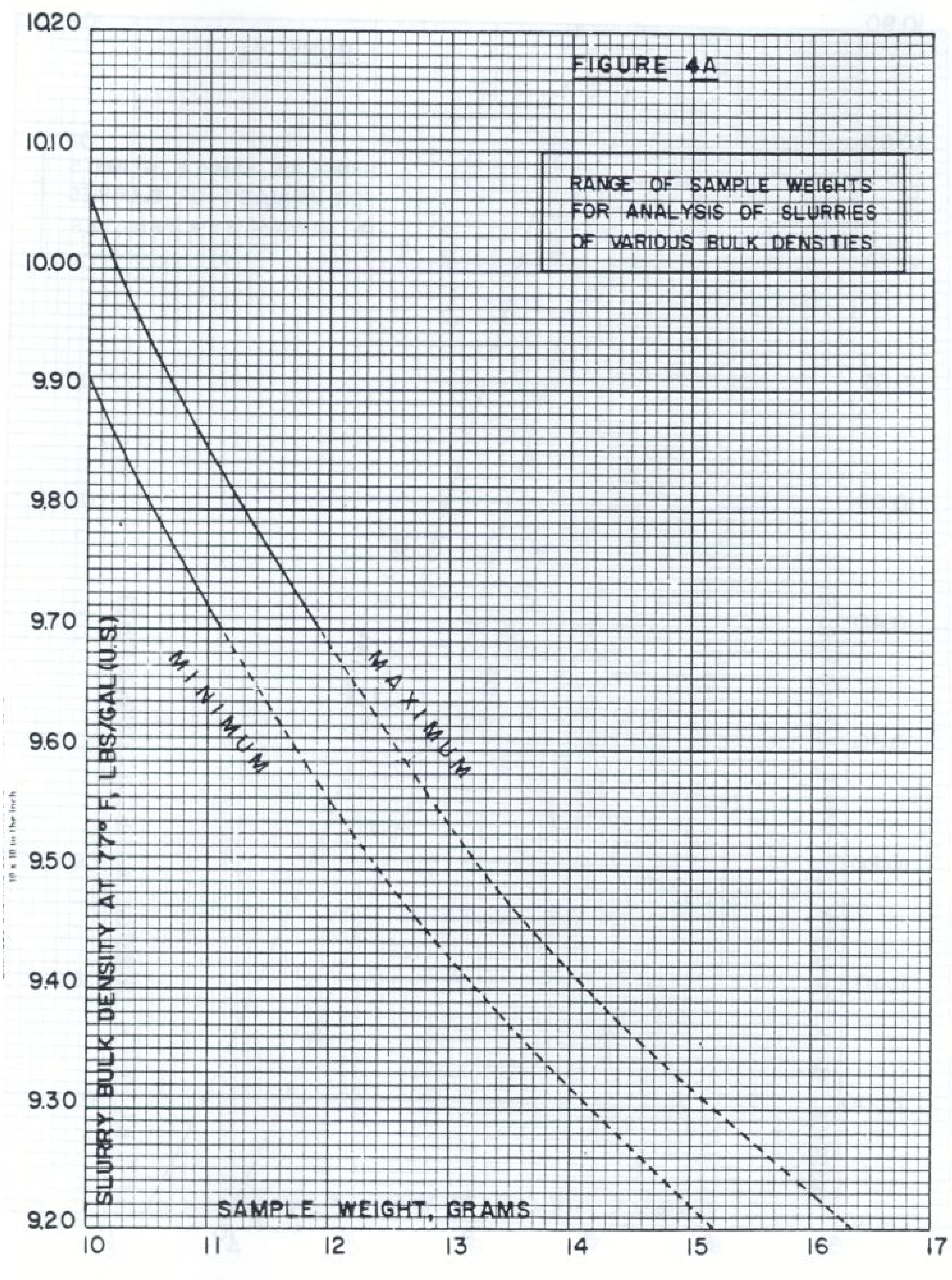

Figure 1—Range of Sample Weights for Analysis of Slurries—No. 1

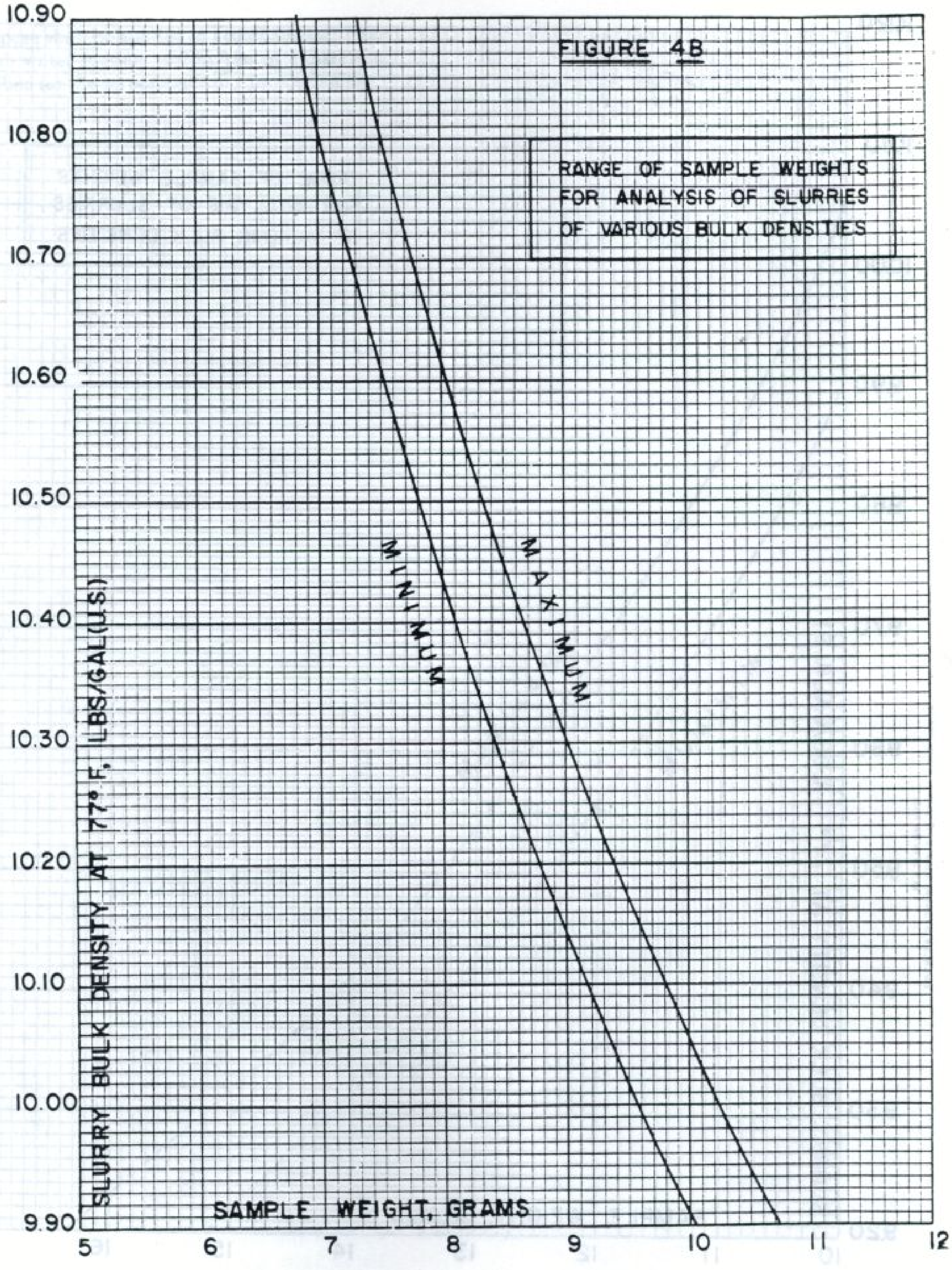

Figure 2—Range of Sample Weights for Analysis of Slurries—No. 2

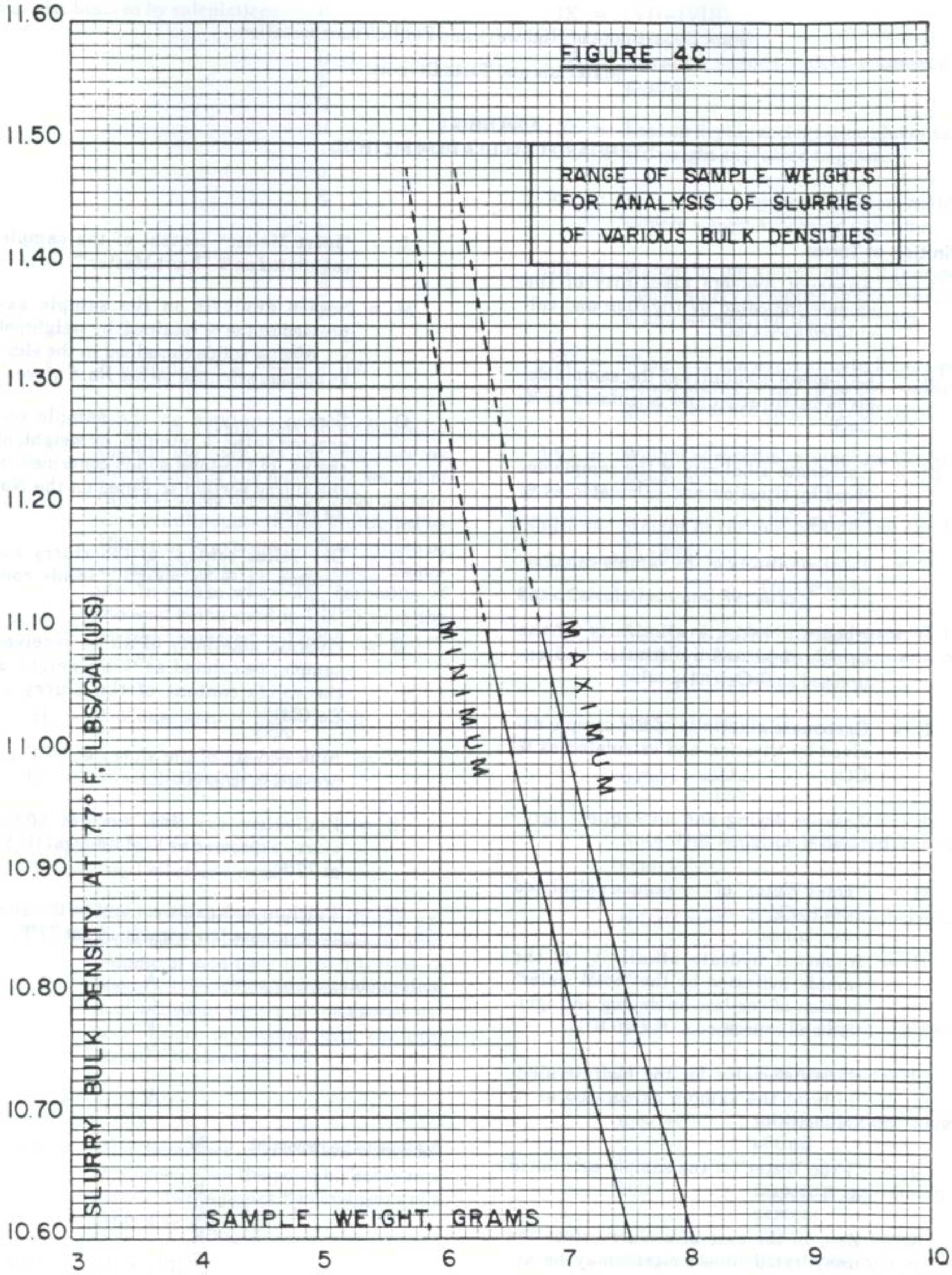

Figure 3—Range of Sample Weights for Analysis of Slurries—No. 3

17. TESTING PROCEDURES

17.1 *Titration to pH 8.3:*

17.1.1 Determine the sample size using the method indicated in Section 16.3.

17.1.2 Tare a 400-mL tall-form beaker on an analytical balance.

17.1.3 Agitate the 1-pt. bottle for 1 min. Pour the required weight of slurry sample, as determined above, into the 400-mL tall-form beaker. Record the weight of the sample to the nearest 0.0001 g.

17.1.4 Slowly add enough deionized water to bring the volume of mixture to 150 mL. Add a stirring magnet, place the beaker on a magnetic stirrer, and stir the sample.

17.1.5 Calibrate the pH meter using the 4.0 and 9.0 buffer solutions.

17.1.6 Fill the 100-mL buret with 1.0 N hydrochloric acid.

17.1.7 Insert the pH meter probe into the sample. Begin titrating the sample with 1.0 N hydrochloric acid. Add the hydrochloric acid at a rapid drop rate to obtain a pH indication of 9 or slightly less.

Note 26—A suitable rapid drop rate is approximately 12 mL/min.

17.1.8 After reaching a momentary pH of 9, decrease the rate of titration to approximately 2 mL/min. Continue at this rate until the pH is close to 8.5.

17.1.9 Once the pH is close to 8.5, add acid in 4-drop increments until maintaining a pH of 8.3 or less for approximately 35 sec.

17.1.10 Then add 2-drop increments until the endpoint of 8.3 or slightly less persists for 60 sec.

Note 27—The endpoint for the titration to pH 8.3 occurs when the addition of one 2-drop increment of titrant produces a pH of 8.3 or slightly less for exactly 60 sec.

17.1.11 Record the volume of titrant used to reach the endpoint.

17.1.12 Add excess hydrochloric acid to the sample. Record the total volume of acid added to the sample.

Note 28—The sample mixture contains a “measured excess” of hydrochloric acid.

17.2 *Back-Titration to pH 4.4:*

17.2.1 Fill a 50-mL buret with 1.0 N sodium hydroxide solution.

17.2.2 Titrate the sample with 1.0 N sodium hydroxide to the endpoint. The endpoint occurs when a pH of 4.4 or slightly greater occurs exactly 10 sec. after a 1-drop addition of titrant.

17.2.3 Record the volume of sodium hydroxide used to reach the endpoint.

17.3 *Loss on Ignition:*

17.3.1 Weigh a crucible using an analytical balance, record the weight, and tare the balance.

17.3.2 Determine the required slurry sample size from Section 16.3. Weigh that amount into the tared crucible. Record the weight of the sample to the nearest 0.0001 g.

17.3.3 Place the crucible with slurry into a 212 ± 4°F (100 ± 2°C) drying oven for 1 hr.

17.3.4 Remove sample from oven and place in a muffle furnace at 2,000 ± 20°F (1093 ± 11°C) for a minimum of 2 hr.

17.3.5 Use the long tongs to remove the crucible from the muffle furnace. Place the crucible on a hard-surfaced asbestos board. Allow the crucible to cool on the board for 10–30 sec.

17.3.6 Transfer the crucible to a vacuum desiccator for cooling. Allow 20 min. for cooling.

Note 29—If running several samples simultaneously, transfer the samples to the desiccator and replace the lid with the vacuum stopcock open. The heated air will expand through the opening for several seconds. Then close the stopcock.

17.3.7 After the sample has cooled, remove the sample from the desiccator. Weigh the crucible on an analytical balance as rapidly as possible to minimize hydration of residue. Record the weight to the nearest 0.0001 g.

17.4 *Sieve Analysis:*

17.4.1 Weigh a 1000-mL glass beaker on a top-loading balance, record the weight to the nearest 0.1 g, and tare the balance.

17.4.2 Pour 1 L of the well-mixed slurry into the beaker. Weigh the beaker with the slurry on the balance and record the weight to the nearest 0.1 g.

17.4.3 Weigh a No. 6 and a No. 30 sieve on a top loading balance. Record each weight to the nearest 0.1 g.

17.4.4 Wash the slurry through the No. 6 and No. 30 sieves simultaneously using regular tap water.

Note 30—Apply the wash water in a fantail spray pattern from the 3/8-in. ID plastic tubing fitted with a Bunsen “fan-type” burner attachment at the outlet. Monitor the water pressure with a water gauge at 12 ± 0.3 psi.

17.4.5 Continue washing the sample for 30 min. or until the water coming through the sieve is clear, whichever occurs first.

17.4.6 Oven-dry the residue on each sieve for 1 hr. at 212 ± 4°F (100 ± 2°C).

17.4.7 Remove the sieves from the oven and allow them to cool for 20 min. Weigh each sieve on a top loading balance. Record the weight to the nearest 0.1 g.

18. CALCULATIONS

18.1 *Percent Solids:*

18.1.1 Calculate all factors A through H and J. Then use the following equation to calculate percent solids:

$$\%\ \text{Solids} = \frac{100 - J}{100}$$

Where:

J = free water of the sample, %.

18.1.2 Calculate A, apparent hydrate alkalinity of the sample, calculated as percent calcium oxide:

$$A = \frac{2.8054(\text{mL of HCl to } 8.3)}{\text{sample wt.}}$$

Where:

mL of HCl to 8.3 = amount of hydrochloric acid used to reach 8.3 pH endpoint, mL

sample wt. = the weight of the sample used in the titration.

18.1.3 Calculate B, carbonate alkalinity of the sample derived by titration and calculated as percent calcium oxide:

$$B = C - A = (total\ alkalinity) - (apparent\ hydrate\ alkalinity, CaO)$$

Where:

C = total alkalinity of the sample calculated as percent calcium oxide

A = apparent alkalinity of the sample indicated by titration calculated as percent calcium oxide.

18.1.4 Calculate C, total alkalinity of the sample calculated as percent calcium oxide:

$$C = \frac{2.8054\ (\text{total ml of HCl - ml of NaOH to } 4.4)}{\text{sample wt.}}$$

Where:

total mL of HCl = amount of hydrochloric acid used, including the “measured excess,” mL

mL of NaOH to 4.4 = amount of sodium hydroxide used to reach 4.4 pH endpoint, mL

sample wt. = titration sample weight, g.

18.1.5 Calculate *D*, apparent hydrate alkalinity indicated by titration of the sample expressed as percent calcium hydroxide:

$$D = (1.32126)A = (1.32126)(apparent\ hydrate\ alkalinity, CaO)$$

Where:

A = apparent hydrate alkalinity of the sample calculated as percent calcium oxide.

18.1.6 Calculate *E*, carbonate alkalinity of the sample calculated as percent carbon dioxide:

$$E = (0.78479)B = (0.78479)(cabonate\ alkalinity, CaO)$$

Where:

B = carbonate alkalinity of the sample derived by titration and calculated as percent calcium oxide.

18.1.7 Calculate F, loss on ignition:

$$\mathrm{F} = \frac{\text{(sample wt. - residue wt.) 100}}{\text{sample wt.}}$$

Where:

sample wt. = original weight of the loss on ignition sample, g

residue wt. = weight of the residue after the loss on ignition test, g.

18.1.8 Calculate *G*, total water of the sample, expressed in percent:

$$G = F - E = (loss\ on\ ignition) - (carbonate\ alkalinity, CO_2)$$

Where:

F = loss on ignition

E = carbonate alkalinity of the sample calculated as percent carbon dioxide.

18.1.9 Calculate *H*, apparent hydrate alkalinity of the sample indicated by the total water portion of the loss on ignition and expressed as percent calcium hydroxide:

$$H = (4.1128)G = (4.1128)(total\ water)$$

Where:

G = total water of the sample, %.

18.1.10 Calculate *J*, free water of the sample, %:

$$J = 0.24314\left(H - D\right)$$

$$J = \left(0.24314\right)\left(apparent\ hydrate\ alkalinity, total\ water - apparent\ hydrate\ alkalinity, titration\right)$$

Where:

H = apparent hydrate alkalinity indicated by the total water portion of the loss on ignition

D = apparent hydrate alkalinity indicated by titration of the sample expressed as percent calcium hydroxide.

18.2 *Lime Slurry Alkalinity, % $Ca(OH)_2$:*

$$\%\,\mathrm{Ca(OH)_2} = \frac{\mathrm{D}}{\%\ \mathrm{solids}} = \frac{\left(\text{apparent hydrate alkalinity, titration}\right)}{\%\ \text{solids}}$$

Where:

D = apparent hydrate alkalinity of the sample indicated by titration and expressed as percent calcium hydroxide.

Note 31—D is the same value calculated for the percent solids determination.

18.3 *Loss on Ignition:*

$$\mathrm{LOI} = \frac{\left(\text{sample wt. - residue wt.}\right)100}{\text{sample wt.}}$$

Where:

sample wt. = original weight of the loss on ignition sample, g

residue wt. = weight of the residue after the loss on ignition test, g.

Note 32—This is the same calculation used for factor F in the percent solids determination.

18.4 *Sieve Analysis:*

$$\%\ \text{retained} = \frac{\text{residue retained}}{(\text{sample wt.})(\%\ \text{solids})} \times 100$$

Where:

% retained = weight of residue retained on the sieve, %

residue retained = weight of residue retained on the sieve, g

sample wt. = weight of the sample before being sieved, g

% solids = percent solids as calculated above.

PART IV—TESTING QUICKLIME

19. SCOPE

19.1 This part discusses the method for testing quicklime.

WARNING: Quicklime is extremely hazardous and capable of inflicting severe caustic burns to skin, lung damage, and eye injury. It can cause blindness if handled improperly. Personnel handling, sampling, testing, or working around quicklime should wear proper protective clothing, respirators, and dust-proof goggles to prevent injury.

20. APPARATUS

20.1 *Equipment,* listed under Part II, Section 6.

20.2 *Laboratory grinder.*

20.3 *Mechanical shaker,* with timer, to accommodate 8-in. diameter sieves.

20.4 *Mixer,* with 1-gal. capacity mixing bowl and type D wire whip attachment.

20.5 *Sample splitter,* (alternate slot type), *two catch pans of appropriate size, and one pour pan.*

20.6 *Erlenmeyer flasks,* 500 mL.

20.7 *Rubber stoppers,* No. 10 size.

20.8 *Ointment type cans,* 6 oz., with lids.

21. REAGENTS

21.1 Use chemical reagents listed for the testing of bulk hydrate or powdered lime in Part III, Section 7, of this method.

22. PREPARING SOLUTIONS

22.1 Use chemical solutions listed for the testing of bulk hydrate or powdered lime in Section 8 of this procedure with the following addition:

22.1.1 *Sugar Solution:*

22.1.1.1 Prepare a fresh sugar solution for each lime slurry sample.

22.1.1.2 In a 200-mL beaker, add 60 g of granulated cane sugar and 120 mL of deionized or distilled water. Add a stirring bar and place the beaker on a magnetic stirrer. Stir until all of the sugar dissolves.

22.1.1.3 Add one drop of phenolphthalein indicator.

22.1.1.4 Add 1.0 N sodium hydroxide, drop by drop, until observing a faint pink color.

23. SAMPLE PREPARATION

23.1 Divide the 1-gal. sample into two portions through the splitter.

23.2 Return and set aside one of the portions to the 1-gal. can.

23.3 Measure 300 g of the remaining portion.

23.4 Grind the 300-g portion for chemical analysis to pass a No. 30 sieve. Transfer the ground lime to a sample vial and close the lid tightly.

23.5 Measure 800 g from the remainder of this portion. Set aside this 800-g portion in a sealed container.

Note 33—Protect all portions from contamination. Crush samples that contain flint or other extremely hard material to pass through a 1/8-in. sieve in a jaw-type crusher before grinding.

24. TESTING PROCEDURES

24.1 *Titration to Phenolphthalein Endpoint:*

24.1.1 Boil 60 mL of deionized or distilled water. Add 10 mL of boiled water to a 500-mL Erlenmeyer flask.

24.1.2 Weigh 2.8 ± 0.3 g of the ground quicklime sample in a weighing boat. Record the exact amount of quicklime to the nearest 0.0001 g.

24.1.3 Transfer the lime sample to the Erlenmeyer flask.

24.1.4 Add 50 mL of boiled water to the Erlenmeyer flask.

24.1.5 Stopper the flask with a No. 10 rubber stopper and swirl for a few seconds.

24.1.6 Place the sample on a hot plate and boil for 2 min.

24.1.7 Remove the sample from the hot plate and allow it to cool to room temperature.

24.1.8 Add 150 mL of the sugar solution to the flask, stopper the flask, and let it stand for 15 min. Swirl the flask every 5 min.

24.1.9 Add five drops of phenolphthalein indicator solution and a stirring magnet to the sample. Stir the sample on a magnetic stir.

24.1.10 Fill a 100-mL buret with 1.0 N hydrochloric acid. Titrate the sample with the hydrochloric acid until the first instance the pink color disappears. This is the endpoint.

24.1.11 Record the volume of hydrochloric acid used to reach the endpoint. Use the calculations to determine the percent active CaO to the nearest 0.1%.

24.2 *Dry Sieve Analysis:*

24.2.1 Weigh the previously reserved 800-g portion on a top loading balance to the nearest 0.1 g.

24.2.2 Shake the 800-g portion through a nest of sieves into a catch pan in 200-g increments. The nest of sieves includes the 1 in., 3/4 in., No. 6, and No. 100.

Note 34—Perform this operation under a hood, while wearing a dust/mist mask.

24.2.3 Weigh the amount caught in each sieve to the nearest 0.1 g, and then recombine them for the wet sieve analysis.

24.3 *Wet Sieve Analysis:*

24.3.1 Add 2600 mL tap water to the mixer bowl, return to the mixer, and attach the wire whip.

24.3.2 Weigh 800 g of quicklime, in increments of 80 g, into ten tin containers.

24.3.3 Lower the wire whip, turn on the mixer, and add one of the increments. Lower the shield. Let stir for 3 min.

24.3.4 Continue adding the 80-g increments every 3 min. until 10 min. have lapsed. At 10 min., stop the mixer for 30 sec. and record the temperature of the slurry. After 30 sec., restart the mixer and mix the slurry for 2 min. to complete the 3 min. cycle of the last added increment.

24.3.5 Add the next increment. Continue the cycle of adding the 80-g increments every 3 min. and stopping every 10 min. for 30 sec. to read the temperature, until all the quicklime is mixed.

24.3.6 When finished, take the bowl out of the mixer and rinse the wire whip.

24.3.7 Weigh the No. 6 sieve on a top loading balance. Record the weight of the sieve to the nearest 0.1 g.

24.3.8 Using tap water, wash the reacted sample onto a No. 6 sieve. Ensure that none of the slurry sample is lost over the sides of the sieve. Use a water pressure gauge to maintain 14–18 psi water pressure.

Note 35—Apply the wash water in a fantail spray pattern from the 3/8 in. ID plastic tubing fitted with a Bunsen “fan-type” burner attachment at the outlet.

24.3.9 Continue to wash the sample for 30 min. or until the wash water is clear, whichever occurs first.

24.3.10 After washing, place the sieve in a drying oven at a temperature of 212 ± 4°F (100 ± 2°C) for 1 hr.

24.3.11 After drying, cool the sieve to room temperature and weigh it on a top loading balance to the nearest 0.1 g.

25. CALCULATIONS

25.1 Calculate Unhydrated Lime Content, % CaO:

$$\% \text{ CaO} = \frac{2.804 \text{ V N}}{\text{sample wt.}}$$

Where:

V = volume of hydrochloric acid, mL

N = normality of hydrochloric acid

sample wt. = weight of quicklime used for titration, g.

25.2 Calculate the percent of the quicklime retained on each sieve. This calculation applies to both wet and dry sieve analysis. Report values to the nearest whole percent:

$$\% \text{ retained} = \frac{\text{residue retained}}{\text{quicklime sample wt.}} \times 100$$

Where:

% retained = weight percent of residue retained on the sieve

residue retained = weight of residue retained on the sieve, g

quicklime sample wt. = weight of the quicklime sample being sieved, g.

PART V—TESTING CARBIDE LIME SLURRY

26. SCOPE

26.1 This part discusses the method for testing high-calcium type carbide lime slurry and assumes that the principal constituents present in the lime are:

- calcium hydroxide,
- water,
- calcium carbonate, and
- inert matter such as silica dioxide.

27. APPARATUS

27.1 *Equipment,* listed under Part II, Section 6.

27.2 *Eyedropper,* large bore polyethylene.

27.3 *Weight-per-gallon cup,* stainless steel, with tare weight.

27.4 *Polyethylene bottles with screw caps,* 1 pt.

27.5 *Glass rod.*

27.6 *Beaker,* 2000 mL.

28. REAGENTS AND SOULTIONS

28.1 Use chemical reagents and solutions listed for the testing of bulk hydrate or powdered lime in Part III, Sections 7 and 8, of this method.

29. SAMPLE PREPARATION

29.1 *Carbide Lime Slurry Sample Preparation:*

29.1.1 Set the slurry jug in a roller and agitate for 3–4 hr.

29.1.2 Use a glass rod to determine if the lime sample is in suspension. If any lime remains caked on the bottom or sides, place the slurry jug back on the roller and agitate.

29.1.3 When the slurry is well mixed, pour a representative sample in a 1-pt. polyethylene bottle with a screw top.

Note 36—Clean the threaded portion of the bottles and the inside threads of the cap. If slurry remains on these places, the seal will not be airtight and the slurry may dehydrate.

29.2 *Determining Bulk Density:*

29.2.1 Weigh a clean and empty weight-per-gallon cup on a top loading balance. Tare the weight of the cup.

29.2.2 Shake the 1-pt. bottle of slurry vigorously for 1 min. Rapidly pour the slurry from the 1-pt. bottle into the weight-per-gallon cup Fill the cup to just below the top rim.

29.2.3 Tap the bottom of the filled cup against a hard surface to remove air bubbles.

29.2.4 Place the lid on the cup, gently pressing it into place. Excess slurry will flow out the small center opening in the top.

Note 37—If slurry does not flow out of the opening, use additional slurry. Agitate the sample before adding more slurry.

29.2.5 Carefully wipe off all of the excess slurry from the outside of the cup and lid.

29.2.6 Place the weight-per-gallon cup on a top loader balance. Weigh the cup and its contents to the nearest 0.1 g.

Note 38—Make sure the tared weight remains displayed on the balance just before measuring the weight of the filled cup.

29.2.7 Divide the result by 10. Report this value as the bulk density of the slurry.

29.3 *Calculations:*

29.3.1 Calculate bulk density:

$$BD = \frac{(\text{slurry wt.})}{10}$$

Where:
BD = bulk density, lb./gal.
slurry wt. = weight of the slurry in the weight-per-gallon cup, g.

29.4 *Determination of Sample Size:*

29.4.1 Refer to Part III, Section 16.3.

30. TESTING PROCEDURES

30.1 *Titration to pH 8.3:*

30.1.1 Determine the sample size using the method indicated in Section 29.4.

30.1.2 Tare a 400-mL tall-form beaker on an analytical balance.

30.1.3 Agitate the 1-pt. bottle for 1 min. Pour the required weight of slurry sample, as determined above, into the 400-mL tall-form beaker. Record the weight of the sample to the nearest 0.0001 g.

30.1.4 Slowly add enough deionized water to bring the volume of mixture to 150 mL. Add a stirring magnet, place the beaker on a magnetic stirrer, and stir the sample.

30.1.5 Calibrate the pH meter using the 4.0 and 9.0 buffer solutions.

30.1.6 Fill the 100-mL buret with 1.0 N hydrochloric acid.

30.1.7 Insert the pH meter probe into the sample. Begin titrating the sample with 1.0 N hydrochloric acid. Add the hydrochloric acid at a rapid drop rate to obtain a pH indication of 9 or slightly less.

Note 39—A suitable rapid drop rate is approximately 12 mL/min.

30.1.8 After reaching a momentary pH of 9, decrease the rate of titration to approximately 2 mL/min. Continue at this rate until the pH is close to 8.5.

30.1.9 Once the pH is close to 8.5, add acid in 4-drop increments until maintaining a pH of 8.3 or less for approximately 35 sec.

30.1.10 Then add 2-drop increments until the endpoint of 8.3 or slightly less persists for 60 sec.

Note 40—The endpoint for the titration to pH 8.3 occurs when the addition of one 2-drop increment of titrant produces a pH of 8.3 or slightly less, for exactly 60 sec.

30.1.11 Record the volume of titrant used to reach the endpoint.

30.1.12 Add excess hydrochloric acid to the sample. Record the total volume of acid added to the sample.

Note 41—The sample mixture contains a "measured excess" of hydrochloric acid.

30.2 *Back-Titration to pH 4.4:*

30.2.1 Fill a 50-mL buret with 1.0 N sodium hydroxide solution.

30.2.2 Titrate the sample mixture with 1.0 N sodium hydroxide to the endpoint. The endpoint occurs when a pH of 4.4 or slightly greater occurs exactly 10 sec. after a one-drop addition of titrant.

30.2.3 Record the volume of sodium hydroxide used to reach the endpoint.

30.3 *Loss on Ignition:*

30.3.1 Weigh a crucible using an analytical balance, record the weight, and tare the balance.

30.3.2 Determine the required slurry sample size from Section 28.4. Weigh the determined amount of slurry into the tared crucible. Record the weight of the sample to the nearest 0.0001 g.

30.3.3 Place the platinum crucible with slurry into a 212 ± 4°F (100 ± 2°C) drying oven for 1 hr.

30.3.4 Remove sample from oven and place in a muffle furnace at 2,000 ± 20°F (1093 ± 11°C) for a minimum 2 hr.

30.3.5 Use the long tongs to remove the crucible from the muffle furnace. Place the crucible on a hard-surfaced asbestos board. Allow the crucible to cool on the board for 10–30 sec.

30.3.6 Transfer the crucible to a vacuum desiccator for cooling. Allow 20 min. for cooling.

Note 42—If running several samples simultaneously, transfer the samples to the desiccator and replace the lid with the vacuum stopcock open. The heated air will expand through the opening for several seconds. Then close the stopcock.

30.3.7 After the sample has cooled, remove the sample from the desiccator. Weigh the crucible on an analytical balance as rapidly as possible to minimize hydration of residue. Record the weight to the nearest 0.0001 g.

30.4 *Sieve Analysis:*

30.4.1 Weigh a 2000-mL glass beaker on a top-loading balance, record the weight to the nearest 0.1 g, and tare the balance.

30.4.2 Pour 2 qt. of the well-mixed slurry from the 2-gal. polyethylene bottle into the beaker. Weigh the beaker with the slurry on the balance and record the weight to the nearest 0.1 g.

30.4.3 Weigh a No. 6 and a No. 30 sieve on a top loading balance. Record the weights to the nearest 0.1 g.

30.4.4 Wash the slurry through the nested No. 6 and No. 30 sieves using regular tap water.

Note 43—Apply the wash water in a fantail spray pattern from the 3/8-in. ID plastic tubing fitted with a Bunsen "fan-type" burner attachment at the outlet. Monitor the water pressure with a water gauge at 12 ± 0.3 psi.

30.4.5 Continue washing the sample for 30 min. or until the water coming through the sieve is clear, whichever occurs first.

30.4.6 Oven-dry the residue on each sieve for 1 hr. at 212 ± 4°F (100 ± 2°C).

30.4.7 Remove the sieves from the oven and allow them to cool for 20 min. Weigh each sieve on a top loading balance. Record the weight to the nearest 0.1 g.

31. CALCULATIONS

31.1 *Percent Solids:*

31.1.1 Calculate all factors A through H and J. Then use the following equation to calculate percent solids:

$$\% \text{ Solids} = \frac{100 - J}{100}$$

Where:

J = free water of the sample, %.

31.1.2 Calculate A, apparent hydrate alkalinity of the sample calculated as percent calcium oxide:

$$A = \frac{2.8054(\text{mL of HCl to } 8.3)}{\text{sample wt.}}$$

Where:

mL of HCl to 8.3 = amount of hydrochloric acid used to reach 8.3 pH endpoint, mL

sample wt. = the weight of the sample used in the titration.

31.1.3 Calculate B, carbonate alkalinity of the sample derived by titration and calculated as percent calcium oxide:

$$B = C - A = (\textit{total alkalinity}) - (\textit{apparent hydrate alkalinity}, CaO)$$

Where:

C = total alkalinity of the sample calculated as percent calcium oxide

A = apparent alkalinity of the sample indicated by titration calculated as percent calcium oxide.

31.1.4 Calculate C, total alkalinity of the sample calculated as percent calcium oxide:

$$\text{C} = \frac{\text{2.8054(total mL of HCl - mL of NaOH to 4.4)}}{\text{sample wt.}}$$

Where:

total mL of HCl = amount of hydrochloric acid used, including "measured excess," mL

mL of NaOH to 4.4 = amount of sodium hydroxide in milliliters used to reach 4.4 pH end point

sample wt. = titration sample weight, g.

31.1.5 Calculate *D*, apparent hydrate alkalinity indicated by titration of the sample expressed as percent calcium hydroxide:

$$D = (1.32126)A = (1.32126)(\textit{apparent hydrate alkalinity}, CaO)$$

Where:

A = apparent hydrate alkalinity of the sample calculated as percent calcium oxide.

31.1.6 Calculate *E*, carbonate alkalinity of the sample calculated as percent carbon dioxide:

$$E = (0.78479)B = (0.78479)(\textit{carbonate alkalinity}, CaO)$$

Where:

B = carbonate alkalinity of the sample derived by titration and calculated as percent calcium oxide.

31.1.7 Calculate F, loss on ignition:

$$F = \frac{(\text{sample wt. - residue wt.})100}{\text{sample wt.}}$$

Where:

sample wt. = original weight of the loss on ignition sample, g

residue wt. = weight of the residue after the loss on ignition test, g.

31.1.8 Calculate *G*, total water of the sample expressed in percent:

$$G = F - E = (loss\ on\ ignition) - (carbonate\ alkalinity, CO_2)$$

Where:

F = loss on ignition

E = carbonate alkalinity of the sample calculated as percent carbon dioxide.

31.1.9 Calculate *H*, apparent hydrate alkalinity of the sample indicated by the total water portion of the loss on ignition and expressed as percent calcium hydroxide:

$$H = (4.1128)G = (4.1128)(total\ water)$$

Where:

G = total water of the sample, %.

31.1.10 Calculate *J*, free water of the sample, %:

$$J = 0.24314(H - D)$$

$$J = (0.24314)(apparent\ hydrate\ alkalinity, total\ water - apparent\ hydrate\ alkalinity, titration)$$

Where:

H = apparent hydrate alkalinity indicated by the total water portion of the loss on ignition

D = apparent hydrate alkalinity indicated by titration of the sample expressed as percent calcium hydroxide.

31.2 Calculate carbide lime slurry alkalinity, % $Ca(OH)_2$:

$$\% \ Ca(OH)_2 = \frac{D}{\% \text{ solids}} = \frac{\text{(apparent hydrate alkalinity, titration)}}{\% \text{ solids}}$$

Where:

D = apparent hydrate alkalinity of the sample indicated by titration and expressed as percent calcium hydroxide.

Note 44—D is the same value calculated for the percent solids determination.

31.3 Calculate loss on ignition:

$$\text{LOI} = \frac{\text{(sample wt. - residue wt.) 100}}{\text{sample wt.}}$$

Where:

sample wt. = original weight of the loss on ignition sample, g

residue wt. = weight of the residue after the loss on ignition test, g.

Note 45—This is the same calculation used for factor F in the percent solids determination.

31.4 Calculate residue retained by sieve analysis, %:

$$\% \text{ retained} = \frac{\text{residue retained}}{\text{(sample wt.)(\% solids)}} \times 100$$

Where:

% retained = weight percent of residue retained on the sieve

residue retained = weight of residue retained on the sieve, g

sample wt. = weight of the sample before being sieved, g

% solids = percent solids as calculated above.

32. ARCHIVED VERSIONS

32.1 Archived versions are available.