# SB102 Field Specialist

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# HMAC CERTIFIED SPECIALIST & APPLICANTS FOR CERTIFICATION: RIGHTS AND RESPONSIBILITIES

This agreement (the "Agreement") affirms that, ______________________________, the below Applicant for Certification or the current Certified Specialist (collectively, the "Specialist") seeks to meet or has successfully met the requirements for certification by the Texas Asphalt Pavement Association ("TXAPA"), a/k/a the Hot Mix Asphalt Center (HMAC) as a certified technician. In that connection, the Specialist agrees as follows:

***RIGHTS AND RESPONSIBILITIES -*** HMAC certification includes the exclusive right to perform certified sampling, testing and reporting on Texas Department of Transportation (TxDOT) projects involving Hot Mix Asphalt (HMA) and Soils & Base (SB) in accordance with TxDOT specifications and test procedures (as may be amended by TxDOT from time to time) for the level of certification issued. Specialists are required to perform and report test results with the accuracy and precision required of a certificated HMA or SB Specialist. It is important that the Specialist fully understands the significance of performing these duties in accordance with the certification level received by the Specialist.

Each Specialist is responsible for performing their own independent sampling, testing and reporting in accordance with TxDOT specifications, test procedures and standard operating procedures. These duties must be performed in a diligent and professional manner to produce TxDOT projects of the highest possible quality.

***CONTACT INFORMATION*** - It is the Specialist's responsibility to provide the HMAC with current contact information by logging into www.txhmac.org, Communications from the HMAC will primarily be sent electronically to the most recent contact information provided by the Specialist.

***ANNUAL PROFICIENCY*** - Once certified, Specialists are required to complete annual proficiency testing and the reporting of results to the HMAC. The testing and reporting must be timely and independently performed by the Specialist and, where applicable, in conformance with the requirements of the Specialist's certifications. TxDOT will ship the annual proficiency samples to the Specialist's address of record. (If this address is not current, the Specialist may not timely receive a proficiency sample and his/her certification may be danger of lapsing.)

Failure to submit proficiency test results or to respond to low rating(s) by the appointed deadlines may result in a change of certification status from active to inactive. Specialists whose certifications are inactivated because their annual proficiency testing/reporting is not current or whose certifications have been revoked or inactivated are prohibited from performing the duties associated with all certifications held by that Specialist.

***RECERTIFICATION*** - Certification(s) are valid for three (3) years from the date originally issued, after which the Specialist must be recertified by again passing the requirements for certification. Specialists may seek recertification up to one year prior to the expiration of their certificate(s).

Failure to timely obtain recertification will cause all dependent certifications held by a Specialist to be inactivated. It is the Specialist's responsibility to maintain an active certification(s). (The HMAC will not provide reminders of pending expiration dates.)

***ALLEGATIONS OF MISCONDUCT*** - Allegations of misconduct should be submitted to the HMAC, P.O. Box 149, Buda, TX 78610 and must include the name, address and signature of the individual asserting the allegations as well as a brief description of the allegations.

If the allegations are properly submitted and appear to have merit, the HMAC Steering Committee (the "Committee") the individual asserting misfeasance and the person so accused will be asked to meet in person (but at separate dates/times) with members of the Committee. At the conclusion of the meeting(s), the Committee will issue its determination.

Misconduct generally consists of (i) neglect, (ii) abuse and/or (iii) breach of trust which are generally defined as:

1. Neglect: unintentional deviation(s) from specifications or testing procedures;

2. Abuse: careless or deliberate deviation from specifications or testing procedures; and

3. Breach of Trust: violation of the trust placed in Certified Specialists including, but not limited to, acts such as:

    a. Falsification of or deliberate omission from material records or information; or

    b. Awareness of improprieties in sampling, testing and/or production by others and the failure to timely report those improprieties to the appropriate project supervision.

The Committee may issue written reprimands (private or public) and/or revoke or inactivate a certification (if the Specialist has made a false representation or misstatement to the Committee or to the public or has engaged in misconduct) or take such other actions as the Committee, in its sole discretion, determines to be appropriate with respect to the Specialist's certification(s).

Specialists who do not achieve recertification or whose certification is revoked or inactivated by the Committee may appeal to a separate appeals committee comprised of industry members (the "Appeal Committee"). The exhaustion of this right of appeal to the Appeal Committee is a prerequisite to the exclusive remedy of administrative review by final and binding arbitration in Hays County, Texas, as administered by the American Arbitration Association by a single-member panel. Any and all other claims related in any way to this Agreement are exclusively subject to final and binding arbitration in Hays County, Texas as administered by the American Arbitration Association by a single-member panel.

In consideration for the HMAC certification/recertification process, Specialist hereby waives any and all claims of whatsoever kind or character related, directly or indirectly, to this Agreement, against the TXAPA, HMAC (including, but not limited to, the Committee and the Appeal Committee and their members), that s/he may have (including claims for attorney's fees) and further agrees to save, indemnify and hold TXAPA, the HMAC (including, but not limited to, the Committee and the Appeal Committee and their members) harmless from any claim, action or cause of action arising as a result of, or relating to this Agreement including, but not limited to, Specialist's certification, or any refusal, reprimand, revocation and/or suspension of certification or recertification of Specialist.

**ACKNOWLEGED & AGREED:**

**SPECIALIST:**

______________________________ ______________________

**SIGNATURE** **DATE**

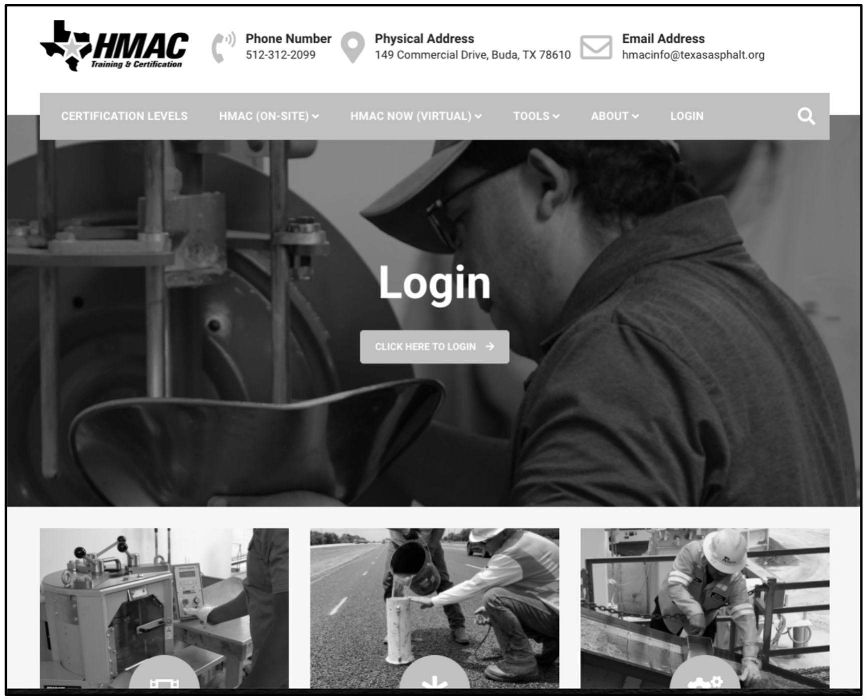
______________________________

**PRINTED NAME**

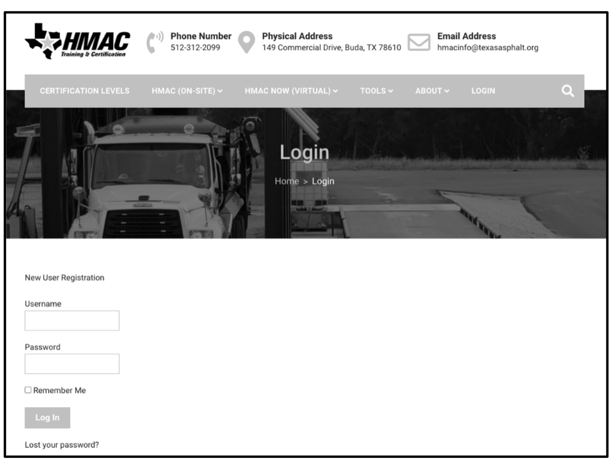
# WEBSITE

www.txhmac.org

**IT IS YOUR RESPONSIBILITY TO KEEP YOUR INFORMATION UP TO DATE**




# FIRST TIME LOGIN



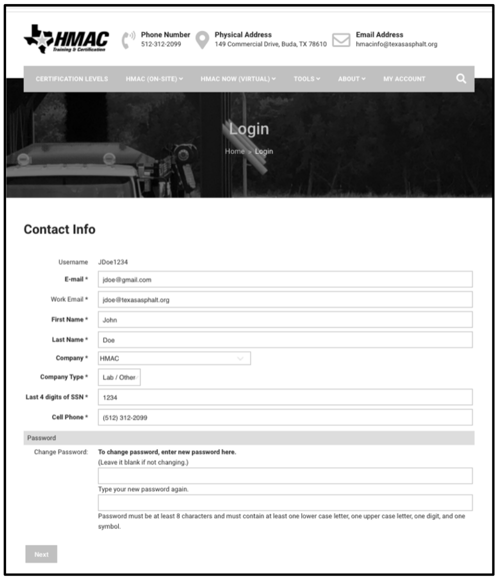

- Username: Capitalize the **FIRST LETTER OF YOUR FIRST AND LAST NAME** then spell out the rest of your last name in lower case, followed by the last 4 digits of your SSN.
- Password: The same but add a "+" at the end.

Example: **John Doe**

Username: **JDoe1234**

Password: **JDoe1234+**

# UPDATE INFORMATION

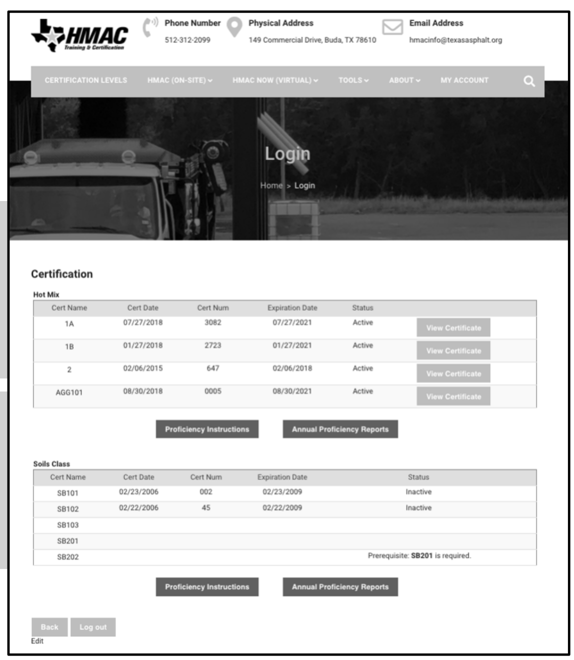



- Verify that all information is correct.

# UPDATE INFORMATION

VIEW/PRINT CERTIFICATIONS

PROFICIENCY



**Test Procedure for**

# SURVEYING AND SAMPLING SOILS FOR HIGHWAYS

## TxDOT Designation: Tex-100-E

***Effective Date: August 1999***

## 1. SCOPE

1.1 This method provides definitions of some basic terms and describes the procedures for surveying and sampling soils for highways. It describes the information required from the survey and the sampling methods required, and it discusses the apparatus necessary to carry out the sampling process.

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.

## 2. DEFINITIONS

2.1 *Aggregate*—Aggregate is granular material of mineral composition such as sand, gravel, shell, slag, crushed stone or lightweight. Aggregate may be used with a cementing medium to form mortars or concrete, or alone in base courses or as a surface treatment.

2.2 *Bank Gravel*— Bank Gravel is found in natural deposits, usually intermixed with fine material, such as sand or clay, or combinations thereof; the terms “gravelly clay,” “gravelly sand,” “clayey gravel,” or “sandy gravel” indicate the varying proportions of the materials in the mixture.

2.3 *Blast Furnace Slag*—Blast furnace slag is a non-metallic by-product, developed in a blast furnace simultaneously with iron in a molten condition, essentially consisting of silicates and alumina-silicates of calcium and other bases.

2.4 *Bottom Ash*—Bottom Ash is the heavy residue from the combustion of ground or powdered coal or lignite.

2.5 *Clay*—Clay is a fine grained soil that can be made to exhibit plasticity (putty-like properties) within a range of water contents and that exhibits considerable strength when air dry.

2.6 *Clay Size*—Clay size refers to any material finer than 0.002 mm (2 µm), though not necessarily exhibiting clay characteristics.

2.7 *Coarse Aggregate*—Coarse aggregate is the portion of aggregate retained on the 2.00 mm (No. 10) sieve for Bituminous Concrete or retained on the 4.75 mm (No. 4) sieve for Portland Cement Concrete.

2.8 *Concrete*—Concrete is a composite material consisting of a binding medium within which are embedded particles or fragments of aggregate; in hydraulic cement concrete, the binder is formed from a mixture of hydraulic cement and water.

2.9 *Conglomerate*—Conglomerate is the coarse grained, clastic, sedimentary accumulation of particles, composed of rounded to sub-angular fragments larger than 2 mm (0.08 in.) in diameter, set in a fine-grained matrix of sand or silt and commonly cemented by calcium carbonate, iron oxide, silica or hardened clay.

2.10 *Crushed Face*—Crushed face is a fractured surface produced by the mechanical crushing of an aggregate. Crushed aggregate faces are identified by fresh fractures and lack of evidence of weathering.

2.11 *Crushed Gravel*—Crushed gravel is the product resulting from the mechanical crushing of gravel, with substantially all fragments having at least one face resulting from a fracture.

2.12 *Crushed Stone*—Crushed stone is the product excavated from an in-situ deposit of rock, crushed and processed for construction purposes with substantially all faces resulting from the crushing operation.

2.13 *Detrital (Weathered)*—Detrital material consists of particles that have been formed through the disintegration of other particles by erosion or weathering.

2.14 *Fine Aggregate*—Fine aggregate is the portion of the aggregate passing the 2.00 mm (No. 10) sieve for Bituminous Concrete or passing the 4.75 mm (No. 4) sieve for Portland Cement Concrete.

2.15 *Fly Ash*—Fly ash is the finely divided residue from the combustion of ground or powdered coal or lignite that is transported from the firebox through the boiler by flue gases.

2.16 *Granite*—Granite is an igneous rock consisting of quartz and alkali feldspars.

2.17 *Gravel*—Gravel consists of unconsolidated or loose detrital sediment (aggregate resulting from natural disintegration and abrasion of rock) with particle sizes passing the 76.2 mm (3 in.) sieve and retained on the 2.00 mm (No. 10) sieve.

2.18 *Lightweight Aggregate*— Lightweight aggregate consists of expanded shale, clay, or slate, and is produced by the rotary kiln method.

2.19 *Limestone Rock Asphalt*—Limestone rock asphalt is limestone impregnated with naturally occurring asphalt.

2.20 *Lithification*—Lithification is the process of hardening, induration, and compaction of sediments**,** leading to the formation of solid materials.

2.21 *Mineral Filler*—Mineral filler is a fine aggregate or manufactured material used to supply particle sizes where the mix design is deficient in gradation.

2.22 *Quarry*—A quarry is an open surface excavation of minerals or construction materials.

2.23 *Riprap*—Riprap is quarried stone especially selected, graded, and placed to prevent erosion and thereby preserve the shape of a surface, slope, or underlying structure.

2.24 *Rock*—Rock is a mass of solid, naturally occurring material from an in-situ deposit, excluding conglomerates. The formation may or may not be laminated.

2.25 *Sand*—Sand consists of fine aggregate particles that are retained on the 75 µm (No. 200) sieve, either as natural sand resulting from natural disintegration and abrasion of rock, or as manufactured sand, which is produced by the crushing of rock, gravel, slag, etc.

2.26 *Sandstone*—Sandstone is sedimentary rock consisting predominantly of weathered sand-sized particles naturally cemented together.

2.27 *Silt*—Silt is soil passing the 75 µm (No. 200) sieve that is non-plastic or very slightly plastic and that exhibits little or no strength when air dry.

2.28 *Silt Size*—Silt size is any material passing the 75 µm (No. 200) sieve that is coarser than 0.002 mm (2 µm), though not necessarily exhibiting silt characteristics.

2.29 *Slag*—Slag is a non-metallic by-product of the smelting or refining of metals and consists of calcium and alumina-silicates.

2.30 *Soil*—Soil is a superficial, unconsolidated deposit of disintegrated and decomposed rock material produced by surface weathering.

2.31 *Source*—A source is a geographical location of naturally occurring material that can be mined or quarried from the original in-situ deposit. In the case of manufactured or by-product material, it is the location of the plant at which the material is produced.

2.32 *Stone*—Stone consists of crushed**,** angular particles of rock.

2.33 *Traprock*—Traprock consists of various fine-grained, dense, dark colored igneous rocks, typically basalt or diabase; also called “trap.”

2.34 *Virgin Material*—Virgin material is material not previously used in construction.

**Note 1**—The Specification Committee has approved the above definitions.

## 3. APPARATUS

3.1 Many factors, such as the nature of the terrain, the kind of material, the depth of material below the surface, the equipment available, and the use to be made of the survey information, will affect the type and amount of equipment to be used in sampling.

3.2 Small hand tools are satisfactory for sample collection where the materials are at a shallow depth and can be easily dug. However, if the materials are very hard, power equipment may be more economical. The only feasible method of sampling strata located at a considerable depth below the surface is the use of a power drill machine with a core or auger attachment.

3.3 The following equipment should suffice for ordinary conditions:

- *Sample bags and moisture cans* for disturbed samples
- *Materials,* to maintain moisture content, and *boxes* for packing undisturbed cores
- *Power drill rig*, with core and/or auger attachments
- *Metallic tape*, 30 m (100 ft.) long
- *Post hole digger, shovel, prospector's pick, other hand tools*
- *Jackhammer and air compressor*
- *Sample splitter or quartering cloth*
- *Engineer's level and level rod*
- *Stakes*
- *Gasoline burner and pan*
- *Ruler*, 2 m (6 ft.)
- *Soil auger.*

## 4. SOIL SURVEY

4.1 The soil survey is an important part of the engineering survey for the design, location and construction of a highway. The investigation should furnish the following information:

- The extent and location of each type of soil or rock in the subsurface
- The condition of subsoils (moisture and density) upon which embankments will be constructed
- The design of ditches and backslopes in cut sections to prevent slides
- The location and selection of suitable material for fills, sub-grade treatment and backfill adjacent to structures
- The location of local material for base and aggregate
- The need for stabilization of sub-grade, sub-base and base materials
- The supporting values of soils as foundation materials.

## 5. SAMPLING

5.1 A representative sample of disturbed soil consists of a combination of the various particles in exactly the same proportion as they exist in the natural ground, roadway, or pit.

5.1.1 The proper method of obtaining a sample will depend on the place, the quantity of material, the proposed treatment, and tests to be performed in the laboratory.

5.1.2 Unless different types of materials are to be uniformly mixed in certain proportions, samples should contain only materials of like color and texture, and should not be composite of materials apparently different in character.

5.2 It is impossible to obtain a sample from the earth that is entirely undisturbed, because the removal of the surrounding soil releases the pressure from the specimen, which causes a certain amount of disturbance.

5.3 The intent of sampling, however, is to obtain a core of soil from the earth with as little disturbance as possible to the natural density, moisture content, and structural arrangement of the particles.

5.3.1 Such a soil core is satisfactory for all practical purposes and can be classified as an undisturbed sample of soil.

## Test Procedure for

# PREPARING SOIL AND FLEXIBLE BASE MATERIALS FOR TESTING

## TxDOT Designation: Tex-101-E

***Effective Date: January 2010***

## 1. SCOPE

1.1 This method describes three procedures for preparing of soil and flexible base samples for soil constants and particle size analysis, compaction and triaxial, and sieve analysis of road-mixed material.

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.

## 2. DEFINITIONS

2.1 *Soil Binder*—Soil binder is any material passing the 425 µm (No. 40) sieve.

2.2 *Percent Soil Binder*—Percent soil binder is equal to 100 times the ratio of the oven-dry mass of the soil binder to the oven-dry mass of the total.

## 3. APPARATUS

3.1 *Set of standard U.S. sieves*, meeting the requirements of Tex-907-K.

3.2 *Scale*, with a minimum capacity of 36 kg (80 lb.), with a minimum accuracy and readability of 5 g or 0.1% of the test load, whichever is greater.

3.3 *Drying oven*, maintained at 60°C (140°F).

3.4 *Crusher* (optional), which can be adjusted to produce material passing a 2.00 mm (No. 10) sieve.

3.5 *Mechanical pulverizer* (optional).

3.6 *Wedgewood mortar and pestl*e.

3.7 *Scoop.*

3.8 *Small siphon tube* (optional).

3.9 *Sample containers, metal pans, cardboard cartons.*

3.10 *Filter paper*, non-fibrous.

3.11 *Sample splitter, quartering machine, or quartering cloth.*

3.12 *Mechanical mixer* (stirring device).

3.13 *Dispenser cup.*

3.14 *Plaster of Paris molds* (optional).

**Note 1**—Dry the plaster of Paris molds at a temperature not to exceed 60°C (140°F) after forming, and wash and dry after each use.

## 4. MATERIALS

4.1 *Tap water.*

## 5. SAMPLE IDENTIFICATION

5.1 Give each sample an identification number on a suitable card. This card should remain with the sample throughout the processing and testing.

# PART I—PREPARING SAMPLES FOR SOIL CONSTANTS AND PARTICLE SIZE ANALYSIS

## 6. SCOPE

6.1 If only soil constants are desired, use either "Dry Preparation (Method A)" or "Wet Preparation (Method B)." However if particle size analysis or percent soil binder is also desired, use Method A. Furthermore, use Method A when preparing a referee test.

## 7. PREPARING SAMPLES

7.1 *Dry Preparation (Method A)*—to be used for analysis of soil constants, particle size, or percent soil binder, or when preparing a referee test

7.1.1 Select a representative sample according to the appropriate test method (Tex-100-E or Tex-400-A) large enough to yield at least 300 g of soil binder.

7.1.2 Dry the sample in a 60°C (140°F) oven.

7.1.3 Examine the sample by visual inspection or slake small portions in water to determine if the material has any particles larger than 425 µm (No. 40).

7.1.4 If the amount of aggregate larger than 425 µm (No. 40) is easily distinguishable, remove these particles by hand and proceed to Section 7.1.27.

7.1.5 For materials containing a considerable amount of aggregate, separate the fine loose binder from the coarse particles by sieving over a 425 µm (No. 40) sieve.

7.1.6 Set the soil binder passing the sieve aside to recombine with the additional binder obtained from Sections 7.1.8 through 7.1.24.

7.1.7 If desired, slake the total material.

7.1.8 Place the material to be slaked into a pan.

7.1.9 Cover the material completely with water and soak for a minimum of 12 hours, unless Tex-102-E determines a shorter time.

7.1.10 Place the empty 425 µm (No. 40) sieve into a clean pan and pour the liquid from the wet sample through it.

7.1.11 Transfer the wet sample to the sieve in increments not exceeding 450 g.

7.1.12 Pour water over the sieve until the water level is about 12.5 mm (0.5 in.) above the sample on the sieve.

7.1.13 Alternately agitate the sieve up and down and stir the sample by hand.

7.1.14 If the material retained on the sieve contains lumps that have not disintegrated, crumple any that can be broken down between thumb and fingers and wash through the sieve.

7.1.15 After all the soil binder appears to have passed through the sieve, hold the sieve above the pan and wash the retained aggregates clean by pouring a small amount of water over it and letting the water drain into the pan.

7.1.16 Transfer the retained aggregate from the sieve to a clean pan.

7.1.17 Repeat the procedure in Sections 7.1.10 through 7.1.16 until all of the soaked sample has been washed.

7.1.18 Dry the retained aggregate portion of the sample in a 60°C (140°F) oven.

7.1.19 Re-screen over the 425 µm (No. 40) sieve and add the binder passing the sieve to the soil binder obtained in Section 7.1.6.

7.1.20 Weigh the mass of the aggregate and retain for use in Tex-110-E, Part I.

7.1.21 Place the pan containing the soil binder and wash water aside, where it will not be disturbed, until all the soil has settled to the bottom of the pan and the water above the soil is clear.

7.1.22 Decant the water off the soil.

7.1.23 Dry the remaining soil in a 60°C (140°F) oven.

7.1.24 In cases where the materials fail to settle overnight, evaporate the water by placing the sample in a 60°C (140°F) oven until it is dry, or siphon the water on to a plaster of Paris mold lined with filter paper.

7.1.25 When the water has disappeared, place the filter paper with adhering soil in a pan and dry in the oven.

7.1.26 Sweep the dry soil from the filter paper with a stiff brush into the pan of fines.

7.1.27 Break down the dried soil binder with a mortar and pestle or use a suitable mechanical pulverizer with an opening set from 635 to 889 µm (0.025 to 0.035 in.)

7.1.28 If a pulverizer is used, any material still aggregated in lumps larger than 425 µm (No. 40) should be broken down with a mortar and pestle.

7.1.29 Combine all of the soil binder obtained and weigh the mass to the nearest 5 g.

7.1.30 Mix thoroughly to produce a uniform sample of all of the particles.

7.1.31 Add the masses obtained in Sections 7.1.20 and 7.1.29, and record the sum as the Total Dry Mass of the sample.

7.2 *Wet Preparation (Method B)*—to be used for the analysis of soil constants only

7.2.1 Select a representative sample according to Tex-100-E or Tex-400-A.

7.2.2 Make the sample large enough to yield at least 300 g of soil binder.

7.2.3 Place the sample in a clean pan.

7.2.4 Cover the sample completely with clear water.

7.2.5 Soak soils with moderate to high Plasticity Indices (PI) for a minimum of 12 hours, unless Tex-102-E determines a shorter time.

7.2.6 Soak flexible base and low PI materials for a minimum of two hours.

7.2.7 Sieve the wet sample into a clean pan in increments of approximately 450 g over a 2.00 mm (No. 10) sieve to remove large aggregate particles.

7.2.8 Wash the aggregate retained on the sieve with a small amount of water.

7.2.9 Discard the portion of material retained on the sieve.

7.2.10 For soils and base materials with low PI, go to Section 7.2.14.

7.2.11 For soils with moderate to high PI, place the wet material passing the 2.00 mm (No. 10) sieve into the dispersion cup of a mechanical malt mixer.

7.2.12 Do not fill the cup more than half-full.

7.2.13 Mix the material for three to five minutes or until the soil binder is separated.

7.2.14 Pour the material through a 425 µm (No. 40) sieve into a plaster of Paris bowl lined with filter paper.

7.2.15 Vigorously agitate the sieve up and down over the bowl while occasionally stirring the sample by hand to allow as much material as possible to pass the 425 µm (No. 40) sieve.

7.2.16 If the material retained on the sieve contains lumps that have not disintegrated, return to the dispersion cup, and remix and wash through the sieve.

7.2.17 Sieve until at least 95% of the soil binder appears to have passed through the sieve.

7.2.18 Reduce the water content of the material in the plaster of Paris bowl to below the liquid limit.

7.2.19 When the sample can be divided into pie-like wedges, and each wedge can be easily removed, it can be used for testing of soil constants.

7.2.20 If the soil constants are not to be determined immediately, place the material into an airtight container to prevent moisture loss.

## 8. CALCULATION

8.1 Use the following to calculate the percent soil binder:

$$Percent\ Soil\ Binder = 100(W_1/W_T)$$

Where:
$W_1$ = dry mass of soil binder
$W_T$ = dry mass of total sample.

## 9. REPORT

9.1 Report the percent soil binder to the nearest whole percent.

# PART II—PREPARING SAMPLES FOR COMPACTION AND TRIAXIAL TESTS

## 10. SCOPE

10.1 Use this part to prepare samples for the compaction and triaxial tests. This procedure applies to all materials, except stabilized material, in the roadway or stockpile condition.

## 11. PREPARING SAMPLES

11.1 Select approximately a 90 kg (200 lb.) representative sample according to Tex-100-E or Tex-400-A.

11.2 Check specifications for maximum aggregate size.

11.3 Spread sample on a clean floor to air dry or use a forced draft of warm air not to exceed 140°F (60°C) for soils and 230°F (110°C) for flexible base material.

11.4 Dry soils in accordance with Section 11.4.1 and flexible base in accordance with Section 11.4.2.

11.4.1 Reduce the water content of soil samples to slightly below the estimated optimum moisture content.

11.4.2 Dry flexible base materials to constant weight. Constant weight will be considered achieved when the weight loss is less than 0.1% of the sample weight in four hours of drying.

11.5 Process soils in accordance with Section 11.5.1 and flexible base in accordance with Section 11.5.2.

11.5.1 Process moist clay and other soils (which form hard lumps when dried or contain aggregates) so that it will pass a 6.3 mm (1/4 in.) wire-mesh.

11.5.2 Separate flexible base by dry sieving into the following sizes:

- 1 3/4 in. (44.5 mm)
- 1 1/4 in. (31.7 mm)
- 7/8 in. (22.2 mm)
- 5/8 in. (16 mm)
- 3/8 in. (9.5 mm)
- No. 4 (4.75 mm)
- No. 40 (0.425 mm).

**Note 2**—Do not overload the screens. The material passing the No. 4 and retained on the No. 40 sieve may need to be shaken separately and in several small batches to avoid overloading the screen.

11.5.3 When material contains aggregate retained on the 44.5 mm (1-3/4 in.) sieve, add the material passing the 1-3/4 in. (44.5 mm) sieve and retained on the 1-1/4 in. (31.7 mm) sieve for recombining individual specimens.

**Note 3**—Do not use particles larger than 1-3/4 in. (44.5 mm) in the compacted specimens.

11.6 When aggregate between 1- 3/4 in. (44.5 mm) and 1-1/4 in. (31.7 mm) is needed, crush particles larger than 1-3/4 in. (44.5 mm) or obtain additional material from the project.

**Note 4**—Do not crush the material if it is an uncrushed gravel.

11.7 Mix each size to make moisture as uniform as possible.

11.8 Weigh each size of material to the nearest 0.1 lb (5 g).

11.9 Calculate the cumulative percentages retained on each sieve size as shown under Section 12.

11.10 These values are to be used in recombining the sample for compaction specimens.

## 12. CALCULATION

12.1 Calculate the cumulative percentages retained on each sieve.

$$Percent\ Retained = 100(Mass\ Retained / Total\ Mass\ of\ Sample)$$

# PART III—SIEVE ANALYSIS OF ROAD-MIX STABILIZED MATERIAL

## 13. SCOPE

13.1 This procedure applies to chemically stabilized materials sampled from the roadway during construction. These materials may be tested in the roadway condition for adequate pulverization, using sieves required by the governing Department Standard Specifications.

## 14. PROCEDURE

14.1 Select a representative sample from the roadway according to Tex-100-E.

14.2 Reduce the sample, using a sample splitter or other approved method, to a minimum of 4.5 kg (10 lb.)

14.3 Remove all non-slakable aggregates retained on the specified sieves.

14.4 Weigh the sample to the nearest 5 g and record as the Total Mass under Section 15.

14.5 Use the specified sieves to separate the sample into different size fractions.

14.6 Use either a sieve shaker or the hand method in the sieving operation.

14.7 Sieve the sample with a lateral and vertical motion of the sieves, accompanied by a jarring action to keep the material moving over the surface of the sieve.

14.8 Continue the sieving operation until no more than one percent of the test sample, by mass, passes through any sieve after one minute of continuous shaking.

14.9 Weigh the material passing each of the required sieves and record its mass to the nearest 5 g.

## 15. CALCULATION

15.1 Use the following to determine percent material passing each sieve.

$$Percent\ Passing = 100(Mass\ Passing\ a\ Sieve / Total\ Mass\ of\ Sample)$$

## 16. REPORT

16.1 Report test results to the nearest whole percent.

## 17. ARCHIVED VERSIONS

17.1 Archived versions are available.

# Tex-101-E Part III, Sieve Analysis of Road-Mix Stabilized Material

| Sieve Size | Weight Retained | Weight Passing | % Passing | Specification | |
|---|---|---|---|---|---|
| 1 ¾” | | | | | |
| ¾” | | | | | |
| #4 | | | | | |

**Calculation**

% Passing = 100 (Mass Passing a Sieve / Total Mass of Sample)

**Test Procedure for**

# DETERMINING MOISTURE CONTENT IN SOIL MATERIALS

## TxDOT Designation: Tex-103-E

***Effective Date: August 1999***

## 1. SCOPE

1.1 This method determines the moisture (water) content of soil, rock, and soil-aggregate mixtures, expressed as a percentage of the mass, by means of either a conventional oven or a microwave oven.

1.2 This method does not give true representative results for materials containing significant amounts of holloysite, montimorillonite, or gypsum minerals, highly organic soils, or materials in which the pore water contains dissolved solids (such as salt in case of marine deposits). For the above named materials, establish a modified method of testing or data calculation to give results consistent with the purpose of the test.

1.3 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.

## 2. DEFINITIONS

2.1 *Water Content*—Water content of a material is equal to the ratio of the mass of “pore” or “free” water in a given mass of material to the mass of the solid particles in the same mass of material, expressed as a percentage.

2.2 *Heat Sink*—Heat sink is a solid or liquid placed in the microwave oven to absorb energy after the moisture has been driven from a test specimen. The heat sink reduces the possibility of over-heating the specimen.

## 3. APPARATUS

3.1 *Drying oven*, maintained at 110 ± 5°C (230 ± 9°F).

3.2 *Microwave oven*, variable power controls and input power ratings of 700 watts preferred.

3.3 *Balance*

- Class G1 in accordance with Tex-901-K for specimens with a mass of 200 g or less

- Class G2 in accordance with Tex-901-K for specimens with a mass greater than 200 g.

3.4 *Specimen containers for conventional ovens*, with close fitting lids if specimen is less than 200 g.

3.5 *Specimen containers for microwave ovens*, non-metallic, non-absorbent.

3.6 *Container handling apparatus*, such as glove or holder suitable for removing hot containers from the ovens.

3.7 *Desiccator cabinet, or jar*, containing silica gel or anhydrous calcium sulfate.

3.8 *Heat sink*, for microwave ovens.

## 4. PREPARATIONS

4.1 *Preparing Test Specimens:*

4.1.1 Store samples prior to testing in airtight containers at a temperature between 2.8°C (37°F) and 30°C (86°F) and in an area that prevents direct contact with sunlight.

**Note 1**—Determine test specimen selection and mass by the proposed application, type of material, and type of sample. Always select a representative sample in all cases. For determination of water contents in conjunction with other test methods, the method of specimen selection is that which is specified in that method.

4.1.2 Make water content determination as soon as practical after sampling, especially if potentially corrodible containers (thin-walled Shelby tubes, paint cans, etc.) or sample bags are used.

4.1.3 For bulk samples, select the test specimen from the material after thoroughly mixed. The mass of moist material selected should be in accordance with Table 1.

4.2 *Preparing Small Jar Samples:*

4.2.1 Cohesionless Soils:

- Thoroughly mix the material, and then select a test specimen having a mass of moist material in accordance with Table 1.
- Remove about 3 mm (0.12 in.) of material from the exposed periphery of the sample and slice it in half (to check if the material is layered) prior to selecting the test specimen.

4.2.2 Layered soils:

- Select an average portion or individual portions or both, and note which was tested in the report of the results. The mass of moist material selected should not be less than 25 g or if coarse-grained particles are noted, should be according to Table 1.

**Note 2**—In many cases, when working with a small sample containing a relatively large coarse-grained particle, it is appropriate not to include this particle in the test specimen. If a large particle is not included, note this fact in the results.

4.2.3 Using a test specimen smaller than the minimum mass indicated previously requires discretion, though it may be adequate for the purpose of the test. If a specimen has a mass less than the previously indicated value, note this fact in the report of the results.

**Table 1—Minimum Mass of Moist Specimens by Sieve Size**

| Sieve Size Retaining More Than 10% of Sample | Recommended Minimum Mass of Moist Specimen, g |
|---|---|
| 2.00 mm (No. 10) | 100–200 |
| 4.75 mm (No. 4) | 300–500 |
| 19.00 mm (3/4 in.) | 500–1000 |
| 38.10 mm (1 1/2 in.) | 1500–3000 |
| 76.20 mm (3 in.) | 5000–10,000 |

# PART I—CONVENTIONAL OVEN METHOD

## 5. SCOPE

5.1 This part outlines the procedures for determining the moisture (water) content of soil, rock, and soil-aggregate mixtures by using the conventional oven method.

## 6. PROCEDURE

6.1 Select a representative test specimen of the mass designated in Table 1.

6.2 Determine the tare mass of a clean, dry container and lid, and record as $W_C$ under Section 7.

6.3 Place the moist specimen in the container and secure the lid onto the container.

6.4 Determine and record the mass of the container, lid, and moist specimen as $W_1$ under Section 7.

6.5 Remove the lid and place the container with the sample in the drying oven.

6.6 Dry for a minimum of 16 hours or to a constant mass.

6.7 To oven-dry large test specimens, place the material in containers having a large surface area (such as a pan) and break into smaller aggregations.

6.8 After the material has dried to a constant mass, remove the container from the oven and replace the lid firmly.

6.9 Allow the material and container to cool to room temperature or until the container can be handled comfortably with bare hands and the operation of the balance will not be affected by the convection currents.

6.10 Determine the mass of the container, lid, and dry specimen using the same balance as in Section 6.2 and record as $W_2$ under Section 7.

## 7. CALCULATIONS

7.1 Mass of the Water:

$$W_w = W_1 - W_2$$

7.2 Mass of the Solid Particle:

$$W_s = W_2 - W_c$$

7.3 Water Content (%):

$$WC = 100(W_w / W_s)$$

Where:
$W_c$ = mass of container and lid, g
$W_1$ = mass of container, lid, and moist specimen, g
$W_2$ = mass of container, lid, and oven-dried specimen, g.

## 8. REPORT

8.1 Include the following information on the report (data sheet):

- identification of the sample (material) being tested by boring number, sample number, test number, etc.
- water content of the specimen to the nearest 0.1% or 1%, depending on the purpose of the test
- indication of any test specimen having a mass less than the minimum indicated
- indication of any test specimen containing more than one soil type (layered, etc.)
- indication of the method of drying, Part I or Part II
- indication of any material (size and amount) excluded from the test specimen
- time and setting of initial drying period and subsequent incremental drying periods when Part II is used
- initial mass of the test specimen prior to drying, and the mass after the incremental drying periods when Part II is used
- identification of the microwave oven and the drying settings and cycles used when standardized drying is used.

# PART II—MICROWAVE OVEN METHOD

## 9. SCOPE

9.1 This part outlines the procedures for determining the moisture (water) content of soil, rock, and soil-aggregate mixtures by using the microwave oven method.

## 10. HAZARDS

10.1 Handle hot containers with insulated container holder. Some soil types can retain considerable heat, and serious burns can result from improper handling.

10.2 Observe any safety precautions supplied by the manufacturer of the microwave. Pay particular attention to keeping the door sealing gasket and door interlocks clean and in good working condition.

*CAUTION*: Manufacturers of microwave ovens may consider using their products to dry soils to be abusive and to constitute the voiding of warranties. Drying soils containing metallic materials may cause arcing in the oven. Highly organic soils and those containing oils and coal may ignite and burn during drying. Continued operation of the oven after the soil has reached constant weight may also cause damage or premature failure of the unit.

10.3 When first introduced, microwave ovens were reported to affect heart pacemakers, primarily because of the operating frequencies of the two devices. Since that time, pacemakers have been redesigned, and the microwave oven is not regarded as the health hazard it once was; however, it may be advisable to post warnings that a microwave is in use.

*CAUTION:* Highly organic soils and soils containing oil or other contaminates may ignite into flames during microwave drying. Means for smothering flames to prevent operator injury or oven damage should be available during testing. Fumes given off from contaminated soils or wastes may be toxic, and the oven should be vented accordingly.

10.4 Do not use metallic containers in a microwave oven. Arcing and oven damage may result.

10.5 Observe manufacturer's instructions when installing and using the oven.

*CAUTION:* Placement of the test specimens directly on the glass liner tray provided with some ovens is strongly discouraged. Concentrated heating of specimens may shatter the glass tray, possibly causing injury to the user.

10.6 The use of a microwave oven is acceptable in place of a 110 ± 5°C (230 ± 9°F) oven for drying soil specimens. Experience and good judgment should dictate sufficient drying time related to using a microwave oven.

## 11. PROCEDURE

11.1 Determine the tare mass of a clean, dry container and lid, and record as $W_C$ under Section 12.

11.2 Place the soil specimen in the container.

11.3 Replace the lid.

11.4 Determine and record the combined mass as $W_1$ under Section 12.

11.5 Place the soil and container, without lid, in a microwave oven.

11.6 Turn on the microwave oven for 3 minutes.

11.7 Adjust the initial and subsequent drying times if experience with a particular soil type and specimen size indicates that shorter or longer initial drying times can be used without over heating.

- The three-minute initial setting is for a minimum sample mass of 100 g, as indicated in Table 1.
- Smaller samples are not recommended when using the microwave oven because drying may be too rapid for proper control.

11.8 It may be necessary to split the sample into segments and dry them separately to obtain the dry mass of the total sample when very large samples are needed to represent soil containing large gravel particles.

11.9 Most ovens have a variable power setting. For the majority of the soils tested, a setting of “high” should be satisfactory; however, for some soils such a setting may be too severe. The proper setting can be determined only through experience with a particular oven for various soil types and sample sizes.

11.10 The energy output of microwave ovens may decrease with age and usage; therefore, establish power settings and drying times for each oven.

11.11 After set time has elapsed, remove the containers and soil from oven and cool the specimen in the desiccator to allow handling and to prevent damage to the balance.

**Note 3**—If containers with close-fitting lids are used, the desiccator is not necessary.

11.12 Determine and record the combined mass of the container, lid, and oven-dried specimen.

11.13 With a small spatula, knife, or short length of glass rod, carefully mix the soil.

11.14 Return the container and soil to the oven and reheat in the oven for one minute.

11.15 Repeat drying, cooling, and weighing, until the change between two consecutive mass determinations has an insignificant effect on the calculated moisture content. A change of 0.1% or less of the initial wet mass of the soil should be acceptable for most specimens.

11.16 Record the final combined mass as $W_2$ under Section 12.

11.17 Standardize the drying times and number of cycles for each oven when routine testing of similar soils is contemplated.

11.18 Periodically verify standardized drying times and cycles to assure that the results of the final dry mass determination are equivalent to those obtained with the repeated drying, cooling, and weighing procedure.

11.19 Minimize overheating and localized drying of the soil with incremental heating and stirring, thereby achieving results more consistent with those obtained by Part 1. The recommended time increments have been suitable for most specimens having particles smaller than a 4.75 mm (No. 4) sieve and with a mass of approximately 200 g; however, they may not be appropriate for all soils and ovens, and adjustment may be necessary.

11.20 Discard water content specimens after testing and do not use them in any other tests, since they may suffer particle breakdown, chemical changes or losses, melting, or losses of organic constituencies.

## 12. CALCULATIONS

12.1 Use the same calculations as shown in Section 7

## 13. REPORT

13.1 Include the same report information as detailed in Section 8.

# Tex-103-E, Determining Moisture Content in Soil Materials

**Calculation**

Mass of the Water

$$W_W = W_1 - W_2$$

Mass of the Solid Particle:

$$W_S = W_2 - W_C$$

Water Content:

$$WC = (W_W / W_S) \times 100$$

**Where**

| | | |
|---|---|---|
| $W_W$ | = | Mass of the water |
| $W_S$ | = | Mass of the solid particle |
| $WC$ | = | Water content |
| $W_C$ | = | Mass of container and lid |
| $W_1$ | = | Mass of container, lid and moist specimen |
| $W_2$ | = | Mass of container, lid and oven-dried specimen |

Test Procedure for

# FIELD METHOD FOR DETERMINING IN-PLACE DENSITY OF SOILS AND BASE MATERIALS

## TxDOT Designation: Tex-115-E

***Effective Date: August 1999***

## 1. SCOPE

1.1 This method determines the density of untreated and treated soil and granular material compacted in the roadway or in the natural state, as exists in a cut section and borrow source prior to excavation.

1.2 The principal use of the in-place density is to determine the degree of compaction or percentage of the density obtained by the method outlined in Tex-113-E and Tex-114-E.

1.3 The term "soils" used in this procedure includes all base materials, as well as fine grain soils.

1.4 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.

## 2. TEST RECORD FORMS

2.1 Tx115.xlsm, "Nuclear Density and Moisture Determination: Tex-115-E"

# PART I—NUCLEAR GAUGE METHOD

## 3. APPARATUS

3.1 *Nuclear testing gauge,* capable of making density and moisture determinations.

3.2 *Portable reference standard.*

3.3 *Calibration curves,* for the nuclear gauge.

3.4 *Scraper plate and drill rod guide.*

3.5 *Drill rod and driver or hammer.*

3.6 *Shovel, sieve, trowel, or straightedge and miscellaneous hand tools.*

3.7 *Gauge logbook.*

## 4. STANDARDIZING EQUIPMENT

4.1 Standardize equipment to a reference standard at the start of each day's use and when test measurements are suspect.

4.1.1 Set the standardizing block 1.5 m (5 ft.) from any object and 7.62 m (25 ft.) from any other nuclear gauge.

4.1.2 Place the gauge on the standardizing block in the closed (safe) mode and take four one-minute density counts.

4.1.3 Repeat the four one-minute counts for moisture in the backscatter position.

4.1.4 Record in the gauge logbook.

4.1.5 When the nuclear gauge is equipped with electronic circuitry capable of automatically averaging four one-minute density and moisture standard counts simultaneously, place the gauge on the standardizing block in the closed (safe) mode and take the averages on the field form and in the gauge logbook.

**Note 1**—Any field form suitable for use with the gauge is acceptable. For additional gauge operation information not covered in this paragraph, follow instructions given in the manufacturer's manual.

**Note 2**—Each individual count (taken using the appropriate time base) must be within the statistical tolerance of:

$$\pm 1.96 \times (average\ count\ rate)^{1/2}$$

4.1.6 Since some gauges display one or two digits less than the gauge is actually counting, multiply the count on those gauges by either 10 or 100 before calculating the tolerance. Then divide this tolerance by 10 or 100 to determine the statistical tolerance.

**Table 1—Example of Individual Count and Its Statistical Tolerance**

| Count/Time Base | Tolerance |
|---|---|
| 23500 | ± 300 |
| 2350(0) | ± 30 |
| 235(00) | ± 3 |

## 5. PROCEDURE

5.1 Prepare the test area by creating a surface plane free of loose material and deformations that extends laterally not less than 152 mm (6 in.) beyond the gauge housing.

5.2 Fill in minor depressions with sand or native fines. Proper test site preparation is closely related to testing accuracy. Make every effort to smooth the site as much as possible.

5.3 Make a hole using the pin and guide plate.

5.4 Extract the pin by hand or with a pin puller or pipe wrench. The depth of hole should be 51 mm (2 in.) greater than the transmission depth being used. This hole must be as close as possible to 90° from the plane of the testing surface. If the plate is rotated slightly around the pin and the plate does not make contact with the ground, or if it appears that the hole is crooked, make a new hole.

5.5 Place the nuclear gauge on the prepared surface so that the bottom of the gauge seats firmly, in full contact with the soil or base material.

5.6 Insert the rod into the hole to the predetermined depth.

5.7 Adjust the gauge so that the rod is firmly against the side of the hole that is nearest to the source or detector tubes.

5.8 After seating the probe, record the number of readings required for the particular instrument being used.

5.9 Rotate the gauge 90° and repeat. (Rotating gauge 90° is optional.)

## 6. CALCULATIONS

6.1 Determine the wet density and moisture by dividing the field counts by the standard counts and use the appropriate calibration curves.

6.2 On programmable models, read the density and moisture directly.

## 7. REPORTING TEST RESULTS

7.1 Report dry density to the nearest 1 $kg/m^3$ (0.1 $lb./ft.^3$) and moisture to the nearest 0.1%.

**Note 3**—This procedure is intended to be a general guide method.

**Note 4**—Follow the instruction manual furnished with a particular gauge for specific operation of that gauge. This is essential, because several different models and different brands are in standard use by the Department.

**Note 5**—Check nuclear density gauges for density at regular time intervals by taking readings using the limestone and granite blocks located in each district. Variation in readings will indicate a possible problem with the gauge. The test position of the gauge on the block and the location of the blocks should be as near the same as practical each time.

**Note 6**—If moisture results obtained by the nuclear gauge are suspect, use Tex-103-E to determine the correct moisture content of the soil.

**Note 7**—Use of these gauges must be according to all applicable State and Federal rules and regulations, and the terms of the radioactive materials license issued to the Department.

**Note 8**—Chemical composition of the materials tested may affect the test results of the gauge.

**Note 9**—Where the material contains chemically bound water (i.e., gypsum) and other hydrogen atoms, such as in asphalt, iron ore, coal, mica and vegetation, use of nuclear gauges may not provide accurate results.

**Note 10**—When used for trench measurements or near obstructions such as abutments, follow the instructions provided in the gauge manual carefully.

# PART II—SAND-CONE METHOD

## 8. SCOPE

8.1 This method is applicable for soils without appreciable amounts of rock or coarse materials in excess of 38 mm (1.5 in.) in diameter, natural soil deposits, aggregates, soil mixtures, and other similar material, and for undisturbed (or in-situ) soils, provided natural void or pore openings in the soil are small enough to prevent sand used in the test from entering the voids.

8.2 Use this test method to determine the density of compacted soils placed during construction of earth embankments, road fill, and structural backfill. It is often used as a basis of acceptance for soils compacted to a specified density or percentage of a maximum density determined by Tex-113-E and Tex-114-E.

8.3 The soil or other materials being tested should have sufficient cohesion or particle attraction to maintain stable sides on a small hole or excavation, and be firm enough to withstand the minor pressures exerted in digging the hole and placing the apparatus over it, without deforming or sloughing.

**Note 11**—This test method is not suitable for:

- organic, saturated, or highly plastic soils that would deform or compress during the excavation of the test hole,
- soils consisting of unbound granular materials that will not maintain stable sides in the test hole, or
- granular soils having high void ratios.

8.4 The use of this method is generally limited to soils in an unsaturated condition.

8.4.1 It is not recommended for soils that are soft or friable (crumble easily) or in a moisture condition such that water seeps into the hand excavated hole.

8.4.2 The accuracy of this test may be affected for soils that deform easily or that undergo a volume change in the excavated hole from vibration, or from standing or walking near hole during test.

**Note 12**—When testing in soft conditions or in soils near saturation, volume changes may occur in the excavated hole.

## 9. APPARATUS

9.1 *Attachable jar*, or other sand container having a volume capacity in excess of that required to fill the test hole and apparatus during the test.

9.2 *Metal base plate or template*, with a flange, center hole cast or machined to receive the large funnel (cone) of the appliance described above.

9.2.1 The base plate may be larger than the funnel (sand-cone).

9.2.2 The plate will be flat on the bottom and have sufficient thickness or stiffness to be rigid.

9.2.3 Plates with raised edges, ridges, ribs, or other stiffeners or approximately 10–13 mm (0.375–0.5 in.) high may be used.

9.3 *Detachable appliance*, consisting of a cylindrical valve with an orifice approximately 13 mm (0.5 in.) in diameter, attached to a metal funnel and sand container on one end, and a large metal funnel and sand container on the other end.

9.3.1 The valve will have stops to prevent rotating past the completely open or completely closed position.

9.3.2 The appliance should be constructed of metal sufficiently rigid to prevent distortion or volume changes in the cone.

9.3.3 The walls of the cone will form an angle of approximately 60° with the base to allow uniform filling with sand.

9.4 *Balance*, Class G5 in accordance with Tex-901-K with a minimum capacity of 20 kg (50 lb.)

9.5 *Drying equipment*, as specified in Tex-103-E.

9.6 *Calibration container*, of known volume, approximately the same size and allowing sand to fall approximately the same distance as the hole excavated during a field test.

9.7 *Miscellaneous equipment*, including:

- knife
- small pick
- chisel
- small trowel
- screwdriver or spoons
- large nails or spike
- metal straightedge about 51 mm (2 in.) wide and 1.5 times the length of the calibration container
- buckets with lids

- plastic-lined cloth sacks, or other suitable containers
- small paint brush
- calculator
- notebook or test forms, etc.

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## 10. MATERIALS

10.1 *Sand*, clean, dry, and uniform in density and grading, un-cemented, durable, and free flowing.

10.1.1 Use any gradation that has a coefficient of uniformity ($C_U = D_{60} / D_{10}$) less than 2.0, a maximum particle size passing the 2.00 mm (No. 10) sieve and less than 3% by mass passing the 250 µm (No. 60) sieve.

10.1.2 Use uniformly graded sand to prevent segregation during handling, storage, and use.

10.1.3 Sand free of fines and fine sand particles is required to prevent significant bulk-density changes with normal daily changes in atmospheric humidity.

10.1.4 Sand comprised of durable, natural sub-rounded, or rounded particles may not be free flowing, a condition that can cause bridging resulting in inaccurate density determinations.

10.2 In selecting sand from a potential source, make a gradation and five separate bulk-density determinations according to the procedure in Section 12 on each container or bag of sand.

10.2.1 The bulk-density variation between any one determination must not be greater than 1% of the average.

10.2.2 Before using sand in density determinations, it should be dried and then allowed to reach an air-dried state in the general location of use.

10.3 Do not re-use sand without removing contaminants.

**Note 13**—As a general rule, reclaiming sand after testing is not desirable.

10.4 Make bulk-density tests of the sand at least every 14 days, after any significant changes in atmospheric conditions, before reusing, and before use of a new batch from a previously proven supplier.

**Note 14**—Most sands have a tendency to absorb moisture from the atmosphere. A very small amount of absorbed moisture can make a substantial difference in bulk-density. In areas of high humidity, or where the humidity changes frequently, the bulk-density may need to be determined more often than the 14-day maximum interval indicated. The need for more frequent checks can be determined by comparing the results of different bulk-density tests on the same sand made in the same conditions of use over a period.

**Note 15**—Some manufactured (crushed) sands, such as blasting sand, have been successfully used with good reproducibility. Check the reproducibility of test results using singular sand under laboratory-controlled testing situations before selecting an angular sand.

**Note 16**—Many organizations have found it beneficial to store sands in moisture-resistant containers. Sand should be stored in dry areas protected from weather. The use of a lighted bulb or other heat source in or adjacent to the storage containers is also beneficial in areas of high humidity.

## 11. CALIBRATING SAND-CONE APPARATUS

11.1 *Method A:*

11.1.1 Since the mass of the sand contained in the apparatus funnel and base plate is dependent on the bulk density of the sand, if using Method A, it must be repeated whenever the bulk-density of the sand changes.

11.1.2 Fill the apparatus with sand that is dried and conditioned to the state anticipated during use in testing.

11.1.3 Determine the mass of the apparatus filled with sand, kg (lb.)

11.1.4 Place the base plate on a clean, level, plane surface.

11.1.5 Invert the container/apparatus and seat the funnel in the flange center hole in the base plate.

11.1.6 Mark and identify the apparatus and base plate so that the same apparatus and plate can be matched and reseated in the same position during testing.

11.1.7 Open the valve fully until the sand flow stops, making sure the apparatus, base plate, or plane surface is not jarred or vibrated before the valve is closed.

11.1.8 Close the valve sharply, remove the apparatus, and determine the mass of the apparatus and remaining sand.

11.1.9 Calculate the mass of sand used to fill the funnel and base plate as the difference between initial and final mass.

11.1.10 Repeat the procedure a minimum of three times. The maximum variation between any one determination and the average may not exceed 1%.

11.1.11 Use the average of the three determinations for this value in the test calculations.

11.2 *Method B*—(optional):

11.2.1 Determine the mass of sand required to fill the apparatus funnel and base plate according to Sections 11.1.2–11.1.7 of Method A.

11.2.2 Calculate the volume of the funnel and base plate by dividing the bulk-density of the sand (as determined in Section 12) by the mass of sand found in Section 11.1 7 of Method A.

11.2.3 Perform a minimum of three determinations and calculate an average value.

11.2.4 The maximum volume variation between any one determination and the average may not exceed 1%.

11.2.5 Use the average of the values when performing test calculations.

## 12. CALIBRATING DENSITY SAND APPARATUS

12.1 *Method A:*

12.1.1 Fill the apparatus with sand conditioned to the same state anticipated during use.

12.1.2 Determine and record the mass of the calibration container when empty.

12.1.3 When calibration container has the same diameter as the flange hole in the base plate, invert and center the sand filled apparatus and base plate on the calibration container.

12.1.4 Fully open the valve and allow the sand to fill the container. When the sand flow stops, close the valve.

12.1.5 Determine the mass of the apparatus and remaining sand. Calculate the net mass of sand in the calibration container by subtracting the mass of sand contained in the cone and base plate (as determined in Section 11) and record.

12.2 *Method B:*

12.2.1 Fill the apparatus with sand conditioned to the same state anticipated during use.

12.2.2 Determine and record the mass of the calibration container when empty.

12.2.3 Invert and support the apparatus over the calibration container so that the sand falls approximately the same distance and location as in a field test, and fully open valve.

12.2.4 Fill the container until it just overflows and close the valve.

12.2.5 Clean any sand from outside of the container.

12.2.6 Determine the mass of container and sand.

12.2.7 Record the net mass of the sand by subtracting the mass of the empty container.

12.2.8 Perform at least three bulk-density determinations. Any one determination and the average will not exceed 1% in difference.

12.2.9 Repeated determinations not meeting these requirements indicate non-uniform sand density, and the sand source should be reevaluated for suitability.

12.2.10 Use the average value obtained in the test calculations.

## 13. CALCULATIONS

13.1 Calculate the bulk-density of the sand:

$$\rho_1 = M_5 / V_1$$

Where:

$M_5$ = mass of the sand to fill the calibration container, kg (lb.)

$V_1$ = volume of the calibration container, $m^3$ ($ft.^3$)

## 14. PROCEDURE

14.1 Select a location and elevation that is representative of the area to be tested.

14.2 Inspect cone apparatus for damage, free rotation of valve, and properly matched baseplate.

14.3 Fill the cone container with conditioned sand for which the bulk-density has been determined in accordance with Section 12.

14.4 Determine the total mass, $M_2$ as shown under Section 15.

14.5 Prepare the surface of the location to be tested so that it is a level plane.

14.6 Use the base plate as a tool for striking off the surface to a smooth level plane.

14.7 Seat the base plate on the plane surface, making sure there is contact with the ground surface around the edge of the flange center hole.

14.8 In soils where leveling is not successful or surface voids remain, determine the volume horizontally bounded by the funnel, plate, and ground surface by a preliminary test.

14.9 Fill the space with sand from the apparatus.

14.10 Determine the mass of sand used to fill the space.

14.11 Refill the apparatus.

14.12 Determine a new initial mass of apparatus and sand before proceeding with the test.

14.13 After this measurement is completed, carefully brush the sand from the prepared surface.

14.14 Dig the test hole through the center hole in the base plate, being careful to avoid disturbing or deforming the soil that will bound the hole.

14.15 Test hole volumes are to be as large as practical to minimize errors and will in no case be smaller than the volumes indicated in Table 2 for the maximum size of soil particle removed from the test hole.

14.16 The sides of the hole should slope slightly inward and the bottom should be reasonably flat or concave.

14.17 Keep the hole as free as possible of pockets, overhangs, and sharp obtrusions, since these affect the accuracy of the test.

14.18 Soils that are essentially granular require extreme care and may require digging a conical shaped test hole.

14.19 Place all excavated soil, and any soil loosened during digging, in a moisture tight container that is marked to identify the test number.

14.20 Take care to avoid losing any materials.

14.21 Protect this material from any loss of moisture until determining the mass and obtaining a specimen for a water content determination.

14.22 Clean flange of base plate hole, invert sand-cone apparatus, and seat sand-cone funnel into flange hole at the same position as marked during calibration. (See Section 11.)

14.23 Eliminate or minimize vibrations in test area by personnel or equipment.

14.24 Open valve and allow sand to fill hole, funnel, and base plate.

14.25 Take care to avoid jarring or vibrating apparatus while sand is running.

14.26 When sand stops flowing, close the valve.

14.27 Determine mass of apparatus with remaining sand, record, and calculate mass of sand used.

14.28 Determine and record the mass of the moist material removed from the test hole.

14.29 When oversized material corrections are required, determine the mass of the oversized material on the appropriate sieve and record, taking care to avoid moisture losses.

14.30 Mix the material thoroughly, and obtain a representative specimen for determining water content or use the entire sample.

14.31 Determine the water content in accordance with Tex-103-E, Section 7.

**Note 17**—Select water content specimens to represent all material obtained from the test hole, and their minimum mass must provide water content values accurate to 1.0%.

**Table 2—Minimum Test Hole Volumes and Minimum Moisture Content Samples Based on Maximum Size of Particle**

| Maximum Particle Size | Minimum Test Hole Volume, $m^3$ | Minimum Test Hole Volume, $ft.^3$ | Minimum Moisture Content Sample, g |
|---|---|---|---|
| No. 4 Sieve (4.75 mm) | 0.0007 | 0.025 | 100 |
| 1/2 in. (12.5 mm) | 0.0014 | 0.050 | 300 |
| 1 in. (25 mm) | 0.0021 | 0.075 | 500 |
| 2 in. (50 mm) | 0.0028 | 0.100 | 1000 |

## 15. CALCULATIONS

15.1 Calculate the volume of the test hole ($V$):

$$V = (M_1 - M_2) / \rho_1, m^3 (ft^3)$$

Where:

$M_1$ = mass of the sand used to fill the test hole, funnel and base plate, kg (lb.)

$M_2$ = mass of the sand used to fill the funnel and base plate (from calibration of sand-cone apparatus), kg (lb.)

$\rho_1$ = bulk density of the sand, kg/m$^3$ (lb./ft.$^3$)

15.2 Calculate the dry mass of the material ($M_4$) removed from the test hole:

$$M_4 = 10[M_3 / (w + 100)], kg\ (lb.)$$

Where:

$w$ = water content of the material removed from the test hole, %

$M_3$ = moist mass of the material removed from the test hole, kg (lb.)

15.3 Calculate the in-place wet density ($\rho_m$) of the tested material removed from the hole:

$$\rho_m = M_3 / V, kg / m^3 (lb / ft^3)$$

15.4 Calculate the in-place dry density ($\rho_D$) of the tested material removed from the hole:

$$\rho_D = M_4 / V, kg / m^3 (lb / ft^3)$$

15.5 It may be desired to express the in-place density as a percentage of some other density, for example, the laboratory densities. Determine this relationship by dividing the in-place density by the laboratory density and multiplying by 100.

## 16. REPORTING TEST RESULTS

16.1 Report density to nearest 1 kg/m$^3$ (0.1 lb./ft.$^3$) and moisture content to the nearest 0.1%.

# PART III—VOLUMETER METHOD

## 17. APPARATUS

17.1 *Volumeter,* a calibrated metal chamber mounted between a top and bottom assembly.

17.1.1 The base plate has an opening designed to accept a rubber membrane, which fits over the base insert.

17.1.2 The base is fastened to the bottom assembly by means of thumbscrews.

17.1.3 A pump provides pressure to fill the rubber membrane or vacuum to remove the water.

17.1.4 A four-way valve controls these operations.

17.1.5 The compound gauge indicates the kilopascals (pounds) of pressure (or inches of mercury) applied.

17.1.6 A transparent gauge tube and graduated metal tape connected through the upper and lower base assembly measures the quantity of water used, which is the volume of the material removed.

17.1.7 A level is attached to the upper assembly.

17.2 *Equipment,* for digging holes, such as post hole auger, soil auger, density-in-place digging tools.

17.3 *Buckets,* 4 L (1 gal.), with lids, or plastic bags,

17.4 *Drying pans,*

17.5 *Drying oven,* with temperature maintained at 110 ± 5°C (230 ± 2°F),

17.6 *Balance,* Class G2 in accordance with Tex-901-K, with a minimum capacity of 15 kg (33 lb.)

## 18. PREPARING THE VOLUMETER

Use the operating manual furnished with each volumeter for complete description of parts, how to disassemble and reassemble the device, and general information about the care and use of the instrument.

18.1 Depress the pump handle into "hold" position to interlock the control valve and to prevent the water from entering the pump or escaping from the volumeter.

18.2 Invert volumeter and set on top six legs.

18.3 Loosen thumbscrews and remove base plate.

18.4 Carefully center base insert.

18.5 Use clean tap or deionized water and fill the metal chamber to within 6.5 mm (0.25 in.) of the top of the inverted lower base.

18.6 Remove the protective talc from a rubber membrane and fold the open end inside itself to obtain a length approximately 25 mm (1 in.) longer than the depth of hole to measure.

18.7 Examine base quad ring for twists and check surfaces of base and base plate for grit.

18.8 Place base on volumeter with flat side out; align holes and finger tighten all thumbscrews.

18.9 Invert and tilt volumeter so the pump cylinder is on top of the water chamber.

18.10 Lift pump handle from interlock and shift control valve to vacuum position.

18.11 Use short and slow strokes of pump handle to apply vacuum, while using the other hand to assist the rubber membrane to retract and flatten out over base insert.

18.12 Place control valve in "hold" position and lower pump handle into curved notch.

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## 19. PROCEDURE

19.1 Select a density test location that is reasonably smooth and free from loose soil or pebbles.

19.2 Place matching tray flat side down on test site.

19.3 Fasten to surface of compacted soil with spikes through boss holes.

19.4 Zero the volumeter tape before excavating the test hole. This compensates for any irregularity in surface of test site, the temperature of water, the volume of the rubber membrane and the tray thickness.

19.5 Place the volumeter upright on the tray between the three bosses.

19.6 Rotate entire volumeter until the bubble level centers.

19.7 Open memory valve at the bottom of the gauge tube.

19.8 Lift the pump handle, turn the control valve to "open" for a few seconds and then shift to "pressure."

19.9 Place one knee on center of the top of volumeter, hold volumeter firmly down against tray, and apply 20.7 kPa (3 psi) pressure.

19.10 While maintaining this pressure, reach down and close memory valve.

19.11 Retract membrane by turning valve to “vacuum.”

19.12 Pump with short, slow strokes of handle until 25 mm (1 in.) of vacuum registers on gauge.

19.13 Turn control valve to hold position and depress knob on pump handle into curved notch.

19.14 Remove knee.

19.15 Rotate the upper or lower tape spool until the 76.2 lineal mm (0.4 lineal ft.) mark coincides with the top of the meniscus of the water in the adjacent gauge tube. This will automatically compensate for the 0.00113 $m^3$ (0.04 $ft.^3$) in the bulged upper chamber of the 220 model volumeter.

19.16 While holding lower tape spool, securely tighten tape lock nut. Set volumeter aside in upright position.

19.17 Use the various tools available and excavate the soil inside the limit of the hole in center of tray.

19.18 To ensure accuracy of the density determination, dig the hole as large as possible but be sure not to exceed the capacity of the volumeter.

19.18.1 The walls should be as smooth as possible and round into the bottom surface.

19.18.2 Use caution in digging holes in freshly compacted materials so as not to enlarge the hole due to excessive effort applied with digging tools.

19.18.3 Avoid having cavities so deep that the membrane might not fill them, or cave-ins directly under the tray, or considerable variation in diameter of the hole.

19.19 Recover all of the material removed from the hole and seal in a gallon bucket or plastic bag to prevent loss of moisture.

19.20 Measure the volume of the test hole by repeating the operations outlined in Sections 19.4 through 19.17, and record as V under Section 20.

19.21 Weigh the material excavated from the test hole to determine the wet mass.

19.22 Dry the soil at 110°C (230°F), obtain the dry mass, and record as $W_D$, under Section 20.

## 20. CALCULATIONS

20.1 Calculate moisture content in accordance with Tex-103-E.

20.2 Calculate the mass of the water ($W_W$):

$$W_W = W_1 - W_2$$

20.3 Calculate the mass of the solid particles ($W_S$):

$$W_S = W_2 - W_C$$

20.4 Calculate the water content (%) ($WC$):

$$WC = (100 \bullet W_W \ / \ W_S)$$

Where:
$W_C$ = mass of container and lid, g
$W_1$ = mass of container, lid, and moist specimen, g
$W_2$ = mass of container, lid, and oven-dried specimen, g.

20.5 Calculate in-place density:

$$In\ Place\ Density = W_D \ / \ V$$

Where:
$W_D$ = mass of oven-dried soil, kg (lb.)
$V$ = volume of test hole, $kg/m^3$ ($lb./ft.^3$)

**Note 18**—Carefully read operating manual for the volumeter.

**Note 19**—Use only clean, pure water in volumeter and change often to prevent corrosion. Add only antifreeze selected from manufacturer's recommended list.

**Note 20**—When retracting the rubber membrane with the volumeter in a tilted position, always have the cylinder of the pump on top of the metal chamber.

**Note 21**—To obtain accuracy in measuring the volume of the hole, the volumeter must be held solidly against the tray and not be allowed to rise when pressure is applied to rubber membrane.

**Note 22**—Other methods of in-place density tests that correlate satisfactorily with this method for the soil material in question will be satisfactory for use.

## 21. TEST REPORT

21.1 Report density to the nearest 1 $kg/m^3$ (0.1 $lb./ft.^3$) and moisture to the nearest 0.1%.

Test Procedure for

# MEASURING THICKNESS OF PAVEMENT LAYER

## TxDOT Designation: Tex-140-E

***Effective Date: August 1999***

## 1. SCOPE

1.1 This method determines the thickness of a base, subbase, or subgrade when a core cannot be obtained.

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.

## 2. APPARATUS

2.1 *Drill with auger bit, grubbing hoe, or other acceptable tool*.

2.2 *Nail, blade, knife, or other suitable tool*, not to exceed 3 mm (1/8 in.) in thickness, and approximately 75 mm (3 in.) in length.

2.3 *Folding scale*, 2 m (6 ft.), or other scale with 3 mm (1/8 in.) or smaller divisions.

2.4 *Depth measurement indicator*, DHT No. 2238 (not to be used for pay purposes).

## 3. PROCEDURE

3.1 Drill or dig a hole to sufficiently penetrate the layer immediately below the layer being measured. Determine the thickness of the layer after the finished grade (bluetops) has been obtained.

3.2 Make a vertical groove on the side of the hole to remove loose material and expose interface of both layers.

3.3 Locate interface visually. When the interface is clearly defined, push a six-penny nail or blade horizontally into the interface approximately 25 mm (1 in.)

3.4 Measure from the clearly defined interface or top of nail (blade) to the top of the layer being measured to nearest 3 mm (1/8 in.) and record as $t_1$. A straightedge or surveyor's stake may be used to place across top of hole to determine the top of layer.

3.5 Move the nail to another location on the interface and measure the thickness. Record as $t_2$, etc.

3.6 Repeat until obtaining a minimum of three measurements per hole.

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## 4. REPORT

4.1 Report the average of three measurements ($t_1$, $t_2$, and $t_3$) to the nearest 3 mm (1/8 in.) as the thickness of the pavement layer.

**Note 1**—If lime or cement is used as a soil stabilizer in neutral or acid soils, the bottom of this layer may be emphasized by a light application of 1% phenolphthalein indicator solution prepared in accordance with Tex-600-J. This solution should be applied after the vertical groove is made and from the bottom up. Once the interface is identified, application of the solution should stop.

**Note 2**—If depth measurement indicator is used:

- Drive the indicator into soil deeper than the specified requirement.
- Rotate using a pin through the shaft.
- Carefully remove indicator and lay on ground with groove up.
- Identify bottom and top of layer.
- Measure layer thickness.
- Record layer thickness to nearest 3 mm (1/8 in.)

# Tex-140-E, Measuring Thickness of Pavement Layer

T1: ____________________

T2: ____________________

T3: ____________________

Total: ____________________ Reported Average: ______________________

Report to the nearest $^1/_8$”

$^1/_8$” = 0.125

$^1/_4$" = 0.250

$^3/_8$” = 0.375

$^1/_2$" = 0.500

$^5/_8$” = 0.625

$^3/_4$" = 0.750

$^7/_8$” = 0.875

**Test Procedure for**

# SAMPLING AGGREGATE, FLEXIBLE BASE, AND STONE RIPRAP

**TxDOT Designation: Tex-400-A**

***Effective Date: April 2021***

## 1. SCOPE

1.1 Use this test procedure for sampling completed stockpiles of coarse and fine aggregate used to produce concrete and retaining wall select backfill, stockpiles of flexible base, and stone riprap. This procedure may also be used to sample concrete aggregate during production when stored in bays or stockpiles that are not accessible in three directions.

1.2 Use this test procedure to sample completed coarse or fine aggregate stockpiles and materials using a front-end loader or shovel. The preference for sampling is to use a front-end loader. Only use a shovel where a front-end loader is not available.

## 2. SAMPLE SIZE

2.1 Use Table 1 to determine the minimum sample size to perform the required tests listed in the applicable specification.

2.2 Obtain samples that show the true nature and condition of the materials that they represent. Do not combine materials that apparently differ in property or character to make a composite sample. Differences may be indicated by color or texture.

2.3 Use a sample splitter or quartering cloth to reduce the field sample to laboratory test size.

| **Nominal Maximum Size of Aggregate[1]** | **Minimum Weight of Field Samples[2] (lbs.)** |
|---|---|
| No. 8 | 25 |
| No. 4 | 25 |
| 3/8 in. | 25 |
| 1/2 in. | 35 |
| 3/4 in. | 55 |
| 1 in. | 110 |
| 1-1/2 in. | 165 |
| 2 in. | 220 |
| 2-1/2 in. | 275 |
| 3 in. | 330 |
| **Nominal Maximum Size of Flexible Base[1]** | **Minimum Weight of Field Samples[2] (lbs.)** |
| 1-3/4 in. | 400 |

1. The maximum aggregate size is the largest sieve size listed in the applicable specification upon which any material can be retained.
2. The minimum sample mass depends upon the maximum aggregate size as well as the number and type of tests required.

## 3. SAMPLING PROCEDURES

3.1 *This test procedure does not claim to address the safety concerns associated with its use. It is the responsibility of the user of this test procedure to establish the appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

3.2 *Sampling procedure for aggregate from completed stockpiles using a front-end loader.*

3.2.1 Use this procedure to sample completed stockpiles of coarse or fine aggregate using a front-end loader.

3.2.2 Determine the minimum number of samples required for approval based on TxDOT's Guide Schedule frequency for concrete or retaining wall select backfill.

3.2.3 Identify the sampling locations by dividing the stockpile in approximately equal sectors based on the number of samples determined from Section 3.2.2. Aggregate must be sampled from a minimum of two sectors or locations for acceptance.

3.2.4 Clean and level the ground at the sampling location to prevent contamination of the sampling pile.

3.2.5 Cut approximately at the ground level to the top edge of the stockpile until a clean face is exposed. This represents the full height of the stockpile.

**Note 1**-The exposed vertical face should be perpendicular to the top edge of the stockpile, but this may not be possible with stockpiles of dry, coarse aggregates.

3.2.6 Discard the material cut away while exposing the clean face.

3.2.7 Build a sample pad by cutting into the vertical face at the ground level to the full height of the stockpile to obtain material.

3.2.8 Lower the bucket as close as possible to the ground to avoid segregation and empty the entire contents of the bucket in one motion onto the ground.

3.2.9 Using the loader bucket, strike and level the sample pad at mid-height in the direction the bucket was emptied to create a flat surface for sampling. Back-drag the sampling pad only once.

3.2.10 Divide the sample pad into four quadrants of similar size.

3.2.11 Place clean sample bags or containers near the center of the sampling pad and obtain the sample across the flat area staying more than 1 ft. away from the edges.

3.2.12 Sample equal amounts of aggregate evenly across each quadrant. Fully insert the shovel as near as vertical as possible and then slowly roll the shovel back and lift slowly to avoid coarse aggregate rolling off the sides of the shovel.

**Note 2**-Spade-tip shovels are not recommended for sampling. Square-tip shovels work well preventing aggregate from rolling from the side.

3.2.13 Obtain additional shovelfuls from different quadrants of the sampling pad, and in areas avoiding previous shovel holes.

3.2.14 Place the aggregate into the clean sample bags or containers.

3.2.15 Seal and label the sample bags or containers.

3.2.16 Repeat Section 3.2.4 to 3.2.15 for each additional location as determined from Section 3.2.3.

3.3 *Sampling procedure for aggregate from completed stockpiles using a shovel.*

3.3.1 Use this procedure to sample completed stockpiles of coarse or fine aggregate using a shovel where a front-end loader is not available. The preference for sampling is to use a front-end loader.

3.3.2 Determine the minimum number of samples required for approval based on TxDOT's Guide Schedule frequency for concrete or retaining wall select backfill.

3.3.3 Identify the sampling locations by dividing the stockpile in approximately equal sectors based on the number of samples determined from Section 3.3.2. Aggregate must be sampled from a minimum of two sectors or locations for acceptance.

3.3.4 Identify locations within each sector from the top third, at the mid-point, and bottom third of the stockpile. Sampling must be performed in no less than these three increments.

3.3.5 Dig a small trench into the stockpile at each location approximately 1 ft. deep and 3 ft. in diameter.

**Note 3**-A board may be shoved vertically into the stockpile just above the point of sampling to prevent segregation from coarser aggregate rolling down during sampling.

3.3.6 Do not use the aggregate removed from the trenched area as a part of the sample.

3.3.7 Shovel aggregate from the stockpile into clean sample bags or containers from the innermost part of the trench. Minimize larger sized aggregate falling back into the trench.

**Note 4**-When sampling sands, sampling tubes of at least 1-1/4 in. diameter may be used where experience has indicated representative samples cannot be obtained otherwise.

3.3.8 Seal and label the sample bags or containers.

3.3.9 Repeat Section 3.3.4 to 3.3.8 for each additional location as determined from Section 3.3.3.

3.4 *Sampling procedure for aggregate when sampling during concrete production at mixing plant/site.*

3.4.1 Use this procedure to sample coarse or fine aggregate at concrete mixing plants/sites during production from bays or stockpiles that are not accessible in three directions and for Optimized Aggregate Gradation (OAG) concrete. The preference for sampling is to use a front-end loader. Only use a shovel when a front-end loader is not available.

3.4.2 Identify the center of the open face of the aggregate bay.

3.4.3 Sample at this location using a front-end loader according to Section 3.2.

3.4.4 Sample at this location using a shovel when a front-end loader is not available according to Sections 3.3.

3.4.5 Sample at additional locations when the face of the open bay or face of the stockpile is significantly wide. Chose a minimum of two locations approximately equal offset from the center.

3.5 *Sampling Procedure for Flexible Base Stockpiles.*

3.5.1 Identify four locations around the perimeter of the stockpile that represent the approximate quarter-points of the stockpile.

**Note 5**– When the locations cannot be obtained from around the entire perimeter due to limited space, use four equally spaced locations.

3.5.2 Clean and level the ground at these four locations to prevent contamination of the sampling pile.

3.5.3 Sample each quarter-point of the stockpile using a front-end loader to cut into each quarter-point.

3.5.4 Cut at the ground level to the top edge of the stockpile until a clean vertical face is exposed that is perpendicular to the top edge of the stockpile. This represents the full height of the stockpile.

3.5.5 Discard this material cut away while exposing the clean face.

3.5.6 Build a sample pad by cutting into the vertical face at the ground level of the full height of the stockpile to obtain material.

3.5.7 Lower the bucket as close as possible to the ground to avoid segregation and empty the entire contents of the bucket in one motion onto the ground in one motion.

3.5.8 Using the loader bucket, strike and level the sample pad at mid-height in the direction the bucket was emptied to create a flat surface for sampling. Back-drag the sampling pad only once.

3.5.9 If material is visually segregated, discard the material and repeat Sections 3.5.6 to 3.5.8.

3.5.10 Place clean sample bags or containers near the center of the sampling pad and obtain the sample across the flat area staying more than 1 ft. away from the edges.

3.5.11 Divide the sample pad into four quadrants and sample equal amounts of aggregate evenly across each quadrant.

3.5.12 Using a square-tip shovel, fully insert the shovel as near as vertical as possible and then slowly roll the shovel back and lift slowly to avoid coarse aggregate rolling off the sides of the shovel.

**Note 6**-Spade-tip shovels are not allowed for sampling flexible base stockpiles.

3.5.13 Obtain additional shovelfuls from different quadrants of the sampling pad, and in areas avoiding previous shovel holes. Remove material from each quadrant to fill one sample bag or container. Minimize loose material falling from the sides of the hole and loss of material from the shovel into the hole or adjacent ground while filling sample bags or containers.

3.5.14 Place the aggregate into the clean sample bags or containers.

3.5.15 Repeat Sections 3.5.13 to 3.5.15 until a minimum of 100 lbs. of material is sampled from each sample pad.

3.5.16 Seal and label the sample bags or containers, and properly secure for transportation to avoid any loss of material.

3.5.17 Repeat Sections 3.5.4 to 3.5.16 at each of the stockpile quarter-points to provide a minimum of 400 lbs. of sampled flexible base material.

**Note 7**-The minimum amount of 400 lbs. of material is representative of the entire stockpile sampled. This minimum amount of material is required for a testing laboratory to perform all the required test procedures for stockpile approval.

3.6 *Preparation of Sampled Flexible Base for Testing.*

3.6.1 Allow the material to air dry or oven dry at a maximum temperature of 140°F for a minimum of 4 hours until the material is sufficiently dry for handling.

3.6.2 Quarter the material by emptying each container or sample bag onto a clean floor or a clean tarp. Optionally, use a mechanical quartering device or sample splitter to quarter the material in accordance with ASTM C702, Method A. Proceed to Section 3.6.6.

3.6.3 Thoroughly mix the material using a shovel. When using a tarp, the ends of the tarp may also be used to also mix the material.

3.6.4 Spread the material into the shape of a circle of uniform thickness and homogenous with no segregation.

3.6.5 Using a shovel or straightedge, visibly trace lines on top of the material to outline four evenly sized quarters.

3.6.6 Use a quarter of the sample and proceed to Tex-101-E, Part I to prepare and test the material for gradation (Tex-110-E); and the liquid limit (Tex-104-E) and plastic limit (Tex-105-E) tests to determine the plasticity index (Tex-106-E).

3.6.7 Combine the remaining three quarters with any remaining material from Section 3.4.15 and proceed to Tex-101-E, Part II to prepare the material for Moisture-Density curve (Tex-113-E), wet ball mill (Tex-116-E), and compressive strength (Tex-117-E) testing.

3.7 *Sampling Procedure for Stone Riprap.*

3.7.1 Obtain six to eight pieces of the stone riprap that is representative of the size of the riprap.

3.7.2 Crush or break down the entire sample to a maximum particle size of 6 in.

3.7.3 Select approximately 275 lbs. of the riprap and place into sample bags or containers.

3.7.4 Seal and label the sample.

## 4. ARCHIVED VERSIONS

4.1 Archived versions are available.

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 × 0.991 = 18.334
- 18.500 - 18.334 = 0.166
- 0.166 × 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 × 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:

$$Uncorrected Ca(OH)_2 = 94.8$$
$$CaCO_3 = 3.1$$
$$Assumed inert = 2.2$$
$$\overline{100.1}$$

$$Subtotal 100.1 - 100 = 0.1$$
$$\%CaCO = 0.1x3.11 = 0.3$$

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

- 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.

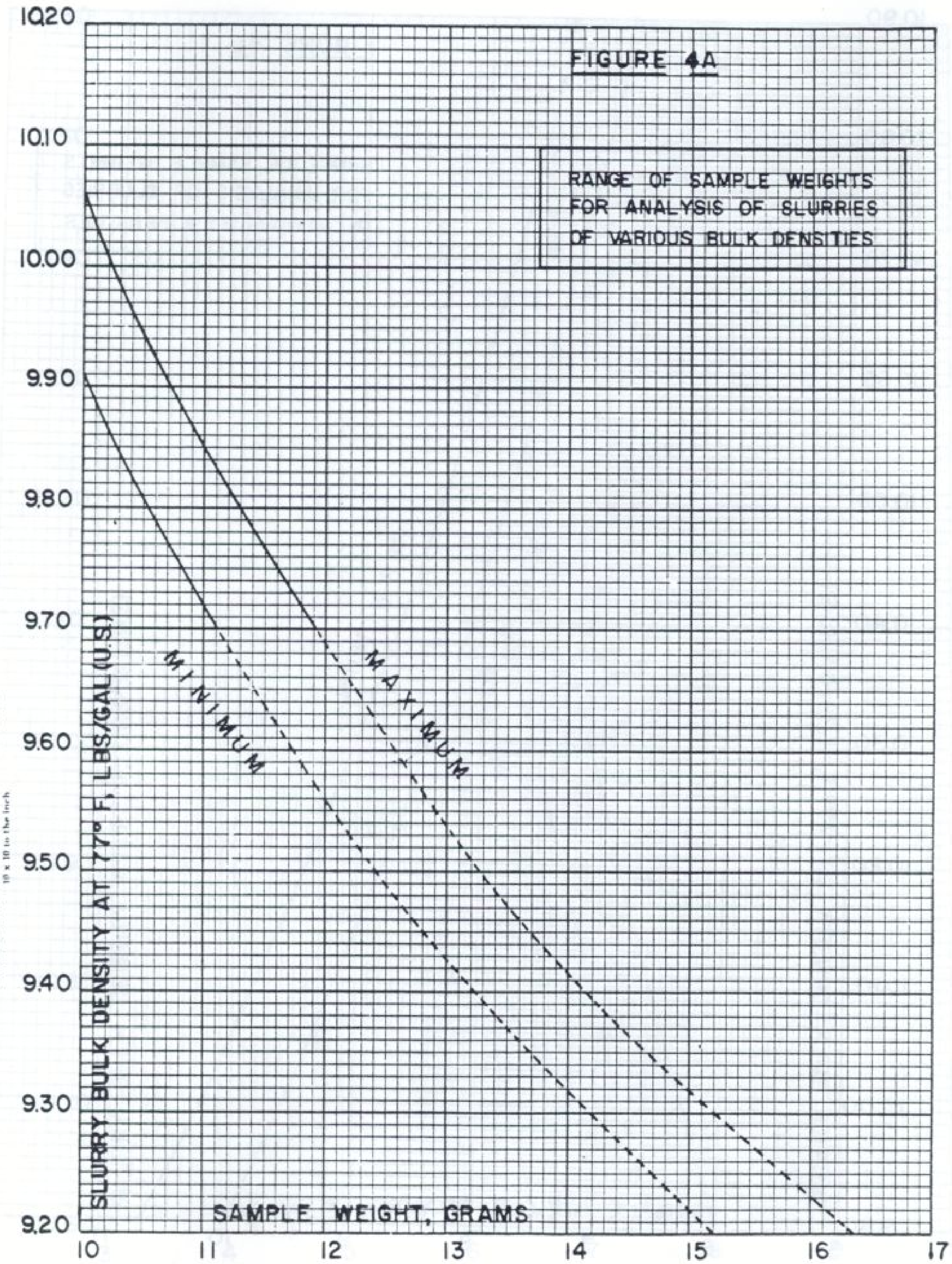


**Figure 1—**Range of Sample Weights for Analysis of Slurries—No. 1

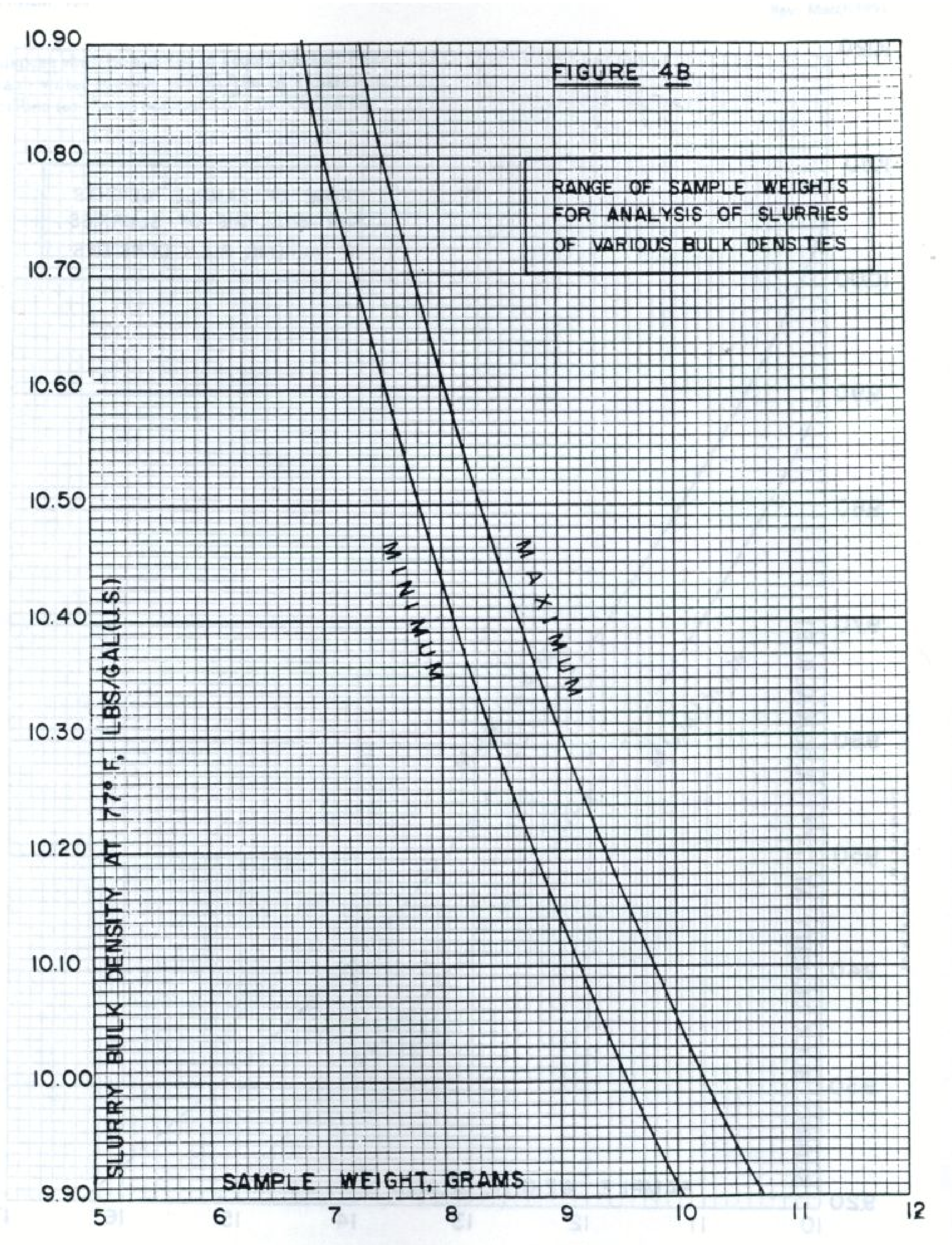


**Figure 2—**Range of Sample Weights for Analysis of Slurries—No. 2

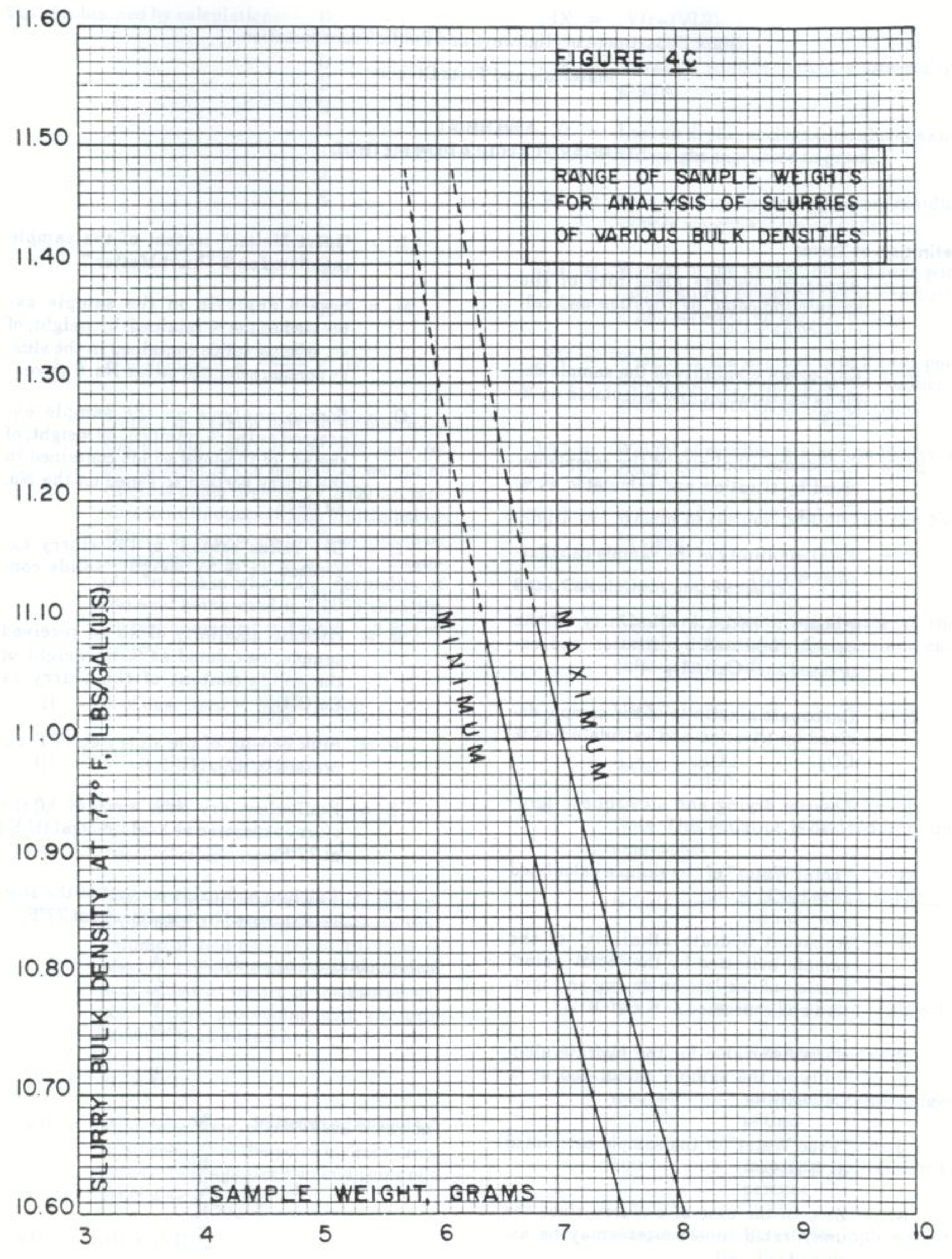


**Figure 3—**Range of Sample Weights for Analysis of Slurries—No. 3

## 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:

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

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, %:

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

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

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.

18.2 *Lime Slurry Alkalinity, % Ca(OH)₂:*

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

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:*

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

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:*

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

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.

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## 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:

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

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.

31.1.4 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 “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:

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

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:

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

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:

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

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.