

# SB101 Properties 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](http://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.

**ACKNOWLEDGED & AGREED:**

**SPECIALIST:**

\_\_\_\_\_  
**SIGNATURE**

\_\_\_\_\_  
**DATE**

\_\_\_\_\_  
**PRINTED NAME**

# WEBSITE

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



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www.txhmac.org

# 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+**

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**Contact Info**

Username: jDoe  
 E-mail: jdoe@txhmac.org  
 First Name: John  
 Last Name: Doe  
 Company: HMAC  
 Company Type: Lab / Other  
 Last 4 Digits of SSN: 1234

**Work Address**

Address: 140 Commercial Drive  
 City: Buda  
 State: Texas  
 Zip: 78610  
 District:

Work Phone: (512) 312-2889  
 Work Email: jdoe@txhmac.org

**Freight Address**  Same as Work Address

Address: 140 Commercial Drive  
 City: Buda  
 State: Texas  
 Zip: 78610  
 Freight Phone:

**Physical Address**  Same as Work Address

Address: 123 Hobbs Drive  
 City: Buda  
 State: Texas  
 Zip: 78610  
 Home Phone: (512) 305-5333  
 Cell Phone: (512) 305-5333

**Password**

Change Password: To change password, enter new password here. (Leave it blank if not changing)  
 Type your new password again.  
 Password must be at least 8 characters and must contain at least one lower case letter, one upper case letter, one digit, and one symbol.

# UPDATE ALL YOUR INFORMATION

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**Certification**

**Hot Mix**

CERT NAME	CERT DATE	CERT NUM	EXPIRATION DATE	STATUS	
1A	07/27/2015	3082	07/27/2018	Active	<a href="#">View Certificate</a>
1B	01/27/2015	2723	01/27/2018	Active	<a href="#">View Certificate</a>
2	02/06/2015	647	02/06/2018	Active	<a href="#">View Certificate</a>

[Proficiency Instructions](#)      [Annual Proficiency Reports](#)

**Soils Class**

CERT NAME	CERT DATE	CERT NUM	EXPIRATION DATE	STATUS	
SB101	02/23/2006	002	02/23/2009	Inactive Expired	<a href="#">View Certificate</a>
SB102	02/22/2006	45	02/22/2009	Inactive Expired	<a href="#">View Certificate</a>
SB103					
SB201					
SB202				Prerequisite: SB201 is required.	

[Proficiency Instructions](#)      [Annual Proficiency Reports](#)

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**Test Procedure for****SURVEYING AND SAMPLING SOILS FOR  
HIGHWAYS****TxDOT Designation: Tex-100-E****Effective Date: August 1999**

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**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.
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- 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  $\mu\text{m}$  (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  $\mu\text{m}$  (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  $\mu\text{m}$  (No. 200) sieve that is coarser than 0.002 mm (2  $\mu\text{m}$ ), 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.

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### 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.
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## 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.

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**Test Procedure for****PREPARING SOIL AND FLEXIBLE BASE  
MATERIALS FOR TESTING****TxDOT Designation: Tex-101-E****Effective Date: January 2010**

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**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.
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**2. DEFINITIONS**

- 2.1 *Soil Binder*—Soil binder is any material passing the 425  $\mu\text{m}$  (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.
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**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 pestle*.
- 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.

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## 4. MATERIALS

4.1 *Tap water.*

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## 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.

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

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## 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.

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## 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  $\mu\text{m}$  (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  $\mu\text{m}$  (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  $\mu\text{m}$  (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  $\mu\text{m}$  (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  $\mu\text{m}$  (0.025 to 0.035 in.)
- 7.1.28 If a pulverizer is used, any material still aggregated in lumps larger than 425  $\mu\text{m}$  (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  $\mu\text{m}$  (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  $\mu\text{m}$  (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.
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## 8. CALCULATION

- 8.1 Use the following to calculate the percent soil binder:

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

Where:

$W_1$  = dry mass of soil binder

$W_T$  = dry mass of total sample.

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## 9. REPORT

- 9.1 Report the percent soil binder to the nearest whole percent.
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# PART II—PREPARING SAMPLES FOR COMPACTION AND TRIAXIAL TESTS

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## 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.
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**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.

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

---

## 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.

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

---

**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 II, Preparing Samples for Compaction and Triaxial

Sieve Size	Individual Weight Retained	Individual % Retained	Cumulative % Retained
1 3/4"			
1 1/4"			
7/8"			
5/8"			
3/8"			
#4			
#40			
- #40			
Total			

---

**Test Procedure for****DETERMINING SLAKING TIME****TxDOT Designation: Tex-102-E****Effective Date: August 1999**

---

**1. SCOPE**

- 1.1 This method determines the slaking time to use in preparing flexible base materials.
  - 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 *Sample splitter, quartering machine, or quartering cloth.*
  - 2.2 *Set of standard U.S. sieves, meeting the requirements of Tex-907-K.*
  - 2.3 *Balance, Class G5 in accordance with Tex-901-K, with a minimum capacity of 36 kg (80 lb.)*
  - 2.4 *Drying oven, maintained at 60°C (140°F).*
  - 2.5 *Crusher (optional), which can be adjusted to produce material passing a 2.00 mm (No. 10) sieve.*
  - 2.6 *Mechanical pulverizer (optional).*
  - 2.7 *Wedgewood mortar and pestle.*
  - 2.8 *Scoop.*
  - 2.9 *Small siphon tube (optional).*
  - 2.10 *Sample containers, metal pans, cardboard cartons.*
  - 2.11 *Filter paper, non-fibrous.*
  - 2.12 *Sample splitter, quartering machine, or quartering cloth.*
  - 2.13 *Mechanical mixer (stirring device).*
  - 2.14 *Dispenser cup.*
-

2.15 *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.

---

### 3. MATERIALS

3.1 *Clear potable water.*

---

### 4. PROCEDURE

4.1 Select a field sample of approximately 14 kg (30 lb.)

4.2 Dry the field sample at  $60 \pm 5^\circ\text{C}$  ( $140 \pm 9^\circ\text{F}$ ).

4.3 Use a sample splitter and divide the field sample into six equal test samples.

4.4 Slake the test samples in clean water for different lengths of time (Ex: 1, 2, 4, 6, 8, and 24 hours).

4.5 Wash the samples and determine the soil binder according to Tex-101-E, Part I.

---

### 5. TEST RESULTS

5.1 The slaking time required for flexible base material will be the shortest time interval that produced the maximum amount of soil binder. Use this minimum slaking time for all control tests of materials from the particular source investigated.

---

**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, montmorillonite, 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  $110 \pm 5^{\circ}\text{C}$  ( $230 \pm 9^{\circ}\text{F}$ ).
  - 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

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### 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 contaminants 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  $110 \pm 5^\circ\text{C}$  ( $230 \pm 9^\circ\text{F}$ ) 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

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**Test Procedure for****DETERMINING LIQUID LIMITS OF SOILS****TxDOT Designation: Tex-104-E****Effective Date: August 1999**

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**1. SCOPE**

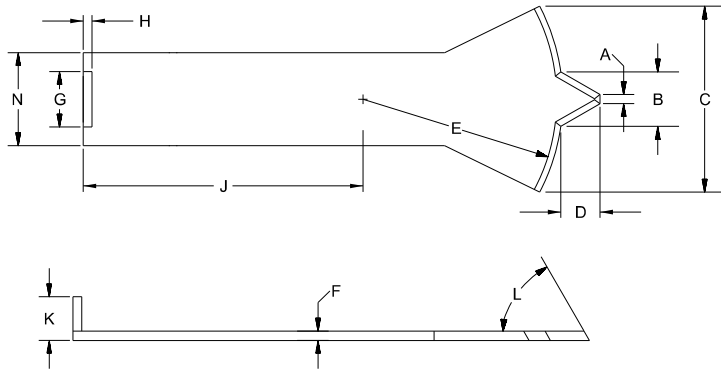
- 1.1 This procedure determines the liquid limit of soils, defined as the water content of a soil at the arbitrarily determined boundary between the liquid and plastic states, expressed as a percentage of the oven-dried mass of the soil.
- 1.2 The values given in parentheses (if provided) are not standard 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 *Mixing dish*, porcelain, 102–127 mm (4–5 in.) in diameter.
- 2.2 *Spatula*, flexible, with blade approximately 102 mm (4 in.) long and 19 mm (0.75 in.) wide.
- 2.3 *Balance*, Class G1 in accordance with Tex-901-K, minimum capacity of 100 g.
- 2.4 *Drying oven*, vented, maintained at  $110 \pm 5^\circ\text{C}$  ( $230 \pm 9^\circ\text{F}$ ).
- 2.5 *Weighing dishes*, non-absorbent, with lids.
- 2.6 *Grooving tool*, dimensions shown in Figure 1, made of non-absorbent, non-reactive material. (This grooving tool may be used in combination with the grooving tool specified by AASHTO T 89.)
- 2.7 *Liquid limit device*, consisting of a brass cup having a weight including cup hanger of 185–215 g and carriage conforming to the dimensions shown in Figure 4.  
**Note 1**—Wear is excessive when the point of contact on the cup or base exceeds 13 mm (0.5 in.) in diameter, or when any point on the rim of the cup is worn to half of the original thickness. Replace the cup when the groove-wear in the cup is 0.1 mm (0.004 in.) Refinish a base that is excessively worn, as long as the thickness meets the tolerance specified in Figure 4.
- 2.8 *Height-of-drop metal gauge block*, 10 mm (0.4 in.) thick and approximately 51 mm (2 in.) long and 25 mm (1 in.) wide.
-

2.9 Spray bottle.

ASTM Grooving Tool



AASHTO Grooving Tool

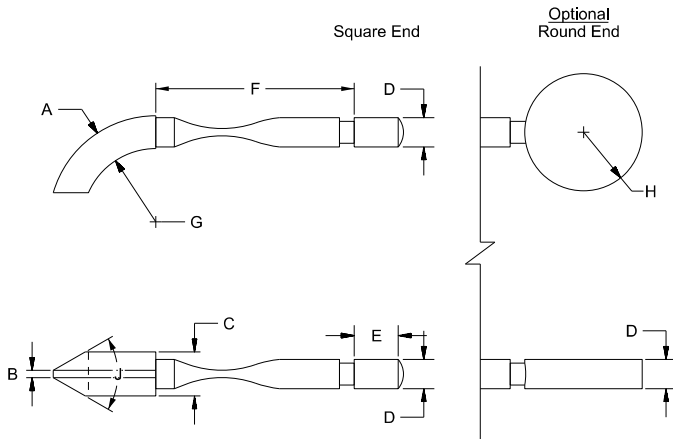
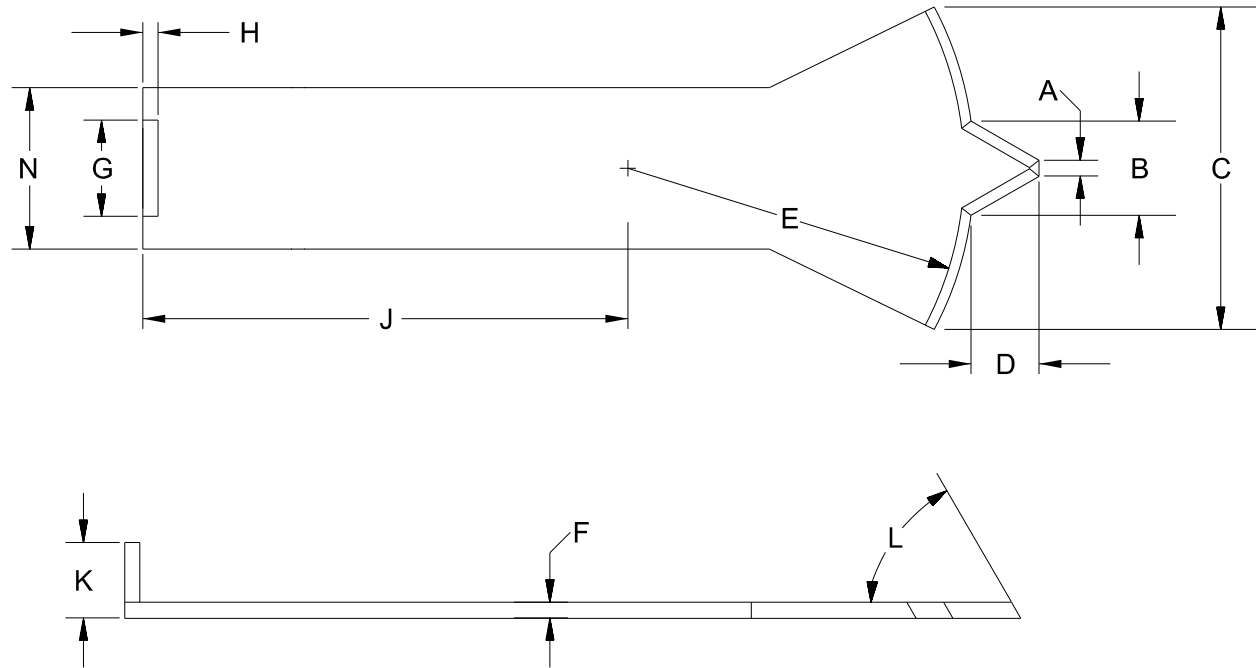


Figure 1—Grooving Tool Dimensions



Letter	A <sup>Δ</sup>	B <sup>Δ</sup>	C <sup>Δ</sup>	D <sup>Δ</sup>	E <sup>Δ</sup>	F <sup>Δ</sup>
Measurement, mm	2 ± 0.1	11 ± 0.2	40 ± 0.5	8 ± 0.1	50 ± 0.5	2 ± 0.1
Letter	G	H	J	K <sup>Δ</sup>	L <sup>Δ</sup>	N
Measurement, mm	10 (min)	13	60	10 ± 0.05	60° ± 1°	20

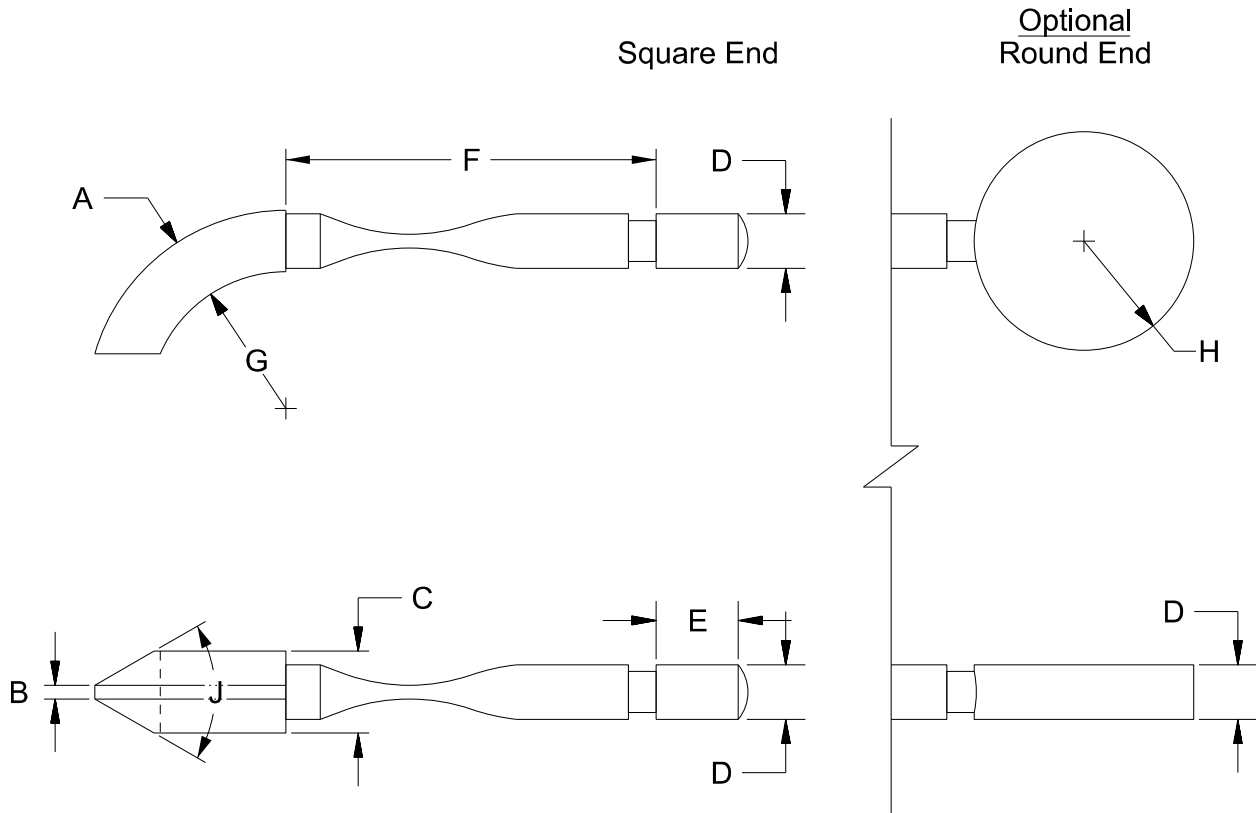
<sup>Δ</sup> Essential Dimensions

<sup>□</sup> Back at least 15 mm from tip.

NOTE: Dimension A should be 1.9 - 2.0 and dimension D should be 8.0 - 8.1 when new to allow for adequate service life.

**Figure 2**—Grooving Tool (ASTM)





Letter	A	B	C	D	E
Measurement, mm	10 ± 0.1	2 ± 0.1	13.5 ± 0.1	10 ± 0.2	15.9
Letter	F	G	H	J	
Measurement, mm	76.2	22.2	25.4	60°	

Figure 3—Grooving Tool (AASHTO)

OVERALL DIMENSIONS										
Letter	A	B	C	E	F	G	H	J	K	L
Measurement, in	54 ± 0.5	2 ± 0.1	27 ± 0.5	56 ± 2.0	32	10 ± 2.0	16 ± 2.0	60 ± 1.0	50 ± 2.0	150 ± 2.0
Letter	M	N	P	R	T	U	V	W	Z	
Measurement, in	125 ± 2.0	24 ± 1.0	28 ± 1.0	24 ± 1.0	45 ± 2.0	47 ± 1.0	3.8 ± 0.5	13 ± 2.0	6.5 ± 2.0	

CAM DIMENSIONS							
Cam Angle Degrees	0	30	60	90	120	150	180
Cam Radius	0.742 R	0.753 R	0.764 R	0.773 R	0.784 R	0.796 R	0.818 R
Cam Angle Degrees	210	240	270	300	330	360	
Cam Radius	0.854 R	0.901 R	0.945 R	0.974 R	0.995 R	1.000 R	

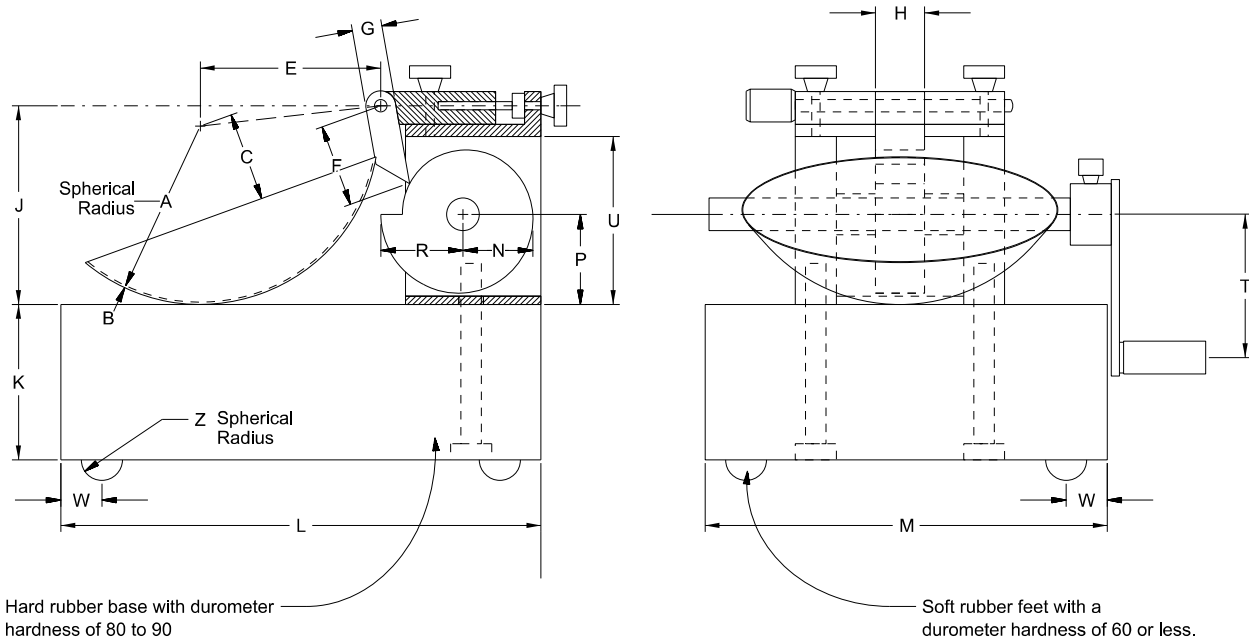


Figure 4—Mechanical Liquid Limit

### 3. REPORT FORM

- 3.1 Use [Form 1748](#), “Worksheet for Tex-104-E, Determining Liquid Limit of Soils” for the 1-point method and the 3-point method.

### 4. MATERIAL

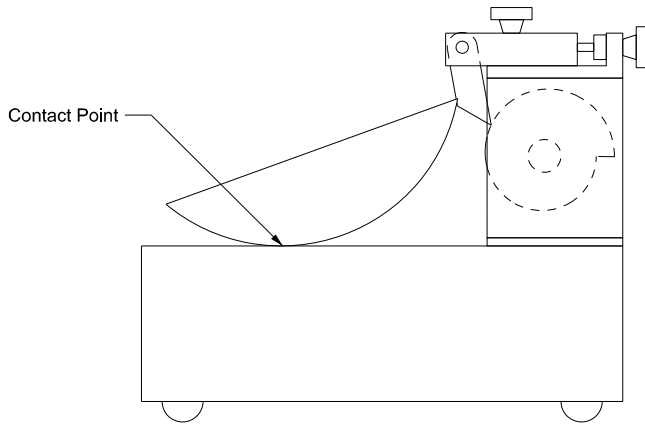
- 4.1 *Distilled or deionized water.*

### 5. PROCEDURE

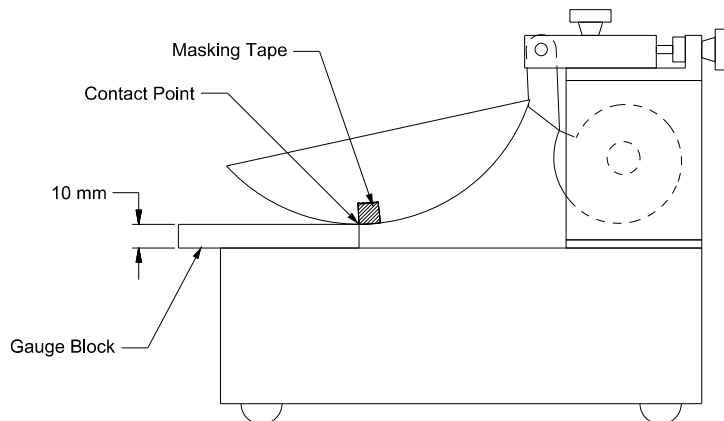
- 5.1 Inspect the Liquid Limit Device to ensure it is in good working condition by checking that:
- the pin connecting the cup is not worn sufficiently to permit side play;
  - the screws connecting the cup to the hanger arm are tight;

- the points of contact on the cup and base are not excessively worn; and
  - a groove has not been worn into the cup from long usage.
- 5.2 Inspect the grooving tool and the liquid limit device frequently to ensure that the critical dimensions meet those shown in Figure 1 and Figure 4, respectively.
  - 5.3 Use the height-of-drop gauge block and the adjustment plate (H, shown in Figure 4) to adjust the height of the cup.
  - 5.4 Secure the adjustment plate by tightening the screws.
  - 5.5 With the gauge block in place, check the adjustment by revolving the crank rapidly several times. If the height of drop for the cup is correct, a slight ringing sound will be heard when the cam strikes the cam follower. Make further adjustment if no sound is heard or the cup is raised off the gauge. (See Note 2.)

Cup Resting on Base of Liquid Limit Device



Cup Resting on Gauge Block with Tape in Place



**Figure 5**—Adjusting Liquid Limit Device

**Note 2**—A convenient procedure for adjusting the height of drop is as follows:

- Place a piece of masking tape across the outside bottom of the cup parallel with the axis of the cup hanger pivot.
- The edge of the tape away from the cup hanger should bisect the spot on the cup that contacts the base.
- For new cups, placing a piece of carbon paper on the base and allowing the cup to drop several times will mark the contact spot.
- Attach the cup to the device and turn the crank until the cup is raised to its maximum height.
- Slide the height gauge under the cup from the front, and observe whether the gauge contacts the cup or the tape. See Figure 4 for a side view.
- If the tape and cup are both contacted, the height of drop is approximately correct. If not, adjust the cup until simultaneous contact is made.
- Check adjustment by turning the crank at two revolutions per second while holding the gauge in position against the tape and cup.
- If a ringing or clicking sound is heard without the cup rising from the gauge, the adjustment is correct.
- If no ringing is heard, or if the cup rises from the gauge, readjust the height of drop.
- If the cup rocks on the gauge during this checking operation, the cam follower pivot is excessively worn, and the worn parts should be replaced.
- Always remove the tape after completion of the adjustment operation.

---

## 6. PREPARING SAMPLE

- 6.1 Prepare the soil binder in accordance with Tex-101-E, “Dry Preparation (Method A) or “Wet Preparation (Method B).” Prepare a referee sample according to Method A only.

---

## PART I, MULTI-POINT METHOD

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### 7. SCOPE

- 7.1 Use the multi-point method (Part I) as a referee method. The results of this method overrule the results of the one-point method (Part II) in cases of dispute.

---

### 8. PROCEDURE

**Note 3**—Go immediately to Section 8.6. if Tex-101-E, Method B was used to prepare the dry soil sample.

- 8.1 Place approximately 100 g of the soil sample in the mixing dish.
- 8.2 Thoroughly mix the soil with small increments of distilled or deionized water. Use a spray bottle to apply a uniform mist of water to the sample.
- 8.3 Alternately stir, knead, and chop the sample with a spatula, thoroughly mixing each increment of water with the soil before adding another increment of water.
- Note 4**—Do not use the cup of the liquid limit device for mixing the soil and water.
- 8.4 Add water and mix thoroughly until the soil-water mixture produces a groove closure with 25 to 35 blows by following Sections 8.7 through 8.14.
- 8.5 Cover the mixing dish and allow the sample to stand for at least 12 hours.
- Note 5**—Some soils are slow to absorb water; therefore, it is possible to add increments of water so fast that a false liquid limit is obtained. Avoid this by allowing more mixing and/or time for soil to absorb moisture sufficiently. If more soil is added to dry out the sample, mix thoroughly and allow ample time for the soil to absorb moisture sufficiently. Judgment may be used to shorten the soaking time for low Plasticity Index (PI) materials. All referee samples will be soaked for the full term.
- 8.6 Place a sufficient quantity of thoroughly mixed material in the cup of the liquid limit device and above the spot where the cup rests on the base.
- 8.7 Squeeze and spread the soil-water mixture with the spatula to level and at the same time trim to a depth of about 10 mm (0.4 in.) at the point of maximum thickness.
- 8.8 Use as few strokes of the spatula as possible, being careful not to entrap air bubbles within the mass. Return the excess soil to the mixing dish.
- 8.9 Divide the soil in the cup by a firm stroke of the grooving tool along the diameter through the centerline of the cam follower, so a clean sharp groove of the proper dimensions is formed. The grooving tool should be drawn in an arc and perpendicular to the surface of the cup through its movement.
- 8.10 To avoid tearing the sides of the groove or slipping of the soil pat in the cup, use no more than six strokes.
- 8.11 The depth of the groove should be increased with each stroke and only the last stroke should scrape the bottom of the cup.
- 8.12 When using the AASHTO grooving tool, the depth of the groove should be measured to assure a groove depth of  $8 \pm 0.1$  mm ( $0.3 \pm 0.05$  in.)
- 8.13 Lift and drop the cup by turning the crank at the approximate rate of two revolutions per second until the two halves of the soil pat come in contact at the bottom of the groove for a distance of about 13 mm (0.5 in.)
- 8.14 Record the number of blows required to close the groove. Observe at least two groove closures before one is accepted for the record.

**Note 6**— Do not hold the base of the machine while the crank is being turned. Some soils tend to slide on the surface of the cup instead of flowing. If this occurs, remove the sample from the cup and place in the mixing bowl, then add more water to the sample and remix. Place the soil-water mixture in the cup. Spread and smooth the soil with the spatula. Cut a groove with the grooving tool, and repeat the above step. If the soil continues to slide on the cup with 20 blows or fewer, this test is not applicable and Tex-107-E should be performed.

- 8.15 Remove a slice of soil approximately the width of the spatula, extending from edge to edge of the soil pat, at a right angle to the groove in which the soil flowed together.
- 8.16 Place at least 10 g of the sample in a tared container. Weigh and record the mass of the sample and container to the nearest 0.01 g. (See Section 9.)
- 8.17 Dry the sample to a constant mass in the oven. Weigh and record the mass of the dry soil and container to the nearest 0.01 g. (See Section 9.)
- 8.18 Calculate and record the loss in mass due to drying as the mass of water under Section 9.
- 8.19 Transfer the remaining soil in the cup to the mixing dish. Wash and dry the cup and grooving tool in preparation for the next trial.
- 8.20 Add small increments of water to the soil, mix thoroughly, allow sufficient time for the moisture to be absorbed, and repeat Sections 8.6 through 8.19 with at least two additional trials. Obtain one trial with the required groove closure in each of the following ranges:
- 15–25 blows
  - 20–30 blows
  - 25–35 blows

The minimum range of blows for the three trials should be ten blows.

## 9. CALCULATION

- 9.1 Use the following calculation to determine percent water content:

$$W = \frac{(A - B)}{(B - C)}(100)$$

Where:

$W$  = percent water content

$A$  = mass of wet sample + tare, g

$B$  = mass of dry sample + tare, g

$C$  = mass of tare, g.

---

**10. DATA REDUCTION**

- 10.1 *Plot data:*
- 10.1.1 Plot the relationship between the water content and the corresponding number of blows on a semi-logarithmic graph with the water content on the arithmetical scale, and the number of blows on the logarithmic scale.
- 10.1.2 Draw a “best-fit” straight line through the three or more plotted points.
- 10.1.3 Repeat the procedure to add or replace points necessary to find the “best-fit” straight line.
- 10.2 *Liquid Limit:*
- 10.2.1 Take the water content corresponding to the intersection of the line with the 25-blow abscissa as the liquid limit of the soil.
- 10.2.2 Report this value to the nearest whole percent.

---

**PART II—ONE-POINT METHOD**

---

**11. PROCEDURE**

- 11.1 Use Part I except for the following:
- 11.1.1 Use approximately 50 g of the prepared soil sample.
- 11.1.2 For accuracy equal to that obtained by the standard multi-point method, apply between 20 and 30 blows for groove closure.
- 11.1.3 Observe at least two groove closures before one is accepted for the record to ensure the accepted number is truly characteristic of the soil being tested.
- Note 7**—Some soils tend to slide on the surface of the cup instead of flowing. If this occurs, remove the sample from the cup and place in the mixing bowl, then add more water to the sample and remix. Place the soil-water mixture in the cup. Spread and smooth the soil with the spatula. Cut a groove with the grooving tool, and repeat the above step. If the soil continues to slide on the cup with 20 blows or fewer, this test is not applicable and Tex-107-E should be performed.
- 11.1.4 Take one moisture sample only for the final (accepted) trial.

**12. CALCULATIONS**

12.1 Calculate the water content of the soil at the time of the accepted groove closure according to Part I:

$$W_N = 100[(\text{Mass of Water})/(\text{Mass of Oven Dry Soil})]$$

-OR-

$$100[(A - B)/(B - C)]$$

12.2 Calculate the liquid limit by one of the two following equations:

$$LL = W_N (N/25)^{0.121}$$

-OR-

$$LL = K(W_N)$$

Where:

$N$  = No. of blows causing closure of the groove

$K$  = A factor given in Table 1

$W_N$  = Water content at  $N$  blows

$A$  = mass of wet sample + tare, g

$B$  = mass of dry sample + tare, g

$C$  = mass of tare, g

**Table 1—Factors for Obtaining Liquid Limit from Water Content & Number of Blows Causing Closure of Groove**

N (Number of Blows)	K (Factor for LL)
20	0.974
21	0.979
22	0.985
23	0.990
24	0.995
25	1.000
26	1.005
27	1.009
28	1.014
29	1.018



12.3 For low PI and non-plastic materials, when the two halves close or slide prior to 20 blows, determine the liquid limit (LL) as follows:

12.3.1 Perform Tex-107-E.

12.3.2 Calculate the Plasticity Index (LS = linear shrinkage in percent):

$$PI = LS(1.6)$$

12.3.3 Determine the Plastic Limit (PL) in accordance with Tex-105-E.

12.3.4 Calculate the Liquid Limit:

$$LL = PI + PL$$

---

**13. REPORT**

13.1 Report the liquid limit to the nearest whole percent.

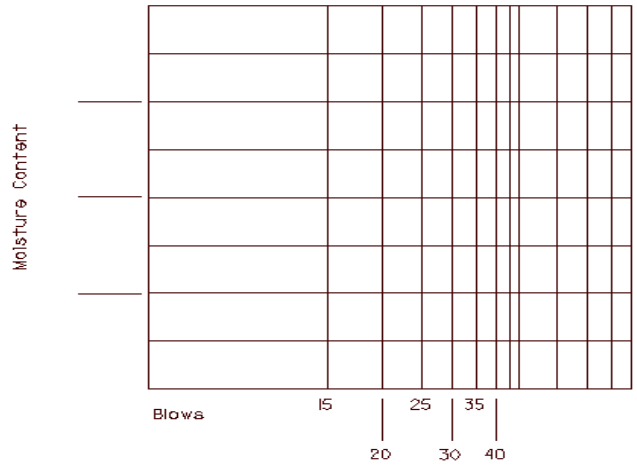


# Atterberg Limits

## Tex-104-E Part I, Multi-Point Method

<b>Dish 1</b>	A - Mass of wet sample + tare	<u>24.87</u>	B - Mass of wet sample + tare	<u>21.97</u>	# of Blows	<u>17</u>
	B - Mass of dry sample + tare	- <u>21.97</u>	C - Mass of tare	- <u>11.19</u>		
		<u>                    </u>	/	<u>                    </u>	X 100 =	<u>                    </u> % Water Content
<b>Dish 2</b>	A - Mass of wet sample + tare	<u>26.14</u>	B - Mass of wet sample + tare	<u>23.19</u>	# of Blows	<u>24</u>
	B - Mass of dry sample + tare	- <u>23.19</u>	C - Mass of tare	- <u>11.09</u>		
		<u>                    </u>	/	<u>                    </u>	X 100 =	<u>                    </u> % Water Content
<b>Dish 3</b>	A - Mass of wet sample + tare	<u>22.08</u>	B - Mass of wet sample + tare	<u>20.10</u>	# of Blows	<u>33</u>
	B - Mass of dry sample + tare	- <u>20.10</u>	C - Mass of tare	- <u>11.11</u>		
		<u>                    </u>		<u>                    </u>	X 100 =	<u>                    </u> % Water Content

(LL) Liquid Limit @ 25 Blows:                     



## Tex-105-E, Determining Plastic Limit of Soils

<b>Dish 3</b>	A - Mass of wet sample + tare	<u>24.87</u>	B - Mass of wet sample + tare	<u>21.97</u>
	B - Mass of dry sample + tare	- <u>21.97</u>	C - Mass of tare	- <u>11.19</u>
		<u>                    </u>	/	<u>                    </u>

## Tex-106-E, Calculating the Plasticity Index of Soils

Plasticity Index (PI) = Liquid Limit (LL) – Plastic Limit (PL)

Plastic Index (PI) =

### Tex-104-E, DETERMINING LIQUID LIMIT SOILS (1 Point)

Dish # \_\_\_\_\_

W.W.	_____	D.W.	_____	No. of	_____
D.W.	-	T.W.	-	Blows	_____
	_____	÷	_____	x 100 = Moisture Content:	_____

Δ Liquid Limit: \_\_\_\_\_

### Tex-105-E, DETERMINING PLASTIC LIMIT SOILS

DISH # \_\_\_\_\_

W.W.	_____	D.W.	_____		
D.W.	-	T.W.	-		
	_____	÷	_____	X 100 =	Plastic Limit _____

### Tex-106-E, CALCULATING THE PLASTIC INDEX OF SOILS

Plasticity Index: \_\_\_\_\_

### Tex-106-E, CALCULATING THE PLASTIC INDEX OF SOILS

Determine the liquid limit for each water content specimen using one of the following equations:

$$LL = W_N (N/25)^{0.121} \quad \text{or} \quad LL = K(W_N)$$

**Where:**

N = the number of blows causing closure of the groove at water content

$W_N$  = water content

K = a factor given in Table 1

Table 1 - Factors for Obtaining Liquid Limit from Water Content and Number of Drops Causing Closure of Groove

N (Number of Drops)	K (Factor for Liquid Limit)
20	0.974
21	0.979
22	0.985
23	0.990
24	0.995
25	1.000
26	1.005
27	1.009
28	1.014
29	1.018
30	1.022

---

**Test Procedure for****DETERMINING PLASTIC LIMIT OF SOILS****TxDOT Designation: Tex-105-E****Effective Date: August 1999**

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**1. SCOPE**

- 1.1 This method determines the plastic limit of soils.
  - 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 *Plastic Limit of Soils*—Plastic limit is defined as the lowest moisture content and expressed as a percentage of the weight of the oven dried soil at which the soil can be rolled into threads one-eighth inch in diameter without the soil breaking into pieces. This is also the moisture content of a solid at which a soil changes from a plastic state to a semisolid state.
- 

**3. APPARATUS**

- 3.1 *Porcelain evaporating dish*, 102–127 mm (4–5 in.) in diameter.
  - 3.2 *Flexible spatula*, blade approximately 102 mm (4 in.) long × 19 mm (0.75 in.) wide.
  - 3.3 *Plastic Limit Rolling Device (PLRD)*, and paper.
  - 3.4 *Balance*, Class G1 in accordance with Tex-901-K, 100 g minimum capacity.
  - 3.5 *Drying oven*, maintained at  $110 \pm 5^{\circ}\text{C}$  ( $230 \pm 9^{\circ}\text{F}$ ).
  - 3.6 *Weighing dishes*, non-absorbent, with lids.
  - 3.7 *Plaster of Paris disks*, approximately 102 mm (4 in.) in diameter (optional).
  - 3.8 *Rolling surface*, minimum area of  $300 \times 300$  mm ( $12 \times 12$  in.), non-absorbent, non-corrosive, light surface texture (e.g., ground glass plate, linoleum, or plastic-faced plywood).
- Note 1**—The hand method may be used; however, PLRD will be used as the referee.
-

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#### 4. PREPARING SAMPLE

- 4.1 Select approximately 20 g from material prepared for Tex-104-E.
- 4.2 Reduce water content of soil until it reaches a consistency at which it can be rolled without sticking to hands. Water content can be reduced by one of the following methods:
- place the soil in between two plaster of Paris disks,
  - expose the soil to the air current from an electric fan, or,
  - roll on paper that does not add any fiber to the soil, such as hard surface paper.

---

#### 5. PROCEDURE

- 5.1 From the 20 g mass, select four to five portions of 1.5–2 g.
- 5.2 Form each portion into an ellipsoidal mass.
- 5.3 Place the masses in a row on a piece of paper on the PLRD, spaced evenly apart.
- 5.4 Roll the masses in the device with just sufficient pressure to form threads of uniform diameter.
- Note 2**—A normal rate of rolling for most soils is 80–90 strokes per minute, counting a stroke as one complete motion forward and backward to the starting position. This rate of rolling may have to be decreased for very fragile soils.
- 5.5 Taking no more than two minutes, further deform the threads on each stroke so the diameters are continuously reduced and the lengths are extended, until the diameters reach  $3.2 \pm 0.5$  mm ( $1/8 \pm 0.02$  in.)
- 5.6 Reduce the diameter of the threads to  $3.2 \pm 0.5$  mm ( $1/8 \pm 0.2$  in.) Break each thread into several pieces. Squeeze the pieces of each thread together, knead between the thumb and first finger, and reform into an ellipsoidal mass.
- 5.7 Repeat Sections 5.3–5.6 until the soil crumbles under the pressure required for rolling and can no longer be rolled into a 3.2 mm ( $1/8$  in.) diameter thread. There is no problem if the thread breaks into shorter segments before reaching the 3.2 mm ( $1/8$  in.) diameter. Roll each of these shorter segments to 3.2 mm ( $1/8$  in.) diameter.
- 5.8 Gather the portions of the crumbled threads and place in a tared container. Immediately cover the container.
- 5.9 Continue the procedure to accumulate at least 10 g of sample rolled to the 3.2 mm ( $1/8$  in.) diameter.
- 5.10 Weigh and record the mass of the sample and container to the nearest 0.01 g. (See Section 6.) Dry the soil sample in the container to a constant mass in a 110°C (230°F) oven.

- 5.11 Weigh and record the mass of the dry soil and container to the nearest 0.01 g. (See Section 6.)
- The operator should at no time attempt to produce failure at exactly 3.2 mm (1/8 in.) by reducing the rate of rolling and/or pressure, while continuing the rolling without further deformation.
  - For low Plasticity Index (PI) soils, it is permissible to reduce the initial diameter of the ellipsoidal mass to near the required 3.2 mm (1/8 in.) final diameter.
  - Use palm, finger, or a flexible spatula to roll low PI materials.
  - If crumbling occurs when the thread has a diameter greater than 3.2 mm, this should be considered a satisfactory endpoint, provided the soil has been previously rolled to a 3.2 mm (1/8 in.) thread.
  - Crumbling of the thread will manifest itself differently with various types of soil. Some soils fall apart into numerous small aggregations of particles. Others may form an outside tubular layer that starts splitting at both ends. The splitting progresses toward the middle, and finally the thread falls apart in many small platy particles.
  - Fat clay soils require much pressure to deform the thread, particularly as they approach the plastic limit. With these soils, the thread breaks into a series of barrel-shaped segments about 3.2–9.5 mm (1/8–3/8 in.) in length.

**NOTE 3**—The only requirement for continuing the test is that the sample can be reformed into an ellipsoidal mass and rerolled.

## 6. CALCULATIONS

6.1 Mass of water:

$$W = A - B$$

6.2 Plastic Limit (%):

$$PL(\%) = 100[W/(B - C)]$$

Where:

$A$  = mass of wet soil + tare, g

$B$  = mass of dry soil + tare, g

$C$  = mass of tare, g.

## 7. REPORT

7.1 Report the PL to the nearest whole percent.

---

**Test Procedure for****CALCULATING THE PLASTICITY INDEX OF SOILS****TxDOT Designation: Tex-106-E****Effective Date: August 1999**

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**1. SCOPE**

- 1.1 This method determines the plasticity index of soils.
  - 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 *Plasticity Index*—Plasticity index is a test conducted on soil samples as set out in this test method. The plasticity index is a range of moisture in which a soil remains in a plastic state while passing from a semisolid state to liquid state. Numerical difference between Liquid Limit and Plastic Limit of a soil ( $PI = LL - PL$ ) using Tex-106-E.
- 

**3. PROCEDURE**

- 3.1 Determine liquid limit in accordance with Tex-104-E.
  - 3.2 Determine plastic limit in accordance with Tex-105-E.
- 

**4. CALCULATION**

- 4.1 Use the following calculation to determine plasticity index:

$$PI = \text{Liquid Limit} - \text{Plastic Limit}$$

---

**5. REPORTING**

- 5.1 Record results to the nearest whole number.

---

**Test Procedure for****DETERMINING THE BAR LINEAR SHRINKAGE OF SOILS****TxDOT Designation: Tex-107-E****Effective Date: August 1999**

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**1. SCOPE**

- 1.1 This method allows the user to determine bar linear shrinkage of soils. It shows how to prepare the sample, take measurements, and calculate the linear shrinkage.
  - 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 *Porcelain evaporating dish*, approximately 115–140 mm (4.5–5.5 in.) in diameter.
  - 2.2 *Flexible spatula*, with a blade approximately 102 mm (4 in.) long × 19 mm (0.75 in.) wide.
  - 2.3 *Straight edge*, stainless steel shrinkage gauge.
  - 2.4 *Balance*, Class G1 in accordance with Tex-901-K, minimum capacity of 200 g.
  - 2.5 *Drying oven*, maintained at  $110 \pm 5^{\circ}\text{C}$  ( $230 \pm 9^{\circ}\text{F}$ ).
  - 2.6 *Grooving tool*, made of non-absorbent, non-reactive material, as shown in Tex-104-E.
  - 2.7 *Bar linear shrinkage mold*, stainless steel or aluminum, with section  $19 \times 19 \times 127$  mm ( $0.75 \times 0.75 \times 5$  in.)
  - 2.8 *Number 20 scale* (optional).
- 

**3. MATERIALS**

- 3.1 *Petroleum jelly*.
- 3.2 *Distilled or deionized water*.



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#### 4. PREPARING SAMPLE

- 4.1 The bar linear shrinkage test is made on soil binder.
- 4.2 The moist soil sample remaining after the completion of Tex-104-E may be used to form the soil bar.
- 4.3 If there is not a sufficient amount of soil remaining from the liquid limit test, prepare a soil sample according to Tex-101-E, Part I, and mix uniformly with water to reach the consistency as outlined in the procedure of this test.

---

#### 5. PROCEDURE

- 5.1 Place enough material in the evaporating dish to fill the shrinkage mold.
- 5.2 Add water, realizing that a considerable amount of manipulation is required to mix plastic soils.  
**Note 1**—It is very important to mix the soil and water thoroughly and uniformly.
- 5.2.1 Test wet soil for the proper molding consistency by shaping the sample into a smooth layer about 13 mm (0.5 in.) thick on the bottom of the dish and making a groove with the grooving tool.
- 5.2.2 If the material immediately flows of its own accord and just closes the groove at the bottom, the sample is ready for molding.
- 5.2.3 If a slight jarring is required to close the groove or if the soil is obviously too wet, add more water or dry soil and remix the sample.
- 5.3 Grease the inside walls of the bar linear shrinkage mold with a thin layer of petroleum jelly to prevent the adhesion of the soil to the mold.
- 5.4 Shape the soil bar by placing a small portion of the wet soil evenly into the mold and gently jarring the mold to cause the soil to flow and to assist in the removal of entrapped air bubbles.
- 5.5 When the mold has been completely filled, remove the excess soil from the bar by means of the straightedge and smooth the surface level with the top of the mold.
- 5.6 Air dry the soil bar at room temperature until color changes slightly, place in  $110 \pm 5^{\circ}\text{C}$  ( $230 \pm 9^{\circ}\text{F}$ ) oven and dry to a constant mass. Remove specimen from oven, allow to cool, and measure the length of the bar.

---

## 6. CALCULATIONS

6.1 The linear shrinkage can be obtained as a direct reading from the stainless steel shrinkage gauge or calculated as below:

6.1.1 If measured in mm (in.):

$$LS = 100(L_W - L_D) / L_W$$

Where:

$L_W$  = length of the wet soil bar, 127 mm (5 in.)

$L_D$  = length of the dry soil bar, mm (in.)

6.1.2 If measured in percent:

$$LS = L_W - L_D$$

Where:

$L_W$  = length of the wet soil bar, 100%

$L_D$  = length of the dry soil bar, %.

---

## 7. REPORT

7.1 Report to the nearest whole percent.



## Tex-107-E, Determining Bar Linear Shrinkage of Soils Calculation

### Calculation

If measured in Inches:

$$LS = 100 (L_w - L_d) / L_w$$

If measured in percent:

$$LS = L_w - L_d$$

### Where

- LS = Linear shrinkage
- L<sub>w</sub> = Length of the wet soil bar (5" or 100%)
- L<sub>d</sub> = Length of the dry soil bar (in. or %)

---

**Test Procedure for****PARTICLE SIZE ANALYSIS OF SOILS****TxDOT Designation: Tex-110-E****Effective Date: August 1999**

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**1. SCOPE**

- 1.1 This method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75  $\mu\text{m}$  (No. 200) is determined by sieving, while the distribution of particle sizes smaller than 75  $\mu\text{m}$  is determined by a sedimentation process, using a hydrometer to secure the necessary data.
- 1.2 If hydrometer analysis is not required, but a determination of material passing the 75  $\mu\text{m}$  (No. 200) sieve is desired, refer to Tex-401-A for low Plasticity Index (PI) materials or Tex-111-E for clay materials.
- 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.
- 

**PART I—SIEVE ANALYSIS OF MATERIAL RETAINED ON THE 425  $\mu\text{M}$  (NO. 40) SIEVE**

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**2. SCOPE**

- 2.1 Part I details the necessary steps for sieve analysis of material retained on the 425  $\mu\text{m}$  (No. 40) sieve.
- 

**3. APPARATUS**

- 3.1 *Drying oven, maintained at  $110 \pm 5^\circ\text{C}$  ( $230 \pm 9^\circ\text{F}$ ).*
- 3.2 *Mechanical sieve shaker.*
- 3.3 *Balance, Class G2 in accordance with Tex-901-K, minimum capacity of 15 kg (33 lb.)*
- 3.4 *Sample splitter, quartering machine, or quartering cloth.*
- 3.5 *Standard U.S. sieves, meeting the requirements of Tex-907-K.*
- 3.6 *Pans.*
-

---

## 4. SAMPLES

- 4.1 The mass of sample should be sufficient for particle size analysis. The minimum amount required of material retained on the 425  $\mu\text{m}$  (No. 40) sieve depends on the maximum particle size. The size should not be less than the amount shown in Table 1. When the nominal maximum size is between sizes shown, use next larger minimum mass.

**Table 1—Mass Requirement for Sieve Analysis**

Nominal Maximum Size	Approximate Minimum Mass
9.5 mm (3/8 in.)	0.5 kg (1 lb.)
25 mm (1 in.)	2 kg (4 lb.)
37.5 mm (1-1/2 in.)	4 kg (8 lb.)
50 mm (2 in.)	5 kg (10 lb.)
75 mm (3 in.)	6 kg (12 lb.)

**Note 1**—The size of the portion passing the 425  $\mu\text{m}$  (No. 40) sieve should be:

- for the hydrometer test, approximately 100 g for sandy soil and approximately 50 g for silty or clayey soils.
- for hygroscopic moisture determination, at least 10 g.

---

## 5. PROCEDURE

- 5.1 Prepare a sample of material for analysis in accordance with Tex-101-E, Method A). Record the mass of the material passing the 425  $\mu\text{m}$  (No. 40) sieve (soil binder) as  $W_s$  under Section 6.
- 5.2 Obtain all sieve sizes required by the material specification. Stack sieves in descending order with the sieve having the largest opening on top and a pan on the bottom.
- 5.3 Pour the plus (+) 425  $\mu\text{m}$  (No. 40) portion of the sample into the sieves. Use a mechanical shaker and shake the sieves for five minutes.
- 5.4 After shaking, remove the top sieve from the stack without losing any of the retained material. Over a clean pan, hand sieve until not more than one percent, by weight, of the material retained on the sieve continues to pass through the sieve. Combine any material passing the sieve with the material retained on the next smallest size sieve.
- 5.5 Weigh the portion retained on the first sieve and record the mass as  $W_1$  under Section 6. Repeat Section 5.4 for the next largest sieve size and then add the material retained to the portion retained on the first sieve and record the combined weight as  $W_2$  under Section 6. Continue hand sieving and recording the combined masses, as  $W_3$ ,  $W_4$ , etc., until all sieving is complete.

---

## 6. CALCULATIONS

6.1 Calculate the total mass of the sample:

$$W_T = W_S + W$$

Where:

$W_T$  = total mass of sample, g

$W_S$  = mass of material passing the 425  $\mu\text{m}$  (No. 40) sieve, g

$W$  = cumulative mass of smallest sieve size, g.

6.2 Calculate cumulative percent retained for each sieve:

$$\text{Cumulative \% Retained first sieve} = 100 \bullet W_1 / W_T$$

$$\text{Cumulative \% Retained second sieve} = 100 \bullet W_2 / W_T, \text{ etc.}$$

6.3 Calculate individual percent retained for each sieve by subtracting the cumulative percent retained of one sieve size larger from the cumulative percent retained of the sieve size:

$$\text{Individual \% Retained} = \text{Cum. \% of } W_2 - \text{Cum. \% of } W_1, \text{ etc.}$$

Plot the cumulative percent retained, from above, versus the sieve size, on [Tx110.xlsm](#), “Cumulative Mechanical Analysis” tab or on semi-logarithmic paper.

**Table 2—Sieve Analysis Calculations**

Sieve Size	Cumulative Weight Retained (g)	Cumulative Percent Retained	Individual Percent Retained
12.5 mm (1/2 in.)	108.4	2.8	2.8
9.5 mm (3/8 in.)	412.5	10.8	8.0
4.75 mm (No. 4)	2285.0	59.6	48.8
2.36 mm (No. 8)	3523.0	91.9	32.3
Total – including minus (-) 425 $\mu\text{m}$ (No. 40) material	3832.0	100	

---

## 7. TEST REPORT

7.1 Report the individual percent retained on each sieve to the nearest whole number.

---

**PART II—HYDROMETER ANALYSIS OF SOILS PASSING 425  $\mu\text{M}$   
(NO. 40) SIEVE**

---

**8. SCOPE**

- 8.1 This part describes the analysis of soils passing the 425  $\mu\text{m}$  (No. 40) sieve using a hydrometer.
- 

**9. APPARATUS**

- 9.1 *Balance*, Class G1 in accordance with Tex-901-K, minimum capacity of 200 g.
- 9.2 *Stirring apparatus*, either a mechanical stirring device or an air dispersion device, as shown in Figure 1.
- 9.3 *Hydrometer*, graduated in grams per liter, Type 151 H or 152 H, as shown in Figure 3.
- 9.4 *Sedimentation cylinder*, a glass hydrometer cylinder approximately 457 mm (18 in.) in height and 63.5 mm (2.5 in.) in diameter, graduated for a volume of 1000 mL, as shown in Figure 2.
- 9.5 *Mercury thermometer*, range of 0–104°C (1–220°F), accurate to 0.5°C (1°F).
- 9.6 *Standard U.S. sieves*, meeting the requirements of Tex-907-K in the following sizes:
- 75 mm (3 in.)
  - 50 mm (2 in.)
  - 25 mm (1 in.)
  - 9.5 mm (3/8 in.)
  - 4.75 mm (No. 4)
  - 2 mm (No. 10)
  - 425  $\mu\text{m}$  (No. 40)
  - 75  $\mu\text{m}$  (No. 200).
- 9.7 *Oven*, maintained at  $110 \pm 5^\circ\text{C}$  ( $230 \pm 9^\circ\text{F}$ ).
- 9.8 *Evaporating dishes*.
- 9.9 *Water bath, or constant temperature room*.
- 9.10 *Timing device*, with a second hand.
- 9.11 *Beaker*, 250 mL (7.5 oz.)
-



**Figure 1**—Mechanical Stirring Device



**Figure 2**—Air Dispersion Device



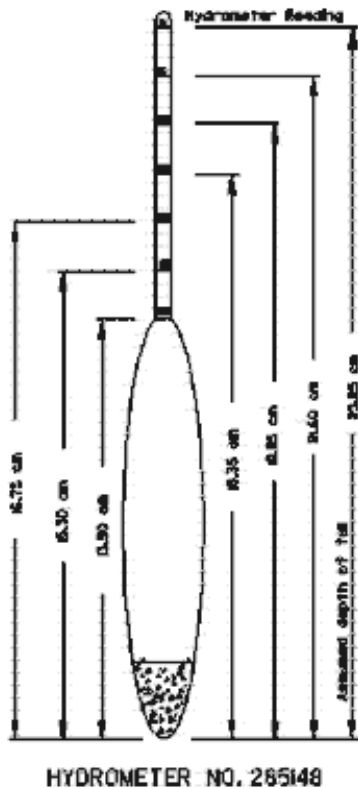


Figure 3—Hydrometer with Dimensions

## 10. MATERIAL

- 10.1 *Dispersing agent*, a solution of sodium hexametaphosphate in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate per liter of solution.
- 10.1.1 Solution of this salt, if acidic, should be slowly reverted or hydrolyzed back to the orthophosphate form with a resultant decrease in dispersing action.
- 10.1.2 Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate.
- 10.1.3 Bottles containing solutions should have the date of preparation marked on them.
- 10.2 *Distilled or demineralized water*.
- 10.3 *Source of compressed air*, if air dispersion device is used.

## 11. DETERMINING COMPOSITE CORRECTION FOR HYDROMETER READING

- 11.1 Equations for percentages of soil remaining in suspension, as given in Section 13 are based on the use of distilled or demineralized water.

- 11.2 A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.
- 11.3 The manufacturer calibrates the soil hydrometers at 20°C (68°F), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.
- 11.4 The manufacturer graduates hydrometers to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.
- 11.5 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.
- 11.6 For convenience, a graph or table of composite corrections for a series of one-degree temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.
- 11.7 Prepare 1000 mL (30 fl. oz.) of liquid composed of distilled water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test.
- 11.8 Place the liquid in a sedimentation cylinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used.
- 11.9 When the temperature of the liquid becomes constant, insert the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151 H the composite correction is the different between this reading and one; for hydrometer 152 H it is the difference between the reading and zero.
- 11.10 Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.
- 

## 12. PROCEDURE

- 12.1 *Hydrometer Analysis:*
- 12.1.1 Use soil binder prepared in Part I, or prepare the soil binder in accordance with Tex-101-E if Part I was not used. Record the mass of the air-dried sample as  $W_A$  under Section 13.
- 12.1.2 Determine the hygroscopic moisture of the soil binder in accordance with Tex-103-E. Record the percent hygroscopic moisture as  $P_H$  under Section 13.
- 12.1.3 Determine the specific gravity of the soil binder in accordance with Tex-108-E.

- 12.1.4 Use a sample splitter to obtain 50 g (for silty or clayed soils) or 100 g (for sandy soil) of soil binder to the nearest 0.01 g. Place the sample in a 250 mL beaker or in the hydrometer cylinder if the dispersion tube is available. Add approximately 125 mL of the sodium hexametaphosphate solution and cover with distilled water. Stir the soil thoroughly and then allow to soak for at least 12 hours.
- 12.1.5 After the soaking period, disperse the soil with the stirring device or a soil dispersion tube as follows:
- 12.1.5.1 *Stirring Device:*
- 12.1.5.1.1 Wash the soil into the dispersion cup and add distilled water until the cup is slightly more than half-full.
- 12.1.5.1.2 Disperse the contents for a period of 1 minute in the mechanical stirring device.
- 12.1.5.1.3 After dispersion, transfer the soil slurry to the hydrometer cylinder and add sufficient distilled water (having the same temperature as the water bath) to bring the level of the water to the 1000 mL mark on the cylinder.
- 12.1.5.1.4 Place the cylinder in the constant temperature water bath.
- 12.1.5.2 *Soil Dispersion Tube:*
- 12.1.5.2.1 Add about 300 mL of distilled water to the soaked sample and carefully place the dispersion tube into the hydrometer cylinder.
- 12.1.5.2.2 Adjust the air pressure by means of the valve and disperse the soil.
- 12.1.5.2.3 Disperse the soil-water mixture using an air pressure of 152 kPa (20 psi).
- 12.1.5.2.4 Disperse soils with a Plasticity Index (PI) of 5 or less for 5 minutes; soils with a PI between 6 and 20 for 10 minutes; and soils with a PI greater than 20 for 15 minutes.
- 12.1.5.2.5 Soils containing large percentage of mica need be dispersed for 60 seconds only.
- 12.1.5.2.6 Wash the soil from the dispersion tube into cylinder and add sufficient amount of distilled water to bring the level of the water to the 1000 mL mark before placing into the constant temperature bath.
- 12.1.6 When the soil suspension reaches the temperature of the bath, remove the graduate and thoroughly shake its contents for one minute, using the palm of the hand or a stopper over the open end of the cylinder. Mix the contents of the cylinder by alternately turning the cylinder upside-down, then right side up and by loosening any material remaining on the bottom while in the inverted position.
- 12.1.7 At the conclusion of this shaking, place the hydrometer cylinder on the table, immediately lower the hydrometer into the suspension and record the time. Read the hydrometer at the peak of the meniscus formed on the stem to the nearest 0.5 g per liter at the end of two minutes from the time the graduate was set on the table. Remove the hydrometer and carefully place the cylinder with contents into the constant temperature

bath. Obtain hydrometer readings at time intervals of 5, 15, 30, 60, 250, and 1440 minutes after the beginning of sedimentation. About 15 seconds before the time of each reading, slowly and carefully lower the hydrometer into the soil suspension and read the hydrometer after it has come to rest. After each reading, remove the hydrometer from the cylinder in such a manner as to cause as little disturbance as possible. Determine and record the temperature of the suspension each time a hydrometer reading is taken. Record data on work card, to the nearest 0.1%.

## 12.2 *Fine Sieve Analysis:*

- 12.2.1 At the conclusion of the final hydrometer reading, pour the soil suspension onto a 75  $\mu\text{m}$  (No. 200) sieve and rinse the retained particles with tap water until the wash water is clear.
- 12.2.2 Flush any material retained on the 75  $\mu\text{m}$  (No. 200) sieve to an evaporating dish and dry to a constant mass at a temperature of  $110 \pm 5^\circ\text{C}$  ( $230 \pm 9^\circ\text{F}$ .) No water should be decanted from the evaporating dish to avoid loss of material; after the material has settled and the water is clear, carefully decant water from the evaporating dish avoiding any loss of material. A sieve analysis is made using the 425  $\mu\text{m}$  (No. 40) and 75  $\mu\text{m}$  (No. 200) sieves, and such other sieves as may be required by the material under test.

## 13. CALCULATIONS

- 13.1 Calculate the percentage of hygroscopic moisture to the nearest 0.001 percent:

$$P_H = 100 \bullet (W_{Air\ Dry} - W_{Ovenr\ Dry}) / W_{Ovenr\ Dry}$$

Where:

$W_{Air\ Dry}$  = mass of air-dry soil for hygroscopic moisture test, g

$W_{Ovenr\ Dry}$  = mass of oven-dry soil for hygroscopic moisture test, g.

- 13.2 Calculate the mass of oven-dry soil sample for the hydrometer test:

$$W_O = 100 \bullet W_A / (100 + P_H)$$

Where:

$W_O$  = mass of oven-dry soil sample for the hydrometer test

$P_H$  = percent hygroscopic moisture

$W_A$  = mass of air-dry sample for the hydrometer test.

- 13.3 Calculate the percent soil binder in the total sample:

$$P_B = 100 \cdot W_S / W_T$$

Where:

$P_B$  = percent soil binder in the total sample

$W_S$  = mass of material passing the 425  $\mu\text{m}$  (No. 40) sieve, g

$W_T$  = total mass of the sample, g (as calculated in Part I).

- 13.4 Calculate the percentage of the total original material that is retained on any given fine sieve analysis sieves:

$$\% \text{ of Original Retained} = P_B \cdot W_1 / W_B + (100 - P_B), \text{ etc.}$$

Where:

$P_B$  = percent soil binder in the total sample

$W_B$  = mass of oven-dry soil sample for the hydrometer test, g

$W_1$  = mass of portion retained on the first sieve from fine sieve analysis, g.

- 13.5 Calculate the percentage of soil in suspension:

$$P_S = (P_B \cdot R \cdot a / W_O)$$

Where:

$P_S$  = percentage of soil binder in suspension

$P_B$  = percent soil binder in the total sample

$R$  = corrected hydrometer reading

$a$  = constant depending on the density of the suspension and varies with the specific gravity of the soil (Gr.), (shown in Table 3)

$W_O$  = mass of oven-dry soil sample for the hydrometer test.

**Table 3—Constant a**

Specific Gravity, Gr.	Constant, a
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

- 13.6 Calculate the maximum diameter, *d*, of the particles in suspension, corresponding to the percentages indicated by a given hydrometer reading, using modified Stoke's Law:

$$d = (K / 1000) \bullet (L / T)^{1/2}$$

Where:

*L* = distance from the surface of the suspension is being level at which the density of the suspension is being measured, cm. For a given hydrometer and sedimentation cylinder, the values vary according to the hydrometer readings. These values of distance *L*, known as the effective depth, are given in Table 4.

*T* = an interval of time from beginning of sedimentation to the taking of the reading, minutes (2, 5, 15, 30, 60, 250, and 1440 min.)

*K* = a constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of *K* for a range of temperatures and specific gravities are given in Table 5. The value of *K* does not change for a series of readings constituting a test, while values of *L* and *T* do vary.

**Table 4—Effective Depth, L**

Actual Hydrometer reading	Effective Depth, L, cm	Actual Hydrometer reading	Effective Depth, L, cm
0	16.3	31	11.2
1	16.1	32	11.1
2	16.0	33	10.9
3	15.8	34	10.7
4	15.6	35	10.6
5	15.5	36	10.4
6	15.3	37	10.2
7	15.2	38	10.1
8	15.0	39	9.9
9	14.8	40	9.7
10	14.7	41	9.6
11	14.5	42	9.4
12	14.3	43	9.2
13	14.2	44	9.1
14	14.0	45	8.9
15	13.8	46	8.8
16	13.7	47	8.6
17	13.5	48	8.4
18	13.3	49	8.3
19	13.2	50	8.1
20	13.0	51	7.9
21	12.9	52	7.8
22	12.7	53	7.6
23	12.5	54	7.4
24	12.4	55	7.3

Table 4—Effective Depth, L

Actual Hydrometer reading	Effective Depth, L, cm	Actual Hydrometer reading	Effective Depth, L, cm
25	12.2	56	7.1
26	12.0	57	7.0
27	11.9	58	6.8
28	11.7	59	6.6
29	11.5	60	6.5
30	11.4		

Table 5—Constant K

Temp	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	15.10	15.05	14.81	14.57	14.35	14.14	13.94	13.74	13.56
17	15.11	14.86	14.62	14.39	14.17	13.96	13.76	13.56	13.38
18	14.92	14.67	14.43	14.21	13.99	13.78	13.59	13.39	13.21
19	14.74	14.49	14.25	14.03	13.82	13.61	13.42	13.23	13.05
20	14.56	14.31	14.08	13.86	13.65	13.44	13.25	13.07	12.89
21	14.38	14.14	13.91	13.69	13.48	13.28	13.09	12.91	12.73
22	14.21	13.97	13.74	13.53	13.32	13.12	12.94	12.76	12.58
23	14.04	13.81	13.58	13.37	13.17	12.97	12.79	12.61	12.43
24	13.88	13.65	13.42	13.21	13.01	12.82	12.64	12.46	12.29
25	13.72	13.49	13.27	13.06	12.86	12.67	12.49	12.32	12.15
26	13.57	13.34	13.12	12.91	12.72	12.53	12.35	12.18	12.01
27	13.42	13.19	12.97	12.77	12.58	12.39	12.21	12.04	11.88
28	13.27	13.04	12.83	12.64	12.44	12.25	12.08	11.91	11.75
29	13.12	12.90	12.69	12.49	12.30	12.12	11.95	11.78	11.62
30	12.98	12.76	12.56	12.36	12.17	11.99	11.82	11.65	11.49

## 14. TEST REPORT

14.1 Report the total percentages retained on each sieve, or the grain diameter to the nearest whole number, as follows:

14.1.1 Sieves, opening in millimeters (inches or sieve numbers):

- 75 mm (3 in.)
- 50 mm (2 in.)
- 25 mm (1 in.)
- 9.5 mm (3/8 in.)

- 4.75 mm (No. 4)
- 2.00 mm (No. 10)
- 425  $\mu\text{m}$  (No. 40)
- 75  $\mu\text{m}$  (No. 200).

14.1.2 Grain diameter:

- 0.02 mm
- 0.002 mm
- 0.001 mm.

**Note 2**—The sieve sizes listed above are suggested sizes only and may be specified only in part.

---

## 15. PLOTTING TEST RESULTS

- 15.1 The percentage of grains of different diameters is plotted on semi-logarithmic paper to obtain a grain size accumulation curve.
- 15.2 The data obtained from the hydrometer analysis are plotted as percent of material in suspension (percent passing) against corrected grain diameter in millimeters.
- 15.3 The data from the mechanical analysis are plotted as the percent retained against sieve size.
- 

## 16. REPORT FORM

- 16.1 [Tx110p2.xlsm](#), “Particle Size Analysis of Soil, Part II,” is available to automate the calculation, plot, and report.



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## Test Procedure for

# BALL MILL METHOD FOR DETERMINING THE DISINTEGRATION OF FLEXIBLE BASE MATERIAL



## TxDOT Designation: Tex-116-E

*Effective Date: June 2000*

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### 1. SCOPE

- 1.1 This method determines the resistance of aggregate in flexible base material to disintegration in the presence of water. The test provides a measure of the ability of the material to withstand degradation in the road base and detects soft aggregate that is subject to weathering. The result of this test is the Wet Ball Mill (WBM) value.
  - 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 *Wet Ball Mill machine*, consisting of a watertight steel cylinder, closed at one end, with inside dimensions of  $258.8 \pm 3$  mm ( $10.188 \pm 0.125$  in.) in diameter and  $273.1 \pm 3$  mm ( $10.75 \pm 0.125$  in.) in length.
    - 2.1.1 The cylinder is fitted with a removable lid with watertight gasket attached.
    - 2.1.2 The cylinder is mounted in a rigid support in such a manner that it is rotated about the central axis in a horizontal position.
    - 2.1.3 A steel baffle, projecting radially  $82.6 \pm 3$  mm ( $3.25 \pm 0.125$  in.) into the cylinder and  $273.1 \pm 3$  mm ( $10.75 \pm 0.125$  in.) in length, is welded along one element of the interior surface of the cylinder.
    - 2.1.4 The baffle should be of such thickness and so mounted as to be rigid.
    - 2.1.5 The machine should be operated at a uniform speed of 58–62 rpm.
  - 2.2 *Metal spheres*, used as the abrasive charge, consisting of six steel spheres approximately 47.6 mm (1.875 in.) in diameter, weighing between 390 and 445 g (0.86 and 0.98 lb.)
  - 2.3 *Balance*, Class G5 in accordance with Tex-901-K, with a minimum capacity of 15 kg (33 lb.)
-

- 2.4 *Standard U.S. sieves*, meeting the requirements of Tex-907-K, in the following sizes:
- 45 mm (1 3/4 in.)
  - 31.5 mm (1 1/4 in.)
  - 22.4 mm (7/8 in.)
  - 16.0 mm (5/8 in.)
  - 9.5 mm (3/8 in.)
  - 4.75 mm (No. 4)
  - 2.00 mm (No. 10)
  - 425  $\mu$ m (No. 40).
- 2.5 *Oven*, air-dryer, with temperature set to  $60 \pm 5^{\circ}\text{C}$  ( $140 \pm 9^{\circ}\text{F}$ ).
- 2.6 *Crusher*, (optional).
- 2.7 *Miscellaneous equipment*, includes large pans, wash bottles, etc.
- 2.8 *Container*, 2 L (0.5 gal.)
- 

### **3. TEST RECORD FORM**

- 3.1 Each sample should be given an identification number and a card bearing the number should be placed with each portion of the sample throughout the processing and testing of the material.
- 

## **PART I—DETERMINING DISINTEGRATION**

---

### **4. PROCEDURE**

- 4.1 Secure a representative sample of the total material of approximately 12,000 g (26.5 lb.)
- 4.2 Air-dry the sample at  $60^{\circ}\text{C}$  ( $140^{\circ}\text{F}$ ).
- 4.3 Prepare sample in accordance with Tex-101-E, Part II.
- 4.4 Recombine a 5000 g (11 lb.) sample for soil constants testing as shown in the governing specifications if needed.
- 4.5 Replace oversized particles retained on the 45 mm (1-3/4 in.) sieve with particles passing the 45 mm (1-3/4 in.) and retained on the 31.5 mm (1-1/4 in.) sieve.
- 4.6 Recombine a  $3500 \pm 50$  g ( $7.7 \pm 1.1$  lb.) air dried sample, weigh to the nearest whole gram, and record as A under Section 5. Place sample in pan and cover with 2 L (0.5 gal.)
-

water for one hour. If 2 L (0.5 gal.) of water do not fully cover the sample, use the smallest amount of water possible to do so.

**Note 1**—Use the dry sieve analysis as a rough check for specification compliance for gradation prior to testing for soil constants and Wet Ball Mill.

- 4.7 Decant all free water from sample into a 2 L (0.5 gal.) container, finish filling container with clear water and use to wash sample into the mill.
- 4.8 Place the six steel spheres in the mill, fasten the watertight lid securely, and rotate 600 revolutions at the uniform speed of 58–62 rpm.
- 4.9 When the 600 revolutions are completed, remove cover and empty cylinder contents into a pan.
- 4.10 Remove the steel spheres and separate the sample by washing over the 425 µm (No. 40) sieve.
- 4.11 Dry the aggregate portion retained on the 425 µm (No. 40) sieve to a constant mass at 60°C (140°F). Rescreen over the 425 µm (No. 40) sieve. Weigh the mass to the nearest whole gram and record as B under Section 5.
- Note 2**—Additional sieves may be utilized to determine particle size degradation during the test.

---

## 5. CALCULATIONS

- 5.1 Calculate the percentage of the soil binder from the Wet Ball Mill test:

$$\text{Wet Ball Mill Value, WBM} = 100 \cdot (A - B) / A$$

Where:

*A* = dry weight of total sample

*B* = weight of retained material.

---

## PART II—DETERMINING INCREASE OF MINUS (-) 425 µM (NO. 40) MATERIAL

---

### 6. PROCEDURE

- 6.1 Recombine approximately 3000 g (6.6 lb.) of the original prepared sample in Section 4.3.
- 6.2 Weigh to the nearest whole gram and record the mass as C under Section 7.
- 6.3 Wash the sample over a 425 µm (No. 40) sieve.

- 6.4 Dry the aggregate portion retained on the 425  $\mu\text{m}$  (No. 40) sieve to a constant weight at 60°C (140°F). Rescreen over the 425  $\mu\text{m}$  (No. 40) sieve and weigh the amount retained to the nearest whole gram. Record the mass as D under Section 7.
- 

## 7. CALCULATIONS

- 7.1 Calculate the original percentage of material passing the 425  $\mu\text{m}$  (No. 40) sieve:

$$\text{Original percentage of minus } (-) 425 \mu\text{m (No. 40) material} = 100 \bullet (C - D) / C$$

Where:

*C* = original mass of sample

*D* = mass of material retained on the 425  $\mu\text{m}$  (No. 40) sieve.

- 7.2 Calculate the percent increase of material passing the 425  $\mu\text{m}$  (No. 40) sieve:

$$\text{Percent Increase} = \text{WBM} - \text{Original \% of minus No. 40}$$

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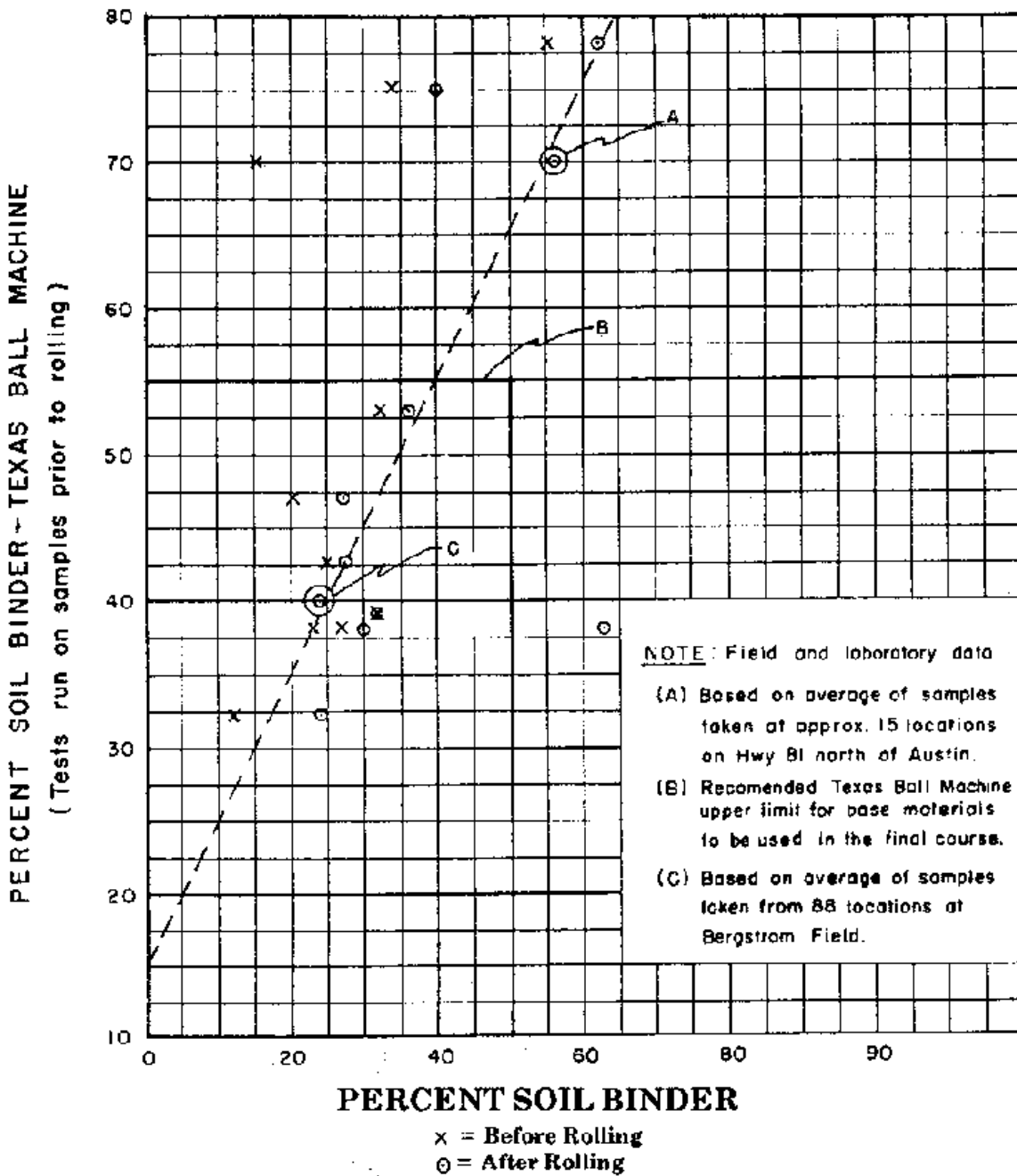
## 8. PRECAUTIONS

- 8.1 Always use dry material in performing test.
- 8.2 Avoid the loss of portions of sample in transferring into or out of cylinder.
- 8.3 Use only 2 L (0.5 gal.) of water to wash the decanted sample into the cylinder.
- 8.4 Check mass of steel spheres periodically for loss due to wear.
- 

## 9. REPORTING TEST RESULTS

- 9.1 Report the Wet Ball Mill value to the nearest whole number.

**Note 3**—This test furnishes valuable supplementary data pertaining to the quality of the aggregate portion of flexible base material. The Wet Ball Mill test is more reliable than the Los Angeles abrasion test in evaluating the quality of base materials.



**Figure 1**—Relation Between Percent Soil Binder from Texas Ball Machine and Percent Soil Binder Before and After Rolling

**10. ARCHIVED VERSIONS**

10.1 Archived versions are available.



## Preparing Samples for Compaction and Triaxial Tests

Sample ID: \_\_\_\_\_

### Tex-101-E, Part II

### Tex-116-E

Sieve Size	Individual Weight Retained	Individual Percent Retained	Cumulative Percent Retained	PART 1 Wet Ball 3500 gr	PART II Increase -#40 3000 gr
1-3/4"					
1-1/4"					
7/8"					
5/8"					
3/8"					
#4					
#40					
- #40					
Total					

#### Part 1: Wet Ball Mill

#### Part II: Washed Sieve Analysis

Initial Weight: \_\_\_\_\_

Initial Weight: \_\_\_\_\_

Sieve Size	Weight Retained (g)	Individual Percent Retained
No. 40		
- No. 40		

Sieve Size	Weight Retained (g)	Individual Percent Retained
No. 40		
- No. 40		

Wet Ball Mill Value: \_\_\_\_\_

Percent Soil Binder: \_\_\_\_\_

% Soil Binder Increase: \_\_\_\_\_

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## Test Procedure for

# SAMPLING FLEXIBLE BASE, STONE, GRAVEL, SAND, AND MINERAL AGGREGATES



TxDOT Designation: Tex-400-A

Effective Date: January 2010

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## 1. SCOPE

- 1.1 This method describes procedures for sampling flexible base material, stone, gravel, sand, and synthetic aggregates including shell, slag, riprap, and mineral filler.
  - 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. SECURING REPRESENTATIVE FIELD SAMPLES

- 2.1 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. Samples taken from a quarry or pit for a source investigation may be taken from various existing layers or strata, and the samples should be combined in proper proportion to represent the entire pit face that will be processed in the same operation.
- 2.2 Use sampling tubes where experience has indicated representative samples cannot be obtained otherwise. Sample fine aggregates (sands) with tubes of at least 31.5 mm (1-1/4 in.) diameter and coarse aggregates up to 19.0 mm (3/4 in.) with tubes of 100 mm (4 in.) diameter.

**Note 1**—The method of sampling will depend on the place, the quantity of the material, and the proposed treatment and tests to be performed in the laboratory. Where practical, take samples of the finished product from commercial plants.

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## 3. RECORD FORM

- 3.1 Identify samples on [Form 202](#), "Identification of Material Samples." In addition to the general information accompanying all samples, show the:
    - name of the producer,
    - name of the pit or quarry,
    - producer code number,
    - location of the stockpile sampled (pit, HMAC or concrete plant, project, etc.),
-

- approximate quantity of the material in the stockpile or unit sampled, and
- specification item number.

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#### **4. SAMPLE SIZE**

- 4.1 The required sample size depends upon the maximum aggregate size and the number and types of tests to be performed. Use Table 1 to determine the minimum size sample required. Use a sample splitter or quartering cloth to reduce the field sample to laboratory test size.

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#### **5. SAMPLING PROCEDURES**

##### *Riprap:*

- 5.1.1 Obtain four to six pieces of riprap representative of the proposed material.
- 5.1.2 Reduce the entire sample to a maximum particle size of 150 mm (6 in.)
- 5.1.3 Submit approximately 50 kg (110 lb.) of the material retained on the 4.75 mm (No. 4) sieve.

##### *From Bins or Belts:*

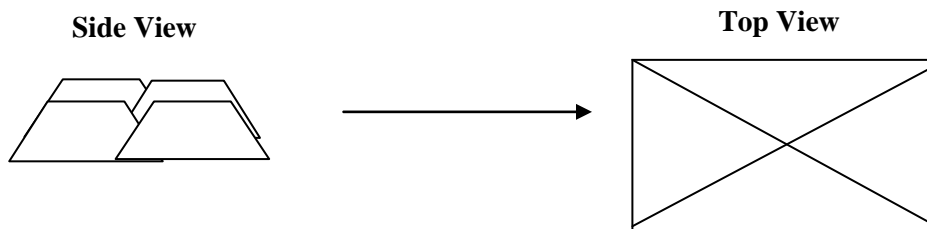
- 5.2.1 For preliminary investigation work, sample aggregates in their natural condition prior to plant processing.
- 5.2.2 If the materials are being fed to the plant from bins, obtain the sample from the collector belt after materials have passed the scalping screen.
- 5.2.3 The preferred method of sampling from the belt is a mechanical belt sampling device approved by the Engineer. This method does not require the belt to be stopped, but simply diverts the flow of aggregate for a designated period where the sample is collected.
- 5.2.4 When mechanical devices are not used, stop the belt prior to sampling, and take the sample from the entire width of the belt and at least one meter of the length, using a square-nosed scoop.
- 5.2.5 After removing the aggregate, brush all fines from the exposed section of the belt using a stiff bristled brush and add to the sample.

##### *From Stockpiles that Contain Coarse and Fine Aggregate:*

- 5.3.1 Identify locations around the perimeter of the stockpile that represent the approximate quarter-points of the stockpile. Clean and level the ground at these locations to prevent contamination of the sampling pile.
- 5.3.2 Sample each quarter-point of the stockpile using the following steps.



- 5.3.2.1 Using a front-end loader large enough to cut into the stockpile from bottom to top in one continuous cut, make enough cuts into the stockpile at the quarter-points so that the cut at ground level is perpendicular to the top edge of the stockpile at that quarter-point. If available, the blade of the loader bucket must be straight and flat, without teeth. Only use a loader with a bucket containing teeth when a loader with a bucket that is straight, flat, and without teeth is not available. Make the cuts from the bottom to the top to expose a clean, interior vertical face representing the full height of the stockpile. Clean out the cut as needed to leave a minimal amount of material on the ground before obtaining the sample.
- 5.3.2.2 Discard this material.
- 5.3.2.3 Using the front-end loader, cut into the exposed interior face to a depth equal to approximately half the depth of the loader bucket, taking care not to overload the bucket in the bottom half of the stockpile. Make the cut from the bottom to the top in one continuous motion.
- 5.3.2.4 Lower the bucket until it is as close as possible to the surface of the ground, and empty the entire contents of the bucket onto the ground in one motion without having to raise the bucket before all contents are emptied. Repeat this process three more times, each time emptying the loader bucket next to the last load. (See Figure 1.)



**Figure 1**—Placement and Sampling of Piles

- 5.3.2.5 Using the loader bucket, strike, and level the sample piles at mid-height in the direction the bucket was emptied (See Figure 1.)
- 5.3.2.6 Using a shovel, identify the approximate mid-point of each bucketload, and dig straight down to remove material for sampling. Deposit the material removed from the hole into sample containers (preferably 5 gal. buckets). 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 containers. Remove enough material from each hole to fill one sample container for each party who will receive a portion of the sample. This will give you four containers of sampled material for each quarter-point of the stockpile sampled.
- 5.3.2.7 Seal and label the sample containers. Include the quarter point location on the label.
- 5.3.3 Repeat Sections 5.3.2.1–5.3.2.7 at each of the stockpile quarterpoints identified in Section 5.3.1.

- 5.3.4 Department personnel will divide the total sample into three equal portions and offer one portion of the sample to the producer, retain one portion for district testing, and retain one portion for CST/M&P for referee and random blind samples.
- 5.4 *From Stockpiles that Contain Only Coarse or Only Fine Aggregate:*
  - 5.4.1 Take samples from stockpiles near the top of the pile, near the base of the pile, and at an intermediate point.
  - 5.4.2 Shove a board into the pile just above the point of sampling to prevent further segregation during sampling.
  - 5.4.3 Do not use the aggregate on the surface of the stockpile as a part of the sample.
  - 5.4.4 In each instance, dig a small trench or hole into the pile approximately 1 ft. deep and take the sample from the innermost part of the hole.
  - 5.4.5 Take samples from these three points at several places around the stockpile and combine them to form a composite sample.
  - 5.4.6 In lieu of using Section 5.4, steps outlined in Section 5.3 are acceptable.
- 5.5 *From Railroad Cars and Trucks:*
  - 5.5.1 Excavate at least three trenches across the width, evenly spaced across the length of the bed.
  - 5.5.2 Each trench bottom should be approximately 0.3 m (1 ft.) in width and 0.3 m (1 ft.) in depth.
  - 5.5.3 Take samples from at least three locations equally spaced along the length of the trench, and combine all samples to create a single field sample as directed in Tex-100-E.
- 5.6 *From the Plant:*
  - 5.6.1 Make a general inspection of the plant and a record of the screening facilities.
  - 5.6.2 For a weigh-batching type plant, take samples from the aggregate bins that feed into the weigh-box.
  - 5.6.3 For a continuous mix type plant, take samples from the aggregate bins that feed the proportioning mechanism.
  - 5.6.4 For a drum type plant, take samples from the collector belt feeding the aggregate into the drum. When an aggregate flow diversion chute is not available, stop the feed belt prior to obtaining the sample.
  - 5.6.5 Take samples from the entire cross-section of the flow of material from each individual bin as it is being discharged. Prior to sampling, permit the plant to produce a minimum of 4.5 Mg (5 tons) of mixture.

**Table 1—Minimum Size of Samples**

Nominal Maximum Size Of Particles <sup>1</sup>	Approximate Minimum Mass Of Field Samples, kg (lb.)
2.00 mm (No. 10)	10 (25)
4.75 mm (No. 4)	10 (25)
9.5 mm (3/8 in.)	10 (25)
12.5 mm (1/2 in.)	15 (35)
19.0 mm (3/4 in.)	25 (55)
25.0 mm (1 in.)	50 (110)
37.5 mm (1-1/2 in.)	75 (165)
50.0 mm (2 in.)	100 (220)
63.0 mm (2-1/2 in.)	125 (275)
75.0 mm (3 in.)	150 (330)

1. For processed aggregate, the nominal maximum size of particles is the largest sieve size listed in the applicable specification upon which any material is permitted to be retained.

**Note 2**—Obtain the test sample from the field sample by quartering or splitting.

## 6. ARCHIVED VERSIONS

6.1 Archived versions are available.

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**Test Procedure for****SAMPLING AND TESTING LIME****TxDOT Designation: Tex-600-J****Effective Date: February 2006**

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**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.

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**PART I—SAMPLING LIME PRODUCTS**

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**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.*

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- 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.*

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## 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.

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## PART II—TESTING HYDRATED LIME

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### 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.

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### 6. APPARATUS

6.1 *Electric muffle furnace*, at  $2,000 \pm 20^\circ\text{F}$  ( $1093 \pm 11^\circ\text{C}$ ).

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  $2,000^\circ\text{F}$  ( $1093^\circ\text{C}$ ).

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  $212^\circ\text{F}$  ( $100^\circ\text{C}$ ).

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  $\pm 0.1$  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  $\times$  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.

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## 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.

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## 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  $1.000 \pm 0.0005$  N, add water or sodium hydroxide to adjust the normality.
- 8.1.2.9 If the sodium hydroxide solution is too:
- weak, follow “Example 1” below to calculate the amount of the 50% sodium hydroxide to add.
  - strong, follow “Example 2” below to calculate the amount of deionized water to add.
- 8.1.2.10 Measure and add the correct amount of sodium hydroxide or deionized water. Stir the solution for at least 4 hr.
- 8.1.2.11 Repeat the standardization procedure. Repeat adjustments, if necessary. After all adjustments are complete, standardize the resulting solution.
- 8.1.3 *Calculations:*
- 8.1.3.1 Calculate the normality of sodium hydroxide solution:
- $$\text{Normality of NaOH} = \frac{\text{Wt. of Potassium Hydrogen Phthalate in (g)}}{\text{mL of NaOH required} \times 0.204228}$$
- 8.1.3.2 Adjusting the sodium hydroxide solution to achieve a 1.0 N solution:
- 8.1.3.2.1 Example 1: If base is too weak, for example, if 18.5 L of 0.9907 N solution needs strengthening to 1.0000 N, calculate the amount of 50% sodium hydroxide to add:
- $18.500 \times 0.991 = 18.334$
  - $18.500 - 18.334 = 0.166$
  - $0.166 \times 52.3 = 8.68$  mL of 50% sodium hydroxide solution needed.
- 8.1.3.2.2 Example 2: If base is too strong, for example, if the 18.5 L of 1.021 N sodium hydroxide solution needs weakening to 1.0000 N, calculate the amount of deionized water to add:
- $18.500 \times 1.021 = 18.888$
  - $18.888 - 18.500 = 0.388$  L or 388 mL of deionized water needed.
- 8.2 *1.0 N Hydrochloric Acid Solution:*
- 8.2.1 *Preparation:*
- 8.2.1.1 Fill a 19-L glass container 1/2–2/3 full with deionized water.

- 8.2.1.2 Add 1625 mL of concentrated hydrochloric acid.  
**Note 11**—It is important to add the acid to a sizable portion of water to dissipate the heat evolved and avoid hazardous spattering of hot acid, fuming, etc.
- 8.2.1.3 Add sufficient water to bring the total volume to 19 L.
- 8.2.1.4 Stir the solution at least 4 hr., but preferably 12 hr.
- 8.2.1.5 Standardize against the sodium hydroxide.
- 8.2.2 *Standardization of a  $1.0 \pm 0.0005$  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  $1.000 \pm 0.0005$  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:

- $19.700 \times 0.991 = 19.523$
- $19.700 - 19.523 = 0.177$
- $0.177 \times 82.5 = 14.6$  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:

- $19.700 \times 1.024 = 20.1732$
- $20.173 - 19.700 = 0.473$  L or 473 mL of deionized water needed.

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

8.3.1 Weigh  $10.21 \pm 0.05$  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  $3.81 \pm 0.01$  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.

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**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.

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**10. TESTING PROCEDURES**

- 10.1 *Titration to pH 8.3:*
- 10.1.1 Weigh out on an analytical balance  $2.804 \pm 0.0050$  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  $2,000 \pm 20^\circ\text{F}$  ( $1093 \pm 11^\circ\text{C}$ ) 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  $12 \pm 0.3$  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  $212 \pm 4^\circ\text{F}$  ( $100 \pm 2^\circ\text{C}$ ).
- 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.  $2.804 \pm 0.0050$  g
  - Acid  $1.000 \text{ N} \pm 0.0005 \text{ N}$
  - Base  $1.000 \text{ N} \pm 0.0005 \text{ N}$ .

- 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  $\text{Ca(OH)}_2$  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:

$$\text{Uncorrected Ca(OH)}_2 = 94.8$$

$$\text{CaCO}_3 = 3.1$$

$$\text{Assumed inert} = 2.2$$

$$\overline{100.1}$$

$$\text{Subtotal } 100.1 - 100 = 0.1$$

$$\% \text{CaCO} = 0.1 \times 3.11 = 0.3$$

$$\text{Uncorrected Ca(OH)}_2 = 94.8$$

$$\text{Ca(OH)}_2 \text{ Correction Value} = -0.4$$

$$\overline{94.4}$$

Analysis shown as :

$$\text{Ca(OH)}_2 = 94.4$$

$$\text{CaCO}_3 = 3.1$$

$$\text{CaO} = 0.3$$

$$\text{Total} = \overline{100.0}$$

- Example where water is present:

$$\begin{array}{rcl}
 \text{Uncorrected } Ca(OH)_2 & = & 93.0 \\
 CaCO_3 & = & 4.6 \\
 \text{Assumed Inert} & = & 1.0 \\
 \text{Total} & = & \overline{98.6} \\
 \% H_2O = 100 - 98.6 & = & 1.4
 \end{array}$$

Analysis is shown as:

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

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) <sub>2</sub>
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
<b>Factor = 1.32126</b>	

11.2.8 Calcium Carbonate Value as CaCO<sub>3</sub> —Whole milliliter difference between two endpoints:

**Table 4—Calcium Carbonate Value**

Carbonate Alkalinity	CaCO <sub>3</sub>
0.0	0.0
1.0	1.8
2.0	3.6
3.0	5.4
4.0	7.1
<b>Factor = 1.78479</b>	

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) <sub>2</sub>
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
<b>Factor = 1.32126</b>	

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 <sub>3</sub>
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
<b>Factor = 1.78479</b>	



- 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  $2.804 \pm 0.0050$  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.

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## PART III—TESTING COMMERCIAL LIME SLURRY

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### 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.

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### 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.

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**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.

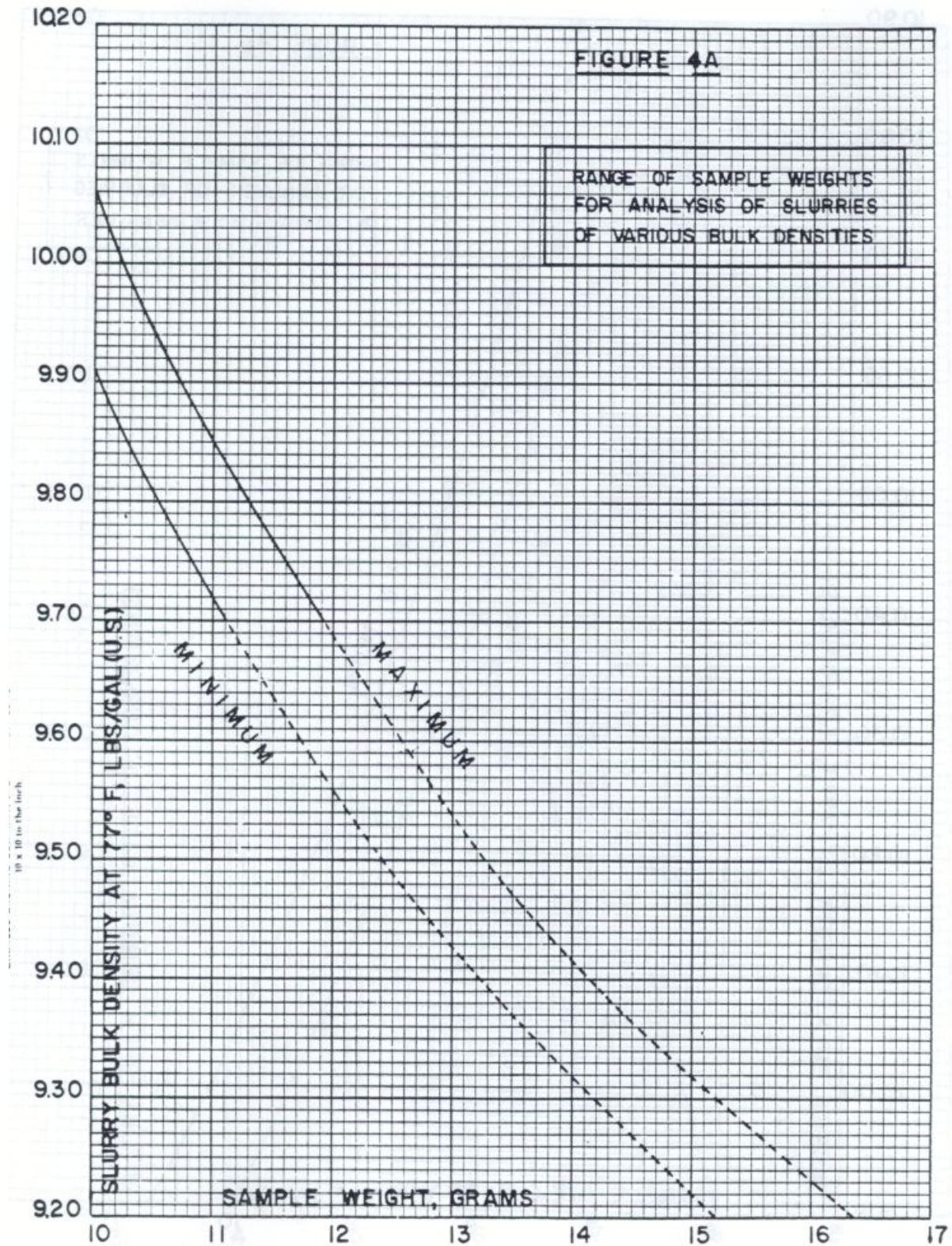


Figure 1—Range of Sample Weights for Analysis of Slurries—No. 1



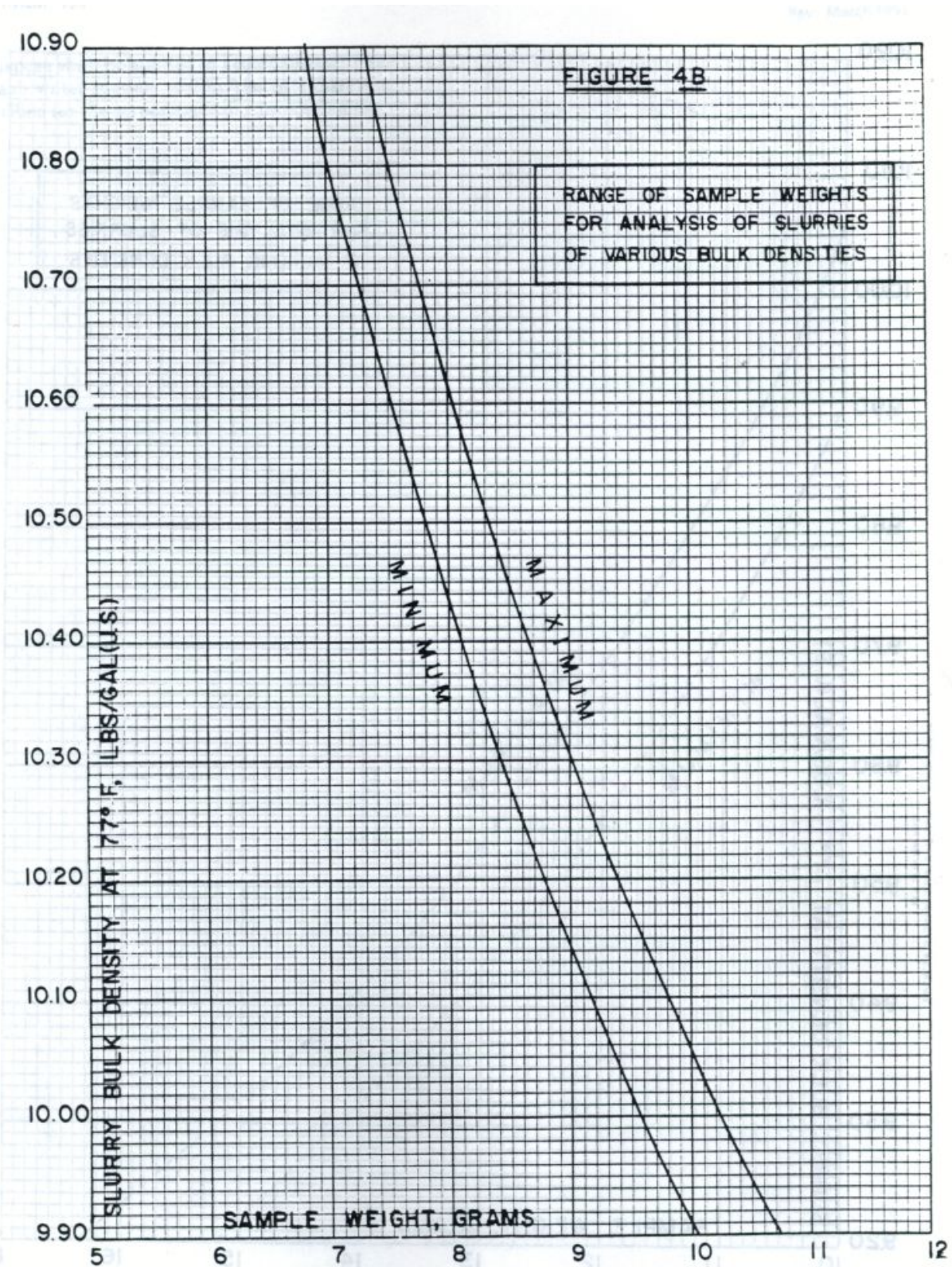


Figure 2—Range of Sample Weights for Analysis of Slurries—No. 2



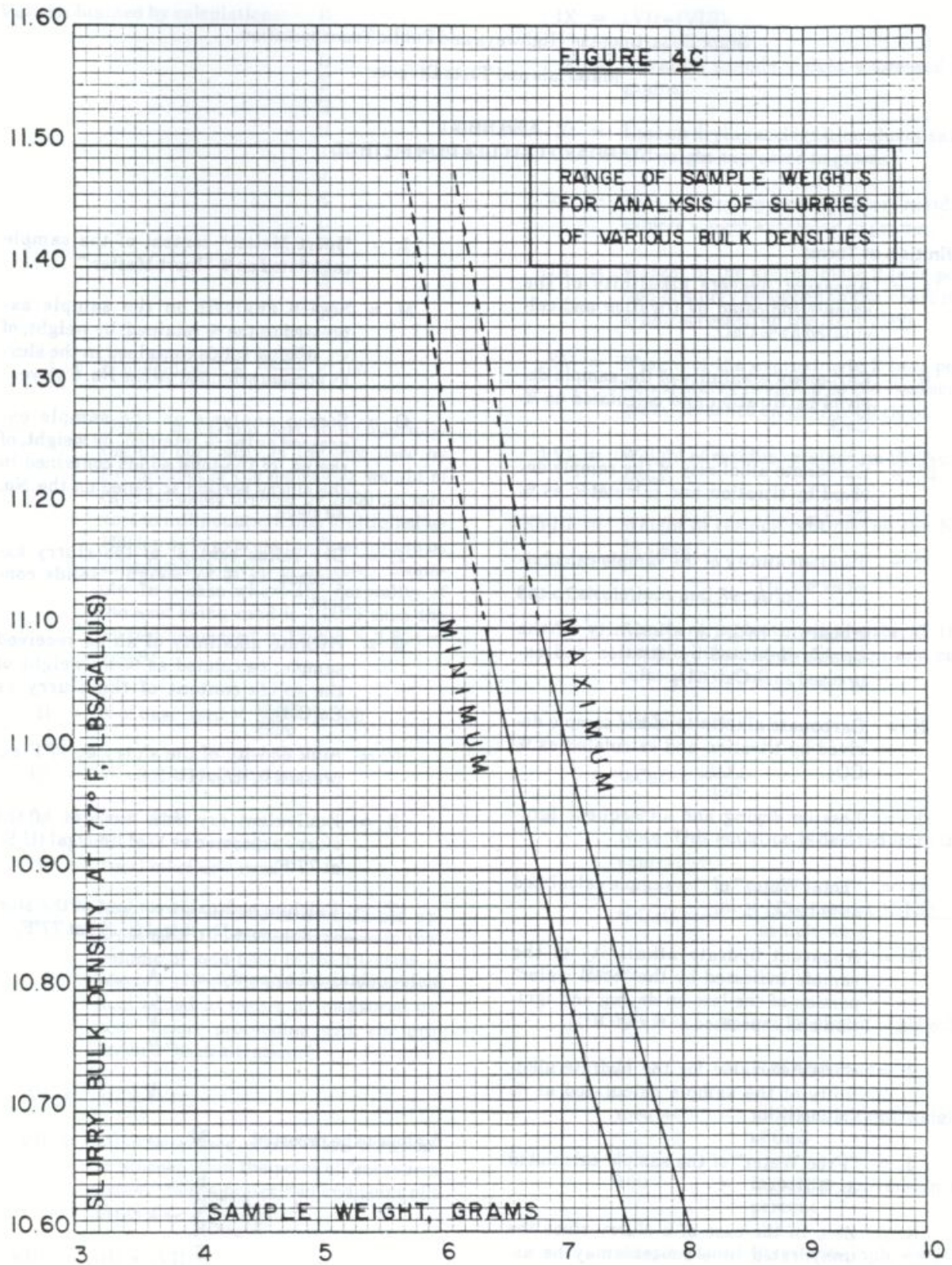


Figure 3—Range of Sample Weights for Analysis of Slurries—No. 3

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**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  $212 \pm 4^\circ\text{F}$  ( $100 \pm 2^\circ\text{C}$ ) drying oven for 1 hr.
- 17.3.4 Remove sample from oven and place in a muffle furnace at  $2,000 \pm 20^\circ\text{F}$  ( $1093 \pm 11^\circ\text{C}$ ) 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  $12 \pm 0.3$  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  $212 \pm 4^\circ\text{F}$  ( $100 \pm 2^\circ\text{C}$ ).
- 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:

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

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:

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

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:

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

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:

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

Where:

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

- 18.1.7 Calculate  $F$ , loss on ignition:

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

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:

$$G = F - E = (\text{loss on ignition}) - (\text{carbonate alkalinity, CO}_2)$$

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:

$$H = (4.1128)G = (4.1128)(\text{total water})$$

Where:

$G$  = total water of the sample, %.

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

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

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

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)<sub>2</sub>:*

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

Where:

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

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

18.3 *Loss on Ignition:*

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

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

18.4 *Sieve Analysis:*

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

Where:

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

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

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

% solids = percent solids as calculated above.

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**PART IV—TESTING QUICKLIME**

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**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.

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**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.

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**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.

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**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.

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- 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  $2.8 \pm 0.3$  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  $212 \pm 4^{\circ}\text{F}$  ( $100 \pm 2^{\circ}\text{C}$ ) 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.*
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## 28.            REAGENTS AND SOLUTIONS

- 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.
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## 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  $212 \pm 4^\circ\text{F}$  ( $100 \pm 2^\circ\text{C}$ ) drying oven for 1 hr.

30.3.4 Remove sample from oven and place in a muffle furnace at  $2,000 \pm 20^\circ\text{F}$  ( $1093 \pm 11^\circ\text{C}$ ) 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  $12 \pm 0.3$  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  $212 \pm 4^\circ\text{F}$  ( $100 \pm 2^\circ\text{C}$ ).
- 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:

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

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:

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

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:

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

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:

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

Where:

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

31.1.7 Calculate  $F$ , loss on ignition:

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

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:

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

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:

$$H = (4.1128)G = (4.1128)(\text{total water})$$

Where:

$G$  = total water of the sample, %.

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

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

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

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)<sub>2</sub>:

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

Where:

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

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

31.3 Calculate loss on ignition:

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

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, %:

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

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.