

SB 102

Field Specialist

CERTIFICATION MANUAL



**Partners
in Quality**





The Hot Mix Asphalt Center exists to certify, train, and equip specialists with the knowledge and skills needed to inspect, test, and design construction materials. Through education and support that continue beyond certification, the HMAC seeks to help people realize their potential to contribute to the future of Texas' infrastructure.

To execute our mission, we constantly aim to:

EDUCATE

Through hands-on learning, training, and testing, our instructors ensure specialists not only have a thorough understanding of the subject, but will be able to apply what they've learned in a real-world setting. We believe our role as an educational resource doesn't stop at the door – it continues throughout a specialist's career.

INNOVATE

From installing the latest, cutting-edge equipment in our labs to streamlining processes and developing new educational tools, we strive to continuously set a higher bar for ourselves and those around us.

EMPOWER

We believe in the potential of every specialist who comes through the center and do all we can to help them be successful before, during, and after certification. Our mantra is that what you do matters. If you put in the work to be the best you can be, we'll be here to cheer you on and support you along the way.

SERVE

Service is at the core of everything we do. Whether it be for individuals, companies, TxDOT, or the traveling public, the goal is for every certification to result in improved quality of asphalt pavements across the state of Texas.



SB102 Field Specialist

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HMAC CERTIFIED SPECIALIST & APPLICANTS FOR CERTIFICATION: RIGHTS AND RESPONSIBILITIES

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

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

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

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

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

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

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

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

ALLEGATIONS OF MISCONDUCT - Allegations of misconduct should be submitted to the HMAC, P.O. Box 1468, 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.



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

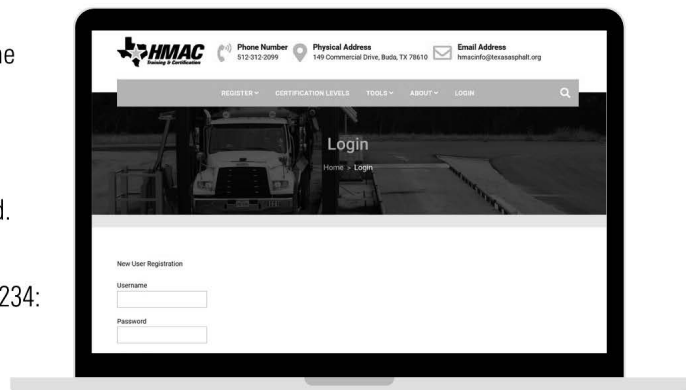
Same as your username, but add a "+" at the end.

Example

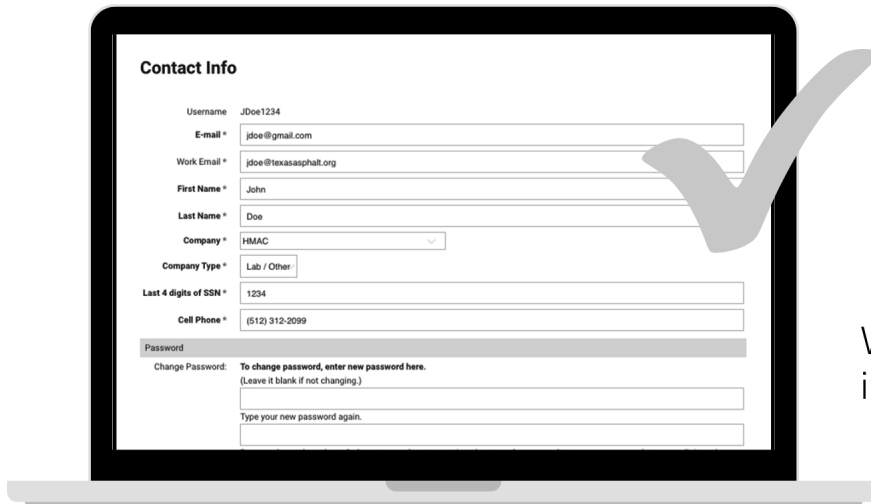
If your name is John Doe and your SSN ends in 1234:

Username: JDoe1234

Password: JDoe1234+



UPDATE INFORMATION



Contact Info

Username JDoe1234

E-mail * jdoe@gmail.com

Work Email * jdoe@texasasphalt.org

First Name * John

Last Name * Doe

Company * HMAC

Company Type * Lab / Other

Last 4 digits of SSN * 1234

Cell Phone * (512) 312-2099

Password

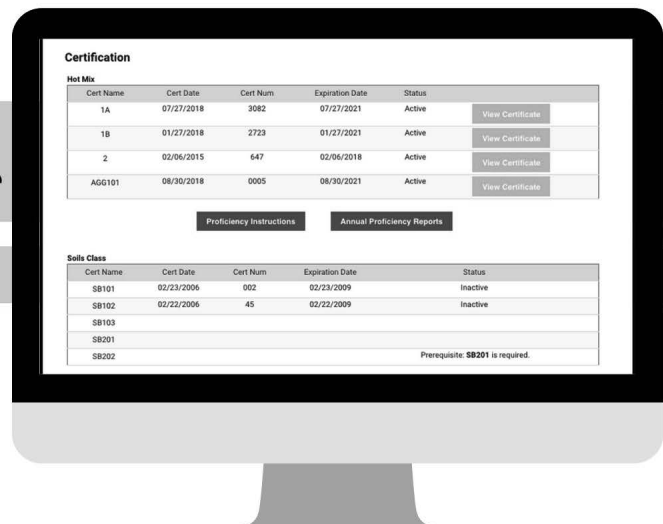
Change Password: To change password, enter new password here.
(Leave it blank if not changing.)

Type your new password again.

Verify that all
information is correct.

UPDATE INFORMATION

**VIEW/PRINT
CERTIFICATIONS
PROFICIENCY**



Certification

Hot Mix

Cert Name	Cert Date	Cert Num	Expiration Date	Status	
1A	07/27/2018	3082	07/27/2021	Active	View Certificate
1B	01/27/2018	2723	01/27/2021	Active	View Certificate
2	02/06/2015	647	02/06/2018	Active	View Certificate
AGG101	08/30/2018	0005	08/30/2021	Active	View Certificate

[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
SB102	02/23/2006	45	02/22/2009	Inactive
SB103				
SB201				
SB202				

Prerequisite: SB201 is required.

Test Procedure for**SAMPLING SOILS AND BASE MATERIALS****TxDOT Designation: Tex-100-E****Effective Date: July 2024**

1. SCOPE

- 1.1 Use this procedure to sample soils that are used as embankment and non-select backfill materials, sample flexible base from completed stockpiles and roadway windrows, prepare flexible base from completed stockpiles and roadway windrows for laboratory testing, and to sample material from the roadway for reclamation and treatment.
 - 1.2 This procedure also provides information to develop a soil survey that may be used for the design, location, and construction of a highway.
 - 1.3 *This test procedure does not claim to address the safety concerns associated with its use. It is the responsibility of the user of this test procedure to establish the appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations before use.*
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2. APPARATUS

- 2.1 The type and amount of equipment to be used for sampling will depend on the nature of the terrain, the material, and the depth of material below the surface.
 - 2.1.1 Use small hand tools for materials that are shallow in depth and can be easily dug. Power equipment may be used when the materials are hard. Use of a power drill machine with a core or auger attachment may be necessary for sampling strata located at a considerable depth below the surface.
 - 2.2 *Containers, jars or plastic bags, capable of being sealed to maintain moisture content.*
 - 2.3 *Hand tools, such as post hole digger, scoop, shovel, anything small to sample material.*
 - 2.4 *Measuring tape, steel or plastic.*
 - 2.5 *Power drill rig, with core or auger attachments.*
 - 2.6 *Ruler, minimum 6 ft.*
 - 2.7 *Sample bags or buckets with lids.*
 - 2.8 *Sample splitter or quartering cloth*
 - 2.9 *Soil Auger - Screw-type, open tubular, or barrel auger types.*
 - 2.10 *Square-Tip Shovel.*
-

3. SAMPLING EMBANKMENT SOIL AND NON-SELECT BACKFILL

- 3.1 Obtain a representative sample from undisturbed or disturbed material in the same proportion as they exist in the embankment soil, non-select backfill, or roadway for reclamation and treatment.
- 3.2 Equipment, method, and tools for obtaining a sample will depend on the location, the quantity of material needed, and the tests to be performed in the laboratory.
- 3.3 Samples should contain only materials of like color and texture. They should not be a composite of materials different in character and properties unless different types of materials are to be uniformly mixed in certain proportions.
- 3.4 When sampling material for treatment (road-mixed), ensure to sample to the depth of treatment as shown on the plans.
- 3.5 When coring, obtain a core of soil from the earth with as little disturbance as possible to the natural density and moisture content.
- 3.5.1 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.
- 3.5.2 A soil core is satisfactory for all practical purposes and can be classified as an undisturbed sample of soil.
- 3.6 Seal and label the sample bags, buckets, containers, or jars, and properly secure for transportation to avoid any loss of material.

4. SAMPLING FLEXIBLE BASE STOCKPILES

- 4.1 Identify four locations around the perimeter of the stockpile that represent the approximate quarter-points of the stockpile.
- 4.1.1 When the locations cannot be obtained from around the entire perimeter due to limited space, use four equally spaced locations.
- 4.2 Clean and level the ground at these four locations to prevent contamination of the sampling pile.
- 4.3 Sample each quarter-point of the stockpile using a front-end loader to cut into each quarter-point.
- 4.4 Cut at the ground level to the top edge of the stockpile until a clean vertical face is exposed that is perpendicular to the top edge of the stockpile. This represents the full height of the stockpile.
- 4.5 Discard this material cut away while exposing the clean face.
- 4.6 Build a sample pad by cutting into the vertical face at the ground level of the full height of the stockpile to obtain material.
- 4.7 Lower the bucket as close as possible to the ground to avoid segregation and empty the entire contents of the bucket onto the ground in one motion.
- 4.8 Using the loader bucket, strike, and level the sample pad at mid-height in the direction the bucket was emptied to create a flat surface for sampling. Back-drag the sampling pad only once.

- 4.9 If material is visually segregated, discard the material and repeat Sections 4.6 – 4.8.
 - 4.10 Place clean sample bags or containers near the center of the sampling pad and obtain the sample across the flat area staying more than 1 ft. away from the edges.
 - 4.11 Divide the sample pad into four quadrants and sample equal amounts of aggregate evenly across each quadrant.
 - 4.12 Fully insert a square-tip shovel as near as vertical as possible and then slowly roll the shovel back and lift slowly to avoid coarse aggregate from rolling off the sides of the shovel.
 - 4.12.1 Spade-tip shovels are not allowed for sampling because they will not prevent material from rolling off the side of the shovel.
 - 4.13 Obtain additional shovelfuls from different quadrants of the sampling pad, and in areas avoiding previous shovel holes. Remove material from each quadrant to fill one sample bag or container. Minimize loose material falling from the sides of the hole and loss of material from the shovel into the hole or adjacent ground while filling sample bags or containers.
 - 4.14 Place the aggregate into the clean sample bags or containers.
 - 4.15 Repeat Sections 4.12 – 4.14 until a minimum of 100 lbs. of material is sampled from each sample pad.
 - 4.16 Seal and label the sample bags or containers, and properly secure for transportation to avoid any loss of material.
 - 4.17 Repeat Sections 4.4 – 4.16 at each stockpile quarter-point to provide a minimum of 400 lbs. of material.
 - 4.17.1 The minimum amount of 400 lbs. of sampled material is representative of the entire stockpile. This amount of material is required for a testing laboratory to perform all the required test procedures for stockpile approval.
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5. SAMPLING FLEXIBLE BASE WINDROWS

- 5.1 Sampling material from a windrow for acceptance may only be tested for liquid limit (Tex-104-E) and plastic limit (Tex-105-E) to determine the plasticity index (Tex-106-E), or gradation (Tex-110-E).
- 5.1.1 When testing material for wet ball mill (Tex-116-E) or compressive strength (Tex-117-E) for acceptance, sampling must be from a stockpile in accordance with Section 4.
- 5.2 Choose two locations that are within 500 ft. apart.
- 5.2.1 Avoid sampling from the ends of the windrow section by choosing a location that is more than 10 ft. from the end.
- 5.2.2 Choose locations that appear uniform and not segregated.
- 5.3 Refer to the test procedure for the minimum sample size.
- 5.4 Use a square-tip shovel and remove approximately the top one ft. of material.
- 5.5 Sample material from the top, flattened area of the windrow from each location.
- 5.5.1 Avoid sampling from the sides of the windrow.

- 5.5.2 Avoid any segregated coarser material.
 - 5.6 Seal and label the sample bags or containers, and properly secure for transportation to avoid any loss of material.
 - 5.7 Proceed to Section 6 to prepare the sample for testing.
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6. PREPARING FLEXIBLE BASE SAMPLES FOR TESTING

- 6.1 Allow the material to air dry or oven dry at a maximum temperature of 140°F for a minimum of 4 hr. until the material is sufficiently dry for handling.
 - 6.2 Quarter the material by emptying each container or sample bag onto a clean floor or a clean tarp. Optionally, use a mechanical quartering device or sample splitter to quarter the material and proceed to Section 6.6.
 - 6.3 Thoroughly mix the material using a shovel. When using a tarp, the ends of the tarp may also be used to also mix the material.
 - 6.4 Spread the material into the shape of a circle of uniform thickness and homogenous with no segregation.
 - 6.5 Using a shovel or straightedge, visibly trace lines on top of the material to outline four evenly sized quarters.
 - 6.6 Use a quarter of the sample and proceed to [Tex-101-E](#), Part I to prepare and test the material for the liquid limit ([Tex-104-E](#)) and plastic limit ([Tex-105-E](#)) tests to determine the plasticity index ([Tex-106-E](#)); and for the gradation ([Tex-110-E](#)).
 - 6.6.1 When the specification requires a #200 sieve test, take a sample from this quarter before proceeding to [Tex-101-E](#) to determine the material passing the No. 200 sieve ([Tex-111-E](#)).
 - 6.6.2 Take the sample by quartering or splitting the material to the minimum required sample weight in [Tex-111-E](#).
 - 6.7 Combine the remaining three quarters with any remaining material from Section 6.6 and proceed to [Tex-101-E](#), Part II to prepare the for Moisture-Density curve ([Tex-113-E](#)), wet ball mill ([Tex-116-E](#)), and compressive strength ([Tex-117-E](#)) testing.
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7. SOIL SURVEY

- 7.1 Conduct the survey during the time of sampling to identify and visualize the material taken from different depths.
 - 7.2 Identify the location which may include the highway, station number, mile marker, direction, location within the lane, and distance from the nearest intersection.
 - 7.3 Identify the materials sampled in accordance with [Tex-141-E](#), Manual Procedure for Description Identification of Soils.
 - 7.4 Include at a minimum the following information in the survey documentation.
 - 7.4.1 The depth and location of each type of soil or rock in the subsurface.
 - 7.4.2 The condition of subsoils (moisture and density) upon which embankments will be constructed.
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- 7.4.3 The location and selection of suitable material for fills, sub-grade treatment and backfill adjacent to structures.
- 7.4.4 Material properties from laboratory testing, such as gradation ([Tex-110-E](#)), liquid limit ([Tex-104-E](#)), plastic limit ([Tex-105-E](#)), plasticity index ([Tex-106-E](#)), and soil classification ([Tex-142-E](#)).
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8. ARCHIVED VERSIONS

- 8.1 Archived versions are available.

Test Procedure for**PREPARING AND TESTING SOILS AND BASE MATERIALS****TxDOT Designation: Tex-101-E****Effective Date: April 2024**

1. SCOPE

- 1.1 This test method consists of three parts for the preparation and testing of embankment (soils), flexible base, roadway salvaged materials, and treated materials from the roadway or stockpile. Part I includes two methods, Method A and B. Method A is for dry preparation, and Method B for wet preparation. Part II is used for preparing untreated material for compaction, wet ball mill, and compression testing. Part III is used to measure the sieve analysis of road-mixed treated material, such as treatment with cement, emulsion, foamed asphalt, or lime.
- 1.2 Part I, Method A is used for preparing untreated material for liquid limit (Tex-104-E), plastic limit (Tex-105-E), plasticity index (Tex-106-E), linear bar shrinkage (Tex-107-E), and gradation (Tex-110-E). This method requires the sample to be oven-dried before preparation.
- 1.2.1 Part I, Method B may be used to prepare samples as received without oven-drying and is only allowed for samples to determine the liquid and plastic limit. Method B cannot be used to prepare samples for gradation testing.
- 1.2.2 Method A is the required method for referee testing and the preferred method to prepare materials. Methods A and B are only used to prepare samples that are not treated with an additive.
- 1.3 Part II is used to prepare samples for compaction (Tex-113-E), wet ball mill (Tex-116-E), and for compressive strength testing (Tex-117-E). This is used to determine the bulk gradation of the sampled base material.
- 1.4 Part III is used to determine the gradation of road-mixed material treated with an additive. This procedure uses sieves as required by the governing standard specification. The results are used to determine if adequate mixing and reclamation is achieved.
- 1.5 *This test procedure does not claim to address the safety concerns associated with its use. It is the responsibility of the user of this test procedure to establish the appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations before use.*

2. APPARATUS AND MATERIALS

- 2.1 *Balance*, Class G2 in accordance with Tex-901-K, minimum capacity 80 lb.
- 2.2 *Containers, metal pans, cardboard cartons.*
- 2.3 *Crusher*, optional to break down material passing the No. 40 sieve.

- 2.4 Dispenser cup.
- 2.5 Filter paper, non-fibrous.
- 2.6 Mechanical mixer, stirring device.
- 2.7 Mortar and pestle.
- 2.8 Oven, capable of maintaining a temperature of $140 \pm 9^{\circ}\text{F}$ for Part I and $230 \pm 9^{\circ}\text{F}$ for Part II.
- 2.9 Plaster of Paris molds (optional).
- 2.9.1 Dry the plaster of Paris molds at a maximum temperature of 140°F after forming, and wash and dry after each use.
- 2.10 Scoop.
- 2.11 Sieves, square, wire cloth openings meeting the requirements of Tex-907-K.
- 2.12 Splitter, quartering machine, or quartering cloth.
- 2.13 Water, approved drinking source.

PART I—PREPARING SAMPLES FOR LIQUID LIMIT, PLASTIC LIMIT, AND GRADATION TESTING

3. DRY PREPARATION, METHOD A

- 3.1 Sample embankment (soils), flexible base, or roadway salvaged materials in accordance with Tex-100-E.
 - 3.1.1 When testing flexible base, prepare the sample in accordance with Section 6 of Tex-100-E.
 - 3.1.2 When testing the sample for the liquid and plastic limits or the linear bar shrinkage, split or quarter material to provide a minimum of 300 g of material passing the No. 40 sieve.
 - 3.1.3 When testing the sample for gradation, split or quarter enough material to provide a minimum weight based on Tex-110-E, Table 1, Minimum Sample Weight. This sample may also be used to test for the liquid and plastic limit to determine the plasticity index.
- 3.2 Dry the sample in an oven at $140 \pm 9^{\circ}\text{F}$ to constant weight.
 - 3.2.1 Constant weight is achieved when the weight loss is less than 0.1% of the sample weight after 4 hr. of drying.
 - 3.2.2 When using the material to test for Tex-110-E, Part I, weigh the sample and record to the nearest 0.1 lb. This weight is the total sample weight before washing the material.
- 3.3 Dry sieve the material over a No. 40 sieve by hand or with a mechanical shaker.
- 3.4 Set aside and save the material passing the No. 40 sieve.
- 3.5 Slake the material retained on the No. 40 sieve in water.

- 3.5.1 Place the dry material into a clean pan.
- 3.5.2 Cover the material completely with water and soak for a minimum of 12 hr.
- 3.5.3 Decant or siphon off the clear water and take care not to lose any material.
- 3.5.4 Place an empty No. 40 sieve into a clean, large pan. Additional pans may be necessary.
- 3.5.5 Pour the liquid from the soaked sample through it.
- 3.5.6 Transfer the soaked sample to the sieve in increments to avoid overloading the sieve.
- 3.5.7 Pour water over the sieve until the water level is approximately 1/2-in. above the material.
- 3.5.8 Agitate the sieve up and down and stir the sample by hand to pass the fines through the sieve.
- 3.5.9 When 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.
- 3.5.10 After the fines have passed through the sieve, wash the retained aggregates clean while holding the sieve above the pan to capture any additional fines.
- 3.5.11 When testing for gradation (Tex-110-E), transfer the retained aggregate from the sieve to a clean pan. This material may be discarded when not testing for gradation.
- 3.5.12 Repeat the procedure in Sections 3.5.6. – 3.5.11. until all the soaked sample has been washed.
- 3.6 Dry the retained aggregate portion of the sample in an oven at $140 \pm 9^{\circ}\text{F}$ to constant weight as defined in Section 3.2.1.
- 3.6.1 When testing for gradation, proceed to Section 3.7.
- 3.6.2 When testing for the liquid and plastic limit, proceed to Section 3.8.
- 3.7 When testing for gradation, determine the gradation of the material retained on the No. 40 sieve in accordance with Tex-110-E, Part I.
- 3.8 Place the pan containing the wash water and material passing the No. 40 sieve aside until all the soil has settled to the bottom of the pan and the water above the soil is clear.
- 3.8.1 When the soil does not settle within 24 hr., proceed to Section 3.11.
- 3.9 Decant the water off the soil and take care not to lose any material.
- 3.10 Dry the remaining soil in an oven at $140 \pm 9^{\circ}\text{F}$ to constant weight as defined in Section 3.2.1. Proceed to Section 3.12.
- 3.11 Evaporate the water by placing the sample in an oven at $140 \pm 9^{\circ}\text{F}$ until it is dry or siphon the water on to a plaster of Paris mold lined with filter paper.
- 3.11.1 When the water has evaporated or absorbed by the filter paper, place the filter paper with adhering soil in a pan and dry in the oven at $140 \pm 9^{\circ}\text{F}$.
- 3.11.2 Sweep the dry soil from the filter paper with a stiff brush into the pan of fines.

- 3.12 Break down the dried soil completely with a mortar and pestle or a suitable crusher.
 - 3.13 Combine this material with the material from Sections 3.4.
 - 3.14 Mix the material thoroughly to produce a uniform sample.
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4. WET PREPARATION, METHOD B

- 4.1 Sample embankment (soils), flexible base, or roadway salvaged materials in accordance with Tex-100-E.
- 4.1.1 When testing flexible base, prepare the sample in accordance with Section 6 of Tex-100-E.
- 4.2 Split or quarter material to provide a minimum of 300 g of material passing the No. 40 sieve.
- 4.3 Place the sample in a clean pan.
- 4.4 Cover the material completely with water and soak for a minimum of 12 hr.
- 4.5 Place an empty No. 8 sieve into a clean, large pan.
- 4.6 Sieve the wet sample to remove large aggregate particles.
- 4.7 Wash the material retained on the sieve with a small amount of water.
- 4.8 Discard the portion of the material retained on the sieve.
- 4.9 When testing material with a low plasticity index (PI) such as 12 or less, proceed to Section 4.12.
- 4.10 When testing material with a moderate to high PI such as greater than 12, place the material into the dispersion cup of a mechanical mixer.
- 4.10.1 Do not fill the cup more than half-full.
- 4.10.2 Mix the material until the soil binder is separated.
- 4.11 Pour the material through a No. 40 sieve into a plaster of Paris bowl lined with filter paper.
- 4.11.1 Agitate the sieve up and down and stir the sample by hand to pass the fines through the sieve.
- 4.11.2 When the material retained on the sieve contains lumps that have not disintegrated, return them to the dispersion cup, and remix and wash through the sieve.
- 4.12 Pour until at least 95% of the soil binder has passed through the sieve.
- 4.13 Reduce the water content of the material in the plaster of Paris bowl to below the liquid limit.
- 4.14 When the sample can be divided into pie-like wedges and each wedge can be easily removed, it may be used for testing the liquid and plastic limit.
- 4.14.1 When the liquid and plastic limit are not to be determined immediately, place the material into an airtight container to prevent moisture loss.

PART II—PREPARING SAMPLES FOR COMPACTION, WET BALL MILL, AND COMPRESSIVE STRENGTH TESTING

5. PREPARING SAMPLES

- 5.1 Sample embankment (soils), flexible base, or roadway salvaged materials in accordance with Tex-100-E.
- 5.1.1 When testing flexible base, prepare the sample in accordance with Article 6, "Preparing Flexible Base Samples for Testing," of Tex-100-E.
- 5.2 When testing soils, spread the material on a clean floor and allow it to air dry to below the anticipated optimum moisture content or oven dry at a maximum temperature of 140°F.
- 5.2.1 Sieve the material using a 1/4 in. sieve, such that there are two sizes available for material retained and passing the 1/4 in. sieve.
- 5.2.1.1 When the material retained on the 1/4-in. sieve contains coarse aggregate, use coarser size sieves as deemed applicable and necessary to separate the material.
- 5.2.2 Process material that may have formed into hard lumps when dried or contain aggregate to pass through a 1/4-in. wire-mesh.
- 5.3 When testing base materials, oven dry the material at $230 \pm 9^\circ\text{F}$ to constant weight.
- 5.3.1 Constant weight is achieved when the weight loss is less than 0.1% of the sample weight after 4 hr. of drying.
- 5.3.2 Sieve base into the following sizes.
- 1-3/4 in.
 - 1-1/4 in.
 - 7/8 in.
 - 5/8 in.
 - 3/8 in.
 - No. 4
 - No. 40
- 5.3.2.1 Do not overload the sieves or screens. The material passing the No. 4 and retained on the No. 40 sieve may need to be sieved separately or in smaller batches to avoid overloading.
- 5.3.3 Weigh the material retained on each sieve and record to the nearest 0.1 lb.
- 5.3.4 Add all the weights retained to determine a total weight and record to the nearest 0.1 lb.
- 5.3.5 Calculate the bulk gradation for the percent retained on each sieve size using the equation in Section 7.1.

PART III—SIEVE ANALYSIS OF ROAD-MIX STABILIZED MATERIAL

6. PROCEDURE

- 6.1.1 Sample road-mixed material after mixing and before roller compaction in accordance with Tex-100-E.
- 6.2 Use a sample splitter, quartering machine, or quartering cloth to reduce the sample size to a minimum of 10.0 lb.
- 6.3 Weigh the sample and record to the nearest 0.1 lb.
- 6.4 Use the sieve sizes required by the specification and arrange a set of sieves in descending order beginning with the largest size on top and a pan at the bottom.
- 6.5 Pour the sample into the top sieve and cover the stack of sieves and pan.
- 6.5.1 Do not overload the sieves or screens, may need to be sieved in smaller batches to avoid overloading.
- 6.6 Shake the set of sieves with a mechanical sieve shaker for 5 min. or by hand.
- 6.7 Shake each sieve size by hand to refusal.
- 6.7.1 Hand sieve for approximately 1 min. with lateral and vertical motion with a jarring action that keeps the material moving continuously over the surface of the sieve.
- 6.7.2 Continue hand sieving until no more than 1% of the test sample by weight passes through the sieve.
- 6.8 Weigh the material passing each sieve and record to the nearest 0.1 lb.
- 6.9 Calculate the percent passing each sieve using the equation in Section 7.2.
-

7. CALCULATION

- 7.1 Use the following equation to determine the percent retained for each sieve size.

$$\text{Percent Retained} = 100 \times \left[\frac{W_{\text{Retained}}}{W_{\text{Total}}} \right]$$

Where:

W_{Retained} = Weight of material retained on the sieve from Section 5.4.3.

W_{Total} = Total weight of the test sample from Section 5.4.4.

- 7.2 Use the following equation to determine the percent passing for each sieve size.

$$\text{Percent Passing} = 100 \times \left[\frac{W_{\text{Passing}}}{W_{\text{Total}}} \right]$$

Where:

W_{Passing} = Weight of material passing the sieve from Section 6.8.

W_{Total} = Total weight of the test sample from Section 6.3.

8. REPORT

8.1 Report test results to the nearest whole percent.

9. ARCHIVED VERSIONS

9.1 Archived versions are available.

Test Procedure for**MOISTURE CONTENT OF SOILS AND BASE MATERIALS****TxDOT Designation: Tex-103-E****Effective Date: February 2024**

1. SCOPE

- 1.1 Use this test procedure to determine the moisture content of embankment (soils), flexible base, roadway salvaged materials, and treated materials. Moisture content is the water content of a material. It is a ratio of the weight of water to the weight of the dry, solid particles of the material and is reported as a percentage.
 - 1.2 This test consists of two parts. Part I is for using a drying oven and Part II is for using a microwave oven the preferred method to determine the moisture content of material is Part I.
 - 1.2.1 A microwave oven may be used to expedite the test to provide results rapidly. It is not recommended to use a microwave oven for testing treated materials using asphalt, cement, or lime because this may damage the oven. Be attentive to the composition of samples, such containing highly organic material or contaminants that may ignite into flames.
 - 1.2.2 Do not use a microwave oven to dry material when testing it for the plasticity index and organic content.
 - 1.3 *This test procedure does not claim to address the safety concerns associated with its use. It is the responsibility of the user of this test procedure to establish the appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations before use.*
-

2. APPARATUS

- 2.1 *Balance*
 - 2.1.1 Class G1 in accordance with Tex-901-K, for specimens with a mass of 200 g or less.
 - 2.1.2 Class G2 in accordance with Tex-901-K for specimens with a mass greater than 200 g.
- 2.2 *Container or pan with lid.*
 - 2.2.1 When using an oven, the container or pan and lid must be resistant to corrosion, will not change in weight from heating and cooling, and have close-fitting lids.
 - 2.2.2 When using a microwave oven, the container or pan and lid must be non-metallic and non-absorbent.
- 2.3 *Desiccator cabinet or jar, containing silica gel or anhydrous calcium sulfate.*

- 2.3.1 Use a desiccator when using a microwave oven to allow the container and sample to cool after use to allow handling and prevent damage to the balance. A desiccator is not required when using containers with close-fitting lids.
- 2.4 *Gloves, tongs, or holder*, suitable for moving and handling hot containers or pans after drying.
- 2.5 *Heat sink*, small bowl, cup, or container of water or sponge.
- 2.6 *Microwave oven*, variable power controls and input power ratings of 700 watts.
- 2.7 *Oven*, capable of maintaining a temperature of $230^{\circ} \pm 9^{\circ}\text{F}$.
- 2.8 *Spatula or scoop*.

3. PREPARING SAMPLE

- 3.1 Sample embankment (soils), flexible base, roadway salvaged materials, and treated materials in accordance with Tex-100-E and split or quarter to the minimum weight listed in Table 1.

Table 1

Min Weight of Field Sample

Max Particle Size	Min Weight of Test Sample, g
1-3/4 in.	5,000
3/4 in.	1,000
No. 4	500

- 3.1.1 When the maximum particle size is between the sizes listed in the table, use the next larger minimum weight.
- 3.2 Store samples prior to testing in airtight containers at a temperature between 37°F to 86°F and in an area that prevents direct contact with sunlight.
- 3.3 Label and identify each sample separately.
- 3.4 Measure the moisture content as soon as possible after sampling.

PART I—CONVENTIONAL OVEN METHOD

4. PROCEDURE

- 4.1 Weigh a clean, dry container or pan and a lid from Section 2.2.1 and record to the nearest 0.1 g.
- 4.2 Place the sample in the container and spread it around to approximately an even thickness and cover it with the lid immediately.
- 4.3 Weigh the container with the sample and lid, and record to the nearest 0.1 g.
- 4.4 Remove the lid and place the container with the sample in the drying oven.

- 4.5 Dry for a minimum of 16 hours at $230^{\circ} \pm 9^{\circ}\text{F}$ or to a constant weight.
- 4.5.1 When testing the sample after drying for liquid limit (Tex-104-E) and plastic limit (Tex-105-E) or sulfate content (Tex-145-E), dry the sample at a maximum temperature of 140°F .
- 4.5.2 When testing the sample after drying for organic content (Tex-148-E), dry the sample at a maximum temperature of 104°F .
- 4.5.3 Constant weight is achieved when the weight loss is less than 0.1% of the sample weight after 4 hr. of drying.
- 4.6 Remove the container from the oven and replace the lid firmly.
- 4.7 Allow the container and sample 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 heat of the container.
- 4.8 Weigh the container, lid, and sample and record to the nearest 0.1 g.

PART II—MICROWAVE OVEN METHOD

5. PROCEDURE

- 5.1 Weigh a clean, dry container or pan and a lid from Section 2.2.2 and record to the nearest 0.1 g.
- 5.2 Place the sample in the container and spread it around to approximately an even thickness and cover it with the lid.
- 5.3 Weigh the container with the sample and lid, and record to the nearest 0.1 g.
- 5.3.1 The minimum sample weight is 500 g. Samples less than 500 g may dry too rapidly and cause the sample to disperse and scatter throughout the microwave oven during heating.
- 5.4 Remove the lid and place the container with the sample and a heat sink in the microwave oven.
- 5.4.1 A heat sink is a material or liquid that will absorb energy after the moisture has been driven from the sample. This will prevent from over-heating the sample and damaging the microwave oven. A beaker filled with water or sponge may be used.
- 5.5 Turn on the microwave oven for 3 min.
- 5.6 Remove the container and sample from the oven and place it in the desiccator to cool to allow handling and to prevent damage to the balance.
- 5.6.1 A desiccator is not necessary when using containers with close-fitting lids.
- 5.7 Weigh the container and sample and when applicable with the lid and record to the nearest 0.1 g.
- 5.8 Use a spatula and carefully mix the sample.
- 5.9 Return the container and sample and when applicable with the lid to the oven and reheat it for 1 min.

- 5.10 Repeat the heating (drying), cooling, and weighing, until the change between two consecutive weights 0.1% or less of the initial wet mass of the sample.
- 5.11 Record the final weight of the container, lid, and sample to the nearest 0.1 g.
- 5.12 Discard the sample and do not use it for any other test after drying. The sample may have particle breakdown, chemical changes or losses, melting, or loss of organic material. Test results will not be accurate.
-

6. CALCULATIONS

- 6.1 Use the following equation to determine the weight of moisture (water).

$$W_{Water} = W_2 - W_3$$

- 6.2 Use the following equation to determine the weight of the dry sample (material).

$$W_{Dry} = W_3 - W_1$$

- 6.3 Use the following equation to determine the moisture content (%).

$$Moisture\ Content = 100 \times \left[\frac{W_{Water}}{W_{Dry}} \right]$$

Where:

W_{Water} = Weight of water.

W_{Dry} = Weight of the dry material.

W_1 = Weight of container and lid, g. (Section 4.1 or 5.1).

W_2 = Weight of container, lid, and sample containing moisture before drying, g. (Section 4.3 or 5.3).

W_3 = Weight of the container, lid, and dry material after drying, g. (Section 4.8 or 5.11)

7. REPORT

- 7.1 Report the test results to the nearest 0.1 %.
-

8. ARCHIVED VERSION

- 8.1 Archived version.

Test Procedure for

MEASURING IN-PLACE DENSITY AND MOISTURE CONTENT OF SOILS AND BASE MATERIALS



TxDOT Designation: Tex-115-E

Effective Date: January 2025

1. SCOPE

This method determines the in-place density and moisture content of untreated and treated embankment (soils) and granular material compacted in the roadway or in the natural state, as exists in a cut section and borrow source before excavation. In-place density determines the degree of compaction or percentage of the maximum dry density determined by test method [Tex-113-E](#) and [Tex-114-E](#).

- 1.1 Part IV is used to determine random testing locations for determining the density and moisture content. This procedure uses the length and width of the area/land compacted in place.
 - 1.2 *This test procedure does not claim to address the safety concerns associated with its use. It is the responsibility of the user of this test procedure to establish the appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations before use.*
-

2. REPORTING AND DOCUMENTATION

- 2.1 Contact the Materials and Tests Division to request a spreadsheet to determine and report the random density and moisture testing locations from Part IV.
-

PART I—NUCLEAR GAUGE METHOD

3. APPARATUS

- 3.1 *Drill rod (pin) and driver or hammer.*
 - 3.2 *Gauge logbook or field form.*
 - 3.3 *Nuclear testing gauge, used to measure in-place density and moisture.*
 - 3.4 *Reference standard block.*
 - 3.5 *Scraper plate and drill rod guide.*
-

4. STANDARDIZING EQUIPMENT

- 4.1 Standardize the nuclear testing gauge using the reference standard block provided with the gauge.
-

- 4.1.1 Standardize the gauge at the start of each day's use to ensure measurements are accurate.
 - 4.2 Use a dry and clean reference standard block.
 - 4.3 Set the block on a smooth, dry surface that is a minimum of 5 ft. from any object and a minimum of 25 ft. from any other nuclear gauge.
 - 4.4 Place the gauge on the standardizing block in the Safe position.
 - 4.5 Standardize the nuclear testing gauge for density and moisture standard counts according to the manufacturer's recommendations for the gauge.
 - 4.6 Record the counts in the gauge logbook or field form.
 - 4.7 When both the density and moisture standard counts are acceptable, the gauge is in satisfactory operating condition.
 - 4.8 When the density or moisture standard counts are not acceptable, allow additional time for the gauge to stabilize and make sure the area is clear of sources of interference, and thereafter perform another standardization.
 - 4.8.1 If the second standardization is within limits, the gauge may be used.
 - 4.8.2 If the second standardization is not within limits, the gauge must be calibrated or repaired as recommended by the manufacturer.
-

5. PROCEDURE

- 5.1 Use the scraper plate to prepare the test area by creating a level and smooth surface free of loose material that extends laterally more than 6 in. beyond the gauge. This area is approximately 2 ft. in length and width.
- 5.2 Fill in minor depressions or surface voids with dry sand or native fines. Do not use any material with moisture.
- 5.3 Place the scraper plate on the level and smooth surface. Slide the drill rod (pin) through the extraction tool.
- 5.4 Step on the scraper plate and hammer the pin through one of the guides on the scraper plate.
- 5.4.1 Make a hole at least 2 in. deeper than the testing depth and as close as possible to perpendicular from the plane of the testing surface.
- 5.5 Remove the pin by pulling straight up and twisting the extraction tool. Do not loosen the pin by tapping it with a hammer from side to side. This may distort the hole or cause loose material to fall into the hole.
- 5.6 Mark the test area using the pin to outline the scraper plate before removing it.
- 5.7 Remove the scraper plate.
- 5.8 Place the nuclear gauge on the outlined surface with the bottom of the gauge seated firmly in full contact with the soil or base material.
- 5.8.1 When a gauge is not fully seated, it may result in a moisture and density reading lower than what is in-place.

- 5.9 Lower the source rod into the hole. Do not extend and expose the source rod to guide it into the hole.
- 5.9.1 Carefully lower the rod to not disturb material around the hole.
- 5.9.2 Release the trigger and lock the source rod. A click should be heard when the source rod is locked into position. Lightly press the top of the handle to confirm the rod is locked into place.
- 5.9.3 Gently move the gauge so the rod is firmly against the side of the hole that is nearest to the source or detector tubes. This eliminates air so the rod is flush and tight against the hole.
- 5.9.4 Move all tools and equipment at least 5 ft. from the gauge.
- 5.10 Measure the density and moisture content as per the gauge manufacturer's recommendations.
- 5.10.1 When the initial test result does not meet specifications, rotate the gauge 90° and repeat Sections 5.9.3 and 5.10. Proceed to Section 5.10.2.
- 5.10.2 Rotate the gauge 180° and repeat Sections 5.9.3 and 5.10. Proceed to Section 5.10.3.
- 5.10.3 Rotate the gauge 270° and repeat Sections 5.9.3 and 5.10. Proceed to Section 5.10.4.
- 5.10.4 Average the four measurements to determine the density and moisture content of the location tested.
- 5.10.5 When the moisture content is suspect or testing material with other hydrocarbons such as asphalt or emulsion, use Tex-103-E to determine the correct moisture content. Use this moisture content to determine a correction factor.
- 5.10.5.1 Apply the moisture content correction factor to the moisture content measured by the nuclear testing gauge.
- 5.10.5.2 Use equation in Section 6.1 with the corrected moisture content to calculate a new dry density. Use this dry density to calculate the in-place density of the land compacted.
- 5.10.6 When used for trench measurements or near obstructions such as abutments and vertical structures, follow the manufacturer's recommendations.

6. CALCULATIONS

- 6.1 Use the following equation to calculate the dry density using a corrected moisture content.

$$\text{Dry Density} = 100 \times \left(\frac{\text{Wet Density}}{100 + \text{Percent Moisture}} \right)$$

Where:

Wet Density is measured by the nuclear testing gauge from Section 5.10.

Percent Moisture is the corrected moisture content from Section 5.10.5.1.

7. REPORTING TEST RESULTS

- 7.1 Report dry density to the nearest 0.1 lb./ft.³ and moisture content to the nearest 0.1%.

PART II—SAND-CONE METHOD

Part II has been removed from this test procedure.

PART III—VOLUMETER METHOD

Part III has been removed from this test procedure.

PART IV—RANDOM SELECTION OF TESTING LOCATIONS

8. PROCEDURE

- 8.1 Use the random density testing spreadsheet provided by the Materials and Tests Division to determine random locations for density and moisture testing from Part I.
- 8.2 Determine the length and width of the area that is ready for testing.
- 8.3 Multiply the length of the area by the length random number.
 - 8.3.1 Add this distance to the beginning station number.
- 8.4 Multiply the width of the area by the width random number. This is the width offset.
 - 8.4.1 Measure the width offset from the right side of the completed area.
 - 8.4.2 Adjust the width offset by no more than necessary when it is within 2 ft. of each compacted edge.
 - 8.4.3 Adjust the width offset to 2 ft. from the compacted edge when it is outside of the compacted area such as when the width of the compacted area varies.
- 8.5 Measure the density and moisture content in accordance with Part I at the random location determined from Sections 7.3 and 7.4.

9. ARCHIVED VERSIONS

- 9.1 Archived versions are available.

Test Procedure for**MEASURING DEPTH OF COMPACTED BASE AND TREATED MATERIALS****TxDOT Designation: Tex-140-E****Effective Date: July 2024**

1. SCOPE

- 1.1 Use this procedure to measure the depth (thickness) of compacted base and treated materials after the final and finished grade has been obtained and set with grade stakes (blue tops). Treated materials includes reclamation and road-mixed pavements stabilized with asphalt, cement, lime, or a non-standard additive.
 - 1.2 This procedure is required to determine the thickness of compacted pavement layers constructed under the 200 Specification Items for flexible base and treatment (road-mixed) materials.
 - 1.3 *This test procedure does not claim to address the safety concerns associated with its use. It is the responsibility of the user of this test procedure to establish the appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations before use.*
-

2. APPARATUS

- 2.1 *One percent phenolphthalein*, clear, colorless liquid solution, only required when measuring the depth of pavement layers treated with cement, lime, or a non-standard treatment additive blended with cement. This solution will turn a pink or fuchsia color when the pH of the material is greater than 8.0.
 - 2.2 *Drill with auger bit, grubbing hoe, post hole digger, shovel, or any other approved depth measurement tool.*
 - 2.3 *Folding scale, ruler, or tape measure*, with 1/8 in. divisions of enough length.
 - 2.4 *Six-penny nail, blade, or knife*, not to exceed 1/8 in. in thickness and approximately 3 in. in length.
 - 2.5 *Straight edge or surveyor stack*
-

3. PROCEDURE

- 3.1 Drill or dig a hole approximately 6–8 in. below the layer being measured and wide enough to fit a hand in the hold to maneuver around in it.
 - 3.2 Make a vertical groove using a *six-penny nail, blade, or knife* on the side of the hole to remove loose material and to expose and see the interface of both layers.
 - 3.3 When measuring the depth (thickness) of pavement layers treated with cement, lime, or a non-standard treatment additive blended with cement, use a phenolphthalein solution from Section 2.1 to identify the interface.
-

- 3.3.1 Apply the solution from the bottom below the interface up towards the top of the layer. The interface will be identified where the area will turn pink or fuchsia color.
- 3.4 Push a six-penny nail, blade, or knife horizontally into the interface approximately 1 in. such that it doesn't move when measuring the thickness.
- 3.5 Place a straightedge or surveyor's stake across the top of the hole.
- 3.6 Use a folding scale, ruler, or tape measure and measure the thickness from the top of the nail, blade, or knife to the bottom of the straightedge or stake and report it to the nearest 1/8 in.
- 3.7 Move the nail, blade, or knife to another location on the interface and repeat Sections 3.4 – 3.6 until three measurements are reported.
- 3.7.1 Repeat Section 3.3 when measuring the thickness of a pavement layer treated with cement, lime, or a non-standard treatment additive blended with cement.
-

4. CALCULATION

- 4.1 Use the following equation to determine the average pavement depth (thickness) from three measurements.

$$\text{Pavement Thickness} = \frac{T_1 + T_2 + T_3}{3}$$

Where:

T_# = Thickness measurement to the nearest 1/8" (0.01 in.) to include a minimum of three measurements.

- 4.1.1 Use the following decimals for the fraction portion of each thickness measurement reported.

1/8" = 0.13

1/4" = 0.25

3/8" = 0.38

1/2" = 0.50

5/8" = 0.63

3/4" = 0.75

7/8" = 0.88

5. REPORT

- 5.1 Report the average pavement layer depth to the nearest 0.01 in.
-

6. ARCHIVED VERSIONS

- 6.1 Archived versions are available.

Test Procedure for

SAMPLING AGGREGATE AND LIMESTONE ROCK ASPHALT (LRA)

TxDOT Designation: Tex-221-F

Effective Date: October 2022**1. SCOPE**

- 1.1 Use this test procedure for sampling completed stockpiles of coarse and fine aggregate used to produce hot-mix asphalt (HMA), hot-mix cold-laid asphalt, Limestone Rock Asphalt (LRA), microsurfacing, and for the placement of surface treatments.
- 1.2 Use this test procedure to sample completed coarse or fine aggregate and LRA stockpiles using a front-end loader or shovel. The preference for sampling is to use a front-end loader. Only use a shovel where a front-end loader is not available.

2. SAMPLE SIZE

- 2.1 Use Table 1 to determine the minimum sample size to perform the required tests listed in the applicable specification.
- 2.2 Obtain samples from completed stockpiles that show the true nature and condition of the materials they represent. Do not combine materials that apparently differ in property or character to make a composite sample. Differences may be indicated by color or texture.
- 2.3 Use a sample splitter or quartering cloth to reduce the field sample to laboratory test size.

Table 1—Min Size of Samples

Nominal Max Size of Aggregate ¹	Min Weight of Field Samples ² (pounds)
No. 8	10
No. 4	25
3/8 in.	25
1/2 in.	35
3/4 in.	55
1 in.	110
1-1/2 in.	165

1. Nominal maximum size is one sieve size larger than the first sieve that retains more than 10% of the aggregate.

2. The minimum sample mass depends on the nominal maximum aggregate size as well as the number and type of test required.

3. SAMPLING PROCEDURES

- 3.1 This test procedure does not claim to address the safety concerns associated with its use. It is the responsibility of the user of this test procedure to establish the appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations before use.
- 3.2 *Sampling procedure for aggregate from completed stockpiles using a front-end loader.*
- 3.2.1 Use this procedure to sample completed stockpiles of coarse or fine aggregate and LRA using a front-end loader.
- 3.2.2 Determine the minimum number of samples required for approval based on the Department's Guide Schedule frequency for HMA, hot-mix cold-laid asphalt, seal coat, and LRA.
- 3.2.3 Identify the sampling locations by dividing the stockpile in approximately equal sectors based on the number of samples determined from Section 3.2.2. Stockpiles must be sampled from a minimum of two sectors or locations for acceptance.
- 3.2.4 Clean and level the ground at the sampling location to prevent contamination of the sampling pile.
- 3.2.5 Cut approximately at the ground level to the top edge of the stockpile until a clean face is exposed. This represents the full height of the stockpile.
Note 1-The exposed vertical face should be perpendicular to the top edge of the stockpile, but this may not be possible with stockpiles of dry, coarse aggregates.
- 3.2.6 Discard the material cut away while exposing the clean face.
- 3.2.7 Build a sample pad by cutting into the vertical face at the ground level to the full height of the stockpile to obtain material.
- 3.2.8 Lower the bucket as close as possible to the ground to avoid segregation and empty the entire contents of the bucket in one motion onto the ground.
- 3.2.9 Using the loader bucket, strike and level the sample pad at mid-height in the direction the bucket was emptied to create a flat surface for sampling. Back-drag the sampling pad only once.
- 3.2.10 Using a shovel or straightedge, visibly trace lines on top of the sample pad to outline four evenly sized quarters.
- 3.2.11 Place clean sample bags or containers near the center of the sampling pad and obtain the sample across the flat area staying more than 1 ft. away from the edges.
- 3.2.11.1 Sample equal amounts of aggregate or LRA evenly across each quadrant. Fully insert the shovel as near as vertical as possible and then slowly roll the shovel back and lift slowly to avoid coarse aggregate rolling off the sides of the shovel.
Note 2-Spade-tip shovels are not recommended for sampling. Square-tip shovels work well preventing aggregate from rolling from the side.
- 3.2.11.2 Obtain additional shovelfuls from different quadrants of the sampling pad, and in areas avoiding previous shovel holes.
- 3.2.12 Place the material into the clean sample bags or containers.

- 3.2.13 Seal and label the sample bags or containers.
- 3.2.14 Repeat Section 3.2.4 to 3.2.13 for each additional location as determined from Section 3.2.3.
- 3.3 *Sampling procedure for aggregate from completed stockpiles using a shovel.*
- 3.3.1 Use this procedure to sample completed stockpiles of coarse or fine aggregate and LRA using a shovel where a front-end loader is not available. The preference for sampling is to use a front-end loader.
- 3.3.2 Determine the minimum number of samples required for approval based on the Department's Guide Schedule frequency for HMA, hot-mix cold-laid asphalt, seal coat, and LRA.
- 3.3.3 Identify the sampling locations by dividing the stockpile in approximately equal sectors based on the number of samples determined from Section 3.3.2. Stockpiles must be sampled from a minimum of two sectors or locations for acceptance.
- 3.3.4 Identify locations within each sector from the top third, at the mid-point, and bottom third of the stockpile. Sampling must be performed in no less than these three increments.
- 3.3.5 Dig a small trench into the stockpile at each location approximately 1 ft. deep and 3 ft. in diameter.
Note 3—A board may be shoved vertically into the stockpile just above the point of sampling to prevent segregation from coarser aggregate rolling down during sampling.
- 3.3.6 Do not use the aggregate or LRA removed from the trenched area as a part of the sample.
- 3.3.7 Shovel material from the stockpile into clean sample bags or containers from the innermost part of the trench. Minimize larger sized aggregate falling back into the trench.
Note 4—When sampling sands, sampling tubes of at least 1-1/4 in. diameter may be used where experience has indicated representative samples cannot be obtained otherwise.
- 3.3.8 Seal and label the sample bags or containers.
- 3.3.9 Repeat Section 3.3.4 to 3.3.8 for each additional location as determined from Section 3.3.3.
-

4. ARCHIVED VERSIONS

- 4.1 Archived versions are available.

Test Procedure for**SAMPLING AGGREGATE AND STONE RIPRAP****TxDOT Designation: Tex-400-A****Effective Date: December 2023**

1. SCOPE

- 1.1 Use this test procedure for sampling completed stockpiles of coarse and fine aggregate used to produce concrete and retaining wall select backfill, and stone riprap. This procedure may also be used to sample concrete aggregate during production when stored in bays or stockpiles that are not accessible in three directions.
 - 1.2 Use this test procedure to sample completed coarse or fine aggregate stockpiles and materials using a front-end loader or shovel. The preference for sampling is to use a front-end loader. Only use a shovel where a front-end loader is not available.
 - 1.3 *This test procedure does not claim to address the safety concerns associated with its use. It is the responsibility of the user of this test procedure to establish the appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations before use.*
-

2. APPARATUS

- 2.1 Sample bags or buckets with lids.
 - 2.2 Square-Tip Shovel.
-

3. SAMPLE SIZE

- 3.1 Use Table 1 from Section 3.3 to determine the minimum sample size to perform the required tests listed in the applicable specification.
- 3.2 Obtain samples that show the true nature and condition of the materials that they represent. Do not combine materials that apparently differ in property or character to make a composite sample. Differences may be indicated by color or texture.
- 3.3 Use a sample splitter or quartering cloth to reduce the field sample to laboratory test size.

Table 1
Minimum Field Sample Size

Nominal Max Size of Aggregate ¹	Mini Weight of Field Samples ² (lbs.)
3 in.	330
2-1/2 in.	275
2 in.	220
1-1/2 in.	165
1 in.	110
3/4 in.	55
1/2 in.	35
3/8 in.	25
No. 4	25
No. 8	25

1. The Max aggregate size is the largest sieve size listed in the applicable specification upon which any material can be retained.
2. The Min sample weight depends on the Max aggregate size and the type and number of tests required.

4. SAMPLING AGGREGATE STOCKPILES USING A FRONT-END LOADER

- 4.1 Determine the minimum number of samples required for acceptance based on TxDOT's Guide Schedule frequency for concrete or retaining wall select backfill.
 - 4.1.1 There is no minimum number of samples required for quality control testing.
- 4.2 Identify the sampling locations by dividing the stockpile in approximately equal sectors based on the number of samples determined from Section 4.1. Aggregate must be sampled from a minimum of two sectors or different locations for acceptance.
- 4.3 Clean and level the ground at the sampling location to prevent contamination of the sampling pile.
- 4.4 Cut approximately at the ground level to the top edge of the stockpile until a clean face is exposed. This represents the full height of the stockpile.
 - 4.4.1 The exposed vertical face should be perpendicular to the top edge of the stockpile, but this may not be entirely possible with stockpiles of dry, coarse aggregates.
- 4.5 Discard the aggregate cut away while exposing the clean face.
- 4.6 Build a sample pad by cutting into the vertical face at the ground level to the full height of the stockpile to obtain aggregate.
- 4.7 Lower the bucket as close as possible to the ground to avoid segregation and empty the entire contents of the bucket in one motion onto the ground.
- 4.8 Using the loader bucket, strike and level the sample pad at mid-height in the direction the bucket was emptied to create a flat surface for sampling. Back-drag the sampling pad only once.
- 4.9 Divide the sample pad into four quadrants of similar size.
- 4.10 Place clean sample bags or containers near the center of the sampling pad and obtain the sample across the flat area staying more than 1 ft. away from the edges.
- 4.11 Sample equal amounts of aggregate from each quadrant.

- 4.11.1 Fully insert a square-tip shovel as near as vertical as possible and then slowly roll the shovel back and lift slowly to avoid coarse aggregate rolling off the sides of the shovel.
 - 4.11.2 Spade-tip shovels are not allowed for sampling because they will not prevent aggregate from rolling off the side of the shovel.
 - 4.12 Place the aggregate into the clean sample bags or containers.
 - 4.13 Seal and label the sample bags or containers.
 - 4.14 Repeat Section 4.4 – 4.13 for each additional location as determined from Section 4.2.
-

5. SAMPLING AGGREGATE FROM STOCKPILES USING A SHOVEL

- 5.1 Use this procedure to sample completed stockpiles of coarse or fine aggregate using a shovel where a front-end loader is not available. The preference for sampling is to use a front-end loader.
 - 5.2 Determine the minimum number of samples required for acceptance based on TxDOT's Guide Schedule frequency for concrete or retaining wall select backfill.
 - 5.2.1 There is no minimum number of samples required for quality control testing.
 - 5.3 Identify the sampling locations by dividing the stockpile in approximately equal sectors based on the number of samples determined from Section 5.2. Aggregate must be sampled from a minimum of two sectors or different locations for acceptance.
 - 5.4 Identify locations within each sector from the top third, at the mid-point, and bottom third of the stockpile. Sampling must be performed in no less than these three increments.
 - 5.5 Dig a small trench into the stockpile at each location approximately 1 ft. deep and 3 ft. in diameter.
 - 5.6 A board may be shoved vertically into the stockpile just above the point of sampling to prevent segregation from coarser aggregate rolling down during sampling.
 - 5.7 Do not use the aggregate removed from the trenched area as a part of the sample.
 - 5.8 Shovel aggregate from the stockpile into clean sample bags or containers from the innermost part of the trench. Minimize larger sized aggregate falling back into the trench.
 - 5.8.1 When sampling sands, sampling tubes of at least 1-1/4 in. diameter may be used where experience has indicated representative samples cannot be obtained otherwise.
 - 5.9 Seal and label the sample bags or containers.
 - 5.10 Repeat Section 5.4 – 5.9 for each additional location as determined from Section 5.2.
-

6. SAMPLING AGGREGATE DURING CONCRETE PRODUCTION AT MIXING PLANT AND SITE.

- 6.1 Use this procedure to sample coarse or fine aggregate at concrete mixing plants and sites during production from bays or stockpiles that are not accessible in three directions and for Optimized Aggregate Gradation

(OAG) concrete. The preference for sampling is to use a front-end loader. Only use a shovel when a front-end loader is not available.

- 6.2 Identify the center of the open face of the aggregate bay.
- 6.3 Sample at this location using a front-end loader according to Section 4.
- 6.4 Sample at this location using a shovel when a front-end loader is not available according to Sections 5.
- 6.5 Sample at additional locations when the face of the open bay or face of the stockpile is significantly wide. Chose a minimum of two locations approximately equal offset from the center.

7. SAMPLING FLEXIBLE BASE STOCKPILES

- 7.1 Refer to Tex-100-E, Section 4.

8. PREPARING FLEXIBLE BASE SAMPLES FOR TESTING

- 8.1 Refer to Tex-100-E, Section 6.

9. SAMPLING STONE RIPRAP

- 9.1 Obtain six to eight pieces of the stone riprap that is representative of the size of the riprap.
- 9.2 Crush or break down the entire sample to a maximum particle size of 6 in.
- 9.3 Select approximately 275 lbs. of the riprap and place into sample bags or containers.
- 9.4 Seal and label the sample.

10. ARCHIVED VERSIONS

- 10.1 Archived versions are available.

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

**1. SCOPE**

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

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

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

PART I—SAMPLING LIME PRODUCTS

2. SCOPE

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

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

3. APPARATUS

3.1 *Bulk Hydrated Lime:*

3.1.1 *Paint brush, 2 in. wide.*

- 3.1.2 *Bucket*, 1 gal., with double friction type lid and bail.
- 3.1.3 *Top hatch sampling device*, consisting of a 9-ft. length of 1.5-in. IPS PVC 1120 plastic pipe of SDR 1.10 MPa (26,160 psi), meeting ASTM D 2241:
 - 3.1.3.1 Fit the pipe at one end with a rubber stopper drilled with a 0.25-in. diameter hole.
 - 3.1.3.2 Cement the stopper in place using a standard adhesive epoxy.
 - 3.1.3.3 Spot-weld a hook to a 3 × 2-in. half-round plate of 12-gauge steel. Rivet and cement the plate with epoxy to the rubber stopper end of the pipe.
 - 3.1.3.4 The 3-in. long metal hook has a 0.5 × 0.75 in. doubled over section, with a 0.25-in. slot.
 - 3.1.3.5 The opening of the hook should face away from the rubber stopper.
 - 3.1.3.6 The hook will catch the bucket bail, so the sampler can lower the bucket and pipe to the ground. This allows the sampler to alight from the truck safely.
- 3.1.4 *Bottom sampling tube*, consisting of two concentric plastic pipes:
 - 3.1.4.1 The outer pipe is 126 in. long, 1.5 in. inside diameter, IPS PVC plastic pipe, fitted at one end with a tip made from a 7.5-in. length of solid aluminum round stock, 1-5/8 in. in diameter, tapered to a point along 6.5 in. of its length, inserted 1 in. into the tube, and fastened with two screws through the wall of the pipe into tapped holes on either side of the tip.
 - 3.1.4.1.1 Round the point of the tip to a 0.5-in. diameter point for safety.
 - 3.1.4.1.2 Use an adhesive epoxy to mold an epoxy tip in a metal, foil, or cardboard mold.
 - 3.1.4.2 The inner pipe is a 1.25-in. IPS PVC plastic pipe, measuring 10 ft. long.
 - 3.1.4.2.1 Slip this pipe inside the outer pipe. It will extend beyond the outer pipe at the upper end forming a handle to allow the sampler to rotate the inner tube within the outer tube.
 - 3.1.4.2.2 Cut a 1.25 in. wide × 12 in. long sampling port through both pipes 1.5 in. from the lower, plugged end.
 - 3.1.4.2.3 Create index marks on the outside of the outer and inner pipes at the upper end labeled “open” and “closed” to indicate the relative position of the opening in the inner pipe to that of the outer.
- 3.2 *Bagged Hydrated Lime:*
 - 3.2.1 *Paint brush*, 2 in. wide.
 - 3.2.2 *Bucket*, 1 gal., with double friction type lid and bail.
 - 3.2.3 *Bag sampling tube*, made from 0.75-in. diameter steel electrical conduit 3 ft. long. Taper the opening at one end with a 4-in. diagonal cut.

- 3.3 *Quicklime:*
 - 3.3.1 *Safety goggles.*
 - 3.3.2 *Respirator.*
 - 3.3.3 *Rubber gloves.*
 - 3.3.4 *Paint brush, 2 in. wide.*
 - 3.3.5 *Bucket, 1 gal., with double friction type lid and bail.*
 - 3.3.6 *Device designed to hold a sample bucket between the wheel path of a bulk transport discharging quicklime, without allowing the bucket to turn over, but permitting safe, easy removal of the container from the windrow with the sample intact. One suggested design:*
 - 3.3.6.1 *From 0.75-in. plywood, cut into three squares: 18 in., 14 in., and 10 in.*
 - 3.3.6.2 *Cut holes to closely fit the 1-gal. sample bucket in the centers of the smaller two plywood squares.*
 - 3.3.6.3 *Center the 10-in. board on top of the 14-in. board, and fasten them together.*
 - 3.3.6.4 *Center these on top of the 18-in. board, and fasten together.*
 - 3.3.6.5 *To a corner of this unit, fasten an 8-ft. length of 1/8-in. diameter flexible, steel cable.*
 - 3.3.6.6 *To the other end of the cable, attach a 10-in. length of steel conduit or wooden dowel as a "T" handle used to slide the filled bucket from windrowed quicklime.*
 - 3.3.7 *Plastic sample bag, 18 × 9.5 in.—General Warehouse No. 2005000.*
- 3.4 *Commercial Lime Slurry and Carbide Lime Slurry:*
 - 3.4.1 *Safety goggles.*
 - 3.4.2 *Polyethylene bottle, 1/2 gal., large mouth (3.5-in. diameter), with a screw cap—Nalgene Company No. 2234-0020.*
 - 3.4.3 *Plastic electrical tape, PVC 0.75 in. wide.*
 - 3.4.4 *Cloth rag or shop towel.*

4. SAMPLING PROCEDURES

4.1 *Hydrated Lime:*

4.1.1 *Bulk from Loaded Tank Trucks:*

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

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

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

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

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

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

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

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

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

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

4.1.1.3.3 Remove the tube.

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

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

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

Note 1—Exposure to the atmosphere contaminates lime.

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

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

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

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

4.1.2 *Bagged from Bag Trucks:*

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

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

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

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

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

Note 3—Exposure to the atmosphere contaminates lime.

4.1.3 *Bagged from Bagged Shipments:*

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

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

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

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

Note 4—Exposure to the atmosphere contaminates lime.

4.2 *Quicklime:*

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

4.2.1 *Discharged from Tank Trucks:*

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

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

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

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

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

4.2.1.4 Ship samples by motor freight only.

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

4.2.2 *From Tank Trucks:*

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

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

4.2.2.3 Ship samples by motor freight only.

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

4.3 *Commercial Lime Slurry:*

4.3.1 *From the Truck:*

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

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

- 4.3.1.2 Open the sampling valve quickly and completely during sampling.

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

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

- 4.3.1.5 Take one sample to represent the truckload.

- 4.3.1.6 A Department representative must witness sampling.

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

4.3.2 *From the Plant:*

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

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

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

- 4.3.2.4 Take one sample to represent the truckload.

- 4.3.2.5 A Department representative must witness sampling.

4.4 *Carbide Lime Slurry:*

4.4.1 *From the Truck:*

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

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

- 4.4.1.2 Open the sampling valve quickly and completely during sampling.

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

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

4.4.1.5 Take one sample to represent the truckload.

4.4.1.6 A Department representative must witness the sampling.

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

PART II—TESTING HYDRATED LIME

5. SCOPE

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

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

6. APPARATUS

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

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°C).*

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

6.4 *Hard-surfaced temperature resistant board.*

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

6.6 *Vacuum desiccator.*

6.7 *Silica-gel desiccant, indicating type.*

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

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

6.10 *Weighing boats.*

6.11 *Spatula.*

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

- 6.13 *Tall form beakers*, 400 mL, borosilicate type glass.
- 6.14 *Beaker*, 200 mL.
- 6.15 *Buret*, class A, of the following types:
- 100-mL capacity, with a 50-mL bulb at the top and a 50-mL graduated tube
 - 50-mL capacity with 0.1-mL subdivisions.
- 6.16 *Flasks*, class A volumetric, with the following capacities:
- 250 mL
 - 1000 mL.
- 6.17 *Graduated cylinders*, with the following capacities:
- 100 mL
 - 200 mL
 - 1000 mL
 - 2000 mL.
- 6.18 *Stirrer*, magnetic type.
- 6.19 *Stopwatch*, to register up to 30 min.
- 6.20 *Plastic tubing*, 3/8-in. ID, 1/8-in. wall thickness of suitable length, fitted with a Bunsen “fan-type” burner attachment at the outlet end of the hose.
- 6.21 *Water pressure gauge*, 0–30 psi, 3-in. diameter face, with 0.375-in. IPS female thread fitting.
- 6.22 *Standard U.S. sieves*, meeting the requirements of Tex-907-K, in the following sizes:
- No. 6
 - No. 30.
- 6.23 *pH meter*, with an accuracy of ± 0.1 pH unit or better, with automatic temperature compensation and standard combination type electrode.
- 6.24 *pH meter reference electrode filling solution*, KCl, 4 M saturated with AgCl.
- 6.25 *Sample vial*, 1-3/4-in. diameter $\times$ 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.

7. REAGENTS

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

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

8. SOLUTIONS

8.1 *Sodium Hydroxide 1.0 N:*

8.1.1 *Preparation:*

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

8.1.1.2 Add 967.6 mL of 50% sodium hydroxide solution.

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

8.1.1.3 Mix for 5–10 min.

8.1.1.4 Bring to 18.5 L mark on the container.

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

8.1.1.6 Standardize with potassium hydrogen phthalate.

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

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

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

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

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

8.1.2.5 Add five drops of phenolphthalein indicator solution.

- 8.1.2.6 Titrate with the sodium hydroxide solution to a visual endpoint of light pink, which will persist for at least 60 sec.
- 8.1.2.7 Record the amount of sodium hydroxide used.
- 8.1.2.8 Calculate the normality. If the normality is not within the limits 1.000 ± 0.0005 N, add water or sodium hydroxide to adjust the normality.
- 8.1.2.9 If the sodium hydroxide solution is too:
- weak, follow “Example 1” below to calculate the amount of the 50% sodium hydroxide to add.
 - strong, follow “Example 2” below to calculate the amount of deionized water to add.
- 8.1.2.10 Measure and add the correct amount of sodium hydroxide or deionized water. Stir the solution for at least 4 hr.
- 8.1.2.11 Repeat the standardization procedure. Repeat adjustments, if necessary. After all adjustments are complete, standardize the resulting solution.
- 8.1.3 *Calculations:*
- 8.1.3.1 Calculate the normality of sodium hydroxide solution:
- $$\text{Normality of NaOH} = \frac{\text{Wt. of Potassium Hydrogen Phthalate in (g)}}{\text{mL of NaOH required} \times 0.204228}$$
- 8.1.3.2 Adjusting the sodium hydroxide solution to achieve a 1.0 N solution:
- 8.1.3.2.1 Example 1: If base is too weak, for example, if 18.5 L of 0.9907 N solution needs strengthening to 1.0000 N, calculate the amount of 50% sodium hydroxide to add:
- $18.500 \times 0.991 = 18.334$
 - $18.500 - 18.334 = 0.166$
 - $0.166 \times 52.3 = 8.68$ mL of 50% sodium hydroxide solution needed.
- 8.1.3.2.2 Example 2: If base is too strong, for example, if the 18.5 L of 1.021 N sodium hydroxide solution needs weakening to 1.0000 N, calculate the amount of deionized water to add:
- $18.500 \times 1.021 = 18.888$
 - $18.888 - 18.500 = 0.388$ L or 388 mL of deionized water needed.
- 8.2 *1.0 N Hydrochloric Acid Solution:*
- 8.2.1 *Preparation:*
- 8.2.1.1 Fill a 19-L glass container 1/2–2/3 full with deionized water.

- 8.2.1.2 Add 1625 mL of concentrated hydrochloric acid.
Note 11—It is important to add the acid to a sizable portion of water to dissipate the heat evolved and avoid hazardous spattering of hot acid, fuming, etc.
- 8.2.1.3 Add sufficient water to bring the total volume to 19 L.
- 8.2.1.4 Stir the solution at least 4 hr., but preferably 12 hr.
- 8.2.1.5 Standardize against the sodium hydroxide.
- 8.2.2 *Standardization of a 1.0 ± 0.0005 N hydrochloric acid solution:*
- 8.2.2.1 Transfer 40 mL of the hydrochloric acid solution into a 400-mL tall beaker using a pipette for accuracy.
- 8.2.2.2 Add 140 mL of deionized water.
- 8.2.2.3 Add five drops of phenolphthalein indicator.
- 8.2.2.4 Fill a 50-mL buret with 1.0 N sodium hydroxide solution. Titrate hydrochloric acid with the sodium hydroxide solution to a visual endpoint of light pink, which will persist for at least 60 sec. Record the amount of the sodium hydroxide solution used.
- 8.2.2.5 Calculate the normality. If the normality is not within the limits of 1.000 ± 0.0005 N, add hydrochloric acid or water to adjust the normality.
- 8.2.2.6 If the hydrochloric acid solution is too:
- weak, follow “Example 1” below to calculate the amount of the concentrated hydrochloric acid to add.
 - strong, follow “Example 2” below to calculate the amount of deionized water to add.
- 8.2.2.7 Measure and add the correct amount of hydrochloric acid or deionized water. Stir the solution for at least 2 hr.
- 8.2.2.8 Repeat the standardization procedure. Repeat adjustments, if necessary. After all adjustments are complete, standardize the resulting solution.
- 8.2.3 *Calculations:*
- 8.2.3.1 Calculate the normality of hydrochloric acid solution:

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

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

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

- $19.700 \times 0.991 = 19.523$
- $19.700 - 19.523 = 0.177$
- $0.177 \times 82.5 = 14.6$ 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 ± 0.05 g of potassium hydrogen phthalate into a weighing boat.

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

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

8.3.4 Dilute the solution to 1000 mL.

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

Table 1—pH of 0.05 M Potassium Hydrogen Phthalate

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

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

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

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

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

8.4.4 Dilute the solution to 1000 mL.

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

Table 2—pH of 0.01 M Borax Solution

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

8.5 *Phenolphthalein Indicator:*

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

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

8.5.3 Stir until all the phenolphthalein dissolves.

8.5.4 Add 50 mL of deionized water and stir.

8.6 *Bromophenol Blue Indicator:*

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

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

8.6.3 Dilute the mixture with deionized water to 250 mL.

9. SAMPLE PREPARATION

- 9.1 Mix the sample well for 1–2 min.
- 9.2 Invert and shake the bucket at least three times.
- 9.3 Remove the bucket lid and scoop out enough material to fill a sample vial 1/2–3/4 full.
- 9.4 After sample withdrawal, close the bucket and sample vial to minimize atmospheric contamination.

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

10. TESTING PROCEDURES

- 10.1 *Titration to pH 8.3:*
 - 10.1.1 Weigh out on an analytical balance 2.804 ± 0.0050 g of lime from the sample vial. Reseal the vial. Record the weight of the lime sample.
 - 10.1.2 Add 150 mL of deionized water to a 400-mL tall-form beaker.
 - 10.1.3 Transfer the weighed sample to the 400-mL tall-form beaker.

Note 13—To minimize loss of sample, transfer material slowly.
 - 10.1.4 Add a stirring magnet and place the beaker on a magnetic stirrer. Stir the sample mixture.
 - 10.1.5 Calibrate the pH meter using the 4.0 and 9.0 buffer solutions.
 - 10.1.6 Fill a 100-mL buret with 1.0 N hydrochloric acid solution.
 - 10.1.7 Titrate the sample with the first 50 mL of the 1.0 N hydrochloric acid solution to a pH of 9.0. Titrate as rapidly as the reaction rate of the sample will allow.

Note 14—The response or reaction time of the sample dictates the rate of addition. Lime samples will vary in response to additions of titrant. An excess of titrant causes localized pooling of titrant. Carbon dioxide gas evolves from the reaction of acid with calcium carbonate at a pH of less than 5.4. In the titration to pH 8.3, rapidly add the titrant, but slow enough to keep the pH greater than 6.0 at all times. With addition of titrant, the pH will drop and then, in most cases, recover or rise to a higher value as more solids dissolve and enter into the reaction.
 - 10.1.8 Decrease the rate of titration to a rapid drop rate until reaching a pH of between 6.0 and 9.0 that persists for at least 30 sec.
 - 10.1.9 Use smaller increments of titrant to maintain a pH slightly less than 8.3. As the pH increases, add more titrant until a pH of 8.3 or slightly less persists for 60 sec.
-

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

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

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

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

10.2 *Back-Titration to pH 4.4:*

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

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

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

10.3 *Loss on Ignition:*

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

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

10.3.3 Place the crucible containing the sample in an electric muffle furnace set at $2,000 \pm 20^{\circ}\text{F}$ ($1093 \pm 11^{\circ}\text{C}$) 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 ± 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 ± 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 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:

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

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

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

$$\overline{100.1}$$

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

$$\% \text{CaCO} = 0.1 \times 3.1 = 0.3$$

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

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

$$\overline{94.4}$$

Analysis is 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) ₂
68.0	89.8
69.0	91.2
70.0	92.5
71.0	93.8
72.0	95.1
73.0	96.4
74.0	97.8
75.0	99.1
Factor = 1.32126	

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

Table 4—Calcium Carbonate Value

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

11.2.9 Correction for Calcium Hydroxide Value when CaO is Present:

Table 5—Correction for Calcium

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

11.2.10 Amount of Subtotal Over 100%:

- When quicklime is present: $\% \text{CaO} = (\text{Subtotal} - 100\%) \times 3.11$
- When free water is present: $\% \text{free water} = 100\% - \text{Subtotal}$

Table 6—Proportional Parts

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

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

Table 7—Proportional Parts

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

- 11.2.11.1 Use Table 7 if the subtotal (uncorrected calcium hydroxide value + calcium carbonate value + assumed inert value) is greater than 100%. This means that calcium oxide is present and not free water.
- 11.2.11.2 If the subtotal is less than 100%, then assume the uncorrected calcium hydroxide value is correct. Calcium hydroxide and free water are present.
-

12. OPTIONAL INDICATOR METHOD FOR THE TITRIMETRIC ANALYSIS

12.1 Scope:

- 12.1.1 Use this method in lieu of the prescribed pH meter method; however, for all referee tests, or in cases of dispute, the prescribed pH meter method is the official method.
- 12.1.2 In some cases, impurities such as aluminum compounds will prevent or hinder the appearance of the color change in the back-titration portion of this optional method. In these cases, use the prescribed pH meter method.

12.2 Titration to Phenolphthalein Endpoint:

- 12.2.1 Weigh out on an analytical balance 2.804 ± 0.0050 g of lime from the sample vial. Reseal the vial. Record the weight of the lime sample.
- 12.2.2 Add 150 mL of deionized water to a 400-mL tall-form beaker.
- 12.2.3 Transfer the sample to the beaker.
Note 20—To minimize loss of sample, transfer material slowly.
- 12.2.4 Add a stirring magnet and place the beaker on a magnetic stirrer. Stir the sample.
- 12.2.5 Add five drops of phenolphthalein indicator to the sample mixture.
- 12.2.6 Fill a 100-mL buret with 1.0 N hydrochloric acid.
- 12.2.7 Titrate to the point where the color of the solution changes temporarily from pink to clear.
- 12.2.8 Wait for the pink color to return.
- 12.2.9 Add titrant until the color change appears imminent.
- 12.2.10 Add acid in 4-drop increments until the pink coloration disappears for 35 sec.
- 12.2.11 Add acid in 2-drop increments until the endpoint occurs. Record the amount of hydrochloric acid used to reach the endpoint.
Note 21—The endpoint is the failure of the mixture to regain its pink coloration for 60 sec. after the last addition of the titrant. After reaching the endpoint, ignore the reappearance of pink color. The phenolphthalein endpoint is the equivalent to the pH 8.3 endpoint described in the official pH meter method of titration.
-

- 12.2.12 Add an excess of hydrochloric acid to the sample. Record the total volume of acid added.
Note 22—The sample mixture contains a “measured excess” of hydrochloric acid.
- 12.3 *Back Titration to the Bromophenol Blue Endpoint:*
- 12.3.1 Fill a 50-mL buret with 1.0 N sodium hydroxide.
- 12.3.2 Add 2.0 mL of bromophenol blue indicator to the sample mixture.
- 12.3.3 Titrate until the color of the mixture changes from yellow to blue with a slight tinge of purple and maintains the blue color for 10 sec., after a 1-drop addition of titrant.
- 12.3.4 Record the amount of sodium hydroxide used to reach the endpoint.
- 12.3.5 Use the same calculation method to determine active lime content as described under Section 10.

PART III—TESTING COMMERCIAL LIME SLURRY

13. SCOPE

- 13.1 This part discusses the method for testing high-calcium type commercial lime slurry and assumes that the principal constituents present in the lime are:
- calcium hydroxide,
 - water,
 - calcium carbonate, and
 - inert matter such as silica dioxide.

14. APPARATUS

- 14.1 *Equipment*, listed under Part II, Section 6.
- 14.2 *Eyedropper*, large bore polyethylene.
- 14.3 *Weight-per-gallon cup*, stainless steel, with tare weight.
- 14.4 *Polyethylene bottles*, 1 pt., with screw caps.
- 14.5 *Glass rod*.
- 14.6 *Beaker*, 1000 mL.

15. REAGENTS AND SOLUTIONS

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

16. SAMPLE PREPARATION**16.1** *Lime Slurry Sample Preparation:*

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

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

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

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

16.2 *Determining Bulk Density:*

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

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

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

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

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

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

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

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

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

16.2.8 *Calculations:*

16.2.8.1 Calculate bulk density:

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

Where:

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

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

16.3 *Determination of Sample Size:*

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

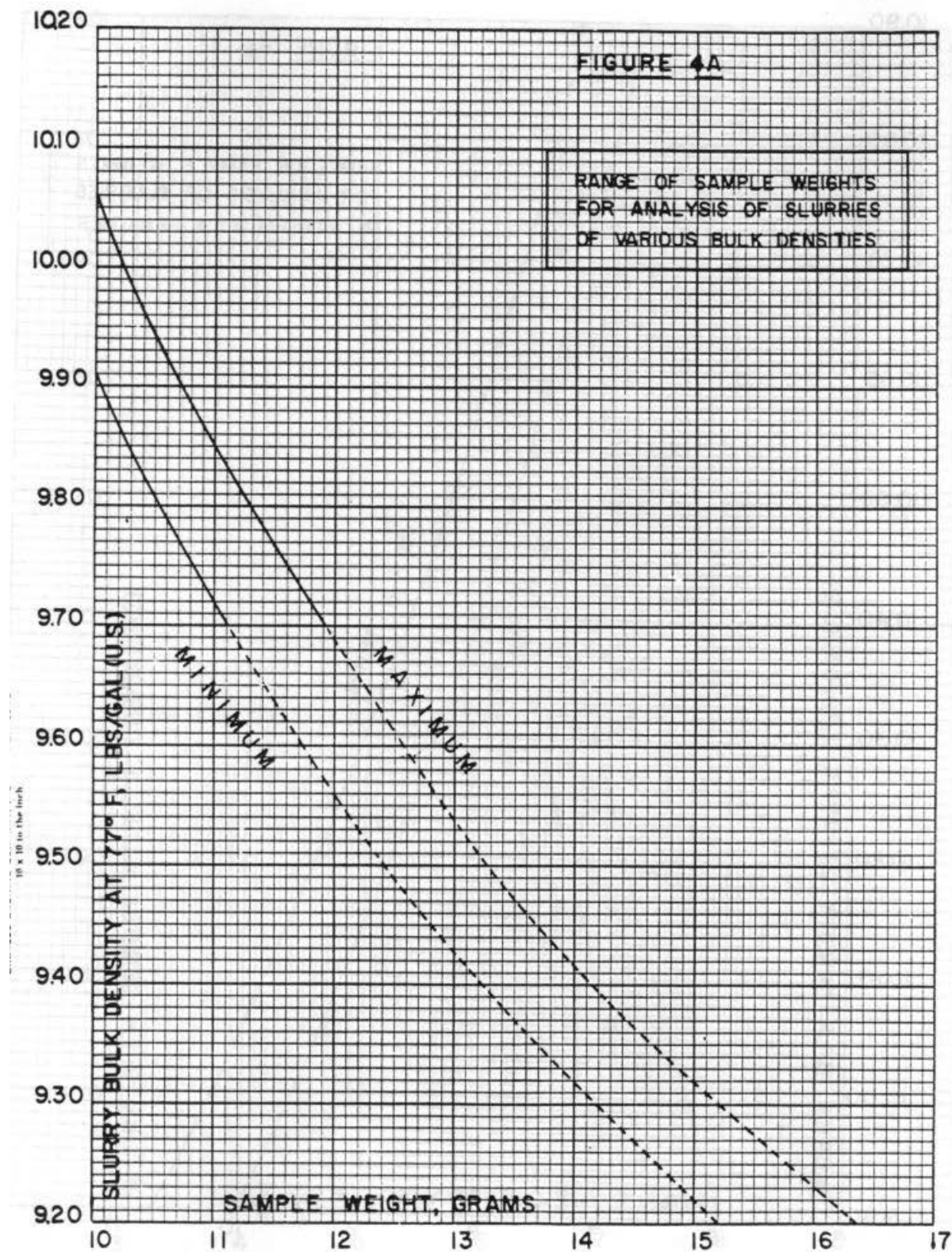


Figure 1—Range of Sample Weights for Analysis of Slurries—No. 1

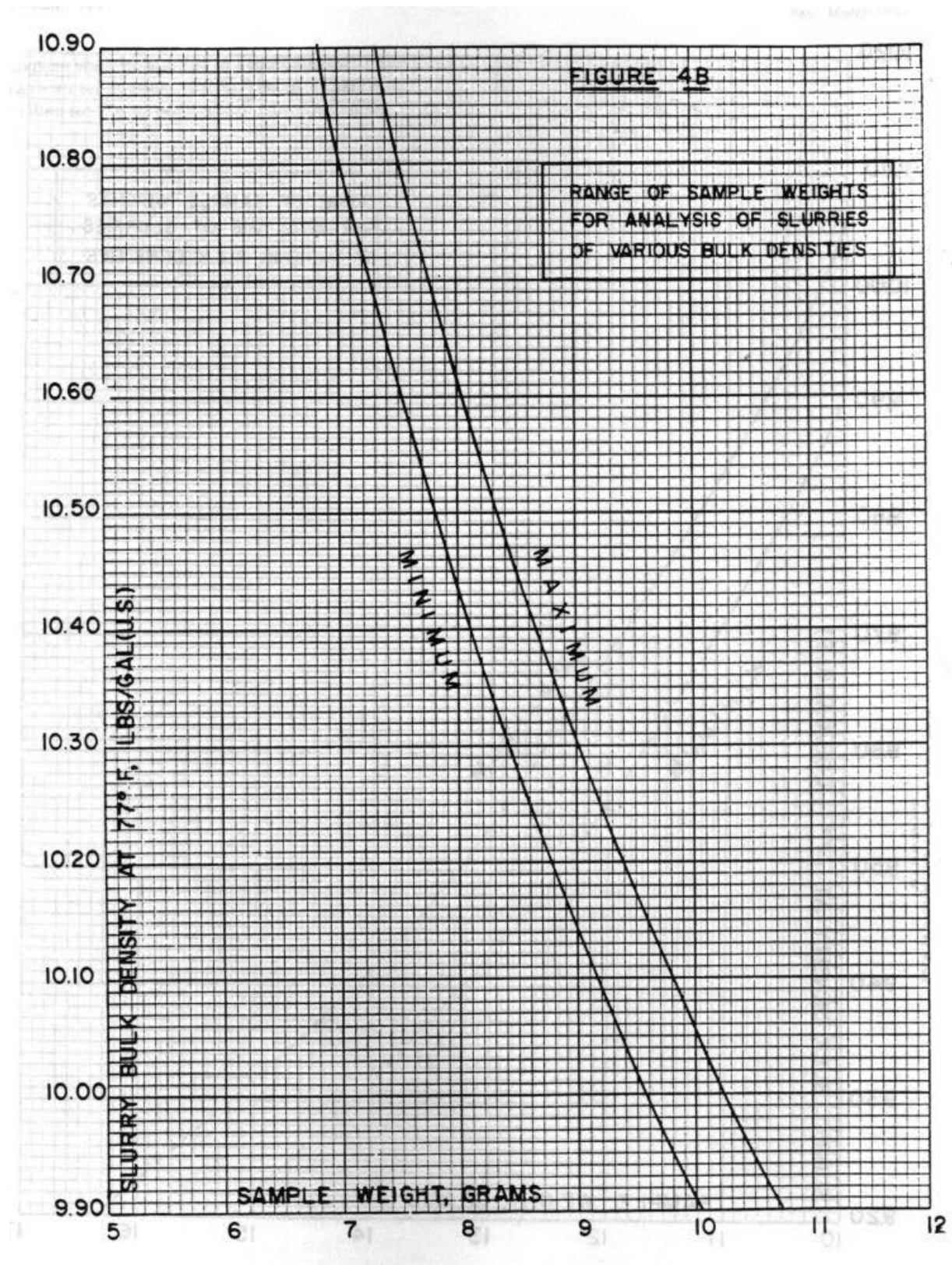


Figure 2—Range of Sample Weights for Analysis of Slurries—No. 2

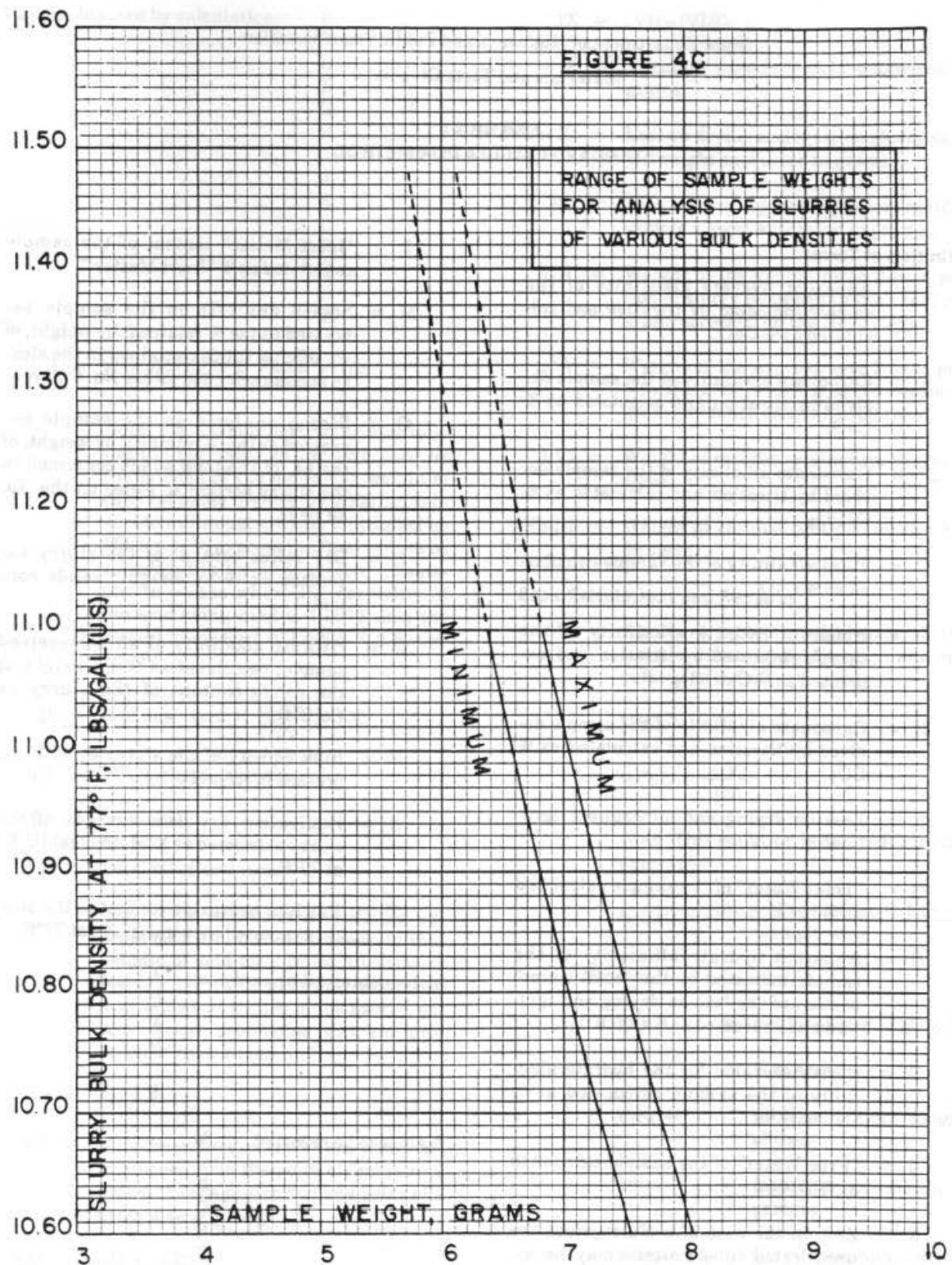


Figure 3—Range of Sample Weights for Analysis of Slurries—No. 3

17. TESTING PROCEDURES**17.1** *Titration to pH 8.3:*

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

17.2 *Back-Titration to pH 4.4:*

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

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

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

17.3 *Loss on Ignition:*

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

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

17.3.3 Place the crucible with slurry into a $212 \pm 4^\circ\text{F}$ ($100 \pm 2^\circ\text{C}$) 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 ± 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, % $\text{Ca}(\text{OH})_2$:

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

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.

PART IV—TESTING QUICKLIME

19. SCOPE

19.1 This part discusses the method for testing quicklime.

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

20. APPARATUS

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

20.2 *Laboratory grinder*.

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

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

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

20.6 *Erlenmeyer flasks*, 500 mL.

20.7 *Rubber stoppers*, No. 10 size.

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

21. REAGENTS

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

22. PREPARING SOLUTIONS

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

22.1.1 *Sugar Solution:*

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

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

- 22.1.1.3 Add one drop of phenolphthalein indicator.
 - 22.1.1.4 Add 1.0 N sodium hydroxide, drop by drop, until observing a faint pink color.
-

23. SAMPLE PREPARATION

- 23.1 Divide the 1-gal. sample into two portions through the splitter.
 - 23.2 Return and set aside one of the portions to the 1-gal. can.
 - 23.3 Measure 300 g of the remaining portion.
 - 23.4 Grind the 300-g portion for chemical analysis to pass a No. 30 sieve. Transfer the ground lime to a sample vial and close the lid tightly.
 - 23.5 Measure 800 g from the remainder of this portion. Set aside this 800-g portion in a sealed container.
Note 33—Protect all portions from contamination. Crush samples that contain flint or other extremely hard material to pass through a 1/8-in. sieve in a jaw-type crusher before grinding.
-

24. TESTING PROCEDURES

- 24.1 *Titration to Phenolphthalein Endpoint:*
 - 24.1.1 Boil 60 mL of deionized or distilled water. Add 10 mL of boiled water to a 500-mL Erlenmeyer flask.
 - 24.1.2 Weigh 2.8 ± 0.3 g of the ground quicklime sample in a weighing boat. Record the exact amount of quicklime to the nearest 0.0001 g.
 - 24.1.3 Transfer the lime sample to the Erlenmeyer flask.
 - 24.1.4 Add 50 mL of boiled water to the Erlenmeyer flask.
 - 24.1.5 Stopper the flask with a No. 10 rubber stopper and swirl for a few seconds.
 - 24.1.6 Place the sample on a hot plate and boil for 2 min.
 - 24.1.7 Remove the sample from the hot plate and allow it to cool to room temperature.
 - 24.1.8 Add 150 mL of the sugar solution to the flask, stopper the flask, and let it stand for 15 min. Swirl the flask every 5 min.
 - 24.1.9 Add five drops of phenolphthalein indicator solution and a stirring magnet to the sample. Stir the sample on a magnetic stir.
 - 24.1.10 Fill a 100-mL buret with 1.0 N hydrochloric acid. Titrate the sample with the hydrochloric acid until the first instance the pink color disappears. This is the endpoint.
-

- 24.1.11 Record the volume of hydrochloric acid used to reach the endpoint. Use the calculations to determine the percent active CaO to the nearest 0.1%.
- 24.2 *Dry Sieve Analysis:*
- 24.2.1 Weigh the previously reserved 800-g portion on a top loading balance to the nearest 0.1 g.
- 24.2.2 Shake the 800-g portion through a nest of sieves into a catch pan in 200-g increments. The nest of sieves includes the 1 in., 3/4 in., No. 6, and No. 100.
Note 34—Perform this operation under a hood, while wearing a dust/mist mask.
- 24.2.3 Weigh the amount caught in each sieve to the nearest 0.1 g, and then recombine them for the wet sieve analysis.
- 24.3 *Wet Sieve Analysis:*
- 24.3.1 Add 2600 mL tap water to the mixer bowl, return to the mixer, and attach the wire whip.
- 24.3.2 Weigh 800 g of quicklime, in increments of 80 g, into ten tin containers.
- 24.3.3 Lower the wire whip, turn on the mixer, and add one of the increments. Lower the shield. Let stir for 3 min.
- 24.3.4 Continue adding the 80-g increments every 3 min. until 10 min. have lapsed. At 10 min., stop the mixer for 30 sec. and record the temperature of the slurry. After 30 sec., restart the mixer and mix the slurry for 2 min. to complete the 3 min. cycle of the last added increment.
- 24.3.5 Add the next increment. Continue the cycle of adding the 80-g increments every 3 min. and stopping every 10 min. for 30 sec. to read the temperature, until all the quicklime is mixed.
- 24.3.6 When finished, take the bowl out of the mixer and rinse the wire whip.
- 24.3.7 Weigh the No. 6 sieve on a top loading balance. Record the weight of the sieve to the nearest 0.1 g.
- 24.3.8 Using tap water, wash the reacted sample onto a No. 6 sieve. Ensure that none of the slurry sample is lost over the sides of the sieve. Use a water pressure gauge to maintain 14–18 psi water pressure.
Note 35—Apply the wash water in a fantail spray pattern from the 3/8 in. ID plastic tubing fitted with a Bunsen “fan-type” burner attachment at the outlet.
- 24.3.9 Continue to wash the sample for 30 min. or until the wash water is clear, whichever occurs first.
- 24.3.10 After washing, place the sieve in a drying oven at a temperature of $212 \pm 4^{\circ}\text{F}$ ($100 \pm 2^{\circ}\text{C}$) 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:

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

Where:

V = volume of hydrochloric acid, mL

N = normality of hydrochloric acid

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

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

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

Where:

% retained = weight percent of residue retained on the sieve

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

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

PART V—TESTING CARBIDE LIME SLURRY

26. SCOPE

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

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

27. APPARATUS

- 27.1 *Equipment*, listed under Part II, Section 6.
- 27.2 *Eyedropper*, large bore polyethylene.

- 27.3 *Weight-per-gallon cup, stainless steel, with tare weight.*
- 27.4 *Polyethylene bottles with screw caps, 1 pt.*
- 27.5 *Glass rod.*
- 27.6 *Beaker, 2000 mL.*
-

28. REAGENTS AND 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.
-

29. SAMPLE PREPARATION

29.1 Carbide Lime Slurry Sample Preparation:

- 29.1.1 Set the slurry jug in a roller and agitate for 3–4 hr.
- 29.1.2 Use a glass rod to determine if the lime sample is in suspension. If any lime remains caked on the bottom or sides, place the slurry jug back on the roller and agitate.
- 29.1.3 When the slurry is well mixed, pour a representative sample in a 1-pt. polyethylene bottle with a screw top.
- Note 36**—Clean the threaded portion of the bottles and the inside threads of the cap. If slurry remains on these places, the seal will not be airtight and the slurry may dehydrate.

29.2 Determining Bulk Density:

- 29.2.1 Weigh a clean and empty weight-per-gallon cup on a top loading balance. Tare the weight of the cup.
- 29.2.2 Shake the 1-pt. bottle of slurry vigorously for 1 min. Rapidly pour the slurry from the 1-pt. bottle into the weight-per-gallon cup. Fill the cup to just below the top rim.
- 29.2.3 Tap the bottom of the filled cup against a hard surface to remove air bubbles.
- 29.2.4 Place the lid on the cup, gently pressing it into place. Excess slurry will flow out the small center opening in the top.
- Note 37**—If slurry does not flow out of the opening, use additional slurry. Agitate the sample before adding more slurry.
- 29.2.5 Carefully wipe off all of the excess slurry from the outside of the cup and lid.
- 29.2.6 Place the weight-per-gallon cup on a top loader balance. Weigh the cup and its contents to the nearest 0.1 g.
- Note 38**—Make sure the tared weight remains displayed on the balance just before measuring the weight of the filled cup.
-

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

29.3 *Calculations:*

29.3.1 Calculate bulk density:

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

Where:

BD = bulk density, lb./gal.

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

29.4 *Determination of Sample Size:*

29.4.1 Refer to Part III, Section 16.3.

30. TESTING PROCEDURES

30.1 *Titration to pH 8.3:*

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

30.2 *Back-Titration to pH 4.4:*

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

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

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

30.3 *Loss on Ignition:*

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

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

30.3.3 Place the platinum crucible with slurry into a $212 \pm 4^{\circ}\text{F}$ ($100 \pm 2^{\circ}\text{C}$) 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 ± 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)₂:

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

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.