

SB 103

Materials Analyst Specialist

CERTIFICATION/TRAINING PROGRAM 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 HMACE 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.



SB103 Materials Analyst Specialist

TABLE OF CONTENTS

Rights and Responsibilities

Website

Tex-100-E Sampling Soils and Flexible Base

Tex-101-E Preparing Soil and Flexible Base Materials for Testing (Parts 1 & 2)

Tex-128-E Determining Soil pH

Tex-129-E Resistivity of Soil Materials

Tex-145-E Determining Sulfate Content in Soils, Colorimetric Method

Tex-400-A Sampling Aggregate and Stone Riprap

Tex-600-J Sampling and Testing Lime (Part 1)

©2023 HMAC - All Rights Reserved

No part of this publication may be reproduced, stored in a retrieval system or transmitted in any form or by any means, mechanical, electronic, photocopying, recording or otherwise without the prior written permission of the Texas Asphalt Pavement Association.



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

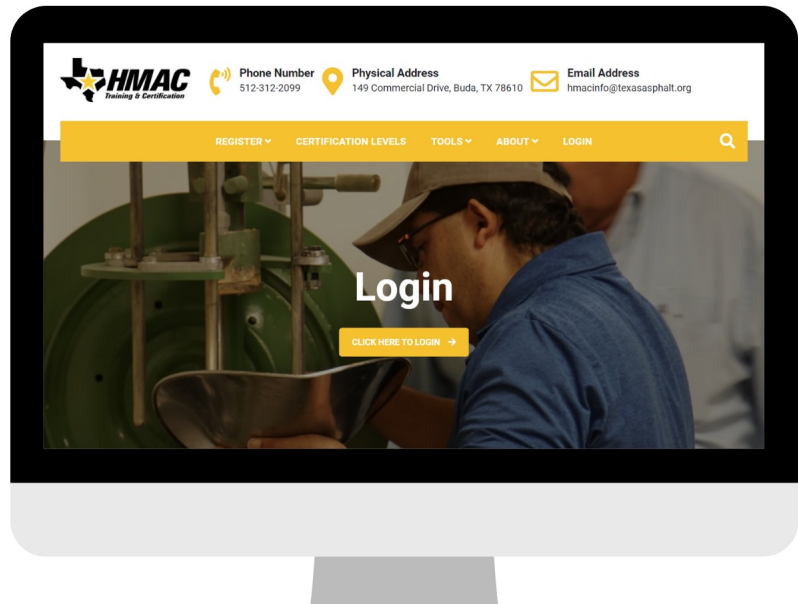
DATE

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

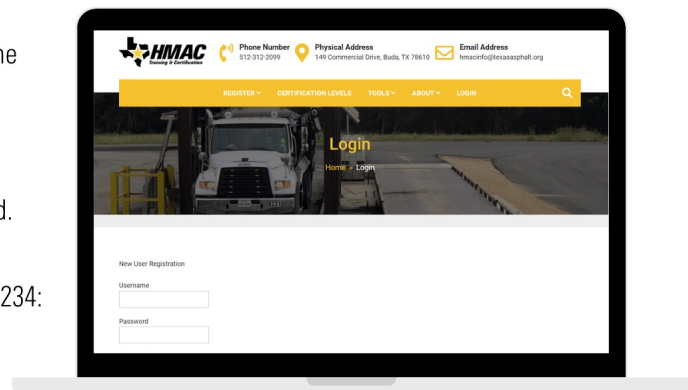
Same as your username, but add a "+" at the end.

Example

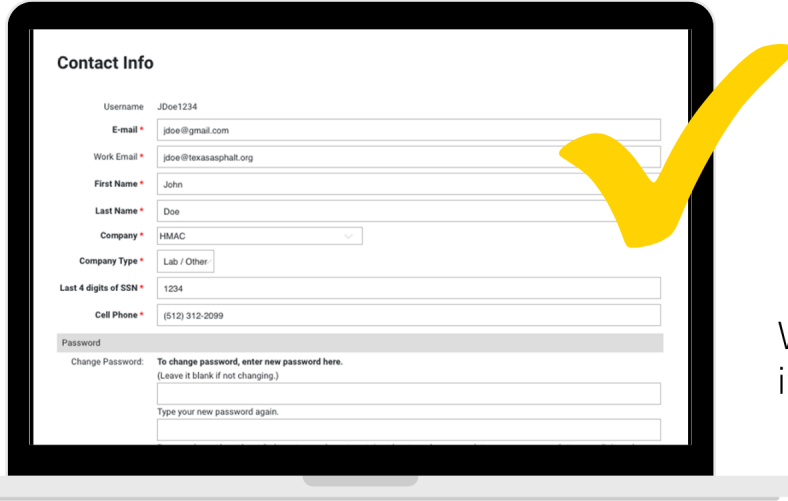
If your name is John Doe and your SSN ends in 1234:

Username: JDoe1234

Password: JDoe1234+



UPDATE INFORMATION



Contact Info

Username

E-mail

Work Email

First Name

Last Name

Company

Company Type

Last 4 digits of SSN

Cell Phone

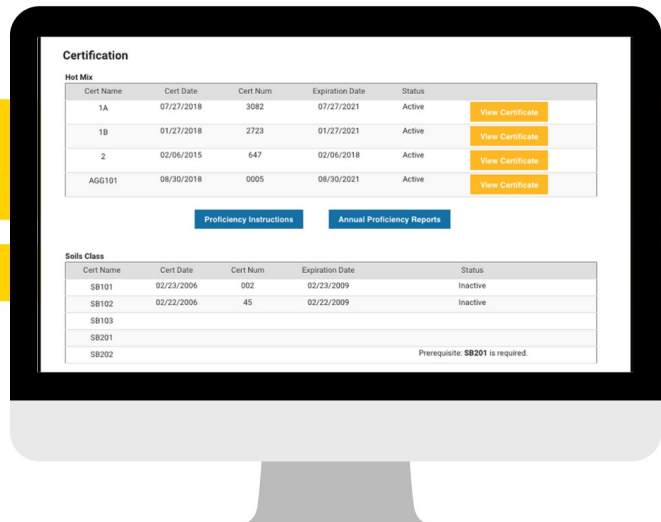
Password

Change Password:

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	3982	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 FLEXIBLE BASE**

TxDOT Designation: Tex-100-E

Effective Date: August 2023

1. SCOPE

- 1.1 Use this test procedure to sample soils that are used as embankment and non-select backfill materials. Use this procedure to sample flexible base from completed stockpiles and roadway windrows, and for the preparation of the samples for laboratory testing.
- 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.*

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 or non-select backfill.
- 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 for soil that will be treated and 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 to 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 to 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 to 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.
-

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.
-

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](#)).
 - 6.7 Combine the remaining three quarters with any remaining material from Section 6.6 and proceed to [Tex-101-E](#), Part II to prepare the material for Moisture-Density curve ([Tex-113-E](#)), wet ball mill ([Tex-116-E](#)), and compressive strength ([Tex-117-E](#)) testing.
-

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.
 - 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](#)).
-

8. ARCHIVED VERSIONS

8.1 Archived versions are available.

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

1. SCOPE

- 1.1 This method describes three procedures for preparing of soil and flexible base samples for soil constants and particle size analysis, compaction and triaxial, and sieve analysis of road-mixed material.
- 1.2 The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.
-

2. DEFINITIONS

- 2.1 *Soil Binder*—Soil binder is any material passing the 425 μm (No. 40) sieve.
- 2.2 *Percent Soil Binder*—Percent soil binder is equal to 100 times the ratio of the oven-dry mass of the soil binder to the oven-dry mass of the total.
-

3. APPARATUS

- 3.1 *Set of standard U.S. sieves*, meeting the requirements of Tex-907-K.
- 3.2 *Scale*, with a minimum capacity of 36 kg (80 lb.), with a minimum accuracy and readability of 5 g or 0.1% of the test load, whichever is greater.
- 3.3 *Drying oven*, maintained at 60°C (140°F).
- 3.4 *Crusher* (optional), which can be adjusted to produce material passing a 2.00 mm (No. 10) sieve.
- 3.5 *Mechanical pulverizer* (optional).
- 3.6 *Wedgewood mortar and pestle*.
- 3.7 *Scoop*.
- 3.8 *Small siphon tube* (optional).
-

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

3.10 *Filter paper, non-fibrous.*

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

3.12 *Mechanical mixer (stirring device).*

3.13 *Dispenser cup.*

3.14 *Plaster of Paris molds (optional).*

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

4. MATERIALS

4.1 *Tap water.*

5. SAMPLE IDENTIFICATION

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

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

6. SCOPE

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

7. PREPARING SAMPLES

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

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

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

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

- 7.1.4 If the amount of aggregate larger than 425 μm (No. 40) is easily distinguishable, remove these particles by hand and proceed to Section 7.1.27.
- 7.1.5 For materials containing a considerable amount of aggregate, separate the fine loose binder from the coarse particles by sieving over a 425 μm (No. 40) sieve.
- 7.1.6 Set the soil binder passing the sieve aside to recombine with the additional binder obtained from Sections 7.1.8 through 7.1.24.
- 7.1.7 If desired, slake the total material.
- 7.1.8 Place the material to be slaked into a pan.
- 7.1.9 Cover the material completely with water and soak for a minimum of 12 hours, unless Tex-102-E determines a shorter time.
- 7.1.10 Place the empty 425 μm (No. 40) sieve into a clean pan and pour the liquid from the wet sample through it.
- 7.1.11 Transfer the wet sample to the sieve in increments not exceeding 450 g.
- 7.1.12 Pour water over the sieve until the water level is about 12.5 mm (0.5 in.) above the sample on the sieve.
- 7.1.13 Alternately agitate the sieve up and down and stir the sample by hand.
- 7.1.14 If the material retained on the sieve contains lumps that have not disintegrated, crumple any that can be broken down between thumb and fingers and wash through the sieve.
- 7.1.15 After all the soil binder appears to have passed through the sieve, hold the sieve above the pan and wash the retained aggregates clean by pouring a small amount of water over it and letting the water drain into the pan.
- 7.1.16 Transfer the retained aggregate from the sieve to a clean pan.
- 7.1.17 Repeat the procedure in Sections 7.1.10 through 7.1.16 until all of the soaked sample has been washed.
- 7.1.18 Dry the retained aggregate portion of the sample in a 60°C (140°F) oven.
- 7.1.19 Re-screen over the 425 μm (No. 40) sieve and add the binder passing the sieve to the soil binder obtained in Section 7.1.6.
- 7.1.20 Weigh the mass of the aggregate and retain for use in Tex-110-E, Part I.
- 7.1.21 Place the pan containing the soil binder and wash water aside, where it will not be disturbed, until all the soil has settled to the bottom of the pan and the water above the soil is clear.
- 7.1.22 Decant the water off the soil.

- 7.1.23 Dry the remaining soil in a 60°C (140°F) oven.
- 7.1.24 In cases where the materials fail to settle overnight, evaporate the water by placing the sample in a 60°C (140°F) oven until it is dry, or siphon the water on to a plaster of Paris mold lined with filter paper.
- 7.1.25 When the water has disappeared, place the filter paper with adhering soil in a pan and dry in the oven.
- 7.1.26 Sweep the dry soil from the filter paper with a stiff brush into the pan of fines.
- 7.1.27 Break down the dried soil binder with a mortar and pestle or use a suitable mechanical pulverizer with an opening set from 635 to 889 μm (0.025 to 0.035 in.)
- 7.1.28 If a pulverizer is used, any material still aggregated in lumps larger than 425 μm (No. 40) should be broken down with a mortar and pestle.
- 7.1.29 Combine all of the soil binder obtained and weigh the mass to the nearest 5 g.
- 7.1.30 Mix thoroughly to produce a uniform sample of all of the particles.
- 7.1.31 Add the masses obtained in Sections 7.1.20 and 7.1.29, and record the sum as the Total Dry Mass of the sample.
- 7.2 *Wet Preparation (Method B)*—to be used for the analysis of soil constants only
- 7.2.1 Select a representative sample according to Tex-100-E or Tex-400-A.
- 7.2.2 Make the sample large enough to yield at least 300 g of soil binder.
- 7.2.3 Place the sample in a clean pan.
- 7.2.4 Cover the sample completely with clear water.
- 7.2.5 Soak soils with moderate to high Plasticity Indices (PI) for a minimum of 12 hours, unless Tex-102-E determines a shorter time.
- 7.2.6 Soak flexible base and low PI materials for a minimum of two hours.
- 7.2.7 Sieve the wet sample into a clean pan in increments of approximately 450 g over a 2.00 mm (No. 10) sieve to remove large aggregate particles.
- 7.2.8 Wash the aggregate retained on the sieve with a small amount of water.
- 7.2.9 Discard the portion of material retained on the sieve.
- 7.2.10 For soils and base materials with low PI, go to Section 7.2.14.
- 7.2.11 For soils with moderate to high PI, place the wet material passing the 2.00 mm (No. 10) sieve into the dispersion cup of a mechanical malt mixer.

- 7.2.12 Do not fill the cup more than half-full.
- 7.2.13 Mix the material for three to five minutes or until the soil binder is separated.
- 7.2.14 Pour the material through a 425 μm (No. 40) sieve into a plaster of Paris bowl lined with filter paper.
- 7.2.15 Vigorously agitate the sieve up and down over the bowl while occasionally stirring the sample by hand to allow as much material as possible to pass the 425 μm (No. 40) sieve.
- 7.2.16 If the material retained on the sieve contains lumps that have not disintegrated, return to the dispersion cup, and remix and wash through the sieve.
- 7.2.17 Sieve until at least 95% of the soil binder appears to have passed through the sieve.
- 7.2.18 Reduce the water content of the material in the plaster of Paris bowl to below the liquid limit.
- 7.2.19 When the sample can be divided into pie-like wedges, and each wedge can be easily removed, it can be used for testing of soil constants.
- 7.2.20 If the soil constants are not to be determined immediately, place the material into an airtight container to prevent moisture loss.
-

8. CALCULATION

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

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

Where:

W_1 = dry mass of soil binder

W_T = dry mass of total sample.

9. REPORT

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

PART II—PREPARING SAMPLES FOR COMPACTION AND TRIAXIAL TESTS

10. SCOPE

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

11. PREPARING SAMPLES

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

11.2 Check specifications for maximum aggregate size.

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

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

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

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

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

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

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

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

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

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

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

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

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

- 11.7 Mix each size to make moisture as uniform as possible.
- 11.8 Weigh each size of material to the nearest 0.1 lb (5 g).
- 11.9 Calculate the cumulative percentages retained on each sieve size as shown under Section 12.
- 11.10 These values are to be used in recombining the sample for compaction specimens.
-

12. CALCULATION

- 12.1 Calculate the cumulative percentages retained on each sieve.

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

PART III—SIEVE ANALYSIS OF ROAD-MIX STABILIZED MATERIAL

13. SCOPE

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

14. PROCEDURE

- 14.1 Select a representative sample from the roadway according to Tex-100-E.
- 14.2 Reduce the sample, using a sample splitter or other approved method, to a minimum of 4.5 kg (10 lb.)
- 14.3 Remove all non-slakable aggregates retained on the specified sieves.
- 14.4 Weigh the sample to the nearest 5 g and record as the Total Mass under Section 15.
- 14.5 Use the specified sieves to separate the sample into different size fractions.
- 14.6 Use either a sieve shaker or the hand method in the sieving operation.
- 14.7 Sieve the sample with a lateral and vertical motion of the sieves, accompanied by a jarring action to keep the material moving over the surface of the sieve.
- 14.8 Continue the sieving operation until no more than one percent of the test sample, by mass, passes through any sieve after one minute of continuous shaking.
-

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

15. CALCULATION

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

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

16. REPORT

- 16.1 Report test results to the nearest whole percent.
-

17. ARCHIVED VERSIONS

- 17.1 Archived versions are available.

Test Procedure for

DETERMINING SOIL pH

**TxDOT Designation: Tex-128-E****Effective Date: August 1999**

1. SCOPE

- 1.1 This method describes the procedure for determining the pH of soils in an aqueous solution.
- 1.2 The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.
-

2. APPARATUS

- 2.1 *pH meter*, with glass electrode, pH range 0–14 ± 0.1.
- 2.2 *Buffer solutions*, such as pH 4.0, 7.0, and 9.0.
- 2.3 *Glass stirring rod*.
- 2.4 *Glass beaker*, 250 mL (8.5 fl. oz.)
- 2.5 *Balance*, Class G1 in accordance with Tex-901-K.
- 2.6 *Thermometer*, 0–100°C (32–212°F).
- 2.7 *Stirring device*, mechanical or magnetic.
-

3. MATERIALS

- 3.1 *Distilled or deionized water*.
-

4. PREPARING SAMPLES

- 4.1 *Soil Sample:*
- 4.1.1 The pH test is performed on the soil binder, minus 425 µm (No. 40) material prepared in accordance with Tex-101-E, Part I.
-

- 4.2 *Compost Sample:*
- 4.2.1 The pH test is performed on minus 4.75 mm (No. 4) material prepared according to the following:
- 4.2.1.1 Obtain a representative sample of approximately 200 g and dry to constant weight at 60°C (140°F).
- 4.2.1.2 Dry sieve material over a 4.75 mm (No. 4) sieve and test material passing.
-

5. PROCEDURE

- 5.1 Heat sample and approximately 200 mL (7 fl. oz.) of distilled water to 45–60°C (112–140°F) in separate containers.
- 5.2 Add 30 g of the prepared dry soil binder to 150 mL (5 fl. oz.) of distilled water.
- 5.3 Stir the sample vigorously and disperse the soil well.
- 5.3.1 Stir the sample every 15 minutes for one hour to disperse the soil and make sure all soluble material is in solution.
- 5.4 After the sample has been added to the water for a period of one hour, record the temperature of the mixture and adjust the pH meter to that temperature.
- 5.5 Standardize the meter (buffer solution of pH 7.0) according to the manufacturer's recommendations.
- 5.6 Clean electrode with water.
Note 1—If scratches are noticed on glass bulb of electrode, replace with new electrode.
- 5.7 Immediately before immersing electrode into mixture, stir and remove glass stirring rod. Place electrode into solution and use stirring bar to make good contact between the solution and the electrode.
- 5.8 Place the tip of the electrode approximately 76 mm (3/4 in.) into the mixture. Allow reading to stabilize.
- 5.9 Read and record the pH value to the nearest tenth of a whole number.
Note 2—If the pH reading appears unstable, leave the electrode immersed until the reading has stabilized, approximately five minutes.
- 5.10 Remove electrode and rinse with water.
- 5.11 If the pH of sample is below 5 or above 9, standardize meter with appropriate buffer and repeat Sections 5.4 through 5.9.

6. REPORT

6.1 Report the pH to the nearest 0.1.



Tex-128-E, Determining Soil pH

pH = _____

Report to the nearest 0.1

Notes:

pH < 7 is acidic

pH = 7 is neutral (distilled water has a pH of 7)

pH > 7 is alkaline

Most soils range from pH 4.5 to pH 8. In this range, pH is generally not considered to be a problem for steel. A pH < 4.5 can cause rapid metal corrosion and serious risks to common construction materials.

Test Procedure for

RESISTIVITY OF SOIL MATERIALS

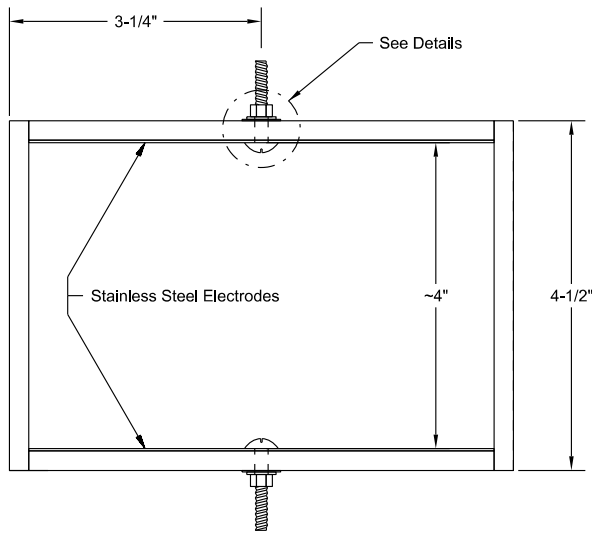
TxDOT Designation: Tex-129-E

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

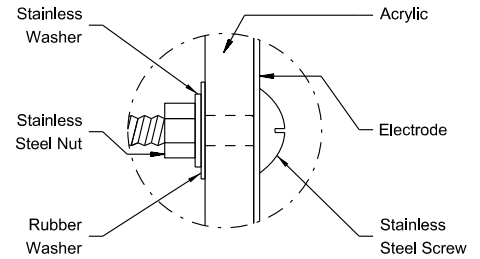
- 1.1 This method determines the resistivity of soil and aggregate materials. Resistivity is an important factor in considering the corrosion potential of soils and aggregates to metal pipe, earth-reinforcing strips, and other metal items in earthwork.
- 1.2 Resistivity is the electrical resistance between opposite faces of a unit cube of material; the reciprocal of conductivity. Resistivity is used in preference to conductivity as an expression of the electrical character of soils (and waters) since it is expressed in whole numbers.
- 1.3 Resistivity measurements indicate the relative ability of a medium to carry electrical currents. When a metallic structure is immersed in a conductive medium, the ability of the medium to carry current will influence the magnitude of galvanic currents and cathodic protection currents. The degree of electrode polarization will also affect the size of such currents.

2. APPARATUS

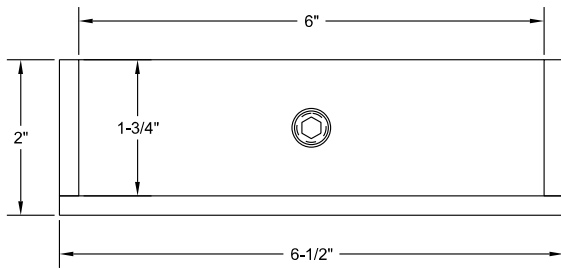
- 2.1 *Balance*, Class G2 in accordance with [Tex-901-K](#), minimum capacity 1,500 g.
- 2.2 *Graduated beaker*, 200 mL
- 2.3 *Miscellaneous equipment*, drying pans, mixing pans, trowel, and small scoop.
- 2.4 *Ohm resistors*, 100, 200, 500, and 900-ohms with a 1% tolerance.
- 2.5 *Resistivity Meter*, alternating current (AC) meter or a 12-V direct current (DC) meter utilizing a Wien Bridge (AC bridge) with a phase sensitive detector and a square wave inverter.
- 2.6 *Soil Box*, two or four electrodes as shown in Figures 1 and 2.
- 2.7 *Standard U.S. Sieve*, No. 8 meeting the requirements of [Tex-907-K](#).
- 2.8 *Straightedge*.
- 2.9 *Thermometer*.
- 2.10 *Wiring*, 18 to 22-gage insulated stranded copper wire with terminal leads.



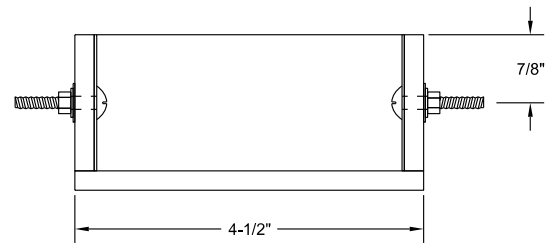
Top View



Screw, Washer & Nut Detail



Front View



Side View

ITEM	QTY.	LENGTH	WIDTH	THICK.
The following items are to be made of plastic (acrylic/plexiglass).				
Bottom	1	6-1/2"	4-1/2"	1/4"
Ends	2	4-1/2"	1-3/4"	1/4"
Sides	2	6"	1-3/4"	1/4"
The following item is to be made of stainless steel.				
Electrodes	2	6"	1-3/4"	20 Gauge
Two of each of the following items are required.				
No. 8-32 x 3/4" Machine Screws, Round Head, Stainless Steel				
Washers, Rubber				
Washer & Nut Set, Stainless Steel or Brass				

Figure 1—Two Electrode Soil Box.

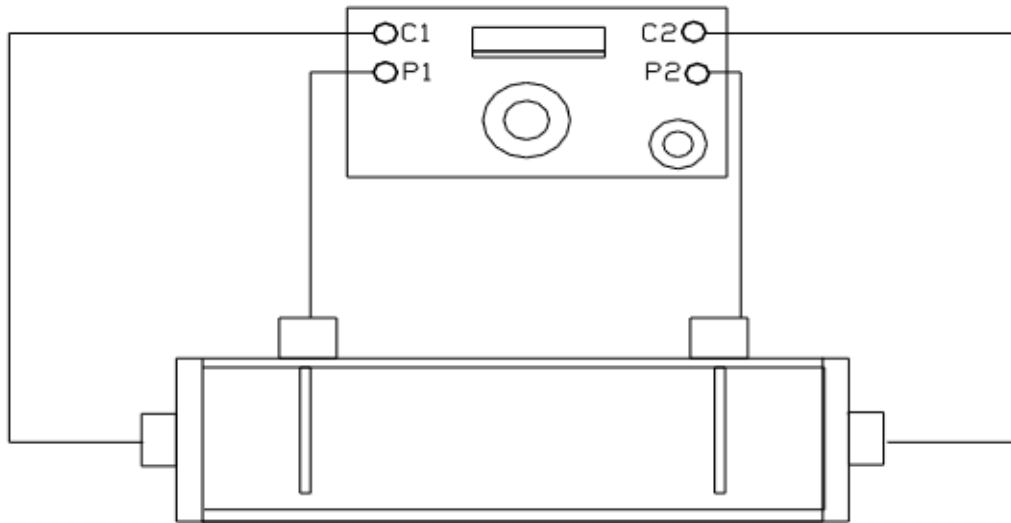


Figure 2—Four Electrode Soil Box Commercially Available. Current Source (Electrode) is Shown as 'C' and Potential Voltage is Shown as 'P'.

3. MATERIAL

- 3.1 Distilled or deionized water.

4. REPORTING AND DOCUMENTATION

- 4.1 Report all data and information pertinent to the resistivity meter verification and testing using SiteManager form [Tx129.xlsm](#).

This form is available from the Materials & Tests Division/Soils & Aggregates Section and online at the following link <https://www.txdot.gov/inside-txdot/forms-publications/consultants-contractors/forms/site-manager.html>.

5. MATERIAL SAMPLING AND PREPARATION

- 5.1 Obtain a representative sample in accordance with [Tex-100-E](#) when testing soil.
- 5.2 Obtain a representative sample in accordance with [Tex-400-A](#) when testing select backfill or material containing coarse or fine aggregate.
- 5.3 Dry the sample to constant mass in an oven at $140 \pm 9^\circ\text{F}$ and allow it to cool at room temperature.
Note 1—Constant mass is achieved when the loss is less than 0.1% of sample mass in 4 hr. of drying.

6. VERIFICATION OF RESISTIVITY METER

- 6.1 Verify the accuracy of the resistivity meter before each series of tests.

- 6.2 Connect the terminal leads to the resistivity meter and the opposite terminal leads to a 100-ohm resistor and read the meter. Repeat this process with a 200, 500, and 900-ohm resistor.
- 6.3 Report the results in the SiteManager template from Section 4.
- 6.4 When the readings are within 5% of the resistance of the resistors, the meter is functioning satisfactory and proceed to Section 7.
- 6.5 When the readings are greater than 5% of the resistance of any of the resistors, repeat the verification procedure.
- 6.5.1 If the readings are within 5% of the resistance of the resistors proceed to Section 7.
- 6.5.2 If the readings are greater than 5% of the resistance of the resistor, calibration of the meter is required.
-

7. PROCEDURE

- 7.1 *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.*
- 7.2 Quarter the sample using a quartering machine or cloth.
- 7.3 Sieve the quartered sample through the No. 8 sieve to obtain 1,300 g or more.
- 7.3.1 When the sample produces less than 1,300 g, crush the coarser aggregate in the sample to obtain the amount needed.
- 7.4 Weigh a sample to $1,300 \pm 0.5$ g and place into a mixing pan.
- 7.5 Add 100 mL of distilled or deionized water to the dry material in the mixing pan at room temperature.
- 7.6 Mix the sample thoroughly until the water is uniformly dispersed.
- 7.7 Fill the soil box by lightly hand-compacting the wet sample to completely fill the box.
- 7.8 Level the top with a straightedge.
- 7.9 Connect the wires to the resistivity meter and the soil box as per manufacturer's recommendations.
Note 2—The dial reads resistance in ohms measured between electrodes separated by the soil.
- 7.10 Operate the resistivity meter as per manufacturer's recommendations to measure the resistance of the sample. Record the reading.
- 7.11 Remove the sample from the soil box into the mixing pan from Section 7.5.
- 7.12 Add distilled or de-mineralized water in increments of 50 mL for sandy soils and 100 mL for clay soils to the sample in the mixing pan and repeat Sections 7.6 to 7.8.
- 7.13 Repeat Sections 7.10 to 7.12 until the resistance reading starts to increase.
Note 3—The resistivity meter readings should decrease for several readings before an increase is noted.

Note 4—The resistivity for sandy soils is generally higher than clay soils. When sandy soils contain higher levels of soluble salts, the readings may not increase after decreasing to the lowest point.

8. CALCULATIONS

8.1 Calculate the Soil Box Factor (SBF):

$$SBF = A / D$$

Where:

A = Area of one side of the electrode (Figure 1 or 2), cm^2

D = Distance between electrodes, cm .

8.2 Calculate Resistivity:

$$R = SBF * R_{ohm}$$

Where:

R = Resistivity, $Ohm\text{-}cm$

SBF = Soil Box Factor

R_{ohm} = Resistance value from meter, Ohm



Tex-129-E, Measuring the Resistivity of Soil Materials

Water Added (mL)	Multiplier	Dial Reading	Resistance (ohms)

Minimum Resistance Reading: _____

Resistivity (Ohm – cm) = Box Factor x Minimum Resistance: _____

Test Procedure for

DETERMINING SULFATE CONTENT IN SOILS — COLORIMETRIC METHOD



TxDOT Designation: Tex-145-E

Effective Date: February 2005

1. SCOPE

- 1.1 This method determines the soluble sulfate content of soil by using turbidimetric techniques.
 - 1.2 The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.
-

2. DEFINITIONS

- 2.1 *Turbidimetric Technique*—The process of measuring the cloudiness of a liquid and translating it into concentration. A reagent (sulfate test tablet) is combined with a sample, which causes cloudiness or turbidity in the sample.
 - 2.2 *PPM*—Parts per million; measure of concentration.
 - 2.3 *Filtrate*—(Soil/water) material that has passed through a filter.
-

3. APPARATUS

- 3.1 *Colorimeter and the following accessories:*
 - 10 mL glass sample vial
 - Sulfate test tablets
 - White plastic rod.
 - 3.2 *9 V battery.*
 - 3.3 *Auger sampler.*
 - 3.4 *Core sampler, 2 in. diameter × 4 ft. long.*
 - 3.5 *Balance, Class G2 in accordance with Tex-901-K, minimum capacity of 3500 g.*
 - 3.6 *Sieves, U.S. Standard No. 4 (4.75 mm) and No. 40 (425 μm).*
-

- 3.7 *Mortar and pestle.*
- 3.8 *Crusher.*
- 3.9 *Oven, capable of maintaining a temperature of $60 \pm 5^{\circ}\text{C}$ ($140 \pm 9^{\circ}\text{F}$).*
- 3.10 *Beakers (glass or plastic), 250 mL.*
- 3.11 *Beakers (glass or plastic), 600 mL.*
- 3.12 *Graduated cylinder TD (ex) (glass or plastic), 10 mL.*
- 3.13 *Graduated cylinder (glass or plastic), 100 mL.*
- 3.14 *Wide-mouth round high-density poly ethylene (HDPE) bottles, 8 oz. (250 mL).*
- 3.15 *Wide-mouth round HDPE bottles, 16 oz. (500 mL).*
- 3.16 *Funnel (glass or plastic), 80 mm diameter with short wide stems.*
- 3.17 *Filter paper (fine porosity), 15 cm diameter.*
- 3.18 *2 mL disposable pipettes.*
- 3.19 *Wash bottle, 16 oz. (500 mL), for distilled or deionized water.*
- 3.20 *Dessicator.*
- 3.21 *Latex gloves.*
- 3.22 *Tweezers.*
- 3.23 *Pans, brush, and spatula.*
- 3.24 *Sample splitter.*

4. MATERIAL

- 4.1 *Distilled or deionized water.*

PART I—FIELD DETERMINATION OF SULFATES

5. SCOPE

- 5.1 The following procedure describes preparing soil samples for determining sulfate content in the field.

6. PROCEDURES

6.1 *Preparing Sample:*

6.1.1 Sample soil within the depth of proposed stabilization with a hand auger at the sampling frequency required by the guide schedule.

6.1.2 Obtain a 300 g representative sample.

6.1.3 Air-dry the sample to constant weight.

6.1.4 Pulverize the 300 g to pass the No. 40 (425 μ m) sieve.

6.1.5 Split the sample and obtain 10 g of representative sample. Do replicates for more accuracy. This means split the material to obtain three samples of 20 g each and run on each sample the colorimetric test.

6.1.6 Weigh 10 g to the nearest 0.1 g.

6.2 *Measuring Sulfate Content:*

6.2.1 Place the 10 g soil sample in an 8 oz. (250 mL) HDPE bottle.

6.2.2 Add 200 mL of distilled or deionized water. This provides an initial dilution ratio of 1:20.

6.2.3 Vigorously shake the bottle of soil/water solution by hand for 1 min. Wait for 12 hr.

6.2.4 After a minimum of 12 hr. has elapsed, place one filter paper along the inside of the funnel and place the funnel into an 8 oz. (250 mL) beaker to capture the filtrate.

6.2.5 Shake the bottle of soil/water solution again for 1 min. and pour the solution into the funnel. Allow all the solution to filter and drain into the beaker.

6.2.6 Using a clean, 2 mL disposable pipette, fill the 10 mL graduated cylinder with the filtrate. Place the 10 mL filtrate into a sample vial. Wipe the outside of the vial clean with a Kimwipe® or equivalent delicate task wipe to remove dirt, fingerprints, or anything else that will obstruct a light beam from passing through the glass and filtrate.

Note 1—Insure the test vial is clean and free from any residue from previous testing.

6.2.7 Switch the colorimeter unit to “ON.”

6.2.8 Press the *MODE* key until “SUL” method is displayed.

6.2.9 Place the sample vial into the sample chamber with the Δ vial mark aligned with the Δ housing mark.

6.2.10 Press the *ZERO/TEST* key. The method symbol flashes for approximately 3 sec. and confirms zero calibration.

- 6.2.11 After zero calibration, remove the vial from the sample chamber.
- 6.2.12 Add one sulfate test tablet to the vial without touching the tablet with hands and crush immediately with the white plastic rod provided.
CAUTION: Use latex gloves and tweezers when handling the sulfate test tablet. The tablets are toxic and care should be taken to avoid contact with the skin, eye, or ingestion.
- 6.2.13 Tamp the sulfate test tablet with the rod in a random motion until it is completely dissolved and dispersed throughout the sample vial.
- 6.2.14 Cap the vial, wipe the outside of the glass clean again, and immediately place the sample in the chamber with the Δ marks aligned.
- 6.2.15 Press the *ZERO/TEST* key. The method symbol flashes for approximately 3 seconds and the result will appear in the display.
- 6.2.16 Take a minimum of three readings per sample and average the results.
Note 2—If an error message is displayed, go to Section 6.2.19.
- 6.2.17 Multiply the average result times the dilution.

(Example: 100 (average reading) \times 20 (1:20 dilution) = 2000 ppm.)

Note 3—This test will only read concentrations from 5-200 mg/l (100-4000 ppm) for a 1:20 dilution.
- 6.2.18 Report the concentration in parts per million (ppm).
- 6.2.19 If the following error messages are displayed, report the following:
- 6.2.19.1 “– *Err message*: The result is below the measuring range limit.”
■ Report the sulfate concentration as <100 ppm.
- 6.2.19.2 “÷ *Err message*: The result exceeds the measuring range.”
■ This requires diluting the sample with more deionized water to bring the reading within the measurable range, as stated below:
- Measure out 5 mL of the filtrate into a 10 mL graduated cylinder and add exactly 5 mL of distilled or deionized water using a clean pipette.
 - The new solution is now at 1:40 dilution ratio, which corresponds to a maximum reading range of 8,000 ppm. Place the new diluted solution in a sample vial and repeat Sections 6.2.8–6.2.18.
 - If a “÷ *Err message*” is displayed again, this means the material has more than 8,000 ppm sulfate content. Report the sulfate concentration as > 8,000ppm.

7. TEST REPORT

- 7.1 Report average sulfate concentration in ppm to the nearest whole number.

PART II—LABORATORY TEST

8. SCOPE

- 8.1 The following procedure describes preparing soil samples for determining sulfate content in the laboratory.

9. PROCEDURES

9.1 *Preparing Sample:*

- 9.1.1 Dry the entire Shelby tube or bagged sample in a $60 \pm 5^\circ\text{C}$ ($140 \pm 9^\circ\text{F}$) oven and allow to cool to $25 \pm 3^\circ\text{C}$ ($77 \pm 5^\circ\text{F}$) in a desiccator to constant weight.
- 9.1.2 After the sample is dried, crush, grind, and split to obtain approximately a 1500 g (3.3 lb.) representative sample to pass the 4.75 mm (No. 4) sieve.
- 9.1.3 Pulverize the 1500g sample to pass the 425 μm (No. 40) sieve.
- 9.1.4 Split the sample to obtain three 40 g representative sample and weigh to the nearest 0.1 g.

9.2 *Measuring Sulfate Content:*

- 9.2.1 Weigh 20 g to the nearest 0.1 g of oven-dried soil and place the soil in a 16 oz. (500 mL) HDPE bottle.
- 9.2.2 Add 400 mL of distilled or deionized water. This provides an initial dilution ratio of 1:20.
- 9.2.3 Vigorously shake the bottle of soil/water solution by hand for 1 min. Wait for 12 hr.
- 9.2.4 After a minimum of 12 hr. has elapsed, place one filter paper along the inside of the funnel and place the funnel into a 600 mL beaker to capture the filtrate.
- 9.2.5 Agitate the sample again for 1 min. and pour the soil/water solution into the funnel. Allow all the solution to filter and drain into the beaker. Ensure the funnel is covered during the filtration process to ensure soil and other substance does not contaminate the solution.
- 9.2.6 Using a clean, uncontaminated pipette, measure out 10 mL of the filtrate into a 10 mL graduated cylinder. Transfer the 10 mL filtrate from cylinder into a sample vial. Wipe the outside of the vial clean with a Kimwipe® or task wipe to remove dirt, fingerprints, or anything else that will obstruct a light beam from passing through the glass and filtrate.

Note 4—Insure the test vial is clean and free from any residue from previous testing.

- 9.2.7 Switch the colorimeter unit to “ON.”
- 9.2.8 Press the *MODE* key until “SUL” method is displayed.
- 9.2.9 Place the sample vial into the sample chamber with the Δ vial mark aligned with the Δ housing mark.
- 9.2.10 Press the *ZERO/TEST* key. The method symbol flashes for approximately 3 sec. and confirms zero calibration.
- 9.2.11 After zero calibration, remove the vial from the sample chamber.
- 9.2.12 Add one sulfate test tablet to the vial without touching the tablet with hands and crush immediately with the white plastic rod provided.
- CAUTION:* Use latex gloves and tweezers when handling the sulfate test tablet. The tablets are toxic and care should be taken to avoid contact with the skin, eye, or ingestion.
- 9.2.13 Tamp the sulfate test tablet with the rod in a random motion until it is completely dissolved and dispersed throughout the sample vial.
- 9.2.14 Cap the vial, wipe the outside of the glass clean again, and immediately place the sample in the chamber with the Δ marks aligned.
- 9.2.15 Press the *ZERO/TEST* key. The method symbol flashes for approximately 3 sec. and the result will appear in the display. Take a minimum of three readings per sample and average the results.
- 9.2.16 If an error message is displayed, go to Section 9.2.19.
- 9.2.17 Multiply the average result times the dilution.
- (Example: 100 (average reading) \times 20 (1:20 dilution) = 2000 ppm.)
- Note 5**—This test will only read concentrations from 5-200 mg/l (100-4000 ppm)
- 9.2.18 Report the concentration in parts per million (ppm).
- 9.2.19 If the following error messages are displayed, take the corresponding actions:
- 9.2.19.1 “- *Err message:* The result is below the measuring range limit.”
- Report the sulfate concentration as < 100 ppm.
- 9.2.19.2 “÷ *Err message:* The result exceeds the measuring range.”
- Dilute the sample with more deionized water to bring the reading within the measuring range, as stated below:
 - Measure out 5 mL of the filtrate into a 10 mL graduated cylinder and add exactly 5 mL of distilled or deionized water using a clean pipette.

- The new solution is now at 1:40 dilution ratio, which corresponds to a maximum reading range of 8,000 ppm. Place the new diluted solution in a sample vial and repeat Sections 9.2.8–9.2.18.
- If “÷ Err message” is displayed again, this means the material has more than 8,000 ppm sulfate content and additional dilutions will be required until an absolute number is obtained. Repeat Sections 9.2.8 through 9.2.15 and Sections 9.2.17 and 9.2.18.

10. TEST REPORT

10.1 Report average sulfate concentration in ppm to the nearest whole number.



Tex-145-E

Determining Sulfate Content in Soils-Colorimetric Method

Dilution Ratio	Meter Reading			Average/ERR	PPM Sulfate Content
	A	B	C		

Dilution Chart			
Filtrate, ml	Plus	Distilled Water	Multiplier
10.00	+	0.00	20
5.00	+	5.00	40
2.50	+	7.50	80
2.00	+	8.00	100
1.25	+	8.75	160
1.00	+	9.00	200

Calculation

$$D_f = D_i \frac{(V_f + V_w)}{V_f}$$

Where:

- Df = Dilution ratio of diluted filtrate
- Di = Initial dilution ratio
- Vf = Volume of undiluted filtrate, ml
- Vw = Volume of water added, ml

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:

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

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

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

$$\overline{100.1}$$

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

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

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

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

$$\overline{94.4}$$

Analysis shown as :

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

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

$$\text{CaO} = 0.3$$

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

- Example where water is present:

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

Analysis is shown as:

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

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

Table 3—Uncorrected Calcium Hydroxide Value

mL of Acid and Hydrated Alkalinity	% Ca(OH)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 CaCO₃ —Whole milliliter difference between two endpoints:

Table 4—Calcium Carbonate Value

Carbonate Alkalinity	CaCO ₃
0.0	0.0
1.0	1.8
2.0	3.6
3.0	5.4
4.0	7.1
Factor = 1.78479	

11.2.9 Correction for Calcium Hydroxide Value when CaO is Present:

Table 5—Correction for Calcium

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

11.2.10 Amount of Subtotal Over 100%:

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

Table 6—Proportional Parts

ml acid	% Ca(OH) ₂
0.1	0.1
0.2	0.3
0.3	0.4
0.4	0.6
0.5	0.7
0.6	0.8
0.7	1.0
0.8	1.1
0.9	1.3
Factor = 1.32126	

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

Table 7—Proportional Parts

Fractional Carbonate Alkalinity	CaCO ₃
0.1	0.2
0.2	0.4
0.3	0.5
0.4	0.7
0.5	0.9
0.6	1.1
0.7	1.3
0.8	1.4
0.9	1.6
Factor = 1.78479	

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

12. OPTIONAL INDICATOR METHOD FOR THE TITRIMETRIC ANALYSIS

12.1 *Scope:*

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

12.2 *Titration to Phenolphthalein Endpoint:*

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

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

PART III—TESTING COMMERCIAL LIME SLURRY

13. SCOPE

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

14. APPARATUS

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

15. REAGENTS AND SOLUTIONS

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

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

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

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

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

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

16.2 *Determining Bulk Density:*

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

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

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

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

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

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

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

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

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

16.2.8 *Calculations:*

16.2.8.1 Calculate bulk density:

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

Where:

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

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

16.3 *Determination of Sample Size:*

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

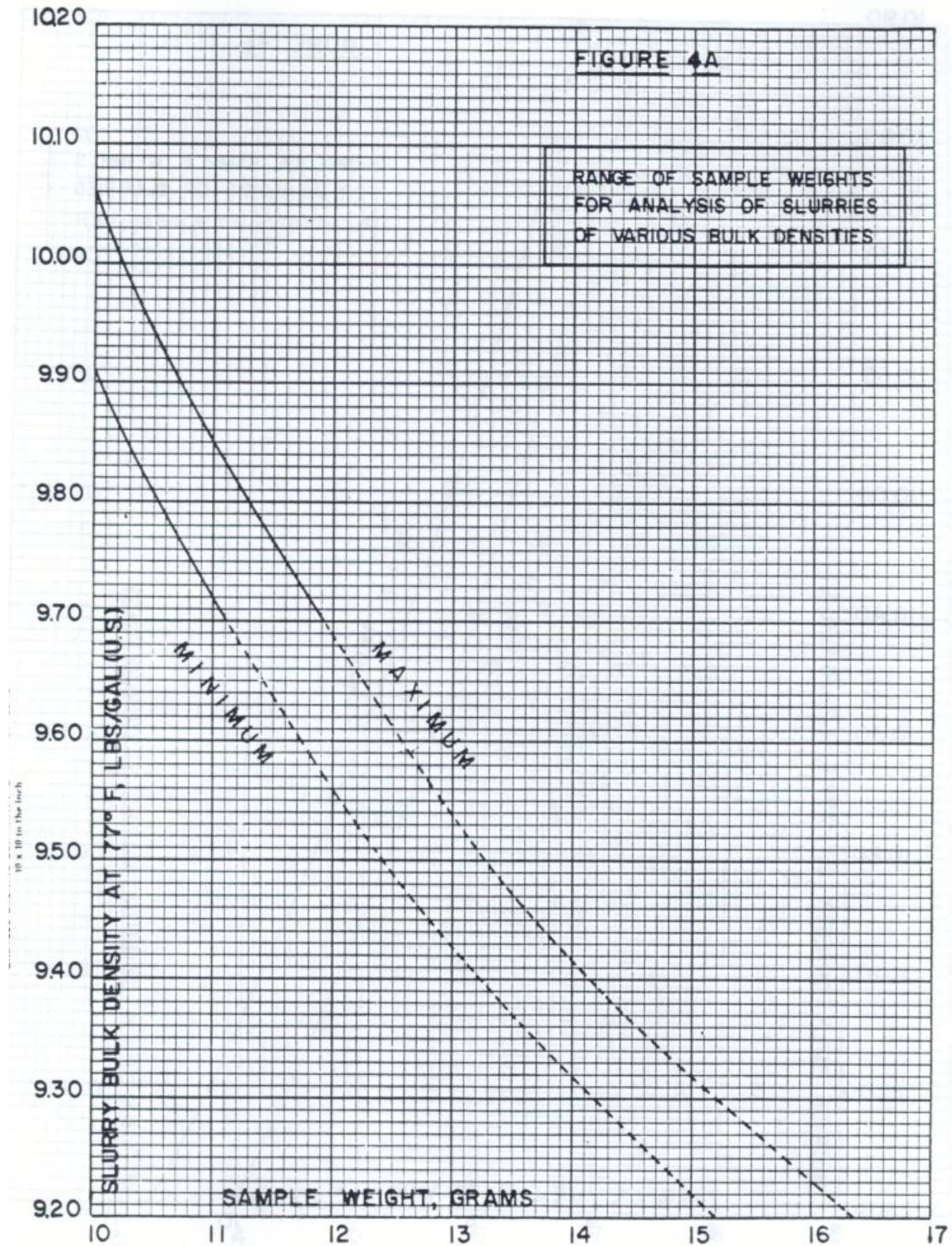


Figure 1—Range of Sample Weights for Analysis of Slurries—No. 1

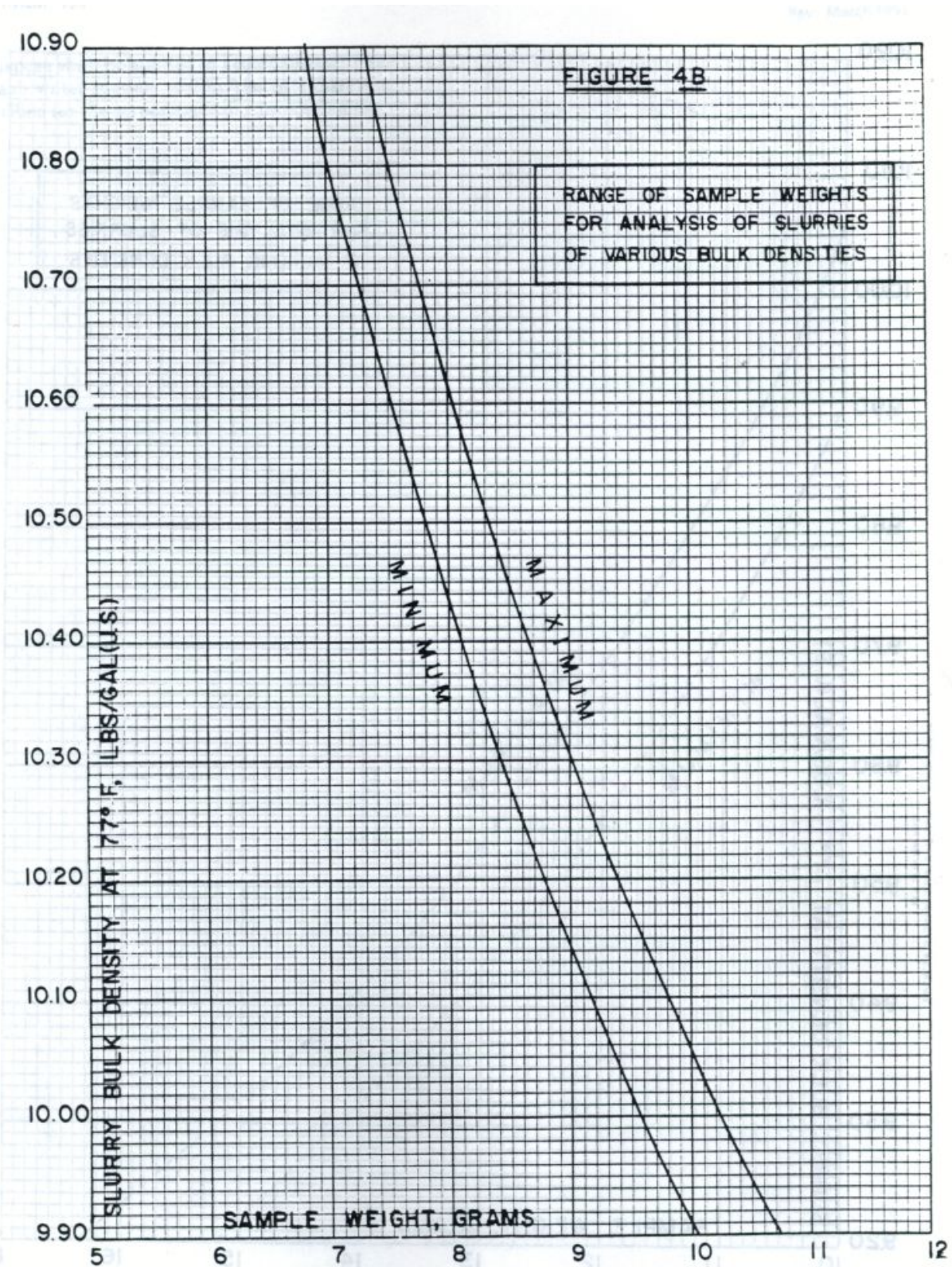


Figure 2—Range of Sample Weights for Analysis of Slurries—No. 2

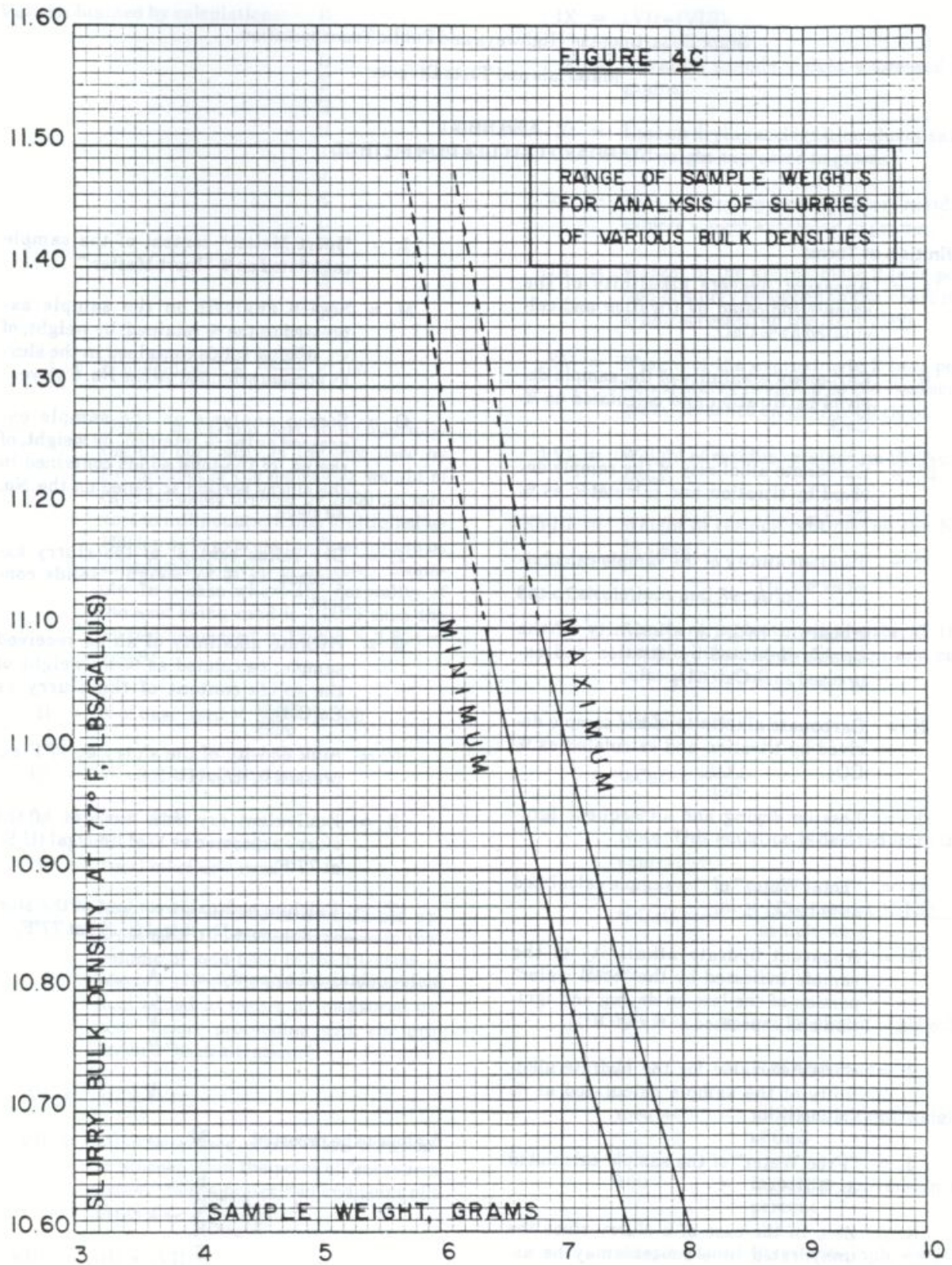


Figure 3—Range of Sample Weights for Analysis of Slurries—No. 3

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

17.2 *Back-Titration to pH 4.4:*

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

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

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

- 17.3 *Loss on Ignition:*
- 17.3.1 Weigh a crucible using an analytical balance, record the weight, and tare the balance.
- 17.3.2 Determine the required slurry sample size from Section 16.3. Weigh that amount into the tared crucible. Record the weight of the sample to the nearest 0.0001 g.
- 17.3.3 Place the crucible with slurry into a $212 \pm 4^\circ\text{F}$ ($100 \pm 2^\circ\text{C}$) 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. CALCULATIONS18.1 *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.