Silo 3 Waste Treatment Phase I
Physical Testing Final Report

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DISCLAIMER

1.0 EXECUTIVE SUMMARY

A characterization study of the Fernald Silo 3 waste was performed at the request of Rocky Mountain Remedial Services, LLC (RMRS) to support treatment of the waste with the Envirobond reagents and Envirobrick process. The Savannah River Technology Center (SRTC) performed the characterization under a Work for Others Agreement WOF-00-007. Physical property testing was subcontracted to the Clemson Environmental Technologies Laboratory (CETL).

This report is intended to transmit the results of the physical property testing conducted at the CETL. Results of the physical property testing are summarized in a final report from Steve Hoeffner, CETL. The report is included in Attachment 1.

Two thirty-gallon containers of Silo 3 waste were shipped from Fernald to the CETL where they were received on September 19, 2000. The shipment consisted of two 30-gallon drums of waste in 55-gallon over pack drums. The 55-gallon drum numbered W173805 contained a 30-gallon drum number W173689. The 55-gallon drum numbered W173822 contained another 30-gallon drum number W173696.

Two cores were taken from each 30-gallon drum. A 3-inch diameter stainless steel sleeve (casing) was first inserted into the drum. Material inside of the sleeve was collected using a 2-inch ID extendible core sampler with a butterfly valve on the auger tip. Material in the top one-half of the container was sampled first. The sampler was reinserted several times (3 to 6, as needed) until a total of 1000 or more grams of sample was collected. Then the bottom half of the sleeve was sampled in the same manner. See the Table 2 for the actual weights. Samples were split and distributed to the various analyses.

Visual description of the material in the as received condition is as follows:
The material is a fine red-brown powder that exhibited some dusting of the finest fraction when disturbed (transferred by pouring or scooping). The particles flowed in a cohesive manner when placed on an inclined surface. The material appeared very dry when the drums were opened. The moisture content of the material collected from the original containers ranged from 2.3 to 4.6 weight percent. The material absorbs moisture from the atmosphere when handled in the work area (radiological hood). This was first noticed by detecting a slight weight increase during the laboratory testing. The appearance of the material exposed to ambient laboratory conditions was the same as the as received material (dry).

Light tamping of the Silo 3 waste resulted in a 53 to 54 % increase in the bulk density relative to the loose material as it came out of the drum. The RMRS results for material sampled in 1997 gave an average increase in bulk density of 42 %. See Table 1.

The specific gravity of the particles was not measured in this study because the specified test method, ASTM D-854, required contact with water. Given the large amount of water-soluble material in the Silo 3 waste, 52 to 56 weight percent, this method was inappropriate. If this information is required in the future, a helium pycnometer (or other dried gas pycnometer) should be used to make the measurements.

Table 1. Bulk Densities and Specific Gravity of Silo 3 Waste.

Test	W173689 A	W173689 B	W173696 A	W173696 B
Specific Gravity	NA	NA	NA	NA
Bulk Density (loose)	0.688 (g/cm^3)	0.697 (g/cm^3)	0.675 (g/cm^3)	0.684 (g/cm^3)
Bulk Density (compacted)	1.061 (g/cm^3)	1.064 (g/cm^3)	1.039 (g/cm^3)	1.045 (g/cm^3)
Increase in Bulk density as the result of tamping	54 %	53 %	54 %	53 %

The engineering/physical properties of the bulk solids are listed in Table 2. These results indicate that in the as received condition, the Silo 3 waste is free flowing. Tests were performed at about 50 % relative humidity. Less than one hour was required to conduct each test so moist laboratory air had minimal effects on the results.

Table 2. Engineering Properties of the Silo 3 Waste.

Test	W173689 A	W173689 B	W173696 A	W173696 B
Angle of Repose (°)	45.71	47.1	46.07	45.68
Angle of Fall (Slump) (°)	25.43	24.22	25.43	26.34
Angle of Spatula/Withdrawal (°)	65.08	65.20	64.61	69.17
Cohesion (%)	74.55	63.65	83.15	66.98
Corrosivity (mm/yr)*	0.14	0.08	0.05	0.05

* A rate of greater than 6.35 mm/yr is considered corrosive per this test.

Since the Silo 3 waste is hygroscopic sensitivity to moisture in the air was evaluated. Weight gains of 12, 25, and 54 percent were measured for a sample exposed for 21 days to air with 51, 71, and 95% relative humidities, respectively. Clumping, color changes (darker reddish brown), and resistance to penetration increased as a function of exposure to moist air.

2.0 SUMMARY

The Fernald Silo 3 waste can be classified as a DOE salt waste stream. New information generated from this study includes documentation of the sensitivity of the Silo 3 waste to water and to moist air. More than 50 percent of the waste consists of water-soluble salts as indicated by weight loss upon leaching in water. In addition the waste is very hygroscopic as indicated by weight gains (greater than 50 percent in the most severe conditions tested). Physical properties of the dried leached material are similar to those of the material in the as received condition. Physical properties of the material exposed to moist air were different than those of the as received waste in that it was no longer free flowing and showed some resistance to penetration.

This work was intended to support the design of the Silo 3 waste retrieval, interim storage operations, waste form processing, and packaging, storage and shipping operations. The Silo 3 material is similar in some respects to other salt waste streams in the DOE complex that have been treated for disposal. Wastes containing large amounts of soluble salts have been successfully processed in the DOE complex but have also presented problems in treatment, storage and shipment throughout the DOE complex.

3.0 QUALITY ASSURANCE

This effort was carried out as a scoping study. Consequently no special M&TE requirements were formally applied. Analyses conducted at the CETL were performed in accordance the CETL Quality Assurance Procedures and are recorded in Laboratory Notebook, DE, Project 226-2002032.

ATTACHMENT 1

Fernald Silo 3 Waste Treatment Phase I

Physical Characterization

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January 9, 2001

Fernald Silo 3 Waste Treatment

Phase 1. Physical Characterization

Prepared for

Westinghouse Savannah River Company

Subcontract No. K47920, WEST 45

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DRUM SAMPLING

On September 19, 2000 a 55-gallon drum (W173805) arrived from Fernald via Federal Express. Radiation from the drums measured about 1 mR/hr on contact. Swipes of the external surface showed no activity above background.

On October 6, 2000 a 55-gallon drum (W173822) arrived from Fernald via Federal Express. Radiation from the drums measured about 1 mR/hr on contact. Swipes of the external surface showed no activity above background.

Each 55 gallon drum contained a 30 gallon drum: 55 gallon drum number W173805 contained 30 gallon drum number W173689 and 55 gallon drum number W173822 contained 30 gallon drum number W173696.

Core Samples
On October 10, 2000 the 30-gallon drum, number W 173689, was removed from drum number W173805. There were measurable levels of alpha activity on the outside of the 30-gallon drum and in the annular space between the two drums. The lid was taken off of the 30-gallon drum and the contents inspected. The material is a fine, dusty, brown powder and visually appears to be homogeneous.

Sampling of the 30-gallon drum, number W173689, occurred on October 12, 2000. There were no measurable levels of alpha activity on the outside of the 30-gallon drum and in the annular space between the two drums. Apparently the activity measured on October 10 was due to the accumulation of radon gas inside of the closed containers. Sampling of the 30-gallon drum, number W173696, occurred on October 12, 2000.

Two cores were taken from each 30-gallon drum. To take the samples a stainless steel sleeve 3 inches in diameter was first inserted into the drum. Core samples were then taken using a 2-inch ID AMS extendible core sampler with butterfly valve on the auger tip. When taking top core sample, the sampler was inserted half way down through the Silo 3 material. For samples from the bottom, the sampler was then inserted, inside the same stainless steel sleeve, completely to the bottom. In each case the sampler was reinserted several times (3 to 6, as needed) until a total of 1000 or more grams of sample were collected. See the table below for actual weights. The number of times the sampler was inserted and the cumulative amounts of sample collected each time are contained in the laboratory notebook. The two core samples were about 180 degrees apart and approximately two inches from the edge of the drum. The cores were divided into top and bottom, generating a total of 4 core samples for each drum. Cores were labeled as follows: last digits of the 30 gallon drum number-core number-T or B for top or bottom core sample, e.g., 689-1-T is the first core sample taken from the top half of drum W173689.

Table 1. Total Weight of Core Samples Collected From Silo 3 Drums

Core	Total Collected, grams
689-1-T	1260
689-1-B	1057
689-2-T	1285
689-2-B	1120
696-1-T	1231
696-1-B	654
696-1a-B	198
696-2-T	1157
696-2-B	1137
696-GRAB	1204

There were three discrepancies of note:

1) The lid on drum W173696 also had the identification W173693

2) We were not able to penetrate completely to the bottom when collecting core sample 696-1-B. Only 654 grams of silo 3 material was collected from this core. A second core was taken immediately next to the stainless steel sleeve (969-1a-B) resulting in an additional 198 grams.

3) Subsequent excavation of the silo 3 material revealed that another bag of material was on the bottom of the drum. The bag prevented the sampler from going to the bottom. A grab sample was taken from the material in this bag (696-GRAB).

Sub-sampling of Core Samples

On October 18, 2000, core samples were blended and split using a riffle splitter. From one split approximately 50 grams were placed in a 125 mL plastic bottle. The remainder from the split was placed in 1 liter bottle. Both samples were sent to General Engineering Laboratories (GEL) for analysis on October 18, 2000, for parameters specified by WSRC. The other split was bottled and archived. For sample 696-1-B the sample was split but then most of the sample was combined and sent to GEL, giving them a total of around 500 grams. Samples 696-1a-B was not combined with 696-1-B and was not sent to GEL. One split from the grab sample 696-GRAB was also sent to GEL.

Approximately 200 grams of sample were taken from the archive samples (all but 696-1a-B) and sent to Cecil May at WSRC on October 24, 2000 for additional testing.

Blending of Remaining Material

The silo 3 material that remained in the drums was transferred to a 55-gallon drum (1 drum for each of the original 30 gallon drums) and then blended by

placing on a drum tumbler for 24 hours. Drum W173696 was tumbled starting on October 26, 2000. Drum W173689 was tumbled starting on October 27, 2000. Samples were then collected for the physical testing: two 5-gallon buckets of silo 3 material were taken from each of the 55-gallon drums. These samples were labeled W173689A, W173689B, W173696A, and W173696B.

PHYSICAL TESTING
Physical testing began on October 31, 2000. Tests performed were as follows:
October 31 - Loose bulk density and compacted bulk density.
November 1 - Angle of repose, angle of fall, angle of spatula, pH
November 2 - cohesion, water leach, % moisture
November 3 - water leach
November 9 - solid and liquid samples from leachate study were shipped to General Engineering Laboratories.
November 16 - Corrosivity
December 13 – Relative Humidity test (in process)

A description of each of the tests follows. Results for each are shown in the following tables. A copy of each method is included in the Appendices.

Task 3.3.1
H2O (110 C), Percent Moisture
Was determined using ASTM method D2216-98, oven drying. (ASTM Method D 4643-00 was specified, but we did not have a microwave dryer). There were no other exceptions or changes to the method.

H2O (1000 C)
Test performed by WSRC

Task 3.3.2
Corrosivity
Was determined using EPA Method 1110. One liter of DI water was mixed with 1000 grams of Silo 3 material. The mixture was shaken by hand for 5 minutes, allowed to settle, and then filtered. The filtrate (500 to 650 mL of leachate, depending on the sample) was used for the corrosivity test. The method requested a minimum volume of 870 mL. Temperature ranged from 48 to 62 degrees C. There were no other exceptions or changes to the method.

pH
Was determined using EPA Method 9045C. There were no exceptions or changes to the method.

Water Leach
Method as specified by WSRC. 50 grams of Silo 3 material was diluted to 1000 mL with DI water. The mixture was stirred overnight (24 hours) at room temperature and then filtered. The solid residue was dried and weighed. The

liquids and dry solids were sent to General Engineering Laboratories for analysis of anions and metals.

Task 3.3.3
Note: All of the following tests that specified ASTM Method 6393-99 required a Carr Indices tester. This unit is expensive (around $25,000), uses fairly simple equipment, and would be difficult to decontaminate after use. WSRC requested that CETL follow the spirit of the test as closely as possible using common laboratory equipment and, in some instances, fabricating components for a given test. The differences between the Carr Indices tester and the equipment and approach we used are documented below for each test. In addition, pictures of the setups are also provided. A Fritsch sieve shaker (Analysette III) was used to vibrate the samples. The amplitude of the vibration is adjustable from 1 to 3 mm.

Loose Bulk Density
Was determined using ASTM Method D 6393-99. Test was performed as written. There were slight differences in the equipment (see Figure 1). ASTM-specified vibrating and stationary chute dimensions: stainless steel conical chute with the dimensions of 75.0 mm top diameter, 55.0-mm height, 50.0-mm bottom diameter. Actual chute dimensions were 72.9 mm top diameter, 76.2 mm height, and 53.8 mm bottom diameter. ASTM-specified cup dimensions: stainless steel cylindrical container, 50.5-mm inside diameter by 49.9-mm height (actual plastic cup was 50.8-mm inside diameter by 49.3-mm height). The volume of the cup was very similar, ASTM method: 100 cm3, actual 99.9 cm3. There were no other exceptions or changes to the method.

Compacted Bulk Density
Was determined using ASTM Method D 6393-99. Test was performed as written with the exception that instead of dropping the cup, it was tapped repeatedly on a ceramic ring stand base until no further decrease in volume was observed (see Figure 2). This process was repeated three times on the initial sample to confirm that this approach was reproducible. There were slight differences in the equipment as detailed in the loose bulk density method, above. In addition, the ASTM-specified cup extension was 55.0-mm in diameter by 48.0-mm in height (actual dimensions were 50.8 mm diameter by 51.6 mm in height).

Angle of Repose
Was determined using ASTM Method D 6393-99. Test was performed as written. There were slight differences in the equipment (see Figure 3). ASTM-specified glass funnel was 55-degree angle bowls as measured from the horizontal with a 7.0-mm outlet diameter and a stem length of 33.5 mm. (actual funnel was 58.1 degrees as measured from horizontal with a 14 mm outlet diameter and a stem length of 37 mm). ASTM-specified platform: chrome-plated brass circular platform with a diameter 80.0 mm and a height of 59.0-mm (actual diameter 78.2 mm and height of 41.4 mm). Height of the cone was measured from the bottom side of the spatula to the top of the cone using a T-square with a level bulb. To

confirm that the differences in cup dimensions did not significantly influence the
test results, the angle of repose was also determined using a larger aluminum
cup, dimensions of 87.9 mm diameter by 52.6 mm in height. The results were
comparable, as shown in the attached table.

Angle of Fall (Angle of Slump)
Was determined using ASTM Method D 6393-99. There were slight differences
in the equipment (see Figure 4, note the T-square with level and the shocker on
the ring stand). The shocker (weight) was formed from several washers glued
together to give a total weight of 109.5 grams (the method specified a weight of
110.0 grams). Height of the cone was measured from the bottom side of the
spatula to the top of the cone using a T-square with a level bulb.

Angle of Spatula (Angle of Withdrawal)
Was determined using ASTM Method D 6393-99. There were slight differences
in the equipment as detailed in the Angle of Fall method. In addition, The ASTM-
specified spatula was 22.0 mm in width by 3.0 mm thick (see Figure 5, note the
calipers and the shocker on the ring stand). The width of the actual spatula was
19.05 mm; the thickness of the spatula was 0.51 mm. Height of the cone was
measured from the bottom side of the spatula to the top of the cone using a
caliper. Thickness of the spatula was subtracted from the measurements.

Cohesion
Was determined using ASTM Method D 6393-99. Test was performed as written.
There were slight differences in the equipment (see Figure 6). The spacer and
vibrating chute were replaced with a bottom pan.

Relative Humidity Test
Was determined in accordance with WSRC instructions. A nominal 40 grams of
Silo 3 material was placed on a 3-inch sieve. The sieve was supported in a small
pan so that air and moisture could reach the sample from the top or bottom of the
sieve. The pan and elevated sieve were placed in a 1-gallon bucket. A total of
four samples were prepared. Each sample was placed in a bucket containing
saturated salt solutions that would produce different relative humidities: $CaCl_2$,
31%; $Ca(NO3)_2$, 51%; $NH4Cl$ + KNO_3, 71.2%, $Na2SO3$, 95%. The buckets were
sealed with a screw top lid. Weight gain was recorded after 1, 4, 7 and 21 days.
Visual observations and photographs were taken after 7 and 21 days. After 7
days a force of 1.85 pounds/in2 was applied to all but the low humidity sample
using a nominal 10-pound weight.

Observations after 7 days:
31 %H – Sample is dry and free flowing
51 %H – Sample feels just slightly "set up". Minor clumping, but free flowing
     overall
71.2 %H – Slightly more clumping. Slight resistance to penetration

95 %H – Clumps up readily. Some resistance to penetration. Sample is[A10]
noticeably darker than the other three.

Figure 7 shows all 4 samples. Note the darker color of the 95%H sample. Figures 8 (31%H and 51%H) and 9 (71.2%H and 95%H) provide a visual comparison of the cohesiveness of the samples.

Observations after 21 days:
31 %H without weight – Sample is dry and free flowing (Figure 10)
51 %H with weight – Clumpy. Like dirt clods (Figure 11).
71.2 %H – Silo 3 material stuck to the bottom of the puck/weight (Figure 12). Clumpy. Like dirt clods (Figure 13). Material is darker in color than the 31 or 51%H samples (both of which have about the dame color).
95 %H – Slight resistance to penetration. No clumping to bottom of puck (Figure 14). Consistency similar to dry paste. Sample is dark brown in color (Figure 15). Figure 16 shows all four samples. Note the increased darkness of samples 71 and 95%H.

Figure 17 shows the weight increase of the Silo 3 material over the 21 days for the four different humidities.

Outstanding Tests

Specific Gravity – To be determined using ASTM method D854-98. Use of fluid other than water is likely.

Compressive Strength after water saturation and drying – Not applicable

Abrasion Resistance – ASTM Method C418-98 was originally specified, but custom method more applicable to this project will likely be developed.

Particle Size Distribution – ASTM Method D422-63 (Reapproved 1998). Use of liquid other than water is likely.

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Table 2. Summary of Physical Test Results

Sample Test	W173689A	W173689B	W173696A	W173696B
H2O (110 C), percent	2.31	2.72	3.15	4.60
pH	7.80	7.82	7.95	7.86
Percent soluble				
Water Leach	47.38	46.13	43.88	44.97
Ethanol Leach	16.43			
Bulk Density (loose), g/cm3	0.688	0.697	0.675	0.684
Bulk Density (compacted), g/cm3	1.061	1.064	1.039	1.045
Angle of Repose, degrees	45.71	47.10	46.07	45.68
Angle of Fall (Slump), degrees	25.43	24.22	25.43	26.34
Angle of Spatula (Withdrawal), degrees	65.08	65.20	64.61	69.17
Cohesion, percent	74.55	63.65	83.15	66.98
Corrosivity, mmpy	<0.20	<0.20	<0.20	<0.20
Humidity Test, % Weight Gain After 21 Days				
At 31%H	2.32			
At 51%H	12.49			
At 71%H	24.59			
At 95%H	53.71			
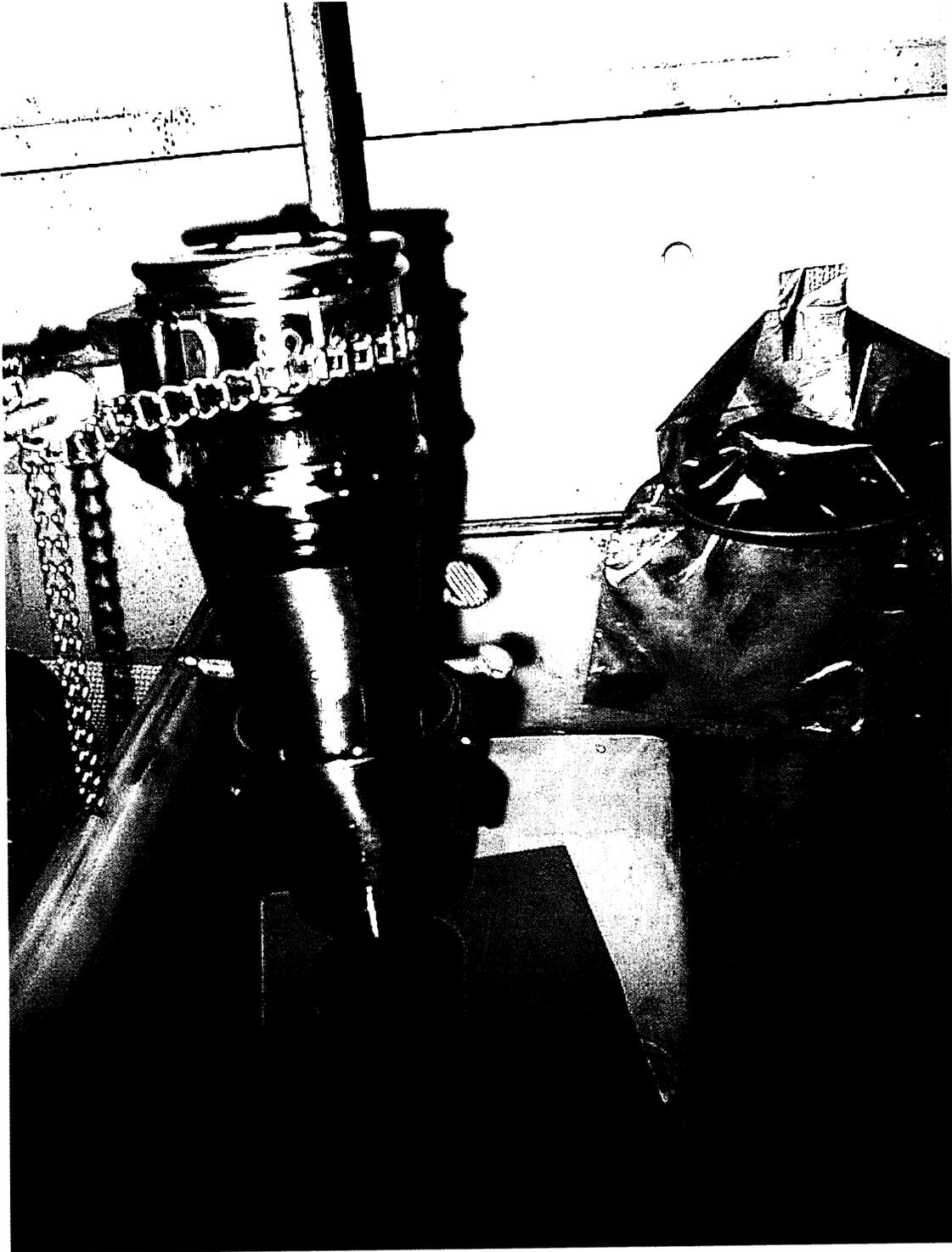
Figure 1. Test Setup for Loose Bulk Density.

Figure 2. Test Setup for Compacted Bulk Density.

Figure 3. Test Setup for Angle of Repose.

Figure 4. Test Setup for Angle of Fall.
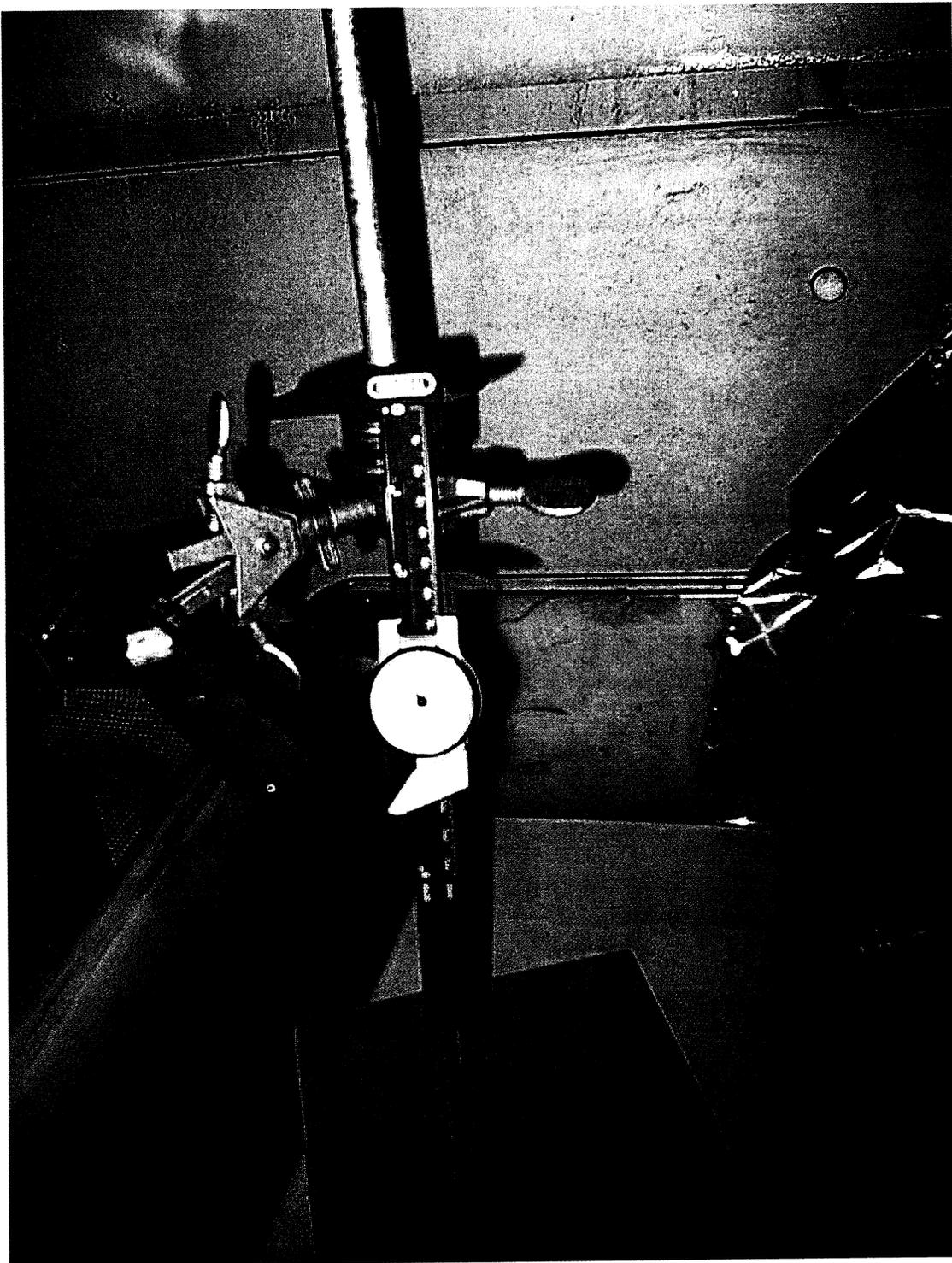
Figure 5. Test Setup for Angle of Spatula.
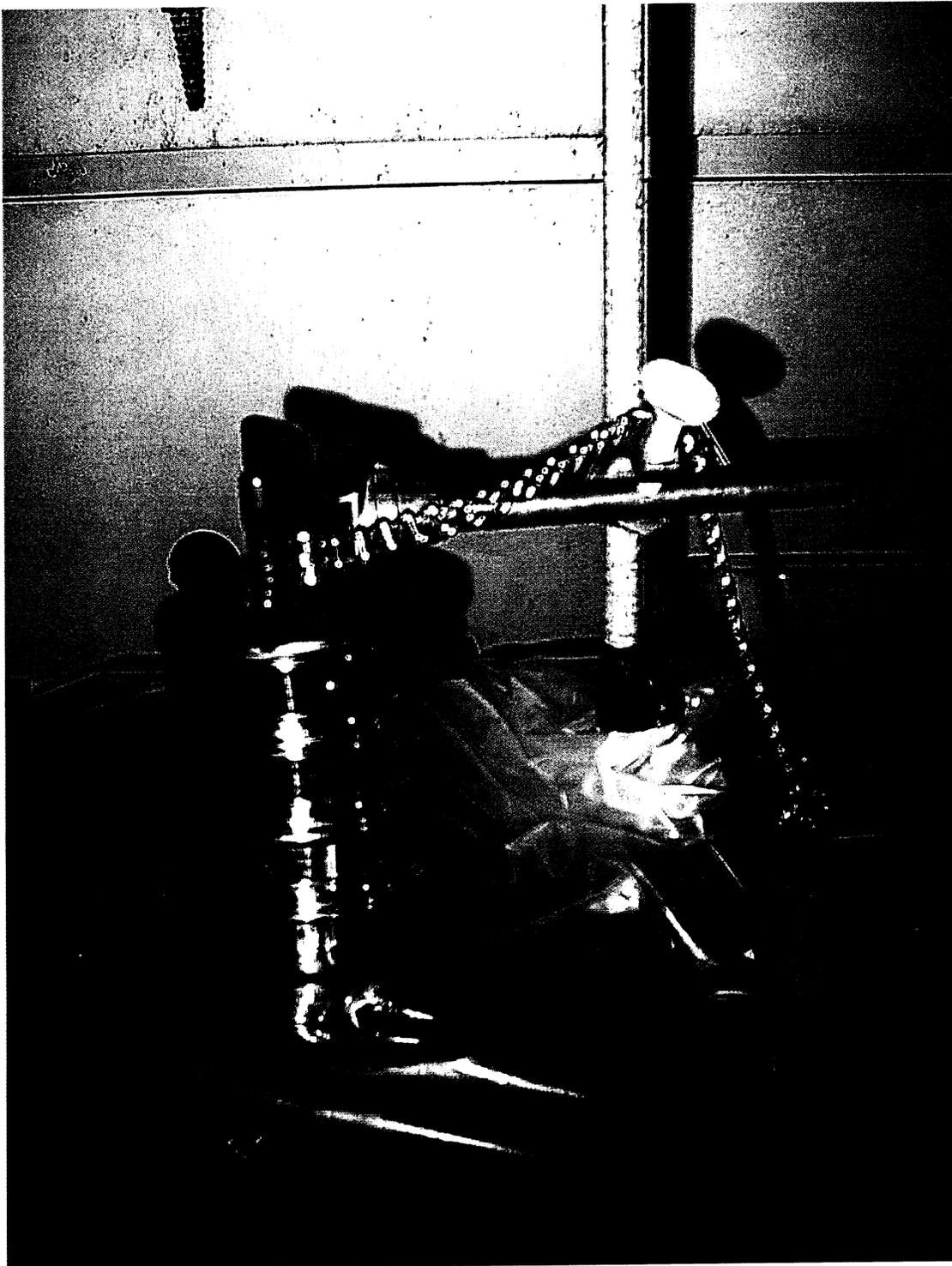
Figure 6. Test Setup for Cohesion.
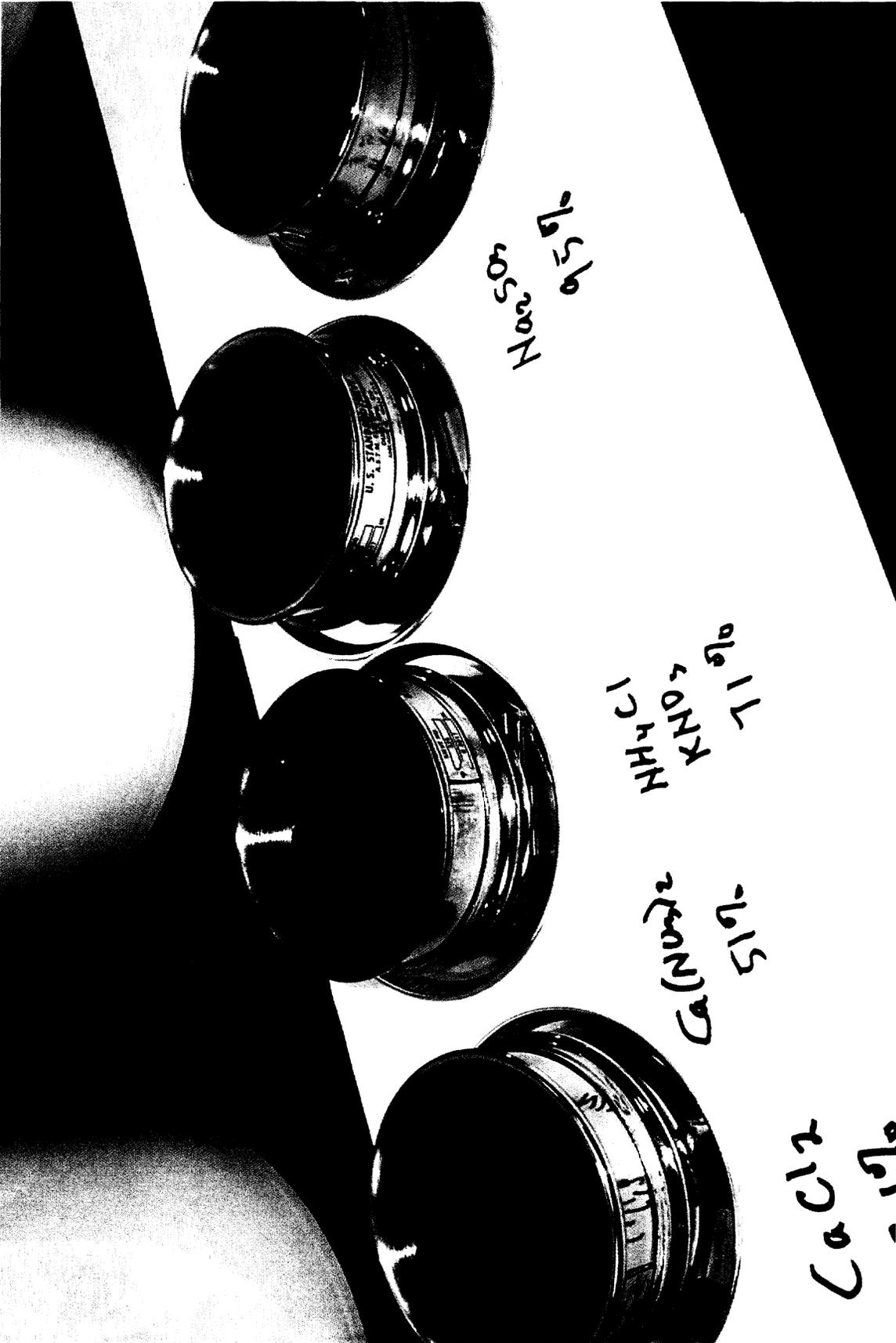
Figure 7. Appearance of Silo 3 Samples After 7 Days at Humidity.
(L to R, 31%H, 51%H, 71%H, 95%H)
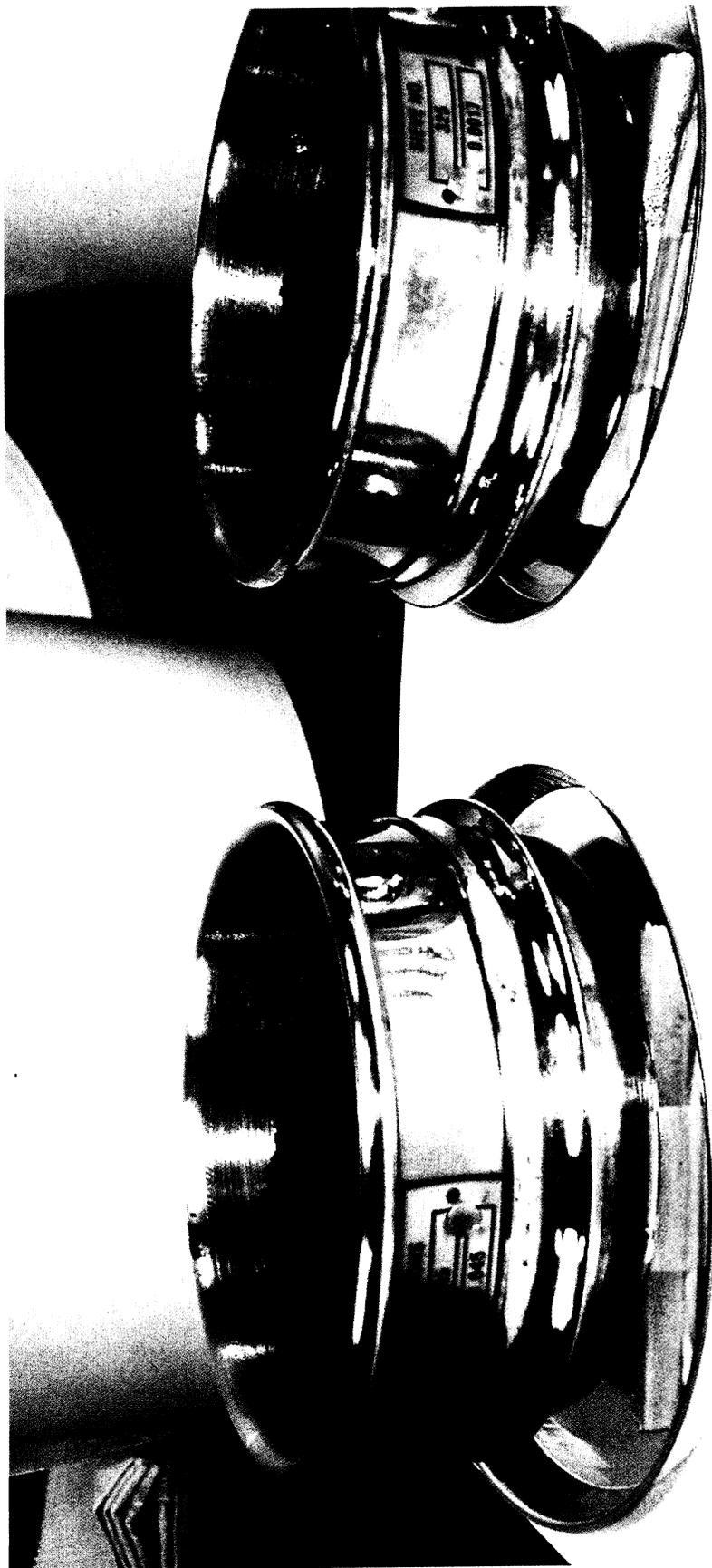
**Figure 8. Comparison of Sample Cohesiveness After 7 Days at Humidity.
(L = 31%H, R = 51%H)**
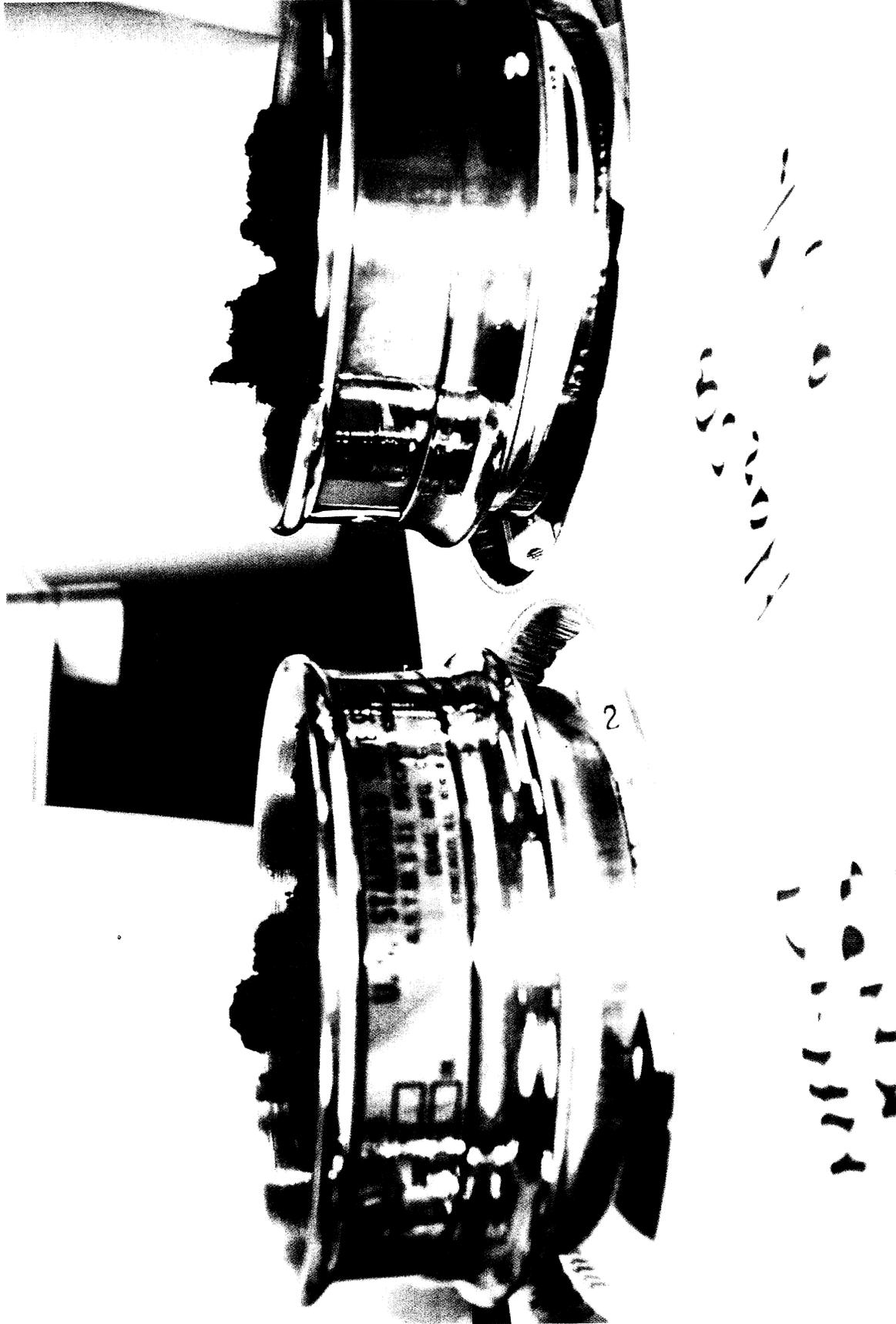
**Figure 9. Comparison of Sample Cohesiveness After 7 Days at Humidity.
(L = 71%H, R = 95%H)**

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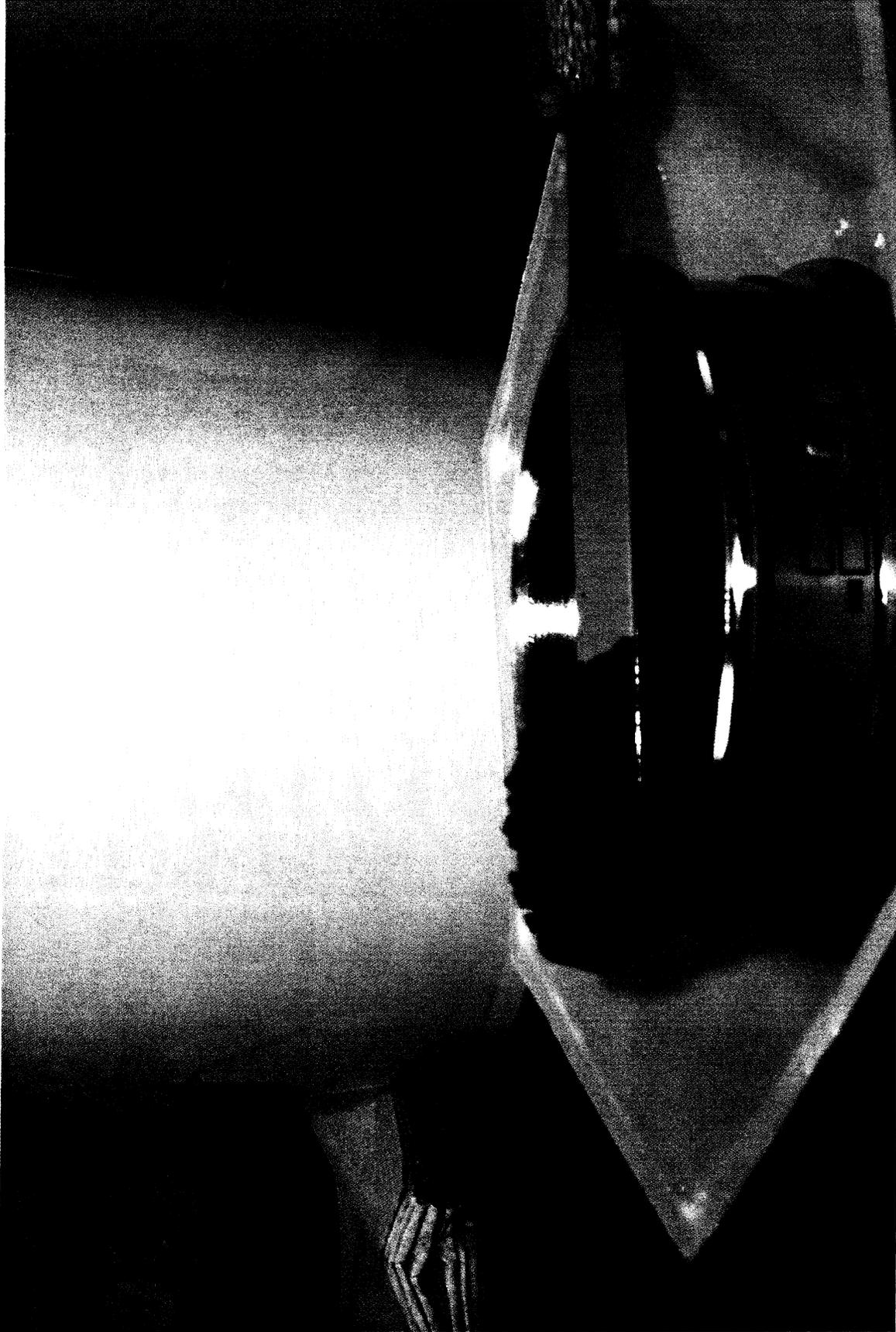
Figure 10. 31%H Sample After 21 Days.

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Figure 11. 51%H Sample After 21 Days.

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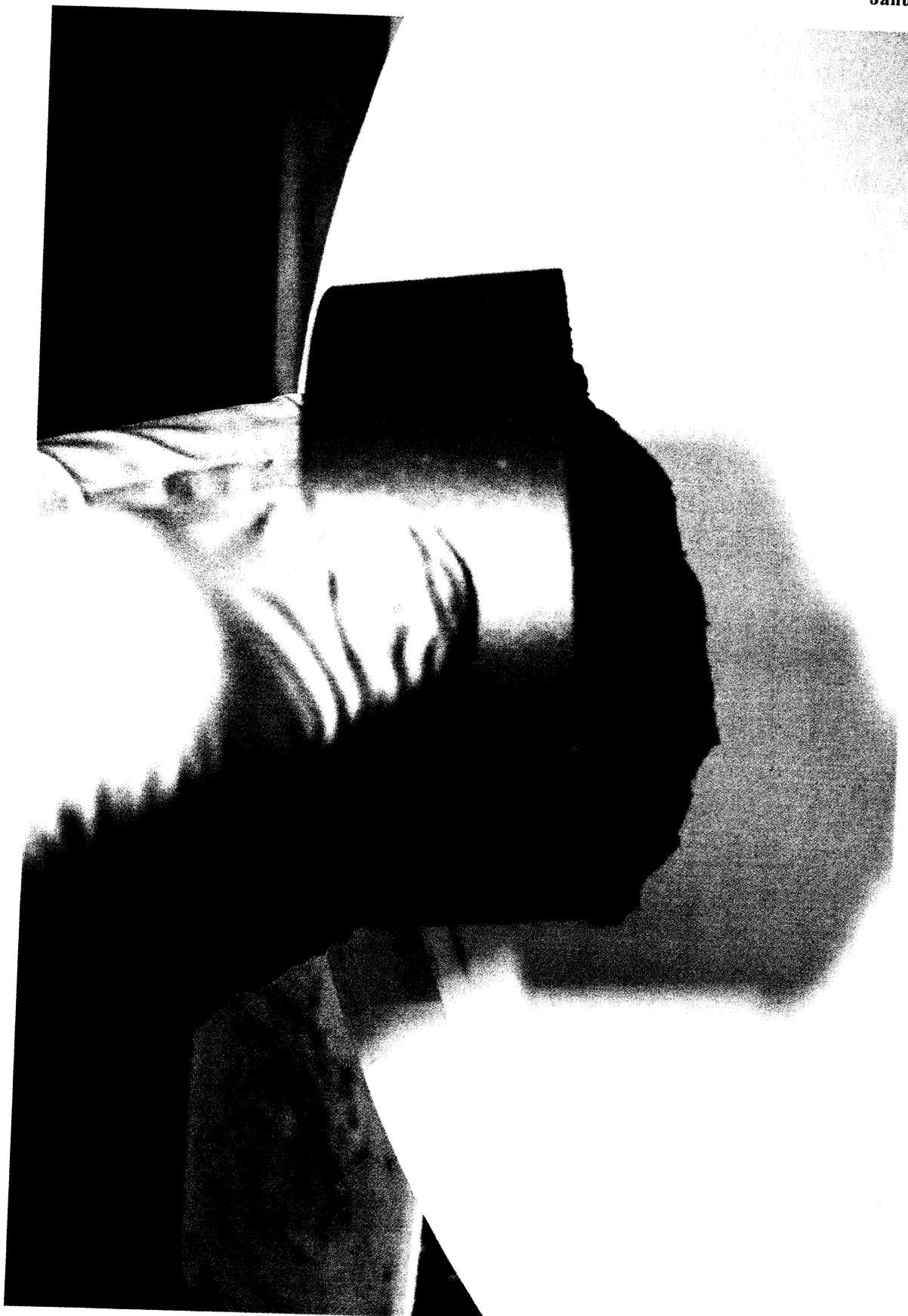
Figure 12. 71%H Sample Stuck to Puck After 21 Days.

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Figure 13. 71%H Sample After 21 Days.
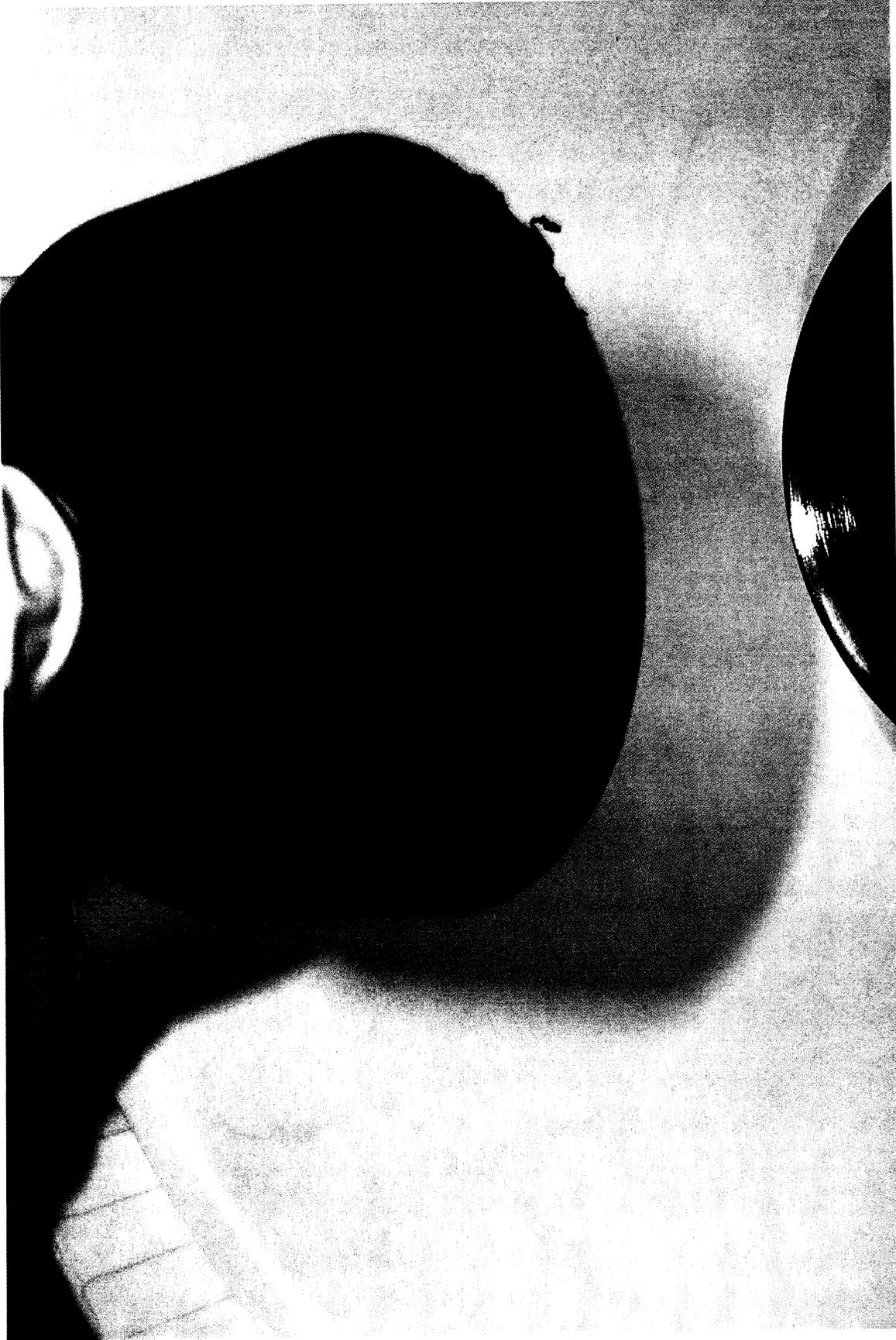
Figure 14. 95%H Sample After 21 Days – Only Minor Amount Stuck to Puck.

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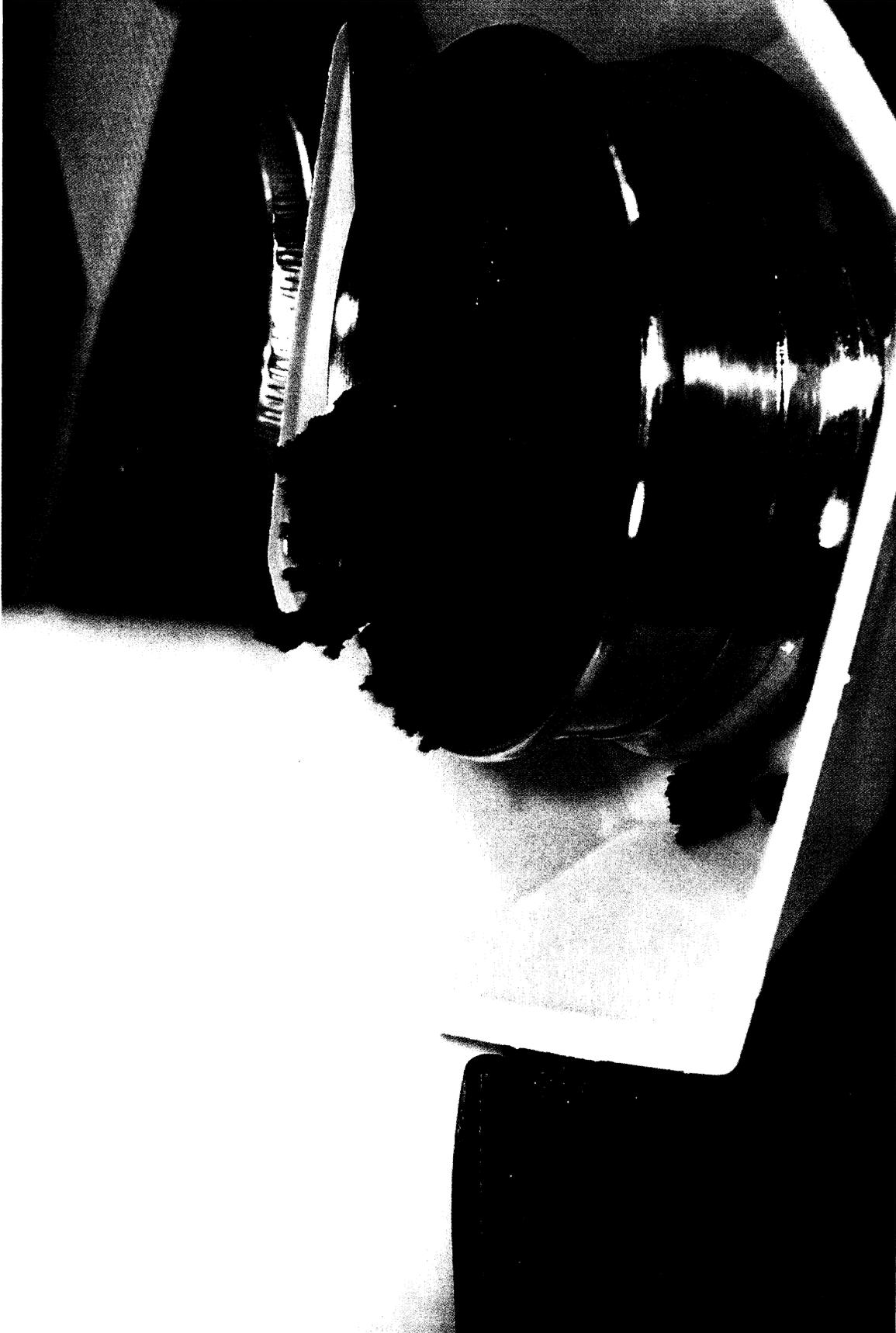
Figure 15. 95%H Sample After 21 Days.
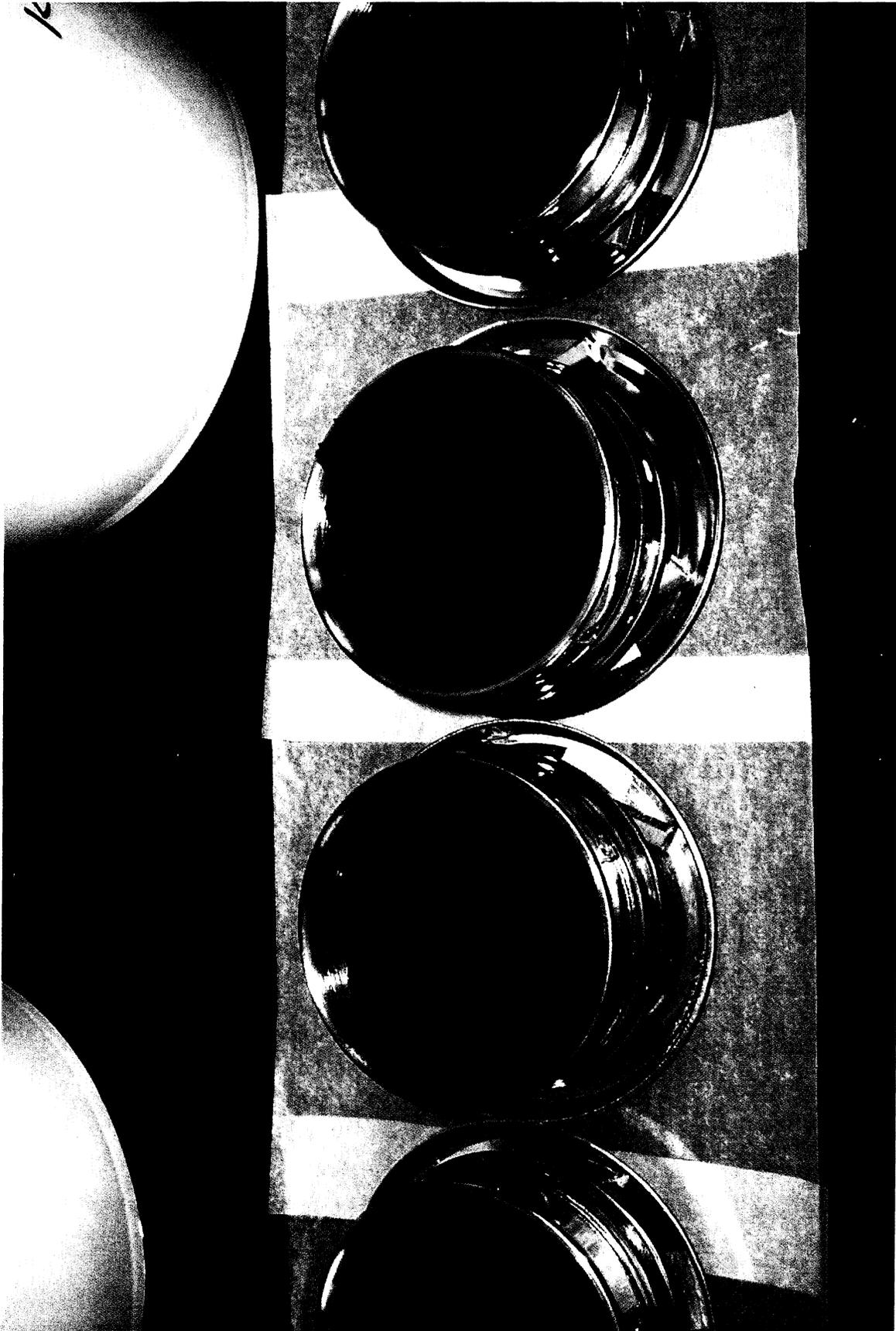
Figure 16. All Four Samples After 21 Days.
(L to R: 31%H; 51%H; 71%H, 95%H)

Figure 17. Percent Weight Gain of Silo 3 Material
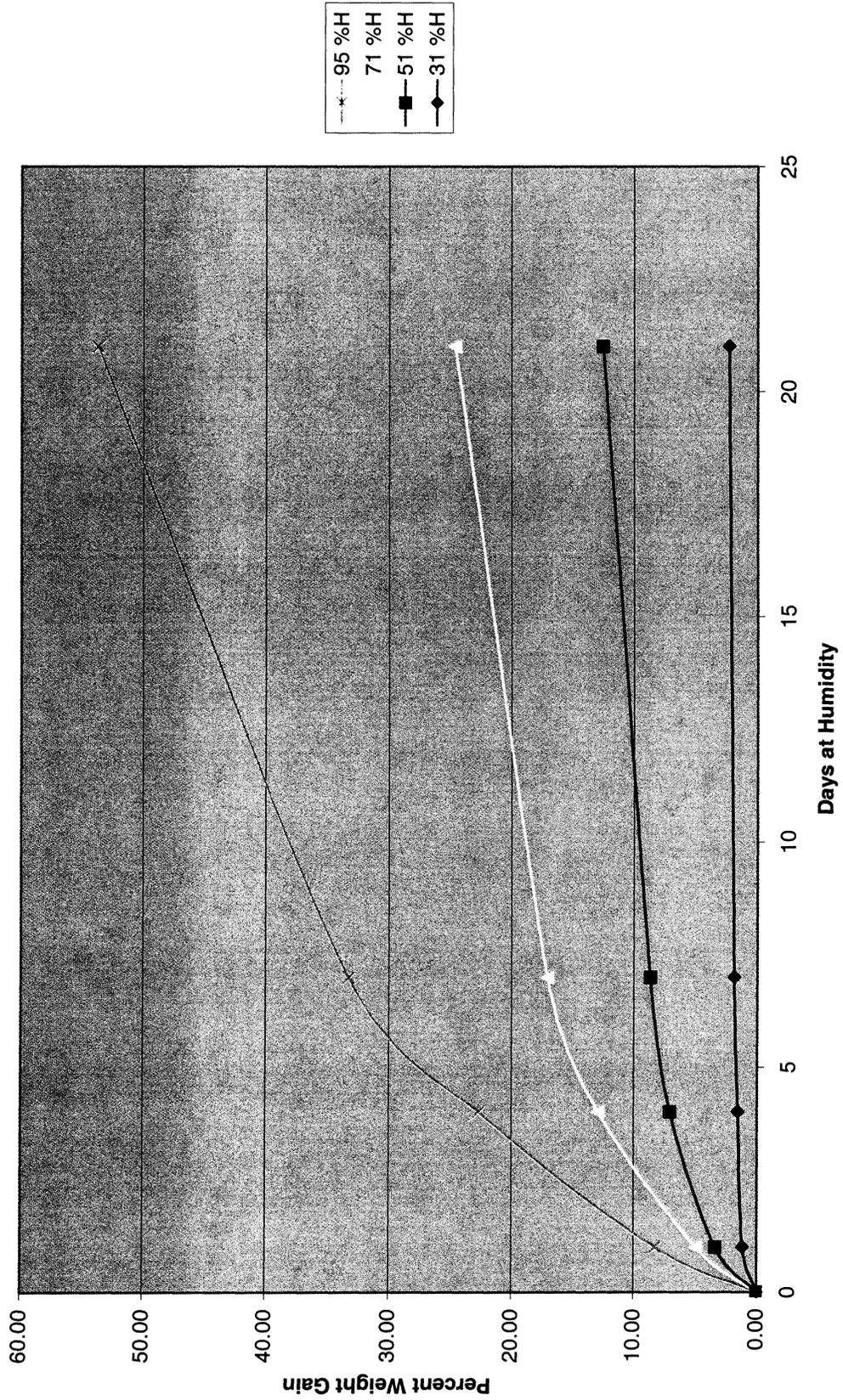
APPENDICES

Appendix A. Analytical Methods

Appendix A. Analytical Methods

ASTM method D2216-98
　　　Percent Moisture
EPA Method 1110
　　　Corrosivity
EPA Method 9045C
　　　pH
ASTM Method D 6393-99
　　　Loose Bulk Density
　　　Compacted Bulk Density
　　　Angle of Repose
　　　Angle of Fall (Angle of Slump)
　　　Angle of Spatula (Angle of Withdrawal)
　　　Cohesion
ASTM method D854-98
　　　Specific Gravity
ASTM Method D422-63
　　　Particle Size Distribution

Designation: D 2216 – 98

Standard Test Method for
Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass[1]

This standard is issued under the fixed designation D 2216; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope *

1.1 This test method covers the laboratory determination of the water (moisture) content by mass of soil, rock, and similar materials where the reduction in mass by drying is due to loss of water except as noted in 1.4, 1.5, and 1.7. For simplicity, the word "material" hereinafter also refers to either soil or rock, whichever is most applicable.

1.2 Some disciplines, such as soil science, need to determine water content on the basis of volume. Such determinations are beyond the scope of this test method.

1.3 The water content of a material is defined in 3.2.1.

1.4 The term "solid material" as used in geotechnical engineering is typically assumed to mean naturally occurring mineral particles of soil and rock that are not readily soluble in water. Therefore, the water content of materials containing extraneous matter (such as cement, and the like) may require special treatment or a qualified definition of water content. In addition, some organic materials may be decomposed by oven drying at the standard drying temperature for this method (110°C). Materials containing gypsum (calcium sulfate dihydrate or other compounds having significant amounts of hydrated water) may present a special problem as this material slowly dehydrates at the standard drying temperature (110°C) and at very low relative humidities, forming a compound (calcium sulfate hemihydrate) which is not normally present in natural materials except in some desert soils. In order to reduce the degree of dehydration of gypsum in those materials containing gypsum, or to reduce decomposition in highly organic soils, it may be desirable to dry these materials at 60°C or in a desiccator at room temperature. Thus, when a drying temperature is used which is different from the standard drying temperature as defined by this test method, the resulting water content may be different from standard water content determined at the standard drying temperature.

NOTE 1—Test Methods D 2974 provides an alternate procedure for determining water content of peat materials.

1.5 Materials containing water with substantial amounts of soluble solids (such as salt in the case of marine sediments) when tested by this method will give a mass of solids which includes the previously soluble solids. These materials require special treatment to remove or account for the presence of precipitated solids in the dry mass of the specimen, or a qualified definition of water content must be used. For example, see Noorany[2] regarding information on marine soils.

1.6 This test method requires several hours for proper drying of the water content specimen. Test Method D 4643 provides for drying of the test specimen in a microwave oven which is a shorter process. Also see Gilbert[3] for details on the background of this test method.

1.7 This standard requires the drying of material in an oven at high temperatures. If the material being dried is contaminated with certain chemicals, health and safety hazards can exist. Therefore, this standard should not be used in determining the water content of contaminated soils unless adequate health and safety precautions are taken.

1.8 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*
D 653 Terminology Relating to Soil, Rock, and Contained Fluids[4]
D 2974 Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils[4]
D 4220 Practice for Preserving and Transporting Soil Samples[4]
D 4318 Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils[4]
D 4643 Test Method for Determination of Water (Moisture) Content of Soil by the Microwave Oven Method[4]
D 4753 Specification for Evaluating, Selecting, and Specifying Balances and Scales for Use in Soil and Rock Testing[4]

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[2] Noorany, I., "Phase Relations in Marine Soils", Journal of Geotechnical Engineering, ASCE, Vol. 110, No. 4, April 1984, pp. 539–543.

[3] Gilbert, P.A., "Computer Controlled Microwave Oven System for Rapid Water Content Determination", Tech. Report GL-88–21, Department of the Army, Waterways Experiment Station, Corps of Engineers, Vicksburg, MS, November 1988.

[4] Annual Book of ASTM Standards, Vol 04.08.

A Summary of Changes section appears at the end of this standard.

D 2216

D 6026   Guide for Using Significant Digits in Calculating and Reporting Geotechnical Test Data[5]

E 145 Specification for Gravity-Convection And Forced-Ventilation Ovens[6]

3. Terminology

3.1 Refer to Terminology D 653 for standard definitions of terms.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *water content (of a material)*—the ratio expressed as a percent of the mass of "pore" or "free" water in a given mass of material to the mass of the solid material. A standard temperature of 110° ± 5°C is used to determine these masses.

4. Summary of Test Method

4.1 A test specimen is dried in an oven at a temperature of 110° ± 5°C to a constant mass. The loss of mass due to drying is considered to be water. The water content is calculated using the mass of water and the mass of the dry specimen.

5. Significance and Use

5.1 For many materials, the water content is one of the most significant index properties used in establishing a correlation between soil behavior and its index properties.

5.2 The water content of a material is used in expressing the phase relationships of air, water, and solids in a given volume of material.

5.3 In fine-grained (cohesive) soils, the consistency of a given soil type depends on its water content. The water content of a soil, along with its liquid and plastic limits as determined by Test Method D 4318, is used to express its relative consistency or liquidity index.

6. Apparatus

6.1 *Drying Oven*, thermostatically-controlled, preferably of the forced-draft type, meeting the requirements of Specification E 145 and capable of maintaining a uniform temperature of 110 ± 5°C throughout the drying chamber.

6.2 *Balances*—All balances must meet the requirements of Specification D 4753 and this section. A Class GP1 balance of 0.01g readability is required for specimens having a mass of up to 200 g (excluding mass of specimen container) and a Class GP2 balance of 0.1g readability is required for specimens having a mass over 200 g. However, the balance used may be controlled by the number of significant digits needed (see 8.2.1 and 12.1.2).

6.3 *Specimen Containers*—Suitable containers made of material resistant to corrosion and change in mass upon repeated heating, cooling, exposure to materials of varying pH, and cleaning. Unless a dessicator is used, containers with close-fitting lids shall be used for testing specimens having a mass of less than about 200 g; while for specimens having a mass greater than about 200 g, containers without lids may be used (see Note 7). One container is needed for each water content determination.

NOTE 2—The purpose of close-fitting lids is to prevent loss of moisture from specimens before initial mass determination and to prevent absorption of moisture from the atmosphere following drying and before final mass determination.

6.4 *Desiccator*—A desiccator cabinet or large desiccator jar of suitable size containing silica gel or anhydrous calcium sulfate. It is preferable to use a desiccant which changes color to indicate it needs reconstitution. See 10.5.

NOTE 3—Anhydrous calcium sulfate is sold under the trade name Drierite.

6.5 *Container Handling Apparatus*, gloves, tongs, or suitable holder for moving and handling hot containers after drying.

6.6 *Miscellaneous*, knives, spatulas, scoops, quartering cloth, sample splitters, etc, as required.

7. Samples

7.1 Samples shall be preserved and transported in accordance with Practice 4220 Groups B, C, or D soils. Keep the samples that are stored prior to testing in noncorrodible airtight containers at a temperature between approximately 3 and 30°C and in an area that prevents direct contact with sunlight. Disturbed samples in jars or other containers shall be stored in such a way as to prevent or minimize moisture condensation on the insides of the containers.

7.2 The water content determination should be done as soon as practicable after sampling, especially if potentially corrodible containers (such as thin-walled steel tubes, paint cans, etc.) or plastic sample bags are used.

8. Test Specimen

8.1 For water contents being determined in conjunction with another ASTM method, the specimen mass requirement stated in that method shall be used if one is provided. If no minimum specimen mass is provided in that method then the values given below shall apply. See Howard[7] for background data for the values listed.

8.2 The minimum mass of moist material selected to be representative of the total sample shall be in accordance with the following:

Maximum particle size (100 % passing)	Standard Sieve Size	Recommended minimum mass of moist test specimen for water content reported to ±0.1 %	Recommended minimum mass of moist test specimen for water content reported to ±1 %
2 mm or less	No. 10	20 g	20 g[A]
4.75 mm	No. 4	100 g	20 g[A]
9.5 mm	⅜-in.	500 g	50 g
19.0 mm	¾-in.	2.5 kg	250 g
37.5 mm	1½ in.	10 kg	1 kg
75.0 mm	3-in.	50 kg	5 kg

[A]To be representative not less than 20 g shall be used.

8.2.1 The minimum mass used may have to be increased to obtain the needed significant digits for the mass of water when reporting water contents to the nearest 0.1 % or as indicated in 12.1.2.

[5] *Annual Book of ASTM Standards,* Vol 04.09.

[6] *Annual Book of ASTM Standards,* Vol 14.02.

[7] Howard, A. K., "Minimum Test Specimen Mass for Moisture Content Determination", *Geotechnical Testing Journal,* A.S.T.M., Vol. 12, No. 1, March 1989, pp. 39-44.

8.3 Using a test specimen smaller than the minimum indicated in 8.2 requires discretion, though it may be adequate for the purposes of the test. Any specimen used not meeting these requirements shall be noted on the test data forms or test data sheets.

8.4 When working with a small (less than 200g) specimen containing a relatively large gravel particle, it is appropriate not to include this particle in the test specimen. However, any discarded material shall be described and noted on the test data forms or test data sheets.

8.5 For those samples consisting entirely of intact rock, the minimum specimen mass shall be 500 g. Representative portions of the sample may be broken into smaller particles, depending on the sample's size, the container and balance being used and to facilitate drying to constant mass, see 10.4. Specimen sizes as small as 200 g may be tested if water contents of only two significant digits are acceptable.

9. Test Specimen Selection

9.1 When the test specimen is a portion of a larger amount of material, the specimen must be selected to be representative of the water condition of the entire amount of material. The manner in which the test specimen is selected depends on the purpose and application of the test, type of material being tested, the water condition, and the type of sample (from another test, bag, block, and the likes.)

9.2 For disturbed samples such as trimmings, bag samples, and the like, obtain the test specimen by one of the following methods (listed in order of preference):

9.2.1 If the material is such that it can be manipulated and handled without significant moisture loss and segregation, the material should be mixed thoroughly and then select a representative portion using a scoop of a size that no more than a few scoopfuls are required to obtain the proper size of specimen defined in 8.2.

9.2.2 If the material is such that it cannot be thoroughly mixed or mixed and sampled by a scoop, form a stockpile of the material, mixing as much as possible. Take at least five portions of material at random locations using a sampling tube, shovel, scoop, trowel, or similar device appropriate to the maximum particle size present in the material. Combine all the portions for the test specimen.

9.2.3 If the material or conditions are such that a stockpile cannot be formed, take as many portions of the material as practical, using random locations that will best represent the moisture condition. Combine all the portions for the test specimen.

9.3 Intact samples such as block, tube, split barrel, and the like, obtain the test specimen by one of the following methods depending on the purpose and potential use of the sample.

9.3.1 Using a knife, wire saw, or other sharp cutting device, trim the outside portion of the sample a sufficient distance to see if the material is layered and to remove material that appears more dry or more wet than the main portion of the sample. If the existence of layering is questionable, slice the sample in half. If the material is layered, see 9.3.3.

9.3.2 If the material is not layered, obtain the specimen meeting the mass requirements in 8.2 by: (1) taking all or one-half of the interval being tested; (2) trimming a representative slice from the interval being tested; or (3) trimming the exposed surface of one-half or from the interval being tested.

Note 4—Migration of moisture in some cohesionless soils may require that the full section be sampled.

9.3.3 If a layered material (or more than one material type is encountered), select an average specimen, or individual specimens, or both. Specimens must be properly identified as to location, or what they represent, and appropriate remarks entered on the test data forms or test data sheets.

10. Procedure

10.1 Determine and record the mass of the clean and dry specimen container (and its lid, if used).

10.2 Select representative test specimens in accordance with Section 9.

10.3 Place the moist test specimen in the container and, if used, set the lid securely in position. Determine the mass of the container and moist material using a balance (see 6.2) selected on the basis of the specimen mass. Record this value.

Note 5—To prevent mixing of specimens and yielding of incorrect results, all containers, and lids if used, should be numbered and the container numbers shall be recorded on the laboratory data sheets. The lid numbers should match the container numbers to eliminate confusion.

Note 6—To assist in the oven-drying of large test specimens, they should be placed in containers having a large surface area (such as pans) and the material broken up into smaller aggregations.

10.4 Remove the lid (if used) and place the container with moist material in the drying oven. Dry the material to a constant mass. Maintain the drying oven at 110 ± 5°C unless otherwise specified (see 1.4). The time required to obtain constant mass will vary depending on the type of material, size of specimen, oven type and capacity, and other factors. The influence of these factors generally can be established by good judgment, and experience with the materials being tested and the apparatus being used.

Note 7—In most cases, drying a test specimen overnight (about 12 to 16 h) is sufficient. In cases where there is doubt concerning the adequacy of drying, drying should be continued until the change in mass after two successive periods (greater than 1 h) of drying is an insignificant amount (less than about 0.1 %). Specimens of sand may often be dried to constant mass in a period of about 4 h, when a forced-draft oven is used.

Note 8—Since some dry materials may absorb moisture from moist specimens, dried specimens should be removed before placing moist specimens in the same oven. However, this would not be applicable if the previously dried specimens will remain in the drying oven for an additional time period of about 16 h.

10.5 After the material has dried to constant mass remove the container from the oven (and replace the lid if used). Allow the material and container to cool to room temperature or until the container can be handled comfortably with bare hands and the operation of the balance will not be affected by convection currents and/or its being heated. Determine the mass of the container and oven-dried material using the same type/capacity balance used in 10.3. Record this value. Tight fitting lids shall be used if it appears that the specimen is absorbing moisture from the air prior to determination of its dry mass.

Note 9—Cooling in a desiccator is acceptable in place of tight fitting lids since it greatly reduces absorption of moisture from the atmosphere during cooling especially for containers without tight fitting lids.

11. Calculation

11.1 Calculate the water content of the material as follows:

$$w = [(M_{cws} - M_{cs})/(M_{cs} - M_c)] \times 100 = \frac{M_w}{M_s} \times 100 \qquad (1)$$

where:

w	=	water content, %,
M_{cws}	=	mass of container and wet specimen, g,
M_{cs}	=	mass of container and oven dry specimen, g,
M_c	=	mass of container, g,
M_w	=	mass of water ($M_w = M_{cws} - M_{cds}$), g, and
M_s	=	mass of solid particles ($M_s = M_{cds} - M_c$), g.

12. Report

12.1 Test data forms or test data sheets shall include the following:

12.1.1 Identification of the sample (material) being tested, such as boring number, sample number, test number, container number etc.

12.1.2 Water content of the specimen to the nearest 1 % or 0.1 %, as appropriate based on the minimum sample used. If this method is used in concert with another method, the water content of the specimen should be reported to the value required by the test method for which the water content is being determined. Refer to Guide D 6026 for guidance concerning significant digits, especially if the value obtained from this test method is to be used to calculate other relationships such as unit weight or density. For instance, if it is desired to express dry unit weight to the nearest 0.1 lbf/f³(0.02 kN/m³), it may be necessary to use a balance with a greater readability or use a larger specimen mass to obtain the required significant digits the mass of water so that the water content can be determined to the required significant digits. Also, the significant digits in Guide D 6026 may need to be increased when calculating phase relationships requiring four significant digits.

12.1.3 Indicate if test specimen had a mass less than the minimum indicated in 8.2.

12.1.4 Indicate if test specimen contained more than one material type (layered, etc.).

12.1.5 Indicate the temperature of drying if different from 110 ± 5°C.

12.1.6 Indicate if any material (size and amount) was excluded from the test specimen.

12.2 When reporting water content in tables, figures, etc., any data not meeting the requirements of this test method shall be noted, such as not meeting the mass, balance, or temperature requirements or a portion of the material is excluded from the test specimen.

13. Precision and Bias

13.1 *Statement on Bias*—There is no accepted reference value for this test method; therefore, bias cannot be determined.

13.2 *Statements on Precision*:

13.2.1 *Single-Operator Precision (Repeatability)*—The single-operator coefficient of variation has been found to be 2.7 percent. Therefore, results of two properly conducted tests by the same operator with the same equipment should not be considered suspect unless they differ by more than 7.8 percent of their mean.[8]

13.2.2 *Multilaboratory Precision (Reproducibility)*[9]—The multilaboratory coefficient of variation has been found to be 5.0 percent. Therefore, results of two properly conducted tests by different operators using different equipment should not be considered suspect unless they differ by more than 14.0 percent of their mean.

14. Keywords

14.1 consistency; index property; laboratory; moisture analysis; moisture content; soil aggregate; water content

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[8] These numbers represent the (1s) and (d2s) limits as described in Practice C 670.

[9] These numbers represent the (1s %) and (d2s %) limits as described in Practice C 670.

SUMMARY OF CHANGES

Committee D-18 has identified the location of selected changes to this standard since the last issue. (D 2216-92) that may impact the use of this standard.

(*1*) Title was changed to emphasize that mass is the basis for the standard.

(*2*) Section 1.1 was revised to clarify "similar materials".

(*3*) New 1.2 was added to explain a limitation in scope. The other sections were renumbered as appropriate.

(*4*) An information reference was included in 1.5.

(*5*) An information reference was included in 1.6

(*6*) A new ASTM referenced document was included in 2.1.

(*7*) New Footnotes 2, 3, and 5 were added and identified. Other footnotes were renumbered where necessary for sequential identification.

(*8*) Information concerning balances was added in 6.2

(*9*) Section 6.3 was revised to clarify the use of close-fitting lids, and a reference to Note 8 was added.

(*10*) In 6.4, "anhydrous calcium phosphate" was changed to "anyhydrous calcium sulfate" to correct an error and to agree with Note 3.

(*11*) A typo in 8.1 was corrected from "before" to "below" and a footnoted reference was added for information.

(*12*) A portion of 8.2 was deleted for clarity.

(*13*) A new 8.2.1 was added to clarify minimum mass requirements.

(*14*) Sections 8.3, 8.4, 9.3.3, and 12.1 were changed to substitute "test data form/sheet" for "report".

(*15*) Footnote seven was identified.

(*16*) Section 9.2.1 was revised to improve clarity and intent.

(*17*) The word "possible" was changed to "practical" in 9.2.3.

(*18*) Section 9.3.1 and 9.3.2 were revised to improve clarity and for practicality.
(*19*) A reference to Guide D 6026 was added in 12.1.2.
(*20*) Footnotes 8 and 9 were added to 13.2.1 and 13.2.2, respectively. These were inadvertently omitted from the 1992 version. These explanations provide clarity and information to the user.
(*21*) A Summary of Changes was added to reflect D-18's policy.

METHOD 1110

CORROSIVITY TOWARD STEEL

1.0 SCOPE AND APPLICATION

1.1 Method 1110 is used to measure the corrosivity toward steel of both aqueous and nonaqueous liquid wastes.

2.0 SUMMARY OF METHOD

2.1 This test exposes coupons of SAE Type 1020 steel to the liquid waste to be evaluated and, by measuring the degree to which the coupon has been dissolved, determines the corrosivity of the waste.

3.0 INTERFERENCES

3.1 In laboratory tests, such as this one, corrosion of duplicate coupons is usually reproducible to within 10%. However, large differences in corrosion rates may occasionally occur under conditions where the metal surfaces become passivated. Therefore, at least duplicate determinations of corrosion rate should be made.

4.0 APPARATUS AND MATERIALS

4.1 An apparatus should be used, consisting of a kettle or flask of suitable size (usually 500 to 5,000 mL), a reflux condenser, a thermowell and temperature regulating device, a heating device (mantle, hot plate, or bath), and a specimen support system. A typical resin flask set up for this type of test is shown in Figure 1.

4.2 The supporting device and container shall be constructed of materials that are not affected by, or cause contamination of, the waste under test.

4.3 The method of supporting the coupons will vary with the apparatus used for conducting the test, but it should be designed to insulate the coupons from each other physically and electrically and to insulate the coupons from any metallic container or other device used in the test. Some common support materials include glass, fluorocarbon, or coated metal.

4.4 The shape and form of the coupon support should ensure free contact with the waste.

1110 - 1
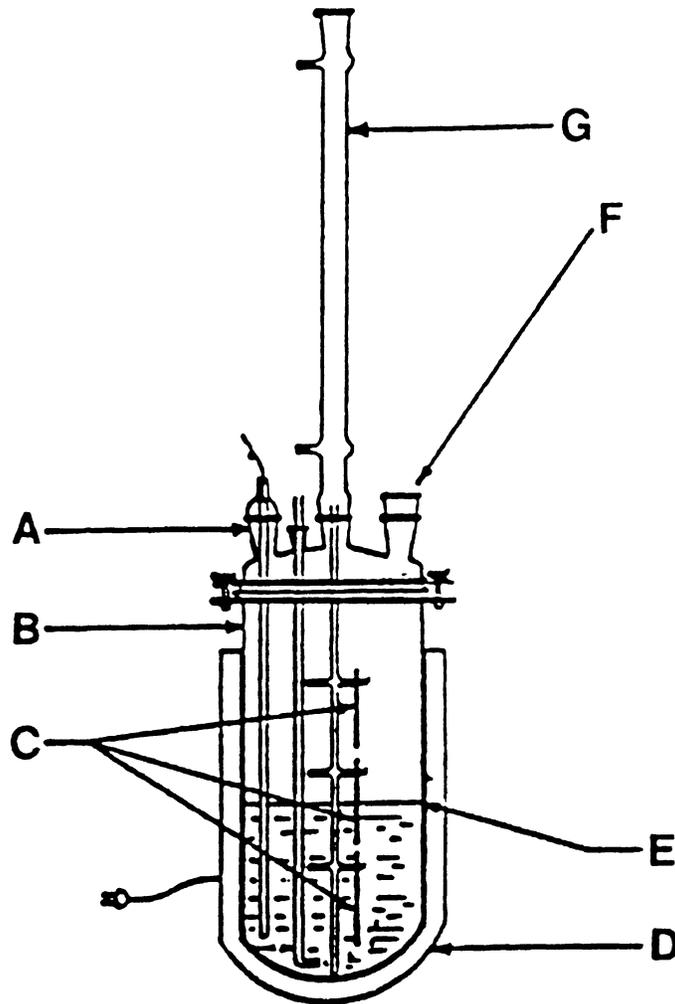
Figure 1.   Typical resin flask that can be used as a versatile and convenient apparatus to conduct simple immersion tests.   Configuration of the flask top is such that more sophisticated apparatus can be added as required by the specific test being conducted.   A = thermowell, B = resin flask, C = specimens hung on supporting device, D = heating mantle, E = liquid interface, F = opening in flask for additional apparatus that may be required, and G = reflux condenser.

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4.5   A circular specimen of SAE 1020 steel of about 3.75 cm (1.5 in.) diameter is a convenient shape for a coupon. With a thickness of approximately 0.32 cm (0.125 in.) and a 0.80-cm (0.4-in.)-diameter hole for mounting, these specimens will readily pass through a 45/50 ground-glass joint of a distillation kettle. The total surface area of a circular specimen is given by the following equation:

$$A = 3.14/2(D^2 - d^2) + (t)(3.14)(D) + (t)(3.14)(d)$$

where:

t = thickness.
D = diameter of the specimen.
d = diameter of the mounting hole.

If the hole is completely covered by the mounting support, the last term in the equation, (t)(3.14)(d), is omitted.

4.5.1   All coupons should be measured carefully to permit accurate calculation of the exposed areas. An area calculation accurate to ±1% is usually adequate.

4.5.2   More uniform results may be expected if a substantial layer of metal is removed from the coupons prior to testing the corrosivity of the waste. This can be accomplished by chemical treatment (pickling), by electrolytic removal, or by grinding with a coarse abrasive. At least 0.254 mm (0.0001 in.) or 2-3 mg/cm^2 should be removed. Final surface treatment should include finishing with #120 abrasive paper or cloth. Final cleaning consists of scrubbing with bleach-free scouring powder, followed by rinsing in distilled water and then in acetone or methanol, and finally by air-drying. After final cleaning, the coupon should be stored in a desiccator until used.

4.5.3   The minimum ratio of volume of waste to area of the metal coupon to be used in this test is 40 mL/cm^2.

5.0 REAGENTS

5.1   Sodium hydroxide (NaOH), (20%): Dissolves 200 g NaOH in 800 mL Type II water and mix well.

5.2   Zinc dust.

5.3   Hydrochloric acid (HCl): Concentrated.

5.4   Stannous chloride (SnCl$_2$).

5.5   Antimony chloride (SbCl$_3$).

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6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples should be collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

7.0 PROCEDURE

7.1 Assemble the test apparatus as described in Paragraph 4.0, above.

7.2 Fill the container with the appropriate amount of waste.

7.3 Begin agitation at a rate sufficient to ensure that the liquid is kept well mixed and homogeneous.

7.4 Using the heating device, bring the temperature of the waste to 55°C (130°F).

7.5 An accurate rate of corrosion is not required; only a determination as to whether the rate of corrosion is less than or greater than 6.35 mm per year is required. A 24-hr test period should be ample to determine whether or not the rate of corrosion is >6.35 mm per year.

7.6 In order to determine accurately the amount of material lost to corrosion, the coupons have to be cleaned after immersion and prior to weighing. The cleaning procedure should remove all products of corrosion while removing a minimum of sound metal. Cleaning methods can be divided into three general categories: mechanical, chemical, and electrolytic.

7.6.1 Mechanical cleaning includes scrubbing, scraping, brushing, and ultrasonic procedures. Scrubbing with a bristle brush and mild abrasive is the most popular of these methods. The others are used in cases of heavy corrosion as a first step in removing heavily encrusted corrosion products prior to scrubbing. Care should be taken to avoid removing sound metal.

7.6.2 Chemical cleaning implies the removal of material from the surface of the coupon by dissolution in an appropriate solvent. Solvents such as acetone, dichloromethane, and alcohol are used to remove oil, grease, or resinous materials and are used prior to immersion to remove the products of corrosion. Solutions suitable for removing corrosion from the steel coupon are:

Solution	Soaking Time	Temperature
20% NaOH + 200 g/L zinc dust	5 min	Boiling
or		
Conc. HCl + 50 g/L $SnCl_2$ + 20 g/L $SbCl_3$	Until clean	Cold

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7.6.3   Electrolytic cleaning should be preceded by scrubbing to remove loosely adhering corrosion products. One method of electrolytic cleaning that can be employed uses:

Solution:	50 g/L H_2SO_4
Anode:	Carbon or lead
Cathode:	Steel coupon
Cathode current density:	20 amp/cm² (129 amp/in.²)
Inhibitor:	2 cc organic inhibitor/liter
Temperature:	74°C (165°F)
Exposure Period:	3 min.

NOTE: Precautions must be taken to ensure good electrical contact with the coupon to avoid contamination of the cleaning solution with easily reducible metal ions and to ensure that inhibitor decomposition has not occurred. Instead of a proprietary inhibitor, 0.5 g/L of either diorthotolyl thiourea or quinolin ethiodide can be used.

7.7   Whatever treatment is employed to clean the coupons, its effect in removing sound metal should be determined by using a blank (i.e., a coupon that has not been exposed to the waste). The blank should be cleaned along with the test coupon and its waste loss subtracted from that calculated for the test coupons.

7.8   After corroded specimens have been cleaned and dried, they are reweighed. The weight loss is employed as the principal measure of corrosion. Use of weight loss as a measure of corrosion requires making the assumption that all weight loss has been due to generalized corrosion and not localized pitting. In order to determine the corrosion rate for the purpose of this regulation, the following formula is used:

$$\text{Corrosion Rate (mmpy)} = \frac{\text{weight loss} \times 11.145}{\text{area} \times \text{time}}$$

where: weight loss is in milligrams,
          area in square centimeters,
          time in hours, and
          corrosion rate in millimeters per year (mmpy).

8.0 QUALITY CONTROL

8.1   All quality control data should be filed and available for auditing.

8.2   Duplicate samples should be analyzed on a routine basis.

9.0 METHOD PERFORMANCE

9.1 No data provided.

10.0 REFERENCES

1. National Association of Corrosion Engineers, "Laboratory Corrosion Testing of Metals for the Process Industries," NACE Standard TM-01-69 (1972 Revision), NACE, 3400 West Loop South, Houston, TX 77027.

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METHOD 1110
CORROSIVITY TOWARD STEEL

(Start)

↓

7.1 Assemble test apparatus.

↓

7.2 Fill container with waste.

↓

7.3 Agitate.

↓

7.4 Heat.

↓

(A)

(A)

↓

7.6 Clean coupons by mechanical, chemical, and/or electrolytic methods.

↓

7.7 Check effect of cleaning treatment on removing sound metal.

↓

7.8 Determine corrosion rate.

↓

(Stop)
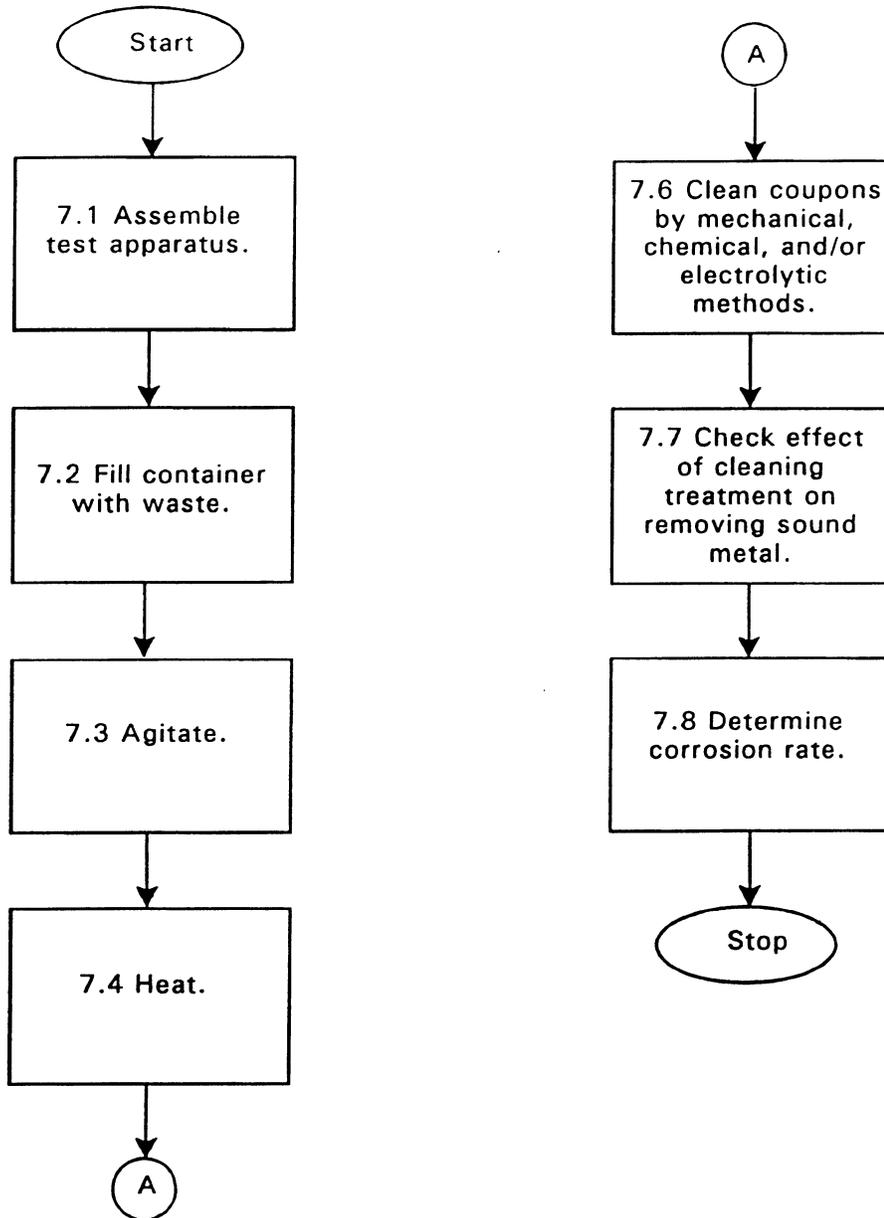
METHOD 9045C

<u>SOIL AND WASTE pH</u>

1.0 SCOPE AND APPLICATION

    1.1 Method 9045 is an electrometric procedure for measuring pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample.

2.0 SUMMARY OF METHOD

    2.1 The sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured.

3.0 INTERFERENCES

    3.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

    3.2 Temperature fluctuations will cause measurement errors.

    3.3 Errors will occur when the electrodes become coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water, or (3) be cleaned per the manufacturer's instructions.

4.0 APPARATUS AND MATERIALS

    4.1 pH Meter with means for temperature compensation.

    4.2 Glass Electrode.

    4.3 Reference electrode: A silver-silver chloride or other reference electrode of constant potential may be used.

            <u>NOTE</u>: Combination electrodes incorporating both measuring and referenced functions are convenient to use and are available with solid, gel-type filling materials that require minimal maintenance.

    4.4 Beaker: 50-mL.

    4.5 Thermometer and/or temperature sensor for automatic compensation.

4.6   Analytical balance: capable of weighing 0.1 g.

5.0   REAGENTS

    5.1   Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

    5.2   Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One.

    5.3   Primary standard buffer salts are available from the National Institute of Standards and Technology (NIST) and should be used in situations where extreme accuracy is necessary. Preparation of reference solutions from these salts requires some special precautions and handling, such as low-conductivity dilution water, drying ovens, and carbon-dioxide-free purge gas. These solutions should be replaced at least once each month.

    5.4   Secondary standard buffers may be prepared from NIST salts or purchased as solutions from commercial vendors. These commercially available solutions, which have been validated by comparison with NIST standards, are recommended for routine use.

6.0   SAMPLE PRESERVATION AND HANDLING

    6.1   All samples must be collected using a sampling plan that addresses the considerations discussed in Chapter Nine of this manual.

    6.2   Samples should be analyzed as soon as possible.

7.0   PROCEDURE

    7.1   Calibration:

        7.1.1   Because of the wide variety of pH meters and accessories, detailed operating procedures cannot be incorporated into this method. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions. Special attention to care of the electrodes is recommended.

        7.1.2   Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart. Repeat adjustments on successive portions of the two buffer solutions until readings are within 0.05 pH units of the buffer solution value. If an accurate pH reading based on the conventional pH scale [0 to 14 at 25°C] is required, the analyst should control sample temperature at 25±1°C when

sample pH approaches the alkaline end of the scale (e.g., a pH of 11 or above).

7.2   Sample preparation and pH measurement of soils:

7.2.1   To 20 g of soil in a 50-mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 minutes. . Additional dilutions are allowed if working with hygroscopic soils and salts or other problematic matrices.

7.2.2   Let the soil suspension stand for about 1 hour to allow most of the suspended clay to settle out from the suspension or filter or centrifuge off the aqueous phase for pH measurement.

7.2.3   Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrodes into the sample solution in this manner. For combination electrodes, immerse just below the suspension.

7.2.4   If the sample temperature differs by more than 2°C from the buffer solution, the measured pH values must be corrected.

7.2.5   Report the results as "soil pH measured in water at __ °C" where "__°C" is the temperature at which the test was conducted.

7.3   Sample preparation and pH measurement of waste materials:

7.3.1   To 20 g of waste sample in a 50-mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 minutes. . Additional dilutions are allowed if working with hygroscopic wastes and salts or other problematic matrices.

7.3.2   Let the waste suspension stand for about 15 minutes to allow most of the suspended waste to settle out from the suspension or filter or centrifuge off aqueous phase for pH measurement.

NOTE: If the waste is hygroscopic and absorbs all the reagent water, begin the experiment again using 20 g of waste and 40 mL of reagent water.

NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned (Step 3.3) if it becomes coated with an oily material.

7.3.3   Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant to establish good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrode into the sample solution

in this manner. For combination electrodes, immerse just below the
suspension.

      7.3.4   If the sample temperature differs by more than 2°C from
the buffer solution, the measured pH values must be corrected.

      7.3.5   Report the results as "waste pH measured in water at __
°C" where "__°C" is the temperature at which the test was conducted.

8.0   QUALITY CONTROL

    8.1   Refer to Chapter One for the appropriate QC protocols.

    8.2   Electrodes must be thoroughly rinsed between samples.

9.0   METHOD PERFORMANCE

    9.1   No data provided.

10.0   REFERENCES

1.   Black, Charles Allen; <u>Methods of Soil Analysis</u>;   American Society of
     Agronomy: Madison, WI, 1973.

2.   National Bureau of Standards, Standard Reference Material Catalog, 1986-
     87, Special Publication 260.

METHOD 9045C

SOIL AND WASTE pH

```
                        ┌──────────────┐
                        │    Start     │
                        └──────┬───────┘
                               │
                               ▼
                        ┌──────────────┐
                        │ 7 1 Calibrate│
                        │each instrument/│
                        │  electrode   │
                        │   system.    │
                        └──────┬───────┘
                               │
                               ▼
┌──────────────┐          ◇────────◇          ┌──────────────┐
│7.2.1 Add 20 mL│  Soil   │  Is the  │         │7.3.1 Add 20 mL│
│water to 20 g soil;◄──────│ sample soil├───────►water to 20 g waste;│◄──────┐
│stir continuously│        │ or waste? │         │stir continuously│      │
│for 5 minutes.│          ◇────────◇          │for 5 minutes.│        │
└──────┬───────┘                               └──────┬───────┘        │
       │                                              │                │
       ▼                                              ▼                │
┌──────────────┐                               ┌──────────────┐        │
│7.2.2 Let soil│                               │7.3.2 Let waste│       │
│ suspension   │                               │ suspension   │        │
│ stand for 1  │                               │ stand for 15 │        │
│hour or filter.│                              │minutes or filter.│    │
└──────┬───────┘                               └──────┬───────┘        │
       │                                              │                │
       │                                              ▼                │
       │                                          ◇────────◇           │
       │                                         │         │  Yes  ┌──────────────┐
       │                                         │ Is waste ├──────►Repeat experiment│
       │                                         │hydroscopic?│     │with 20 g waste│
       │                                          ◇────────◇        │and 40 mL water.│
       │                                              │No           └──────────────┘
       │                                              │
       │      ┌──────────────┐                        ▼
       │      │   Insert     │  No              ◇────────◇          ┌──────────────┐
       └─────►│  electrodes  │◄─────────────────│   Is    │  Yes   │ Decant oily  │
              │ into sample  │                  │supernatant├───────►   phase;     │
              │  solution.   │                  │multiphasic?│       │ measure pH of│
              └──────┬───────┘                   ◇────────◇         │aqueous phase.│
                     │                                │             └──────┬───────┘
                     │                                │                    │Aqueous
                     │                                └────────────────────┘Phase
                     ▼
                 ◇────────◇
┌──────────────┐│   Do    │
│   Correct    ││ sample  │
│ measured pH  │◄──── Yes ─│and buffer│
│   values.    ││sol'n temps│
└──────┬───────┘│vary by  │
       │        │  2C?    │
       │         ◇────────◇
       │             │No
       │             ▼
       │      ┌──────────────┐
       └─────►│    Report    │
              │results and   │
              │ temperature  │
              └──────┬───────┘
                     │
                     ▼
              ┌──────────────┐
              │    Stop      │
              └──────────────┘
```

A∫TM **Designation: D 6393 – 99**

Standard Test Method for
Bulk Solids Characterization by Carr Indices[1]

This standard is issued under the fixed designation D 6393; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the apparatus and procedures for measuring properties of bulk solids, henceforth referred to as Carr Indices.[2]

1.2 This test method is suitable for free flowing and moderately cohesive powders and granular materials up to 2.0 mm in size. Materials must be able to pour through a 7.0 ± 1.0-mm diameter funnel outlet when in an aerated state.

1.3 This method consists of eight measurements and two calculations to provide ten tests for Carr Indices. Each individual test or a combination of several tests can be used to characterize the properties of bulk solids. These ten tests are as follows:

1.3.1 *Test A*—Measurement of Carr Angle of Repose

1.3.2 *Test B*—Measurement of Carr Angle of Fall

1.3.3 *Test C*—Calculation of Carr Angle of Difference

1.3.4 *Test D*—Measurement of Carr Loose Bulk Density

1.3.5 *Test E*—Measurement of Carr Packed Bulk Density

1.3.6 *Test F*—Calculation of Carr Compressibility

1.3.7 *Test G*—Measurement of Carr Cohesion

1.3.8 *Test H*—Measurement of Carr Uniformity

1.3.9 *Test I*—Measurement of Carr Angle of Spatula

1.3.10 *Test J*—Measurement of Carr Dispersibility

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

2.1.1 *Carr angle of difference, n*—the difference between the Carr angle of repose and Carr angle of fall.

2.1.2 *Carr angle of fall, n*—an angle of repose measured from a powder heap to which a defined vibration has been given.

2.1.3 *Carr angle of repose, n*—a measurement from the powder heap built up by dropping the material through a vibrating sieve and funnel above a horizontal plate.

2.1.4 *Carr angle of spatula, n*—a measurement by which a spatula is inserted into a powder heap parallel to the bottom and then lifting it up and out of the material.

2.1.5 *Carr cohesion, n*—a descriptive measure of interparticle forces based on the behavior of the material during sieving.

2.1.6 *Carr compressibility, n*—a calculation made by using Carr loose bulk density and Carr packed bulk density as determined in 5.8.

2.1.7 *Carr dispersibility, n*—a measurement by which a powder sample is dropped through a hollow cylinder above a watch glass and then the amount of powder collected by the watch glass is measured.

2.1.8 *Carr dynamic bulk density, n*—a calculated bulk density of a material. It is used to compute vibration time for the Carr cohesion measurement.

2.1.9 *Carr loose bulk density, n*—a measurement obtained by sieving the sample through a vibrating chute to fill a measuring cup.

2.1.10 *Carr packed bulk density, n*—a measurement obtained by dropping a measuring cup, which is filled with the sample, a specific number of times from the same height. Sometimes known as a *tapped density.*

2.1.11 *Carr uniformity, n*—a measurement calculated from the particle size distribution of the powder as measured by sieving.

3. Significance and Use

3.1 This test method provides measurements that can be used to describe the bulk properties of a powder or granular material.

3.2 The measurements can be combined with practical experience to provide relative rankings of various forms of bulk handling behavior of powders and granular materials for a specific application.

4. Apparatus

4.1 The main instrument includes a *timer/counter* (A), a *vibrating mechanism* (B), an *amplitude gage* (C), a *rheostat* (D), and a *tapping device* (E) (see Fig. 1).[3]

4.1.1 *Timer/Counter*—The timer is used to control the duration of vibration and the number of taps. A minimum 180-s timer for 60 Hz power supply is required. Alternatively, a

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.24 on Characterization and Handling of Powders and Bulk Solids.

Current edition approved April 10, 1999. Published July 1999.

[2] Carr, R.L., "Evaluating Flow Properties of Solids," *Chemical Engineering*, January 18, 1965, pp. 163–168.

[3] Available from Hosokawa Micron International Inc., New York, NY.
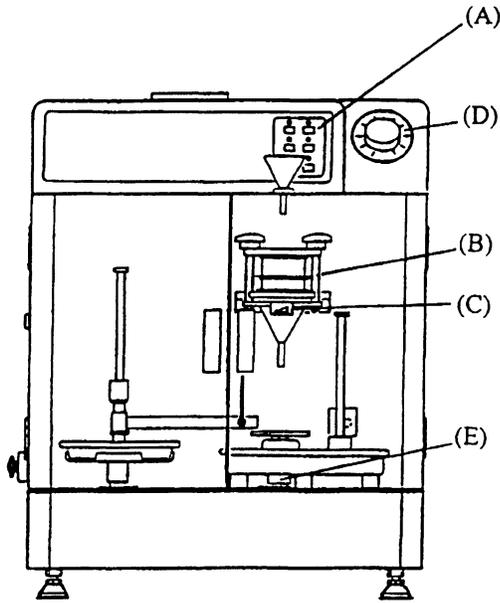
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FIG. 1 Powder Characteristics Tester for Carr Indices

counter can be used to control the number of taps.

4.1.2 *Vibrating Mechanism*, to deliver vibration at 50 to 60 Hz to the vibration plate at an amplitude of 0.0 to 3.0 mm.

4.1.3 *Amplitude Gage*, mounted on the vibration plate to measure the amplitude of the vibration from 0.0 to 4.0 mm.

4.1.4 *Rheostat*—A dial used to adjust the vibration amplitude of vibration plate from 0.0 to 3.0 mm.

4.1.5 *Tapping Device*, consists of tap holder and tapping lift bar (tapping pin), which lifts and free-fall drops a measuring cup a stroke of 18.0 ± 0.1 mm and a rate of 1.0 ± 0.2 taps/s.

4.2 The spatula assembly consists of a *spatula blade* (A), a *pan base/elevator stand* (B), and a *shocker* (C) (see Fig. 2).

4.2.1 *Spatula Blade*—A chrome-plated brass plate mounted on the blade receiver to retain powder while elevator stand

lowers the powder-filled pan. The dimensions of the spatula blade are 80 to 130 mm length, 22.0 ± 0.3-mm width and 3.0 ± 0.3-mm thick.

4.2.2 *Shocker*—A sliding bushing with a mass of 110.0 ± 1.0 g at a drop height of 150.0 ± 10.0 mm, measured from the lower edge of the bushing to the shocker base for the measurement of angle of spatula. The total mass of the shocker assembly including the sliding bushing, pole, spatula blade, and blade receiver is 0.65 ± 0.35 kg.

4.3 A dispersibility measuring unit consists of a *container* (A) with *shutter cover* (B), a *cylindrical glass tube* (C), and a *watch glass* (D), (see Fig. 3).

4.3.1 *Container*—A hopper unit with a shutter cover at the bottom to support a powder sample. The shutter cover opens horizontally to release the powder sample which then falls through the glass tube onto the watch glass.

4.3.2 *Cylindrical Glass Tube*, located vertically 170.0 ± 10.0 mm under the shutter cover to confine the scattering/ dispersed powder. The dimension of the tube is 100.0± 5.0-mm diameter and 330.0 ± 10.0-mm length.

4.3.3 *Watch Glass*, centered 101.0 ± 1.0 mm under the cylindrical glass tube to collect undispersed powder. The dimension of watch glass is 100.0 ± 5.0-mm diameter and 2.0 ± 0.1-mm thickness with the radius of curvature of 96.3 mm, concave upwards.

4.4 *Accessories*:

4.4.1 *Spatula Pan*—A stainless steel pan with at least a 100.0-mm width, a 125.0-mm length, a 25.0 mm height, and a 1.0-mm thickness, used to retain powder for the preparation of the measurement of Carr angle of spatula.

4.4.2 *Scoop*—A stainless steel container used to transport powder.

4.4.3 *Scraper*—A chrome plated brass or stainless steel plate used to scrape off excess powder in the cup.

4.4.4 *Cup*—A 100-cm^3 stainless steel cylindrical container with the inside dimensions of 50.5 ± 0.1-mm diameter and
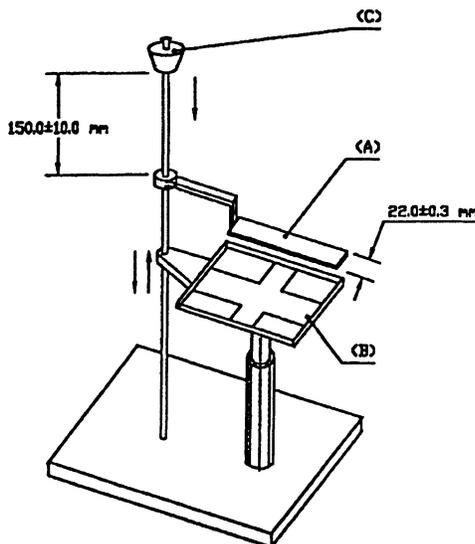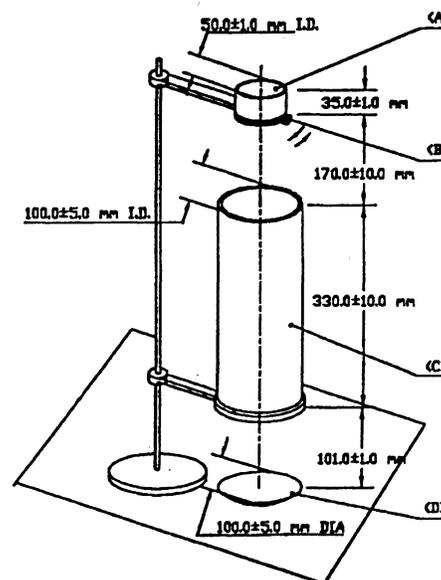

FIG. 2 Carr Spatula Assembly

FIG. 3 Carr Dispersibility Measuring Unit

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49.9 ± 0.1-mm height used for Carr bulk density measurement. The wall thickness of the cup is 1.75 ± 0.25 mm. The interior walls of the cup are sufficiently smooth that machining marks are not evident.

4.4.5 *Cup Extension*—A white Delrin®⁴ extension sleeve for the 100 cm³ measuring cup, 55.0 ± 0.1 mm in diameter by 48.0 ± 1.0 mm in height.

4.4.6 *Funnel for Angle of Repose*—A glass funnel with 55° angle bowls as measured from the horizontal, 7.0 ± 1.0-mm bottom outlet diameter and outlet stem length 33.5 mm for the measurement of Carr angle of repose.

4.4.7 *Stationary Chute*—A stainless steel conical chute with the dimensions of 75.0-mm top diameter, 55.0-mm height, and 50.0-mm bottom diameter to guide the powder flow into the measuring cup (see 4.4.4).

4.4.8 *Vibration Chute*—A stainless steel conical chute with the dimensions of 75.0-mm top diameter, 55.0-mm.height, and 50.0-mm bottom diameter installed on the vibration plate to guide the powder flow to the stationary chute or cup extension.

4.4.9 *Sieves*, certified 76.0-mm diameter stainless steel sieves with the opening of 710 μm, 355 μm, 250 μm, 150 μm, 75 μm, and 45 μm.

4.4.10 *Sieve Extension*—A stainless steel extension piece used as a spacer in the vibration unit when only one sieve is used.

4.4.11 *Spacer Ring*—A white Delrin® spacer inserted between sieve and vibration chute or glass funnel to protect them from damage.

4.4.12 *Sieve Holding Bar*—A chrome-plated brass holding bar used to hold sieve assembly on the vibration plate.

4.4.13 *Pan*, with base for tapping device, measuring cup, and shocker. A stainless steel pan, at least 210.0-mm length, 150.0-mm width, 35.0-mm height, and 1.0-mm thickness, designed to accept tapping device, measuring cup and platform, as well as provide a stand base for shocker.

4.4.14 *Platform*—A chrome-plated brass circular platform with a diameter of 80.0 ± 0.3 mm and a height of 59.0 ± 2.0 mm to be used for the measurement of Carr angle of repose.

4.4.15 *Shocker*—A sliding bushing with a mass of 110.0 ± 1.0 g at a drop height of 150.0 ± 10.0 mm, measured from the lower edge of the bushing to the shocker base for the measurement of Carr angle of fall. The total mass of the shocker, platform, and pan for the measurement of angle of fall is 1.35 ± 0.25 kg.

NOTE 1—The pan has molded-in feet so it is slightly raised from the table top. This helps make vibration more consistent.

4.4.16 *Brush*, a laboratory brush for dust removal.

4.4.17 *Cover*, for measuring dispersibility. A removable enclosure to confine the dust of sample powder when it falls onto the watch glass for the measurement of Carr dispersibility.

4.5 *Balance*, capable of measuring sample mass to an accuracy of ± 0.01 g with a max of 2.0 kg.

4.6 *Data Acquisition Equipment*—A microprocessor or computer may be used to guide the measuring operation, collect data, calculate data, and print test results.

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⁴ Delrin®

5. Procedure

5.1 A representative powder sample from process stream should be riffled carefully into portions for each individual measurement.

5.2 All the measurements should be performed on a strong, horizontally-leveled bench or work table. If possible, use a concrete or stone-topped table.

Test A—Measurement of Carr Angle of Repose

5.3 *Placement of Parts*:

5.3.1 Place the parts onto the vibration plate in the following order starting at the bottom:

5.3.1.1 Glass funnel;

5.3.1.2 Spacer ring;

5.3.1.3 Sieve with opening of 710 μm;

5.3.1.4 Sieve extension; and,

5.3.1.5 Sieve holding bar.

5.3.2 Fasten the vibration assembly with knob nuts located on both sides of sieve holding bar.

5.3.3 Center the platform under the glass funnel.

5.3.4 Position the stem end of the glass funnel 76.0± 1.0 mm above the platform.

5.3.5 Set desired vibration time on timer (usually 180 s on 60 Hz vibrating frequency is selected).

5.3.6 Pour 200 to 300 cm³ of powder over the sieve using the scoop.

5.3.7 Set vibration adjustment dial (Rheostat) to 0.

5.3.8 Turn on the vibrating mechanism and timer.

5.3.9 Gradually increase the amplitude of the vibration, no more than 0.2 mm at a time, by incrementally turning the vibration adjustment dial until powder starts to flow out of the end of the glass funnel and builds up on the circular platform in a conical shape.

5.3.10 Turn off the vibration mechanism when the powder starts to fall from the edge of the platform and the powder pile is completely formed.

5.3.11 If a conical shape is not completely formed, remove the powder pile and repeat steps 5.3.6-5.3.10.

5.3.12 After the cone has been built up, calculate an average angle of the cone (from horizontal) in relation to the edge of the platform by the equation below. This average angle is called the Carr angle of repose.

$$\text{Carr Angle of Repose} = \tan^{-1}[H/R] \qquad (1)$$

where:
H = Height of the powder pile, mm, and
R = Radius of the circular platform, mm.

5.3.13 Indicate the shape of the cone either Concave Up (A), Concave Down (B), or Straight (C) (see Fig. 4) in the report.

5.3.14 If the cone is irregular in shape, repeat the test three times and obtain an average.

5.3.15 If the powder has free-flowing characteristics or has coarse particles larger than 710 μm, the vibration and 710 μm sieve are not necessary. In this case, use the scoop to slowly pour the powder through the funnel. Adjust the pouring rate so that it takes 15 to 30 s to form the conical pile.

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(A) Concave Up   (B) Concave Down   (C) Straight
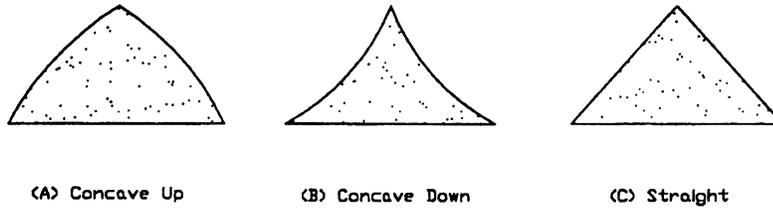
FIG. 4 The Shape of the Powder Pile

Test B—Measurement of Carr Angle of Fall

5.4 After determining the Carr Angle of Repose as in 5.3, place the shocker on the shocker base.

5.5 Then raise the sliding bushing carefully (so that the cone will not be disturbed) to the upper end of the pole (at a drop height of 150.0 ± 10.0 mm) and let it fall to give a shock to the pan. Repeat this three times. The powder layer will be collapsed and exhibit a smaller angle of repose.

5.6 Wait for 30 s after the final shock and then measure the angle as described in 5.3.12-5.3.14. This new, lower angle is called Carr angle of fall.

Test C—Calculation of Carr Angle of Difference

5.7 Subtract the Carr angle of fall from the Carr angle of repose to obtain the Carr angle of difference.

Test D—Measurement of Carr Loose Bulk Density

5.8 *Placement of Parts*:

5.8.1 Place the parts onto the vibration plate in the following order starting at the bottom:

5.8.1.1 Vibration chute;

5.8.1.2 Spacer ring;

5.8.1.3 Sieve with opening of 710 μm;

5.8.1.4 Sieve extension; and,

5.8.1.5 Sieve holding bar.

5.8.2 Fasten the vibration assembly with knob nuts located on both sides of sieve holding bar.

5.8.3 Support the stationary chute below the vibration chute.

5.8.4 Place the pan directly under the stationary chute and position the measuring cup in its base. Make sure the center of the measuring cup is in alignment below the center of the stationary chute and the distance between them is 30.0 ± 5.0 mm.

5.8.5 Use scoop to pour 200 to 300 cm³ of the powder onto the sieve.

5.8.6 Set vibration time on timer (a normal vibration time is about 30 s).

5.8.7 Set vibration adjustment dial (rheostat) to 0.

5.8.8 Turn on the vibrating mechanism and timer.

5.8.9 Adjust the amplitude of vibration to control the powder flow rate so that the powder will fill the cup within 20 to 30 s.

5.8.10 When the cup is filled and overflowing, stop the vibration.

5.8.11 Using the scraper, lift and scrape excess material from the top of the cup as shown in Fig. 5. Remove small quantities at a time, and continue the process until the material is flush with the top of the cup. Do not exert a downward force with the scraper.

5.8.12 Weigh the cup with powder.

5.8.13 Subtract the empty cup mass from that of cup with powder. The difference divided by 100 is the Carr loose bulk density of the powder in g/cm³.
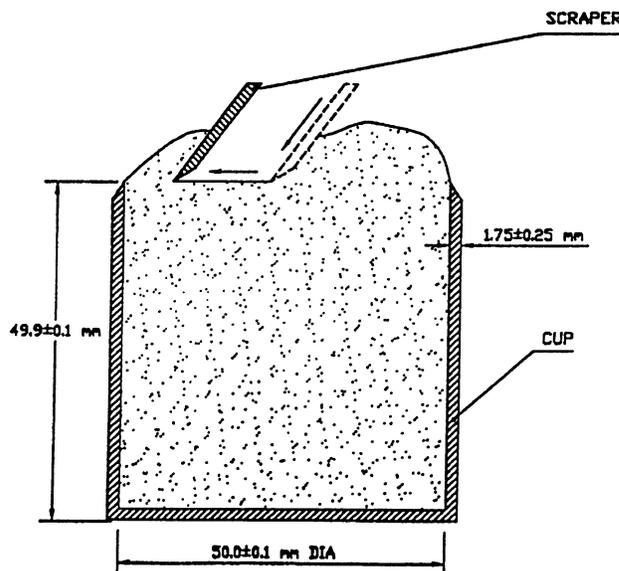
FIG. 5 Scraping Off Excess Powder

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Note 2—The cup is exactly 100 cm³ in volume.

5.8.14 Repeat steps 5.8.5-5.8.13 three to five times and obtain an average value.

5.8.15 When the powder is free-flowing and of fairly coarse particle size, it will not be necessary to use the vibrating sieve. The powder can be poured gently into the cup by the scoop.

Test E—Measurement of Carr Packed Bulk Density

5.9 This test is known in the field as a tapped bulk density even though the sample is dropped instead tapped.

5.9.1 Prepare the parts in the same order as with the measurement for Carr loose bulk density but without using the stationary chute.

5.9.2 Place the cup extension on the top of the measuring cup.

5.9.3 Fill the cup with sample powder to the top with the scoop and place it on the tapping device.

5.9.4 Set timer for a desired tapping duration (usually 180 s on 60 Hz power supply is selected). Alternatively, use a counter to control the number of taps.

Note 3—The optimal number of taps for consistent results is determined by repetitive tests in which the relationship between the tapped bulk density and number of taps is examined. The number of taps should be sufficiently large so that additional taps do not result in an increase in tapped bulk density.

5.9.5 Turn on the tapping device.

5.9.6 During the tapping period, it is necessary to observe the level of the powder and, if necessary, add powder to the cup

extension so that the final powder level will not be below the rim of the measuring cup.

5.9.7 When the tapping is completed, remove the cup and its extension from the tapping device.

5.9.8 Remove the cup extension and scrape off excessive powder from the cup surface as described in 5.8.11.

5.9.9 Weigh the cup with the packed powder and subtract the empty cup mass from it. The difference divided by 100 is the Carr packed bulk density of the powder in g/cm³. (The cup is exactly 100 cm³ in volume).

Test F—Calculation of Carr Compressibility

5.10 Carr compressibility (C) is calculated by the following equation from the Carr loose bulk density (L), in 5.8 and the Carr packed bulk density (P) in 5.9.

$$C = 100\,(P-L)/P \qquad (2)$$

Test G—Measurement of Carr Cohesion

5.11 Determine Carr loose bulk density (L), and Carr packed bulk density (P), as described in 5.8 and 5.9. Determine particle size distribution by sieving.

5.11.1 Refer to the selection guide in Fig. 6 to determine if Carr cohesion measurement is recommended. Proceed to 5.12 for Carr uniformity measurement, if Carr cohesion measurement is not recommended. If Carr cohesion measurement is recommended, select the proper sieve sizes from Fig. 6.

5.11.2 Place the parts on the vibration plate in the following order, starting at the bottom:

FIG. 6 Selection Guide for Carr Cohesion Measurement

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5.11.2.1 Vibration chute.

5.11.2.2 Spacer ring.

5.11.2.3 Sieve 1 (smallest opening).

5.11.2.4 Sieve 2 (midsize opening).

5.11.2.5 Sieve 3 (largest opening).

5.11.2.6 Sieve holding bar.

5.11.3 Fasten the vibration assembly with knob nuts located on both sides of sieve holding bar.

5.11.4 Turn on the vibrating mechanism and adjust the amplitude of vibration to 1.0 mm with vibration adjustment dial. Wait until the vibration amplitude becomes stabilized, then turn off the vibration but keep the position of vibration adjustment dial as it was.
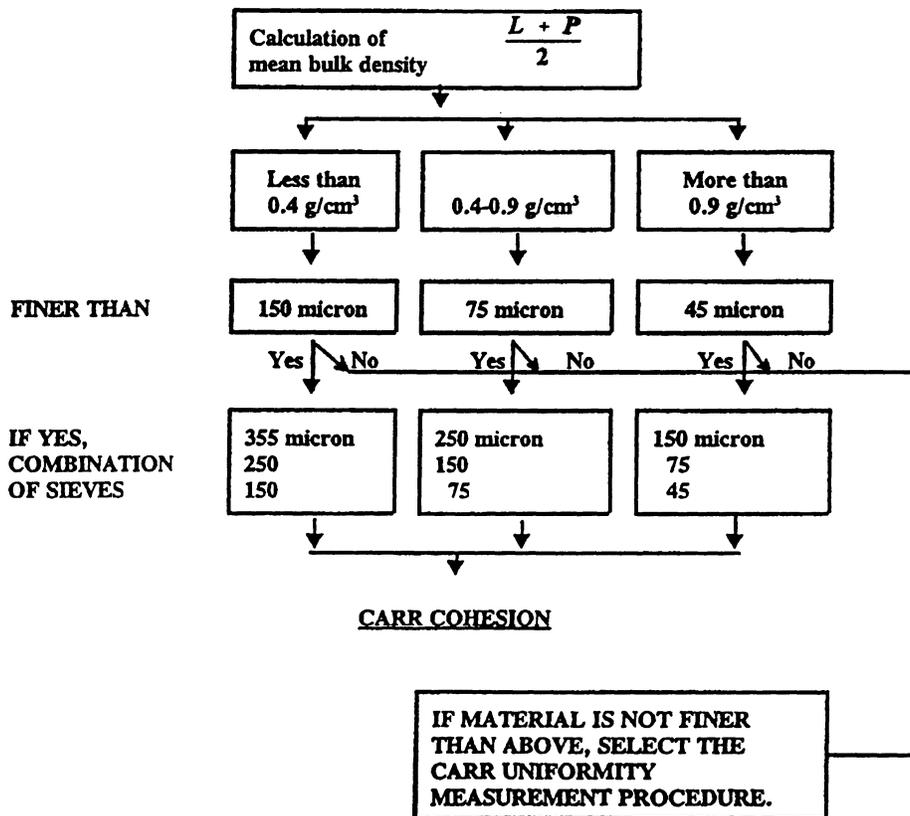
5.11.5 Set timer according to the vibration time calculated as follows:

$$T(s) = 20 + [(1.6 - W)/0.016] \qquad (3)$$

$$W = [(P - L)C/100] + L \qquad (4)$$

where:

T = Vibration time, s,

W = Carr dynamic bulk density, g/cm^3,

C = Carr compressibility, %,

L = Carr loose bulk density, g/cm^3, and

P = Carr packed bulk density, g/cm^3.

NOTE 4—If Carr dynamic bulk density, W, is greater than 1.6 g/cm^3, vibration time, T, should be set at 20 s.

5.11.6 Weigh 2.0 ± 0.01 g of powder and place it on the top sieve.

5.11.7 Turn on the vibration mechanism.

5.11.8 When vibration stops after time T, loosen the knob nuts and remove the three sieves and weigh the amount of powder retained on each sieve. Brush off all material from the sieves.

5.11.9 Carr Cohesion is calculated as follows:

$$[\text{(Powder mass retained on the largest sieve)}/2g] \times 100 \qquad (5)$$

$$[\text{(Powder mass retained on the midsize sieve)}/2g] \times 100 \times (3/5) \qquad (6)$$

$$[\text{(Powder mass retained on the smallest sieve)}/2g] \times 100 \times (1/5) \qquad (7)$$

The sum of the three calculated values (Eq 5-7) will give the Carr cohesion [%].

Test H—Measurement of Carr Uniformity

5.12 This measurement is applied instead of Carr cohesion measurement when the powder is relatively coarse and not so cohesive. See Fig. 6.

5.12.1 Obtain the particle size distribution of the sample powder by sieve analysis.

5.12.2 From the particle size distribution curve, determine a particle size of which 60 % of the powder by volume passes the sieve (d_{60}) and a particle size of which 10 % passes the sieve (d_{10}).

5.12.3 Carr uniformity is calculated below:

$$\text{Carr uniformity} = d_{60}/d_{10} \qquad (8)$$

Test I—Measurement of Carr Angle of Spatula

5.13 Set the Carr spatula assembly in place as described in 4.2.

5.13.1 Put the spatula pan on the pan base.

5.13.2 Raise the pan until the pan bottom contacts the spatula.

5.13.3 Use a scoop to pour the sample powder into the pan so that the spatula is completely covered with several centimeters of powder (about 200 to 300 cm^3 on the spatula). Be consistent about the amount of powder used for each measurement, that is, same depth of powder over the spatula.

5.13.4 Slowly lower the pan away from the spatula. This will expose the spatula with a considerable amount of powder on it.

5.13.5 Calculate an average angle Θ, of the powder pile (from horizontal) in relation to the edge of spatula by the equation below and indicate the shape of the powder pile as described in 5.3.13.

$$\Theta = \tan^{-1}[H/X] \qquad (9)$$

where:

H = height of the powder pile on the spatula, mm, and

X = half width of the spatula, mm.

5.13.6 Raise the sliding bushing to the highest point of the pole (at a drop height of 150.0 ± 10.0 mm), then drop it to give only one shock to the spatula.

5.13.7 Wait for 30 s after the shock and then calculate an average angle of the powder on the spatula again as descried in 5.13.5.

5.13.8 Average the mean angle of spatula before and after the shock to give the Carr angle of spatula.

5.13.9 If the slope of the powder pile is irregular in shape, repeat the test three times and obtain an average.

Test J—Measurement of Carr Dispersibility

5.14 The apparatus should be covered or enclosed in a box to prevent ambient air currents from disturbing the measurement and to contain the powder.

5.14.1 Set the Carr dispersibility measuring unit in place as described in 4.3.

5.14.2 Weigh the watch glass.

5.14.3 Position the watch glass concave upwards and centered under the glass tube.

5.14.4 Make sure the container is closed with the shutter cover.

5.14.5 Weigh 10.0 ± 0.01 g of powder and place it into the hopper of the container.

5.14.6 Release the shutter cover horizontally in 1 to 2 s to allow the powder to fall through the glass tube and onto the watch glass.

5.14.7 Weigh the watch glass and the powder on it.

5.14.8 Carr dispersibility is obtained by the following calculation:

$$\text{Carr dispersibility} = (10 \text{ g} - \text{Mass of powder on watch glass})/10 \text{ g} \times 100 \qquad (10)$$

6. Report

6.1 Reports for each test should include the sample name, the sample source, the moisture content of the sample, and the temperature and relative humidity when the tests are performed. In addition, report the following information:

6.1.1 *Test A*—Carr angle of repose should be reported as

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angle in degrees with an indication of the shape of the powder pile.

6.1.2 *Test B*—Carr angle of fall should be reported as angle in degrees with an indication of the shape of the powder pile.

6.1.3 *Test C*—Carr angle of difference should be reported as angle in degrees.

6.1.4 *Test D*—Carr loose bulk density should be reported in units of g/cm³.

6.1.5 *Test E*—Carr packed bulk density should be reported in units of g/cm³.

6.1.6 *Test F*—Carr compressibility should be reported as a % value.

6.1.7 *Test G*—Carr cohesion should be reported as a % value.

6.1.8 *Test H*—Carr uniformity should be reported as a dimensionless number.

6.1.9 *Test I*—Carr angle of spatula should be reported as angle in degrees. In addition, the average angle and the indication of the shape of the powder pile on the spatula before and after the mechanical shock should be included in the report.

6.1.10 *Test J*—Carr dispersibility should be reported as a % value.

7. Precision and Bias

7.1 *Precision*—Data are being evaluated to determine the precision of this test method.

7.2 *Bias*—No accepted reference value exists for this test method; therefore, bias cannot be determined.

8. Keywords

8.1 angle of difference; angle of fall; angle of repose; angle of spatula; Carr index; Carr indices; Carr procedures; cohesion; compressibility; dispersibility; dynamic bulk density; loose bulk density; packed bulk density; uniformity

ASTM Designation: D 854 – 98

Standard Test Method for Specific Gravity of Soils[1]

This standard is issued under the fixed designation D 854; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope *

1.1 This test method covers the determination of the specific gravity of soils that pass the 4.75-mm (No. 4) sieve, by means of a pycnometer. When the soil contains particles larger than the 4.75-mm sieve, Test Method C 127 shall be used for the material retained on the 4.75-mm sieve and this test method shall be used for the material passing the 4.75-mm sieve.

1.1.1 Two procedures for performing the specific gravity are provided as follows:

1.1.1.1 *Method A*—Procedure for Oven-Dry Specimens, described in 9.1.

1.1.1.2 *Method B*—Procedure for Moist Specimens, described in 9.2. The procedure to be used shall be specified by the requesting authority. For specimens of organic soils and highly plastic, fine-grained soils, Procedure B shall be the preferred method.

1.2 When the specific gravity value is to be used in calculations in connection with the hydrometer portion of Test Method D 422, it is intended that the specific gravity test be made on that portion of the sample which passes the 2.00-mm (No. 10) sieve.

1.3 The values stated in acceptable metric units are to be regarded as standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 127 Test Method for Specific Gravity and Absorption of Coarse Aggregate[2]

C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials[2]

D 422 Test Method for Particle-Size Analysis of Soils[3]

D 653 Terminology Relating to Soil, Rock, and Contained Fluids[3]

D 2487 Classification of Soils for Engineering Purposes (Unified Soil Classification System)[3]

D 3740 Practice for Minimum Requirements for Agencies Engaged in the Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction[3]

D 4753 Specification for Evaluating, Selecting, and Specifying Balances and Scales for Use in Testing Soil, Rock, and related Construction Materials[3]

E 1 Specification for ASTM Thermometers[4]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[5]

E 12 Terminology Relating to Density and Specific Gravity of Solids, Liquids, and Gases[6]

2.2 *AASHTO Standards:*[7]

AASHTO Test Method T 100

3. Terminology

3.1 All definitions are in accordance with Terminology D 653 and E 12.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *specific gravity*—the ratio of the mass of a unit volume of a material at a stated temperature to the mass of the same volume of gas-free distilled water at a stated temperature.

4. Significance and Use

4.1 The specific gravity of a soil is used in calculating the phase relationships of soils (that is, the relative volumes of solids to water and air in a given volume of soil).

4.2 The term solid particles is typically assumed to mean naturally occurring mineral particles that are not readily soluble in water. Therefore, the specific gravity of materials containing extraneous matter (such as cement, lime, and the like), water-soluble matter (such as sodium chloride), and soils containing matter with a specific gravity less than one, typically require special treatment or a qualified definition of their specific gravity.

NOTE 1—Notwithstanding the statements on precision and bias contained in this test method; the precision of this test method is dependent on the competence of the personnel preforming it and the stability of the equipment and facilities used. Agencies which meet the criteria of Practice

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[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity and Density Characteristics of Soils.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.

[3] *Annual Book of ASTM Standards*, Vol 04.08.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Annual Book of ASTM Standards*, Vol 14.02.

[6] Discontinued; see *1996 Annual Book of ASTM Standards*, Vol 15.05.

[7] Available from American Association of State Highway and Transportation Officials, 444 N Capital St., NW, Washington, DC 20001.

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*A Summary of Changes section appears at the end of this standard.

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D 3740 are generally considered capable of competent and objective testing. Users of this method are cautioned that compliance with Practice D 3740 does not in itself ensure reliable testing. Reliable testing depends on several factors. Practice D 3740 provides a means of evaluating some of those factors.

5. Apparatus

5.1 *Pycnometer*—The pycnometer shall be one of the following:

5.1.1 *Volumetric Flask*, having a capacity of at least 100 mL.

5.1.2 *Stoppered Bottle*, having a capacity of at least 50 mL. The stopper shall be of the same material, and shall permit the emission of air and surplus water when it is put in place.

NOTE 2—Flask sizes of larger than the specified minimum capacity are recommended. Larger flasks are capable of holding larger specimens and tend to produce better statistical results.

5.2 *Balance*—Meeting the requirements of Specification D 4753 and readable, without estimation, to at least 0.1 % of the specimen mass.

5.3 *Drying Oven*—Thermostatically-controlled oven, capable of maintaining a uniform temperature of 110 ± 5°C (230 ± 9°F) throughout the drying chamber.

5.4 *Thermometer*, capable of measuring the temperature range within which the test is being performed, graduated in a 0.5°C (1.0°F) division scale and meeting the requirements of Specification E 1.

5.5 *Desiccator*—A desiccator cabinet or large desiccator jar of suitable size containing silica gel or anhydrous calcium sulfate.[8]

NOTE 3—It is preferable to use a desiccant that changes color to indicate when it needs reconstitution.

5.6 *Entrapped Air Removal Apparatus*—To remove entrapped air, use one of the following:

5.6.1 *Hot Plate or Bunsen Burner*, capable of maintaining a temperature adequate to boil water.

5.6.2 *Vacuum System*, a vacuum pump or water aspirator, capable of producing a partial vacuum of 100 min or less absolute pressure.

NOTE 4—A partial vacuum of 100 mm Hg absolute pressure is approximately equivalent to a 660 mm (26 in.) Hg reading on vacuum gauge at sea level.

5.7 *Miscellaneous Equipment*, specimen dishes and insulated gloves.

6. Reagents and Materials

6.1 *Purity of Water*—Where distilled water is referred to in this test method, either distilled or demineralized water may be used.

7. Test Specimen

7.1 The test specimen may be oven-dried or moist soil and shall be representative of the total sample. In either case the specimen shall be large enough that its minimum mass in the oven-dried state is in accordance with the following:

Maximum Particle Size (100 % passing)	Standard Sieve Size	Minimum Mass of Test Specimen, g
2 mm	No. 10	20
4.75 mm	No. 4	100

8. Calibration of Pycnometer

8.1 Determine and record the mass of a clean, dry pycnometer, M_f.

8.2 Fill the pycnometer with distilled water to the calibration mark. Visually inspect the pycnometer and its contents to ensure that there are no air bubbles in the distilled water. Determine and record the mass of the pycnometer and water, M_a.

8.3 Insert a thermometer in the water, and determine and record its temperature, T_a, to the nearest 0.5°C (1.0°F).

8.4 From the mass, M_a, determined at the observed temperature, T_a, prepare a table of values of mass, M_a, for a series of temperatures that are likely to prevail when the mass of the pycnometer, soil, and water, M_b, is determined later. These values of M_a can be determined experimentally or may be calculated as follows:

$$M_a \text{ (at } T_x) = [(\text{density of water at } T_x/\text{density of water at } T_a) \times (M_a \text{ (at } T_a) - M_f)] + M_f \qquad (1)$$

where:
M_a = mass of pycnometer and water, g,
M_f = mass of pycnometer, g,
T_a = observed temperature of water, °C, and
T_x = any other desired temperature,° C.

NOTE 5—This test method provides a procedure that is more convenient for laboratories making many determinations with the same pycnometer. It is equally applicable to a single determination. Bringing the pycnometer and contents to some designated temperature when masses M_a and M_b are taken, requires considerable time. It is important that masses M_a and M_b be based on water at the same temperature. Values for the density of water at temperatures from 16.0 to 30.0°C are given in Table 1.

9. Procedure

9.1 *Test Method A—Procedure For Oven-Dried Specimens:*

9.1.1 Dry the specimen to a constant mass in an oven maintained at 110 ± 5°C (230 ± 9°F) (See Note 6) and cool it in a desiccator.

NOTE 6—Drying of certain soils at 110°C (230°F) may bring about loss of water of composition or hydration, and in such cases drying may be done in reduced air pressure or at a lower temperature.

9.1.2 Determine and record the mass of a clean, dry, calibrated pycnometer, M_f. Select a pycnometer of sufficient capacity that the volume filled to the mark will be at least 50 percent greater than the space required to accommodate the test specimen. Place the specimen in the pycnometer. Determine the mass of the specimen and pycnometer, and subtract the mass of the pycnometer, M_f, from this value to determine the mass of the oven-dry specimen, M_o.

9.1.3 Fill the pycnometer with distilled water to a level slightly above that required to cover the soil and soak the specimen for at least 12 h.

NOTE 7—For some soils containing a significant fraction of organic matter, kerosine is a better wetting agent than water and may be used in

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* Anhydrous calcium sulfate is sold under the trade name Drierite.

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TABLE 1 Density of Water and Correction Factor *K* for Various Temperatures

Temperature, °C	Density of Water (g/mL)	Correction Factor *K*
16.0	0.99897	1.0007
16.5	0.99889	1.0007
17.0	0.99880	1.0006
17.5	0.99871	1.0005
18.0	0.99862	1.0004
18.5	0.99853	1.0003
19.0	0.99843	1.0002
19.5	0.99833	1.0001
20.0	0.99823	1.0000
20.5	0.99812	0.9999
21.0	0.99802	0.9998
21.5	0.99791	0.9997
22.0	0.99780	0.9996
22.5	0.99768	0.9995
23.0	0.99757	0.9993
23.5	0.99745	0.9992
24.0	0.99732	0.9991
24.5	0.99720	0.9990
25.0	0.99707	0.9988
25.5	0.99694	0.9987
26.0	0.99681	0.9986
26.5	0.99668	0.9984
27.0	0.99654	0.9983
27.5	0.99640	0.9982
28.0	0.99626	0.9980
28.5	0.99612	0.9979
29.0	0.99597	0.9977
29.5	0.99582	0.9976
30.0	0.99567	0.9974

place of distilled water for oven-dried specimens. If kerosine is used, the entrapped air should only be removed by use of an aspirator. Kerosine is a flammable liquid that must be used with extreme caution.

NOTE 8—Adding distilled water to just cover the soil makes it easier to control boil-over during removal of entrapped air.

9.1.4 Remove the entrapped air by one of the following methods:

9.1.4.1 Boil the specimen gently for at least 10 min while agitating the pycnometer occasionally to assist in the removal of air. Then cool the heated specimen to room temperature.

9.1.4.2 Subject the contents to a vacuum (air pressure not exceeding 100 mm Hg) for at least 30 min (Note 9) either by connecting the pycnometer directly to an aspirator or vacuum pump or by use of a bell jar. While the vacuum is being applied, gently agitate the pycnometer periodically to assist in the removal of air. Some soils boil violently when subjected to reduced air pressure. It will be necessary in those cases to reduce the air pressure at a slower rate or to use a larger flask.

NOTE 9—Specimens with a high plasticity at the natural water content may require 6 to 8 h to remove entrapped air. Specimens with a low plasticity at the natural water content may require 4 to 6 h to remove entrapped air. Oven-dried specimens may require 2 to 4 h to remove entrapped air.

9.1.5 Fill the pycnometer to just below the calibration mark with distilled water at room temperature. Add the distilled water slowly and carefully to avoid the entrapment of air bubbles in the specimen (Note 10). Allow the pycnometer to obtain a uniform water temperature (Note 11).

NOTE 10—To avoid the entrapment of air bubbles, the distilled water can be introduced through a piece of small-diameter flexible tubing with its outlet end kept just below the surface of the distilled water in the pycnometer.

NOTE 11—To obtain a uniform water temperature the pycnometer may be allowed to sit overnight or be placed in a constant temperature bath.

9.1.6 Fill the pycnometer with distilled water at the same temperature to the mark, clean the outside, and dry with a clean, dry cloth. Determine and record the mass of the pycnometer filled with soil and water, M_b.

9.1.7 Insert a thermometer into the water, and determine and record its temperature, T_b, to the nearest 0.5°C (1.0°F).

9.2 *Test Method B—Procedure For Moist Specimens*:

9.2.1 Place the specimen in a calibrated pycnometer.

9.2.1.1 Disperse specimens of clay soils in distilled water before they are placed in the pycnometer, by use of the dispersing equipment specified in Test Method D 422. The minimum volume of slurry that can be prepared by this dispersing equipment is such that a 500-mL (or larger) flask is needed as a pycnometer.

9.2.2 Proceed as described in Sections 9.1.4 and 9.1.7.

9.2.3 Remove the specimen from the pycnometer. Dry the specimen to a constant mass in a suitable container in an oven maintained at 110 ± 5°C (230 ± 9°F) (See Note 6). Cool the specimen in a desiccator.

9.2.4 Determine and record the mass of the oven-dried soil, M_o.

10. Calculation

10.1 Calculate the specific gravity of the soil, *G*, to the nearest 0.01, based on water at a temperature (T_b) as follows:

$$G \text{ at } T_b = M_o/[M_o + (M_a - M_b)] \tag{2}$$

where:

M_o = mass of sample of oven-dry soil, g,
M_a = mass of pycnometer filled with water at temperature T_b(Note 12), g,
M_b = mass of pycnometer filled with water and soil at temperature T_b, g,
T_b = temperature of the contents of the pycnometer when mass M_b was determined, °C.

NOTE 12—This value can be obtained from the table of values of M_a, prepared in accordance with 8.4, for the temperatures prevailing when mass M_b was determined,° C.

NOTE 13—The equation shown in 10.1 is for computing the specific gravity of the soil tested in water. When kerosine is used, the Eq must be adjusted by multiplying the result by the specific gravity of kerosine at T_b and dividing it by the density of water at T_b.

10.2 Calculate the weighted average specific gravity for soils containing particles both larger and smaller than the 4.75-mm sieve using the following equation:

$$G_{avg} = \frac{1}{\frac{R_1}{100G_1} + \frac{P_1}{100G_2}} \tag{3}$$

where:

G_{avg} = weighted average specific gravity of soils composed of particles larger and smaller than the 4.75-mm sieve,
R_1 = percent of soil particles retained on 4.75-mm sieve,
P_1 = percent of soil particles passing the 4.75-mm sieve,

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G_1 = apparent specific gravity of soil particles retained on the 4.75-mm sieve as determined by Test Method C 127, and

G_2 = specific gravity of soil particles passing the 4.75-mm sieve as determined by this test method.

10.3 Unless otherwise required, specific gravity (G) values reported shall be based on water at 20°C. Calculate the value based on water at 20°C from the value based on water at the observed temperature T_b, as follows:

$$G \text{ at } 20°C = K \times (G \text{ at } T_b) \tag{4}$$

where:

K = a number found by dividing the density of water at temperature T_b by the density of water at 20°C. Values for the range of temperatures are given in Table 1.

10.4 In some cases, it is desired to report the specific gravity value based on water at a different temperature. In these cases, the specific gravity value, based on any temperature T_x, may be calculated as follows:

$$G \text{ at } T_x = \frac{G \text{ at } 20°C}{K} \tag{5}$$

11. Report

11.1 The report (data sheet) shall include the following:

11.1.1 Identification of the sample (material) being tested, such as boring number, sample number, test number, etc.

11.1.2 Specific gravity at 20°C to the nearest 0.01. Test procedure used (A or B).

11.1.3 Maximum particle size of the test specimen.

11.1.4 Specific gravity to the nearest 0.01 at a specified temperature other than 20°C, if applicable.

11.1.5 Type of fluid used, if other than distilled water.

11.1.6 When any portion of the original sample of soil is eliminated in the preparation of the test specimen, the portion on which the test has been made shall be reported.

12. Precision and Bias

12.1 *Precision*—Criteria for judging the acceptability of specific gravity test results obtained by this test method on material passing the 4.75-mm sieve are given as follows:

12.2 *Statement of Precision*—Criteria for judging the acceptability of specific gravity test results obtained by this test method on material passing the 4.75 (No. 4) or 2.00 mm (No. 10) sieve are given in Table 2. The estimates of precision for material passing the 2.00 mm sieve are based on results from the AASHTO Materials Reference Laboratory (AMRL) Proficiency Sample Program, of testing conducted on material passing the 2.00 (No. 10) sieve by this test method and AASHTO Test Method T 100.

12.3 *Bias*—There is no acceptable reference value for this test method; therefore, bias cannot be determined.

13. Keywords

13.1 soil; specific gravity

TABLE 2 Table of Precision Estimates[A]

Material and Type Index	Standard Deviation[B]		Acceptable Range of Two Results[C]	
	Passing 4.75 mm (No. 4)	Passing 2.00 mm (No. 10)	Passing 4.75 mm (No. 4)	Passing 2.00 mm (No. 10)
Single-operator precision:				
Cohesive soils	0.021	0.019	0.06	0.06
Noncohesive soils	D	D	D	D
Multilaboratory precision:				
Cohesive soils	0.056	0.041	0.16	0.12
Noncohesive soils	D	D	D	D

[A]The figures given in Columns 2 and 3 are the standard deviations that have been found to be appropriate for the materials described in Column 1. The figures given in Columns 4 and 5 are the limits that should not be exceeded by the difference between the two properly conducted tests.

[B]These numbers represent, respectively, the (1S) limits as described in Practice C 670.

[C]These numbers represent the d2s limits as described in Practice C 670.

[D]Criteria for assigning standard deviation values for noncohesive soils are not available at the present time.

SUMMARY OF CHANGES

Committee D-18 has identified the location of selected changes to this standard since the last issue (D 854–92[ε1]) that may impact the use of this test method.

(*1*) Added Note 1 referencing Practice D 3740. Subsequently renumbered notes affected by adding Note 1.

Designation: D 422 – 63 (Reapproved 1998)

Standard Test Method for
Particle-Size Analysis of Soils[1]

This standard is issued under the fixed designation D 422; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 μm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 μm is determined by a sedimentation process, using a hydrometer to secure the necessary data (Note 1 and Note 2).

NOTE 1—Separation may be made on the No. 4 (4.75-mm), No. 40 (425-μm), or No. 200 (75-μm) sieve instead of the No. 10. For whatever sieve used, the size shall be indicated in the report.

NOTE 2—Two types of dispersion devices are provided: (1) a high-speed mechanical stirrer, and (2) air dispersion. Extensive investigations indicate that air-dispersion devices produce a more positive dispersion of plastic soils below the 20-μm size and appreciably less degradation on all sizes when used with sandy soils. Because of the definite advantages favoring air dispersion, its use is recommended. The results from the two types of devices differ in magnitude, depending upon soil type, leading to marked differences in particle size distribution, especially for sizes finer than 20 μm.

2. Referenced Documents

2.1 *ASTM Standards:*

D 421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

E 100 Specification for ASTM Hydrometers[4]

3. Apparatus

3.1 *Balances*—A balance sensitive to 0.01 g for weighing the material passing a No. 10 (2.00-mm) sieve, and a balance sensitive to 0.1 % of the mass of the sample to be weighed for weighing the material retained on a No. 10 sieve.

3.2 *Stirring Apparatus*—Either apparatus A or B may be used.

3.2.1 Apparatus A shall consist of a mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 rpm without load. The shaft shall be equipped with a replaceable stirring paddle made of metal, plastic, or hard rubber, as shown in Fig. 1. The shaft shall be of such length that the stirring paddle will operate not less than ¾ in. (19.0 mm) nor more than 1½ in. (38.1 mm) above the bottom of the dispersion cup. A special dispersion cup conforming to either of the designs shown in Fig. 2 shall be provided to hold the sample while it is being dispersed.

3.2.2 Apparatus B shall consist of an air-jet dispersion cup[5] (Note 3) conforming to the general details shown in Fig. 3 (Note 4 and Note 5).

NOTE 3—The amount of air required by an air-jet dispersion cup is of the order of 2 ft³/min; some small air compressors are not capable of supplying sufficient air to operate a cup.

NOTE 4—Another air-type dispersion device, known as a dispersion tube, developed by Chu and Davidson at Iowa State College, has been shown to give results equivalent to those secured by the air-jet dispersion cups. When it is used, soaking of the sample can be done in the sedimentation cylinder, thus eliminating the need for transferring the slurry. When the air-dispersion tube is used, it shall be so indicated in the report.

NOTE 5—Water may condense in air lines when not in use. This water must be removed, either by using a water trap on the air line, or by blowing the water out of the line before using any of the air for dispersion purposes.

3.3 *Hydrometer*—An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per litre of suspension, and conforming to the requirements for hydrometers 151H or 152H in Specifications E 100. Dimensions of both hydrometers are the same, the scale being the only item of difference.

3.4 *Sedimentation Cylinder*—A glass cylinder essentially 18 in. (457 mm) in height and 2½ in. (63.5 mm) in diameter, and marked for a volume of 1000 mL. The inside diameter shall be such that the 1000-mL mark is 36 ± 2 cm from the bottom on the inside.

3.5 *Thermometer*—A thermometer accurate to 1°F (0.5°C).

3.6 *Sieves*—A series of sieves, of square-mesh woven-wire cloth, conforming to the requirements of Specification E 11. A full set of sieves includes the following (Note 6):

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[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

Current edition approved Nov. 21, 1963. Originally published 1935. Replaces D 422 – 62.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] Detailed working drawings for this cup are available at a nominal cost from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428. Order Adjunct No. ADJD0422.

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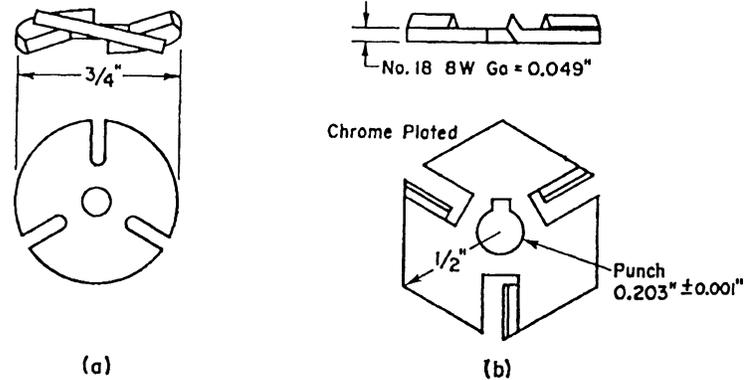
Metric Equivalents

in.	0.001	0.049	0.203	½	¾
mm	0.03	1.24	5.16	12.7	19.0

FIG. 1 Detail of Stirring Paddles
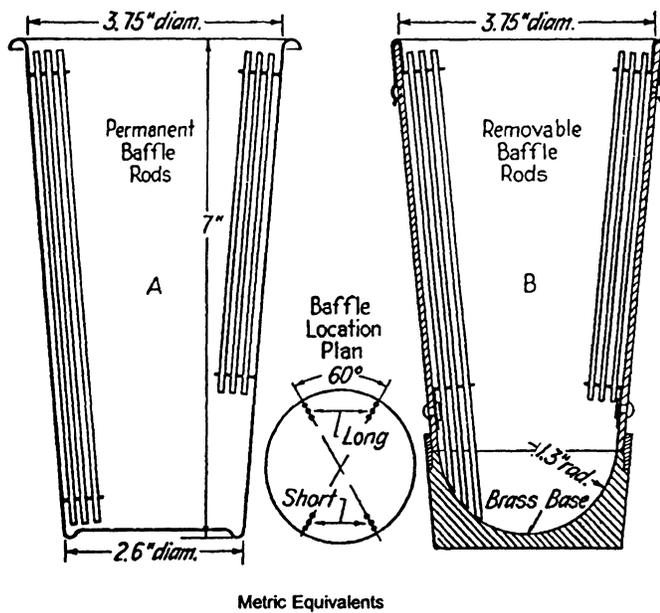
Metric Equivalents

in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

3-in. (75-mm)	No. 10 (2.00-mm)
2-in. (50-mm)	No. 20 (850-μm)
1½-in. (37.5-mm)	No. 40 (425-μm)
1-in. (25.0-mm)	No. 60 (250-μm)
¾-in. (19.0-mm)	No. 140 (106-μm)
⅜-in. (9.5-mm)	No. 200 (75-μm)
No. 4 (4.75-mm)	

Note 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 17, may be used if desired. This set consists of the following sieves:

3-in. (75-mm)	No. 16 (1.18-mm)
1½-in. (37.5-mm)	No. 30 (600-μm)
¾-in. (19.0-mm)	No. 50 (300-μm)
⅜-in. (9.5-mm)	No. 100 (150-μm)
No. 4 (4.75-mm)	No. 200 (75-μm)
No. 8 (2.36-mm)	

3.7 *Water Bath or Constant-Temperature Room*—A water bath or constant-temperature room for maintaining the soil suspension at a constant temperature during the hydrometer analysis. A satisfactory water tank is an insulated tank that maintains the temperature of the suspension at a convenient constant temperature at or near 68°F (20°C). Such a device is illustrated in Fig. 4. In cases where the work is performed in a room at an automatically controlled constant temperature, the water bath is not necessary.

3.8 *Beaker*—A beaker of 250-mL capacity.

3.9 *Timing Device*—A watch or clock with a second hand.

4. Dispersing Agent

4.1 A solution of sodium hexametaphosphate (sometimes called sodium metaphosphate) shall be used in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate/litre of solution (Note 7).

NOTE 7—Solutions of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

4.2 All water used shall be either distilled or demineralized water. The water for a hydrometer test shall be brought to the temperature that is expected to prevail during the hydrometer test. For example, if the sedimentation cylinder is to be placed in the water bath, the distilled or demineralized water to be used shall be brought to the temperature of the controlled water bath; or, if the sedimentation cylinder is used in a room with controlled temperature, the water for the test shall be at the temperature of the room. The basic temperature for the hydrometer test is 68°F (20°C). Small variations of temperature do not introduce differences that are of practical significance and do not prevent the use of corrections derived as prescribed.

5. Test Sample

5.1 Prepare the test sample for mechanical analysis as outlined in Practice D 421. During the preparation procedure

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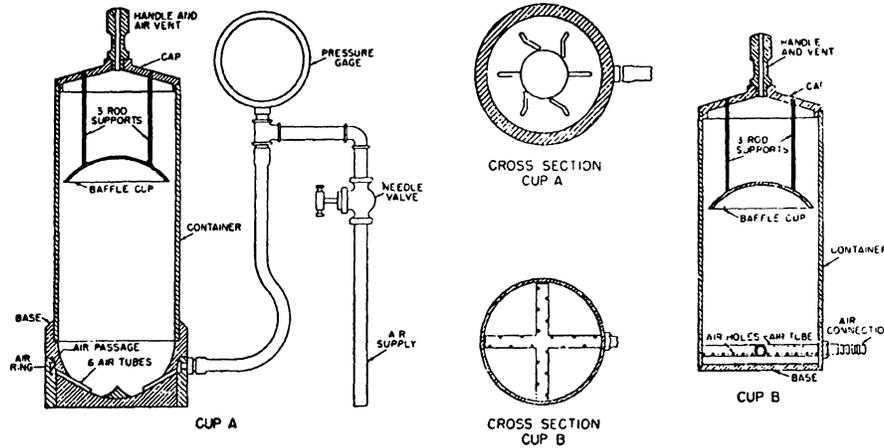
FIG. 3 Air-Jet Dispersion Cups of Apparatus B
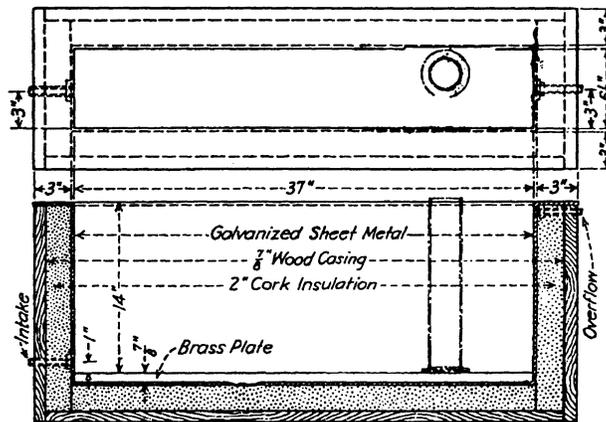
Metric Equivalents						
in.	7/8	1	3	6¼	14	37
mm	22.2	25.4	76.2	158.2	356	940

FIG. 4 Insulated Water Bath

the sample is divided into two portions. One portion contains only particles retained on the No. 10 (2.00-mm) sieve while the other portion contains only particles passing the No. 10 sieve. The mass of air-dried soil selected for purpose of tests, as prescribed in Practice D 421, shall be sufficient to yield quantities for mechanical analysis as follows:

5.1.1 The size of the portion retained on the No. 10 sieve shall depend on the maximum size of particle, according to the following schedule:

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
⅜ (9.5)	500
¾ (19.0)	1000
1 (25.4)	2000
1½ (38.1)	3000
2 (50.8)	4000
3 (76.2)	5000

5.1.2 The size of the portion passing the No. 10 sieve shall be approximately 115 g for sandy soils and approximately 65 g for silt and clay soils.

5.2 Provision is made in Section 5 of Practice D 421 for weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages retained and passing the No. 10 sieve can be calculated in accordance with 12.1.

NOTE 8—A check on the mass values and the thoroughness of pulverization of the clods may be secured by weighing the portion passing the No. 10 sieve and adding this value to the mass of the washed and oven-dried portion retained on the No. 10 sieve.

SIEVE ANALYSIS OF PORTION RETAINED ON NO. 10 (2.00-mm) SIEVE

6. Procedure

6.1 Separate the portion retained on the No. 10 (2.00-mm) sieve into a series of fractions using the 3-in. (75-mm), 2-in.

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(50-mm), 1½-in. (37.5-mm), 1-in. (25.0-mm), ¾-in. (19.0-mm), ⅜-in. (9.5-mm), No. 4 (4.75-mm), and No. 10 sieves, or as many as may be needed depending on the sample, or upon the specifications for the material under test.

6.2 Conduct the sieving operation by means of a lateral and vertical motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve. In no case turn or manipulate fragments in the sample through the sieve by hand. Continue sieving until not more than 1 mass % of the residue on a sieve passes that sieve during 1 min of sieving. When mechanical sieving is used, test the thoroughness of sieving by using the hand method of sieving as described above.

6.3 Determine the mass of each fraction on a balance conforming to the requirements of 3.1. At the end of weighing, the sum of the masses retained on all the sieves used should equal closely the original mass of the quantity sieved.

HYDROMETER AND SIEVE ANALYSIS OF PORTION PASSING THE NO. 10 (2.00-mm) SIEVE

7. Determination of Composite Correction for Hydrometer Reading

7.1 Equations for percentages of soil remaining in suspension, as given in 14.3, are based on the use of distilled or demineralized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

7.1.1 Both soil hydrometers are calibrated at 68°F (20°C), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.

7.1.2 Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

7.1.3 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

7.2 For convenience, a graph or table of composite corrections for a series of 1° temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

7.3 Prepare 1000 mL of liquid composed of distilled or demineralized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cylinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus

formed on the stem. For hydrometer 151H the composite correction is the difference between this reading and one; for hydrometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

8. Hygroscopic Moisture

8.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion of from 10 to 15 g in a small metal or glass container, dry the sample to a constant mass in an oven at 230 ± 9°F (110 ± 5°C), and weigh again. Record the masses.

9. Dispersion of Soil Sample

9.1 When the soil is mostly of the clay and silt sizes, weigh out a sample of air-dry soil of approximately 50 g. When the soil is mostly sand the sample should be approximately 100 g.

9.2 Place the sample in the 250-mL beaker and cover with 125 mL of sodium hexametaphosphate solution (40 g/L). Stir until the soil is thoroughly wetted. Allow to soak for at least 16 h.

9.3 At the end of the soaking period, disperse the sample further, using either stirring apparatus A or B. If stirring apparatus A is used, transfer the soil-water slurry from the beaker into the special dispersion cup shown in Fig. 2, washing any residue from the beaker into the cup with distilled or demineralized water (Note 9). Add distilled or demineralized water, if necessary, so that the cup is more than half full. Stir for a period of 1 min.

Note 9—A large size syringe is a convenient device for handling the water in the washing operation. Other devices include the wash-water bottle and a hose with nozzle connected to a pressurized distilled water tank.

9.4 If stirring apparatus B (Fig. 3) is used, remove the cover cap and connect the cup to a compressed air supply by means of a rubber hose. A air gage must be on the line between the cup and the control valve. Open the control valve so that the gage indicates 1 psi (7 kPa) pressure (Note 10). Transfer the soil-water slurry from the beaker to the air-jet dispersion cup by washing with distilled or demineralized water. Add distilled or demineralized water, if necessary, so that the total volume in the cup is 250 mL, but no more.

Note 10—The initial air pressure of 1 psi is required to prevent the soil-water mixture from entering the air-jet chamber when the mixture is transferred to the dispersion cup.

9.5 Place the cover cap on the cup and open the air control valve until the gage pressure is 20 psi (140 kPa). Disperse the soil according to the following schedule:

Plasticity Index	Dispersion Period, min
Under 5	5
6 to 20	10
Over 20	15

Soils containing large percentages of mica need be dispersed for only 1 min. After the dispersion period, reduce the gage pressure to 1 psi preparatory to transfer of soil-water slurry to the sedimentation cylinder.

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10. Hydrometer Test

10.1 Immediately after dispersion, transfer the soil-water slurry to the glass sedimentation cylinder, and add distilled or demineralized water until the total volume is 1000 mL.

10.2 Using the palm of the hand over the open end of the cylinder (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min to complete the agitation of the slurry (Note 11). At the end of 1 min set the cylinder in a convenient location and take hydrometer readings at the following intervals of time (measured from the beginning of sedimentation), or as many as may be needed, depending on the sample or the specification for the material under test: 2, 5, 15, 30, 60, 250, and 1440 min. If the controlled water bath is used, the sedimentation cylinder should be placed in the bath between the 2- and 5-min readings.

NOTE 11—The number of turns during this minute should be approximately 60, counting the turn upside down and back as two turns. Any soil remaining in the bottom of the cylinder during the first few turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

10.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20 to 25 s before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 12—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

10.4 After each reading, take the temperature of the suspension by inserting the thermometer into the suspension.

11. Sieve Analysis

11.1 After taking the final hydrometer reading, transfer the suspension to a No. 200 (75-μm) sieve and wash with tap water until the wash water is clear. Transfer the material on the No. 200 sieve to a suitable container, dry in an oven at 230 ± 9°F (110 ± 5°C) and make a sieve analysis of the portion retained, using as many sieves as desired, or required for the material, or upon the specification of the material under test.

CALCULATIONS AND REPORT

12. Sieve Analysis Values for the Portion Coarser than the No. 10 (2.00-mm) Sieve

12.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

12.2 To secure the total mass of soil passing the No. 4 (4.75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve. To secure the total mass of soil passing the ⅜-in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the ⅜-in. sieve and retained on the No. 4 sieve. For the remaining sieves, continue the calculations in the same manner.

12.3 To determine the total percentage passing for each sieve, divide the total mass passing (see 12.2) by the total mass of sample and multiply the result by 100.

13. Hygroscopic Moisture Correction Factor

13.1 The hydroscopic moisture correction factor is the ratio between the mass of the oven-dried sample and the air-dry mass before drying. It is a number less than one, except when there is no hygroscopic moisture.

14. Percentages of Soil in Suspension

14.1 Calculate the oven-dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass by the hygroscopic moisture correction factor.

14.2 Calculate the mass of a total sample represented by the mass of soil used in the hydrometer test, by dividing the oven-dry mass used by the percentage passing the No. 10 (2.00-mm) sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

14.3 The percentage of soil remaining in suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows (Note 13): For hydrometer 151H:

$$P = [(100\,000/W)\,[m]]P5\,G/(G - G_1)](R - G_1) \qquad (1)$$

NOTE 13—The bracketed portion of the equation for hydrometer 151H is constant for a series of readings and may be calculated first and then multiplied by the portion in the parentheses.

For hydrometer 152H:

$$P = (Ra/W)\,[m]]P5\,100 \qquad (2)$$

where:

a = correction faction to be applied to the reading of hydrometer 152H. (Values shown on the scale are computed using a specific gravity of 2.65. Correction factors are given in Table 1),

P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension,

R = hydrometer reading with composite correction applied (Section 7),

W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed (see 14.2), g,

G = specific gravity of the soil particles, and

G_1 = specific gravity of the liquid in which soil particles are suspended. Use numerical value of one in both instances in the equation. In the first instance any possible variation produces no significant effect, and in the second instance, the composite correction for R is based on a value of one for G_1.

15. Diameter of Soil Particles

15.1 The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading shall be calculated according to Stokes' law (Note 14), on the basis that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at

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TABLE 1 Values of Correction Factor, α, for Different Specific Gravities of Soil Particles[A]	
Specific Gravity	Correction Factor[A]
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

[A]For use in equation for percentage of soil remaining in suspension when using Hydrometer 152H.

which the hydrometer is measuring the density of the suspension. According to Stokes' law: see Table 2

$$D = \sqrt{[30n/980(G - G_1)]}\,[m[]]P5\,L/T \qquad (3)$$

where:
D = diameter of particle, mm,
n = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),
L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings. This distance is known as effective depth (see Table 2)),
T = interval of time from beginning of sedimentation to the taking of the reading, min,
G = specific gravity of soil particles, and
G_1 = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

NOTE 14—Since Stokes' law considers the terminal velocity of a single sphere falling in an infinity of liquid, the sizes calculated represent the diameter of spheres that would fall at the same rate as the soil particles.

15.2 For convenience in calculations the above equation may be written as follows: see Table 3

$$D = K\sqrt{L/T} \qquad (4)$$

where:
K = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of K for a range of temperatures and specific gravities are given in Table 3. The value of K does not change for a series of readings constituting a test, while values of L and T do vary.

15.3 Values of D may be computed with sufficient accuracy, using an ordinary 10-in. slide rule.

NOTE 15—The value of L is divided by T using the A- and B-scales, the square root being indicated on the D-scale. Without ascertaining the value of the square root it may be multiplied by K, using either the C- or CI-scale.

16. Sieve Analysis Values for Portion Finer than No. 10 (2.00-mm) Sieve

16.1 Calculation of percentages passing the various sieves

TABLE 2 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes[A]

Hydrometer 151H			Hydrometer 152H		
Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
1.000	16.3	0	16.3	31	11.2
1.001	16.0	1	16.1	32	11.1
1.002	15.8	2	16.0	33	10.9
1.003	15.5	3	15.8	34	10.7
1.004	15.2	4	15.6	35	10.6
1.005	15.0	5	15.5		
1.006	14.7	6	15.3	36	10.4
1.007	14.4	7	15.2	37	10.2
1.008	14.2	8	15.0	38	10.1
1.009	13.9	9	14.8	39	9.9
1.010	13.7	10	14.7	40	9.7
1.011	13.4	11	14.5	41	9.6
1.012	13.1	12	14.3	42	9.4
1.013	12.9	13	14.2	43	9.2
1.014	12.6	14	14.0	44	9.1
1.015	12.3	15	13.8	45	8.9
1.016	12.1	16	13.7	46	8.8
1.017	11.8	17	13.5	47	8.6
1.018	11.5	18	13.3	48	8.4
1.019	11.3	19	13.2	49	8.3
1.020	11.0	20	13.0	50	8.1
1.021	10.7	21	12.9	51	7.9
1.022	10.5	22	12.7	52	7.8
1.023	10.2	23	12.5	53	7.6
1.024	10.0	24	12.4	54	7.4
1.025	9.7	25	12.2	55	7.3
1.026	9.4	26	12.0	56	7.1
1.027	9.2	27	11.9	57	7.0
1.028	8.9	28	11.7	58	6.8
1.029	8.6	29	11.5	59	6.6
1.030	8.4	30	11.4	60	6.5
1.031	8.1				
1.032	7.8				
1.033	7.6				
1.034	7.3				
1.035	7.0				
1.036	6.8				
1.037	6.5				
1.038	6.2				

[A]Values of effective depth are calculated from the equation:

$$L = L_1 + 1/2\,[L_2 - (V_B/A)] \qquad (5)$$

where:
L = effective depth, cm,
L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm,
L_2 = overall length of the hydrometer bulb, cm,
V_B = volume of hydrometer bulb, cm³, and
A = cross-sectional area of sedimentation cylinder, cm²

Values used in calculating the values in Table 2 are as follows:
For both hydrometers, 151H and 152H:
L_2 = 14.0 cm
V_B = 67.0 cm³
A = 27.8 cm²

For hydrometer 151H:
L_1 = 10.5 cm for a reading of 1.000
= 2.3 cm for a reading of 1.031

For hydrometer 152H:
L_1 = 10.5 cm for a reading of 0 g/litre
= 2.3 cm for a reading of 50 g/litre

used in sieving the portion of the sample from the hydrometer test involves several steps. The first step is to calculate the mass of the fraction that would have been retained on the No. 10

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TABLE 3 Values of K for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

Temperature, °C	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01510	0.01505	0.01481	0.01457	0.01435	0.01414	0.01394	0.01374	0.01356
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.1323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01255	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149

sieve had it not been removed. This mass is equal to the total percentage retained on the No. 10 sieve (100 minus total percentage passing) times the mass of the total sample represented by the mass of soil used (as calculated in 14.2), and the result divided by 100.

16.2 Calculate next the total mass passing the No. 200 sieve. Add together the fractional masses retained on all the sieves, including the No. 10 sieve, and subtract this sum from the mass of the total sample (as calculated in 14.2).

16.3 Calculate next the total masses passing each of the other sieves, in a manner similar to that given in 12.2.

16.4 Calculate last the total percentages passing by dividing the total mass passing (as calculated in 16.3) by the total mass of sample (as calculated in 14.2), and multiply the result by 100.

17. Graph

17.1 When the hydrometer analysis is performed, a graph of the test results shall be made, plotting the diameters of the particles on a logarithmic scale as the abscissa and the percentages smaller than the corresponding diameters to an arithmetic scale as the ordinate. When the hydrometer analysis is not made on a portion of the soil, the preparation of the graph is optional, since values may be secured directly from tabulated data.

18. Report

18.1 The report shall include the following:

18.1.1 Maximum size of particles,

18.1.2 Percentage passing (or retained on) each sieve, which may be tabulated or presented by plotting on a graph (Note 16),

18.1.3 Description of sand and gravel particles:

18.1.3.1 Shape—rounded or angular,

18.1.3.2 Hardness—hard and durable, soft, or weathered and friable,

18.1.4 Specific gravity, if unusually high or low,

18.1.5 Any difficulty in dispersing the fraction passing the No. 10 (2.00-mm) sieve, indicating any change in type and amount of dispersing agent, and

18.1.6 The dispersion device used and the length of the dispersion period.

NOTE 16—This tabulation of graph represents the gradation of the sample tested. If particles larger than those contained in the sample were removed before testing, the report shall so state giving the amount and maximum size.

18.2 For materials tested for compliance with definite specifications, the fractions called for in such specifications shall be reported. The fractions smaller than the No. 10 sieve shall be read from the graph.

18.3 For materials for which compliance with definite specifications is not indicated and when the soil is composed almost entirely of particles passing the No. 4 (4.75-mm) sieve, the results read from the graph may be reported as follows:

(1) Gravel, passing 3-in. and retained on No. 4 sieve ..... %
(2) Sand, passing No. 4 sieve and retained on No. 200 sieve ..... %
    (a) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve ..... %
    (b) Medium sand, passing No. 10 sieve and retained on No. 40 sieve ..... %
    (c) Fine sand, passing No. 40 sieve and retained on No. 200 sieve ..... %
(3) Silt size, 0.074 to 0.005 mm ..... %
(4) Clay size, smaller than 0.005 mm ..... %
    Colloids, smaller than 0.001 mm ..... %

18.4 For materials for which compliance with definite specifications is not indicated and when the soil contains material retained on the No. 4 sieve sufficient to require a sieve analysis on that portion, the results may be reported as follows (Note 17):

SIEVE ANALYSIS

Sieve Size	Percentage Passing
3-in.	....
2-in.	....
1½-in.	....
1-in.	....
¾-in.	....
⅜-in.	....
No. 4 (4.75-mm)	....
No. 10 (2.00-mm)	....
No. 40 (425-μm)	....
No. 200 (75-μm)	....
HYDROMETER ANALYSIS	
0.074 mm	....
0.005 mm	....
0.001 mm	....

NOTE 17—No. 8 (2.36-mm) and No. 50 (300-μm) sieves may be substituted for No. 10 and No. 40 sieves.

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D 422

19. Keywords

19.1 grain-size; hydrometer analysis; hygroscopic moisture; particle-size; sieve analysis

Appendix B. Copy of Laboratory Notebook Pages

Fernald C of C # 0137/

9/19/00   55 gal drum (W173805) arrived from
Fernald via Fed Ex. Inner container
is 30 gal drum (173689). ~1 mR/hr
on contact. Swipes showed no activity above
background. Drum was transferred to "TSCA (Rm 028)
Room" for storage pending development of
RWP and further definition physical tests.

10/6/00   55 gal drum, Fernald C of C # 01840,
Sample # W173822 arrived from Fernald.
No ID of inner container on chain of custody.
~1 mR/hr on contact. Swipes showed no
activity above background on contact. Drum
was transferred to "TSCA Room for storage   (Rm 028)
pending development of RWP and further
definition of physical tests and sampling plan.

10/9/00   Obtained respirator fit test

10/10/00   Drum W173689 was removed from drum W173805.
There was measureable levels of alpha activity
on the outside of drum the 30 gallon drum
and in the annular space between the two drums.

The lid was taken off of the 30 gallon drum (W173689)
and the contents inspected. Material was grey,
fine dusty powder.

30 gallon drum (W173696) was removed from
the 55 gallon drum (W173822)

10/11/00   Spent day setting up for sampling of the drums.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Steve Heffner	10/12/00

To Page No.

From Page No. _____

① 10/12/00 W 173689 ⟨DRUM 1 Core 1, TOP⟩

C sleeve to w/i 4" of bottom
got ~ 10½ inches in 1st core
~536 g

will take 2nd spl @ same location
to collect slumped in material

Inside is 5" lower than out after
1st sample

got 9" sample

total = 967 g

Inside 7½ lower

3rd core 5½ inches more

total = 1260 g

Inside = 22.5 - 12 = 10.5 inch

W. 173689 ⟨Core 2, TOP (DRUM 1)⟩
sleeve to w/i 4" of bottom
9" from top of sleeve to top of material

went 10½ inches down 1st time

14 - 4½ from top = 9½ collected

530 grams 1st sample

To Page

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Steve Hoffman	10/12/00

10/12/00   2nd core   14 - 5 = 9 inches of spl   _from top_

979 g collected

17 - 8 = 9 inches of lower
inside of the sleeve

3rd core

14 - 8 = 6 inches in 2" sampler

1 295 g collected

19 - 9 = 10 inches lower inside
the sleeve

Removed ½ (12" of material into 55 gal drum

~~∿∿∿∿∿∿∿∿~~

Core i$\cancel{st}$ core   ⟨ CORE 2, BOTTOM, DRUM 1 ⟩
2   14 - 7.5 = 6.5 inches in 2" sampler
Bottom   337 grams
20 - 17 = 3 inches lower inside of
sleeve

2nd core   14 - 7 = 7 inches in 2" sample
663 grams
23 - 17 = 6 inches lower inside sleeve

3rd cor   14 - 10.5 = 3.5 inches in 2" sampler
846 grams
24 - 17 = 7 inches lower inside sleeve

4th core   14 - 11 = 3 inches of 12" sampler
⊕ some material came out of sampler
(tight belt - jerked)

From Page No. ____

10/12/00

total weight = 1065 gram

27.5 - 17 = 10.5 inches

5th core   14 - minimal inches in the 2" sampler
1.120 grams
final meas not taken

Core 1 Bottom DRUM 1

Sleeve place down to ~ 2" from bottom
20" inside & out meas

1st sample 14 - 8 = 6 inches in the 2" sampler
308 grams
23 - 20 = 3 inches

14 - 8 = 6" in sample

2nd sample   617 grams

24 - 20 = 4 inches

3rd sample 14 - 10 = 4 inches
829 grams
26 - 20 = 6 inches

4th sample 14 - 11.5 = 2.5 inches
966 grams
26 - 20 = 6 inches

Note: last 2"s
sound sound like
dull thud =
denser?

Note: No visible
inhomogeneities
in samples

To Page No

Witnessed & Understood by me,	Date	Invented by	Date	
		Recorded by Steve Wooten	10/12/00	

10/12/00

5B Sample
14-12 = 2 inches sample
1057 grams
26½-20 with 6 inches

~~~~~~~~~~

Oct 13, 2000

Sampling Drum W173696 (side) (lid)
(CORE 1, TOP, DRUM 2)
8:15am   Lid W173693 ^30
Sanp

(CORE 1)   Very free flowing. Levels easily

5" down from top of drum ⟹ ~24" of silo 3 material

1st sample   14 - 3½ = 10½ of sample
516 grams
21.5 - 16.5 = 5" lower on inside sleeve

2nd spl   14 - 8 = 6" of sample
796 grams
8" lower on inside sleeve

3rd spl   14 - 9.5 = 4.5" of sample
1030 grams
10" lower on inside sleeve

4th spl   14 - 11 = 3" of sample
1231 grams
11" inches lower inside sleeve

Witnessed & Understood by me,   Date   Invented by   Date

Recorded by Steve Goffney   10/13/00

(CORE 2, TOP, DRUM 2)   W 173 696

1st sample   14 - 2 = 12" sample collected
                  852 grams
                  8" lower on inside of sleeve.

Note: went 14" down (was going 12")
      w/ core sample

2nd sample   butterfly valve did not seat.
                  No spl collected. will Retry

2nd sample   14 - 10.5 = 3.5 inches collected
(2nd try   1157 grams
                  12" lower on inside of sleeve

                  (fluffy - hard to fill top

9:10 am

CORE Holes remain when sleeve is removed
Note: Both Drum appear identical (visually)

(CORE 2 BOTTOM, DRUM 2)   W 1736
          sleeve ~ 3" from bottom
          14 - 2" = 12" sample collected
1st sample   620 grams
                  6" lower
          14 - 9.5 = 4.5 inches collected
2nd Sple   848 grams
                  9" lower

          14 - 11 = 3 inches collected
3rd Sample   997 grams
                  9" lower

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Steve Hoffman	10/13/00

from Page No. ____

10/13/00

4th   14 - 11 = 3 inches collected
                        1137 grams
sample   Δ not measured

W173696   (CORE 1, BOTTOM, DRUM 2)

1st   Steve ^ 4" from bottom
sample   14 - 8 = 6" sample collected
                        282 grams
              7" lower inside ?
2nd   14 - 11.5 = 3.5" collected
sample   409 grams
              8" lower inside
3rd   14 - 10.5 = 4.5" collected
sample   572 grams
              9" lower
4th   14 - 12 = 2" collected
sample   654 grams
              9" lower

W173696 (CORE 1A, BOTTOM, DRUM 2)

              14 - 10 = 4" collected
              198 grams

W173676 (GRAB SAMPLE, BOTTOM BAG, DRUM 2)

              1204 grams

                        SH

To Page No. ____

Witnessed & Understood by me,   Date   Invented by   Date

                                           Recorded by
                                           Steve Huffner   10/13/00

From Page No. _____	

10/16/00   Prepared upstairs lab for physical testing
            ordered some supplies

10/17/00   Prepared upstairs lab for sample splitting.
            Split first sample. Was very dry & dusty.

10/18/00   Blended and split samples. Half of split goes
            to GEL (@ ~50g in one bottle, balance of half in second b
            ~~689-1-T~~ half goes to archive.

            689 - 1 - T
            689 - 1 - B
            689 - 2 - T
            689 - 2 - B

            696 - 1 - T
            696 - 1 - B
            696 - 2 - T
            696 - 2 - B) only 654 grams avail for splitting *
            696 GRAM

            * did not combine with 696-1A-B

            Samples were sent overnight to GEL.

10/ /00   Samples from archive split (~200g each) of
            the 9 samples) sent to Cecil May at WSRC.

10/26/00   10:30 am Started W173696 on drum tumbler
            (in 55 gal drum)

10/27/00   10:30 am took of W173696
            11:00 am started W173689 (in 55 gal drum) on
            drum tumbler

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by *Steve Hoeffner*	10/27/00

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Project No. 226- 2002032
Book No. DE   TITLE Fernald Silo 3 Waste Treatment

From Page No. 56

WSRC-RP-2001-00167, Rev. 0
January 9, 2001
A77

| |

10/30/00 Calculated volume of cup for Carr Indices tester

$$\frac{(51 \text{ mm diam})^2 \times \pi \times 50 \text{mm } H}{50 \; (10 \text{mm}/\text{cm})^3} = 102.1 \text{ cm}^3$$

Filled two 5-gallon buckets of with WI73689
Silo 3 material : W173689 A = 689 A
W173689 B = 689 B

Filled two 5-gallon buckets with WI73696
Silo 3 material: W173696 A = 696 A
W173696 B = 696 B

10/31/00   Began physical testing

~~Samp~~ Loose Bulk Density

Sample	Weight in cup, g	Fritsch Shaker Amplitude	Time
689 A	68.12 g	3.6	> 90 sec
689 A	68.38 g	3.6 → 5.8	90 sec
689 A	69.58 g	5.8	15 sec
689 B	69.17 g	5.6	20 sec
689 B	69.86 g	↓	↓
689 B	69.85 g		
696 A	66.16 g	5.6	20 sec
696 A	68.64 g	↓	↓
696 A	67.47 g		
696 B	68.44 g	5.6	20 sec
696 B	68.36 g	↓	↓
696 B	68.03 g		

Note: material settles, even while moving to the balance

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Steve Hoeffner	10/31/00

To Page No. _____

From Page No. ____

10/31/00 — Compacted bulk density, Tapped until no further decrease in height of column. Did 689 A in triplicate to determine reproducability

Sample	weight in cup, g
689 A	104.92 g
689 A	106.73 g
689 A	106.12 g
689 B	106.31 g
696 A	103.81 g
696 B	104.37 g

11/1/00 — Angle of Repose

Cup dimensions		
ID		75.7 mm
OD		80.8 mm
Height		41.4 mm
Avg. radius		39.2 mm

Heights:	Repose	Fall	Comments
689 A	82	60	slightly concave
	81		
689 B	84	59	v. slightly concave
	83		
696 A	82	60	v. slightly concave
696 B	82	62	v. slightly concave
* 696 B	97	73	v. slightly concave

* using Al cup (ID = 86.9 mm, OD = 88.9 mm, H = 52.6 mm)

To Page No. ____

Witnessed & Understood by me,   Date

Invented by

Recorded by
Steve Hoeffner

Date
11/1/00

Project No. 226-200203

Book No. DE   TITLE Fernald Silo 3 Waste Treatment

From Page No. _____

11/1/00

Angle of Spatula (width = 19.05 mm)

	Before	After
689 A	1.08" (27.4 mm)	0.66" (16.8 mm)
~~689 B~~		
689 B	1.04" (26.4 mm)	0.685" (17.4 mm)
696 A	1.00 (25.4 mm)	0.81 (20.6 mm)
696 B	1.23 (31.2 mm)	0.80 (20.3 mm)
	1.13 (28.7 mm)	0.96 (24.4 mm)

pH of Silo 3 material

	pH	weight
689A	7.80	20.14 g
689 B	7.82	20.39 g
696 A	7.95	19.88 g
696 B	7.86	20.53 g

11/2/00   Cohesion

$$T(s) = 20 + [(1.6 - w)/0.016] = \text{vibration time (sec)}$$
$$w = [(\rho - L)C/100] + L = \text{dynamic bulk density } (g/cm^3)$$
$$C = 100(\rho - L)/\rho = \text{compressibility } (\%)$$
$$L = \text{loose bulk density } (g/cm^3) \approx 0.68 \; g/cm^3$$
$$\rho = \text{packed bulk density } (g/cm^3) \approx 1.05 \; g/cm^3$$

$$C = 100(1.05 - 0.68)/1.05 = 35.24 \%$$
$$w = [(1.05 - 0.68) 35.24/100] + 0.68 = 0.81 \; g/cm^3$$
$$T = 20 + [(1.6 - 0.81)/0.016] = 67.38 \; sec$$

To Page No. _____

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Steve Deffen	11/2/00

TITLE _Fernald Silo 3 Waste Treatment_   Project No. _226-2002032_

Book No. _DE_

From Page No. ____

11/2/00

$$\frac{L+P}{2} = 0.87 \Rightarrow \text{Sieve sizes} \quad 250 \mu m$$
$$150 \mu m$$
$$75 \mu m$$

__Sample 689A__   2.015g start . Held in place by chain clamp

	Weight of sieve + sample	wt of sieve	wt of sample
250 μm	110.336	109.205	1.131 g
150 μm	109.884	109.522	0.362 g
75 μm	104.205	103.761	0.444 g
bottom pan	79.919	79.823	0.096 g
			$\Sigma = 2.033$ g

(689B) Trial 1   1.989 g soil at start

250 μm	110.232	109.205	1.027
150 μm	109.607	109.522	0.085
75 μm	104.634	103.761	0.873
bottom pan	79.848	79.823	0.025
			$\Sigma = 2.010$ g

Sieves held in place by hand

(649B) Trial 2   sieves held in place w/ clamp on top of sieves
(best arrangement - will use this for all samples)
1.998g at start

250 μm	109.653	109.205	0.448
150 μm	109.893	109.522	0.371
75 μm	104.969	103.761	0.208
bottom pan	79.856	79.823	0.033
			2.06

Note: weights of sieves were spot checked to make
sure that tare weight was not changing.

To Page No. ____

Witnessed & Understood by me,   Date   Invented by   Date

Recorded by _Steve Hoffman_   11/2/00

From Page No. ____

11/2/00

696A   initial wt soil = 1.994 g

wt	weight of sieve+sample	weight of sieve	weight of sample
250 μm	110.674	109.205	1.469
150 μm	109.746	109.522	0.224
75 μm	104.059	103.761	0.298
bottom pan	79.823	79.823	0
		Σ =	1.991 g

696B   initial wt = 1.994 g

250 μm	110.291	109.205	1.086
150 μm	109.700	109.522	0.178
75 μm	104.495	103.761	0.734
bottom pan	79.830	79.823	0.007
		Σ =	2.005

689A Trial 2 (repeated using clamp on top of sieves)
initial weight = 2.034 g

20-250 μm	110.151	109.205	0.946
150 μm	110.340	109.522	0.818
75 μm	104.032	103.761	0.271
bottom pan	79.832	79.823	0.009
		Σ =	2.044

689B Trial 3 (repeated since previous value seemed unusually low)
initial weight = 2.044 g

250 μm	110.136	109.205	0.931
150 μm	109.815	109.522	0.293
75 μm	104.592	103.761	0.831
bottom pan	79.845	79.823	0.022
		Σ =	2.077

To Page No. ____

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Steve Hoffman	11/2/00

TITLE _Fernald Silo 3 Waste Treatment_

From Page No. ____

11/2/00
1:30 pm   (Water Leach) (50g diluted to 1000mL with DI water, stir overnight (24 hrs) at room temp. Filter. Dry solids. Send solids and liquids to GEL for analysis.

689 A	50.057 − 0.025g = 50.032g solid
689 B	49.556 − 0.000g = 49.556g solid
696 A	49.943 − 0.000g = 49.943g solid
696 B	49.941 − 0.000g = 49.941g solid

(% Moisture)
2:00 pm

	689A	689 B	696 A	696 B
wt. spl + crucible	182.245	187.440	181.734	173.996
wt. crucible	161.705	163.770	161.569	154.642
wt. sample	20.540g	23.67g	20.165g	19.354g

11/3/00   12:00 pm

	689A	689 B	696 A	696 B
wt. spl + crucible	181.782	186.813	181.119	173.144
wt. crucible	161.705	163.770	161.569	154.642
wt. sample	20.077	23.043	19.550	18.502

(Water Leach)

	689A	689 B	696 A	696 B
glass + f. paper + solid	182.688	122.955	125.655	268.656
watchglass	155.488	95.419	96.790	240.303
filter paper	0.872	0.841	0.837	0.871
wt dry solid				

Note: Solid spls on filter were rinsed out of erlynmeyer w minimula DI water (a few mL)
Prepared water and solid samples from leachate for shipment to GEL.

11/9/00   Shipped solid and liquid samples from leachate study to GEL

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Steve Hoeffner	11/9/00

To Page No. ____

From Page No. ____

11/15/00 Set up for corrosivity test

11/16/00 Mixed 1000 g of 689 A + B ; 696 A + B
w/ 1 liter of deionized water (total of
9:00a 4 samples). Mixed (shook) for 5 minutes.
Let set until most of solids ~~there~~ settled
out.

Length coupon		grams	vol leachate	wt coupon (I)	wt coupon (F)
1.490	686 A	1001.82	645	5.5591	5.5559
1.485	686 B	994.61	520	5.4537	5.4518
1.450	696 A	1003.00	510	5.3629	5.3618
1.485	696 B	1000.53	540	5.4467	5.4453

Coupon dimensions

0.495" wide

$\phi = 0.139$

0.99

2.995" long

0.060" thick

Temp ranged from 48 → 62 °C

Started 1 hour leach 49.745g 689A dilute
to 1 liter of D.I. water

(initial) % moisture   Residue:
spl + cruc. 185.358
cruc. 161.679   watchglass + paper + sample 269.251
spl 23.679   watchglass 240.308
                             filter paper 1.530
(dried)   dry sample
spl + cruc 184.700
cruc. 161.679
spl

To Page No. 74

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Steve Hoffman	11/16/00

From Page No. 72

12/14/00   Began Relative Humidity Test

Weighed nominal 40 grams of silo 3 material
into 3 inch 325 mesh brass sieves. Weighed
4 samples for each Humidity:

	$CaCl_2$ (31% H)	$Ca(NO_3)_2$ (57% H)	$NH_4Cl/$ KNO_3 (71.2% H)	Na_2SO_3 (95% H)
pan+spl	151.517	144.950	156.724	161.667
pan	112.436	110.954	112.099	116.285

3:00 pm Placed in ~~bu~~ 3 gallon buckets containing saturated salt solutions

12/15/00

pan+spl	151.964	146.089	158.913	165.428

12:30 pm

12/18, 7:30am	152.127	147.362	162.470	171.969
12/21, 3:25pm	152.235	147.902	164.317	176.754
1/4 2:00 pm	152.424	149.197	167.696	186.043

12/21 observations

31% H — Sample dry & free flowing

51% H — feels "set up" just a little. Minor clumping but overall is free flowing

71.2% H — Resist ~~some~~ penetration. Slightly more clumping

95% H — clumps up readily. Resists penetration. cakes up

To Page No.

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by Steve Hoffman	12/21/00

12/21 continued
95% H continued. Sample is noticeably darker than
        the other 3.

Placed ᵛlead weights on 51, 71.2, and 95% H samples

1/4/01 31% H - ʷ/ᵒ weight sample is dry + free flowing.

     51% H ʷ/ weight - clumpy. Like dirt clods

     71.2% H ʷ/ weight - silo 3 material stuck to/
     clumped to the bottom of the puck. Clumpy.
     Like dirt clods. Material is darker in color
     than 31 or 51. (both of which have about the
     same color)

     95% H ʷ/ weight - easy to penetrate. No
     clumping to bottom of puck. Consistency similar
     to a dry paste.

SJ+   1/5/01

Witnessed & Understood by me,	Date	Invented by	Date
		Recorded by *Steve Hoeffner*	1/5/01

Appendix C. Raw Data and Calculations for Results in Table 1

Densities

Cup

	Inches	cm
radius	1	2.54
height	1.94	4.9276
Volume	99.87399 cm3	

Loose Bulk Density

Sample	Trail	Weight grams	Density g/cm3	Avg Density g/cm3
W173689A	1	68.12	0.682059	
	2	68.38	0.684663	
	3	69.58	0.696678	0.688
W173689B	1	69.17	0.692573	
	2	69.86	0.699481	
	3	69.85	0.699381	0.697
W173696A	1	66.16	0.662435	
	2	68.64	0.687266	
	3	67.47	0.675551	0.675
W173696B	1	68.44	0.685264	
	2	68.36	0.684463	
	3	68.03	0.681158	0.684
			Overall Average=	0.686

Compacted Bulk Density

Sample	Trail	Weight grams	Density g/cm3	Avg Density g/cm3
W173689A	1	104.92	1.050524	
	2	106.73	1.068647	
	3	106.12	1.062539	1.061
W173689B		106.31	1.064441	1.064
W173696A		103.81	1.03941	1.039
W173696B		104.37	1.045017	1.045
			Overall Average=	1.052

Angles of Repose, Fall and Withdrawal

Angle of Repose = tan(-1)(H/R) where
H = height of powder pile (mm)
R = Radius of circular plateform (mm)

		Inside	Outside	Avg
Glass dish	H			1.63 Inches
				41.402 mm
	D		3.18	Inches
		2.98	80.772	mm
		75.692	78.232	mm
	R	37.846	40.386	39.116 mm
Al cup	R(2)	43.45	44.45	43.95
	H(2)	52.6		87.9

Angle of Repose

Sample	Trail	Total Height, mm	Peak Height, mm	H/R	tan-1(H/R)	Avg
W173689A	1	82	40.598	1.0378873	46.07	
	2	81	39.598	1.0123223	45.35	45.71
W173689A	1	84	42.598	1.0890173	47.44	
	2	83	41.598	1.0634523	46.76	47.1
W173696A	1	82	40.598	1.0378873	46.07	
W173696A (Al cup)	1	82	40.598	1.0378873	46.07	
	2	97	44.4	1.0102389	45.29	45.68

Angle of Fall (Angle of slump)

Total Height, mm	Height, mm	H/R	tan-1(H/R)
60	18.598	0.475458	25.43
59	17.598	0.449893	24.22
60	18.598	0.475458	25.43
62	20.598	0.526588	27.77
73	20.4	0.464164	24.9
			26.335

Angle of Spatula (Angle of Withdrawal)

Width of spatula = 19.05 mm 0.02 inches
Thickness of spatula = 0.508 mm
X = 9.525   X= 0.508 mm

Sample	Height of spatula Inches Before	After	mm Before	After	Before less spatula thickness	After less spatula thickness	HX Before	After	tan-1(H/X) Before	After	Average
W173689A	1.08	0.66	27.432	16.764	26.924	16.256	2.826667	1.706667	70.518	59.632	65.075
W173689A	1.04	0.685	26.416	17.399	25.908	16.891	2.72	1.773333	69.814	60.5809	65.19745
W173696A	1	0.81	25.4	20.574	24.892	20.066	2.613333	2.106667	64.607	64.607	64.607
W173696A	1.23	0.8	31.242	20.32	30.734	19.812	3.226667	2.08	72.781	64.323	68.552
	1.13	0.96	28.702	24.384	28.194	23.876	2.96	2.506667	71.333	68.251	69.792

69.172

Cohesion

Weight

W173689A Trial 1

	Sieve + Sample	Sieve	Sample	Cohesion	
250 um	110.336	109.205	1.131	56.55	C1
150 um	109.884	109.522	0.362	10.86	C2
75 um	104.205	103.761	0.444	4.44	C3
Pan	79.919	79.823	0.096		
		Sum	2.033	71.85	Cohesion

Cohesion

Amt added 2.015

Sieves held in place by chain and clamp

W173689B Trial 1

	Sieve + Sample	Sieve	Sample	Cohesion	
250 um	110.232	109.205	1.027	51.35	C1
150 um	109.607	109.522	0.085	2.55	C2
75 um	104.634	103.761	0.873	8.73	C3
Pan	79.848	79.823	0.025		
		Sum	2.01	62.63	Cohesion
		Amt added	1.989		

Sieves held in place by hand

W173696A

	Sieve + Sample	Sieve	Sample	Cohesion	
250 um	110.674	109.205	1.469	73.45	C1
150 um	109.746	109.522	0.224	6.72	C2
75 um	104.059	103.761	0.298	2.98	C3
Pan	79.823	79.823	0		
		Sum	1.991	83.15	Cohesion
		Amt added	1.994		

Sieves held in place w/ clamp on top of sieves

W173696B

	Sieve + Sample	Sieve	Sample	Cohesion	
250 um	110.291	109.205	1.086	54.3	C1
150 um	109.7	109.522	0.178	5.34	C2
75 um	104.495	103.761	0.734	7.34	C3
Pan	79.83	79.823	0.007		
		Sum	2.005	66.98	Cohesion
		Amt added	1.994		

Sieves held in place w/ clamp on top of sieves

W173689A Trial 2

	Sieve + Sample	Sieve	Sample	Cohesion	
250 um	110.151	109.205	0.946	47.3	C1
150 um	110.34	109.522	0.818	24.54	C2
75 um	104.032	103.761	0.271	2.71	C3
Pan	79.832	79.823	0.009		
		Sum	2.044	74.55	Cohesion
		Amt added	2.034		

Sieves held in place w/ clamp on top of sieves

W173689B Trial 2

	Sieve + Sample	Sieve	Sample	Cohesion	
250 um	109.653	109.205	0.448	22.4	C1
150 um	109.893	109.522	0.371	11.13	C2
75 um	104.969	103.761	1.208	12.08	C3
Pan	79.856	79.823	0.033		
		Sum	2.06	45.61	Cohesion
		Amt added	1.998		

Sieves held in place w/ clamp on top of sieves

W173689B Trial 3

	Sieve + Sample	Sieve	Sample	Cohesion	
250 um	110.136	109.205	0.931	46.55	C1
150 um	109.815	109.522	0.293	8.79	C2
75 um	104.592	103.761	0.831	8.31	C3
Pan	79.845	79.823	0.022		
		Sum	2.077	63.65	Cohesion
		Amt added	2.044		

Sieves held in place w/ clamp on top of sieves

Corrosivity

2(WxT + T*H + W*H) + pi*D*T - 2*pi*(d/2)^2

Calculation of coupon surface area: 10.831851 cm2

W	H	T	D	
0.495	1.4775	0.06	0.189	inches
1.2573	3.75285	0.1524	0.48006	cm

Minimum volume of solution required = 433.27404 mL

Sample

	W173686A	W173686B	W173696A	W173696B
Grams	1001.82	994.61	1003	1000.53
mL of DI	1000	1000	1000	1000
mL of Leachate collected	645	520	510	540
Weight of coupon, grams				
Initial, grams	5.5591	5.4537	5.3629	5.4467
Final, grams	5.5559	5.4518	5.3618	5.4455
Amt Lost, mg	3.2	1.9	1.1	1.2
Time (hours)	24	24	24	24
Corrosion Rate (mmpy)	0.137188	0.08145538	0.04715838	0.051446

(A rate of >6.35 mm per year is considered corrosive)

Miscellaneous Tests

Percent Moisture

pH	W173689	W173689	W173696	W173696B
	7.8	7.82	7.95	7.86

Percent Moisture

	689A	689B	696A	696B
Initial Weights				
Sample + Crucible	182.245	187.44	181.734	173.996
Crucible	161.705	163.77	161.569	154.642
Sample	20.54	23.67	20.165	19.354
Dry (Final) Weights				
Sample + Crucible	181.782	186.813	181.119	173.144
Crucible	161.705	163.77	161.569	154.642
Sample	20.077	23.043	19.55	18.502
Percent Moisture	2.31	2.72	3.15	4.60
	0.414878	0.42478		1.459128

It appears that the sample may rapidly absorb moisture from the air

Water Leach

	Initial Amt Solid	Final Amt Solid	Percent Soluble	Ethanol Leach Initial Amt Solid	Final Amt Solid	Percent Soluble
W173689A	50.032	26.328	47.38	25.082	20.961	16.43
W173689B	49.556	26.695	46.13			
W173696A	49.943	28.028	43.88			
W173696B	49.941	27.482	44.97			

	689A	689B	696A	696B	Ethanol 689A
Final Weights					
Watchglass + filter paper + solid	182.688	122.955	125.655	268.656	262.708
Watchglass	155.488	95.419	96.79	240.303	240.26
Filter paper	0.872	0.841	0.837	0.871	1.487
Dry Solid	26.328	26.695	28.028	27.482	20.961

Relative Humidity Test
All performed on sample 689A

Percent Humidity

Saturated Salt Solution	CaCl2	Ca(NO3)2	KNO3/NH	Na2SO3	sample
	31	51	71.2	95	95

Date	Time	Temp, C	Measured Humidity	Weight
15-Dec	12:15		began, time approximate	
	12:24		51	
	12:30	24.2	58	
	12:40	24	63	
	2:05	23.7	76	
18-Dec	2:40	23.6	78	
	7:35		began 30	
	3:30	22.8		
	3:30			
19-Dec	10:10	23.3	began 50	
	10:10			
20-Dec	9:30	18.4	began 70	
	9:30			
21-Dec	3:25	20.8	began 84	

Weights

Relative H	31	51	71	95
0	39.081	33.996	44.625	45.382
1	39.528	35.135	46.814	49.143
4	39.691	36.408	50.371	55.684
7	39.799	36.948	52.218	60.469
21	39.988	38.243	55.597	69.758

161.667 pan + sample
116.285 pan, etc.

Percent Weight Gain

Relative H	31 %H	51 %H	71 %H	95 %H
0	0.00	0.00	0.00	0.00
1	1.14	3.35	4.91	8.29
4	1.56	7.09	12.88	22.70
7	1.84	8.68	17.02	33.24
21	2.32	12.49	24.59	53.71