Recommended Practices for Testing Sand Used in Hydraulic Fracturing Operations

API RECOMMENDED PRACTICE 56
SECOND EDITION, DECEMBER 1995

Reaffirmed June 2000

American Petroleum Institute

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Exploration and Production Department

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FOREWORD

These recommended practices were prepared by the Task Group on Evaluation of Hydraulic Fracturing Sand under the API Subcommittee on Evaluation of Well Completion Materials. They have been reviewed for content and accuracy by the Subcommittee on Evaluation of Well Completion Materials and by the API Executive Committee on Drilling and Production Practices. This publication is under jurisdiction of the Executive Committee on Drilling and Production Practices, American Petroleum Institute's Exploration and Production Department.

The tests recommended herein have been developed to improve the quality of frac sand delivered to the well site. They are for use in evaluating certain physical properties of sand used in hydraulic fracturing operations. These suggested tests will enable users to compare the physical characteristics of various sands tested under the described conditions and to select materials most useful for application in hydraulic fracturing operations.

The recommendations presented in this publication are not intended to inhibit the development of new technology, materials improvements, or improved operational procedures. Qualified engineering analysis and judgment will be required for their application to fit a specific situation.

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Suggested revisions are invited and should be submitted to the director of the Exploration and Production Department, American Petroleum Institute, 1220 L Street, N.W., Washington, D.C. 20005.

Recommended Practices for Testing Sand Used in Hydraulic Fracturing Operations

1 Scope

The objective of these recommended practices is to provide control of frac sand quality at the well site. As a first step in accomplishing this objective, the recommended tests should be applied at the basic point of supply where quality control is first exercised.

2 References

2.1 STANDARDS

Unless otherwise specified, the most recent editions or revisions of the following standards shall, to the extent specified herein, form a part of this standard.

ASTM[1]

E 11-95 *Specifications for Wire-Cloth Sieves for Testing Purposes*

2.2 OTHER REFERENCES

Krumbein, W.C. and Sloss, L.L., *Stratigraphy and Sedimentation*, Second Edition, 1963, W.N. Freeman and Co., New York, NY.

3 Recommended Sand Sampling Procedure

3.1 DESCRIPTION

The sampling procedure should provide a representative sample of the frac sand supplied by the sand supplier to the service company or by the service company to the user. This sample is to be compiled from a flowing stream of sand as opposed to material sampled at rest.

3.2 EQUIPMENT

The following equipment should be used to compile representative sand samples and conduct physical tests:

a. Box sampling device approximately 8 inches × 6 inches × 4 inches with a 1/2-inch opening. Refer to Figure 1.
b. Sample reducer (of appropriate size for handling sack-size samples and reducing in one pass to 1/16 original weight). Refer to Figure 2.
c. Sample splitter of appropriate size. Refer to Figure 3.
d. Set of recently calibrated sieves, complying with requirements of the U.S.A. Sieve Series, 8-inch diameter. Refer to *ASTM E 11-95: Specifications for Wire-Cloth Sieves for Testing Purposes*. Refer to Figure 4.
e. Testing sieve shaker. Refer to Figure 4.
f. Scale (minimum of 100 gram capacity with precision of 0.1 gram or better).

[1]ASTM, 100 Bar Harbor Drive, West Conshohocken, Pennsylvania 19428-2959.

3.3 NUMBER OF REQUIRED SAMPLES

A minimum of nine samples per rail car load and three samples per truck load should be obtained, combined, and tested. For material sampled at the fracturing job site, a minimum of five samples should be obtained per 100,000 pounds of sand or fraction thereof. These on-site samples should be combined and used as a single sample for subsequent testing operations.

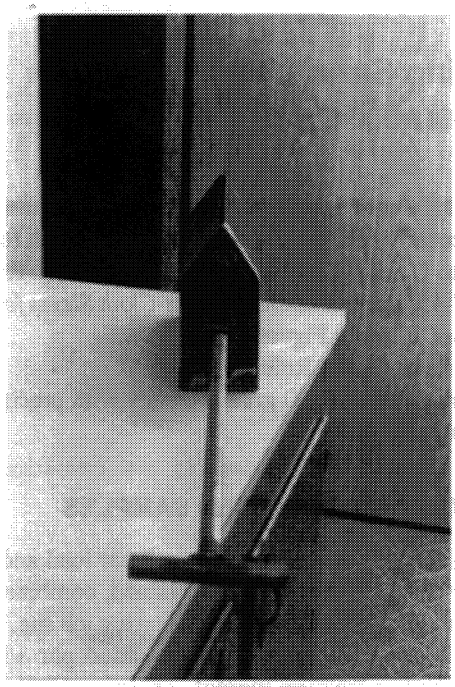
3.4 SAMPLING

The sampling device, with its longitudinal axis perpendicular to the flowing sand stream, should be passed at a uniform rate from side to side through the full stream width of moving sand as the sand falls from a conveyor belt into a blender, truck, or rail car. Sand should be allowed to flow for at least 2 minutes after initial flow prior to taking the first sample. Several samples should be extracted at approximately uniform intervals through the body of sand to ensure a representative sample for analysis. The number of samples taken should comply with the requirements of 3.3. During sampling, the sampling receptacle should be swung completely across the moving sand stream in a brief interval of time so as to take all of the stream part of the time. Under no circumstances should the sampling receptacle be allowed to overflow.

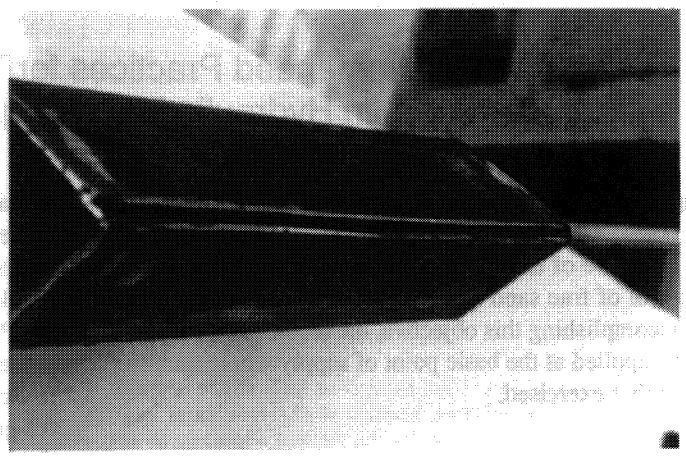
4 Recommended Sand Samples Handling and Storage

4.1 SAMPLE REDUCTION (SACKED MATERIAL)

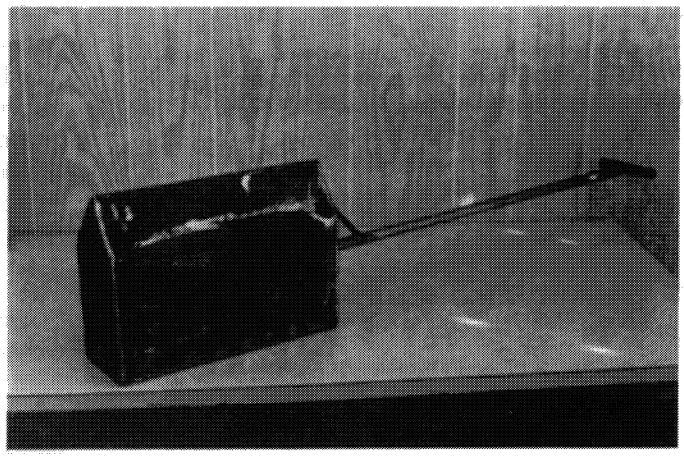
Place the contents of an entire sack of frac sand (approximately 100 pounds) in the sample reducer (refer to Figure 2). Obtain a reduced sample of approximately 6 pounds (approximately 1/16 of the original weight of the total sack's contents).



A—End View



B—Top View



C—Side View

Figure 1—Example Box Sampling Device

4.2 SAMPLE SPLITTING

Place the reduced sand sample in the sample splitter (refer to Figure 3) and split the sample to a suitable testing size. Sufficient sand sample should be split to permit performing recommended tests under all sections of this document. Use of an appropriately sized sample reducer and sample splitter to permit samples to be prepared for testing is an essential step in the recommended procedures.

4.3. SAMPLE RETENTION AND STORAGE

The basic sand producer should maintain written records of tests conducted on each shipment for 1 year. Physical samples of an amount sufficient to conduct all tests recommended herein, but in no case less than 250 grams, should be retained in storage for 3 months for bulk domestic shipments, 6 months for sacked domestic shipments, and 12 months for international shipments. Copies of test results and samples should be furnished by the sand producer, on request, to user companies.

5 Recommended Frac Sand Sieve Analysis

5.1 SIEVE ANALYSIS

Stack six recently calibrated U.S.A. Sieves plus a pan in a nest of decreasing sieve openings from top to bottom (refer to Table 1 for recommended sieves used in testing designated sand sizes). Obtain a split sample of approximately 100 grams and establish an accurate sample weight to within 0.1 gram. Pour the split sample onto the top sieve and place the nest of six sieves plus pan in a Ro-Tap testing sieve shaker (or equivalent) and sieve for 10 minutes. Remove and unload each sieve, being certain to brush each sieve thoroughly with the sieve manufacturer's recommended brush to

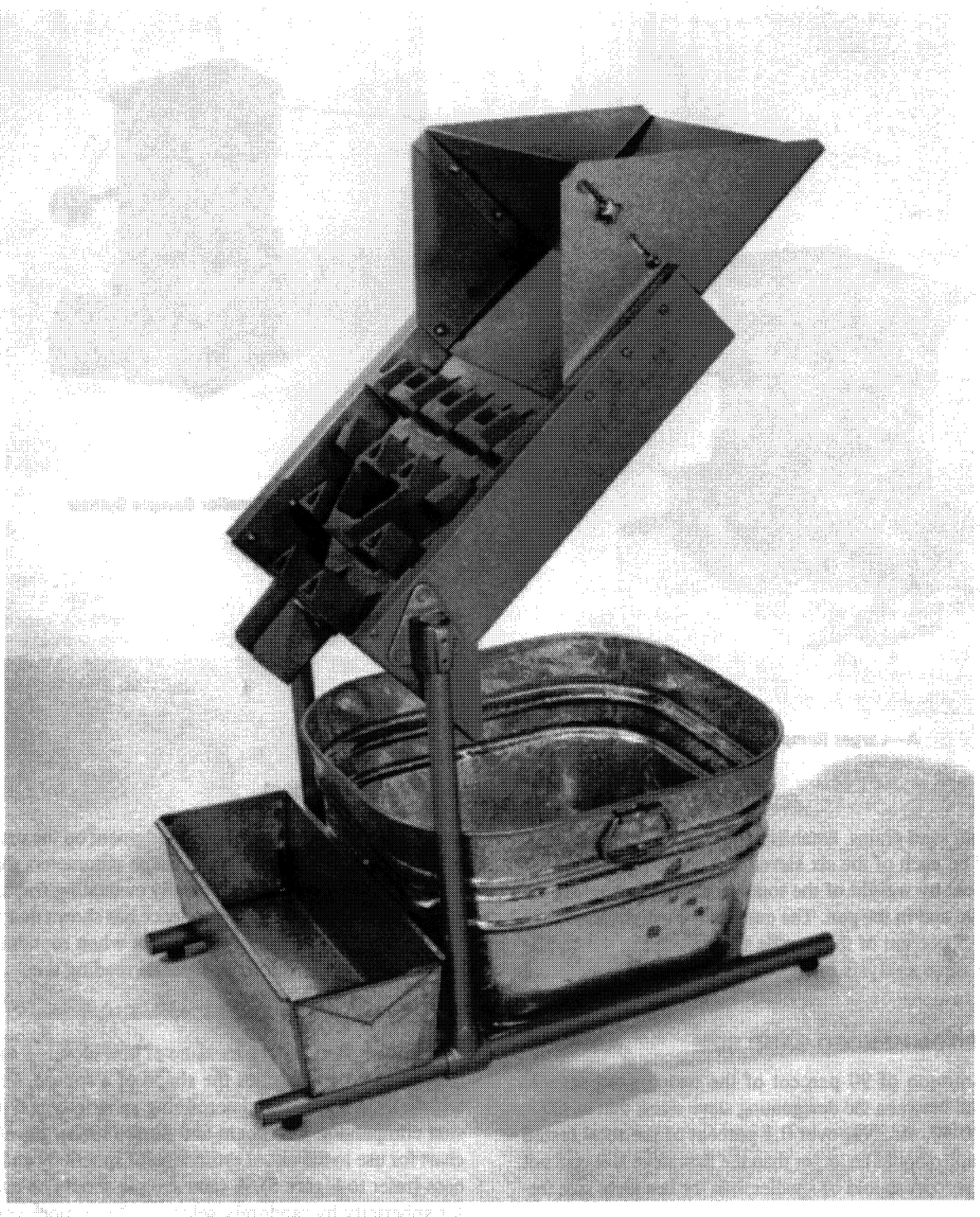
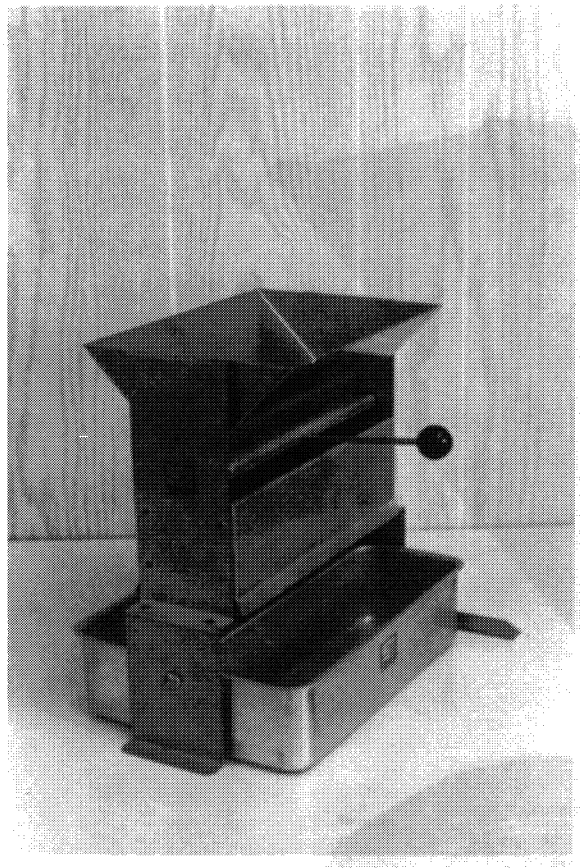
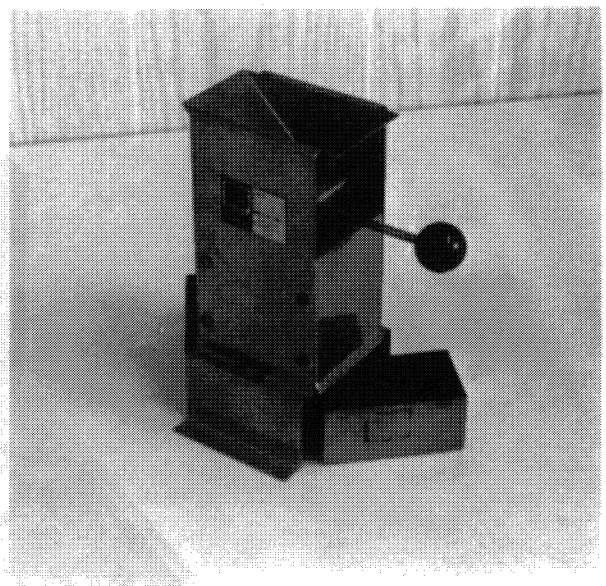


Figure 2—Example Sample Reducer Equipment

Photo courtesy of W.S. Tyler, Inc., Subsidiary of Combustion Engineering, Inc., Mentor, Ohio 44060.



A—Larger Sample Splitter



B—Smaller Sample Splitter

Figure 3—Example Sample Splitter Equipment

remove all sand grains. Establish an accurate weight of sand retained on each of the six sieves and in the pan. Calculate the percent by weight of the total sand sample retained on each sieve and in the pan. The cumulative weight should be within 0.5 percent of the sample weight used in the test. If not, the sieve analysis must be repeated using a different sample.

5.2 RECOMMENDED SAND SIZE

A minimum of 90 percent of the tested sand sample should fall between the designating sieve sizes, that is, 6/12, 12/20, 20/40, etc. Not over 0.1 percent of the total tested sand sample should be larger than the first sieve size and not over 1.0 percent should be smaller than the last sieve size (refer to Table 1).

6 Frac Sand Sphericity and Roundness

6.1 GENERAL

Numerous methods have been published to measure and report sand grain shapes and geometric identities. Some involve tedious measurements; others require visual comparisons. All require some skill and judgment on the part of the technician. The common grain shape parameters that have been found to be useful for visually evaluating frac sand are sphericity and roundness. Experience has shown that the best results are obtained with these tests when roundness and sphericity are determined in separate reading sets.

6.2 SPHERICITY

Particle sphericity is a measure of how closely a sand particle or grain approaches the shape of a sphere. The most widely used method of determining sphericity is with a visual comparator. Krumbein and Sloss (1963)[2] developed a chart for use in the visual estimation of sphericity and roundness (refer to Figure 5). A sand sample should be evaluated for sphericity by randomly selecting 20 or more grains for examination. These grains should be viewed through a 10- to 20-power microscope or examined by photomicrograph of suitable enlargement (refer to 6.6.3). Sphericity of each grain should be determined and recorded, and an average sphericity obtained for the sample.

[2] *Stratigraphy and Sedimentation*, Second Edition, 1963, published by W. H. Freeman & Co., New York, NY.

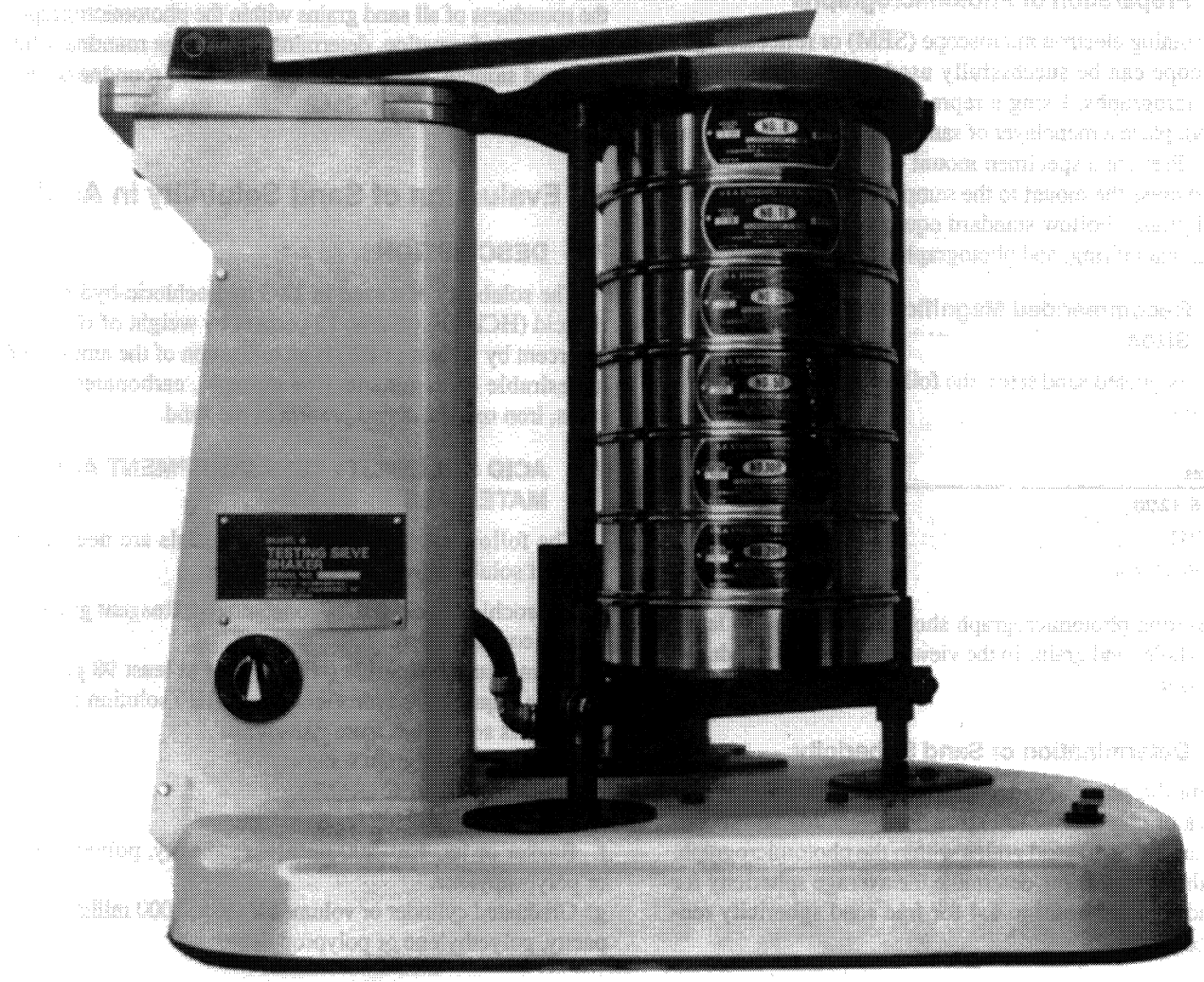


Figure 4—Testing Sieve Shaker and Nest of Six U.S.A. Sieves Plus Pan

Photo courtesy of W.S. Tyler, Inc., Subsidiary of Combustion Engineering, Inc., Mentor, Ohio 44060.

6.3 ROUNDNESS

Grain roundness is a measure of the relative sharpness of grain corners, or of grain curvature. Evaluation of sand grain roundness should be made on the same sample as that used for the sphericity determination. Roundness of each grain should be determined, recorded, and an average roundness obtained for the sample.

6.4 RECOMMENDED SPHERICITY AND ROUNDNESS

Frac sand should have a sphericity of 0.6 or greater and a roundness of 0.6 or greater.

6.5 SAND GRAIN CLUSTERS

Frac sands should consist of single, well-rounded quartz sand grains. Examination of a representative sample should be conducted at low magnification (10X to 20X). The sand should not be considered suitable if it contains 1 percent or more by count of clusters of multiple sand grains.

6.6 ALTERNATE METHOD FOR DETERMINING AVERAGE SPHERICITY AND ROUNDNESS

6.6.1 Use of Photomicrographs

Photomicrographs of a representative frac sand sample may be used to provide identical suitably enlarged repro-

ductions for use to obtain the average sphericity and roundness for the sand sample.

6.6.2 Preparation of Photomicrographs

A scanning electron microscope (SEM) or reflected light microscope can be successfully used to produce suitable photomicrographs. Using a representative split sample of frac sand, place a monolayer of sand grains on a flat, resilient surface. Prepare a specimen mount using double adhesive tape and press the mount to the sample to affix a monolayer of sand grains. Follow standard equipment procedures for coating, magnifying, and photographing the sand sample.

6.6.3 Recommended Magnification for Sand Sizes

For designated sand sizes, the following magnification is suggested:

Sand Sizes	Photomicrograph Magnification
6/12, 8/16, 12/20	15X
16/30, 20/40	30X
30/50, 40/70, 70/140	40X

The resulting photomicrograph should be cropped to leave 20–25 whole sand grains in the viewing area and reproduced as necessary.

6.6.4 Determination of Sand Sphericity

Using the photomicrograph from 6.6.3 and the visual comparator chart (refer to Figure 5), determine and record the sphericity of all sand grains within the photomicrograph. Using this information, determine the average sphericity for the sand sample. Refer to 6.4 for frac sand sphericity recommendations.

6.6.5 Determination of Sand Roundness

Using the photomicrograph from 6.6.3 and the visual comparator chart (refer to Figure 5), determine and record the roundness of all sand grains within the photomicrograph. Using this information, determine the average roundness for the sand sample. Refer to 6.4 for frac sand roundness recommendations.

7 Evaluation of Sand Solubility in Acid

7.1 DESCRIPTION

The solubility of a sand in 12-3 hydrochloric-hydrofluoric acid (HCl-HF) (that is, 12 percent by weight of HCl and 3 percent by weight of HF) is an indication of the amount of undesirable contaminants (for example, carbonates, feldspars, iron oxides, clays) present in the sand.

7.2 ACID SOLUBILITY TEST EQUIPMENT AND MATERIALS

The following equipment and materials are needed to conduct solubility tests on sand samples:

a. Hydrochloric acid (HCl), concentrated. Reagent grade of known concentration.
b. Ammonium bifluoride (NH_4HF_2) of at least 98 percent purity is required. A hydrofluoric acid (HF) solution may be used but is somewhat more hazardous.
c. Balance, 1 milligram accuracy.
d. Water bath, 65.6°C (150°F).
e. Oven, 105°C (221°F).
f. Beaker or jar, 150–200 milliliter capacity, polyethylene or polypropylene.
g. Graduated cylinder or volumetric flask, 1000 milliliter capacity, polyethylene or polypropylene.

Table 1—Recognized Frac Sand Sizes

Sieve Opening Sizes (micrometers)	3350/ 1700	2360/ 1180	1700/ 850	1180/ 600	850/ 425	600/ 300	425/ 212	212/ 106
Frac Sand Size Designations	[b] 6/12	[b] 8/16	[a] 12/20	[b] 16/30	[a] 20/40	[b] 30/50	[a] 40/70	[b] 70/140
Nest of U.S.A. Sieves[c] Recommended for Testing	4	6	8	12	16	20	30	50
	6	8	12	16	20	30	40	70
	8	12	16	20	30	40	50	100
	10	14	18	25	35	45	60	120
	12	16	20	30	40	50	70	140
	16	20	30	40	50	70	100	200
	Pan	Pan	Pan	Pan	Pan	Pan	Pan	Pan

[a]Primary Frac Sand Size.
[b]Alternate Frac Sand Size.
[c]U.S.A. Sieve Series as defined in ASTM E 11-95.

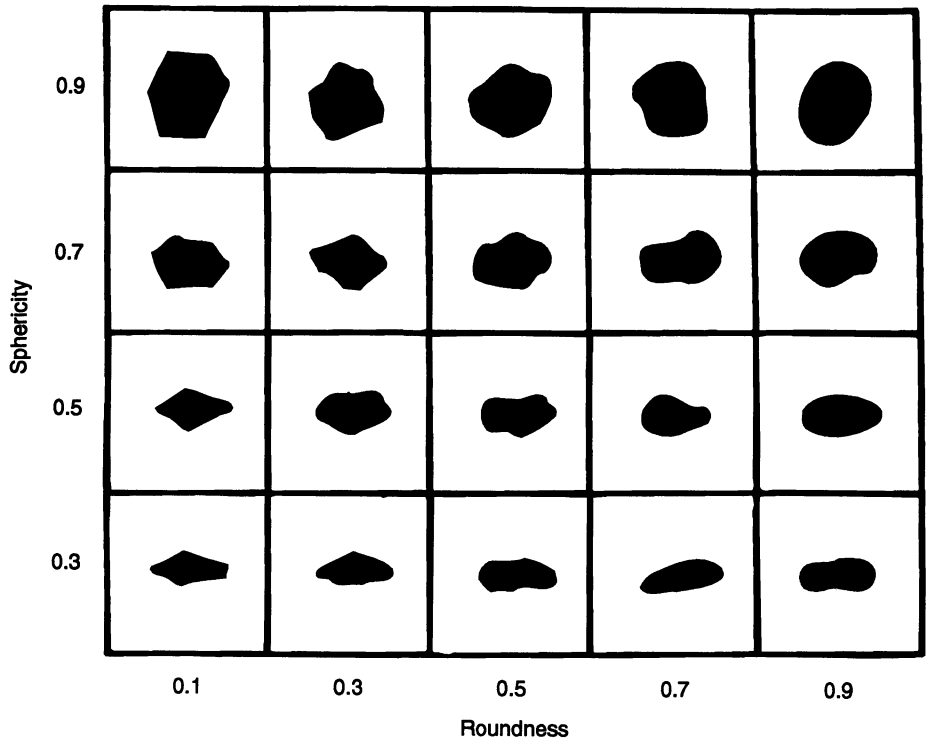


Figure 5—Chart for Visual Estimation of Sphericity and Roundness

h. Analytical filtering apparatus. The following are available and vacuum filtering techniques may be used:

1. Coor's #27004 Gooch crucible with 1/16-inch-thick pad of #40 or #42 Whatman acid-resistant filter paper circles (2.1-centimeter diameter).
2. Gelman filter funnel #4204 using polysulfone filter support #79932 and pad #61756 with 1/16-inch-thick pad of #42 Whatman acid-resistant filter paper circles (1.91-centimeter diameter).
3. Cole-Parmer #6607 filter crucible (by Bel Art) with 1/16-inch-thick pad of #42 Whatman acid-resistant filter paper circles (1.91-centimeter diameter).

7.3 ACID SOLUBILITY TEST PROCEDURE

The following should be used to evaluate the solubility of a representative sand sample in HCl-HF acid.

Note: This procedure is gravimetric in nature and as such requires strict procedures and good laboratory technique to provide reproducibility. Representative sand samples *must* be taken from the sample splitter prior to sieve analysis. Samples should not be subjected to the crush resistance test or ground prior to the acid solubility analysis, but rather the analysis must be performed on the unaltered whole-grain sand.

7.3.1 Prepare a solution of 12-3 HCl-HF acid [specific gravity = 1.08 at 15.6°C (60°F)]. Two examples for preparation of 1000 milliliters of 12-3 HCl-HF are:

a. Using ammonium bifluoride (NH_4HF_2).

1. To 500 milliliters of distilled water contained in a polyethylene or polypropylene 1000-milliliter graduated cylinder or volumeric flask, add 46.23 grams of pure NH_4HF_2 and dissolve. Actual weight of NH_4HF_2 of less than 100 percent purity to be added is equal to 46.23 grams divided by NH_4HF_2 purity, in weight fraction.
2. Add 361 milliliters of 37 percent hydrochloric acid (HCl) [specific gravity = 1.19 at 15.6°C (60°F)].
3. Dilute to 1000 milliliters with distilled water.
4. Stir to ensure complete mixing.

b. Using 52 percent hydrofluoric acid (HF).

1. To 500 milliliters of distilled water contained in a polyethylene or polypropylene 1000-milliliter graduated cylinder or volumetric flask, add 54 milliliters of 52 percent HF [specific gravity = 1.18 at 20°C (68°F)].
2. Add 293 milliliters 37 percent HCl [specific gravity = 1.19 at 15.6°C (60°F)].
3. Dilute to 1000 milliliters with distilled water.
4. Stir to ensure complete mixing.

7.3.2 Weigh 5 grams of sand to the nearest milligram into a tared sample pan. The sand should be dried at 105°C (221°F) to a constant weight and cooled in a desiccator.

7.3.3 To a 150-milliliter polyethylene beaker (jar) containing 100 milliliters of the acid solution from 7.3.1, add the sand sample. The acid and the sample should be at room temperature (22°C ± 3°C or 72°F ± 5°F).

7.3.4 Place beaker (jar) in a 65.6°C (150°F) water bath for a minimum of 30 minutes and a maximum of 35 minutes. Do not stir. Be careful not to allow contamination of the sample.

7.3.5 Prepare the filtering apparatus by adding a 1/16-inch-thick pad of #42 Whatman filter paper to the crucible or filter funnel. Dry the funnel in an oven at 105°C (221°F) for at least 1 hour or to constant weight, weigh, and record the weight. The filter should not be weighed hot but allowed to cool in a desiccator.

7.3.6 Transfer the sand and acid mixture from the beaker (refer to 7.3.4) to the filtering apparatus (refer to 7.3.5). Filter the sample through the preweighed filter crucible (funnel) being sure to transfer all particles from the beaker (jar) to the filter. Vacuum filtering techniques may be used to speed this step.

7.3.7 Wash the sand in the filtering apparatus three times with 20-milliliter portions of distilled water.

7.3.8 Dry the filter and retained sand sample at 105°C (221°F) for a minimum of 1 hour or until constant weight is obtained. Cool the filter and sample in a desiccator before weighing. Weigh filter containing sand and record the weight.

7.3.9 Calculate and report percent sand solubility using the following equation:

$$S = \frac{(W_s + W_r - W_{fs})}{(W_s)} \times 100$$

Where:

S = sand solubility, weight percent

W_s = sand weight, grams (refer to 7.3.2)

W_f = weight of filter, grams (refer to 7.3.5)

W_{fs} = weight of filter containing sand, grams (refer to 7.3.8)

7.4 RECOMMENDED MAXIMUM ACID SOLUBILITY

The acid-soluble material in frac sand should not exceed the values shown in Table 2.

Table 2—Recommended Maximum Acid Soluble Material Content in Frac Sand

Sand Size (mesh)	Maximum Solubility (weight percent)
6/12 through 30/50	2.0
40/70 through 70/140	3.0

8 Recommended Silt Test

8.1 METHOD I: TURBIDITY MEASUREMENT OF SILT- AND CLAY-SIZE PARTICULATE MATTER

8.1.1 Introduction

Turbidity in water is the result of suspended clay, silt, or finely divided inorganic matter being present. Frac sand samples can be placed in distilled water and the turbidity of the resulting liquid measured. Properly washed and processed frac sand will pass the turbidity test described below.

8.1.2 Turbidity Measurement, General

Turbidity tests measure an optical property of a suspension that results from the scattering and absorbing of light by the particulate matter present. The amount of turbidity registered is dependent on such variables as size, shape, and refractive indices of the particles. No direct relationship exists between the turbidity of a sample and the weight concentration of particulate matter present therein.

8.1.3 Turbidity Calibration

Turbidity calibrations were originally based on the Jackson candle turbidimeter, with results expressed in Jackson Turbidity Units (JTU). Since the Jackson candle turbidimeter lacks sensitivity in the low turbidity range, below 25 JTU, the meter scale calibrations have been based on a uniform milky polymer, formazin, that allows accurate calibrations over a wide range. The results are expressed as Formazin Turbidity Units (FTU) and are equivalent to JTU. Suitable spectrophotometers for use in this procedure are the Spectronic Mini 20, Bausch and Lomb Spectrometer 20, Perkin Elmer Coleman Model 35, Hach Model 2100A, or equivalent.

8.1.4 Preparation of Formazin Solution

Prepare a milky white suspension of formazin polymer for use as the turbidity reference standard for conversion of percent transmittance (instrument reading) to FTU. A stock formazin suspension that can be diluted to provide a series of standard solutions covering a range of turbidity values should be prepared as follows:

a. Dissolve 1.0 gram of hydrazine sulfate in demineralized water and dilute to the mark in a 100-milliliter volumetric flask.

b. Dissolve 10.0 grams of hexamethylenetetramine in demineralized water and dilute to the mark in a 100-milliliter volumetric flask.

c. Transfer 5.0 milliliters of each solution prepared in steps a. and b. to a 100-milliliter volumetric flask and mix and allow to stand undisturbed for 24 hours at 25°C (±3°C) or 77°F (±5°F).

d. Use demineralized water to dilute the mixture from step c. to the mark in a 100-milliliter flask and mix. The turbidity of this standard stock solution is 400 FTU. The turbidity of a standard solution prepared by dilution of this stock suspension is proportional to the formazin concentration. For example, the turbidity of a standard solution prepared by diluting 50 milliliters of the 400 FTU stock suspension to 100 milliliters is defined as 200 FTU.
e. The standard stock solution prepared in step d. should be prepared monthly. Dilutions used for standard solutions should be prepared fresh daily.

8.1.5 Equipment Calibration Procedure

The procedure presented herein is general in nature. Testers should check equipment specification manuals for specific and appropriate calibration procedure details.

8.1.5.1 Adjust instrument.

a. Adjust the wave length control to 450 nanometers.
b. Place the opaque rod in the sample compartment and check the zero adjustment.
c. Place a vial containing clear, colorless, turbidity-free water in the sample compartment and adjust the full-scale control to give a meter reading of exactly 100 percent transmittance.

8.1.5.2 Prepare a chart to convert percent transmittance (%T) to FTU.

a. Dilute stock suspension from 8.1.4 to make several standard solutions of known turbidity.
b. For each, place a test vial containing the standard solution in the sample compartment and read the percent transmittance.
c. Plot turbidity (FTU) versus percent transmittance (%T).

8.1.6 Frac Sand Turbidity Measurement

Prepare a sample for turbidity measurement of frac sand as follows:

a. Measure 20 milliliters of dry sand sample and mix with 100 milliliters of demineralized water in a 6-ounce, wide-mouth bottle. Allow to stand for 30 minutes.
b. Shake vigorously by hand for approximately 45–60 shakes in 30 seconds (do not shear in a mechanical mixer). Allow to stand for 5 minutes.
c. Using a syringe, extract 25 milliliters of water-silt suspension from near the center of the water volume.
d. Place the water-silt suspension in the test vial and place in the instrument previously calibrated according to 8.1.5.
e. Determine the sample turbidity in FTU.

8.1.7 Suggested Maximum Frac Sand Turbidity

The turbidity of tested frac sand should be 250 FTU or less.

8.2 METHOD II: FIELD ON-SITE TURBIDITY TEST

8.2.1 Purpose

This test may be used to determine the cleanliness of frac sand at the field location using a minimum of equipment and readily adaptable procedures. The test can be accomplished by carefully observing the cloudiness of the water phase of a mixture of frac sand and water. The procedure uses a marked prescription bottle containing a specified amount of sand sample and water. The test provides a "go, no-go" answer. If the water phase is clear enough to read an identification label on the bottle, the sand should be considered clean and suitable for use. However, if the water phase is cloudy enough to prevent distinguishing the identification label on the bottle, the sand should be considered dirty and unsuitable for use.

8.2.2 Equipment and Materials

The following equipment and materials are necessary for conducting this turbidity test:

a. Frac sand sample.
b. Turbidity-free water (distilled water, if available).
c. Four-ounce, clear-glass prescription bottle with cap closure (refer to Figure 6), calibrated to 100 milliliters in 10-milliliter increments.
d. Black felt tip marking pen.
e. Small funnel.

8.2.3 Test Procedure

8.2.3.1 Using a felt tip marking pen, record the sample identification in characters approximately 1/2 inch high on the flat side of a sample prescription bottle.

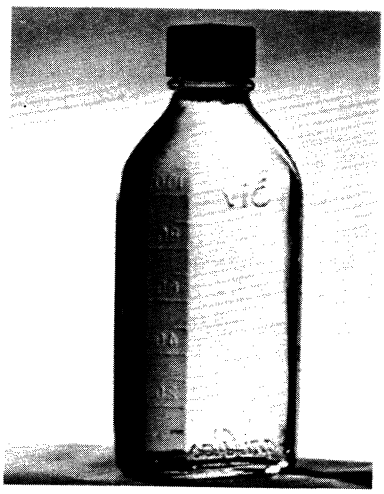


Figure 6—Example Prescription Bottle

8.2.3.2 With the funnel inserted in the prescription bottle, carefully fill the bottle to the 20-milliliter mark with the sand sample. Gently tap and level the sand and add sand to achieve the 20-milliliter mark, but do not fill above the 20-milliliter level. It is extremely important to use the proper sample size and care should be exercised in this step.

Note: 20 milliliters of sand weighs approximately 40 grams.

8.2.3.3 Add turbidity-free water (distilled water, if available) to the 100-milliliter mark on the bottle.

8.2.3.4 Cap the bottle and shake vigorously for 10 seconds.

8.2.3.5 Hold the bottle at arm's length toward a moderate light source, for example, an outside window or the horizon on a clear, bright day. Do not face the sun directly. The flat side of the bottle, with the sample identification information thereon, should be faced toward the light source.

8.2.4 Interpretation of Test Results

a. If the sample identification information can be read through the water phase, the sand should be judged clean and suitable for use.
b. If the sample identification information is not legible, the sand should be judged dirty and unsuitable for use.
c. If the sample identification information can be read but with difficulty, let the sample stand for 10 minutes and repeat operations prescribed in 8.2.3.4 and 8.2.3.5. If now legible, the sand should be judged clean and suitable for use. However, if the sample identification information cannot be read, additional material was dispersed by the longer exposure time and the sand should be judged dirty and unsuitable for use.

8.3 METHOD III: CENTRIFUGAL MEASUREMENT OF CLAY AND SOFT PARTICLE CONTENT

8.3.1 Procedure

The clay and soft particle content of frac sand should be determined by washing 10 milliliters of the frac sand sample in a total volume of 50 milliliters of distilled water. The 10-milliliter sand sample should be placed in a 50-milliliter graduated centrifuge tube and washed by adding 10–15 milliliters of the distilled water and hand shaking the sand-water mixture for 30 seconds. The wash water should be carefully decanted into a second graduated centrifuge tube. The sand sample washing procedure should be repeated until the total 50 milliliters of distilled water is used. The 50-milliliter sample of collected wash water should be centrifuged for 10 minutes, using a centrifuge capable of operating at 3000 (±200) revolutions per minute and supplying a centrifugal force of 1500 (±100) gravity (G). The clay and soft particle content in the bottom of the centrifuge tube should be noted and recorded. One milliliter of sediment in the centrifuge tube is equal to 10 percent clay and soft particle content; 0.5 milliliters is equal to 5 percent clay and soft particle content, etc.

8.3.2 Suggested Maximum Frac Sand Clay and Soft Particle Content

Frac sand clay and soft particle content should not exceed 1 percent, for example, 0.1 milliliters of sediment in a 10-milliliter sand sample.

9 Recommended Frac Sand Crush Resistance Test

9.1 GENERAL

Silica sand varies in composition and strength. The following test is useful for comparing the crush resistance of different samples of sand. The test is to be conducted using a given volume of sand particles, all of which have been sieved and found to be within the specified frac sand size range.

9.2 EQUIPMENT AND MATERIALS

The following equipment and materials are necessary for the recommended frac sand crush resistance test:

a. Frac sand sample.
b. Press capable of applying load required to accomplish the stress levels specified in Table 3. *The press must have platens that can be maintained parallel during application of load to the cell. The press must be calibrated to ensure that stress measurements are accurate to within 5 percent, or an independent, calibrated load-measuring device should be used when the load is applied to the cell.*
c. Cell for sand crush resistance test as described in Figure 7, or equivalent. The piston length should be 3.5 inches regardless of the diameter of the piston used in the cell.
d. Pan and two U.S.A. Sieves of the mesh size opening for the specified sand size range, for example, the No. 20 and No. 40 sieves for use with a 20/40 sand; the No. 12 and No. 20 sieves for use with a 12/20 sand.
e. Balance for weighing sand sample to 0.1-gram tolerance.
f. Ro-Tap testing sieve shaker, or equivalent.

9.3 RECOMMENDED TEST PROCEDURE

9.3.1 Stack the two U.S.A. Sieves and pan, with the larger sieve opening size on top, and pour a sufficient quantity of split frac sand sample on the top sieve to provide in the test cell (refer to Figure 7) a concentration of 4 pounds per square foot of the mesh size specified for the sample being tested (for example, a 2-inch inside diameter test cell requires a 40-gram sample). For test cell inside diameters other than 2 inches, equation (1) should be used to determine the

appropriate quantity of sand to be placed in the test cell. Place the sieves in a Ro-Tap testing sieve shaker (or equivalent) and sieve for 10 minutes.

$$W = 40.0\left(\frac{d}{2}\right)^2 \quad (1)$$

Where:

W = weight of split frac sand sample, grams

d = inside diameter of test cell, inches

9.3.2 Discard all of the sieved sand sample material except that remaining on the lower screen.

9.3.3 Place the sieved sand (obtained under 9.3.1) equivalent to 4 pounds per square foot (weighed to the nearest 0.1 gram) in the test cell (for example, a 2-inch inside diameter test cell requires a 40-gram sand sample). Pour the sand sample into the test cell, constantly moving the source of the sand to keep the surface in the cell as level as possible.

9.3.4 Level the surface of the sand in the cell. This is to be done by inserting the piston in the cell and, without applying any force, rotating the piston 180 degrees (in one direction only).

Note: To ensure uniformity in leveling the surface of the sand in the cell, the piston length should be 3.5 inches.

Table 3—Stress to Be Applied and Suggested Maximum Fines for Frac Sand Crush Resistance Tests

Mesh Size	Load on Cell[a] (lb force)	Stress on Sand (psi)	Suggested Maximum Fines (% by weight)
6/12	6,283	2,000	20
8/16	6,283	2,000	18
12/20	9,425	3,000	16
16/30	9,425	3,000	14
20/40	12,566	4,000	14
30/50	12,566	4,000	10
40/70	15,708	5,000	8
70/140	15,708	5,000	6

[a]Note: Indicated loads are for cells with a 2-inch diameter piston. For cells of other sizes, the cell load should be adjusted by the factor $\left(\frac{\text{diameter of cell, in.}}{2}\right)^2$.

For example, a 3-inch diameter cell, loads shown in Table 3 should be multiplied by a factor, $\left(\frac{3}{2}\right)^2 = 2.25$. Thus, to achieve a stress of 2,000 pounds per square inch requires a load of (6,283) (2.25) = 14,137 pounds force. Similarly, a test cell with a 1.5-inch diameter requires an applied load of $\left(\frac{1.5}{2}\right)^2 = 0.5625$ multiplied by the load for a 2-inch diameter cell, that is, (6,283) (0.5625) = 3,534 pounds force. To ensure uniformity in leveling the sand surface, for any piston diameter, the piston length must be 3.5 inches (refer to 9.3.4).

9.3.5 Without shaking or jarring the cell, place the cell containing the piston and sand sample in the press.

9.3.6 Apply the required load (dependent on the cell size being used) to attain a stress corresponding to the stress prescribed in Table 3 for the sand size being tested. The cell load should be applied taking 1 minute to reach the prescribed level and that level should be held for 2 minutes. If the recommended load is exceeded, the test should be aborted.

9.3.7 Reduce the load to zero and remove the cell from the press.

9.3.8 Stack the sieve with smaller openings on the pan (refer to 9.3.1) and transfer the cell contents onto the sieve using a small brush to ensure transfer of the sample and all fines. Place the sieve and pan in a Ro-Tap testing sieve shaker (or equivalent) and sieve for 10 minutes.

9.3.9 Weigh to the nearest 0.1 gram the crushed material collected in the pan from the sieve shaker. Calculate, as a percentage, the weight of the crushed material in the pan to the weight of sand sample originally placed in the cell.

9.3.10 Report as percent fines the average of three crush resistance tests conducted according to 9.3.1 through 9.3.9.

9.4 SUGGESTED FINES

Samples of frac sand subjected to the stress specified in Table 3 should not produce more than the suggested maximum fines (percent by weight) as prescribed in Table 3 for the sand size being tested.

10 Recommended Sand Mineralogical Analysis

10.1 TEST PROCEDURE

A qualitative x-ray diffraction test should be conducted on a representative sample of frac sand. The sample should be ground so as to pass through a No. 200 sieve (U.S.A. Sieve Series as defined in ASTM E 11-95) and split into two parts. One sample portion should be used for a powder x-ray and scanned from an angle of 4 degrees to 40 degrees, 2θ (CuK). The other sample portion should be used to prepare an oriented clay slide. This sample should be dispersed in deionized water and allowed to hydrate. If the liquid above the solids is clear, there is little or no clay present and an oriented clay slide will not be necessary. If the liquid above the solids is cloudy, extract a sample of the liquid suspension, place it on a glass slide, and allow it to dry. This glass slide sample should be scanned through an angle of 4 degrees to 14 degrees, 2θ.

10.2 REPORTED RESULTS

The relative peak heights should be recorded and used to estimate the amount of clay present in the sample. Report by mineral type any mineral present in excess of approximately 1 percent.

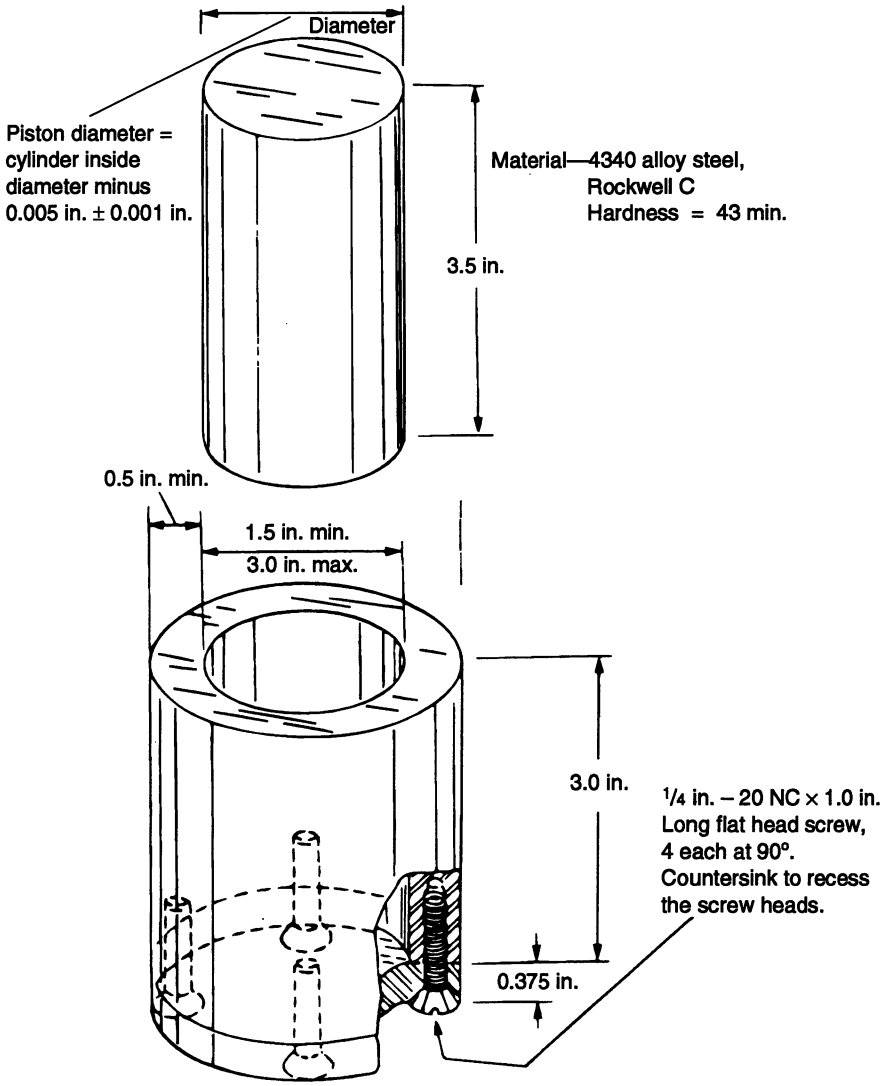


Figure 7—Example Test Cell Frac Sand Crush Resistance Test

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