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APÊNDICE A - API RP 56

Recommended Practices for Testing Sand Used in Hydraulic Fracturing Operations

API RECOMMENDED PRACTICE 56
SECOND EDITION, DECEMBER 1995

American Petroleum Institute

API RP*56 95 ■ 0732290 0551677 115 ■

Recommended Practices for Testing Sand Used in Hydraulic Fracturing Operations

Exploration and Production Department

API RECOMMENDED PRACTICE 56
SECOND EDITION, DECEMBER 1995

American Petroleum Institute

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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).

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.

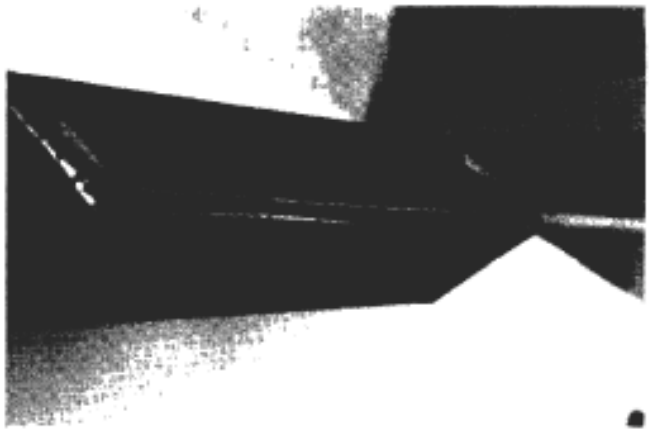
4 Recommended Sand Samples Handling and Storage

4.1 SAMPLE REDUCTION (SACKED MATERIAL)

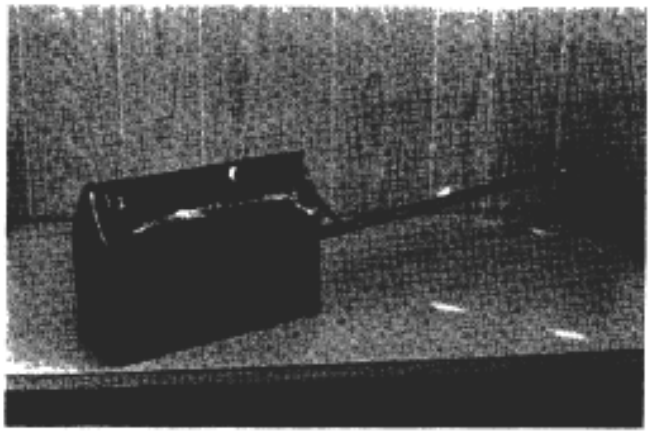
Place the contents of an entire sack of frac sand (approximately 100 pounds) in the sample reducer (refer to Figure 2). Obtain a reduced sample of approximately 6 pounds (approximately 1/16 of the original weight of the total sack's contents).

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

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

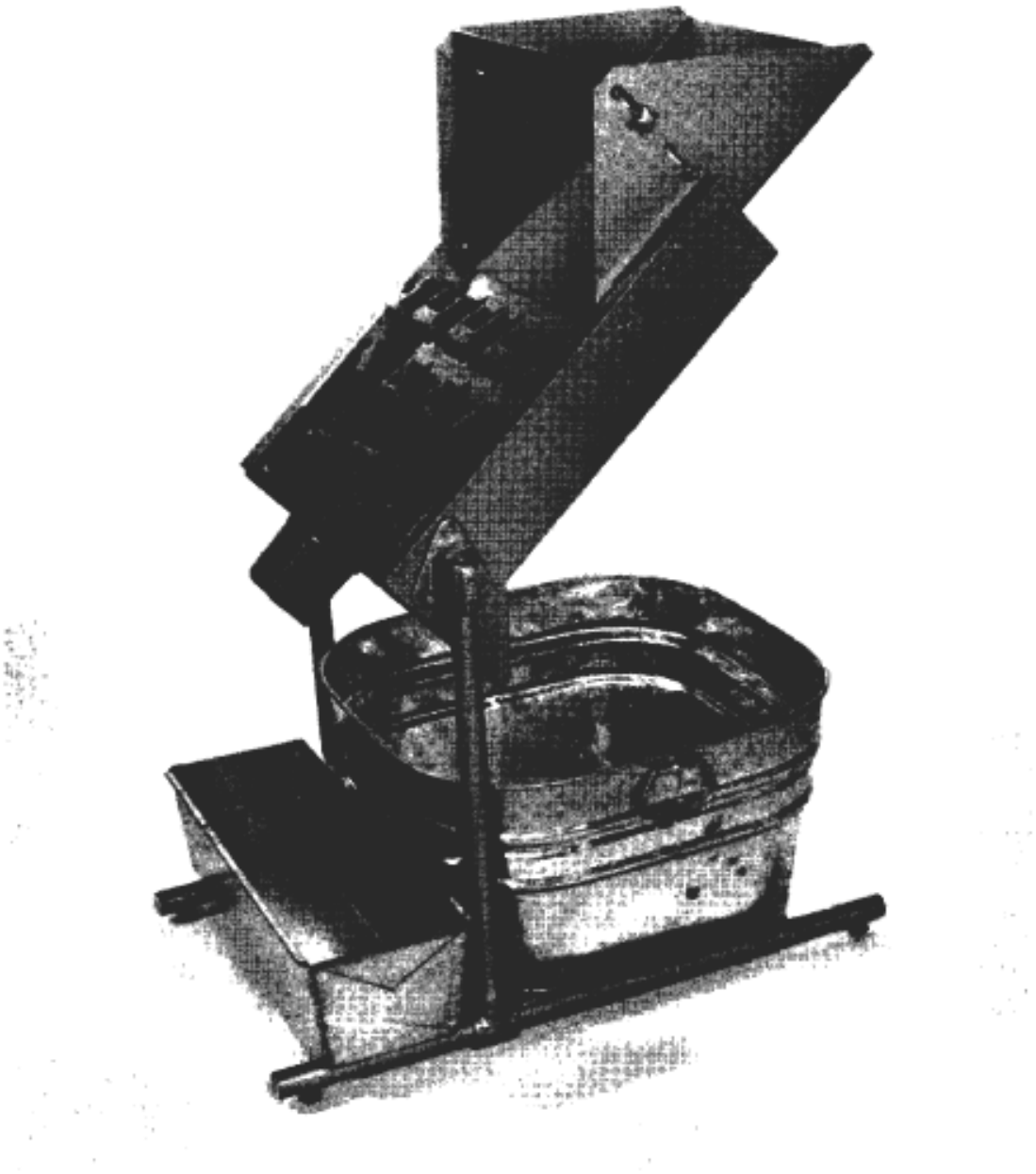
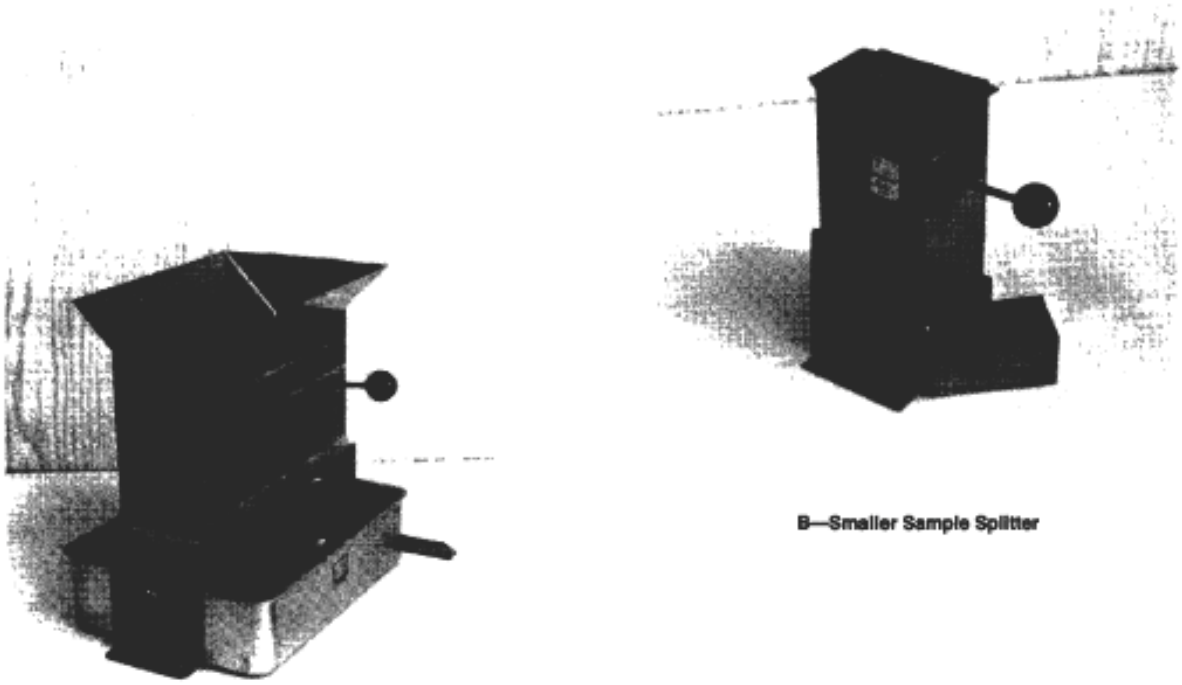


Figure 2—Example Sample Reducer Equipment

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.

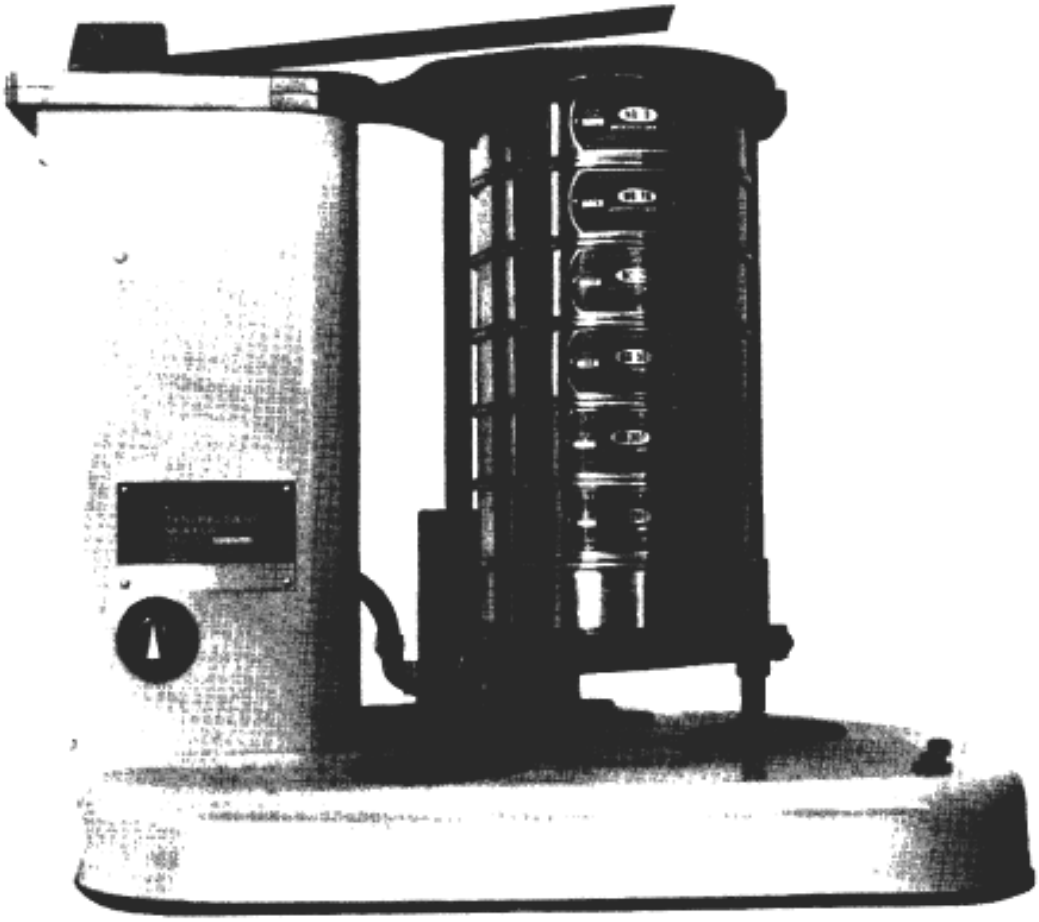


Figure 4—Testing Sieve Shaker and Nest of Six U.S.A. Sieves Plus Pan

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.

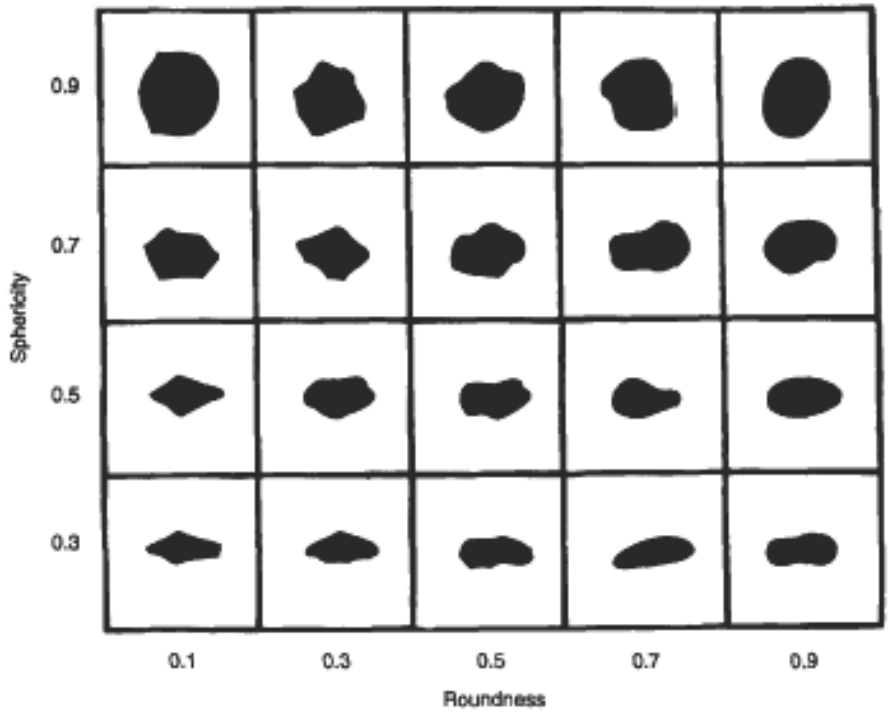


Figure 5—Chart for Visual Estimation of Sphericity and Roundness

From *Stratigraphy and Sedimentation*, Second Edition, Krumbein, W.C. and Sloss, L.L. Copyright © 1951, 1963 by W.H. Freeman and Co., New York, N.Y. All rights reserved.

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 volumetric 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:

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

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.

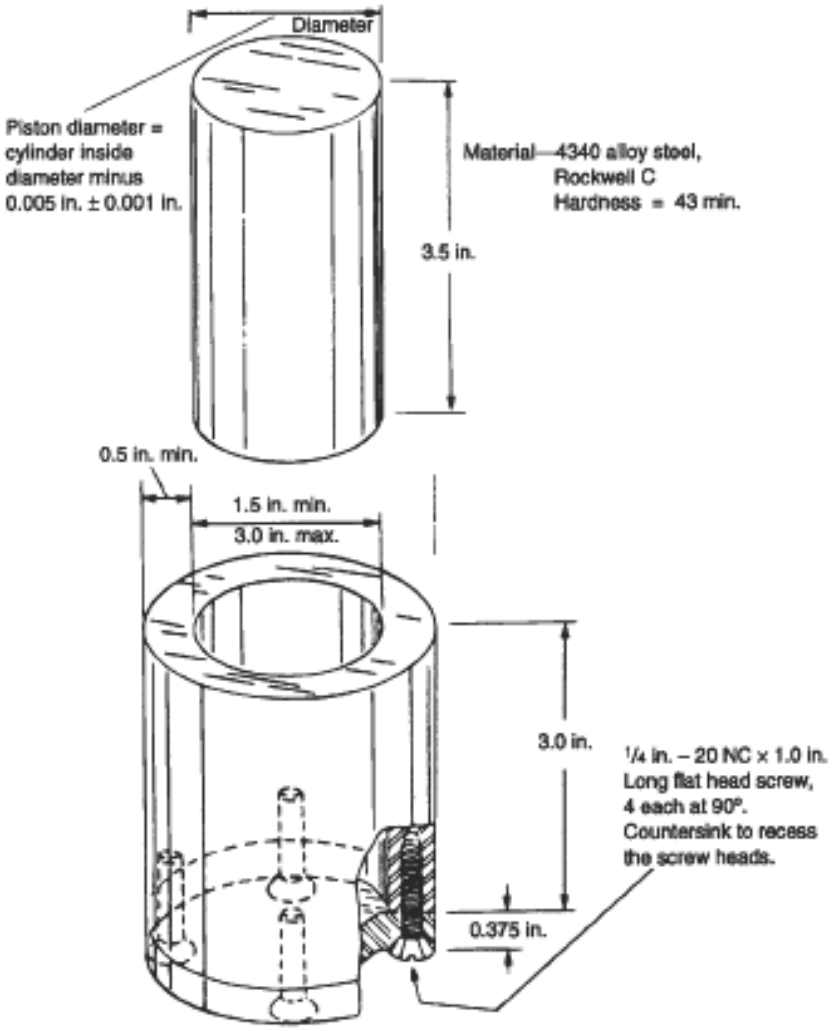


Figure 7—Example Test Cell Frac Sand Crush Resistance Test

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APÊNDICE B - API RP 60

Recommended Practices for Testing High-Strength Proppants Used in Hydraulic Fracturing Operations

API RECOMMENDED PRACTICE 60
SECOND EDITION, DECEMBER 1995

Recommended Practices for Testing High-Strength Proppants Used in Hydraulic Fracturing Operations

Exploration and Production Department

API RECOMMENDED PRACTICE 60
SECOND EDITION, DECEMBER 1995

American Petroleum Institute

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FOREWORD

These recommended practices for testing high-strength proppants (i.e., proppants stronger than sand) were prepared by the API Subcommittee on Evaluation of Well Completion Materials. This publication is a companion to API RP 56: *Recommended Practices for Evaluating Sand Used in Hydraulic Fracturing Operations.* It is published under the jurisdiction of the Executive Committee on Drilling and Production Practices, American Petroleum Institute's Exploration and Production Department.

The recommended tests have been developed to improve the quality of high-strength proppants delivered to the well site. They are for use in evaluating certain physical properties of high-strength proppants used in hydraulic fracturing operations. These tests should enable users to compare the physical characteristics of various high-strength proppants tested under the described conditions and to select materials useful for hydraulic fracturing operations.

The recommended practices 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 a specific situation.

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Recommended Practices for Testing High-Strength Proppants Used in Hydraulic Fracturing Operations

1 Scope

1.1 SCOPE

The purpose of these recommended practices is to provide standard testing procedures for evaluating high-strength proppants, that is, proppants stronger than silica sand.

1.2 OBJECTIVE

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

1.3 TEST PROCEDURES

The use of good, safe laboratory procedures and maintenance and use of good, calibrated equipment is essential to the accuracy and reproducibility of these tests.

2 References

2.1 STANDARDS

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

API

RP 56 *Recommended Practices for Testing Sand Used in Hydraulic Fracturing Operations*

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.H. Freeman and Co., New York, NY.

[1]ASTM, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428.

3 Recommended Proppant Sampling Procedure

3.1 DESCRIPTION

The sampling procedure should provide a representative sample of the high-strength proppant material as provided by the supplier or service company at the time the proppant material is transferred to the bulk transport container or bin. The samples may need to be obtained from three potential sources:

a. From the supplier after the proppant material has been initially screened;
b. From the service company during filling of the transport container with previously sacked or bulk proppant material;
c. On-site at the well where the material is to be used.

When bulk containers are filled from a flowing stream of proppant material, sampling procedures set forth in 3.4 should be applied. If bulk containers are filled using sacked proppant material, sampling procedures set forth in 3.5 should be applied.

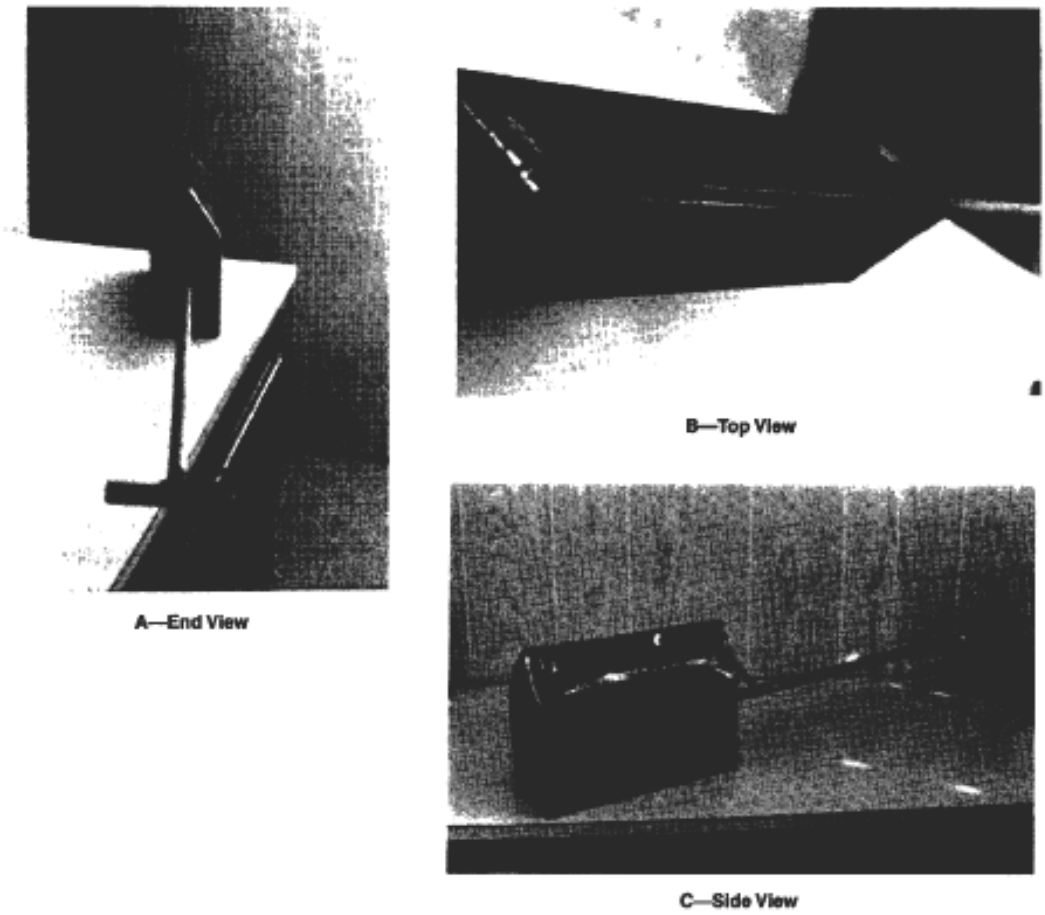
3.2 EQUIPMENT

The following equipment should be used to compile representative proppant material 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 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 that provides simultaneous rotating and tapping action and accepts the sieves specified in Item d. Refer to Figure 4.
f. Scale (minimum of 100 grams capacity with precision of 0.1 gram or better).

3.3 RECOMMENDED NUMBER OF SAMPLES

At the basic source of supply, a minimum of three samples per truck load should be obtained and tested. These basic source-of-supply samples should be combined and used



A—End View

B—Top View

C—Side View

Figure 1—Example Box Sampling Device

as a single sample for subsequent testing operations. For proppant material sampled at the job site, a minimum of one sample should be obtained per 20,000 pounds or fraction thereof of proppant used, with a minimum of five samples per job. These on-site samples should be combined and used as a single sample for subsequent testing operations.

3.4 SAMPLING (BULK MATERIAL)

The sampling device, with its longitudinal axis perpendicular to the falling stream, should be passed at a uniform rate from side to side through the full stream width of moving proppant material as it falls from a conveyor belt into the bulk container. Proppant material should be allowed to flow 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 proppant material to ensure a complete and accurate analysis. The number of samples taken should comply with 3.3. During sampling, the sampling receptacle should be swung completely across the moving proppant 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.

3.5 SAMPLING (SACKED MATERIAL)

Only whole sack samples are to be used for sacked high-strength proppant materials.

4 Recommended Samples Handling and Storage

4.1 SAMPLE REDUCTION (SACKED MATERIAL)

Place the contents of an entire sack of proppant material in the sample reducer (refer to Figure 2). Obtain a reduced sample of approximately $^{1}/_{16}$ of the original weight of the total sack's contents.

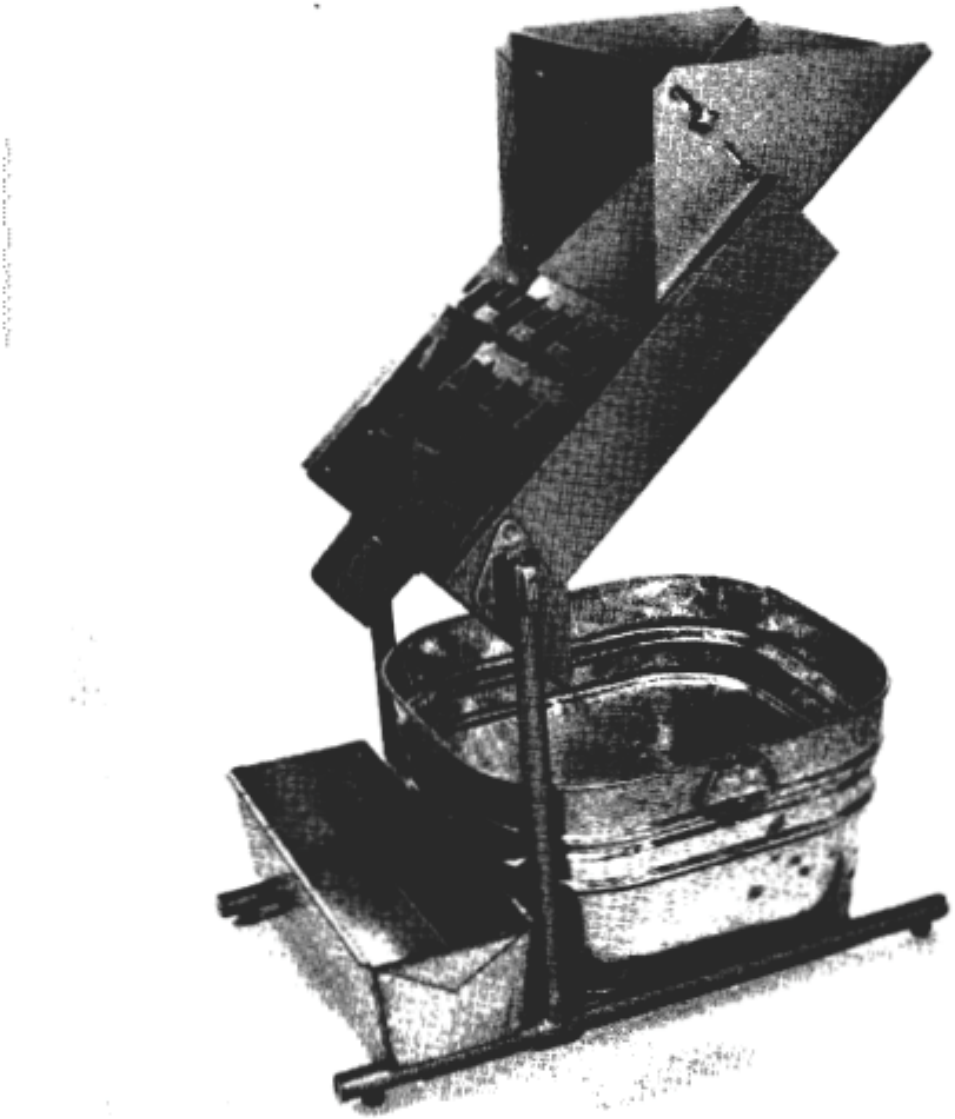
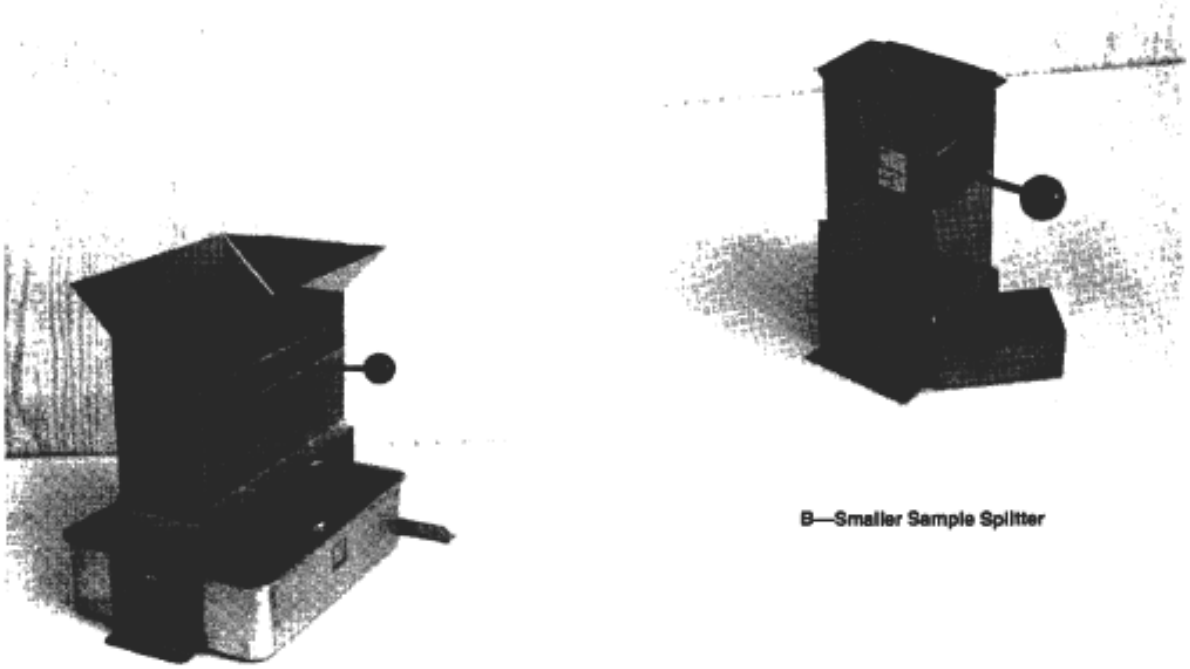


Figure 2—Example Sample Reducer Equipment

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

4.2 SAMPLE SPLITTING

Place the reduced sample obtained according to 4.1 or the sample obtained during bulk material loading operations (refer to 3.4) in the sample splitter (refer to Figure 3) and split the sample to a testing size of approximately 500 grams minimum. Sufficient proppant material 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 high-strength proppant source of supply should maintain written records of all 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 1000 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. Samples and copies of test results should be furnished by the proppant source of supply, on request, to user companies.

5 Recommended Proppant Sizes

5.1 SIEVE ANALYSIS

Stack six recently calibrated U.S.A. Sieves plus a pan in a nest of decreasing sieve opening sizes from top to bottom. Table 1 establishes recommended sieve sizes for use in testing designated recognized high-strength proppant sizes. Using a split sample of approximately 100 grams, obtain an accurate sample weight (6 0.1 gram), pour the sample onto the top sieve, place the nest of sieves plus pan in the testing sieve shaker and shake for 10 minutes. Remove and unload each sieve, being certain to brush each sieve thoroughly with the sieve manufacturer's recommended brush to remove all proppant grains. Establish an accurate weight of proppant retained on each of the six sieves and in the pan. Calculate the percent by weight of the total proppant sample retained on each sieve and in the pan. The cumulative weight should be

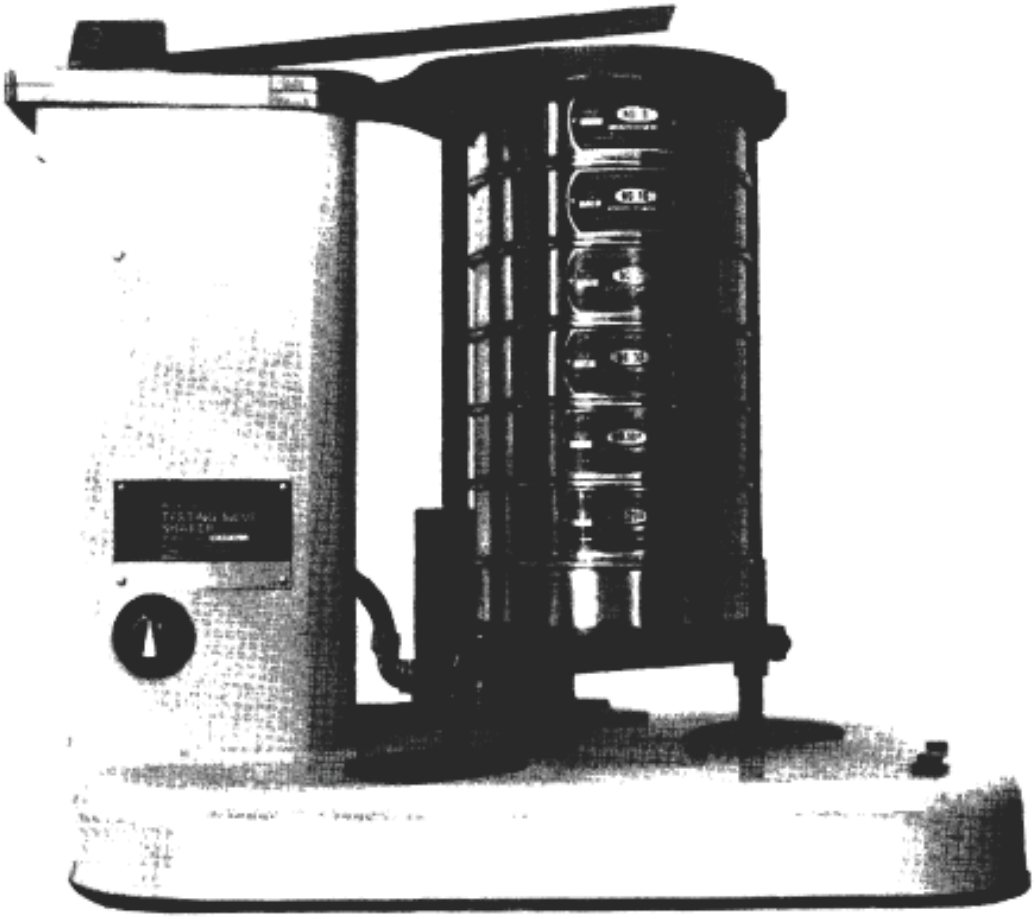


Figure 4—Example of Testing Sieve Shaker Equipment and Nest of Six U.S.A. Sieves Plus Pan

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

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 PROPPANT SIZE

A minimum of 90 percent of the tested proppant sample should fall between the designating sieve sizes, that is, 12/20, 16/20, 20/40, 40/70. Not over 0.1 percent of the total tested proppant sample should be larger than the first sieve size in the nest specified in Table 1 and not over 1.0 percent of the tested sample should be smaller than the last sieve size in the nest specified in Table 1.

6 Proppant Sphericity and Roundness

6.1 GENERAL

Numerous methods have been published to measure and report 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 proppants are sphericity and roundness. Experience has shown that the best results are obtained with these tests when sphericity and roundness are determined in separate reading sets.

Table 1—Recognized High-Strength Proppant Sizes

Proppant Size Designation	12/20	16/20	20/40	40/70
Opening Size (micrometers)	1700/850	1180/850	850/425	425/212
Opening Size (in.)	0.0661/0.0331	0.0469/0.0331	0.0331/0.0165	0.0165/0.0083
Nest of U.S.A. Sieves[a] Recommended for Testing	8	12	16	30
	12	16	20	40
	16	18	30	50
	18	20	35	60
	20	25	40	70
	30	30	50	100
	Pan	Pan	Pan	Pan

[a]U.S.A. Sieve Series as defined in ASTM E 11-95: *Specification for Wire-Cloth Sieves for Testing Purposes.*

6.2 SPHERICITY

Particle sphericity is a measure of how close a proppant 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 visual estimation of sphericity and roundness (refer to Figure 5). A proppant 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 6.5.3). Sphericity of each grain should be determined, recorded, and an average sphericity obtained for the sample.

6.3 ROUNDNESS

Grain roundness is a measure of the relative sharpness of grain corners or of grain curvature. Evaluation of proppant grain roundness should be made on the same sample as that used for sphericity determination (refer to 6.2). Roundness of each grain should be determined, recorded, and an average roundness obtained for the sample.

6.4 RECOMMENDED SPHERICITY AND ROUNDNESS

High-strength proppants should have an average sphericity of 0.7 or greater and an average roundness of 0.7 or greater.

6.5 ALTERNATIVE METHOD FOR DETERMINING AVERAGE SPHERICITY AND ROUNDNESS

6.5.1 Use of Photomicrographs

Photomicrographs of a representative proppant sample may be used to provide identical suitably enlarged reproductions for use to obtain the average sphericity and roundness of the proppant sample.

6.5.2 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 proppant, place a monolayer of grains on a flat, resilient surface. Prepare a specimen mount using transparent, double-sided adhesive tape and press the mount to the sample to affix a monolayer of proppant grains. Follow standard equipment procedures for coating, magnifying, and photographing the proppant sample.

6.5.3 Recommended Magnification for Proppant Sizes

For designated proppant sizes, the following magnification is suggested:

Proppant Size	Photomicrograph Magnification
12/20	15×
16/20	30×
20/40	30×
40/70	40×

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

6.5.4 Determination of Proppant Sphericity

Using the photomicrograph from 6.5.2 and the visual comparator chart (refer to Figure 5), determine and record the sphericity of all proppant grains within the photomicrograph. Using this information, determine the average sphericity for the proppant sample. Refer to 6.4 for proppant sphericity recommendations.

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

6.5.5 Determination of Proppant Roundness

Using the photomicrograph from 6.5.2 and the visual comparator chart (refer to Figure 5), determine and record the roundness of all proppant grains within the photomicrograph. Using this information, determine the average roundness for the proppant sample. Refer to 6.4 for proppant roundness recommendations.

7 Acid Solubility Considerations

7.1 GENERAL

A test to determine the solubility in acid of high-strength proppants has not been included in this standard because of insufficient data upon which to base a recommendation. However, this omission does not imply the unimportance of acid solubility of high-strength proppants. For example, refer to Cheung[3] for an evaluation of such solubility. Rather, exposing a propped fracture to acid, particularly one containing a mixture of hydrofluoric and hydrochloric acids, may result in dissolution of part of the proppant, a deterioration in propping capabilities, and a reduction in fracture conductivity in the zone contacted by such acid. The loss of fracture conductivity near the wellbore may cause a dramatic reduction in well productivity, as has been demonstrated by Raymond and Binder.[4]

7.2 ACID SOLUBILITY TEST CAUTIONS

While exposure of high-strength proppants to acid is generally discouraged, should such exposure be considered, it should not be undertaken without some knowledge of the solubility of the proppant in the acid with which it is to be contacted. One way of determining proppant solubility in acid is described in API RP 56: *Recommended Practices for Testing Sand Used in Hydraulic Fracturing Operations*. Such an evaluation represents only a first step, however. If the proppant is found to be appreciably soluble in the chosen acid at the expected temperature, pressure, and time of exposure, then the critical test is to determine how much fracture conductivity is reduced by such acid exposure. The latter requires work to evaluate fracture conductivity.

8 Recommended Proppant Crush Resistance Test

8.1 GENERAL

Proppants vary in composition, density, and strength. The following test is useful for determining and comparing the crush resistance of proppants. A series of crush resistance tests are conducted on samples of proppant to determine the stress at which the proppant material shows excessive fines generation. Tests are conducted on samples which have been sieved so that all particles tested are within the specified size range. Four specific stress levels, 7,500; 10,000; 12,500; and 15,000 pounds per square inch, are used in the recommended test. The amount of proppant material crushed at each stress level is measured. Evaluation of test results should provide indications of the stress level where proppant crushing is excessive and the maximum stress to which the proppant material should be subjected.

8.2 EQUIPMENT AND MATERIALS

The following equipment and materials are suggested for conducting the proppant crush resistance test:

a. Proppant sample.
b. Press with the capacity to apply the load required to accomplish the stress levels set forth in Table 2. *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 proppant crush resistance test as described in Figure 6, or equivalent. The piston length should be 3.5 inches regardless of the diameter of the piston used in the cell. Permissible piston diameter ranges from 1-1/2 inches to 3 inches.
d. Pan and two U.S.A. Sieves of the mesh size opening for the specified proppant size range, for example, the No. 12 and No. 20 sieves for use with 12/20 proppant and the No. 20 and No. 40 sieves for use with 20/40 proppant.
e. Balance for weighing proppant sample to 0.1 gram precision or better.
f. Testing sieve shaker. Refer to 3.2, Item e and Figure 4.

8.3 RECOMMENDED TEST PROCEDURE

8.3.1 Determine the bulk density of the proppant sample using the recommended procedure in Section 9.

8.3.2 The volume of proppant to be used in a test is equivalent to the volume occupied by 4 pounds of 20/40 frac sand per square foot in the test cell piston area. Thus, each test requires 1.22 cubic centimeters of proppant per square centimeter[5] of test cell piston area. Calculate the weight of proppant material needed for each test (to the nearest 0.1 gram) as follows:

[3]Cheung, S. K., "Effect of Acids on Gruvels and Proppants," SPE 13842, presented at the SPE 1985 California Regional Meeting, held in Bakersfield, California, March 27–28, 1985, Society of Petroleum Engineers, Richardson, Texas.

[4]Raymond, L. R., and Binder, G. G., Jr. "Productivity of Wells in Vertically-Fractured, Damaged Formations," *Journal of Petroleum Technology* (January 1967) 120-130, Society of Petroleum Engineers, Richardson, Texas.

[5]This volume is calculated as follows:

1. The bulk density of 20/40 proppant frac sand averages 100 lb/ft^3 or 1.60 g/cm^3.

2. 4 lb/ft^2 = 1.95 g/cm^3.

3. Volume of 20/40 frac sand required per unit of piston area of the test cell is 1.95/1.60 = 1.22 cm^3 of proppant per cm^2. In a 2-in. inside diameter test cell, the volume needed is 24.7 cm^3.

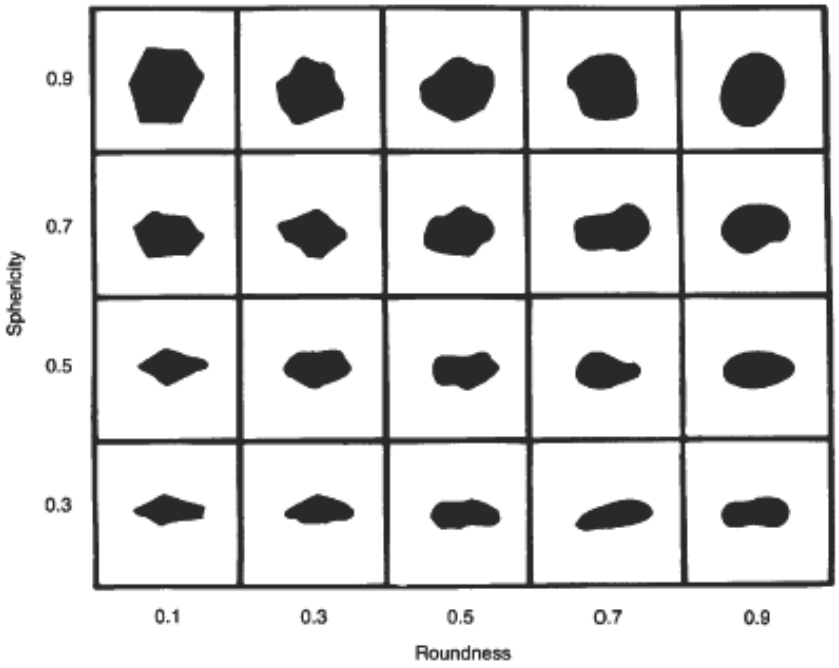


Figure 5—Chart for Visual Estimation of Sphericity and Roundness

From *Stratigraphy and Sedimentation*, Second Edition, Krumbein, W.C., and Sloss, L.L. Copyright © 1951, 1963. Reprinted with the permission of W.H. Freeman and Co., New York, New York. All rights reserved.

$$W_p = 6.18 \times \rho_b \times D^2 \quad (1)$$

Where:

W_p = Weight of proppant, g

ρ_b = Proppant bulk density, g/cm3

D = Test cell inside diameter, in.

$$6.18 = \frac{1.22(2.54)^2\pi}{4}$$

8.3.3 Stack the two U.S.A. Sieves and pan described in 8.2.d, with the sieve having the larger opening size on top. Pour a sufficient quantity of proppant material on the top sieve to provide in the test cell (refer to Figure 6) a concentration of 1.22 cubic centimeters of proppant per square centimeter of the mesh size specified for the sample being tested. Place the sieve stack in the testing sieve shaker (refer to 3.2, Item e and Figure 4) and sieve for 10 minutes.

8.3.4 Discard all proppant material retained on the top screen and pan. Use only proppant material retained on the lower screen.

8.3.5 Sieve sufficient material so that eight tests may be conducted (two tests each at four stress levels).

8.3.6 Weigh a sample of the sieved material to the calculated weight (refer to 8.3.2) and pour the weighed sample into the test cell, constantly moving the source of the proppant stream so that the surface in the test cell is as level as possible.

8.3.7 Level the surface of the proppant in the cell. This is 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 proppant in the cell using the piston, the piston length should be 3.5 inches regardless of the diameter of the piston used in the cell (refer to 8.2, Item c).

8.3.8 Without shaking or jarring the cell, place the cell containing the piston and proppant sample in the press.

Table 2—Suggested Fines Limit According to Proppant Size for Stated Stress Levels[a]

Proppant Size Designation	Suggested Maximum Allowable Fines (% by weight)
12/20	25
16/20	25
20/40	10
40/70	8

[a]Suggested test stress levels are 7,500; 10,000; 12,500; and 15,000 psi.

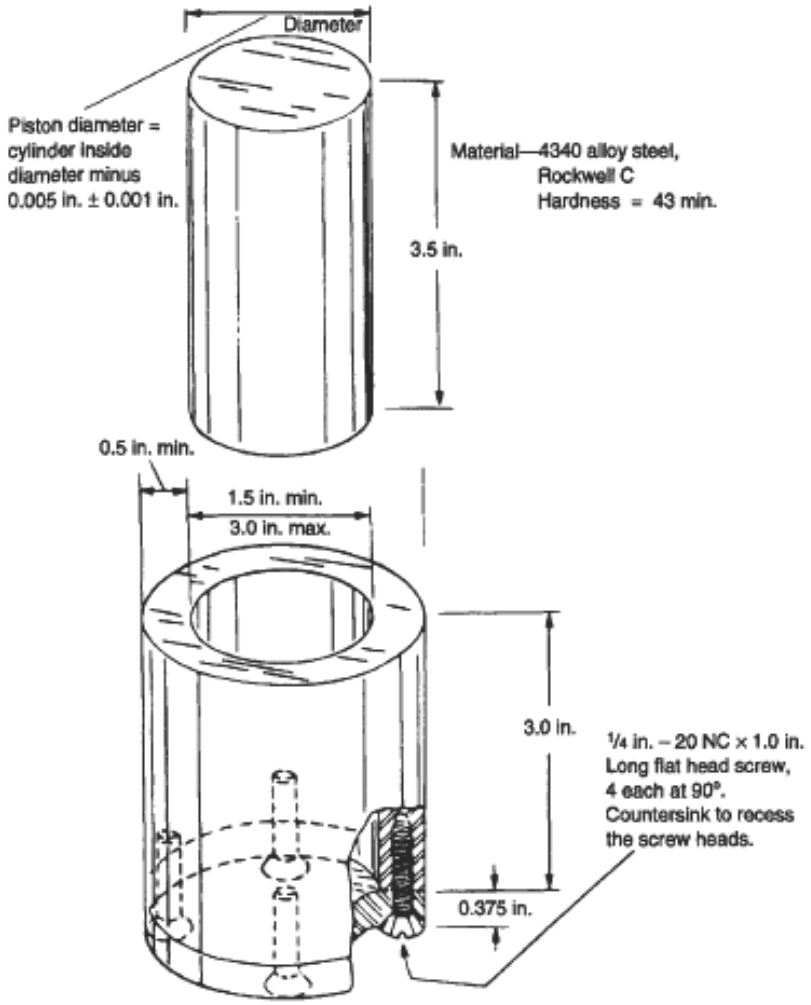


Figure 6—Example Test Cell for Proppant Crush Resistance Test

8.3.9 Using Equation 2, determine the force (pounds) required on the press to attain the prescribed stress on proppant samples:

$$F = \frac{S(3.14)D^2}{4} \qquad (2)$$

Where:

F = Force required on test cell, lb

S = Stress on proppant sample, psi

D = Test cell inside diameter, in.

The complete series consists of subjecting two samples to a stress of 7,500 pounds per square inch, two samples to a stress of 10,000 pounds per square inch, two samples to a stress of 12,500 pounds per square inch, and two samples to a stress of 15,000 pounds per square inch, making a total of eight tests.

8.3.10 The required cell load should be applied at a uniform loading rate to attain the stress levels shown in Table 3, 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.

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

8.3.12 Stack the two U.S.A. Sieves and pan with the screen with the largest screen openings on top (refer to 8.3.3). Transfer the cell contents onto the top sieve using the

sieve manufacturer's recommended brush to ensure transfer of all the sample and all fines. Place the sieve stack in a testing sieve shaker and shake for 10 minutes.

8.3.13 Weigh the crushed material from the pan to the nearest 0.1 gram. Using Equation (3), calculate and report fines generated as a percentage of the weight of proppant sample placed in the cell. Each proppant sample tested should be run in duplicate at each stress level and the results averaged.

$$f = \frac{100 W_f}{W_p} \tag{3}$$

Where:

f = Fines generated in the test, percent (%)

W_f = Weight of fines, g

W_p = Weight of proppant, g

8.4 SUGGESTED MAXIMUM FINES

It has been determined that fines generated by proppant crushing will affect propped fracture performance and that long-term migration of these fines may be damaging to permeability. However, there are only limited data available on specific effects (short- and/or long-term) of fines on performance of propped fractures and on the severity of damage from these fines.

Conductivity of propped fractures is expected to decline when proppant crush fines are excessive. As a general guide, suggested fines generated in the recommended crush resistance tests should not exceed values shown in Table 2. The stress level at which the generated fines exceed the suggested limits shown in Table 2 should be used to evaluate closure stress levels where proppant performance may be adversely affected by crushing.

Note: These values for "suggested maximum allowable fines" (refer to Table 2) should be considered as broad guidelines. It is recommended that additional fracture conductivity tests be made with the proppant under appropriate stress to estimate propped fracture performance.

Each proppant sample should be tested at a minimum of four stress levels, 7,500; 10,000; 12,500; and 15,000 psi. Other stress levels may be used, by specific agreement between user and supplier, to more clearly and specifically define proppant crush behavior.

8.5 VARIABILITY OF CRUSH RESISTANCE TEST RESULTS

Crush resistance test results are subject to variability. Tests performed within a given laboratory by the same personnel, using the same equipment, and following the same test procedures have produced more consistent comparative results. However, testing between laboratories where different personnel, equipment, and varying test procedures can affect results has not produced the same degree of data reliability or repeatability. Variances in crush resistance data should be considered when interpreting and using the suggested limits shown in Table 2.

9 Recommended Procedures for Determining Proppant Bulk Density, Apparent Density, and Absolute Density

9.1 GENERAL

The bulk density and absolute density are important properties of proppants. Bulk density describes the weight of proppant that will fill a unit volume, and includes both proppant and porosity void volume. It is used to determine the weight of a proppant needed to fill a fracture or a storage tank. Apparent density and absolute density are usually very nearly the same. Apparent density includes internal porosity of a particle as part of the particle volume. It is measured with a low viscosity fluid that wets the particle surface. On the other hand, the absolute density excludes internal/interconnected porosity as a part of the particle volume.

9.2 BULK DENSITY

9.2.1 Equipment and Materials

The following are needed to obtain the bulk density of proppant samples:

Table 3—Equivalent Load on Cell Versus Stress on Proppant Pack

Stress on Proppant Pack (psi)	Load on Cell (lbs force)
7,500	23,562
10,000	31,416
12,500	39,270
15,000	47,124

Note: Indicated cell 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, for 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 10,000 pounds per square inch on a proppant pack requires a load of (31,416)(2.25) = 70,686 pounds force. Similarly, a test cell with a 1.5-inch diameter piston would require a cell load of $(31{,}416)\left(\frac{1.5}{2}\right)^2 = 17{,}672$ pounds force to achieve 10,000 pounds per square inch stress on a proppant pack.

a. Analytical balance, 0.01 gram precision or better.
b. 100-milliliter volumetric flask [100 ml ≃ 100 cm^3 at 75°F (24°C)].
c. Proppant sample, dry and free-flowing.
d. Wide-mouth funnel, stem to fit inside the volumetric flask.

9.2.2 Procedure

The following procedure is suggested for determining bulk density of proppants.

a. Weigh the clean, dry 100-milliliter volumetric flask to 0.01 gram precision using the analytical balance.
b. Place the funnel in the neck of the volumetric flask and fill it with proppant to the 100 milliliters mark. Do not shake the flask or tamp the proppant.

Note: This is a critical step and must be done the same way by each person measuring bulk density.

c. Weigh the volumetric flask containing proppant to 0.01 gram precision.
d. Calculate the proppant bulk density using the following equation:

$$\rho_b = \frac{W_{f,p} - W_f}{100} \quad (4)$$

Where:

ρ_b = Proppant bulk density, g/cm^3
$W_{f,p}$ = Weight of flask and proppant (step c), g
W_f = Weight of flask (step a), g

e. Report proppant bulk density in g/cm^3 and lb/ft^3. (Note: lb/ft^3 = g/cm^3 × 62.4)

9.3 APPARENT DENSITY (MEASURED IN KEROSINE OR WATER)

9.3.1 Equipment and Materials

The following are needed to determine apparent density of proppants in kerosine or water:

a. Analytical balance, 0.01 gram precision.
b. Weighing dish.
c. 25-milliliter volumetric flask, or pycnometer [25 ml = 25 cm^3 at 75°F (24°C)].
d. Test liquid, kerosine or equivalent; water with surfactant [0.1 percent of an ethylene oxide (9–10 mole) adduct of nonylphenol, or equivalent].
e. Proppant sample.
f. Wide-mouth funnel, stem to fit inside the volumetric flask.

9.3.2 Procedure

The following procedure is suggested for determining the apparent density of proppants.

a. Weigh the clean, dry volumetric flask or pycnometer to 0.01 gram precision.
b. Carefully fill the volumetric flask or pycnometer to the fill line with test fluid at ambient temperature. Make certain that no air bubbles are trapped in the liquid and that all liquid has been wiped off the outer surface of the flask or pycnometer.
c. Weigh the filled flask and liquid to 0.01 gram precision.
d. Tare the weighing dish, then add approximately 10 grams of proppant sample and weigh the dish and sample to 0.01 gram precision.
e. Pour out approximately one-half the volume of liquid in the volumetric flask and transfer the weighed proppant sample from the weighing dish to the flask or pycnometer. A funnel that fits into the neck of the volumetric flask should be used.
f. Carefully add sufficient test liquid at ambient temperature to the flask or pycnometer and fill to the fill line. Rotate the flask about its vertical axis until all air bubbles have been dislodged from the proppant. Refill with test liquid to the fill line, if necessary, and wipe off any test liquid on the flask surface.
g. Weigh the flask containing proppant and test liquid to 0.01 gram precision.
h. Calculate the test liquid density and apparent density of the proppant as follows:

$$D_l = \frac{W_{f,l} - W_f}{25} \quad (5)$$

Where:

D_l = Test liquid density, g/cm^3
$W_{f,l}$ = Weight of flask filled with test liquid (step c), g
W_f = Weight of empty flask (step a), g
25 = Volume of pycnometer, cm^3

$$D_p = \frac{W_p}{25 - \left(\frac{W_{f,l,p} - W_f - W_p}{D_l}\right)} \quad (6)$$

Where:

D_p = Apparent density of proppant, g/cm^3
W_p = Weight of proppant (step d), g
$W_{f,l,p}$ = Weight of flask, liquid, and proppant (step g), g
W_f = Weight of empty flask (step a), g
D_l = Test liquid density, g/cm^3
25 = Volume of pycnometer, cm^3

i. Report apparent density in g/cm^3 and lb/ft^3 and denote the liquid used in the test.

9.4 ABSOLUTE DENSITY

9.4.1 Equipment and Materials

A schematic of apparatus for measuring absolute density of proppant materials is shown in Figure 7. The following equipment and materials are needed for construction and operation:

a. Test gauge (0–100 pounds per square inch range) with at least 0.5 percent precision.
b. Three ball valves.
c. Reference cell (20 to 50 cubic centimeters) sample cylinder is satisfactory.
d. Sample cell (40 to 100 cubic centimeters cell is satisfactory).

Note: The sample cell used should be approximately twice the size of the reference cell for maximum accuracy.

e. Copper or stainless steel tubing ($^1/_8$-inch or $^1/_4$-inch diameter) with appropriate required fittings.
f. Analytical balance (0.01 gram precision).
g. High pressure gas source (air, nitrogen, or helium is satisfactory).
h. Volumetric flask (100 milliliters).
i. Pasteur pipette.
j. Proppant sample, dry and free-flowing.
k. Weighing dish.

The listed equipment is commercially available. However, for ease of operations, a sample cell with a removable, wide-mouth top can be constructed.

Note: Commercially available air comparison pycnometer equipment, or equivalent, is available to quickly and efficiently measure absolute density of proppant samples. If this equipment is used, follow the equipment manufacturer's instructions for calibration and operation of the equipment to determine absolute density of proppant samples.

9.4.2 Calibration

The following procedure is used for measuring the volume of the reference cell and sample cell (refer to Figure 7).

a. Weigh to 0.01 gram precision the dry and clean reference cell with valve V_3 attached.
b. Carefully fill the reference cell and valve V_3 (up to the ball of the valve) with water. A Pasteur pipette can be used in this operation.
c. Weigh the water-filled reference cell and valve V_3 to 0.01 gram precision.
d. Empty the water from the reference cell and valve and dry thoroughly. Disassembly of the equipment may be required to effect drying.
e. Weigh a dry 100-milliliter volumetric flask to 0.01 gram precision.
f. Fill the volumetric flask with water to the marked line (bottom of the meniscus just touching the line).
g. Weigh the water-filled volumetric flask to 0.01 gram precision.
h. Calculate the volume of the reference cell as follows:

$$V_r = \frac{100\,(W_{rw} - W_r)}{(W_{fw} - W_f)} \tag{7}$$

Where:

V_r = Volume of reference cell, cm^3
W_{rw} = Weight of reference cell filled with water, g
W_r = Weight of empty reference cell, g
W_f = Weight of empty volumetric flask, g
W_{fw} = Weight of volumetric flask filled with water, g

i. Connect the reference cell to the absolute density apparatus.
j. With valve V_1 closed and valves V_2 and V_3 open, pressurize the system to approximately 100 pounds per square inch; record this pressure accurately.
k. Close valves V_3 and V_2; open valve V_1.
l. When the pressure, as indicated by the pressure gauge, returns to zero (atmospheric pressure), close valve V_1.
m. Open valve V_3 and record the stabilized pressure accurately.
n. Open valve V_1 to depressurize the apparatus.
o. Calculate the volume of the sample cell as follows:

$$V_s = V_r\left(\frac{P_i + P_{atm}}{P_f + P_{atm}} - 1\right) \tag{8}$$

Where:

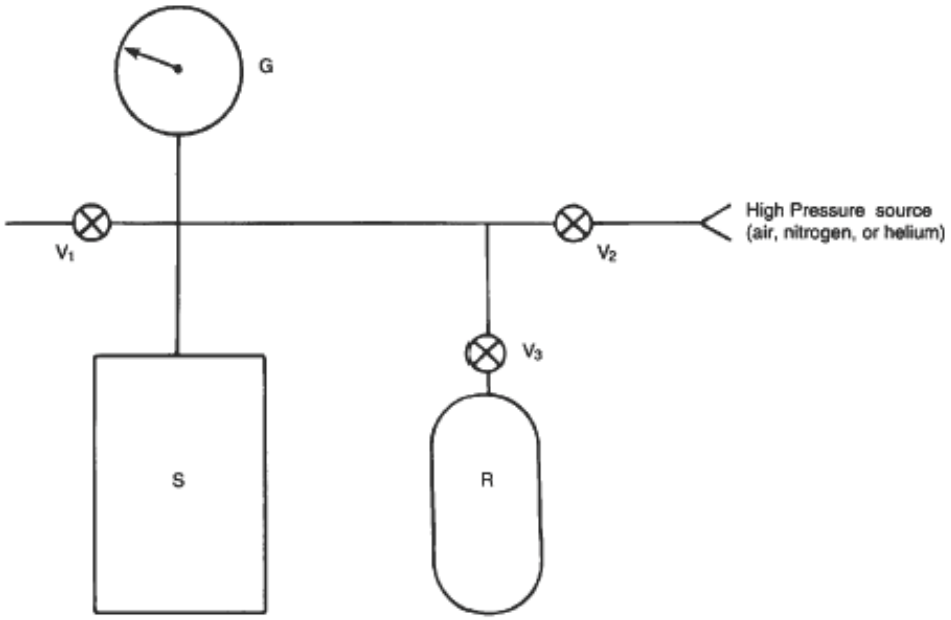
V_s = Volume of the sample cell, plumbing, and gauge, cm^3
V_r = Volume of reference cell, cm^3
P_{atm} = Atmospheric pressure, psi
P_i = Initial pressure of the reference cell, psi
P_f = Final pressure of the reference and sample cells, psi

Note: V_s includes the plumbing and gauge volumes. The plumbing and gauge volume should be as small as practicable.

9.4.3 Measurement of Absolute Density

After the reference cell volume (V_r) and sample cell volume (V_s) are determined, absolute density of the proppant sample should be determined using the following procedure.

a. Obtain sufficient proppant to fill at least 80 percent of the sample cell volume. Weigh this proppant sample to 0.01 gram precision.
b. Carefully place the proppant material in the sample cell and attach the cell to the absolute density apparatus (refer to Figure 7).
c. Close valve V_1 and open valves V_3 and V_2.



G = 0–100 psi pressure guage (±0.5 % precision)
S = Sample cell
R = Reference cell
V_1, V_2, V_3 = Ball valves

Figure 7—Example Apparatus for Measuring Proppant Absolute Density

d. Pressure the apparatus to approximately 100 pounds per square inch and record the pressure (P'_i) accurately.
e. Close valves V_3 and V_2.
f. Slowly open valve V_1 and allow the pressure, as indicated by the pressure gauge, to return to zero (atmospheric pressure).

CAUTION: If the pressure is relieved too rapidly, the proppant material will be displaced out of the sample cell.

g. Close valve V_1.
h. Open valve V_3.
i. After the pressure has stabilized, record it accurately as P'_f.
j. Slowly open valve V_1 to return the apparatus to atmospheric pressure.
k. Calculate the absolute density of the proppant material as follows:

$$\rho_p = \frac{W_p}{V_s - V_r\left(\frac{P'_i + P_{atm}}{P'_f + P_{atm}} - 1\right)} \quad (9)$$

Where:

ρ_p = Absolute density of proppant material, g/cm³
W_p = Proppant sample weight, g
V_s = Volume of the sample cell, plumbing, and gauge, cm³
V_r = Volume of reference cell, cm³
P'_i = Initial pressure of the reference cell, psi
P_{atm} = Atmospheric pressure, psi
P'_f = Final pressure of the reference and sample cells, psi

l. Report the proppant absolute density in g/cm³, as well as the gas used in the test measurements, for example, air.

9.4.4 Accuracy

The pressure gauge should be checked with a dead weight tester at least once every six months. If a gauge with 0.5 percent precision or better is used, absolute density measurements of better than 3.5 percent precision are possible. If a gauge with 0.1 percent precision or better is used, absolute density measurements with precision of 1 percent or better are possible.

APPENDIX A—DERIVATION OF EQUATION (8)

Ideal Gas Law, $P_1V_1 = P_2V_2$

$P_f^\circ V_f = P_i^\circ V_i$

Where:

$P_f^\circ = P_f + P_{atm}$

$V_f = V_s + V_r$

$V_i = V_r$

$P_i^\circ = P_i + P_{atm}$

$(P_f + P_{atm})(V_s + V_r) = (P_i + P_{atm})V_r$

$$V_s + V_r = \frac{V_r(P_i + P_{atm})}{P_r + P_{atm}}$$

$$V_s = V_r\left(\frac{P_i + P_{atm}}{P_f + P_{atm}} - 1\right) \qquad \text{(Equation 8)}$$

DERIVATION OF EQUATION (9)

$P_f^\circ V_f = P_i^\circ V_i$

Where:

$P_f^\circ = P'_f + P_{atm}$

$V_f = V_r + V_S - V_p$

$V_i = V_r$

$P_i^\circ = P'_i + P_{atm}$

$(P'_f + P_{atm})(V_r + V_s - V_p) = (P'_i + P_{atm})V_r$

$$V_p = V_s - V_r\left(\frac{P'_i + P_{atm}}{P'_f + P_{atm}} - 1\right)$$

Since density, ρ_p, is given by

$$\rho_p = \frac{\text{Proppant wt.}}{\text{Proppant vol.}} = \frac{W_p}{V_p}$$

Then,

$$\rho_p = \frac{W_p}{V_s - V_r\left(\frac{P'_i + P_{atm}}{P'_f + P_{atm}} - 1\right)} \qquad \text{(Equation 9)}$$

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APÊNDICE C - API RP 61

Recommended Practices for Evaluating Short Term Proppant Pack Conductivity

API RECOMMENDED PRACTICE 61 (RP 61)
FIRST EDITION, OCTOBER 1, 1989

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RECOMMENDED PRACTICES FOR EVALUATING SHORT TERM PROPPANT PACK CONDUCTIVITY

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RECOMMENDED PRACTICES FOR EVALUATING SHORT TERM PROPPANT PACK CONDUCTIVITY

FOREWORD

a. These recommended practices were prepared by the Task Group on Conductivity Testing of Proppants under the API Subcommittee on Evaluation of Well Completion Materials. This publication is under jurisdiction of the Executive Committee on Drilling and Production Practices, American Petroleum Institute's Production Department.

b. The tests and test apparatus recommended herein have been developed to establish standard procedures and conditions for use in evaluating the short term conductivity of various fracture proppant materials under laboratory conditions. These suggested tests will enable users to compare the conductivity characteristics under the specifically described test conditions. The test results can aid users in selecting proppant materials for use in hydraulic fracturing operations.

c. ***CAUTION:*** The testing procedures in this publication are not designed to provide absolute values of proppant conductivity under downhole reservoir conditions. Long-term test data (refer to Appendix D, References 1-8) have shown that time (within a few days), elevated temperature, fracturing fluid residues, embedment, and formation fines may reduce fracture proppant pack conductivity by up to 90% or more. A recommended testing procedure to address longer term conductivity reduction may be considered in future API work.

d. 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 sound judgment will be required for their application to fit a specific situation.

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RECOMMENDED PRACTICES FOR EVALUATING SHORT TERM PROPPANT PACK CONDUCTIVITY

SECTION 1
GENERAL

1.1 Objective. The objective of these recommended practices is to establish suggested standard test apparatus, test conditions, and procedures for use in evaluating the short term conductivity of fracture proppants under laboratory conditions. These recommended procedures may be used to evaluate and compare the conductivity of packs of proppant samples under laboratory conditions but are not intended for use in obtaining absolute values of proppant pack conductivities under downhole reservoir conditions. The effects of fines, formation temperature, formation hardness, resident fluids, time, or other factors are beyond the scope of this recommended procedure.

1.2 Test Procedures. This procedure uses deionized or distilled water as the test fluid. Normally, ambient temperature [75 F(24 C)] is employed in the test. Fluids other than deionized water can be utilized to evaluate different characteristics of proppant materials, and, therefore, could be expected to produce differing test results. Tests using other fluids or temperatures may be of value in evaluating proppant pack conductivity. These tests may be conducted by agreement between user and supplier.

1.3 Discussion. In these test procedures, a closure stress is applied across a test unit for sufficient time to allow the proppant sample bed to reach a semi-steady state condition (refer to Par. 2.6). Test fluid is forced through the proppant bed. Proppant pack width, differential pressure, and flow rates are measured at each stress level as test fluid is forced through the proppant bed. Proppant pack permeability and conductivity are calculated. Three different flow rates are tested at each closure stress; an average of data at these three flow rates is reported. At stipulated flow rates and ambient temperature conditions, no appreciable non-darcy flow or inertial effects should be encountered. After completing three flow rates at a closure stress level, the closure stress is increased to a new level; sufficient time is allowed for the proppant bed to reach a semi-steady state condition, and three flow rates are introduced to gather data required to determine proppant pack conductivity at this stress level. The procedure is repeated until all desired closure stresses and flow rates have been evaluated.

SECTION 2
RECOMMENDED CONDUCTIVITY TEST

2.1 Test Medium. This test uses deionized or distilled water as the test fluid. This test procedure is used for evaluating proppant pack conductivity under laminar (darcy) flow conditions. To achieve accurate conductivity measurements, it is essential that single phase flow occur under laminar (darcy) flow conditions.

2.2 Equipment and Materials. The following equipment and materials should be used in this test procedure.

1. **Test Unit.** The test unit should be a linear flow design with a 10 square inch (64.5 cm^2) proppant and bed area. Figures 2.1, 2.2, 2.3, and 2.5 illustrate details of the recommended test unit. The pistons (or platens), platen shims, and test chamber should be constructed of 316 stainless steel material. Figure 2.4 is a schematic diagram of the flow path(s) through the test unit. Filters for the test unit may be constructed using 0.125 inch (0.318 cm) thick stainless steel filter material stock. A five-ton hand punch (or equivalent equipped with a $^{15}/_{64}$ inch punch filed flat can be used to cut the filters from the flat filter material stock. Nominal particle retention sizes are 3 to 10 micron for the exit and 65 micron for all other ports. Ready-made filters are available and can be purchased from numerous commercial sources.

2. **Hydraulic Load Frame.** The hydraulic load frame should have sufficient capacity to develop 150,000 pounds force (667,200 N). To ensure uniform stress distribution, the platens must be parallel to each other. The hydraulic pressurizing source must be capable of holding any desired closure stress [± 0.5% or 20 psi (140 kPa), whichever is greater] over extended time periods. The hydraulic load frame should be capable of loading rate changes of 5,000 lb_f/minute (2200 N/minute), that is 500 psi/minute (3500 kPa/minute) on a 10 square inch (64.5 cm^2) cell. A properly sized press equipped with a pressure compensated hydraulic pump is suitable for this testing. In lieu of a pressure compensated hydraulic pump, the press can be equipped with a conventional hydraulic pump in combination with an air/oil booster system capable of the aforelisted stress level and force control. Appendix A provides information and a listing of necessary items for fabrication and modification of an existing load frame to incorporate air/oil booster pump equipment.

3. **Pack Width Measurement Device(s).** Pack width measurements should be made at each end of the test unit. Dial indicators, micrometers, linear variable differential transformers (LVDT), or linear potentiometers capable of measuring to 0.001 inch (0.0025 cm) accuracy or better can be used.

4. **Test Fluid Drive System.** The test fluid (deionized or distilled water) should be driven at a constant flow rate ranging from 1 to 10 mL/minute (± 1.0%). Constant flow rate pumps (e.g., chromatographic pumps) have been found satisfactory for this application. An alternate system suitable for this application includes a controlled gas pressure source (e.g., a nitrogen tank and pressure regulator) driving test fluid at a constant pressure from a piston or bladder accumulator. Pressure pulsation dampening may be necessary on some chromatographic pumps and can be accomplished by use of a piston, bladder accumulator, or other effective means. Pressure fluctuations during differential pressure and flow rate measurements (for conductivity calculations) should be maintained less than 2.0%. Each laboratory must determine the best technique and charging pressure (if a bladder accumulator or similar device is used) for pulsation dampening. Large pressure spikes may be indicative of pump problems or trapped gas in the flow system and should be corrected before proceeding.

5. **Pressure Indicators.** Measurement of the differential pressure within the test cell requires use of very sensitive devices. Differential pressure transducers with a range of 0-1.0 psi (0-7kPa) are usually satisfactory. The transducer should be capable of measuring the differential pressure to ± 5% of any data point. If low differential pressures [i.e., below 0.1 psi (0.7 kPa)] are encountered, more sensitive transducers may be required. (Refer to *CAUTION* statement below.)

   Figure 2.4 illustrates a plumbing schematic which will allow calibration during a test using a column of water. Calibration must be accomplished with the actual test fluid in contact with both sides of the differential pressure measuring diaphragm.

   CAUTION: *Most manufacturers of differential pressure transducers classify the accuracy as a percent of full scale. A 0-1.0 psi (0-7 kPa) transducer with ±0.5% precision normally implies ± .005 psi (± 0.035 kPa) accuracy of any reading. Since a reading of 0.01 psi (0.07 kPa) may be common, this is only ± 50% precision. However, laboratory experience has shown that, with proper calibration, many of these types of transducers are capable of ± 5% [or ± .0005 psi (± 0.0035 kPa)] accuracy at a reading of 0.01 psi (0.07 kPa). Thus, it is important to calibrate transducers for operating at 10% of full scale or less.*

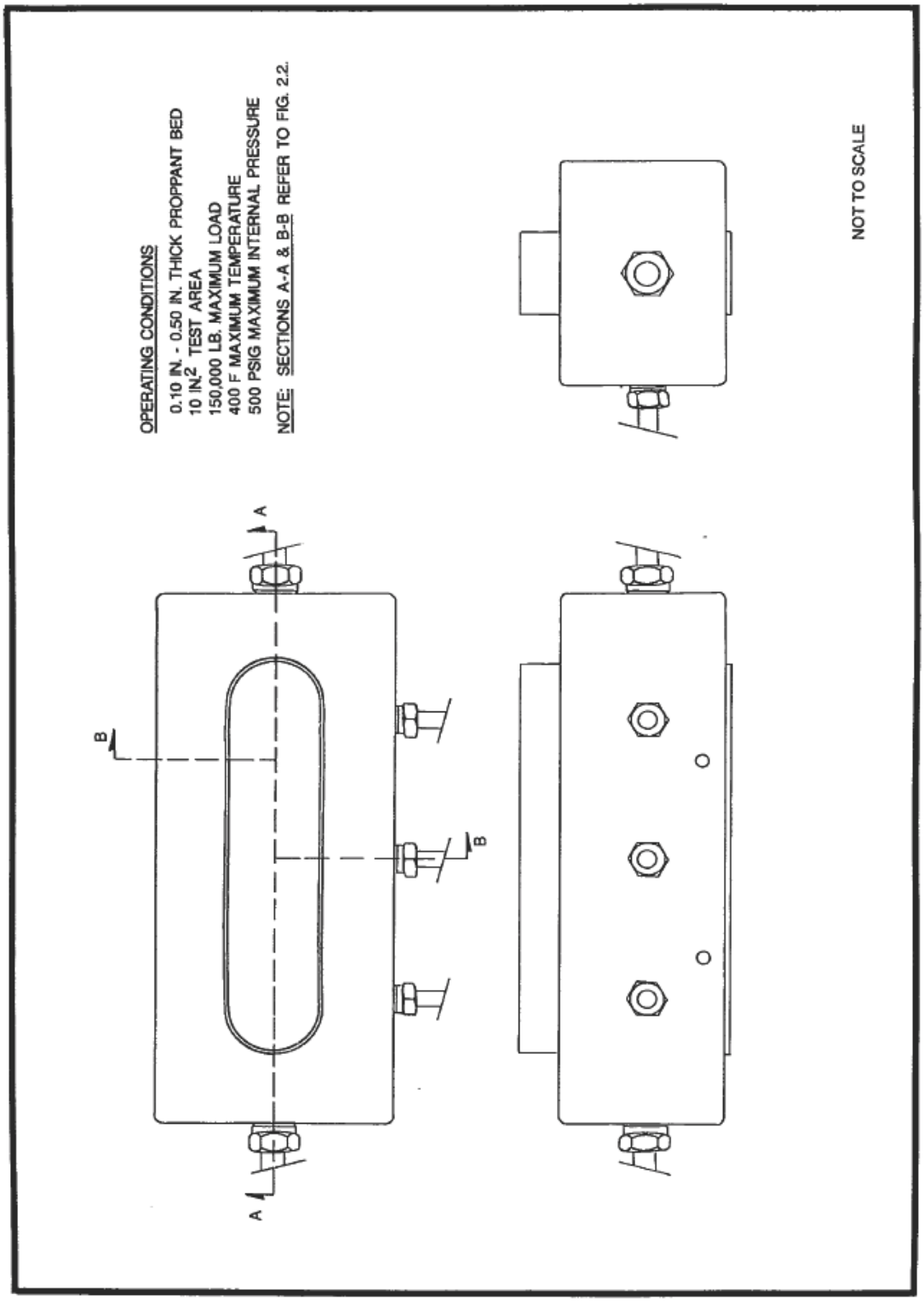

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FIG. 2.1
API PROPPANT PACK CONDUCTIVITY TEST UNIT
(Sheet 1 of 3, Refer to Figs. 2.2 and 2.3)

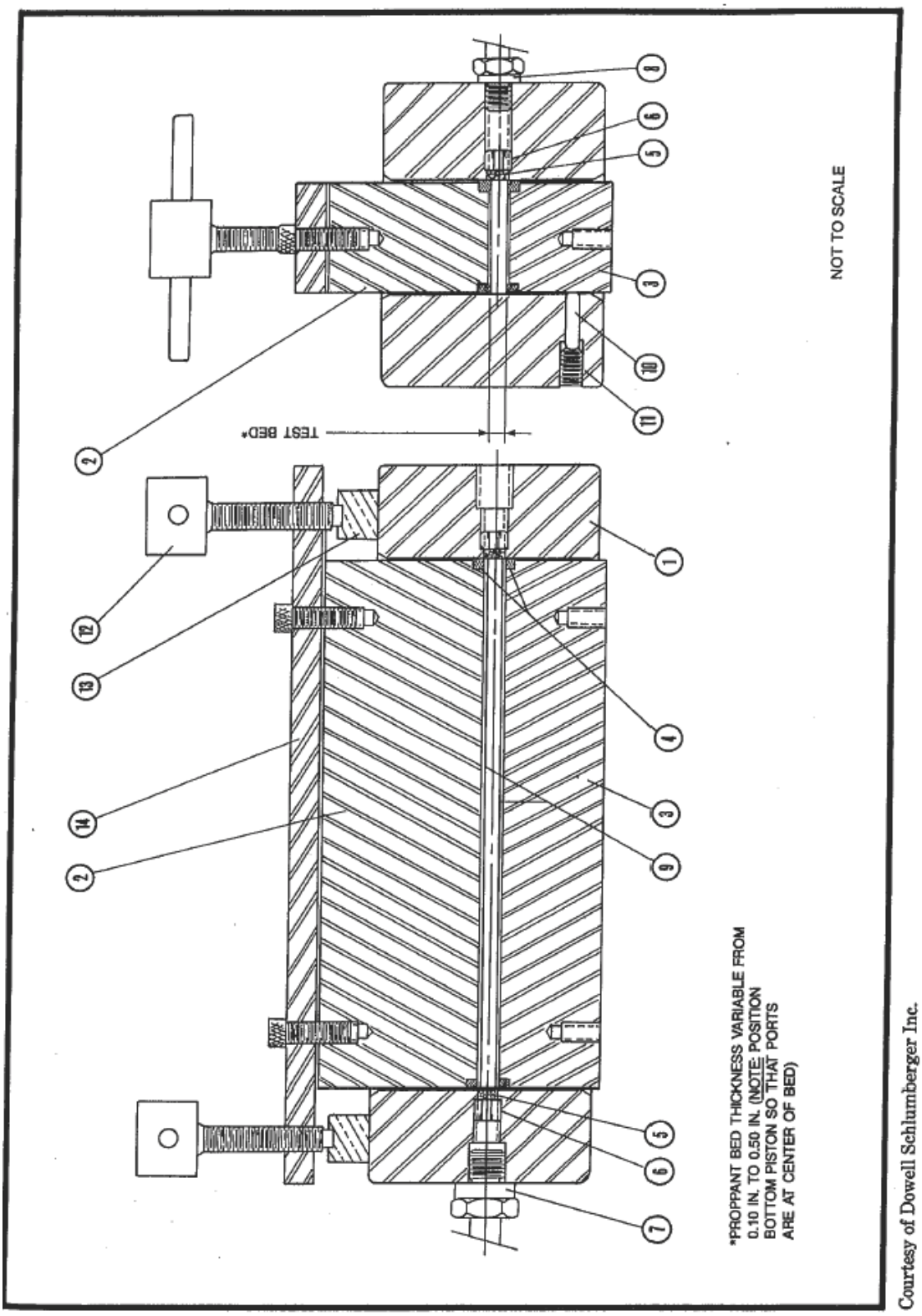

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FIG. 2.2
API PROPPANT PACK CONDUCTIVITY TEST UNIT WITH PISTON PULLER, SECTIONED ASSEMBLY
(Sheet 2 of 3, Refer to Figs 2.1 and 2.3)

6. **Back Pressure Regulators.** The back pressure regulator should be capable of maintaining a downstream pressure 50 psi (350 kPa) ± 5% greater than the vapor pressure of the test fluid (deionized water).

7. **Balance.** The balance used should be capable of accommodating a minimum capacity of 100 g with a precision of 0.1 g or better.

8. **Test Fluid.** The test fluid should be freshly degassed [i.e., 1 hour at 25 mm mercury absolute pressure (3.3 kPa) at 75 F (24 C)] deionized or distilled water. Viscosity and density of any test fluid must be known or measured at test temperature. Table 2.3 should be used to obtain values for viscosity and density of water.

9. **Proppant.** Conductivity may be tested on a volume equivalent to 0.25 inch (0.64 cm) pack width (unstressed) or on a mass per unit cell surface area, such as 2.0 lb_m/ft^2 (9.76 kg/m^2). However, comparison testing should be conducted on an equivalent volume of proppant material rather than an equivalent mass. The bulk density of proppant material to be tested will determine the amount required for comparison testing. Proppant bulk density should be determined using procedures shown in Appendix B, "Measurement of Bulk Density of Proppants."

10. **Temperature Control.** The test cell and proppant pack should be maintained at an ambient temperature of 75 ± 5 F (24 ± 3 C). The temperature of the test fluid at the inlet and outlet ports (refer to Figure 2.4) should be measured and the average of these measurements reported as the test temperature in Figures 2.6 and 2.7. This temperature will be used to determine the fluid viscosity from Table 2.3.

11. **Load Measuring Device.** A temperature compensated electronic load cell should be incorporated in-line between the hydraulic ram and the opposing platen of the load frame. This type of device is preferred over use of hydraulic pressure gauges as a method of determining closure stress applied to the test cell.

    Note: If hydraulic gauges are used as a method of determining closure stress, in conjunction with use of a secondary method of applying closure stress (e.g., air/oil booster), care should be exercised to ensure hydraulic fluid is not trapped on the return side of the ram causing erroneous calculations of closure stress.

2.3 Equipment Calibration.

1. Pressure indicators in the test fluid flow stream should be calibrated initially and rechecked at each test as described in Par. 2.2.5. Other components of the test apparatus must be calibrated initially and at least once per year thereafter. The hydraulic load measuring device should be calibrated using gauge rings. Dial indicators, micrometers, LVDT, or linear potentiometers should be calibrated using gauge blocks. Constant flow rate pumps should be tested at several flow rates with suitable accurate balance, containers, and timing device (stop watch). High range pressure gauges and transducers should be dead weight tested. Low range pressure transducers should be calibrated with an inclined manometer or column of water. Use only that portion of the transducer range which is repeatable and linear. The use of backup mechanical calibration or measuring devices is recommended for each component in the system.

2. Prior to testing the proppant sample, measure the vertical dimension of the complete test unit [± 0.001 inch (± 0.0025 cm)] equipped with platens, but without proppant, at each test closure stress level. These values will serve as the base line when calculating proppant pack widths.

2.4 Leak Tests.

1. **Hydraulic Load Frame.** The hydraulic system, i.e., lines, fittings, and pumps, should be tested initially and periodically at regular intervals thereafter to make sure there are no leaks. This may be done by placing an appropriate block of high strength material [having at least 10 square inches (64.5 cm^2) surface area] between the platens of the press at maximum load; shut in and observe to see if the pressure or load change is greater than ± 2% of maximum reading during a 30 minute period. If the pressure or load varies significantly, inspect all lines and fittings to find the leak. If no line leaks are evident, there may be an internal leak in the control valve or the hydraulic ram.

   Note: Slight changes in pressure or load can be caused by temperature changes of a few degrees. Air in the hydraulic lines can appear to be a leak and should be removed prior to initiation of the test.

2. **Test Fluid System.** The complete test fluid system consisting of pump, lines, fittings, and conductivity test unit should be checked for leaks initially and at the start of each conductivity test. To conduct a leak test, the conductivity test unit should contain at least a monolayer of proppant material.

   Note: With no proppant between the platens, neither the square seal rings nor the downstream equipment can be tested.

   Apply at least 500 psi (3500 kPa) closure stress to the conductivity unit and evacuate the entire system, i.e., between the pump discharge and the back pressure regulator, to an absolute pressure of 25 mm mercury (3.3 kPa) at 75 F (24 C). Turn

off the vacuum pump and determine if the system holds pressure. The pressure in the system should not change more than 1 mm mercury (0.13 kPa) in 5 minutes.

Note: If an absolute pressure below 23 mm mercury (3.1 kPa) is attempted and water is present in the evacuated system, an increase in pressure will occur once the vacuum pump is turned off until the vapor pressure of water [22.2 mm mercury (2.96 kPa) at 75 F (24 C)] is attained.

2.5 Preparation of the Test Unit. The following detailed procedure should be used to assemble and prepare the test unit for proppant pack conductivity testing.

1. Place a stainless steel filter (item 5, Fig. 2.3) in the fluid entry (65 micron) and exit (3-10 micron) ports (ports 1 and 5, Fig. 2.4) and in each of the differential pressure ports (65 micron in ports 2, 3, and 4, Fig. 2.4) from the inside of the conductivity test unit. The set screws (item 6, Fig. 2.3) should be adjusted so that the filter is flush with the inside surface of the test unit. Filters should be replaced periodically as they can become plugged with crushed proppant. Plugged filters are evidenced by an increase in the drive pressure necessary to flow through the test unit and by erratic differential pressure measurements.

   Note: When testing proppants at stress levels where significant crushing occurs, the downstream filter (port 5) will often become completely plugged with fine crushed material, thereby preventing flow through the test unit. If this occurs or is anticipated, the downstream filter should be removed (and not replaced) and the filter port packed with proppant material. This can be accomplished by turning the test cell on end with discharge port down, pouring a small amount of proppant into the filter port, and tamping lightly with a punch or blunt instrument.

   Continue filling and tamping until the port is full and packed. If this method is used, fine crushed material will be transported out of the test unit along with the test fluid. Back pressure regulators or other instrumentation which might be damaged by the fine crushed material should not be located directly on the discharge line. Back pressure may be safely supplied by using a pressurized accumulator on the discharge line.

2. Place the bottom piston equipped with square ring in the test chamber.

   Note: A very small amount of grease applied to the beveled edge at the bottom of the chamber greatly aids efforts to insert the piston and square ring into the chamber.

3. Position a proppant platen on top of the bottom piston. The platen must be flat in order for the proppant bed to have a uniform cross-sectional area. In order to assure uniformity, the depth from the platen to the top of the test chamber should be measured with a depth gauge in a minimum of three places along the length of the platen. There should be no more than 0.01 inch (0.025 cm) difference in these measurements between the platen surface and the top of the test chamber.

4. Load the test cell with the desired amount of proppant material using one of the procedures described below (note that procedure b should be used for comparison of propping agents):

   a. Mass per unit area (lb_m/ft^2 or kg/m^2). Load the desired amount of proppant which can be calculated as follows:

   $$W_p = 31.50\,C \quad \text{..... (2.1)}$$

   where:

   W_p = Proppant weight, g

   C = Proppant loading, lb_m/ft^2

   $31.50 = 10.0 \times 453.6/12^2$

   If C is in kg/m^2 and W_p is in grams, the conversion factor is 6.452.

   ***CAUTION:** For best reproducibility, the recommended minimum and maximum unstressed proppant pack widths are:*

   Minimum pack width = 0.10 inch (0.25 cm).
   Maximum pack width = 0.50 inch (1.3 cm).

   Operation of the test cell outside these recommended limits may cause damage to the seals and test cell. ***CAUTION:** The test cell must be operated outside the recommended unstressed proppant pack widths in order to determine the base line measurements for fracture width (refer to Par. 2.3.2).* The unstressed proppant pack width can be approximated as follows:

   $$W_f = 0.1922\,\frac{C}{\rho} \quad \text{..... (2.2)}$$

   where:

   W_f = Proppant pack width, inch

   C = Proppant loading, lb_m/ft^2

   ρ = Proppant bulk density (refer to Appendix B), g/cm^3

   If C is in kg/m^2, ρ is in g/cm^3, and W_f is in cm, the conversion factor is 0.100.

   b. Unstressed proppant pack width equal to 0.25 inch (0.64 cm). There are two methods for obtaining conductivity data at an initial, unstressed pack width of 0.25 inch (0.64 cm).

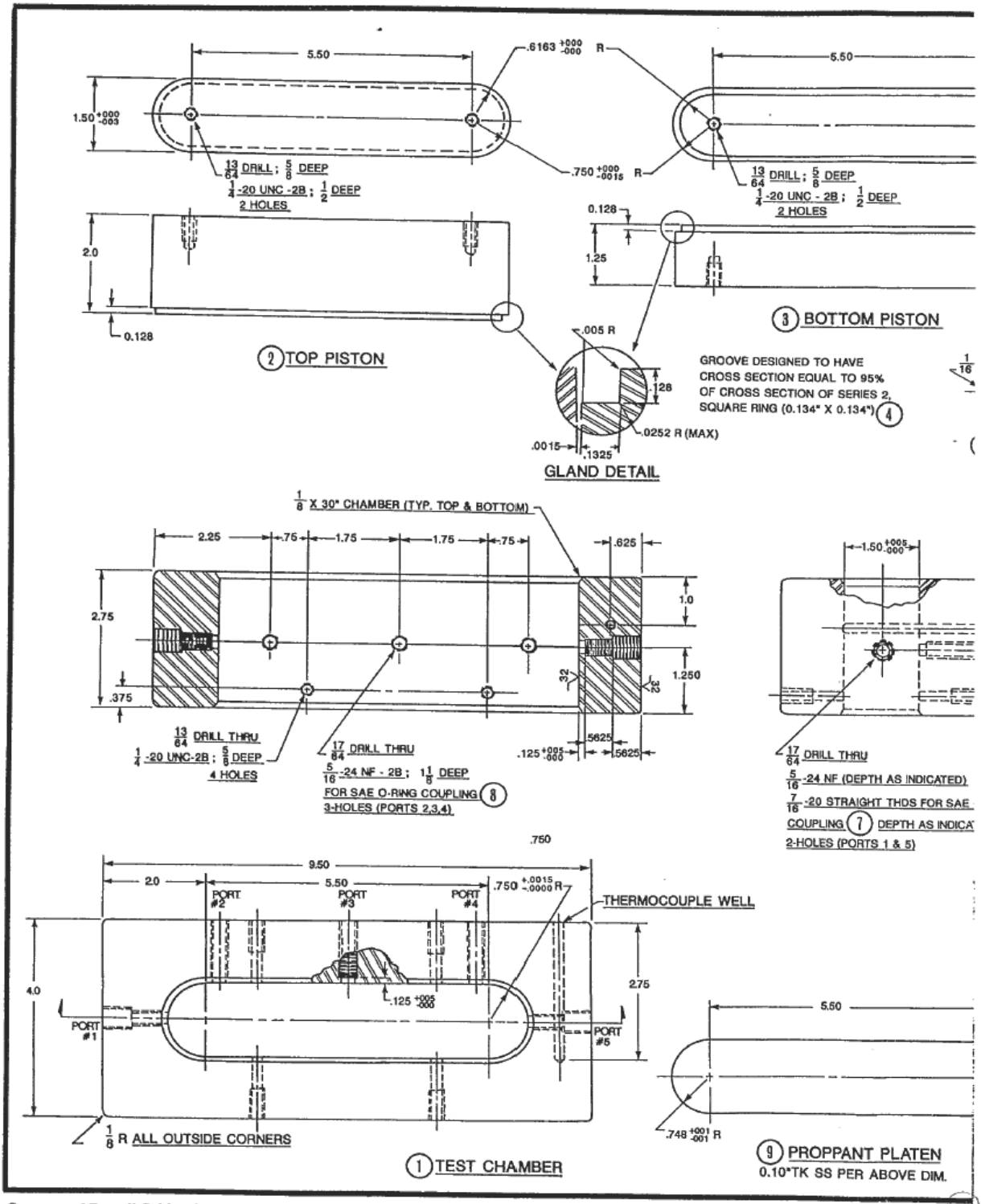

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FIG.
API PROPPANT PACK CONDU
(Sheet 3 of 3, Refer t

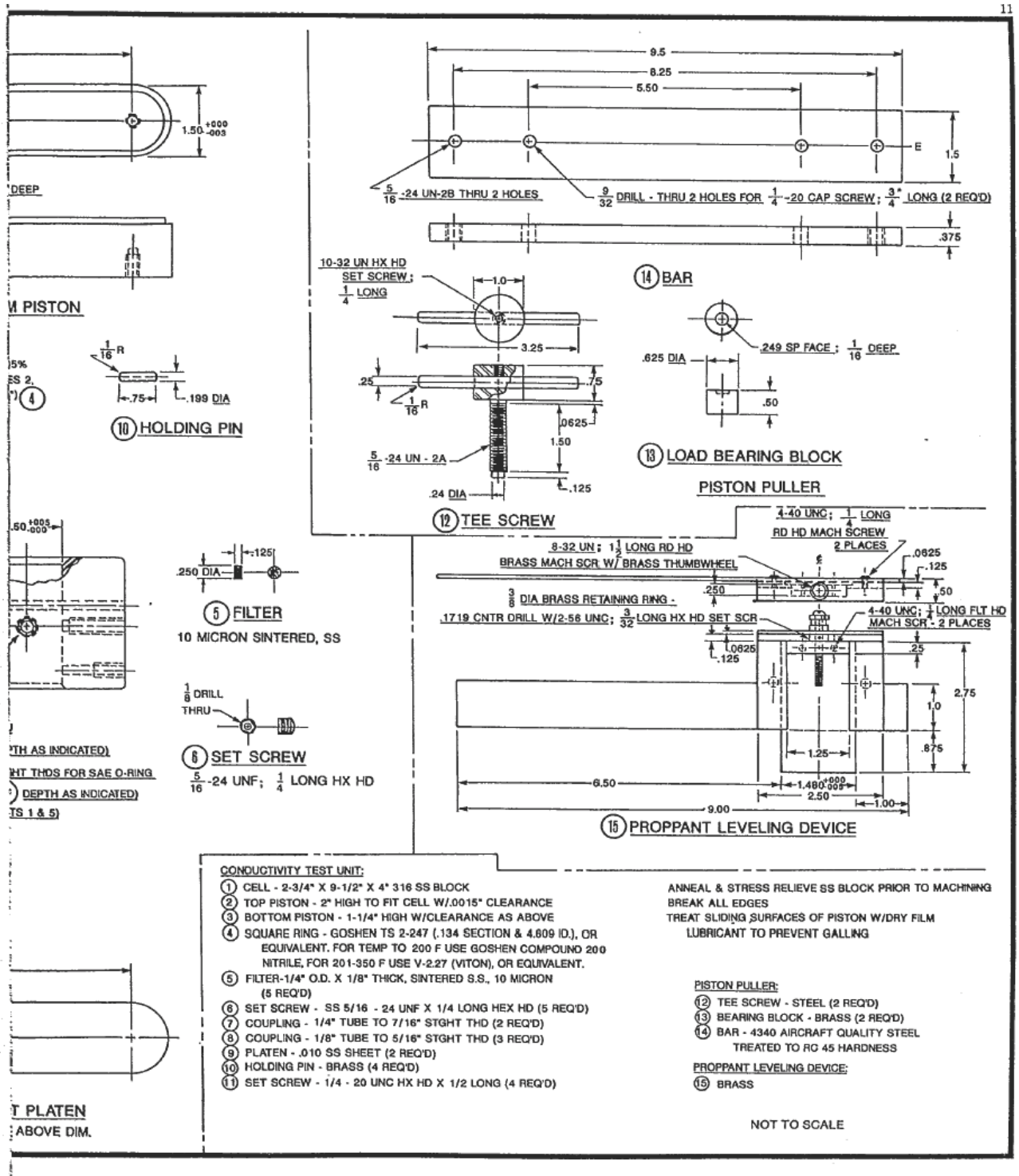


FIG. 2.3
PACK CONDUCTIVITY TEST EQUIPMENT
3 of 3, Refer to Figs. 2.1 and 2.2)

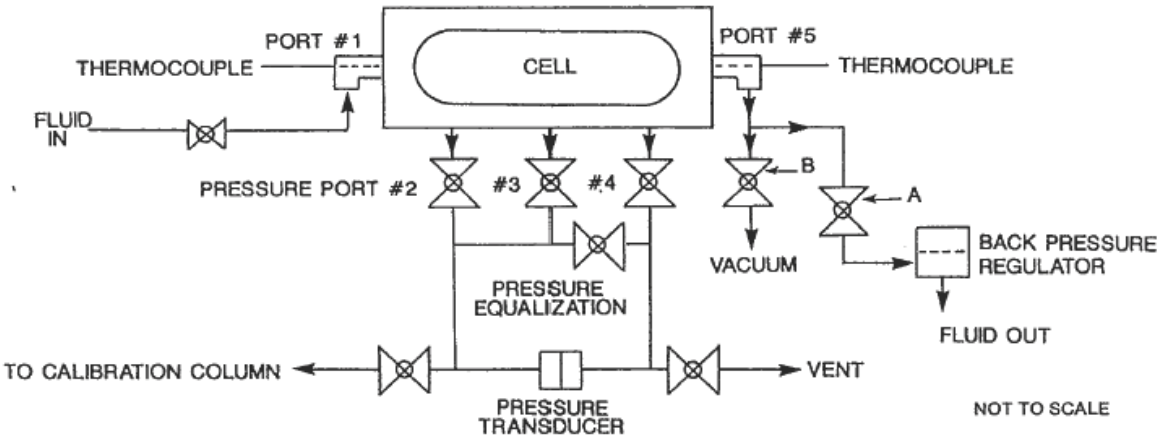
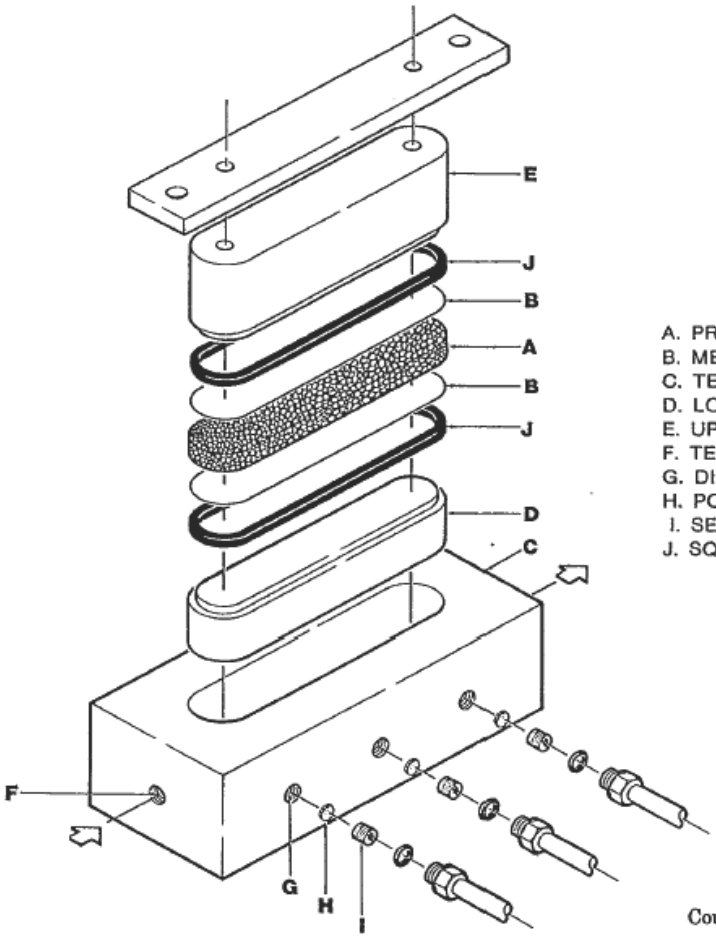


FIG. 2.4
SCHEMATIC OF FLOW PATHS THROUGH
THE API PROPPANT PACK CONDUCTIVITY TEST UNIT

Courtesy of The Western Company of North America.



A. PROPPANT PACK 7 INCH × 1.5 INCH × W_f
B. METAL PLATEN
C. TEST UNIT BODY
D. LOWER PISTON
E. UPPER PISTON
F. TEST FLUID ENTRY/EXIT PORT
G. DIFFERENTIAL PRESSURE SENSING PORT
H. POROUS METAL FILTER
I. SET SCREW
J. SQUARE RING SEAL

NOT TO SCALE

Courtesy of The Western Company of North A:

FIG. 2.5
EXPLODED SCHEMATIC DIAGRAM OF
API PROPPANT PACK CONDUCTIVITY TEST UNIT

1) **Method I.** Load the test cell with 41.0 ± 0.1 cm^3 of proppant material. The approximate weight of the required proppant material can be calculated as follows:

$$W_p = 41.0\rho \quad \text{..... (2.3)}$$

where:

W_p = Proppant weight, g

ρ = Proppant bulk density (refer to Appendix B), g/cm^3

2) **Method II.** Conductivity data at an initial unstressed proppant pack width of 0.25 inch (0.64 cm) can be calculated by interpolating between two sets of conductivity data generated by using the procedure described in Par. 2.5.4.a. The proppant loading can be calculated for an unstressed proppant pack width of 0.25 inch (0.64 cm) by rearranging Equation (2.2) (Par. 2.5.4.a) and solving for C, while letting W_f = 0.25 inch (0.64 cm).

$$C = 1.301\rho \quad \text{..... (2.4)}$$

The interpolated conductivity at the desired stress is calculated as follows:

$$kW_f = kW_{f1} + \left(kW_{f2} - kW_{f1}\right)\left(\frac{C - C_1}{C_2 - C_1}\right) \quad \text{.. (2.5)}$$

where:

kW_f = Conductivity of proppant pack having an initial unstressed pack width of 0.25 inch (0.64 cm), md·ft

kW_{f1} = Conductivity of proppant pack loaded with C_1 lb_m/ft^2 of proppant, md·ft

kW_{f2} = Conductivity of proppant pack loaded with C_2 lb_m/ft^2 of proppant, md·ft

C_1 = Proppant loading used in test 1, lb_m/ft^2

C_2 = Proppant loading used in test 2, lb_m/ft^2

C, C_1, and C_2 can also be expressed in units of kg/m^2. If kW_{f1} and kW_{f2} are expressed in μm^2, then kW_f will be expressed in μm^2.

Note: To minimize interpolation errors, the proppant loading used in tests 1 and 2 should differ by no more than 0.5 lb_m/ft^2 (2.44 kg/m^2) and C should fall between C_1 and C_2.

The interpolated proppant pack width at the desired stress can be calculated by the same procedure used to calculate the interpolated conductivity, kW_f, as follows:

$$W_f = W_{f1} + \left(W_{f2} - W_{f1}\right)\left(\frac{C - C_1}{C_2 - C_1}\right) \quad \text{... (2.6)}$$

where:

W_f = Width of proppant pack having an initial unstressed pack width of 0.25 inch (0.64 cm), inch

W_{f1} = Width of proppant pack loaded with C_1 lb_m/ft^2 of proppant material, inch

W_{f2} = Width of proppant pack loaded with C_2 lb_m/ft^2 of proppant material, inch

Note: Refer to Appendix C for conversion factors.

5. Level the proppant material layer in the test unit with a blade type device (refer to Fig. 2.3). The proppant material should not be leveled by vibration or tamping as this will concentrate fines toward the bottom of the test unit.

6. Place another proppant platen on top of the leveled proppant pack. This must be done very carefully without disturbing the proppant material. A tab made of masking tape (or equivalent) and placed in the center of the platen can be used as a "handle" for positioning the platen on the proppant material layer. Once the platen is in place, carefully remove the tape "handle," again being certain to prevent disturbing the proppant material.

7. Place the top piston equipped with the square ring in the test chamber. Lubricate the ring lightly with grease and push it down by hand until it contacts the upper proppant platen.

8. Place the test unit between the platens of the load frame. Apply hydraulic pressure and raise the lower platen until 1,000 psi (6900 kPa) closure stress is applied to the test unit, using a maximum loading rate of 500 psi/minute (3500 kPa/minute).

9. Remove residual gas by closing the inlet valve and exit valve A and pulling a vacuum [25 mm mercury (3.3 kPa) at 75 F (24 C)] on the outlet end of the test unit until all gas has been removed from the unit and transducer lines. Slowly open the inlet valve and flow degassed test fluid into the unit until the cell, flow lines, and pressure transducer lines are full. Close vacuum valve B and *slowly* bring the test fluid pressure to operating level or a minimum of 10 psig (69 kPa). Using the bleed ports on the transducer, ascertain that no gas is trapped in the transducer or its associated plumbing.

10. Check for leaks at the test unit fittings or associated plumbing. Repair any leaks and do not disturb the proppant pack.

11. Check for leaks around the pistons. If leakage is observed, the test should be terminated and the test unit repacked with new material. Leakage problems should be corrected at this time (refer to Par. 2.5.2).

12. Slowly open exit valve A and adjust the back pressure, if used.

13. Check uniformity of the proppant pack using one of the following procedures:

a. Measure pack bed width at each end of the test unit. If there is a difference of 5% or more between these width measurements, non-uniform leveling is indicated and the test should be terminated and the unit repacked with new material and leveled (refer to Par. 2.5.2).

b. Flow test fluid through the test unit at a constant rate and compare the pressure drop between ports 2 and 3 to the pressure drop between ports 3 and 4 (refer to Fig. 2.4). Differential pressures which vary by 5% or more indicate non-uniform leveling. If this occurs, the test should be terminated and the test unit repacked with new material and leveled (refer to Par. 2.5.2). Note that the differential pressure between ports 2 and 3 can be calculated by subtracting the differential pressure between ports 3 and 4 from the differential pressure between ports 2 and 4 (refer to Fig. 2.4).

2.6 Test Parameters. Tables 2.1 and 2.2 show the recommended test closure stresses, flow rates, and time at stress to reach a semi-steady state condition for sand and high strength proppant materials, respectively. A maximum loading rate of 500 psi/minute (3500 kPa/minute) should be used to increase closure stresses in these tests.

2.7 Calculations.

1. Equation (34) from *API RP 27: Recommended Practice for Determining Permeability of Porous Media*, Third Edition, September 1952 (Reissued August 1956)* (check latest edition), can be used to calculate the permeability of proppant packs to liquid under laminar (darcy) flow conditions.

$$k = \frac{\mu QL}{A(\Delta P)} \qquad \text{..... (2.7)}$$

*Available from American Petroleum Institute, Publications and Distribution, 1220 L St., N. W., Washington, D.C. 20005.

TABLE 2.1
RECOMMENDED TEST PARAMETERS FOR SAND PROPPANTS

Closure Stress,* psi (kPa)	Flow Rates, cm³/minute	Time at Stress for Various Sand Sizes, hr				
		12/20	20/40	30/50	40/70	70/140
1,000 (6,900)	2.5, 5.0, 10.0	1.0	0.25	0.25	0.25	0.25
2,000 (13,800)	2.5, 5.0, 10.0	1.5	0.25	0.25	0.25	0.25
4,000 (27,600)	2.5, 5.0, 10.0	1.5	1.00	0.25	0.25	0.25
6,000 (41,400)	1.25, 2.5, 5.0	1.5	1.00	0.25	0.25	0.25
8,000 (55,200)	1.0, 2.0, 4.0	1.5	1.00	0.75	0.75	0.75
10,000 (69,000)	1.0, 2.0, 4.0	1.5	1.00	1.00	1.00	1.00

**Note:* Use a maximum loading rate of 500 psi/minute (3500 kPa/minute) to achieve closure stress level. The closure stress is equal to the stress applied to the conductivity test unit minus the pore pressure of the test fluid pressure.

TABLE 2.2
RECOMMENDED TEST PARAMETERS FOR HIGH STRENGTH PROPPANTS (ALL SIZES)

Closure Stress,* psi (kPa)	Flow Rates, cm³/minute	Time at Stress, hr
1,000 (6,900)	2.5, 5.0, 10.0	0.25
2,000 (13,800)	2.5, 5.0, 10.0	0.25
4,000 (27,600)	2.5, 5.0, 10.0	0.25
6,000 (41,400)	2.5, 5.0, 10.0	0.25
8,000 (55,200)	2.5, 5.0, 10.0	0.25
10,000 (69,000)	2.5, 5.0, 10.0	0.25
12,000 (82,700)	2.5, 5.0, 10.0	0.25
14,000 (96,500)	2.5, 5.0, 10.0	0.25

**Note:* Use a maximum loading rate of 500 psi/minute (3500 kPa/minute) to achieve closure stress level. The closure stress is equal to the stress applied to the conductivity test unit minus the pore pressure of the test fluid pressure.

where:

k = Proppant pack permeability, darcy

μ = Viscosity of test liquid at test temperature, cP

Q = Flow rate, cm^3/s

L = Length between pressure ports, cm

A = Cross-sectional area of test unit perpendicular to flow, cm^2

ΔP = Pressure drop (pressure upstream minus pressure downstream), atm

If ΔP is in kPa and k is in μm^2, the conversion factor is 9.74×10^{-3}.

When the cross-sectional shape of the proppant bed is rectangular as it is in a fracture, then:

$$A = w\ W_f \quad \text{..... (2.8)}$$

where:

A = Cross-sectional area perpendicular to flow, cm^2

w = Test unit width, cm

W_f = Pack width, cm

Equation (2.7) can be rewritten so that either proppant pack permeability or conductivity can be calculated.

To calculate proppant pack permeability use:

$$k = \frac{\mu Q L}{w(\Delta P)\dot{W}_f} \quad \text{..... (2.9)}$$

To calculate proppant pack conductivity use:

$$kW_f = \frac{\mu Q L}{w(\Delta P)} \quad \text{..... (2.10)}$$

To convert kW_f from darcy × centimeters (d·cm) to millidarcy × feet (md·ft), multiply by the constant, 32.8.

2. The following information and simplified equations can be used when making determinations using the recommended API test unit and procedures.

a. Proppant Pack Permeability:

$$k = \frac{321.4\ \mu Q}{\Delta P W_f} \quad \text{..... (2.11)}$$

where:

k = Proppant pack permeability, md

μ = Viscosity of test liquid at test temperature, cP (refer to Table 2.3)

Q = Flow rate, cm^3/minute

W_f = Pack width, inch

ΔP = Pressure drop (pressure upstream minus pressure downstream), psi

If W_f is in cm, ΔP is in kPa, and k is in μm^2, the conversion factor is 5.411×10^{-4}.

TABLE 2.3
VISCOSITY AND DENSITY OF WATER AT TEMPERATURE*
(Reference *Handbook of Chemistry and Physics*, 66th Edition, 1985-86, CRC Press Inc., Boca Raton, Fla.)

Temperature, C (F)	Viscosity, cP (mPa·s)**	Density, g/cm^3
20.0 (68)	1.002	0.9982
21.0 (70)	0.978	0.9980
22.0 (72)	0.955	0.9978
23.0 (73)	0.932	0.9975
24.0 (75)	0.911	0.9973
25.0 (77)	0.890	0.9970
26.0 (79)	0.870	0.9968
27.0 (81)	0.851	0.9965
38.0 (100)	0.678	0.9930
49.0 (120)	0.556	0.9885
60.0 (140)	0.466	0.9832
71.0 (160)	0.399	0.9775
82.0 (180)	0.346	0.9705
93.0 (200)	0.304	0.9633
104.0 (220)	0.270	0.9554
116.0 (240)	0.240	0.9464
127.0 (260)	0.217	0.9376
138.0 (280)	0.198	0.9281
149.0 (300)	0.181	0.9182

*These data may be approximated by the following equations:

Density of water, -30 C to 150 C (-22 F to 302 F)

$\rho = (0.99983952 \times 0.016945176\ T - 7.9870401\ E\text{-}6T^2 - 4.6170461\ E\text{-}8T^3 + 0.10556302\ E\text{-}9T^4 - 0.28054253\ E\text{-}12T^5)/(1 + 0.01687985\ T)$

where:

ρ = Density of water, g/cm^3

T = Average fluid temperature, C

Viscosity of water, 20 C to 150 C (68 F to 302 F)

$\mu = e^x$

where:

μ = Viscosity of water, cP

e = Base of natural logarithm = 2.7182818.

$x = (60.359768 - 2.9570089\ T - 0.0024246\ T^2)/(105 + T)$

**1 cP = 1mPa·s

b. Proppant Pack Conductivity:

$$kW_f = \frac{26.78\,\mu Q}{\Delta P} \quad \text{..... (2.12)}$$

where:

kW_f = Proppant pack conductivity, md.ft

μ = Test liquid viscosity at test temperature, cP (refer to Table 2.3)

Q = Flow rate, cm^3/minute

ΔP = Pressure drop (pressure upstream minus pressure downstream), psi

If ΔP is in kPa, and kW_f is in $\mu m^2 \cdot cm$, the conversion factor is 5.411×10^{-4}.

Note: The following dimensions were used in calculating the constants shown in equations (2.11) and (2.12):

Test unit width, w = 1.5 inch (3.81 cm).

Length between pressure ports 2 and 4, L = 5.0 inch (12.70 cm).

Note: Refer to Appendix C for conversion factors.

2.8 Data Reporting. Data sheets shown in Figures 2.6 and 2.7 should be used to record and report test data. If Method II (refer to Par. 2.5.4.b.2) is used to calculate interpolated data for a 0.25 inch (0.64 cm) initial unstressed proppant pack width, a pair of data sheets should be completed for the test data at each proppant loading. The interpolated data for the 0.25 inch (0.64 cm) initial unstressed proppant pack width should be recorded on a "Short Term Proppant Pack Conductivity Data Reduction Sheet" (refer to Fig. 2.7).

FIGURE 2.6
SHORT TERM PROPPANT PACK CONDUCTIVITY TEST DATA

Proppant Type: _____ **Size:** _____

TEST CONDITIONS:

_____ g/cm^3 **Bulk Density**

_____ $inch^2(cm^2)$† **Cell Test Area**

_____ **Test Fluid**

_____ $lb_m/ft^2(kg/m^2)$† **Proppant Loading**

_____ g **Proppant Weight**

Pretest Proppant Sieve Analysis

Sieve No.	Weight on Sieve, g	Percent of Total
PAN		
TOTAL		

DIFFERENTIAL PRESSURE AND FLOW RATE

Stress, psi	Stress, (kPa)	Pack Width, in. (cm)*† W_{fa}	W_{fb}	Avg. W_f	ΔP_1, psi (kPa)†	Q_1, cm^3/minute	ΔP_2, psi (kPa)†	Q_2, cm^3/minute	ΔP_3, psi (kPa)†	Q_3, cm^3/minute	Fluid Temp., F (C)†	Fluid Viscosity, cP (mPa·s)†
1,000	(6,900)											
2,000	(13,800)											
4,000	(27,600)											
6,000	(41,400)											
8,000	(55,200)											
10,000	(69,000)											
12,000	(82,700)											
14,000	(96,500)											

*W_{fa} and W_{fb} are the measured proppant pack widths taken at each end of the test cell.

†Where dual units are given, indicate unit used.

FIGURE 2.7
SHORT TERM PROPPANT PACK CONDUCTIVITY DATA REDUCTION

Proppant Type: _____ **Size:** _____

Proppant Amount: _____ lb_m/ft^2 (kg/m^2)† _____ ft^3/ft^2(m^3/m^2)†

Bulk Density: _____ g/cm^3, _____ lb_m/ft^3 (kg/m^3)†

Average Temperature: _____ F (C)† **Fluid Type:** _____

Initial Pack Width:* _____ inch (cm)†

Stress, psi (kPa)	Avg. W_f, inch (cm)†	Avg. Conductivity, kW_f, md·ft (μm^2 · cm)†	Permeability, k**, md (μm^2)†	Fluid Viscosity, cP (mPa ·s)†
1,000 (6,900)				
2,000 (13,800)				
4,000 (27,600)				
6,000 (41,400)				
8,000 (55,200)				
10,000 (69,000)				
12,000 (82,700)				
14,000 (96,500)				

*If these data are to be interpolated from other test data to an initial (no load) pack width of 0.25 inch (0.64 cm), write "interpolated" beside the initial pack width of 0.25 inch (0.64 cm). The initial pack width, W_{fo}, is calculated as follows:

W_{fo} (inch) = (12 inches/ft) × Proppant Loading (lb_m/ft^2)/Bulk Density (lb_m/ft^3), or

W_{fo} (cm) = 0.100 × Proppant Loading (kg/m^2)/Bulk Density (g/cm^3).

*The permeability is calculated from the average conductivity and average width as follows:

k (md) = 12 (inches/ft) × kW_f (md · ft)/W_f (inch), or

k (μm^2) = kW_f μm^2·cm/W_f (cm).

†Where dual units are given, indicate the unit used.

APPENDIX A
HYDRAULIC PRESSURE CONTROL

A.1 Components of an air/oil pressure control for regulating the hydraulic press with a maximum working pressure for the hydraulic ram loading system of 6,000 psi (41,000 kPa). If other equipment is to be used in the tests, consult the equipment manufacturer for proper equipment pressure ranges and appropriate plumbing apparatus.

Three basic component systems are part of this control system: (1) air/oil booster pump, (2) air pressure control system, (3) hydraulic pressure control system. Figure A.1 is a schematic drawing of the complete control system. The necessary components are available from many hydraulic control supply organizations.

1. **Air/Oil Booster Pump:**

   The booster pump should have a pressure ratio of 1:60. The booster pump should be equipped with a so-called "low air pressure control kit" so that it will operate efficiently at air pressures of less than 20 psi (138 kPa). This modification effectively allows the entire cycling (start/stop) and pilot valve system to be independent of regulated air pressure to the air drive piston. Refer to Item 7.

2. **Air Pressure Control System:**

   Inlet air shut-off or plug valve. Refer to Item 1.

   Air filter. Refer to Item 2.

   Air regulator, non-adjustable, set at a pressure equal to maximum allowable hydraulic pressure divided by booster pump ratio, i.e., 5300 psi/60 = 88.3 psi (36,544 kPa/60 = 609 kPa). This is a safety device. Refer to Item 3.

   Air regulator, adjustable. Refer to Item 4.

   Air pressure gauge, 0-100 psi or 0-1,000 kPa. Refer to Item 5.

   Speed control valve, to control speed of booster pump. Refer to Item 6.

3. **Hydraulic Pressure Control System Rated at 6,000 psi (41,400 kPa) Maximum Allowable Working Pressure.**

   Pressure gauge or transducer 0-6,000 psi or 0-50,000 kPa. Refer to Item 8.

   Flow check valve. Refer to Item 9.

   Shut-off valve to pressure frame hydraulic ram. Refer to Item 10.

   Pressure relief or pop-off valve set at 5,400 psi (37,233 kPa). This is a safety device. Refer to Item 11.

   Pressure relief, rupture disc, 5,600 psi (38,612 kPa). This is a safety device. Refer to Item 12.

   Shut-off valves to low and high range hydraulic regulators. Refer to Items 13 and 14.

   Adjustable hydraulic pressure regulators or relief valves. Low-range — 75 to 750 psi or 520 to 5,200 kPa; high range — 600 to 6,000 psi or 4,200 to 42,000 kPa. These regulators are used to compensate for the inevitable hydraulic pressure leaks in the pressure frame loading system. Refer to Items 15 and 16.

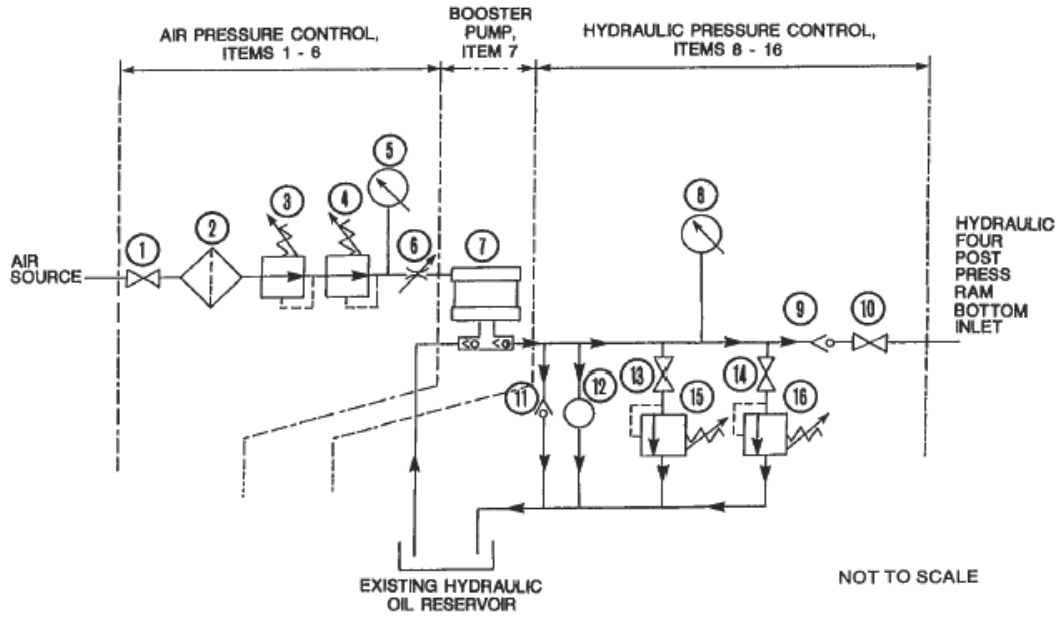


FIGURE A.1*
SCHEMATIC OF HYDRAULIC PRESSURE CONTROL EQUIPMENT

*Courtesy of The Western Company of North America.

APPENDIX B
MEASUREMENT OF BULK DENSITY OF PROPPANTS

B.1 BULK DENSITY

1. **Equipment and Materials.** The following are needed to obtain the bulk density of proppant samples:
   a. Analytical balance, 0.01 g precision.
   b. 100 mL volumetric flask (100 mL = 100 cm^3 at 75 F).
   c. Proppant sample, dry and free-flowing.
   d. Wide mouth funnel, stem to fit inside the volumetric flask.
2. **Procedure.** The following procedure may be used for determining bulk density of proppants:
   a. Weigh the clean, dry 100 mL volumetric flask to 0.01 g precision using the analytical balance.
   b. Place the funnel in the neck of the volumetric flask and fill it with proppant to the 100 mL mark. Do not shake the flask or tamp the proppant. *Note: This is a critical step and must be done the same way by each person measuring bulk density.*
   c. Weigh the volumetric flask containing proppant to 0.01 g precision.
   d. Calculate the proppant bulk density using the following equation:

$$\rho = [W_{f1,p} - W_{f1}]/100 \quad \text{(B.1)}$$

where:

ρ = Proppant bulk density, g/cm^3

$W_{f1,p}$ = Weight of flask and proppant (step c), g

W_{f1} = Weight of flask (step a), g

   e. Report proppant bulk density in g/cm^3 and lb_m/ft^3 (refer to Fig. 2.6). *Note:* $lb_m/ft^3 = g/cm^3 \times 62.4$.

APPENDIX C
CONVERSION FACTORS

1 ft	=	0.3048 m
1 inch	=	2.54 cm
1 darcy	=	1000 md = 0.9869 μm^2
1 lb_m	=	453.6 g
1 lb_f	=	4.448 N
1 psi	=	6.895 kPa
1 atm	=	14.7 psi = 101.3 kPa
1 mm Hg	=	0.1333 kPa (absolute)
1 mL	=	1.000 cm^3
F	=	(1.80 × C) + 32
1 cP	=	1 mPa·s
1 $lb_f \cdot s/ft^2$	=	47.88 Pa·s

APPENDIX D
REFERENCES

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