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

API RECOMMENDED PRACTICE 60
SECOND EDITION, DECEMBER 1995

American Petroleum Institute

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Exploration and Production Department

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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.

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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.

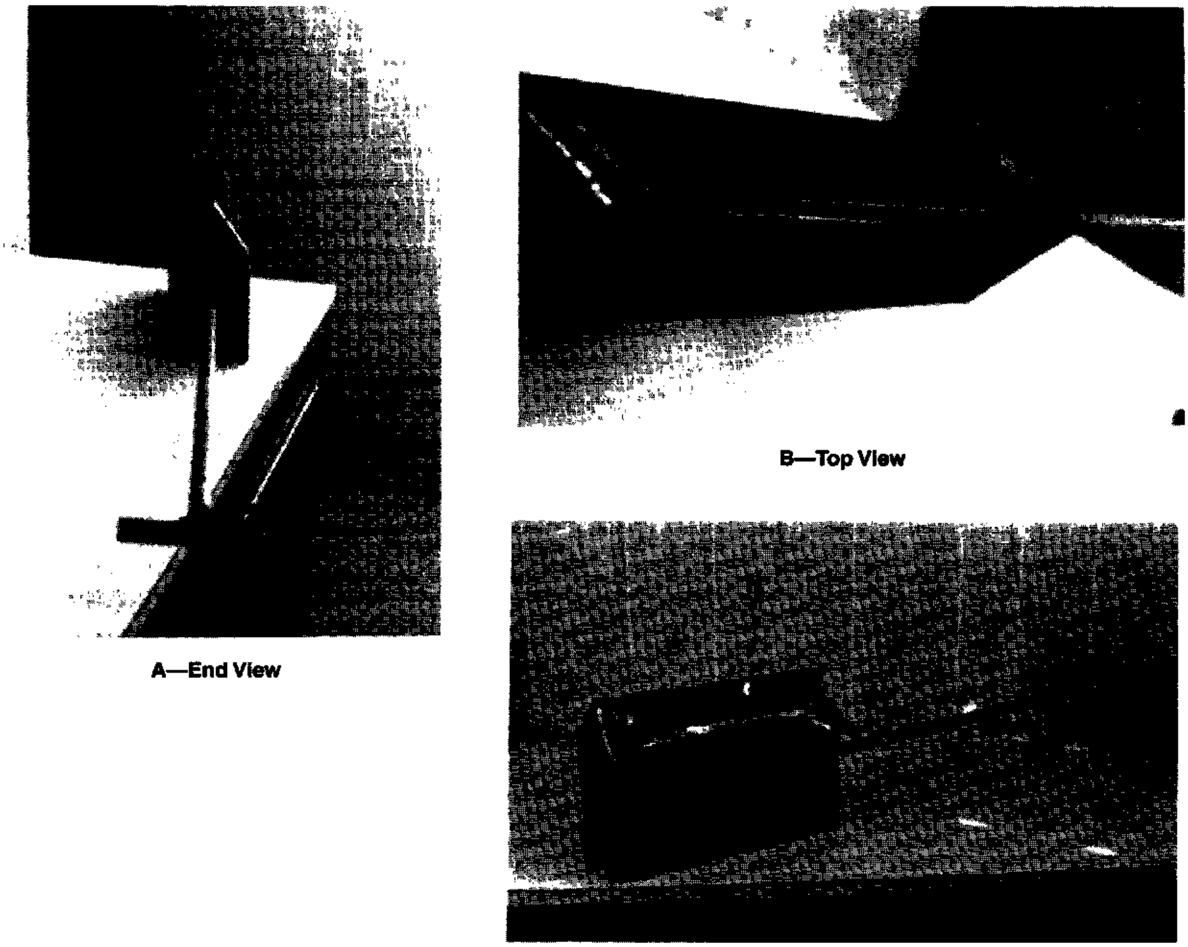
3.2 EQUIPMENT

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.

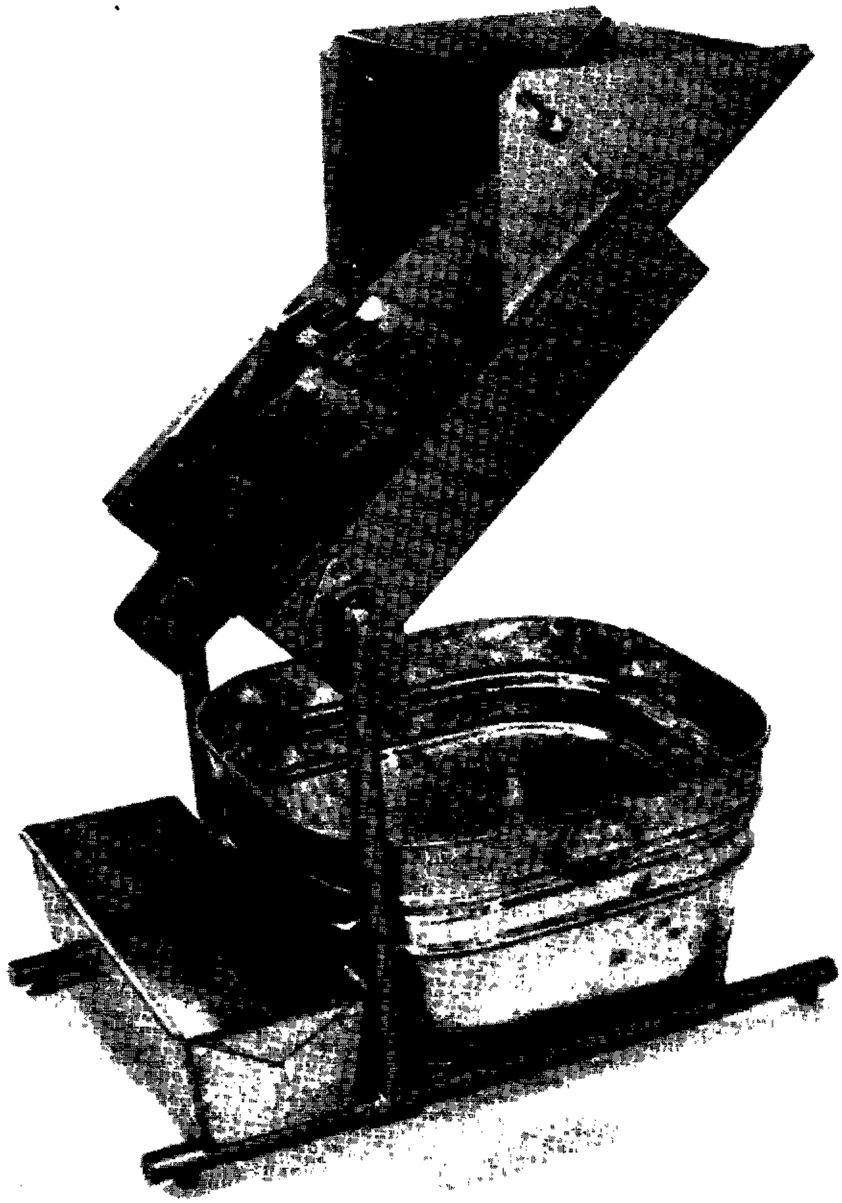
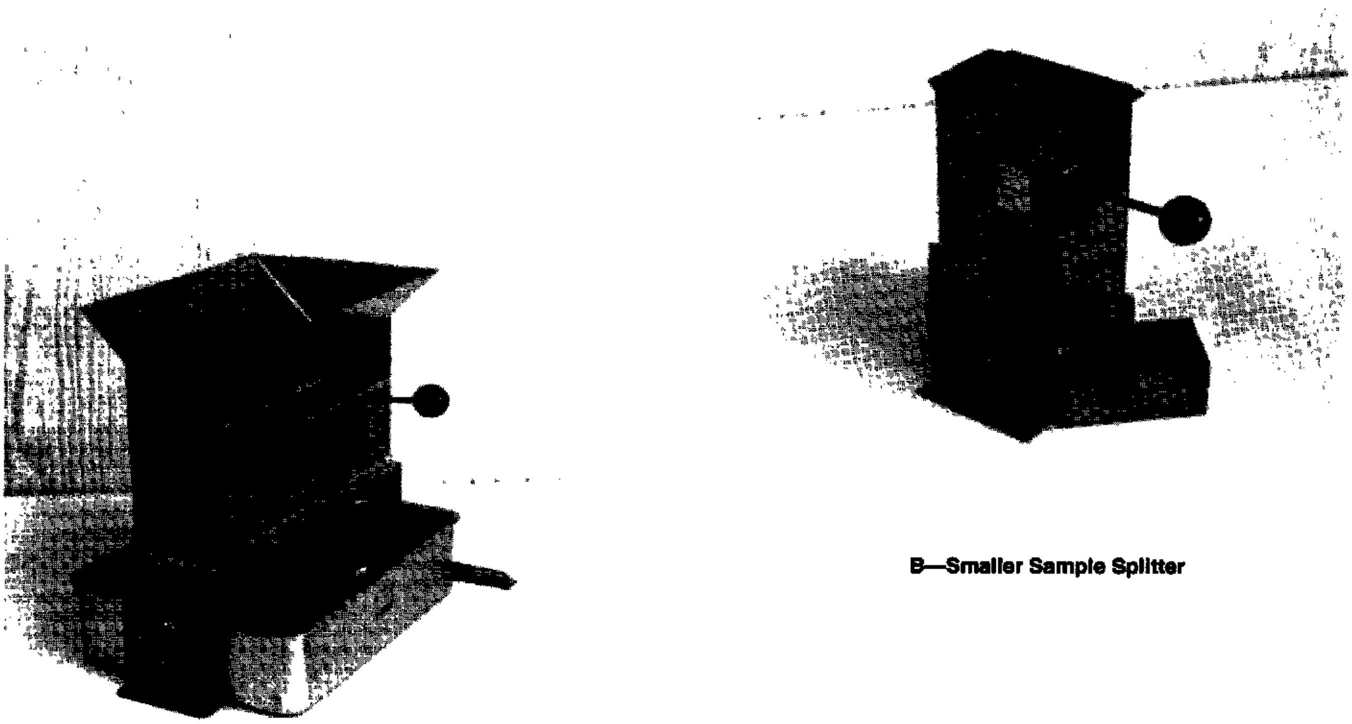


Figure 2—Example Sample Reducer Equipment

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



B—Smaller Sample Splitter

A—Larger 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

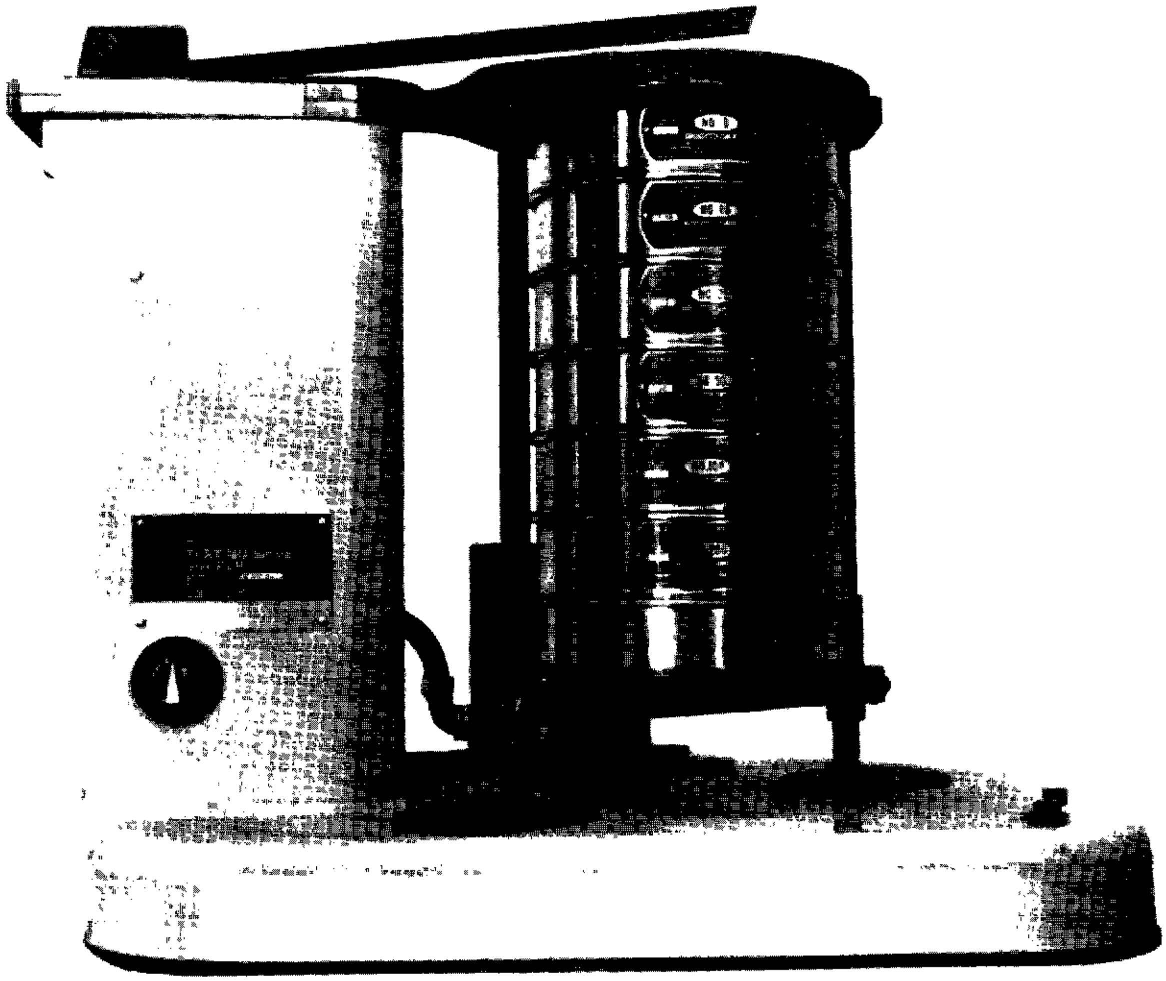


Figure 4—Example of Testing Sieve Shaker Equipment and Nest of Six U.S.A. Sieves Plus Pan

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 Gravels 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^2.

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.

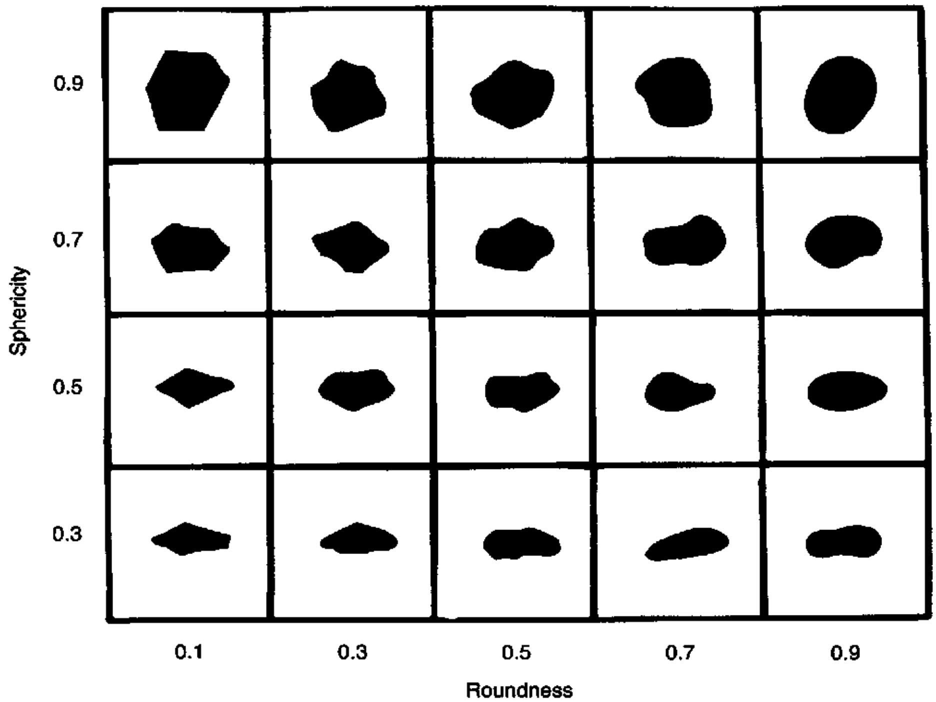


Figure 5—Chart for Visual Estimation of Sphericity and Roundness

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

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.

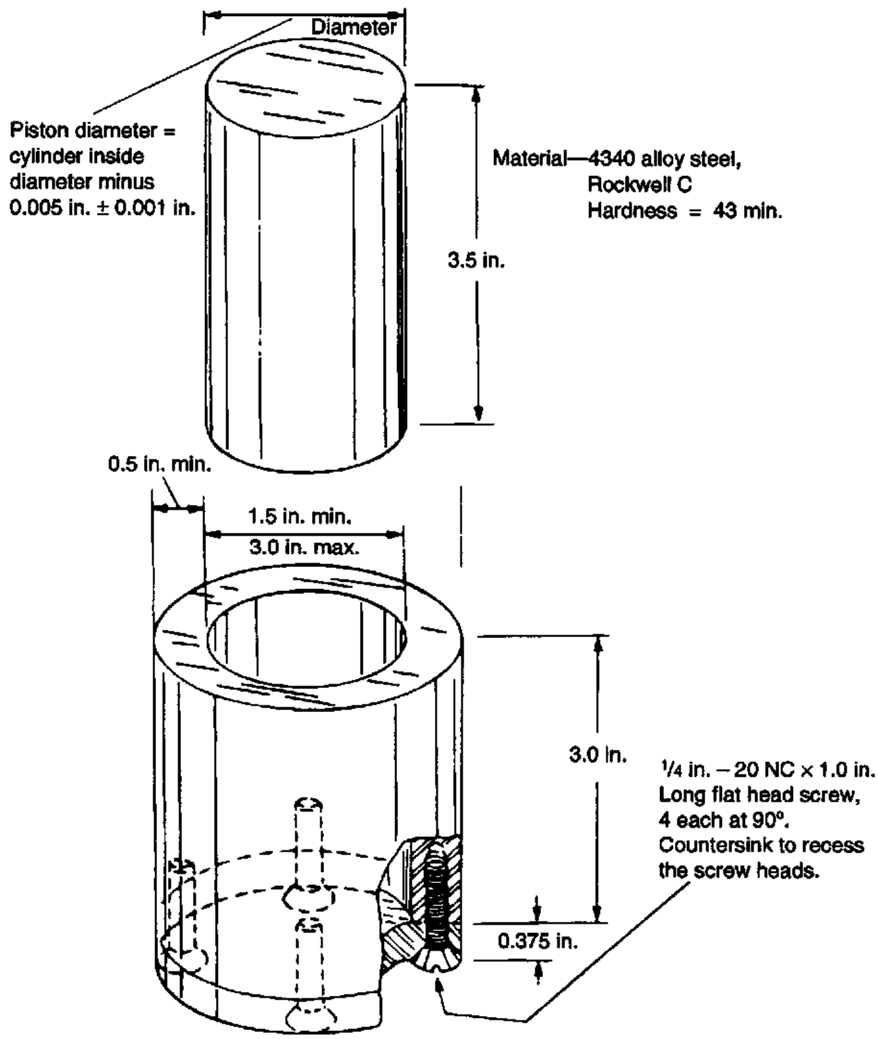


Figure 6—Example Test Cell for Proppant Crush Resistance Test

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

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

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.

$$f = \frac{100W_f}{W_p} \qquad (3)$$

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 cm³ 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:

$$\rho_b = \frac{W_{f,p} - W_f}{100} \tag{4}$$

Where:

ρ_b = Proppant bulk density, g/cm³
$W_{f,p}$ = Weight of flask and proppant (step c), g
W_f = Weight of flask (step a), g

e. Report proppant bulk density in g/cm³ and lb/ft³. (Note: lb/ft³ = g/cm³ × 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 cm³ 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:

$$D_l = \frac{W_{f,l} - W_f}{25} \tag{5}$$

Where:

D_l = Test liquid density, g/cm³
$W_{f,l}$ = Weight of flask filled with test liquid (step c), g
W_f = Weight of empty flask (step a), g
25 = Volume of pycnometer, cm³

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

Where:

D_p = Apparent density of proppant, g/cm³
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/cm³
25 = Volume of pycnometer, cm³

i. Report apparent density in g/cm³ and lb/ft³ 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:

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

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:

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

Where:

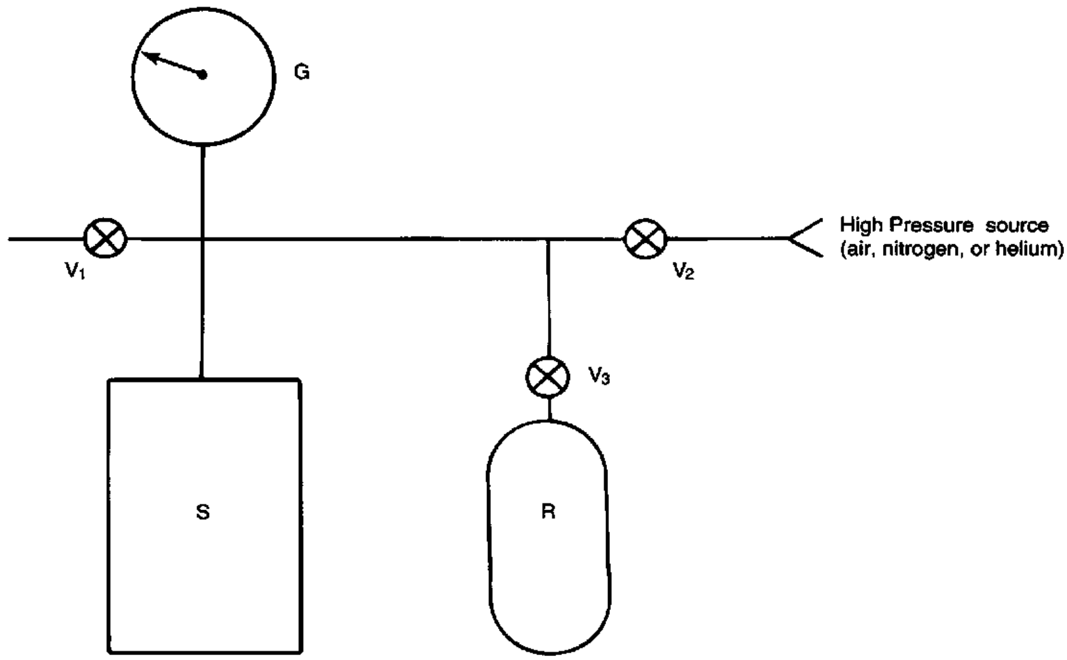
V_s = Volume of the sample cell, plumbing, and gauge, cm^3
V_r = Volume of reference cell, cm^3
P_{atm} = Atmospheric pressure, psi
P_i = Initial pressure of the reference cell, psi
P_f = Final pressure of the reference and sample cells, psi

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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