Measurement of and Specifications for Proppants Used in Hydraulic Fracturing and Gravel-packing Operations

1 Scope

This document provides specifications and standard testing procedures for evaluating proppants used in hydraulic fracturing and gravel-packing operations.

The objective of this document is to provide specifications and a consistent methodology for testing performed on hydraulic fracturing and/or gravel-packing proppants. Methodologies and specifications (where applicable) are provided for:

— sieve analysis and median diameter determination;

— sphericity and roundness;

— acid solubility;

— turbidity;

— loose pack bulk density, apparent density, and absolute density;

— crush resistance;

— loss on ignition.

Proppant size designation is as per industry standard. It is based on the maximum and minimum size determination by sieve analysis and ASTM [1] sieve number (and not as per standard opening expressed in micrometers).

Proppants in this document are sand, ceramic media, resin-coated proppants, gravel-packing media, and other similarly used materials for hydraulic fracturing and gravel-packing operations.

It is noteworthy that natural sand-based proppants that do not pass all of the criteria detailed in this document have been used in great quantities in recent hydraulic fracturing completions. These natural sand materials often do not meet a conventional sieve distribution, show unacceptable acid solubility, or fail to pass other criteria. Use of off-spec natural sand in fracturing is not recommended and should be only done with an understanding of the impact of those off-spec qualities on productivity of the associated completions.

2 Normative References

The following referenced documents are indispensable for the application of this document. For dated reference, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

[1] ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, www.astm.org

ASTM E11, *Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves*

3 Abbreviations

ANSI [2] American National Standards Institute

API American Petroleum Institute

ASTM American Society for Testing and Materials

FTU Formazin turbidity unit

LOI loss on ignition

NTU nephelometric turbidity unit

4 Standard Proppant Sampling Procedure

4.1 General

Before any sample is taken, consider what tests will be performed; each test will require different volumes. It is important that both the supplier and customer obtain the best representative sample possible. Unless the sample is truly representative of a total shipment or container, testing and correlation with specifications/standards is difficult. It is unlikely that sampling/testing methods in the field duplicate the producer's system. The standard procedures included within this document assist in obtaining representative samples; however, there are inherent variations associated with sampling, testing equipment, and procedures that can lead to inconsistent results. A sample that is representative of a truckload, 23,000 kg (50,700 lb), or a railcar load, 90,000 kg (198,000 lb), can be the initial source of wide variation when making comparisons. All parties shall ensure uniform samples. The customer and supplier shall agree on sampling and testing methods/techniques.

For the best representation, continuous sampling is ideal. Although many proppant suppliers utilize automatic sampling, it is usually impractical at the job site. If sampling is conducted while unloading a container or at the well site, consideration should be given to the number and frequency of samples.

If bulk containers are filled from a flowing stream of proppant material, sampling procedures in accordance with 4.5 shall be applied. If bulk containers are filled using sacked proppant material, sampling procedures in accordance with 4.6 shall be applied.

4.2 Particle Segregation

It is important to have a basic understanding of segregation when sampling proppant. Depending on the size, shape, distribution, and mechanisms involved, there is usually a certain amount of error or variability involved in sampling due to segregation. The sampling procedures described here are the result of much experience and are designed to minimize the effects of segregation of particles by size. While proppant has been historically provided as a dry material, wet processing of mined sand ("wet sand") is a common practice in high volume fracture sand production; despite reduced segregation in a wet sand, it remains critical to exercise caution and utilize appropriate techniques to capture representative samples of the wet sand.

[2] ANSI (Operations), 25 West 43rd Street, 4th Floor, New York, NY 10036, www.ansi.org

Particles, such as proppants, will naturally find the path of least resistance when moved or when force is applied. During transfer or movement, particles of differing size and mass naturally separate or segregate. The degree of segregation depends on the mechanisms involved in the transfer or movement.

There are several forces, such as gravity, acting on a stream of particles as it flows. Within a moving stream, fine particles drop through the voids or gaps and coarser particles move to the outside. The fine particles migrate and usually rest close to the area where they land. The heavier, coarser particles bounce or roll much further, stratifying the material by particle size.

4.3 Equipment

The following equipment shall be used to compile representative proppant material samples.

4.3.1 Sampling Device

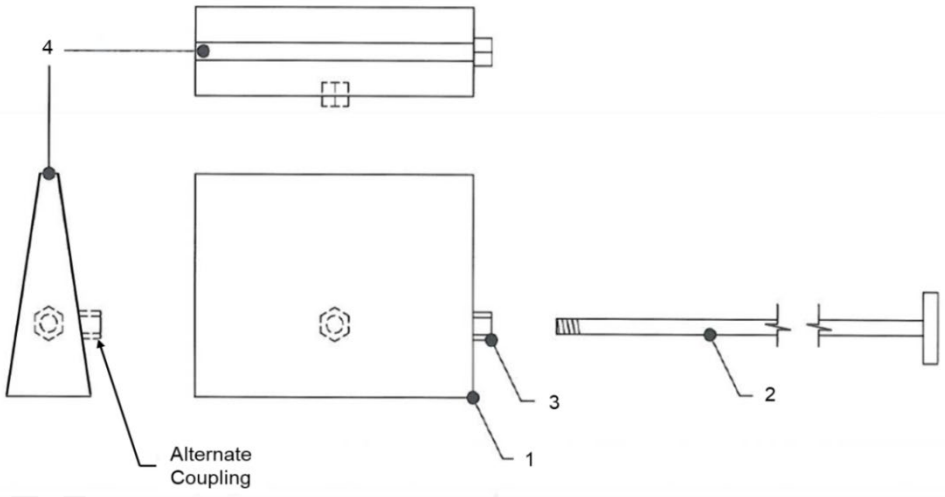
With a minimum slot opening six times the largest particle diameter sampled.

For example, a 12/20 fracturing proppant may contain particles as large as 2.36 mm, the size of the opening in a U.S. alternative No. 8 (ASTM 2.36 mm) test sieve.

The minimum slot opening is defined as:

6 x 2.36 mm = 14.2 mm or 0.557 in.

The length of the slot shall be longer than the thickness of the stream being sampled. The volume of the sampler shall be large enough so as to not overflow while cutting through the entire stream. An example of a sampling device is shown in Figure 1.



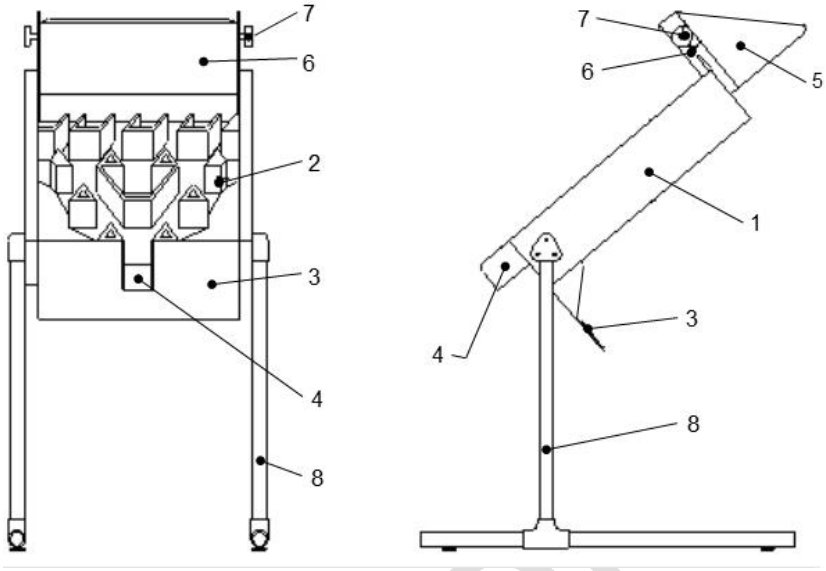
Key

1 Sampler body: aluminum; 160 mm x 210 mm x 65 mm (6.25 in. x 8.25 in. x 2.5 in.)
2 Handle: common round pipe of sufficient strength to support the sampler plus about 1.6 kg (3.5 lb) proppant; length as needed
3 Pipe coupling to accept handle (two locations)
4 Sample opening: 15 mm (0.6 in.)

Figure 1—Typical Box Sampling Device

4.3.2 Sample Reducer (16:1)

Of appropriate size for handling sack-size samples and reducing the material to $^{1}/_{16}$ of the original mass. See Figure 2.



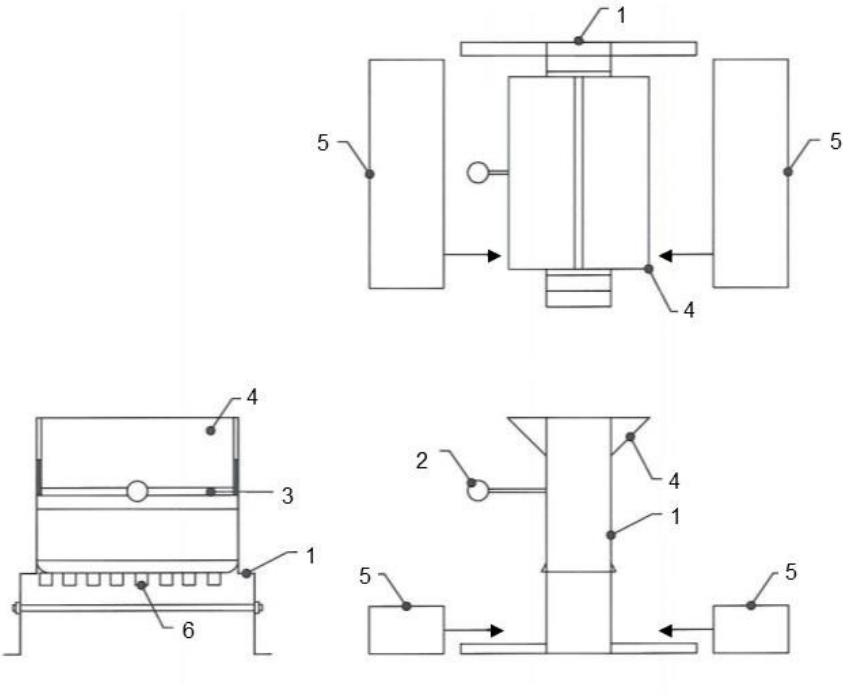
Key

1 Main body: 370 mm x 480 mm x 110 mm (14.5 in. x 19 in. x 4.5 in.)
2 Splitter plate: eight locations, 51 mm x 51 mm x 51 mm (2 in. x 2 in. x 2 in.)
3 Discharge plate: 370 mm x 300 mm x 3.2 mm (14.5 in. x 12 in. x 0.125 in.)
4 Discharge chute: 57 mm x 57 mm x 76 mm (2.25 in. x 2.25 in. x 3 in.)
5 Hopper: 370 mm x 240 mm x 150 mm (14.5 in. x 9.5 in. x 6 in.)
6 Gate: 370 mm x 190 mm x 3.5 mm (14.5 in. x 7.5 in. x 0.125 in.)
7 Hand knob: diameter 38 mm (1.5 in.)
8 Support stand assembly: 710 mm x 380 mm x 690 mm (28 in. x 15 in. x 27 in.)

Figure 2— Typical 16:1 Sample Reducer

4.3.3 Sample Splitter

Of appropriate size.



Key

1 Main body: 290 mm x 280 mm x 165 mm (11.5 in. x 11 in. x 6.5 in.)
2 Handle
3 Gate plate
4 Hopper
5 Pan
6 Splitter vanes: 16 locations, 15 mm (0.6 in.)

Figure 3—Typical Sample Splitter

4.4 Number of Required Samples—Bulk

4.4.1 Proppants for Hydraulic Fracturing

A minimum of one sample per 9000 kg (20,000 lb), or fraction thereof, shall be obtained. All samples per bulk container shall be combined and tested.

4.4.2 Gravel-packing Proppant

A minimum of one sample per 4500 kg (10,000 lb), but no fewer than two samples per job, shall be obtained, combined, and tested.

4.5 Sampling—Bulk Material

All samples shall be obtained from a flowing stream of proppant by a manual or automatic sampler. Samples shall not be taken from a static pile. The sampling device shall be used with its length perpendicular to the flowing proppant stream. The sampler shall be passed at a uniform rate from side to side through the full stream width of moving proppant. This shall be done as the material is moving to or from a conveyor belt into a blender, truck, railcar, or bulk container. Approximately 2000 kg (4400 lb) of proppant material shall be allowed to flow prior to taking the first sample. The number of samples taken shall comply with 4.4. During sampling, the sampling receptacle shall be passed completely across the moving proppant stream in a brief interval of time so as to sample the entire stream with each pass. Under no circumstances shall the sampling receptacle be allowed to overflow.

4.6 Sampling—Sacked and Bagged Material

4.6.1 Sacks Up to 50 kg (110 lb)

Only whole sacks are to be used for sampling proppant materials.

4.6.2 Totes/Bulk Bags/Super Sacks: Weighting up to 2000 kg (4400 lb)

Unless the product can be sampled in a free-flowing state, the sample of large bags presents the same problems as a static pile. Follow the same sample frequency as described in 4.4, using the sampling method described in 4.5, but allow approximately 50 kg (110 lb) to be discharged from the bulk bag before sampling.

5 Sample Handling and Storage

5.1 Sample Drying

When drying wet sand samples, it's essential to note that moisture content can vary significantly, ranging from 1% to 20%. Samples need to be thoroughly dried prior to proceeding with any testing in the following sections. It's recommended that the drying temperature does not exceed 200°F (93°C). Moreover, it's crucial to be mindful of the presence of elevated levels of clays in "wet sand", as they can lead to clumping during the drying process.

5.2 Sample Reduction

Place the contents of the combined bulk sample of proppant, or an entire sack up to 50 kg (110 lb), in the 16:1 sample reducer (see Figure 2) or equivalent. Obtain a reduced sample of approximately $^{1}/_{16}$ of the original mass of the total sack's contents, typically 3 kg (6.6 lb).

5.3 Sample Splitting

An appropriately sized sample reducer and sample splitter shall be used to prepare representative samples for testing. Place the reduced sample, obtained according to 5.1, or the sample obtained during bulk

material loading operations (refer to 4.5) in the sample splitter (see Figure 3) or equivalent. Split the sample to the desired quantity to permit performance tests as specified in this document.

5.4 Sample, Record Retention, and Storage

The proppant supplier shall maintain records of all tests conducted on each shipment for a minimum of one year. Physical samples of an amount sufficient to conduct all tests recommended herein, but in no cases less than 0.25 kg (0.5 lb), shall be retained in storage for a minimum of six months. Any material subsequently taken for testing shall be split from the retained sample. Samples shall be sealed in a type of container that is sufficient to protect the sample from contamination and moisture. Samples shall be stored in a cool, dry place.

6 Sieve Analysis

6.1 Purpose

The purpose of this section is to provide specifications for recognized proppant sizes and a standard procedure to ensure a consistent methodology for sieve analysis.

6.2 Description

The procedure and equipment described in 6.3 through 6.6 are the most widely utilized in the hydraulic fracturing and gravel-packing industry.

6.3 Apparatus and Materials

6.3.1 Sieve Sets (Two)

Complying with the requirement of ASTM Sieve Series, 200 mm (8 in.) diameter or equivalent.

One set is a working or compliance set, and the other a master or calibration set to be used for calibration or standardization only. Compliance and calibration sieves are defined in ASTM E11.

Selection of the sieve sizes to use for testing most mesh sizes using this method should follow the recommendations in Table 1. As detailed in 6.5.1, a variety of specific distributions are offered in operations for fine natural sand proppants. For those mesh sizes not listed in Table 1, an alternative set of seven sieve sizes should be selected for this measurement. The largest mesh size in the stack should be selected as two mesh sizes larger than the largest mesh (per ASTM E11) in the listed distribution; an example is selecting 30-mesh sieve as the largest to test a sample listed as 40/70 mesh. The smallest mesh size, stacked above the pan, should be two mesh sizes smaller than the smallest mesh (per ASTM E11) in the listed distribution; an example is selecting 200-mesh sieve as the smallest to test a sample listed as 50/140 mesh. The five middle sized sieves should include the two sizes in the listed distribution of the sample with three intermediate mesh sizes between. In an example of selecting sieves for a custom mesh size, a sample listed as “40/200” might be assessed with a stack series comprising 30, 40, 60, 80 140, 200, and 270 mesh over the pan.

NOTE For informational purposes, an excerpt from E11 is included in this document as Annex A.

Table 1—ASTM Test Sieve Sizes

Sieve-opening Sizes μm											
	3350/ 1700	2360/ 1180	1700/ 1000	1700/ 850	1180/ 850	1180/ 600	850/ 425	600/ 300	425/ 250	425/ 212	212/ 106
Typical Proppant/Gravel-pack Size Designations											
	6/12	8/16	12/18	12/20	16/20	16/30	20/40	30/50	40/60	40/70	70/140
Stack of ASTM Sieves [a, b]											
Upper designating sieve in bold type	4	6	8	8	12	12	16	20	30	30	50
	6	**8**	**12**	**12**	**16**	**16**	**20**	**30**	**40**	**40**	**70**
	8	10	14	14	18	18	25	35	45	45	80
Lower designating sieve in bold type	10	12	16	16	**20**	20	30	40	50	50	100
	12	14	**18**	18	25	25	35	45	**60**	60	120
	14	**16**	20	**20**	30	**30**	**40**	**50**	70	**70**	**140**
	16	20	30	30	40	40	50	70	100	100	200
	pan	pan	pan	pan	pan	pan	pan	pan	pan	pan	pan

[a] Sieve series as defined in ASTM E11 (U.S. Alternative designation); refer to Annex A for opening size in μm.

[b] Test sieve stacked in order from top to bottom, largest opening on top.

6.3.2 Testing Sieve Shaker

To provide simultaneous rotating and tapping action, and accept the sieves specified in Table 1.

To mitigate the effect of static charge, the stack of sieves should be grounded. The preferred shaker will have performance characteristics as follows:

— oscillations per minute: 278 ±10

— oscillation displacement: 28.6 ±11.1 mm (1.125 in. ±0.4375 in.)

— taps per minute: 150 ±10

— height of the tapper: 33.3 ±1.6 mm (1.3125 in. ±0.0625 in.)

— hammer weight: 2.44 kg (5.38 lb)

— timer accuracy: ±5 seconds over a 10-minute interval

6.3.3 Balance

Minimum capacity of 100 g with precision of 0.01 g or better.

6.3.4 Brushes

Nylon or equivalent.

6.4 Procedure

6.4.1 Stack a minimum of seven sieves, checked against a master set, plus a pan and cover, in a stack of decreasing sieve opening sizes from top to bottom. Table 1 establishes sieve sizes for use in testing designated example proppant sizes. Table 1 should be used as a guide and does not attempt to preclude the use of other grades that are or may become available.

6.4.2 Weigh each clean sieve and record the mass.

6.4.3 Using a split sample of 100 g ±20 g, obtain an accurate initial mass, m_{init}, and record the mass to the nearest 0.01 g.

6.4.4 Pour the split and weighed sample onto the top sieve. Place the stack of sieves plus the pan and lid in the test sieve shaker and shake for 10 minutes ±5 seconds.

6.4.5 Remove the sieve stack from the test sieve shaker.

6.4.6 Weigh each sieve with the proppant. Invert the sieve on a collection device and remove the proppant by thoroughly brushing the sieve to ensure that grains are not adhering to the wire cloth. Calculate the mass retained by subtracting the empty sieve mass from the sieve plus proppant mass. Record the mass of proppant retained on each of the sieves and the pan, m_i.

6.4.7 The cumulative mass shall be within 0.5 % of the initial sample mass, m_{init}, used in the test. If not, the sieve analysis shall be repeated using a different sample.

6.4.8 Calculate the percent by mass or frequency, n_i, of the total proppant sample retained on each sieve and in the pan using Equation (1).

$$n_i = 100 \times \frac{m_i}{\sum m_i} \tag{1}$$

where

m_i is the measured mass of proppant retained on the sieve of interest, expressed in grams

Σm_i is the sum of each of the measured masses retained on the sieves and pan, expressed in grams

6.5 Specifications—Sieve Analysis of Proppants

6.5.1 Fracturing Proppant Sizes

A minimum of 90.0 % of the tested proppant sample shall pass through the coarse designated sieve and be retained on or above the fine designated sieve, i.e. 12/20, 20/40, 40/70. Not over 0.1 % of the total tested proppant sample shall be larger than the first sieve in the stack specified in Table 1, and not over 1.0 % of the total tested proppant sample shall be retained in the pan. For example, a 20/40 proppant sample shall have no more than 0.1 % of the total tested proppant sample retained on the No. 16 sieve and no more than 1.0 % of the total tested proppant sample shall be retained in the pan. The median diameter, d_{50}, and the particle size distribution of each grade shall be made available.

6.5.2 100 Mesh Sizes

The term “100 mesh” has been used for years to detail the finest mesh sizes of proppant used in practice, commonly natural sands. With time, many specific sub-distributions are offered, i.e. 40/140, 40/170, 70/140 that have been classified under the broader term “100 mesh” proppant. This document should not reference

sand mesh size in the broader “100 mesh” term and instead samples should be described by the specific sub-distribution of sizes which the sample is confirmed to meet the acceptance criteria listed in 6.5.1.

6.5.3 Gravel-packing Proppant Sizes

A minimum of 96.0 % of the tested proppant sample shall pass the coarse designated sieve and be retained on the fine designated sieve, i.e. 12/20, 20/40, 40/60. Not over 0.1 % of the total tested proppant sample shall be larger than the first/top sieve in the stack specified in Table 1, and not over 1.0 % of the total tested proppant shall be smaller than the last designated sieve size. For example, a 20/40 proppant sample shall have no more than 0.1 % of the total tested sample retained on the No. 16 sieve and no more than 1.0 % of the total tested sample shall be retained in the pan. The median diameter, d_{50}, and the particle size distribution of each grade shall be made available.

6.5.4 Example Sieve Analysis and Calculations

In Table 2, the first column is a listing of the sieve numbers used to test a sample of 16/30 sand. The numbers and labels are lifted directly from Table 1.

— Column 2 is the position of each sieve in the stack or nest assembled before testing begins. The largest holes (smallest sieve number) are on the top of the stack, position 1, with the other sieves below in sequence: smallest sieve number down to the largest sieve number, then the pan at the bottom of the nest, position 8.

— Column 3 is the specification listed next to the appropriate sieves or labels.

— Column 4 shows the proppant mass retained on each of the sieves and the pan.

— Column 5 shows the result of calculating the weight percent retained for each of the sieves and the pan.

— Column 6 shows the cumulative percent retained numbers.

Table 2—Example Sieve Analysis and Calculations (Fracturing 16/30 Proppant)

ASTM Sieve Series U.S. Alternative Sieve Number (opening size)		**Sieve in Stack Position** i	**Specification** % by mass	**Mass Retained** m_i g	**Mass Retained** or **Frequency** n_i % by mass	**Cumulative Mass Retained** % by mass
12	(1.7 mm)	1 (top)	≤ 0.1	0.00	**0.0**	0.0
16	(1.18 mm)	2		1.20	1.19	1.2
18	(1.0 mm)	3	≥ 90.0	37.90	37.63	38.8
20	(850 µm)	4		48.70	48.36	87.2
25	(710 µm)	5		11.90	11.82	99.0
30	(600 µm)	6		0.30	0.30	99.3
40	(425 µm)	7		0.20	0.20	99.5
Pan		8 (bottom)	≤ 1.0	0.50	**0.50**	100.0
Total				100.70	100.0	
Initial Mass, m_{init}, g			80 to 120 g	**100.50**		
Difference			≤ 0.5	–0.20	**–0.2**	
In-size			≥ 90.0		**98.1**	

The bottom rows of Table 2, beginning with the row labeled “total,” are various calculations used to evaluate the sieve analysis results against the specifications. It is helpful to the observer that the results related to each of the five specifications for sieve analysis are in bold font with bold cell margins.

— The first specification is the initial sample mass and is recorded in column 4, opposite the label “Initial Mass, m_{init}, g.”

— The second specification is the difference between the initial sample mass, m_{init}, and the sum of all the retained masses, and is recorded in column 5 opposite the label “Difference.” The negative number –0.2 % is within specification and indicates that a small amount of proppant was picked up from material left in the sieves or handling equipment from the last time it was used.

— The third specification is the amount of material retained on the top sieve and is recorded in column 5 opposite the No.12 sieve. Note that 0.0 % is less than 0.1 % and is within specification.

— The fourth specification is the mass of proppant retained in the pan and is recorded in column 5 opposite the label “Pan.” Note that 0.5 % is less than 1.0 % and is within specification.

— The fifth and last specification is the mass of proppant considered to be “in-size.” In this case, the cumulative mass of proppant passing through the No. 16 sieve and retained on the No. 30 sieve is calculated and recorded in column 5 opposite the label “In-size.” Note that 98.1 % is greater than the 90 % and is within specification.

Since each of the five specification items passes the limitations required by this section, the sample of 16/30 sand has the properties of an acceptable 16/30 fracturing proppant with respect to particle size distribution.

6.5.5 Proppant Median Diameter, d_{50}

The median diameter, d_{50}, is used to characterize proppant size distribution for hydraulic fracturing and gravel-packing and is derived from the particle size characterization or sieve analysis described in 6.4, and reported in addition to that analysis.

In gravel-packing, the median diameter, d_{50}, the 50th mass percentile, is commonly used to evaluate the suitability of gravel to retain formation sand. In hydraulic fracturing, this same value is used to predict long-term conductivity performance, as well as other properties directly related to demonstrable flow capacity or potential productivity of the well. Both disciplines calculate the number as follows:

6.5.5.1 Calculating Median Diameter, d_{50}

The sieve analysis obtained in 6.4 and demonstrated in Table 2 is repeated for clarity in Table 3, columns 1, 5, and 6 for the 16/30 proppant. The cumulative percent of the material retained by all the sieves and the pan should be 100 %. Refer to Table 3, column 6.

The median diameter is calculated using a variable called phi, Φ_i, calculated in column 7 of Table 3, using Equation (2) as one-half the difference in the negative logarithm (base 2) of the opening sizes of the sieve of interest and the just previous sieve multiplied by the frequency of that fraction.

$$\Phi_i = -1/2 \times \log_2\left(O_{i-1}\right) + \log_2\left(O_i\right) \times n_i \tag{2}$$

where

O_i is the sieve opening size of interest, expressed in millimeters

n_i is the relative mass retained or frequency of occurrence, expressed as percent by mass

Median phi, Φ_{50}, is the sum of those differences divided by the sum of the frequencies and is calculated by Equation (3):

$$\Phi_{50} = \sum \Phi_i \Big/ \sum n_i \tag{3}$$

Median diameter, d_{50}, is the anti-logarithm (base 2) of median phi, $\Phi_{50,}$ and is calculated using Equation (4):

$$d_{50} = 2^{-\Phi_{50}} \tag{4}$$

Table 3—Example Calculation of Median Diameter (Fracturing 16/30 Proppant)

ASTM Sieve Series		Sieve in Stack Position	Mass Retained	Mass Retained or Frequency	Cumulative Mass Retained	Phi
Sieve Number	Sieve Opening O_i mm	i	g	n_i % by mass	% by mass	Φ_i
12	1.700	1	0.00	0.0	0.0	0.0000
16	1.180	2	1.20	1.19	1.2	-0.5984
18	1.000	3	37.90	37.63	38.8	-4.4936
20	0.850	4	48.70	48.36	87.2	5.6695
25	0.710	5	11.90	11.82	99.0	4.3049
30	0.600	6	0.30	0.30	99.3	0.1834
40	0.425	7	0.20	0.20	99.5	0.1958
pan	0.213 [a]	8	0.50	0.50	100.0	0.8604
		Total	100.70	100.0		6.1220
					Median phi, Φ_{50}	0.061
					Median Diameter, d_{50} mm (in.)	0.958 (0.038)

[a] For calculation, pan is considered with an opening of 1/2 of the opening of the last bottom test sieve.

6.5.5.2 Graphical Determination of Median Diameter, d_{50}

The median diameter can be determined using a graphical technique as depicted in Figure 4. Plot the particle-size distribution curve with cumulative percent retained on the dependent axis (y-axis) as a function of the log of the average sieve opening on the independent axis (x-axis). The independent axis (x-axis) shall be inverted or flipped, left to right, so that the scale is smaller to the right and larger to the left. The sieve size can be plotted as the logarithm (base 2) of microns, millimeters, or inches. A plot of the example in Table 2 and Table 3 is illustrated in Figure 4. Reading the graph at 50 % cumulative mass (on the y-axis) gives the median particle diameter, d_{50}, (on the *x*-axis) of 0.96 mm (0.038 in.).

d_{50} is the diameter at which 50 % of the particles are smaller and 50 % are larger.

From Figure 4, other common criteria, such as d_{90} (used in gravel-packing design) can be determined. Reading the graph at 90 % cumulative mass (on the y-axis) gives the d_{90} a grain diameter (on the x-axis) of 0.83 mm (0.033 in.).

Refer to columns 2 and 7 in Table 3 for example calculations illustrating this graphical technique.

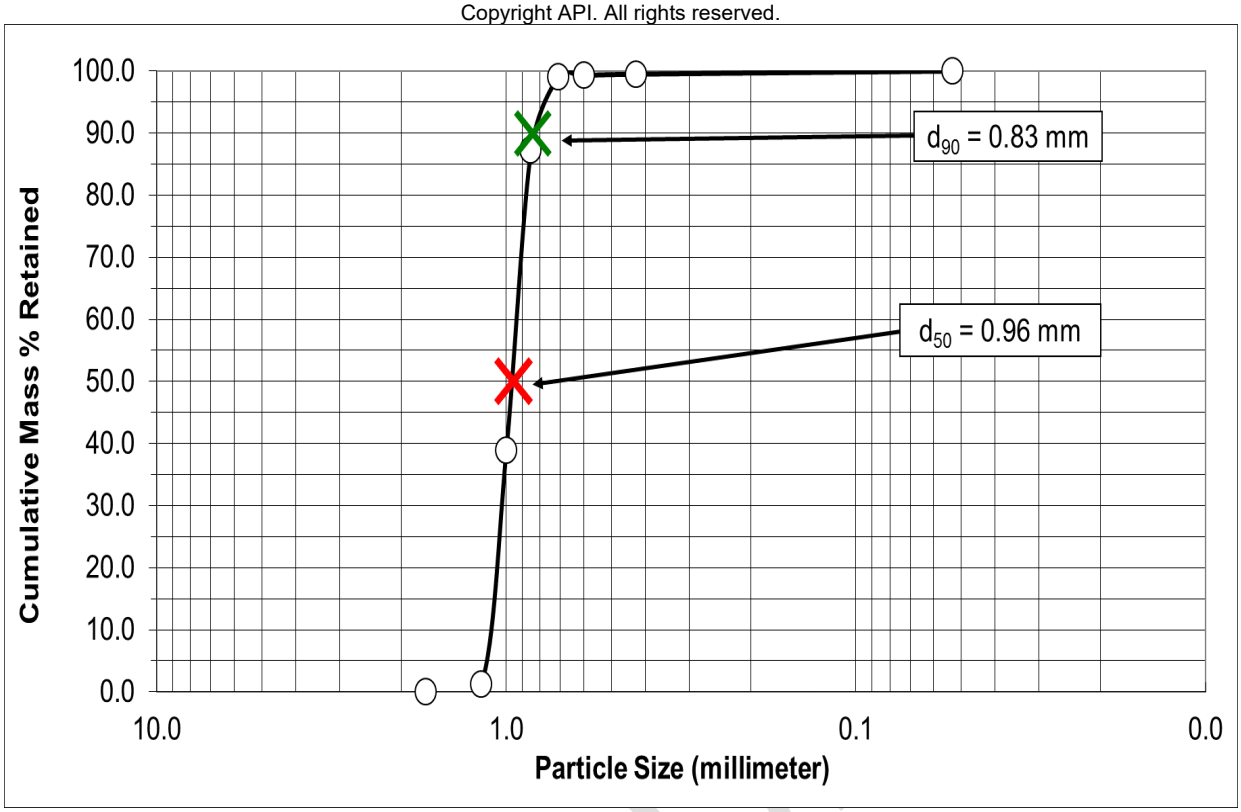


Figure 4—Graphical Method for Determination of Median Diameter

6.6 Sieve Calibration

6.6.1 Purpose

It is necessary to check sieves against a master set because sieves are not perfect and are subject to wear. This is true for both new and used sieves and for matched and unmatched sieves. Optical examination of any sieve shows the openings vary both in size and shape. Calibration is a means by which the extent and the effect of the opening differences can be determined. It is also a means to compensate for manufactured differences in sieves to ensure consistency in sieve analysis by comparing one sieve set with another.

6.6.2 Description

A stack of master sieves is used to check working sieves. This master stack (calibration sieves) should be used very sparingly to prevent major wear and changes in the openings. The master stack consists of certified sieves that are optically calibrated on an annual basis, typically by the original equipment manufacturer, to ensure the sieves are in accordance with ASTM E11. If any sieve fails to meet the ASTM specification, the sieve shall be replaced with a new calibrated sieve. Storage of sieves shall be done in such a manner as to prevent deterioration and/or damage.

6.6.3 Preparing Calibration Samples

A sieve calibration sample is prepared by blending sized samples with a minimum of 0.6, but preferably 0.8, in sphericity and roundness. To prepare the sized samples, determine the specific size of material(s) needed, then assemble a sieve stack that covers these sizes and place a pan at the bottom.

6.6.3.1 Place 100 g ±20 g of proppant that has been identified as a source for calibration material onto the top sieve and cover with a lid. Place the sieve stack into the sieve shaker and shake for 10 minutes ±5 seconds. The mass of media used should be adjusted to a maximum of 35 g on any single sieve.

6.6.3.2 Remove the stack from the sieve shaker, then remove the lid. Carefully remove the top sieve and invert onto a recovery pan. Brush the sieve thoroughly, removing any grains that adhere to the wire cloth. Place the material from the recovery pan into a storage container labeled specifically for this size of material. Repeat for each sieve.

6.6.3.3 Select the sieve sizes to be tested based on Table 1. For each sieve size, weigh approximately 10 g of correspondingly sized material. Blend the sized material to form a calibration standard sample.

6.6.4 Calibration Procedure

6.6.4.1 Place the calibration standard sample from 0 into the master sieve stack and complete sieve analysis according to 6.4, recombining each of the sized fractions after they are weighed. The load retained on each of the master sieves will be considered the master load for calculation purposes.

6.6.4.2 The recombined calibration sample is then placed into the working sieve stack, and sieve analysis is completed according to 6.4. The load retained on each working sieve is considered the working load for calculation purposes.

6.6.4.3 If the total test sample mass exceeds ±0.5 % difference from the master stack to the working stack, repeat the test.

6.6.4.4 For each sieve in the stack, calculate:

a) the difference, δ_i, expressed in grams, using Equation (5)

b) the relative difference, Δ_i, in percent, using Equation (6)

c) the absolute difference, δ_{Ai}, expressed in grams, using Equation (7)

d) the absolute relative difference, Δ_{Ai}, in percent, using Equation (8)

If the absolute relative difference value exceeds 15 %, the working sieve shall be replaced.

$$\delta_i = m_{Wi} - m_{Mi} \tag{5}$$

$$\Delta_i = 100 \times \left(\frac{m_{Wi}}{m_{Mi}} - 1 \right) \tag{6}$$

where

m_{Mi} is the mass retained on each sieve in the master set, expressed in grams

m_{Wi} is the mass retained on each sieve in the working set, expressed in grams

NOTE Both δ_i and Δ_i can be positive or negative.

The absolute difference of the sieve of interest, δ_{Ai}, expressed in grams, is calculated from Equation (7):

$$\delta_{Ai} = \delta_{Ai-1} + \delta_i \quad \delta_{Ai} = \delta_{Ai-1} + \delta_i \tag{7}$$

where

δ_{Ai-1} is the absolute difference of the preceding sieve, expressed in grams;

δ_i is the difference for the sieve of interest, expressed in grams.

The absolute relative difference of the sieve of interest, Δ_{Ai}, is calculated, in percent, from Equation (8):

$$\Delta_{Ai} = 100 \times \frac{\delta_{Ai}}{m_{Mi}} \quad (8)$$

where

δ_{Ai} is the absolute difference for the sieve of interest from Equation (7)

m_{Mi} is the mass retained in the master sieve of interest, expressed in grams

NOTE Use of the word “absolute” is not referring to the mathematical function.

EXAMPLE Example calculations for the sixth sieve in the working stack, No. 30 (highlighted), in Table 4:

— From Equation (5): δ_6 = 10.30 – 10.05 = 0.25 g

— From Equation (6): Δ_6 = (10.30 / 10.05 – 1) x 100 = 2.5 %

— From Equation (7): δ_{A6} = 1.33 + 0.25 = 1.58 g

— From Equation (8): Δ_{A6} = (1.58 / 10.05) x 100 = 15.7 %

Table 4—Example of Absolute Relative Difference Calculations

ASTM Sieve Series Sieve Number	Sieve in Stack Position i	Master Load m_{Mi} g	Working Load m_{Wi} g	Difference δ_i g	Relative Difference Δ_i %	Absolute Difference δ_{Ai} g	Absolute Relative Difference Δ_{Ai} %
12	1 (top)	9.42	10.00	0.58	6.2	0.58	**6.2**
16	2	10.90	11.05	0.15	1.4	0.73	**6.7**
18	3	9.51	9.51	0.00	0.0	0.73	**7.7**
20	4	9.50	9.70	0.20	2.1	0.93	**9.8**
25	5	10.10	10.50	0.40	4.0	1.33	**13.2**
30	6	10.05	10.30	0.25	2.5	1.58	**15.7**
35	7	11.44	10.95	-0.49	-4.3	1.09	**9.5**
40	8	9.33	9.19	-0.14	-1.5	0.95	**10.2**
50	9	10.12	9.18	-0.94	-9.3	0.01	**0.1**
70	10	8.80	8.90	0.10	1.1	0.11	**1.2**
Pan	11 (bottom)	0.90	0.47				
Total		**100.07**	**99.75**				

In conclusion, the relative difference between the working set total and the master set total is -0.3 % [(99.75 / 100.07 – 1) x 100] and is within tolerance specified in 0 and 0 of less than ±0.5 %. Absolute relative difference for the sixth sieve, No. 30 (highlighted), in the working stack exceeds the allowable 15 % and shall be replaced with a new calibrated sieve. All other sieves in the working stack are in calibration, as indicated by the bold numbers in the right-most column of Table 4.

7 Proppant Sphericity and Roundness

7.1 Purpose

The purpose of this procedure is to evaluate and report proppant particle shapes.

7.2 Description

The common particle shape parameters that have been found to be useful for visually evaluating proppants are sphericity and roundness. This procedure finds its greatest utility in the characterization of new proppant deposits and new sources of man-made proppants. The most widely used method of determining sphericity and roundness is the use of the Krumbein and Sloss chart (see Figure 5). Sphericity is a measure of how close a proppant particle approaches the shape of a sphere. Roundness is a measure of the relative sharpness of corners or curvature. These measurements shall be determined separately. Distinct measurement methods utilizing photographic or digital technology are available and are acceptable as long as they actually measure and calculate the properties described here.

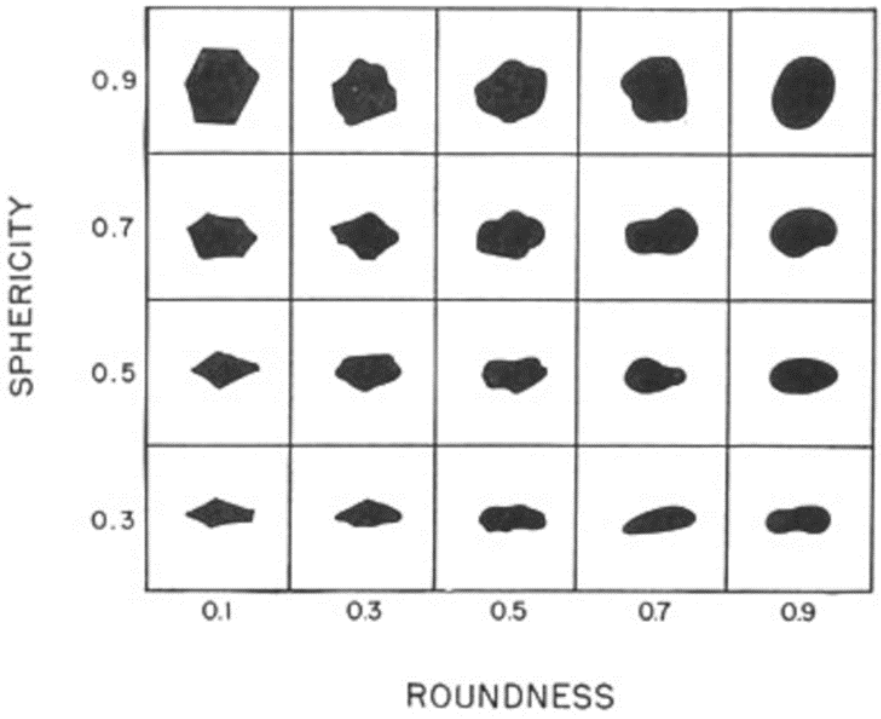


Figure 5—Chart for Visual Estimation of Sphericity and Roundness

7.3 Apparatus Capability

A microscope or equivalent shall have 10 times to 40 times magnification

7.4 Procedure

7.4.1 Using the split sample (see 5.2) and the sample splitter, further reduce the sample to a monolayer of proppant.

7.4.2 Place the reduced sample on a suitable background, spread it out to a one-particle-thickness layer, and view it through a microscope at low magnification (10 times to 40 times magnification). Choose a darker background color for a light-colored proppant and, conversely, choose a lighter background color for dark particles.

7.4.3 To prevent inadvertent skewing of the sample and data, every particle in the central field of view shall be evaluated. At least 20 particles should be assessed for roundness and sphericity in each sample. Particles should not be touching (if possible).

7.4.4 Determine the roundness and sphericity of each selected particle by comparing with the chart provided (refer to Figure 5). Assessment of the roundness and sphericity can be carried out manually or using digital technology both assessment types should adhere to the standard shapes in Figure 5. Record the assigned roundness and sphericity number for each particle selected.

7.4.5 Calculate the arithmetic average of the recorded sphericity numbers and report as average particle sphericity to the nearest 0.1 unit.

7.4.6 Calculate the arithmetic average of the recorded roundness numbers and report as average particle roundness to the nearest 0.1 unit.

7.5 Alternate Method for Determining Average Sphericity and Roundness

7.5.1 Procedure

Follow the procedure in 7.4, but use the photographic equipment of choice. Select a representative field of view under the lens with the camera angle perpendicular to the samples and capture a photograph of the proppant. Numbering each selected particle on the printed image limits confusion and assures the same particles are used for both the sphericity and the roundness determinations. Continue with the measurements as in 7.4.

7.5.2 Suggested Magnification for Photomicrographs

For designated proppant size ranges, the suggested magnifications are shown in Table 5.

Table 5—Suggested Magnification

Proppant Size Ranges	Photomicrograph Magnification
6/12 through 12/20	15 times
16/12 through 20/40	30 times
30/50 through 70/140	40 times

7.6 Specifications—Proppant Roundness and Sphericity

Ceramic proppants and resin-coated ceramic proppants shall have an average sphericity of 0.7 or greater and an average roundness of 0.7 or greater. All other proppants shall have an average sphericity of 0.6 or greater and an average roundness of 0.6 or greater.

8 Acid Solubility

8.1 Purpose

Acid-solubility procedures are used to determine the suitability of a proppant for use in applications where the proppant can come into contact with acids. In the case of naturally occurring crystalline α-quartz frac sand, the solubility of the proppant in a mixture of hydrofluoric acid in hydrochloric acid is used to estimate the effectiveness of mineral separation techniques in removing the softer and less desirable cementitious minerals from the quartz sand. In the case of man-made ceramic proppants, the amorphous glass phases are also soluble in the HF/HCl acid mixtures, as are other compounds, such as magnetite, alkalis, and iron oxides.

8.2 Description

The preferred method of testing acid solubility is to use a solution of 12:3 HCl:HF acid; i.e., 12 % by mass of hydrochloric acid (HCl) and 3 % by mass of hydrofluoric acid (HF) (see 8.4.2). However, the following procedural example shall not preclude the testing of materials in other acids.

The solubility of a proppant in 12:3 HCl:HF is an indication of the amount of soluble materials; e.g. carbonates, feldspars, iron oxides, clays, etc., present in the proppant.

Caution—Hydrochloric acid and 12:3 HCl:HF acid solutions are highly corrosive. Always handle them with care to prevent harm or injury. Take specific safety precautions when handling, transporting, and storing HCl and 12:3 HCl:HF. Get medical help immediately if accidental contact occurs.

8.3 Apparatus and Materials

The following equipment and materials are needed to conduct acid-solubility tests on proppant samples.

8.3.1 Hydrochloric Acid

HCl, CAS No. 7647-01-0, concentrated, 37 % in water, reagent grade of known assay.

NOTE Hydrochloric acid is a mixture/solution of hydrogen chloride gas in water; the same CAS registry number is used for the gas and aqueous solutions.

8.3.2 Ammonium Hydrogen Difluoride (ammonium bifluoride)

NH_4HF_2, CAS No. 1341-49-7, reagent grade of known assay.

8.3.3 Deionized (or Distilled) Water

For dilution of the acid and washing of the filtered sample.

8.3.4 Balance

0.0001 g accuracy.

8.3.5 Water Bath

66 °C (150 °F).

8.3.6 Oven

105 °C (220 °F).

8.3.7 Beaker

250-ml capacity, high-density polyethylene or polypropylene.

8.3.8 Graduated Cylinder or Volumetric Flask

1000 ml, high-density polyethylene or polypropylene.

8.3.9 Analytical Filtering Apparatus

Acid-resistant (porcelain, high-density polyethylene, or polysulfone), using a vacuum-filtering technique with a Gooch-style crucible or Buchner-style funnel.

8.3.10 Filter Paper

Circles, cellulose-based, acid-resistant sufficient to prevent the loss of the sample.

The test requires a pad of filter paper about 1.6 mm ($^1/_{16}$ in.) thick or about five layers of filter paper circles with a diameter to lay flat in the crucible or funnel. Suitable filter paper is commonly known as Whatman No. 42 or equivalent.

8.3.11 Timer

Accurate to ±5 seconds over a 30-minute period.

8.3.12 Desiccator

With standard drying agent (anhydrous calcium sulfate or silica gel).

8.4 Procedure

Representative proppant samples shall be taken from the sample splitter (see 5.2). Do not sieve the sample. Samples should not be subjected to the crush resistance test or ground prior to the acid solubility analysis; the analysis shall be performed on the unaltered proppant.

Warning—Extreme caution and proper protective equipment shall be used at all times when handling acids. For more information, consult material safety data sheets or the chemical manufacturer.

8.4.1 Preparation of the 12:3 HCl:HF Acid Solution

Prepare the 12:3 HCl:HF acid solution, specific gravity = 1.08 at 15.6 °C (60 °F), as follows:

8.4.1.1 To 500 ml of water contained in a polyethylene or polypropylene 1000-ml graduated cylinder or volumetric flask, add 46.23 g of pure NH_4HF_2 and mix. The actual mass of NH_4HF_2, of assay less than 100 % purity, to be used is equal to 46.23 g divided by the assay of NH_4HF_2, in mass fraction. For example, 46.23 g / 0.94 = 49.18 g of NH_4HF_2 having an assay of 94 % purity shall be used.

8.4.1.2 Add 361 ml 37 % HCl, (specific gravity of 1.19). Adjust volume for different concentrations of hydrochloric acid. For example, using 33 % HCl (specific gravity 1.17), the correct amount of acid would be (361 ml x 1.19 x 0.37) / (0.33 x 1.17) = 411.7 ml of HCl having an assay of 33 %.

8.4.1.3 Dilute to 1000 ml with water. Recheck the volume when the acid has cooled to room temperature and top off as needed.

8.4.1.4 Stir to ensure complete mixing and be sure the acid has cooled to room temperature before use.

8.4.2 Proppant Solubility Test Procedure

8.4.2.1 Dry the funnel and filter paper in an oven at 105 °C (220 °F) for a minimum of one hour and until constant mass is achieved; weigh and record the mass, m_f, to the nearest 0.1 mg. The filter medium should not be weighed hot; it should first be allowed to cool in a desiccator.

8.4.2.2 Weigh 5 g ±0.01 g of proppant to the nearest 0.1 mg into a tared sample pan. The proppant should be dried at 105 °C (220 °F) to a constant mass and cooled in a desiccator. Record the mass, m_s, to the nearest 0.1 mg.

8.4.2.3 Add the proppant sample to a 250-ml polyethylene beaker containing 100 ml of the acid solution prepared in 8.4.1. Cover the beaker with a polyethylene watch glass. The acid and the samples shall be at room temperature, 22 °C ±3 °C (72 °F ±5 °F).

8.4.2.4 Place the beaker in a 66 °C (150 °F) water bath for 30 minutes +5 seconds. Do not stir.

8.4.2.5 Transfer the proppant and acid mixture from the beaker to the filtering apparatus. Filter the sample through the pre-weighed filter crucible/funnel, being sure to transfer all particles from the beaker to the filter. Vacuum-filtering techniques shall be used to remove the acid from the sample within one minute. A larger filter area may be required for some samples to be able to filter this rapidly.

8.4.2.6 Wash the sample in the filtering apparatus at least three times with sufficient volume of water to flood the remaining solids; 20-ml portions of water should be sufficient for the sample size and the filters listed.

8.4.2.7 Dry the filter and retained sample at 105 °C (220 °F) for a minimum of one hour and until a constant mass is obtained. Cool the filter and sample in a desiccator before weighing. Weigh the filter and sample and record the mass, m_{fs}, to the nearest 0.1 mg.

8.4.2.8 Calculate $S_{12:3}$, the acid solubility (expressed in percent), of the proppant using Equation (9) and report the result to one decimal place:

$$S_{12:3} = \frac{m_s + m_f - m_{fs}}{m_s} \times 100 \quad (9)$$

where

m_s is the sample mass, expressed in grams

m_f is the mass of the filter, expressed in grams

m_{fs} is the dried mass of the filter containing proppant, expressed in grams

8.5 Specifications—Acid Solubility

Acid-soluble material in proppants shall not exceed the values shown in Table 6.

Table 6—Proppant Maximum Acid Solubility

Proppant Type and Size	Maximum Acid Solubility % by weight
Hydraulic fracturing sand, resin-coated proppant, and gravel-packing sand proppants	
— Larger or equal to 30/50	2.0
— Smaller than 30/50	3.0
Ceramic proppants and resin-coated ceramic proppants	7.0

9 Turbidity

9.1 Purpose

The purpose of this procedure is to determine the amount of suspended particles or other finely divided matter present.

9.2 Description

In general, turbidity tests measure an optical property of a suspension that results from the scattering and absorption of light by the particulate matter suspended in the wetting fluid. The higher the turbidity number, the more suspended particles are present. In most commercial turbidity meters, the incident light path is normal to the detection path; this is the preferred method of measurement. The results are expressed in nephelometric turbidity units (NTU), or the equivalent formazin turbidity units (FTU) when formazin is used as the calibration standard. Alternate methods may be used, but shall be correlated with the method given in this document.

The updated method includes steps of sonication that do not introduce additional turbidity to the proppant sample itself but may mobilize fines previously immobile within the sample to become available for more accurate measurement. This process can result in a higher turbidity reading versus previous methods due to the enhanced ability to measure existing fines more accurately. This change aims to provide a more precise assessment of the sample's turbidity, reflecting the true extent of fine particles within the proppant.

9.3 Apparatus and Materials

The following is a list of equipment and materials for turbidity measurements:

9.3.1 Turbidimeter

9.3.2 Bath Sonicator

— The bath sonicator will have a frequency of 40 kHz.

9.3.3 Double or Wrist Action Flask Shaker

The preferred shaker will have performance characteristics similar to these:

— oscillations per minute: 385 ±10 %;

— angle of movement: 10 degrees ±1 degree.

9.3.4 Syringe

9.3.5 Erlenmeyer (Conical) Flask

250-ml, wide-mouth flask with screw cap.

9.3.6 Deionized (or Distilled) Water

For suspension of fine particles.

9.4 Apparatus Calibration

Apparatus calibration shall be performed in accordance with procedures designated by the original equipment manufacturer.

9.5 Procedure—Turbidity Measurement

9.5.1 Using the sample splitter, reduce the proppant sample size to 50 g ±20 g. Measure 20 ml ±1 ml of a dry proppant sample and add it, plus 100 ml ±5 ml of water, to the 250-ml Erlenmeyer flask having a wide mouth and a screw cap.

9.5.2 **For Natural Sands Only:** place Erlenmeyer flask in sonicator and sonicate for 1 minute ±5 seconds. Allow to stand for 30 minutes ±1 minute.

For Ceramic and Resin Coated Proppants: allow Erlenmeyer flask to stand for 30 minutes ±1 minute.

9.5.3 Set the frequency of the shaker to the maximum range. Place the flask in the shaker. Allow the shaker to shake for 30 seconds ±5 seconds. Remove the flask and let stand for 5 minutes ±1 minute.

9.5.4 Using a syringe, extract 25 ml of the water and solids suspension from near the center of the water volume. Care should be taken not to extract any proppant particles, as this distorts the results.

9.5.5 Place the suspended-particle sample in a test vial and place in a recently calibrated turbidimeter.

9.5.6 Record the sample turbidity in FTU (NTU) to the nearest unit.

9.6 Specification—Turbidity

The turbidity for ceramic and resin coated proppant shall not exceed 250 FTU (NTU).

The turbidity for natural sands shall not exceed 950 FTU (NTU).

10 Procedures for Determining Proppant Loose Pack Bulk Density, Apparent Density, and Absolute Density

10.1 Purpose

The purpose of this procedure is to determine the loose pack bulk density, ρ_{bulk}, apparent density, ρ_{ap}, and absolute density, ρ_{abs}, of proppant.

10.2 Description

The loose pack bulk density, apparent density, and absolute density are important properties of proppants. Loose pack bulk density describes the mass of proppant that fills a unit volume, and includes both proppant and porosity. It is used to determine the mass of a proppant needed to fill a fracture, an annulus for gravel packing, or storage tank. Apparent density is measured with a low-viscosity fluid that wets the particle surface and includes the pore space inaccessible to the fluid. The absolute density excludes pores that can be in the proppant, as well as void spaces between proppant.

10.3 Loose Pack Bulk density (ρ_{bulk})

This procedure is based on ANSI B74.4.

10.3.1 Apparatus and Materials

An apparatus consisting of the following parts (see Figure 6) is used to determine the loose pack bulk density. Nonessential dimensions of the apparatus can be adjusted at the convenience of the user.

10.3.1.1 Funnel Stand

The funnel stand consists of a square metal base, 305 mm × 305 mm (12 in. × 12 in.), with a tripod 305 mm (12.0 in.) high. The upper part of the tripod shall consist of a horizontal circular platform, 203 mm (8 in.) in diameter, forming a support for a funnel, which is replaceable and adjustable and fastened in place by screws.

10.3.1.2 Funnel

The funnel is stainless steel, having smooth inside seams and a shut-off at the outlet consisting of a 35 mm (1.378 in.) rubber ball attached to the funnel by two coil springs of such strength that a firm seal is made.

The funnel shall have the following dimensions:

— top diameter (inside): 114.3 mm ±0.4 mm (4.5 in. ±0.0156 in.);

— bottom diameter (inside): 12.7 mm ±0.4 mm (0.5 in. ±0.0156 in.);

— height of conical section: 76.2 mm ±0.4 mm (3.0 in. ±0.0156 in.).

— height of parallel section: 12.7 mm ±0.4 mm (0.5 in. ±0.0156 in.).

10.3.1.3 Brass Cylinder

The brass cylinder has a capacity of approximately 100 cm^3 (6.10 $in.^3$), calibrated with water as described in 10.3.2.

The cylinder shall be made of No. 17 gauge brass and seamless tubing, and shall have the following dimensions:

— inside diameter: 38.9 mm (1.53 in.) brass tube; inside diameter shall be within the tolerances normal to high-grade commercial tubing;

— height: 84.1 mm ±0.4 mm (3.3125 in. ±0.0156 in.);

— base thickness: 12.7 mm ±0.4 mm (0.5 in. ±0.0156 in.).

The bottom surface of the base shall be recessed to center the cylinder directly under the funnel discharge by means of a mating pin in the bottom plate.

10.3.1.4 Balance

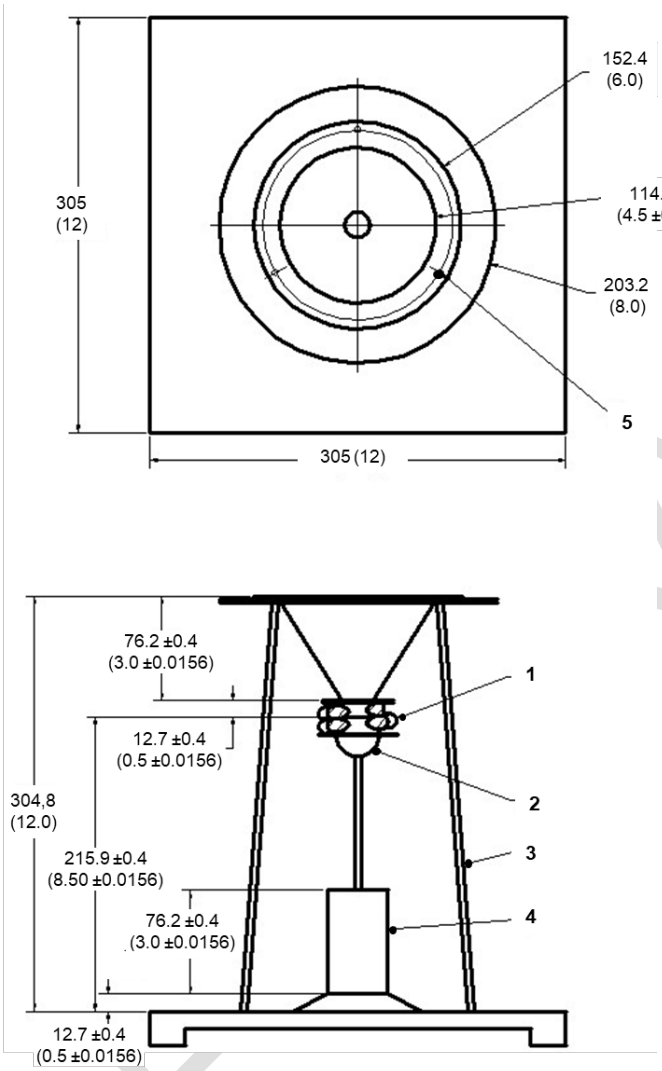
Minimum capacity of 100 g, with precision of 0.01 g or better.

10.3.1.5 Thermometer

Accurate at ±0.5 °C (±1 °F).

10.3.1.6 Deionized (or Distilled) Water

Dimensions in millimeters (inches)



Key

1 Spring diameter, 9.65 mm (0.38 in.)
2 Rubber ball stop, diameter 31.75 mm (1.25 in.)
3 Leg diameter, 5 mm (0.19 in.), welded to the base plate
4 Cylinder internal diameter, 38.1 mm (1.5 in.)
5 Three holes on a diameter of 139.7 mm (5.5 in.)

Figure 6—Loose Pack Bulk Density Device

10.3.2 Calibration of Cylinder Volume

Prior to use, determine the volume of the cylinder as follows:

10.3.2.1 Weigh the dry, empty cylinder with a flat glass plate (slicker plate) and record as m_1.

10.3.2.2 Fill the cylinder with water and allow the temperature to come to equilibrium. Use of a thermostat is advisable to control the time required for temperature equilibrium to be established. As with most laboratory volumetric ware, the temperature at calibration should be in the vicinity of 25 °C ±1 °C (77 °F ±2 °F).

10.3.2.3 Measure and record the temperature of the water in the cylinder.

10.3.2.4 Slide the glass plate into contact with the upper edge of the cylinder, cutting off the water precisely in the plane of the edge.

10.3.2.5 With the glass plate held firmly in place, wipe off the excess water and obtain the gross mass m_2.

10.3.2.6 Calculate the volume, V_{cyl}, expressed in cubic centimeters, of the cylinder as given in Equation (10):

$$V_{cyl} = \frac{m_2 - m_1}{\rho_w} \tag{10}$$

where

m_1 is the mass of the dry cylinder and glass plate, expressed in grams

m_2 is the mass of the cylinder, glass plate, and water, expressed in grams

ρ_w is the density of the calibration fluid, water at test temperature, expressed in grams per cubic centimeter.

The density of water at various temperatures is available in Table 7.

Table 7—Density of Water at Various Temperatures

Temperature °C	Density g/cm^3	Temperature °F	Density g/cm^3
15.0	0.9991	59	0.9991
15.5	0.9990	60	0.9990
16.0	0.9989	61	0.9989
16.5	0.9989	62	0.9988
17.0	0.9988	63	0.9987
17.5	0.9987	64	0.9986
18.0	0.9986	65	0.9985
18.5	0.9985	66	0.9984
19.0	0.9984	67	0.9983
19.5	0.9983	68	0.9982
20.0	0.9982	69	0.9981
20.5	0.9981	70	0.9980
21.0	0.9980	71	0.9978
21.5	0.9979	72	0.9977
22.0	0.9978	73	0.9976
22.5	0.9977	74	0.9975
23.0	0.9975	75	0.9973
23.5	0.9974	76	0.9972
24.0	0.9973	77	0.9970
24.5	0.9972	78	0.9969
25.0	0.9970	79	0.9968
25.5	0.9969	80	0.9966
26.0	0.9968	81	0.9965
26.5	0.9966	82	0.9963
27.0	0.9965	83	0.9961
27.5	0.9964	84	0.9960
28.0	0.9962	85	0.9958
28.5	0.9961	86	0.9956
29.0	0.9959	87	0.9955
29.5	0.9958	88	0.9953
30.0	0.9956		

NOTE Water density data referenced from *Handbook of Chemistry and Physics, 66th Edition*, CRC Press Inc., Boca Raton, Florida (1985–1986)

10.3.3 Procedure—Loose Pack Bulk Density Determination

10.3.3.1 Weigh the dry, empty cylinder and record the mass as m_1.

10.3.3.2 The sample of proppant to be tested shall have a temperature of not less than 18 °C (65 °F) and not more than 29 °C (85 °F). Fill a 150-ml beaker with the split sample of proppant (see 5.2).

10.3.3.3 With the funnel outlet closed and the cylinder centered under the outlet of the funnel, pour the sample from the beaker into the funnel.

10.3.3.4 Move the ball to the right or left of the opening at the bottom of the funnel and allow the proppant to fall freely, filling the cylinder.

10.3.3.5 Immediately after the funnel is emptied, smoothly pass a straight edge once across in contact with the edge of the cylinder to level the surface of the proppant. It is important to avoid vibration and shock or any disturbing factor.

10.3.3.6 Weigh the cylinder and the contained proppant, and record as m_2.

10.3.3.7 Calculate the loose pack bulk density, ρ_{bulk}, expressed in grams per cubic centimeter, as given in Equation (11):

$$\rho_{bulk} = \frac{m_2 - m_1}{V_{cyl}} \tag{11}$$

where

m_1 is the mass of the dry cylinder, expressed in grams

m_2 is the mass of the cylinder filled with proppant from 0, expressed in grams

V_{cyl} is the volume of the cylinder, expressed in cubic centimeters

To express ρ_{bulk} in lb/ft^3, multiply ρ_{bulk} expressed in g/cm^3 by 62.428.

10.4 Apparent Density (ρ_{ap})

10.4.1 Apparatus and Materials

The following equipment and materials are needed to determine apparent density, ρ_{ap}, of proppants in oil.

10.4.1.1 Le Chatelier Flask

As described in ASTM C188; clean.

10.4.1.2 Displacement Liquid

Low-viscosity, paraffinic oil, kerosene, or similar oil with a maximum viscosity of less than or equal to 5 centipoises at temperature of use.

10.4.1.3 Balance

Capacity 80 g to 100 g, with precision of 0.01 g or better.

10.4.1.4 Weighing Boat

10.4.1.5 Funnel

With stem to fit inside the flask.

Stem length should be long enough [63.5 mm (2½ in.)] that displacement liquid can be poured into the flask without touching the sides of the upper half of the neck of the flask.

10.4.1.6 Proppant

Approximate quantity of proppant required by the procedure:

Natural sands: 48 g to 54 g

Resin-coated sands: 63 g to 68 g

High-density ceramics: 63 g to 68 g

Lightweight ceramics: 42 g to 48 g

Resin-coated ceramics: See 10.4.2.4

Neutral buoyancy: may require a different fluid density

10.4.2 Procedure—Apparent Density Determination

10.4.2.1 Fill the flask with test liquid to a point on the neck between the 0 and 1-ml mark without the fluid wetting the wall of the flask neck. If the neck of the flask above the lower bulb is wetted, the proppant may bridge when introduced.

10.4.2.2 Read and record the initial volume of liquid, V_{init}, to the nearest 0.05 ml.

10.4.2.3 Weigh the proppant and record the mass as m_{p} to the nearest 0.01 g.

10.4.2.4 Carefully add all of the weighed proppant to the flask. The final liquid level in the flask with all the proppant in place must be within the range of the upper graduated markings.

10.4.2.5 After all of the proppant has been placed into the flask, put the stopper into the flask.

10.4.2.6 Gently tilt and roll the flask until all the air bubbles are removed and there is no proppant in the neck of the flask.

10.4.2.7 Read the final volume of liquid and record as V_{f} to the nearest 0.05 ml.

10.4.2.8 Calculate the apparent density, ρ_{ap}, expressed in grams per milliliters, in Equation (12):

$$\rho_{\text{ap}} = \frac{m_{\text{p}}}{V_{\text{f}} - V_{\text{init}}} \tag{12}$$

where

m_{p} is the mass of dry proppant, expressed in grams

V_{f} is the final volume of liquid, expressed in milliliters

V_{init} is the initial volume of liquid, expressed in milliliters

10.4.2.9 Report the apparent density, ρ_{ap}, expressed in grams per milliliter.

NOTE One milliliter is considered equivalent to one cubic centimeter.

To express ρ_{ap} in lb/ft^3, multiply the value in g/ml by 62.428.

10.5 Absolute Density (ρ_{abs})

10.5.1 Description

The preferred method to determine absolute density, ρ_{abs}, is based on Boyle's Law: The pressure upon filling the sample chamber with a gas and then discharging the gas into a second empty chamber allows the computation of the sample solid phase volume. Since the density and volume of gas are very low compared to the proppant, the result is very close to the true density of the proppant, excluding the connected internal porosity common in some proppant types.

10.5.2 Apparatus

10.5.2.1 Pycnometer

A gas-comparison pycnometer with a typical reproducibility within 0.02 % of the nominal full-scale volume on clean, dry, thermally-equilibrated sample. The preferred gas is helium, He_2, but dry nitrogen, N_2, is an acceptable alternative.

10.5.2.2 Balance

0.0001 g accuracy.

10.5.2.3 Drying Oven

Capable of holding 105 °C (220 °F).

10.5.2.4 Desiccator

With standard drying agent (anhydrous calcium sulfate or silica gel).

10.5.3 Procedure—Absolute Density Determination

10.5.3.1 The volume of proppant is dependent upon the volume of the sample cup. The proppant should be dried to constant mass at 105 °C (220 °F), then cooled to room temperature in a desiccator. Record the sample mass, m_p, to the nearest 0.1 mg.

10.5.3.2 Check the gas-comparison pycnometer for zero measurement and calibration as specified in the instruction manual for the specific pycnometer being used**.**

10.5.3.3 Place the sample cup with proppant in the pycnometer sample compartment and lock firmly in place. Purge the sample compartment with gas at pressures not exceeding 15 kPa (2 psi).

10.5.3.4 Measure the sample volume by the standard manufacturer's procedures. Ten gas purges of the sample and five measurements shall be observed and recorded to the accuracy of the instrument.

10.5.3.5 Record the five measurements as the volume of the proppant sample, V_i, in cubic centimeters. Calculate the average volume, V_{avg}, of the proppant.

10.5.3.6 The absolute density, ρ_{abs}, expressed in grams per cubic centimeters, is calculated by using Equation (13):

$$\rho_{abs} = \frac{m_p}{V_{avg}} \tag{13}$$

where

m_p is the mass of the proppant sample, expressed in grams

V_{avg} is the average of the five volume measurements, expressed in cubic centimeters

10.5.3.7 Report the absolute density, ρ_{abs}, in grams per cubic centimeter to two decimal places.

To express ρ_{abs} in lb/ft^3, multiply the value in g/cm^3 by 62.428.

11 Proppant Crush Resistance

11.1 Purpose

Crush resistance tests are conducted on samples to determine the amount of proppant crushed at a given stress.

11.2 Description

This test is useful for determining and comparing the crush resistance of proppants. Tests are conducted on samples that have been sieved so that all particles tested are within a specified size range, which is usually below the coarse primary screen and on or above the fine primary screen. The amount of proppant material crushed at each stress level is measured. Even with finer and wider mesh distributions of natural sand proppant, the same method steps can determine the crush resistance as a function of stress. Evaluation of test results from the current method provides indications of the stress level where proppant crushing is more than 10 %.

Since the previous update to Section 11 (Proppant Crush Resistance), the scope of proppant distribution has broadened significantly. This expansion has led to increased variability in specifications of proppants available in the market. Consequently, the 2018 revision, which was initially designed under a narrower scope, now results in comparisons that are not truly representative of the proppants' performance under similar conditions.

The revised method addresses this issue by focusing on the distribution of the proppant within the sand. By considering this distribution, the new approach allows for a more accurate and equitable comparison between proppants, ensuring that the test results better reflect the actual performance of the proppant under stress. This refinement provides clearer guidance and more reliable data for both suppliers and end-users, ultimately enhancing the standard's relevance in today's diverse proppant landscape.

11.3 Apparatus

11.3.1 Hydraulic Load Frame

With the capacity to apply the load required for accomplishing the stress levels up to 103 MPa (15,000 psi).

The load frame shall have platens that can be maintained parallel during application of the load to the cell. The load frame shall be calibrated at least annually and after all major repairs, to ensure that stress measurements are accurate to within 5 % (±2.5 %). Alternatively, an independent load measuring device calibrated at least annually and after all major repairs may be used when the load is applied to the proppant in the test cell. The stress shall be within 5 % (±2.5 %) of target (see Table 11). Automated load frames are highly recommended.

11.3.2 Crush Cell

Refer to Figure 7 or equivalent.

The crush cell is composed of three parts: cell body, piston, and cell plate. All parts shall be machined from alloy steel and hardened to a minimum of 43 Rockwell C; 60 Rockwell C is preferred for reduced wear of

the crush cell and longer service life. The piston length shall be 88.9 mm (3.5 in.) and the piston diameter shall be 50.67 mm (1.995 in.). The cell body length is 76.2 mm (3 in.). The internal diameter is 50.8 mm (2.000 in.). Periodic inspection for wear to the cell internal diameter indicates when the cell should be replaced. When the internal diameter of the lower portion of the cell exceeds the designed diameter by more than 3.25 % [e.g. 52.45 mm (2.065 in.)]—about a 10 % increase in cross-sectional area—the cell shall be replaced.

11.3.3 Test Sieves

Appropriate sieve numbers (sizes), pan and lid (see Table 1).

11.3.4 Test Sieve Shaker

See 6.3.2.

11.3.5 Timer

Accurate to ±5 seconds over a period of 30 minutes.

11.3.6 Balance

Minimum capacity of 100 g, with precision of 0.01 g or better.

11.3.7 Sample Splitter

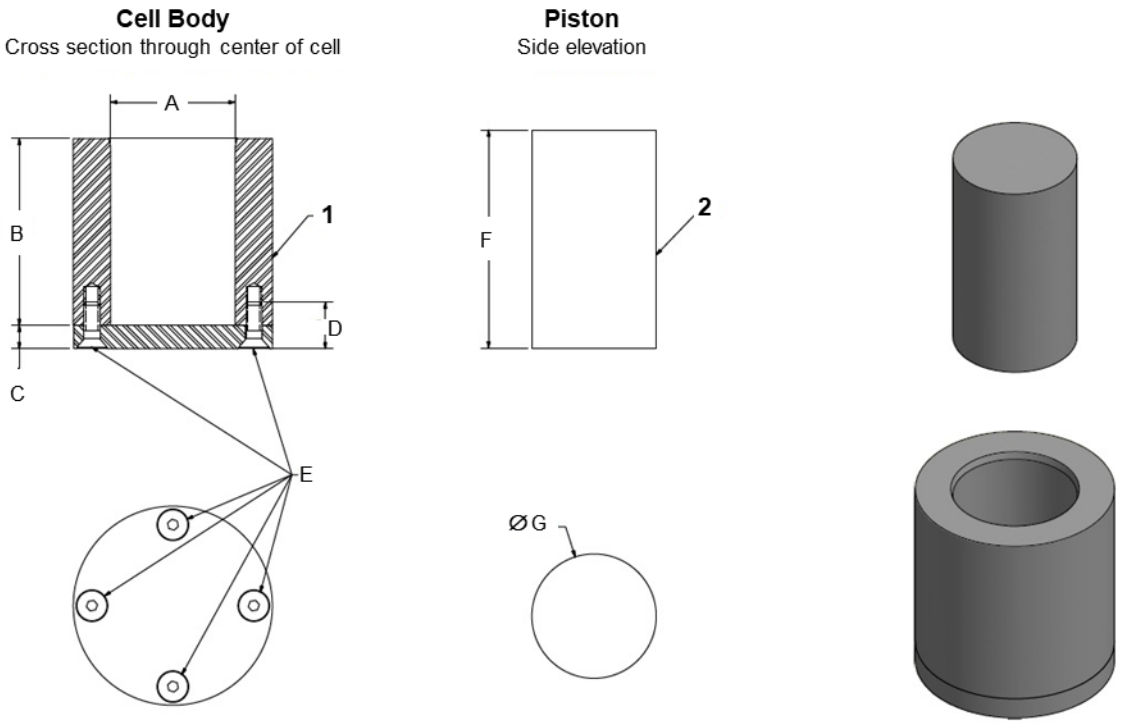
Metal; see 4.3.3.

11.3.8 Metal Beaker or Weighing Boat

Plastic, glass, or paper containers tend to generate static electrical charge and shall not be used.

11.3.9 Pluviator Crush Cell Loading Device

With parts and accessories listed in Table 8; also refer to Figure 8, Figure 9, Figure 10, and Table 9. Material: stainless steel.



Key

All dimensions in millimeters (inches). Tolerances ±0.13 (0.005) unless otherwise noted.

1 Cell body; minimum wall thickness 12.70 (0.50):

- **A** Bore diameter: 50.80 ±0.03 (2.000 ±0.001).
- **B** Height: 76.20 (3.00).
- **C** Cell plate: Thickness 0.953 (0.375); 4-off holes 90° apart to accept M6 machine screws 10 x 25.4 (0.25-20 x 1.00), counter sunk (screw heads shall not protrude past level of bottom plate).
- **D** Four off-threaded holes 90° apart to accept M6 machine screws 10 x 25.4 (0.25-20 x 1.00); depth 15.88 (0.625).

2 Piston:

- **F** Height: 88.90 (3.50).
- **G** Diameter: 50.67 ±0.03 (1.995 ±0.001).

NOTE 1 Material: Steel hardened to 60 Rockwell hardness (minimum 43 Rockwell hardness).

NOTE 2 Assembled cell body and cell plate shall sit level and square on the work surface.

Figure 7—Design of the Crush Cell

Table 8—Pluviator Cell Loading Device, Parts, and Accessories

Part Number [a]	Description
1	**Cap Screw,** socket head, stainless steel #10.
2	**Lock Washer,** standard, stainless steel #10.
3A	**Screen Carrier Assembly**[b], ASTM E11, Standard Sieve Number 5, 6, 8, 14, 18, 20, and 30.
3B	**Screen Carrier Assembly**[c], ASTM E11, Standard Sieve Number 6, 8, 20, 30 and blank.
A	**Tube Assembly,** lower.
B	**Tube Assembly,** upper.
C	**Taper Sleeve**.
D	**Sand Gate,** female, lower.
E	**Sand Gate,** male, upper.

[a] Parts are marked for identification.

[b] All pieces marked for use in the flange at the top of the lower Tube A.

[c] All pieces marked for use in the flange at the top of the upper Tube B.

Dimensions in millimeters (inches)

Material: Stainless steel

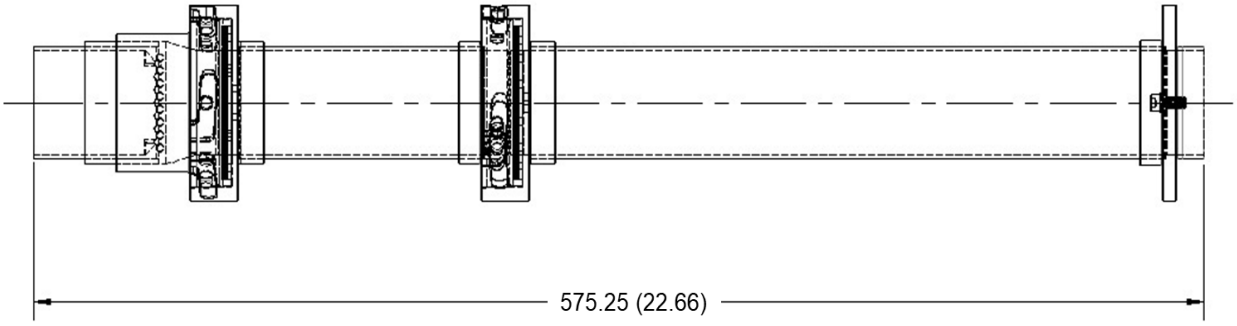
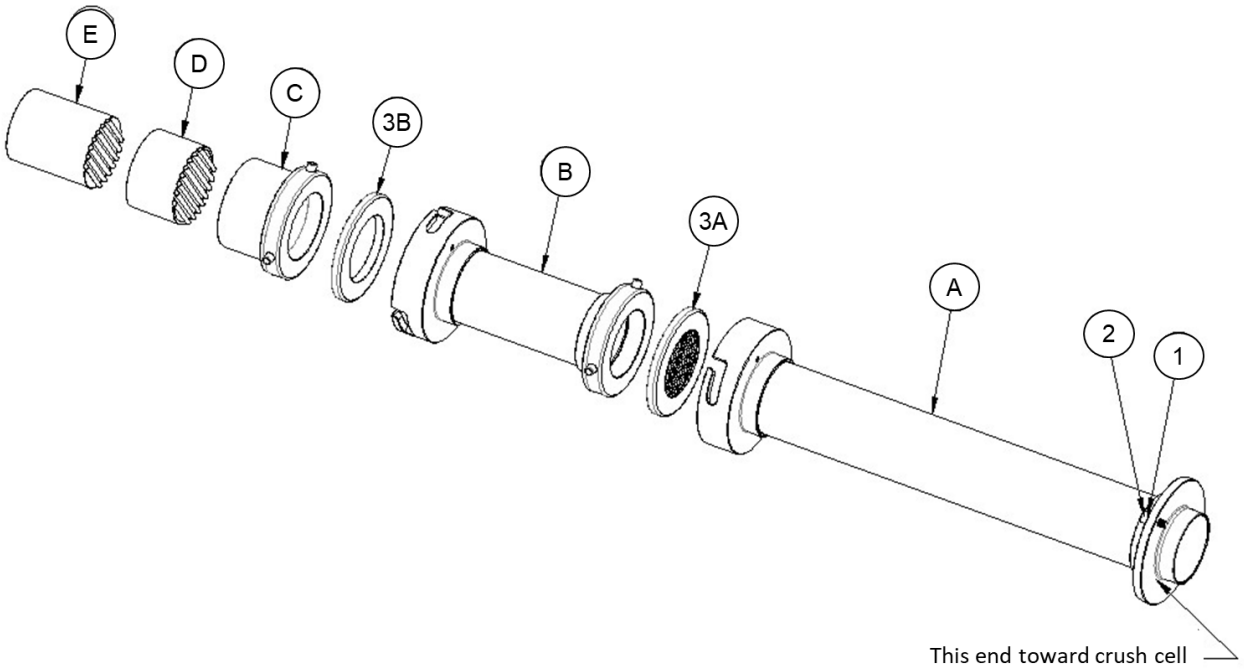


Figure 8—Assembled View of Pluviator Device



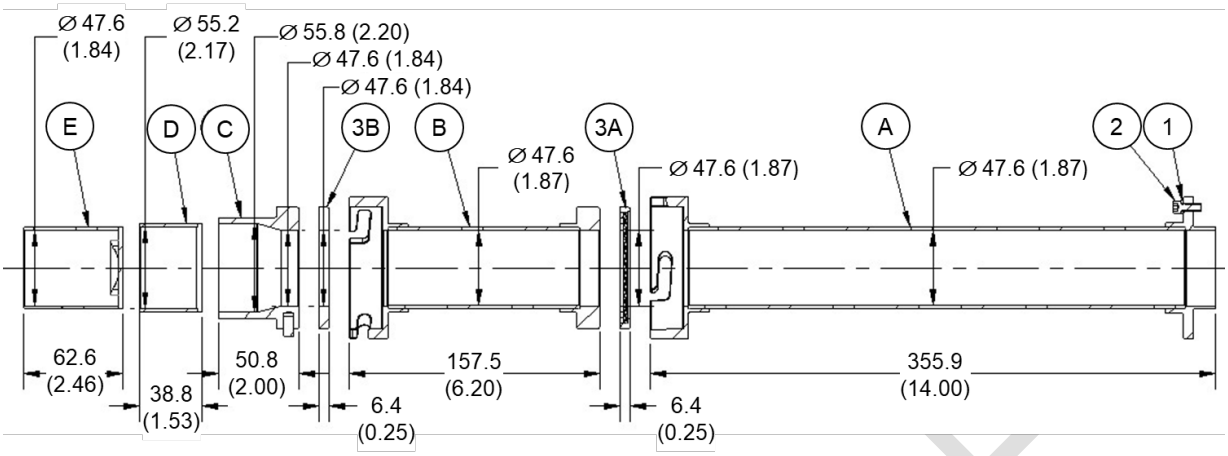
Key

A	Tube assembly (lower)	**3A**	Screen carrier assembly
B	Tube assembly (upper)	**3B**	Screen carrier assembly
C	Taper sleeve	**1**	Cap screw
D	Sand gate (lower)	**2**	Lower washer
E	Sand gate (upper)		

NOTE See Tables 8 and 9 for identification of parts and accessories.

Figure 9—Exploded View of Pluviator Device

Dimensions in millimeters (inches)



Key

A	Tube assembly (lower)	**3A**	Screen carrier assembly
B	Tube assembly (upper)	**3B**	Screen carrier assembly
C	Taper sleeve	**1**	Cap screw
D	Sand gate (lower)	**2**	Lower washer
E	Sand gate (upper)		

NOTE See Tables 8 and 9 for identification of parts and accessories.

Figure 10—Dimensional View of the Pluviator Device

11.4 Sample Preparation

11.4.1 Using the sample splitter, reduce the sample size to 70 g ±10 g. Too large a quantity of proppant can block or blind the sieve openings and skew the results.

11.4.2 Refer to Table 1 and the steps following to select three testing sieves from the list appropriate for the proppant being tested. The sieves to select include a) the upper designating sieve size in the product sample as measure by the d1 (selected in the following step); b) the sieve size that approximates (choosing the next sieve size smaller than) the d90 (selected in the following step; and c) the middle sieve from the list. Prepare the sieve stack in the normal order by placing the sieves on the pan in ascending opening size order – smallest openings on the bottom and largest openings on the top. The purpose of the intermediate sieve is to minimize the effects of sieve blinding, which occurs when a large amount of proppant, greater than 35 g, is resting on a single sieve.

- Identification of d1 (representing 1% cumulative mass retained) is used to determine the upper bound sieve (with largest opening size) for that method. That sieve will be selected as the ASTM E11 sieve above (larger opening than) the d1 value.
    - In the example shown in Table 2, 12-mesh sieve would be selected as the upper bound sieve size.
- Identification of d90 (representing 90% cumulative mass retained) is to determine the lower bound sieve (with smallest opening size) for that method. That sieve will be selected as the ASTM E11 sieve below (smaller opening than) the d90 value.
    - In the example shown in Table 2, 25-mesh sieve would be selected as that lower bound sieve size.

11.4.3 Pour the reduced sample into the top sieve, and cover with the lid.

11.4.4 Place the sieve stack in the sieve shaker, properly secure the stack, and shake for at least 15 minutes. Note this is a longer shake time than required for the sieve analysis described in Section 6**,** and is designed to assure that all the pan material is removed from the proppant sample prior to crush testing.

11.4.5 Remove the sieve stack from the shaker and discard all material retained on the upper sieve and in the pan. Only the material retained on the intermediate sieve(s) and the lower designating sieve is used in the crush test.

11.4.6 Determine the exact mass of the proppant to be used in the crush resistance test. The objective of the following procedure is to determine the amount of proppant, of any loose bulk density, required to provide a proppant pack concentration in the crush cell of approximately 1.953 g/cm^2 (4 lb/ft^2) of the piston area. If the loose bulk density of the proppant being tested is known, skip to Equation (14) and determine the mass required; otherwise, proceed as follows:

11.4.6.1 Determine the loose pack bulk density of the proppant sample using the procedure in 10.3.

11.4.6.2 The cross-sectional area of the crush cell is (π/4) x (5.08 cm)^2 = 20.27 cm^2. The proppant mass needed to achieve the targeted proppant pack concentration is 1.953 g/cm^2 x 20.27 cm^2 = 39.59 g. Using a loose pack bulk density constant of 1.60 g/cm^3, the volume of sand required per unit of piston area of the test cell is 39.59 g / 1.6 g/cm^3 = 24.7 cm^3.

Other proppants with different bulk densities require different masses. The mass, m_p, expressed in grams, of proppant material needed for each test, to the nearest 0.01 g, is calculated according to Equation (14):

$$m_p = 24.7 \times \rho_{bulk} \quad (14)$$

where

24.7 is the volume of proppant required for a single crush test, expressed in cubic centimeters

ρ_{bulk} is the proppant bulk density, expressed in grams per cubic centimeter

11.4.6.3 Using the sample splitter, reduce the sieved sample to the calculated m_p plus no more than 5 g.

11.4.6.4 From the material prepared in step 11.4.6.3, weigh the sample mass determined in 11.4.6.2, m_p to the nearest 0.01 g, and retain for crush testing.

11.5 Assemble and Set Up the Crush Cell and Pluviator Loading Device

11.5.1 Using a bubble level, ensure the surface on which the crush cell is to be placed is level. The crush cell-pluviator column shall be at a right angle to the horizontal/flat/level surface on which it is resting. A crush cell leveling platform is commercially available.

11.5.2 Assemble the pluviator loading device and the crush cell. Check to be sure the pluviator and crush cell is actually level in all directions, side-to-side and front-to-back. Adjust as necessary.

11.5.3 The pluviator is designed to fit the most common crush cell bore dimensions of slightly larger than 50.8 mm (2 in.). Be sure the upper internal bore of the cell is clean and free of any rust or other foreign material. If it is not clean, brush with a wire brush or other suitable device. Carefully fit the cell insertion fitting on the bottom of the lower tube, A (see Figures 8, 9, and 10), into the existing crush cell. Do not misalign the fitting or use extra force. The fitting should slide easily into the cell and, when fully inserted, it should freely rotate inside the cell. If the fit is very tight and the pluviator device will not rotate easily, be sure the cell bore is clean. Do not attempt to modify the crush cell or the pluviator device; send them back to the manufacturer for adjustment as needed.

11.5.4 The pluviator device shall sit vertically on top of a properly leveled crush cell.

11.5.5 Always use the grounding lug on the lower flange. Attach a length of 12–14 gauge stranded copper wire, using crimp or solder terminals, between the pluviator device and a known electrical ground. Since the pluviator's proppant gate may leak very fine proppants, always have the crush cell loading device fully assembled on the crush cell before adding the proppant sample.

11.5.6 All work with the pluviator device should be done on a work surface that is low enough for easy observation of the inside of the top valve when the pluviator is assembled, mated with the crush cell, and ready for loading.

11.5.7 Remove all proppant grains from the mating surfaces of the pluviator device before assembly. Determine the specific screen sizes from Table 9, using the top sieve appropriate to the largest grain size in the sample.

Table 9—Wire Cloth Sizes Used in the Pluviator Device

Proppant Size	Captured Wire Cloth Screens [a]		Gate Bar Diameter mm (in.)
	Upper [b] Top of Tube B	Lower [c] Top of Tube A	
70/140 (and custom fine ranges)	30	30	3.175 (0.125)
40/70 and 40/80	18	20	
30/50 and 30/70	14	18	
20/40	8	8	
16/30 and 16/20	6	8	
16/20 and 12/18	blank	8	
8/16	blank	5	4.763 (0.1875) [d]

[a] **ASTM** E11 standard sieves numbers.

[b] **Part** No. 3B.

[c] **Part** No. 3A.

[d] **Not** supplied with the current commercial pluviator device.

NOTE Wire cloth screens are marked “top” and “bottom,” which is the intended orientation of the screen in the mounting flange at the top of the lower (A) and upper (B) tubes. All of the framed screens are labeled as A or B to assist in getting the recommended sieves in the correct positions. The correct position of the sieves in the pluviator device are indicated in the table by the column headers, with “Upper” referring to the upper flange on the top of the upper (B, short) tube.

11.5.8 Assemble the lower screen into the flange at the top of tube A.

11.5.9 Install the upper tube B, ensuring the proper locking of the pins of the bayonet flange.

11.5.10 Install the upper captured screen or the blank frame as required into the flange at the top of tube B. Make sure the word “TOP” is visible.

11.5.11 Install the upper portion of the upper flange, tapered sleeve C, into the bayonet connector and lock the pins into proper position.

11.5.12 Test the column to assure the device is stable. If not vertically stable, replace the support base with a more suitable object.

11.5.13 Assemble the proppant gate, D and E, and twist to make sure it is in a closed position. Install the valve by inserting it into part C.

11.6 Procedure—Crush Resistance Testing

11.6.1 Check the proppant gate to make sure it is properly installed and in the closed position. Pour the premeasured proppant sample, from 0, into the top of the proppant gate and level proppant.

11.6.2 Transfer the proppant sample into the crush cell by lifting straight up on the part E of the proppant gate.

11.6.3 Remove part E and inspect to make sure the entire sample has completely exited the valve and entered the pluviator. Slowly remove part E without jarring the pluviator assembly. Tilt part E and brush it, using an appropriately sized brush, to remove any adhering proppant grains. Do not tap part E.

11.6.4 Remove part D and inspect it to be sure all adhering grains of proppant have entered the pluviator device. Brush any adhering grains into the top of the column. Inspect the upper screen and brush any adhering grains through the screen. If a majority of the proppant has failed to pass through the screen, select an alternative screen, and begin again at step 11.6.1. Alternative screens may include sizes not shown in Table 9.

11.6.5 Continue to disassemble the pluviator in reverse order to the assembly instructions to ensure the entire sample has been transferred to the crush cell. Inspect inside sections A and B of the pluviator to ensure all the proppant sample has completely exited the pluviator and entered the crush cell. If any of the sieved and weighed sample has spilled outside of the crush cell, begin again at step 11.6.1.

11.6.6 Remove the pluviator from the crush cell and lay it down on the bench top. Visually ensure the proppant is sufficiently level (absent of visually detectable high and/or low spots) within the crush cell. If proppant is not sufficiently level, begin again at step 11.6.1.

11.6.7 Insert the piston into the crush cell and let the piston settle onto the proppant sample in the cell. Do not apply any additional force other than gravity. Once the cell is loaded, care should be taken to avoid agitation (tapping, jarring, shaking), as these activities will change the packing in the cell, and variance in crush results is largely associated with the method of loading the crush cell.

11.6.8 Carefully lift the test cell and place it directly into the press, centering it under the upper platen. In order to maintain a loose pack bulk density, do not shake or jar the cell, as this tends to settle the proppant pack and change the particle packing during the stress application.

11.6.9 Crush stress-level guidelines are given in Table 10.

Table 10—Guidelines for Testing Various Proppant Types

Proppant Type	**Crush Stress Minimum** MPa (psi)
	Minimum
Hydraulic Fracturing	
Man-made proppants	34.5 (5000)
Natural sand proppants	13.8 (2000)
Gravel-packing	
Natural sand proppants	13.8 (2000)

11.6.10 Other stress levels may be used, by specific agreement between user and supplier, to more clearly and specifically define proppant crush behavior. Determine the force, F_{tc}, expressed in newtons (pounds-force) required on the cell to attain the prescribed stress using Equation (15):

$$F_{tc} = \frac{\pi \times \sigma \times d_{cell}^2}{4} \tag{15}$$

where

σ is the stress on the proppant sample, expressed in megapascals (pound-force per square inch)

d_{cell} is the test cell inside diameter, expressed in millimeters (inches)

With the test crush cell (see 11.3.2) with an inside diameter of 50.80 mm (2.0 in.), the force F_{tc}, required is given per Equation (16) when expressed in newtons.

$$F_{tc}=2026.8 \times \sigma \quad (16)$$

Or per Equation (17) when F_{tc}, is expressed in pounds-force.

$$F_{tc}=3.14159 \times \sigma \quad (17)$$

Where σ is the stress on the proppant sample, expressed in megapascals Equation (16) or in lbf/in². [Equation (17)].

11.6.11 Apply the appropriate stress (see 11.6.10) to the test cell piston at a constant rate of 13.8 MPa (2000 psi) per minute until the final pressure is reached. Arrival time at the final stress should be within 5 % of the ramp time for that stress. If the targeted stress deviates by 5 % (±2.5 %), abort the test and start over with a new sample. Automated load frames are highly recommended.

11.6.12 Hold the stress constant for 120 ±5 seconds.

11.6.13 Release the stress, and remove the test cell from the press.

11.6.14 Carefully transfer the content of the cell into the same sieve stack used in 11.4. Scrape the bottom of the test cell to ensure the sample is completely removed.

11.6.15 Place the sieve stack into the shaker and shake for at least 15 minutes.

11.6.16 Carefully weigh all sieve fractions and the pan, m_{pan}, recording the weight of each fraction to the nearest 0.01 g. The total weight recovered, m_s, is calculated by adding all the fractions weighed. If the amount of material retained on the top sieve is greater than 0.1 g, sieve blinding is indicated and the sample and data shall be discarded. Begin again with a new sample at 11.4 and extend the shake time to ensure removal of all oversized material as necessary. Compare the total amount recovered with the initial weight of the crush sample and discard the results if the difference is more than ±0.5%.

11.6.17 Using Equation (18), calculate and report the amount of crushed material, m'_{pan}, as a percentage of the mass of proppant sample recovered from the cell. Each proppant sample tested shall be run in triplicate, at the same stress, and the results averaged. If the absolute variance between any two of the replicates is greater than 0.5 %, repeat the replicates.

$$m'_{pan} = 100 \times \frac{m_{pan}}{m_s} \quad (18)$$

where

m_{pan} is the mass of fines generated in the test, expressed in grams

m_s is the mass of proppant recovered from the cell, expressed in grams

m'_{pan} is the mass of fines generated in the crush test, expressed in percent

11.7 Specification—Crush Resistance of Fracturing Proppants

Determine the highest stress level at which the proppant generates no more than 10.0 % crushed material, and round this value down to the nearest 6.9 MPa (1000 psi). This value represents the maximum stress that the material can withstand without exceeding 10.0 % crush.

Using Table 11, establish the 10 % crush classification of the proppant, referred to as the K-Value, by finding the classification that corresponds to the maximum stress. This apparent K-Value is reported specific to the d90 value of the sand, rather than to the smaller sieve size in the product label. For example, if a proppant generates 10 % crush at 33.27 MPa (4926 psi), then, after rounding down, the maximum stress that the proppant can withstand without exceeding 10 % would be 27.6 MPa (4000 psi). Using Table 11, the 10 % crush classification of this proppant shall be reported as 4K.

Table 11—10 % Crush Classification Table for Fracturing Proppants

10% Crush Classification	Closure Stress	
	MPa	psi
1K	6.9	1000
2K	13.8	2000
3K	20.7	3000
4K	27.6	4000
5K	34.5	5000
6K	41.4	6000
7K	48.3	7000
8K	55.2	8000
9K	62.1	9000
10K	68.9	10,000
11K	75.8	11,000
12K	82.7	12,000
13K	89.6	13,000
14K	96.5	14,000
15K	103.4	15,000
16K	110.3	16,000
17K	117.2	17,000
18K	124.1	18,000
19K	131.0	19,000
20K	137.9	20,000

11.8 Specification—Crush Resistance of Gravel-packing Proppants

A sample of gravel-packing proppant subjected to this test shall not produce average fines in excess of the fines specified in Table 12, at a stress of 13.8 MPa (2000 psi).

Table 12—Maximum Fines for Gravel-packing Proppant

Gravel-packing Proppant Size	Maximum Crushed Material at 13.8 MPa (2000 psi) % by weight
8/16	8.0
12/20	4.0
16/30	2.0
20/40	2.0
30/50	2.0
40/60	2.0

12 Loss on Ignition (LOI) of Whole Grain Proppant

12.1 Purpose

This test is used for the determination of amount of organic material on proppant.

12.2 Apparatus and Materials

The following equipment and materials are required for the LOI test:

12.2.1 Muffle Furnace

Conventional or microwave, capable of at least 927 °C (1700 °F).

12.2.2 Ashing Crucibles

Ceramic or quartz fiber, with lids.

12.2.3 Desiccator

With standard drying agent (anhydrous calcium sulfate or silica gel).

12.2.4 Balance

Analytical, 0.0001 g accuracy.

12.2.5 Sample Splitter

See Figure 3.

12.2.6 Crucible Tongs or Spatula

Long.

12.3 Procedure

12.3.1 Pre-condition a series of crucibles with lids in a preheated furnace at 927 °C (1700 °F) for about 15 minutes. Place the preconditioned crucibles with lids in a desiccator containing standard desiccants and allow to cool to room temperature. Preheating of the furnace to a minimum of 927 °C (1700 °F) is an absolute necessity.

12.3.2 Weigh on an analytical balance, a conditioned crucible with a lid. Record this tare mass, m_t, in grams.

12.3.3 Dry the proppant sample to a constant weight. Use the sample splitter to obtain a representative 6 g to 10 g sample and place it in the crucible.

12.3.4 Weigh the sample plus the crucible and lid on the balance. Record the mass, m_1, in grams.

12.3.5 Place the covered crucible and sample in the muffle furnace at 927 °C (1700 °F) using the long crucible tongs or spatula.

12.3.6 Allow the furnace to heat back up to 927 °C (1700 °F). This may take some time depending on the type and condition of the furnace.

12.3.7 Keep the samples in the furnace for 2 hours (15 minutes when using a microwave furnace) after the furnace temperature has returned to 927 °C (1700 °F).

12.3.8 Transfer the crucible with the lid and sample to the desiccator and cool to room temperature. Inspect the content of the fired crucible. If any black or dark-colored residue remains in the crucible, firing may not be complete. Put the crucible and lid with the sample back in the furnace for at least 30 minutes or until the black residue has been burned to off-white or gray ash.

12.3.9 Re-weigh the crucible with the lid containing the heat-treated proppant, m_f, using the balance. Record the mass, in grams.

12.3.10 Run triplicate samples of each proppant.

12.3.11 Calculate the LOI for each sample. If the values differ by more than 0.2 %, repeat the analysis. After the proppant is carefully removed from the ceramic crucibles, they are stored in a desiccator.

12.3.12 Calculate the LOI, expressed in percent using Equation (19):

$$LOI = 100 \times \frac{m_1 - m_f}{m_1 - m_t} \tag{19}$$

where

m_1 is the initial mass of the sample plus the crucible and lid, expressed in grams

m_t is the original mass of the crucible and lid, expressed in grams

m_f is the final weight of the heat-treated sample, crucible, and lid, expressed in grams

12.3.13 Example LOI calculation:

8.8242 g m_1, original mass of the sample with the crucible and lid

–0.8039 g less, m_t, tare or mass of the original crucible and lid

8.0203 g m, original sample mass

8.8242 g m_1, original mass of the sample with the crucible and lid

–8.5742 g less, m_f, mass of the heat-treated sample with crucible and lid

0.2500 g mass lost

LOI = 100 x ($m_1 - m_f$) / ($m_1 - m_t$) = 100 x 0.2500 / 8.0203 = 3.12 %

Repeat these calculations for the remaining two replicates. If any two replicates differ by more than 0.2 %, repeat the analysis.

Annex A

(informative)

ASTM E11 Sieve Table

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Sieve Designation			Nominal Sieve Opening	±Y Variation for Average Opening	+X Maximum Variation for Opening	Resulting Maximum Individual Opening	Compliance Sieves		Inspection Sieves		Calibration Sieves		Typical Wire Diameter	Permissible Average Wire Diameter	
Standard	U.S. Alternative	Supplementary Size					Sample Openings per 100 ft²	Maximum Standard Deviation	Sample Openings per Sieve	Maximum Standard Deviation	Sample Openings per Sieve	Maximum Standard Deviation		Min	Max
mm		mm	in.	mm	mm	mm							mm		
125	5 in.		5.00	3.30	4.06	129.06	20	–	all	–	all	–	8.00	6.8	9.2
		112	4.41	2.96	3.74	115.74	20	–	all	–	all	–	8.00	6.8	9.2
106	4.24 in.		4.24	2.80	3.59	109.59	20	–	all	–	all	–	6.30	5.4	7.2
100	4 in.		4.00	2.65	3.44	103.44	20	–	all	–	all	–	6.30	5.4	7.2
90	3½ in.		3.50	2.39	3.18	93.18	20	–	all	–	all	–	6.30	5.4	7.2
		80	3.15	2.13	2.91	82.91	20	–	all	–	all	–	6.30	5.4	7.2
75	3 in.		3.00	2.00	2.78	77.78	20	–	all	–	all	–	6.30	5.4	7.2
		71	2.80	1.89	2.67	73.67	20	–	all	–	all	–	5.60	4.8	6.4
63	2½ in.		2.50	1.69	2.44	65.44	20	–	all	–	all	–	5.60	4.8	6.4
		56	2.20	1.50	2.24	58.24	20	–	all	–	all	–	5.00	4.3	5.8
53	2.12 in.		2.12	1.42	2.15	55.15	20	–	all	–	all	–	5.00	4.3	5.8
50	2 in.		2.00	1.34	2.06	52.06	20	–	all	–	all	–	5.00	4.3	5.8
45	1¾ in.		1.75	1.21	1.91	46.91	20	–	all	–	all	–	4.50	3.8	5.2
		40	1.57	1.08	1.75	41.75	20	–	all	–	all	–	4.50	3.8	5.2
37.5	1½ in.		1.50	1.01	1.67	39.17	20	1.103	all	–	all	–	4.50	3.8	5.2
		35.5	1.40	0.961	1.60	37.10	20	1.035	all	–	all	–	4.00	3.4	4.6
31.5	1¼ in.		1.25	0.855	1.47	32.97	20	0.907	all	–	all	–	4.00	3.4	4.6
		28	1.10	0.762	1.35	29.35	20	0.802	all	–	all	–	3.55	3.0	4.1
26.5	1.06 in.		1.06	0.722	1.29	27.79	20	0.758	all	–	all	–	3.55	3.0	4.1
25	1.00 in.		1.00	0.682	1.24	26.24	20	0.715	all	–	all	–	3.55	3.0	4.1
22.4	⅞ in.		0.875	0.613	1.14	23.54	150	0.641	15	0.431	30	0.460	3.55	3.0	4.1
		20	0.787	0.548	1.05	21.05	150	0.575	15	0.387	30	0.413	3.15	2.7	3.6
19	¾ in.		0.750	0.522	1.01	20.01	150	0.548	15	0.368	30	0.393	3.15	2.7	3.6
		18	0.709	0.495	0.97	18.97	150	0.521	15	0.350	30	0.374	3.15	2.7	3.6
16	⅝ in.		0.625	0.441	0.89	16.89	150	0.467	15	0.314	30	0.335	3.15	2.7	3.6
		14	0.551	0.387	0.81	14.81	150	0.414	15	0.278	30	0.297	2.80	2.4	3.2
13.2	0.530 in.		0.530	0.365	0.78	13.98	150	0.393	15	0.264	30	0.282	2.80	2.4	3.2
12.5	½ in.		0.500	0.346	0.75	13.25	150	0.374	15	0.251	30	0.268	2.50	2.1	2.9
11.2	7/16 in.		0.438	0.311	0.69	11.89	150	0.340	15	0.229	30	0.244	2.50	2.1	2.9
		10	0.394	0.279	0.64	10.64	150	0.308	15	0.207	30	0.221	2.50	2.1	2.9
9.5	⅜ in.		0.375	0.265	0.61	10.11	150	0.294	15	0.198	30	0.211	2.24	1.9	2.6
		9	0.354	0.251	0.59	9.59	150	0.281	15	0.189	30	0.202	2.24	1.9	2.6
8	5/16 in.		0.312	0.224	0.54	8.54	150	0.254	15	0.171	30	0.182	2.00	1.7	2.3
		7.1	0.280	0.200	0.50	7.60	150	0.230	15	0.155	30	0.165	1.80	1.5	2.1
6.7	0.265 in.		0.265	0.189	0.48	7.18	150	0.219	15	0.147	30	0.157	1.80	1.5	2.1
6.3	¼ in.		0.250	0.178	0.46	6.76	150	0.208	15	0.140	30	0.149	1.80	1.5	2.1
5.6	No. 3½		0.223	0.159	0.42	6.02	150	0.189	15	0.127	30	0.136	1.60	1.3	1.9
		5	0.197	0.142	0.39	5.39	150	0.172	15	0.116	30	0.123	1.60	1.3	1.9
4.75	No. 4		0.187	0.135	0.37	5.12	150	0.165	15	0.111	30	0.118	1.60	1.3	1.9
		4.5	0.177	0.128	0.36	4.86	150	0.158	15	0.106	30	0.113	1.40	1.2	1.7
4	No. 5		0.157	0.114	0.33	4.33	150	0.143	15	0.096	30	0.103	1.40	1.2	1.7
		3.55	0.140	0.102	0.30	3.85	200	0.130	20	0.092	40	0.097	1.25	1.06	1.50
3.35	No. 6		0.132	0.096	0.29	3.64	200	0.125	20	0.088	40	0.093	1.25	1.06	1.50
		3.15	0.124	0.091	0.28	3.43	200	0.119	20	0.084	40	0.089	1.25	1.06	1.50
2.8	No. 7		0.110	0.081	0.26	3.06	200	0.108	20	0.076	40	0.081	1.12	0.95	1.30
		2.5	0.0984	0.073	0.24	2.74	200	0.099	20	0.070	40	0.074	1.00	0.85	1.15
2.36	No. 8		0.0937	0.069	0.23	2.59	200	0.095	20	0.067	40	0.071	1.00	0.85	1.15
		2.24	0.0882	0.065	0.22	2.46	200	0.091	20	0.064	40	0.068	0.90	0.77	1.04
2	No. 10		0.0787	0.059	0.20	2.20	250	0.083	25	0.060	50	0.064	0.90	0.77	1.04
		1.8	0.0709	0.053	0.19	1.99	250	0.077	25	0.056	50	0.059	0.80	0.68	0.92
1.7	No. 12		0.0661	0.050	0.18	1.88	250	0.074	25	0.054	50	0.057	0.80	0.68	0.92

Sieve Designation			Nominal Sieve Opening	±Y Variation for Average Opening	+X Maximum Variation for Opening	Resulting Maximum Individual Opening	Compliance Sieves		Inspection Sieves		Calibration Sieves		Typical Wire Diameter	Permissible Average Wire Diameter	
Standard	U.S. Alternative	Supplementary Size					Sample Openings per 100 ft²	Maximum Standard Deviation	Sample Openings per Sieve	Maximum Standard Deviation	Sample Openings per Sieve	Maximum Standard Deviation		Min	Max
		1.6	0.0630	0.047	0.17	1.77	250	0.070	25	0.051	50	0.054	0.80	0.68	0.92
1.4	No. 14		0.0555	0.042	0.16	1.56	400	0.064	40	0.050	80	0.052	0.71	0.60	0.82
		1.25	0.0492	0.038	0.15	1.40	400	0.058	40	0.045	80	0.047	0.63	0.54	0.72
1.18	No. 16		0.0469	0.036	0.14	1.32	400	0.056	40	0.043	80	0.045	0.63	0.54	0.72
		1.12	0.0441	0.034	0.14	1.26	400	0.054	40	0.042	80	0.044	0.56	0.48	0.64
1	No. 18		0.0394	0.030	0.13	1.13	400	0.050	40	0.039	80	0.040	0.56	0.48	0.64
µm		µm	in.	µm	µm	µm							mm		
		900	0.0354	27.6	118	1018	400	45.51	40	35.22	80	36.74	0.500	0.43	0.58
850	No. 20		0.0331	26.2	114	964	400	43.66	40	33.79	80	35.25	0.500	0.43	0.58
		800	0.0315	24.8	109	909	400	41.79	40	32.34	80	33.74	0.450	0.38	0.52
710	No. 25		0.0278	22.2	101	811	500	38.36	50	30.43	100	31.62	0.450	0.38	0.52
		630	0.0248	19.9	93	723	500	35.23	50	27.95	100	29.04	0.400	0.34	0.46
600	No. 30		0.0234	19.0	91	691	500	34.04	50	27.00	100	28.06	0.400	0.34	0.46
		560	0.0220	17.9	87	647	500	32.43	50	25.73	100	26.73	0.355	0.30	0.41
500	No. 35		0.0197	16.2	80	580	600	29.96	60	24.21	120	25.09	0.315	0.27	0.36
		450	0.0177	14.7	75	525	600	27.86	60	22.51	120	23.32	0.280	0.24	0.32
425	No. 40		0.0165	14.0	73	498	600	26.79	60	21.65	120	22.43	0.280	0.24	0.32
		400	0.0157	13.3	70	470	600	25.71	60	20.78	120	21.52	0.250	0.21	0.29
355	No. 45		0.0139	12.0	65	420	800	23.72	80	19.68	160	20.30	0.224	0.19	0.26
		315	0.0124	10.8	60	375	800	21.90	80	18.17	160	18.75	0.200	0.17	0.23
300	No. 50		0.0117	10.4	58	358	800	21.20	80	17.59	160	18.15	0.200	0.17	0.23
		280	0.0110	9.8	56	336	800	20.26	80	16.81	160	17.34	0.180	0.15	0.21
250	No. 60		0.0098	8.9	52	302	800	18.82	80	15.61	160	16.11	0.160	0.13	0.19
		224	0.0088	8.1	49	273	800	17.53	80	14.54	160	15.01	0.160	0.13	0.19
212	No. 70		0.0083	7.8	47	259	800	16.93	80	14.05	160	14.49	0.140	0.12	0.17
		200	0.0079	7.4	45	245	800	16.32	80	13.54	160	13.97	0.140	0.12	0.17
180	No. 80		0.0070	6.8	43	223	1000	15.27	100	12.91	200	13.28	0.125	0.106	0.150
		160	0.0063	6.3	40	200	1000	14.20	100	12.00	200	12.34	0.112	0.095	0.130
150	No. 100		0.0059	6.0	38	188	1000	13.65	100	11.53	200	11.86	0.100	0.085	0.115
		140	0.0055	5.7	37	177	1000	13.09	100	11.06	200	11.38	0.100	0.085	0.115
125	No. 120		0.0049	5.2	34	159	1000	12.23	100	10.33	200	10.63	0.090	0.077	0.104
		112	0.0044	4.8	32	144	1000	11.46	100	9.68	200	9.96	0.080	0.068	0.092
106	No. 140		0.0041	4.7	31	137	1000	11.10	100	9.38	200	9.65	0.071	0.060	0.082
		100	0.0039	4.5	30	130	1000	10.73	100	9.07	200	9.33	0.071	0.060	0.082
90	No. 170		0.0035	4.2	29	119	1000	10.10	100	8.53	200	8.78	0.063	0.054	0.072
		80	0.0031	3.9	27	107	1000	9.45	100	7.99	250	8.33	0.560	0.048	0.064
75	No. 200		0.0029	3.7	26	101	1000	9.12	100	7.70	250	8.04	0.050	0.043	0.058
		71	0.0028	3.6	25	96	1000	8.85	100	7.48	250	7.80	0.050	0.043	0.058
63	No. 230		0.0025	3.4	24	87	1000	8.29	100	7.01	250	7.31	0.045	0.038	0.052
		56	0.0022	3.2	22	78	1000	7.79	100	6.58	250	6.87	0.040	0.034	0.046
53	No. 270		0.0021	3.1	21	74	1000	7.56	100	6.39	250	6.67	0.036	0.031	0.041
		50	0.0020	3.0	21	71	1000	7.34	100	6.20	250	6.47	0.036	0.031	0.041
45	No. 325		0.0017	2.8	20	65	1000	6.95	100	5.87	250	6.13	0.032	0.027	0.037
		40	0.0016	2.7	19	59	1000	6.55	100	5.54	300	5.83	0.032	0.027	0.037
38	No. 400		0.0015	2.6	18	56	1000	6.38	100	5.39	300	5.69	0.030	0.024	0.035
		36	0.0014	2.6	18	54	1000	6.22	100	5.26	300	5.54	0.030	0.024	0.035
32	No. 450		0.0012	2.4	17	49	1000	5.87	100	4.96	300	5.23	0.028	0.023	0.033
25	No. 500		0.0010	2.2	15	40	1000	5.23	100	4.42	300	4.66	0.025	0.021	0.029
20	No. 635		0.0008	2.1	13	33	1000	4.73	100	4.00	300	4.22	0.020	0.017	0.023

[A] Column 3—These numbers are only approximate but are in use for reference; the sieve shall be identified by the standard designation in millimetres or micrometres.
[B] Columns 9 and 11—See Annex A1, which specifies that all openings will be inspected for test sieves having 15 openings or less.
[C] Columns 9 and 11—These number of sample openings are based on an 8-in. diameter test sieve.

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