

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

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Introduction

The procedures presented in this document have been developed to improve the quality of proppants delivered to the well site. They are for use in evaluating certain physical properties used in hydraulic fracturing and gravel-packing operations. These tests should enable users to compare the physical characteristics of various proppants tested under the described conditions and to select materials useful for hydraulic fracturing and gravel-packing operations. Each section contains proppant specifications and replaces API 19C, First Edition.

The procedures presented in this document are not intended to inhibit the development of new technology, material improvements, and improved operational procedures. Qualified engineering analysis and judgment are required for their application to a specific situation.

As with any laboratory procedure requiring the use of potentially hazardous chemicals and equipment, the user is expected to have received proper training and knowledge in the use and disposal of these potentially hazardous materials. The user is responsible for compliance with all applicable local, regional, and national requirements for worker and local health, safety, and environmental liability.

This document uses a format for numbers that follows the examples given in API Document Format and Style Manual, First Edition, June 2007 (Editorial Revision, January 2009). This numbering format is different than that used in API 19C, Second Edition. In this document, the decimal mark is a period and separates the whole part from the fractional part of a number. No spaces are used in the numbering format. The thousands separator is a comma and is only used for numbers greater than 10,000, i.e. 5000 items, 12,500 bags.

In this document, where practical, U.S. Customary units are included in parentheses for information.

The units do not necessarily represent a direct conversion of metric units (SI) to U.S. Customary units, or U.S. Customary to SI. Consideration has been given to the precision of the instrument making the measurement. Consideration has also been given to the nature of the measurement, determining if it is critical or simply informational. The document was originally based on API RP 56, API RP 58, and API RP 60, which are no longer in effect.

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.

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.

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

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

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

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.

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

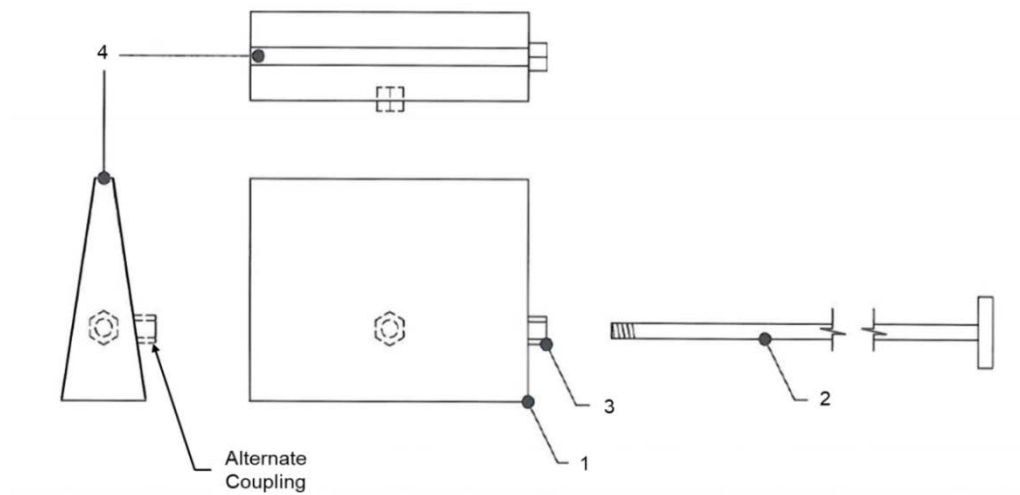
With a minimum slot opening six times the largest particle diameter sampled.

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:

$$6 \times 2.36 \text{ mm} = 14.2 \text{ mm or } 0.557 \text{ in.}$$

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.



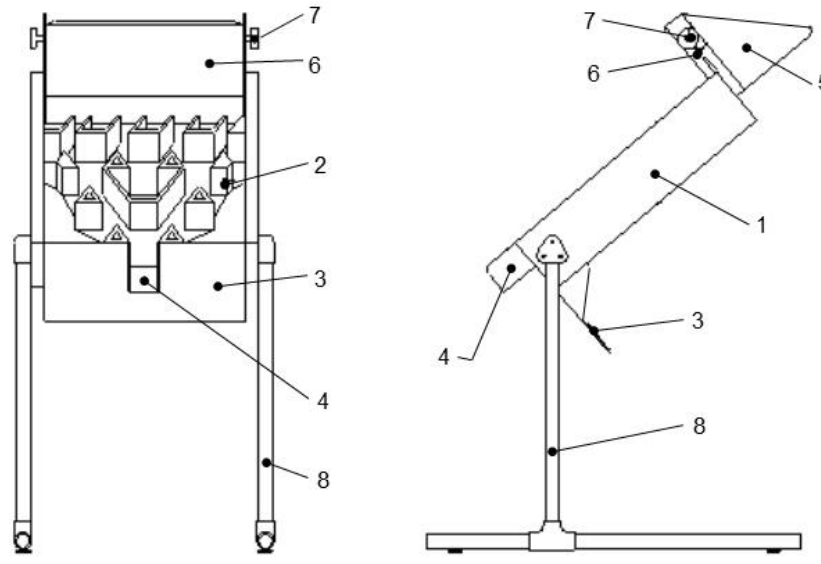
Key

- 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 $\frac{1}{16}$ of the original mass. See Figure 2.



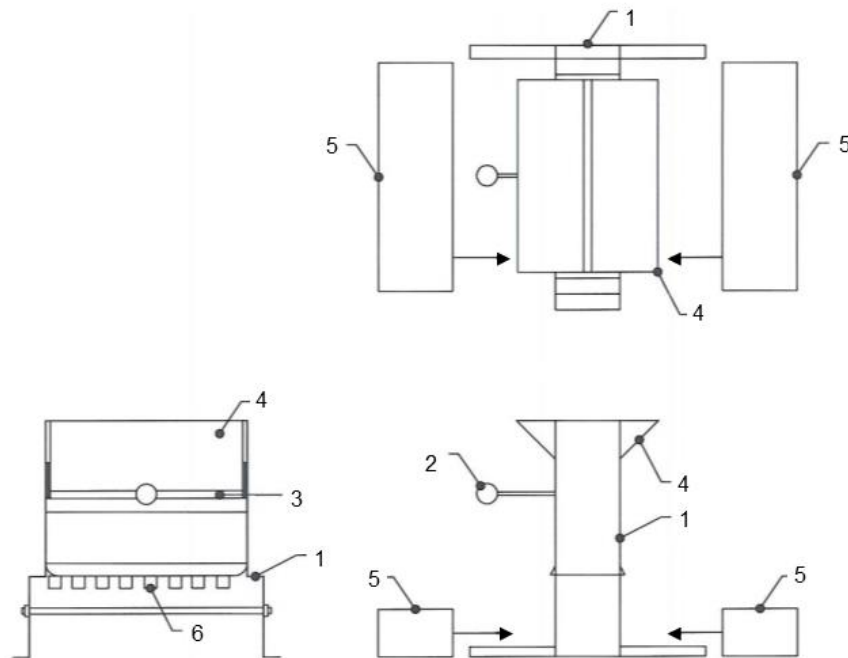
Key

- 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 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 $\frac{1}{16}$ of the original mass of the total sack's contents, typically 3 kg (6.6 lb).

5.2 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.

5.3 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. Alternate methods may be used, but shall be correlated with these standard methods.

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](#).

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 $100 \text{ g} \pm 20 \text{ g}$, 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).

$$n_i = 100 \times \frac{m_i}{\sum m_i} \quad (1)$$

where

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

$\sum m_i$ 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 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.

6.5.3 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.