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Petroleum and natural gas industries — Completion fluids and materials —

Part 2:

Measurement of properties of proppants used in hydraulic fracturing and gravel-packing operations

Industries du pétrole et du gaz naturel — Fluides de complétion et matériaux —

Partie 2: Mesurage des propriétés des matériaux de soutènement utilisés dans les opérations de fracturation hydraulique et de remplissage de gravier

Reference number
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Contents

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13503-2 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cements*.

ISO 13503 consists of the following parts, under the general title *Petroleum and natural gas industries — Completion fluids and materials*:

— *Part 1: Measurement of viscous properties of completion fluids*

— *Part 2: Measurement of properties of proppants used in hydraulic fracturing and gravel-packing operations*

— *Part 3: Testing of heavy brines*

— *Part 4: Procedure for measuring stimulation and gravel-pack fluid leakoff under static conditions*

— *Part 5: Procedures for measuring the long-term conductivity of proppants*

Introduction

This part of ISO 13503 is a compilation and modification of API RP 56 [1], API RP 58 [2] and API RP 60 [3].

The procedures 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.

The procedures presented in this part of ISO 13503 are not intended to inhibit the development of new technology, material improvements or improved operational procedures. Qualified engineering analysis and judgment are required for their application to a specific situation.

In this part of ISO 13503, where practical, US Customary (USC) units are included in brackets for information.

Annex A of this part of ISO 13503 is for information only.

Petroleum and natural gas industries — Completion fluids and materials —

Part 2:

Measurement of properties of proppants used in hydraulic fracturing and gravel-packing operations

1 Scope

This part of ISO 13503 provides standard testing procedures for evaluating proppants used in hydraulic fracturing and gravel-packing operations.

NOTE "Proppants" mentioned henceforth in this part of ISO 13503 refer to sand, ceramic media, resin-coated proppants, gravel-packing media and other materials used for hydraulic fracturing and gravel-packing operations.

The objective of this part of ISO 13503 is to provide a consistent methodology for testing performed on hydraulic fracturing and/or gravel-packing proppants.

2 Normative references

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

ASTM E11, *Standard Specification for Wire Cloth and Sieves for Testing Purposes*

3 Abbreviations

API	American Petroleum Institute
ASTM	American Society for Testing and Materials
ASG	apparent specific gravity
FTU	formazin turbidity unit
HCl	hydrochloric acid
HF	hydrofluoric acid
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, as each test requires a different volume. It is very 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 very difficult. It is unlikely that sampling/testing methods in the field duplicate the producer's system. The standard procedures included within this part of ISO 13503 are to assist in obtaining representative samples. However, there are inherent variations associated with sampling, testing equipment and the 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 an initial source of wide variation when making comparisons. All parties shall take care to insure uniform sampling. The customer and the 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 site, consideration should be given to the number or 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, 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 size.

4.3 Equipment

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

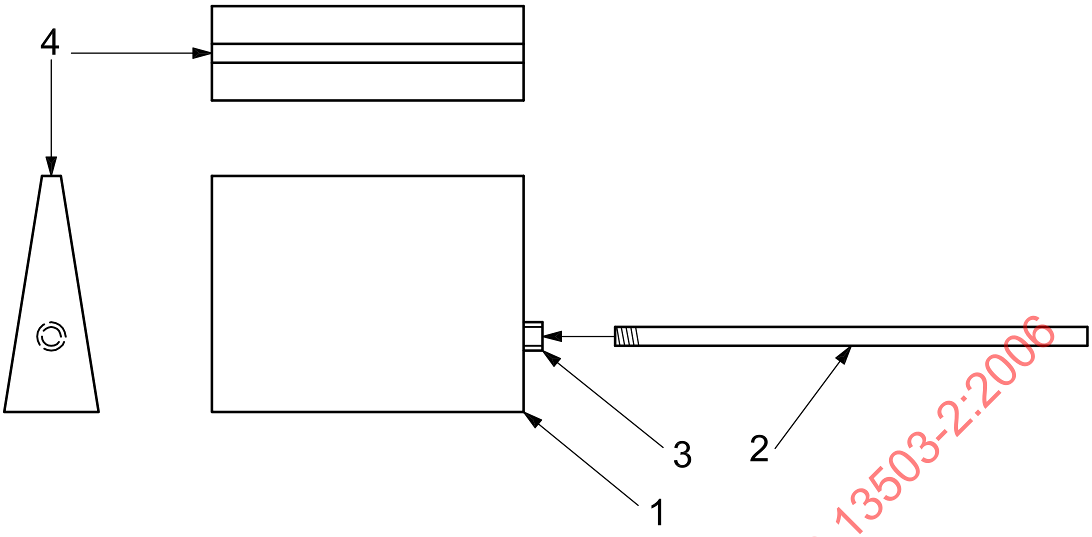
4.3.1 Box sampling device, with a 13 mm (0,50 in) slot opening.

The length of the 13 mm (0,50 in) 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. A box sampling device meeting these criteria is shown in Figure 1.

4.3.2 Sample reducer, of appropriate size for handling sack-size samples and reducing the material to 1/16 of the original mass; see Figure 2.

4.3.3 Sample splitter, of appropriate size; see Figure 3.

Dimensions in centimetres (inches)

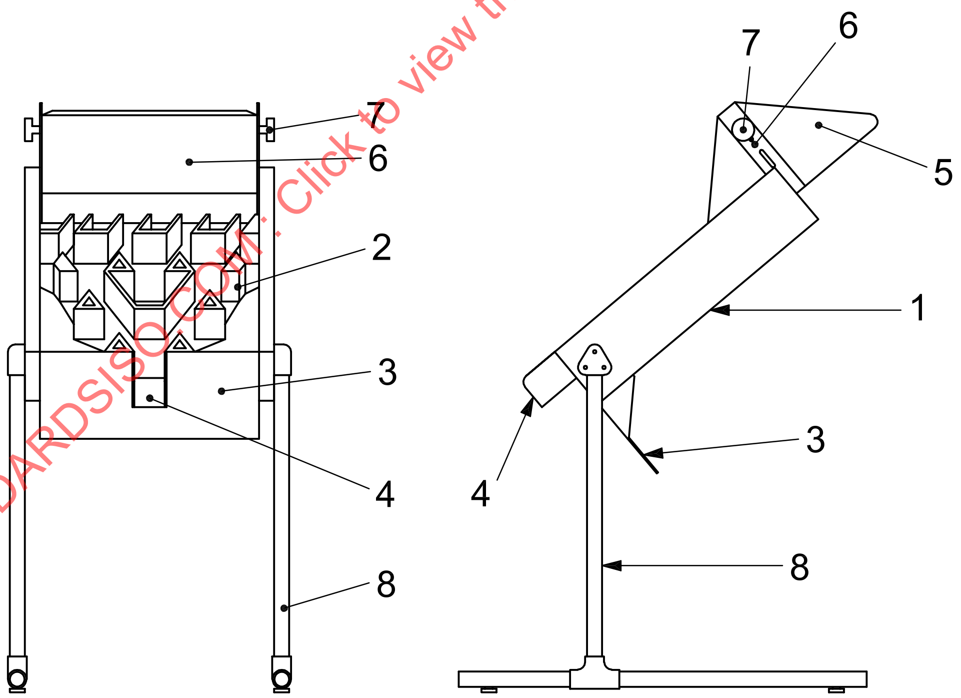


Key

1	sampler body, 15,9 × 20,9 × 6,35 (6,25 × 8,25 × 2,5)	3	pipe coupling
2	handle	4	sample opening, 1,27 (0,50)

Figure 1 — Box sampling device

Dimensions in centimetres (inches)

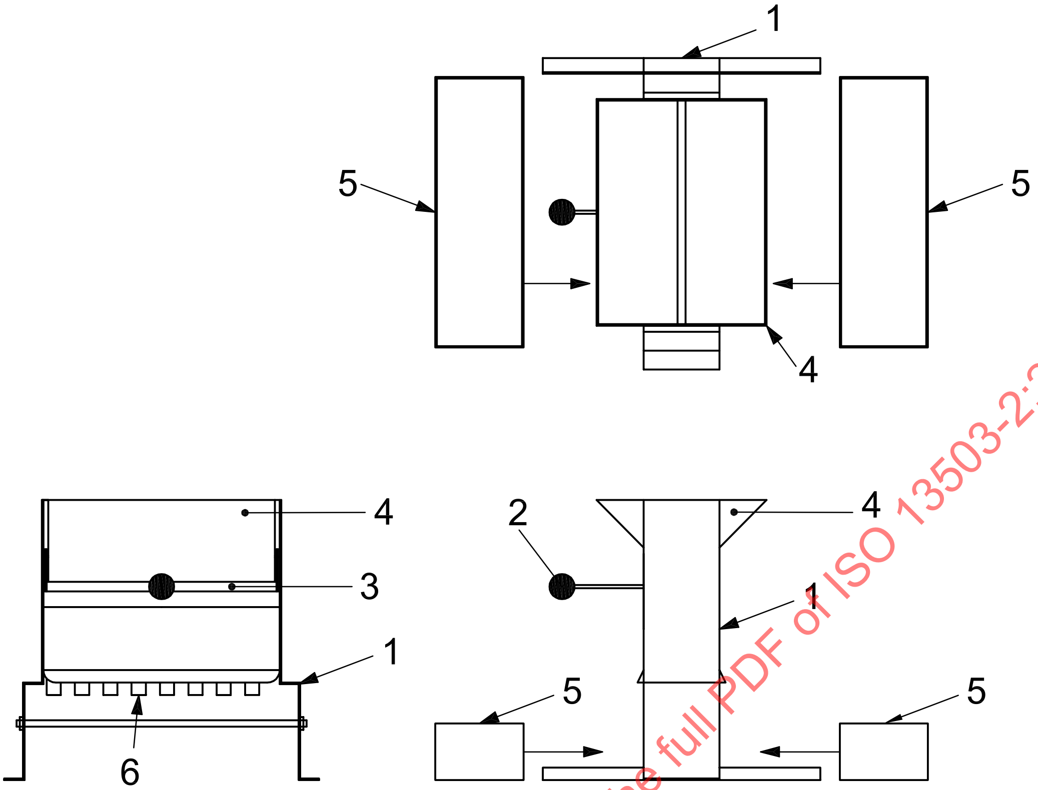


Key

1	main body, 36,8 × 48,3 × 11,4 (14,5 × 19,0 × 4,5)	5	hopper, 36,8 × 24,1 × 15,2 (14,5 × 9,5 × 6,0)
2	splitter plate, 5,1 × 5,1 × 5,1 (2 × 2 × 2)	6	gate, 36,8 × 19,1 × 0,32 (14,5 × 7,5 × 0,125)
3	discharge plate, 36,8 × 30,5 × 0,32 (14,5 × 12 × 0,125)	7	hand knob, 3,8 (1,5) diameter
4	discharge chute, 5,7 × 5,7 × 7,6 (2,25 × 2,25 × 3,0)	8	support stand assembly, 71,1 × 38,1 × 68,6 (28 × 15 × 27)

Figure 2 — Sample reducer

Dimensions in centimetres (inches)



Key

1	main body, 29,2 × 27,9 × 16,5 (11,5 × 11,0 × 6,5)
2	handle
3	gate plate
4	hopper
5	pan
6	splitter vanes, 1,25 (0,5)

Figure 3 — Sample splitter

4.4 Number of required samples — Bulk

4.4.1 Proppants for hydraulic fracturing

A minimum of one sample per 9 000 kg (20 000 lb) or fraction thereof, shall be obtained. A maximum of 10 samples per bulk container shall be obtained, combined and tested.

4.4.2 Gravel-packing media

A minimum of one sample per 4 500 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. Two metric tons 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 take the entire stream with each pass. Under no circumstances shall the sampling receptacle be allowed to overflow.

4.6 Sampling — Bagged material

4.6.1 Bags up to 50 kg (110 lb)

Only whole bags shall be used for sampling bagged proppant materials.

4.6.2 Totes/bulk bags/super sacks weighing up to 2 000 kg (4 400 lb)

Unless the product can be sampled in a free-flowing state, the sampling of large bags presents the same problems as for a static pile. Follow the same sample frequency as described in 4.4, using the sampling method described in 4.5, except for allowing 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 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 permit samples to be prepared 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 (refer to Figure 3) and split the sample to a testing-aliquot size of approximately 1 kg (2,2 lb). Sufficient proppant material shall be split to permit performance of recommended tests as specified in this part of ISO 13503.

5.3 Sample and 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 the procedure in Clause 6 is to ensure a consistent methodology for sieve analysis and to provide a consistent procedure for sieve evaluation.

6.2 Description

The procedure and equipment described in 6.3 to 6.6 are the most widely utilized in the gas and oil industry. Alternate methods may be used but shall be correlated with these standard methods.

6.3 Equipment and materials

6.3.1 Sieve sets, two, complying with the requirements of the ASTM Series, 200 mm (8 in) diameter or equivalent.

One set is a working set of sieves, and the other a master set to be used for standardization only.

Refer to ASTM E11.

6.3.2 Testing sieve shaker, providing simultaneous rotating and tapping action, that accepts the sieves specified in Table 1.

The shaker shall be calibrated to the following specifications: 290 rev/min, 156 taps/min, height of tapper 33,4 mm (1,3 in) and timer accurate to ± 5 s.

6.3.3 Balance, minimum 100 g (0,22 lb) capacity with a precision of 0,1 g or better.

6.3.4 Brushes, nylon or equivalent.

6.4 Procedure

6.4.1 Stack a minimum of seven sieves, recently 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 can become available.

6.4.2 Using a split sample of 80 g to 120 g, obtain an accurate sample to within 0,1 g.

6.4.3 Weigh each sieve and record the mass. Pour the split sample onto the top sieve, place the stack of sieves plus pan and lid in testing sieve shaker and agitate for 10 min.

6.4.4 Remove the sieve stack from the testing sieve shaker.

6.4.5 Weigh and record the mass retained on each of the sieves and in the pan.

Calculate the percent mass of the total proppant sample retained on each sieve and in the pan. The cumulative mass shall be within 0,5 % of the sample mass used in the test. If not, the sieve analysis shall be repeated using a different sample.

Table 1 — Sieve sizes [a]

	Sieve-opening sizes µm										
	3 350/ 1 700	2 360/ 1 180	1 700/ 1 000	1 700/ 850	1 180/ 850	1 180/ 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 [b]										
First primary 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
Second primary 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.

[b] Sieves stacked in order from top to bottom.

6.5 Calculation of the mean diameter, median diameter and standard deviation

6.5.1 General

The mean diameter, d_{av}, shall be used to characterize the proppant distribution for hydraulic fracturing. The median diameter, d_{50}, shall be used to characterize gravel-packing distributions. This is in addition to the mesh-size characterization described in 6.4.

6.5.2 Mean diameter

The mean diameter, d_{av}, expressed in millimetres, is calculated as given in Equation (1):

$$d_{\text{av}} = \sum n \cdot d / \sum n \qquad (1)$$

where $n \cdot d$ is the product of mid-size diameter (d) multiplied by frequency of occurrence (n).

EXAMPLE Calculation of the mean diameter for a 16/30 proppant with the following size distribution; see Table 2:

+12 mesh	(1 700 µm)	0,0 %
+16 mesh	(1 180 µm)	1,2 %
+18 mesh	(1 000 µm)	37,9 %
+20 mesh	(850 µm)	48,7 %
+25 mesh	(710 µm)	11,9 %
+30 mesh	(600 µm)	0,3 %
+40 mesh	(425 µm)	0,0 %
Pan		0,0 %
TOTAL		100,0 %

Table 2 — Parameters for mean diameter calculation

US mesh size	Particle-size interval µm	Mid size d µm	Frequency of occurrence n % by mass	$n{\cdot}d$
10 to 12	2 000 to 1 700	1 850	0,0	0
12 to 16	1 700 to 1 180	1 440	1,2	1 728
16 to 18	1 180 to 1 000	1 090	37,9	41 311
18 to 20	1 000 to 850	925	48,7	45 047
20 to 25	850 to 710	780	11,9	9 282
25 to 30	710 to 600	655	0,3	197
	TOTAL		100,0	97 565

Therefore, the mean diameter, $d_{\mathrm{av}} = \sum n \cdot d \big/ \sum n$ = 97 565/100,0 = 975,7 µm = 0,976 mm (0,038 in).

6.5.3 Median diameter

In gravel-packing, the median diameter, d_{50} (the fiftieth mass percentile), which is commonly used, is calculated as follows.

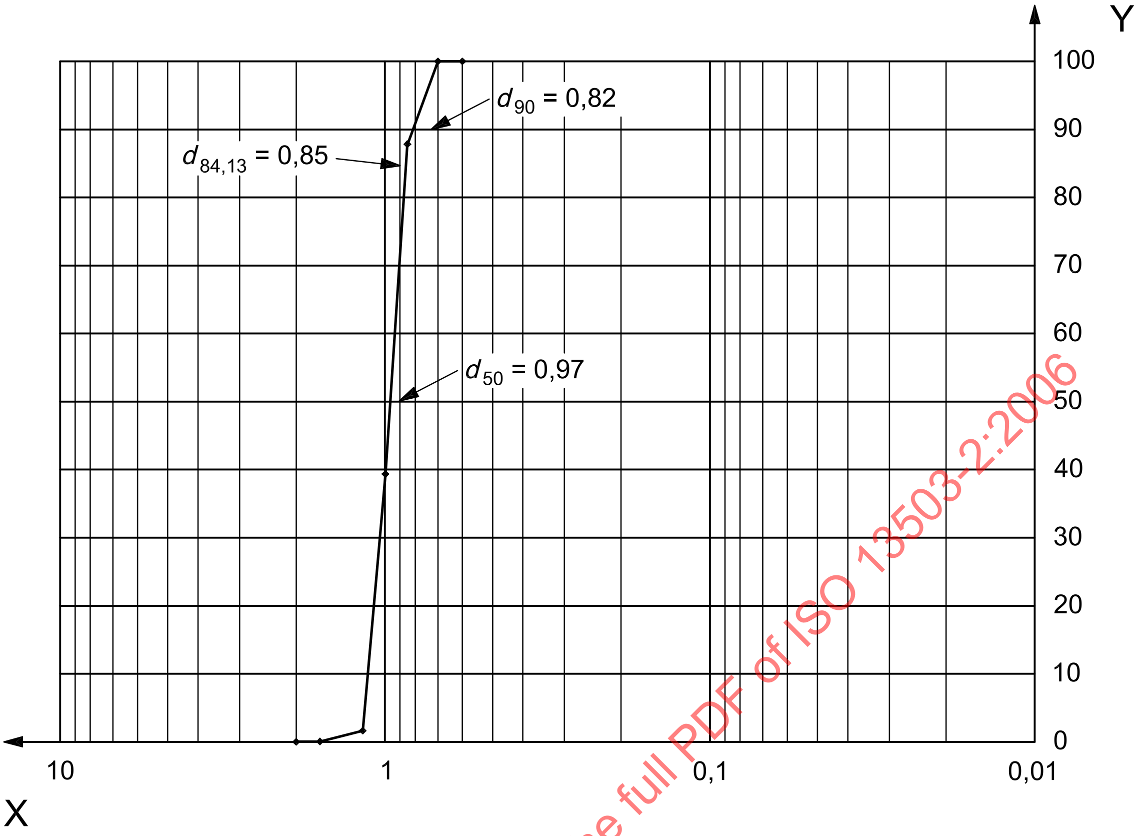
Determine the percent of the sample retained on each screen. This is done by dividing the mass retained on the screen by the total cumulative mass (not the original mass) and multiplying by 100. Determine the cumulative percent retained for each screen by adding the percent retained on that screen with the percents retained on the larger screens that are above it. The cumulative percent of the material retained by all the screens and the pan should be 100 %.

Plot a particle-size distribution curve with cumulative percent on the y-axis versus log of sieve opening on the x-axis, which is inverted so that the scale is smaller to the right and larger to the left. The sieve size can be plotted as microns, millimetres or inches. A plot of the example in 6.5.2 is plotted in Figure 4. Reading the graph at 50 % cumulative mass (on the y-axis) gives the d_{50} grain-size diameter (on the x-axis) of 0,97 mm (0,038 in).

d_{50} is also the median diameter, which is the size at which 50 % of the particles are smaller and 50 % are larger. In this example, the mean and median are very close together. This might not be the case in highly skewed distributions.

From Figure 4 other common criteria, such as d_{90} , and standard deviation, σ, can be determined. Reading the graph at 90 % cumulative mass (on the y-axis) gives the d_{90} grain-size diameter (on the x-axis) of 0,82 mm (0,032 in).

The standard deviation, σ, is calculated from the expression $d_{84,13}/d_{50}$ = 0,85 mm/0,97 mm = 0,88.



Key

X size, expressed in millimetres

Y cumulative percent

Figure 4 — Graphical determination of d_{50}, d_{90} and $d_{84,13}$

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 in both 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.

6.6.2 Description

A stack of master sieves is used to check working sieves. This master stack should be used very sparingly to prevent major 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 that the sieves are in accordance with latest revision of ASTM E11. If any sieve fails the ASTM E11 specification, the sieve shall be replaced with a new certified sieve. Storage of sieves shall be done in such a manner as to prevent deterioration and/or damage.

6.6.3 Procedure

Place a calibration sample (see 6.6.4) into the master sieve stack and complete sieve analysis according to 6.4. The load retained on each master sieve is considered the master load for calculation purposes.

This 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. If the total test sample mass exceeds ± 0,2 % difference from the master stack to the working stack, repeat the test. Calculate the difference, D, as given in Equation (2), the % difference, D', as given in Equation (3), absolute deviation, δ_A, as given in Equation (4), and % absolute deviation, δ'_A, as given in Equation (5). If the percent absolute deviation exceeds 10 % between the master sieve set and the working/supplier sieve, the difference should be considered when comparing results to sieve recommendations. If the absolute deviation exceeds 25 %, the working sieve shall be replaced.

$$D = m_\text{Ws} - m_\text{Ms} \tag{2}$$

$$D' = (D/m_\text{Ms}) \times 100 \tag{3}$$

where

D is the difference, expressed in millimetres;

D' is the difference, expressed in percent;

m_Ms is the mass retained on each sieve in the master set;

m_Ws is the mass retained on each sieve in the working set.

NOTE For the top sieve of the stack, the absolute deviation equals the difference. This number can be positive or negative.

Absolute deviation is calculated from Equation (4):

$$\delta_{\text{A,S}(I)} = \delta_{\text{A,S}(I-1)} + D_{\text{S}(I)} \tag{4}$$

where

$\delta_{\text{A,S}(I)}$ is the absolute deviation of the sieve of interest, expressed in millimetres;

$\delta_{\text{A,S}(I-1)}$ is the absolute deviation of the preceding sieve;

$D_{\text{S}(I)}$ is the difference for the sieve of interest.

Percent absolute deviation, $\delta'_{\text{A,S}(I-1\pm D)}$, is calculated from Equation (5):

$$\delta'_{\text{A,S}(I-1\pm D)} = (\delta_{\text{A,S}(I-1\pm D)}/m_\text{Ms}) \times 100 \tag{5}$$

where $\delta_{\text{A,S}(I-1\pm D)}$ is the absolute deviation of preceding sieve ± $D_{\text{S}(I)}$, the difference for the sieve of interest.

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

EXAMPLE Example calculation for 25-mesh sieve; see Table 3:

- from Equation (2): $D = 10{,}5 - 10{,}1 = 0{,}4$
- from Equation (3): $D' = (0{,}4/10{,}1)\ 100 = 4{,}0$
- from Equation (4): $\delta_{\text{A,S}(I)} = 1{,}8 + 0{,}4 = 2{,}2$
- from Equation (5): $\delta'_{\text{A,S}(I-1\pm D)} = (2{,}2/10{,}1)\ 100 = 22{,}0$

Table 3 —Absolute deviation example

Sieve mesh size	m_{Ms} g	m_{Ws} g	D mm	D' %	$\delta_{\mathrm{A,S}(l)}$ mm	$\delta'_{\mathrm{A,S}(l)}$ %
12	9,4	10,0	0,6	6,4	0,6	6
16	10,9	11,7	0,8	7,3	1,4	13
18	9,5	9,2	−0,3	−3,1	1,1	12
20	9,5	10,2	0,7	7,4	1,8	19
25	10,1	10,5	0,4	4,0	2,2	22
30	10,0	9,9	−0,1	−1,0	2,1	21
35	11,4	10,9	−0,5	−4,4	1,6	14
40	9,3	9,2	−0,1	−1,1	1,5	16
50	10,1	9,2	−0,9	−8,9	0,6	6
70	8,8	8,9	0,1	1,1	0,7	8
Pan	0,9	0,4	—	—	—	—

6.6.4 Preparing calibration samples

A sieve calibration sample is prepared by blending sized samples. To prepare the sized samples, first determine the specific mesh material(s) needed. Assemble a sieve stack that covers these mesh sizes and place a pan at the bottom.

Place 100 g ± 20 g of media that has been identified as a source for test material onto the top sieve and cover with a lid. Place the sieve stack into the sieve shaker and shake for 10 min. The mass of media used should be optimized without exceeding 35 g on any sieve.

Remove the stack from the sieve shaker and then remove the lid. Carefully remove the top sieve and invert onto a recovery pan. Place the material from the recovery pan into a storage container labelled specifically for this mesh-sized material. Return the sieve to the recovery pan. Brush any remaining material from the sieve and discard this material. Repeat for each sieve.

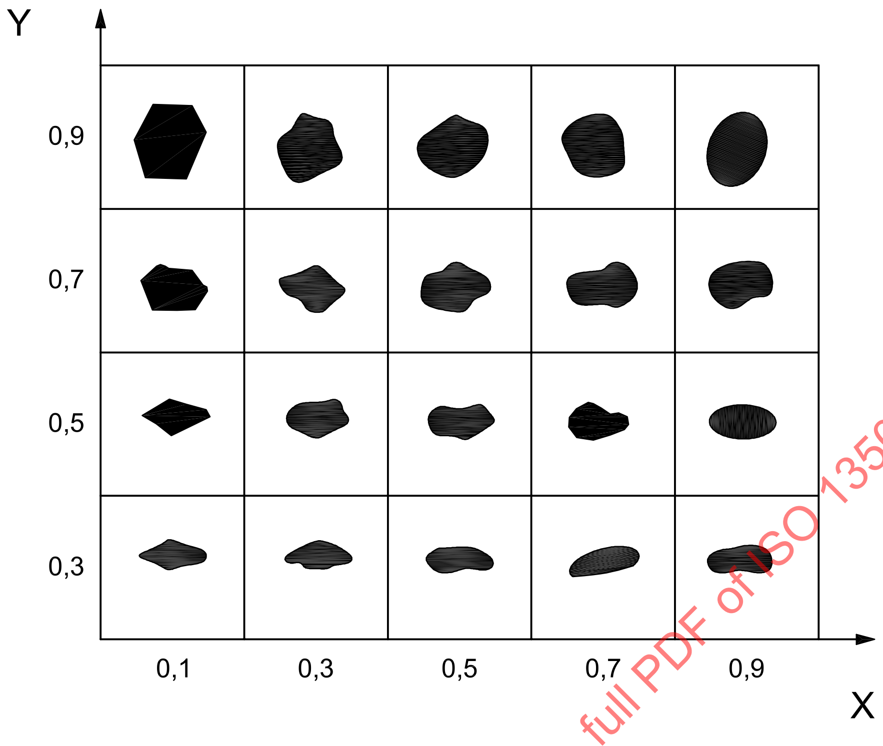
Select the sieve mesh sizes to be tested based on Table 1. For each sieve mesh size, weigh out approximately 10 g of correspondingly sized material. Blend the sized material together to form a calibration standard.

7 Proppant sphericity and roundness

7.1 Purpose

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

7.2 Description



Key

X roundness

Y sphericity

Figure 5 — Chart for visual estimation of sphericity and roundness

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 roundness and sphericity is the use of the Krumbien/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 of curvature. These measurements must be determined separately. Distinct measurement methods utilizing photographic or digital technology are available and are acceptable.

7.3 Apparatus capability

The apparatus shall have the following capabilities:

a) 10 times to 40 times magnification microscope or equivalent;

b) analytical balance, accuracy to 0,001 g.

7.4 Procedure

7.4.1 Using the split sample (see 5.2) and the sample splitter, further reduce the sample to 5 g to 15 g. If a small sample splitter is available, then the reduced sample amount can be safely reduced further to 1 g or 2 g.

7.4.2 Place the reduced sample onto 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 colour for a light-coloured proppant and, conversely, choose a lighter background colour for dark particles.

7.4.3 Randomly select at least 20 individual particles in the field of view for evaluation of particle sphericity.

7.4.4 Determine the sphericity of each selected particle by comparison to the chart provided, refer to Figure 5. Record the assigned 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 units.

7.4.6 Using the same individual particles selected in 7.4.3, determine the roundness of each particle. Record the assigned roundness number for each particle selected.

7.4.7 Calculate the arithmetic average of the recorded roundness numbers and report as average particle roundness to the nearest 0,1 units.

7.5 Alternate method for determining average sphericity and roundness

7.5.1 Use of photomicrographs

Photomicrographs of a representative proppant sample are used to provide identical, suitably enlarged reproductions for the determination of average sphericity and roundness for the reduced proppant sample. A scanning electron microscope, a reflected-light microscope fitted with a camera, a fixed camera with bellows and a wide-angle lens, digital camera and/or other appropriate photographic equipment can be successfully used to produce suitable photomicrographs of known magnification. The choice of equipment to use becomes a matter of individual preference and availability.

7.5.2 Procedure

Follow the procedure in 7.4, except use the photographic equipment of choice. Numbering each selected particle in the image limits confusion and assures the same particles are used for both the sphericity and the roundness determinations.

7.5.3 Suggested magnification for photomicrographs

For designated proppant size ranges, the magnifications are shown in Table 4.

Table 4 — Suggested magnification

Proppant size ranges	Photomicrograph magnification
6/12 through 12/20	15 times
16/20 through 20/40	30 times
30/40 through 70/140	40 times

8 Acid solubility

8.1 Purpose

The 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.

8.2 Description

The preferred method of testing acid solubility is using a solution of 12:3 HCl:HF acid (i.e. 12 % by mass of HCl and 3 % by mass of HF; see 8.4.2). However, the following procedural example shall not preclude the testing of materials in other acids, such as hydrochloric and organic acids, depending on desired application for the proppant.

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

8.3 Equipment and materials

8.3.1 Hydrochloric acid, HCl, concentrated (37 %), reagent grade of known concentration.

8.3.2 Ammonium bifluoride, (NH_4F_2), reagent grade.

8.3.3 Balance, 0,001 g accuracy.

8.3.4 Water bath, 66 °C (150 °F).

8.3.5 Oven, 105 °C (221 °F).

8.3.6 Beaker, 150 ml to 200 ml capacity, high-density polyethylene or polypropylene.

8.3.7 Graduated cylinder or **volumetric flask**, 1 000 ml, high-density polyethylene or polypropylene.

8.3.8 Analytical filtering apparatus, acid-resistant (porcelain, high-density polyethylene or polysulfone), using a vacuum-filtering technique with a Gooch-style [1] or Buchner-style [2] filtering crucible.

8.3.9 Filter paper circles, cellulose-based, acid-resistant, sufficient to prevent the loss of the sample.

The test procedure requires one or more filter papers with a diameter of 21 mm (0,83 in) for the Gooch crucible and 19,1 mm (0,83 in) for the Buchner crucible.

8.3.10 Timer, accurate to ± 5 s.

8.3.11 Desiccator.

8.4 Procedure

8.4.1 General

Representative samples shall be taken from the sample splitter; do not sieve the sample. Samples should not be subjected to the crush-resistance test or ground prior to the acid-solubility analysis, but rather the analysis shall be performed on the unaltered proppant.

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

8.4.2 Preparation of 12:3 HCl:HF solution

8.4.2.1 Prepare a solution of 12:3 HCl:HF acid [specific gravity = 1,08 at 15,6 °C (60 °F)] as follows:

8.4.2.2 To 500 ml of distilled water contained in a polyethylene or polypropylene 1 000 ml graduated cylinder or volumetric flask, add 46,23 g of pure NH_4F_2 and dissolve. The actual mass of NH_4F_2 of less than 100 % purity to be added is equal to 46,23 g divided by the purity of NH_4F_2, expressed as a mass fraction.

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[1] Gooch-style filtering crucible is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of this product.

[2] Buchner-style filtering funnel is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of this product.

8.4.2.3 Add 361 ml 37 % HCl [specific gravity of 1,19 at 15,6 °C (60 °F)]. Adjust volume for different concentrations of hydrochloric acid.

8.4.2.4 Dilute to 1 000 ml with distilled water.

8.4.2.5 Stir to ensure complete mixing.

8.4.3 Solubility test

8.4.3.1 Weigh 5 g of proppant to the nearest mg into a tared sample pan. The proppant should be dried at 105 °C (221 °F) to a constant mass and cooled in a desiccator. Record mass, m_S.

8.4.3.2 Add the proppant sample to a 150 ml polyethylene breaker containing 100 ml of the acid solution prepared in 8.4.2. Cover the breaker with a polyethylene watch glass. The acid and the samples should be at room temperature [22 °C ± 3 °C (72 °F ± 5 °F)].

8.4.3.3 Place the beaker in a 66 °C (150 °F) water bath for 30 min. Do not stir.

8.4.3.4 Dry the funnel and filter paper in an oven at 105 °C (221 °F) for a minimum of 1 h or until constant mass; weigh and record the mass, m_F. The filter medium should not be weighed hot, but should first be allowed to cool in a desiccator.

8.4.3.5 Transfer the sample 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 within 1 min.

8.4.3.6 Wash the sample in the filtering apparatus three times with 20 ml portions of distilled water.

8.4.3.7 Dry the filter and retained sample at 105 °C (221 °F) for a minimum of 1 h or until a constant mass is obtained. Cool the filter and sample in a desiccator before weighing. Weigh filter and sample and record the mass, m_{FS}.

8.4.3.8 Calculate S, the acid solubility, expressed in percent, of the proppant using Equation (6) and report the result:

$$S = \frac{\left(m_S + m_F - m_{FS}\right)}{m_S} \times 100 \qquad (6)$$

where

m_S is the sample mass, expressed in grams;

m_F is the mass of filter, expressed in grams;

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

9 Turbidity test

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 beam is normal to the detection path of the detector; this is the preferred method of measurement. The results are expressed in FTU and/or NTU. Alternate methods may be used but shall be correlated with the method given in this part of ISO 13503.

9.3 Equipment and materials

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

9.3.1 **Formazin calibration standards**; see Annex A.

9.3.2 **Spectronic** [3], set at a wavelength of 450 nm, or **turbidimeter**, such as Hach 2100A [4] or equivalent.

9.3.3 **Wrist-Action® Shaker** [5].

9.3.4 **Syringe**.

9.4 Equipment calibration

Equipment calibration shall be performed in accordance with procedures designated by the original equipment manufacturer or an outside calibration service.

9.5 Procedure

9.5.1 Using the sample splitter, reduce the 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 demineralized water, to a 250 ml Erlenmeyer flask having a wide mouth and a screw cap. Allow to stand for 30 min ± 1 min.

9.5.2 Set the frequency of the Wrist-Action® Shaker to the maximum range of 10. Allow the Wrist-Action® Shaker to shake for 30 s ± 5 s. Remove the flask and allow to stand for 5 min ± 1 min.

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

9.5.4 Place the suspended-particle sample in a test vial and place in a calibrated turbidity meter or Spectronic 20 [6].

9.5.5 Record the sample turbidity in FTU (NTU).

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[3] Spectronic is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of this product.

[4] Hach 2100A is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of this product.

[5] Wrist-Action® Shaker bottle capacity is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of this product.

[6] Spectronic 20 is an example of a suitable product available commercially. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of this product.

10 Procedures for determining proppant bulk density, apparent density and absolute density

10.1 Purpose

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

10.2 Description

The bulk density, apparent density and absolute density are important properties of proppants. 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 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. On the other hand, the absolute density excludes pores that can be in the proppant as well as void spaces between proppant.

10.3 Bulk density

NOTE This procedure is based on ANSI B74.4.

10.3.1 Equipment and materials

An apparatus, consisting of the following parts (see Figure 6), is used to determine the bulk density, ρ_{bulk}.

10.3.1.1 Funnel stand, consisting of a square, 304,8 mm × 304,8 mm (12 in × 12 in) metal base with a tripod 304,8 mm (12 in) high.

The upper part of the tripod shall consist of a horizontal circular platform, 203,2 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, stainless steel, having smooth inside seams and a shut-off at the outlet consisting of a 34,9 mm (1 3/8 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 1/2 in ± 1/64 in);
— bottom diameter (inside):	12,7 mm ± 0,4 mm (1/2 in ± 1/64 in);
— height of sloping section:	76.2 mm ± 0,4 mm (3 in ± 1/64 in);
— height of parallel section:	12,7 mm ± 0,4 mm (1/2 in ± 1/64 in).

10.3.1.3 Brass cylinder, of approximately 100 cm^3 (6,10 in^3) capacity, calibrated with water as described in 10.3.2.

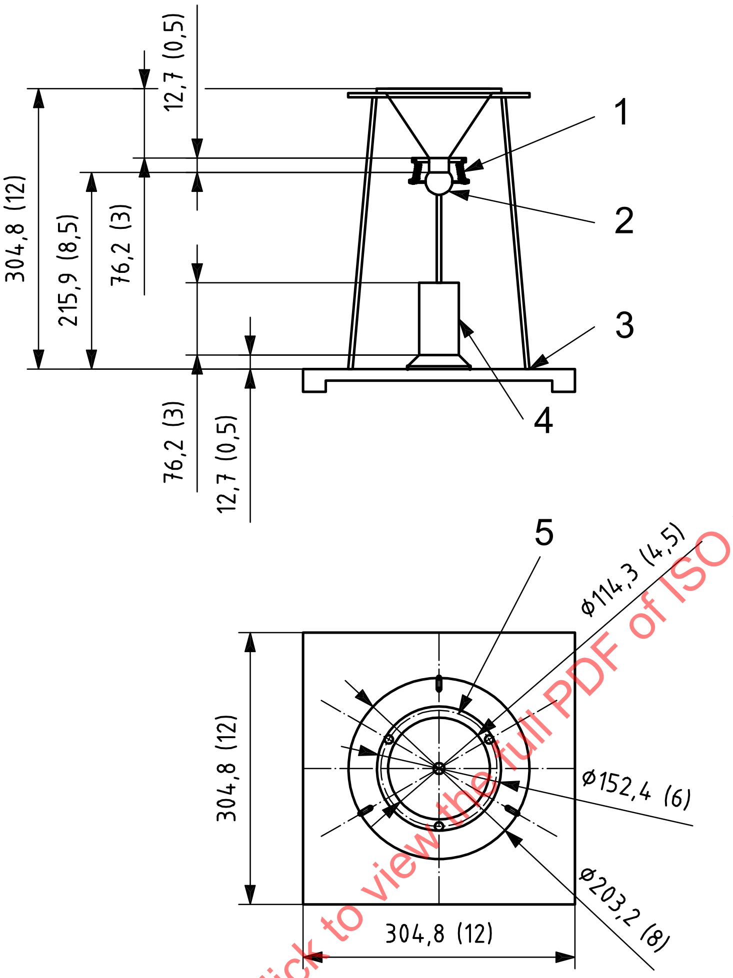
The cylinder shall be made of No. 17 gauge brass, seamless tubing and shall have the following dimensions:

— inside diameter:	38,9 mm (1 17/32 in). Brass tube inside diameter shall be within the tolerances normal to high grade commercial tubing;
— height:	84,1 mm ± 0,4 mm (3 5/16 in ± 1/64 in);
— base thickness:	12,7 mm ± 0,4 mm (1/2 in ± 1/64 in).

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

NOTE Non-essential dimensions of the apparatus can be adjusted at the convenience of the user.

Dimensions in millimetres (inches)



Key

1 spring, 9,525 mm (0,38 in) diameter

2 rubber ball stop, 30,48 mm (1,25 in) diameter

3 legs, 4,8 mm (0,19 in) diameter, welded to base

4 cylinder, 38,1 mm (1,50 in) internal diameter

5 3 holes on 139,7 mm (5,50 in) diameter

Figure 6 — Bulk density device

10.3.2 Calibration of cylinder

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 $m_{\mathrm{f+gp}}$.

10.3.2.2 Fill the cylinder with water and slide the plate into contact with the upper edge of the cylinder cutting off the water precisely in the plane of the edge.

10.3.2.3 With the glass plate held firmly in place, wipe off the excess water and obtain the gross mass, $m_{\mathrm{f+gp+l}}$.

10.3.2.4 Calculate the volume, V_{cyl}, expressed in cubic centimetres, of the cylinder as given in Equation (7):

$$V_{\text{cyl}} = m_{\text{w}} / 0{,}997\,1 \qquad (7)$$

where

m_{w} is the net mass, expressed in grams, of water, equal to $m_{\text{f+gp+l}} - m_{\text{f+gp}}$;

0,997 1 is the density of water; the temperature of the test fluid shall not be less than 18,3 °C (65 °F) and not more than 28,4 °C (85 °F).

10.3.3 Procedure

10.3.3.1 Weigh the dry, empty cylinder and record as m_{f}.

10.3.3.2 The sample of proppant to be tested shall have a temperature of not less than 18,3 °C (65 °F) and not more than 28,4 °C (85 °F). Fill a 150 ml beaker with the proppant sample.

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

10.3.3.4 Move the ball above to the right or left of the opening at the bottom of the funnel and allow the proppant to fall freely to fill 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 proppant-filled cylinder and record as $m_{\text{f+p}}$.

10.3.3.7 Calculate the bulk density, ρ_{bulk}, expressed in grams per cubic centimetre, as given in Equation (8):

$$\rho_{\text{bulk}} = \frac{m_{\text{P}}}{V_{\text{cyl}}} \qquad (8)$$

where

m_{p} is the net mass, expressed in grams, of the proppant, equal to $m_{\text{f+p}} - m_{\text{f}}$;

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

10.4 Apparent density

10.4.1 Equipment and materials

The following equipment is needed to determine apparent density, ρ_{p}, of proppants in kerosene.

10.4.1.1 **Analytical balance**, 0,01 g precision.

10.4.1.2 **Weighing boat**.

10.4.1.3 **Pycnometer**, calibrated, 25 ml or 50 ml.

10.4.1.4 **Test liquid**, low-viscosity, paraffinic oil, kerosene or similar oil, with a maximum viscosity less than 5 cP at the temperature of use.

10.4.1.5 **Funnel**, with stem to fit inside the pycnometer.

10.4.2 Procedure

10.4.2.1 Calibrate the volume of the pycnometer as given in 10.3.2.

10.4.2.2 Weigh the dry, empty pycnometer and record the mass as m_f.

10.4.2.3 Carefully fill the pycnometer to the fill line with test fluid at ambient temperature. Make certain that no air bubbles are trapped in the liquid and that the test fluid has been wiped off the outer surface of the pycnometer.

10.4.2.4 Weigh the test-fluid-filled pycnometer to 0,01 g precision and record the mass as $m_\mathrm{f+l}$.

10.4.2.5 Tare the weighing dish, then add approximately 10 g of proppant sample, weigh the dish and sample to 0,01 g precision, calculate the mass of the proppant and record it as m_p.

10.4.2.6 Pour out approximately one-half the volume of liquid in the pycnometer and transfer the weighed proppant sample from the weighing dish to the pycnometer. A funnel that fits into the neck of the pycnometer should be used.

10.4.2.7 Carefully add sufficient test liquid at ambient temperature to the pycnometer and fill to the fill line. Rotate the flask about its vertical axis until all air bubbles have been dislodged from the proppant. Refill the test liquid to the fill line, if necessary, and wipe off any test liquid on the flask surface.

10.4.2.8 Weigh the flask containing proppant and test liquid to 0,01 g precision and record as $m_\mathrm{f+l+p}$.

Look up the density or calculate the test-liquid density, ρ_l, expressed in grams per cubic centimetre, from Equation (9):

$$\rho_\mathrm{l} = \frac{m_\mathrm{f+l} - m_\mathrm{f}}{V_\mathrm{pyc}} \tag{9}$$

where

$m_\mathrm{f+l}$ is the mass, expressed in grams, of the flask filled with test liquid at room temperature;

m_f is the mass, expressed in grams, of the empty, dry flask;

V_pyc is the volume, expressed in cubic centimetres, of the pycnometer.

Calculate the apparent density, ρ_p, expressed in grams per cubic centimetre, from Equation (10):

$$\rho_\mathrm{p} = \frac{m_\mathrm{p}\rho_\mathrm{l}}{m_\mathrm{f+l} + m_\mathrm{p} - m_\mathrm{f+l+p}} \tag{10}$$

where

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

$m_\mathrm{f+l}$ is the mass, expressed in grams, of the flask and liquid at room temperature;

$m_\mathrm{f+l+p}$ is the mass, expressed in grams, of flask, liquid and proppant at room temperature;

ρ_l the test liquid density, expressed in grams per cubic centimetre.

Report the apparent density, expressed in grams per cubic centimetre, and the amount of liquid used in the test.

10.5 Absolute density

10.5.1 Description

The preferred method to determine absolute density, ρ_{abs}, is a method that is based on Boyle's Law; the pressure upon filling the sample chamber and then discharging into a second empty chamber allows the computation of the sample solid phase volume. The recommended gas is helium, but nitrogen or dry air is an acceptable alternative.

10.5.2 Equipment

10.5.2.1 **Pycnometer**, with a typical recommended reproducibility to within 0,02 % of the nominal full-scale volume on a clean, dry, thermally-equilibrated sample.

Such instruments are commercially available [7].

10.5.2.2 **Analytical balance**, 0,001 g accuracy.

10.5.2.3 **Desiccator**.

10.5.3 Procedures

10.5.3.1 The volume of proppant is dependent upon the volume of the sample cup. The proppant should be dried at 105 °C (221 °F) to a constant mass and cooled to room temperature in a desiccator. Record the sample mass, m_{s}.

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 a pressure not exceeding 13,8 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 are recommended. The absolute density is calculated by dividing m_{s} by the determined sample volume.

10.5.3.5 Report the value of the absolute density to two decimal places.

11 Proppant crush-resistance test

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 the specified size range. The amount of proppant material crushed at each stress level is measured. Evaluation of test results should provide indications of the stress level where proppant crushing is excessive and the maximum stress to which the proppant material should be subjected.

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7) The AccuPyc 1330™ Pycnometer manufactured by Micromeritics Instrument Corporation is an example of a suitable device available commercially. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of this product.

11.3 Equipment and materials

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

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 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 %, or an independent calibrated load-measuring device should be used when the load is applied to the cell. The stress shall never exceed the target value by more than 5 % (see 11.5.10). Automated load frames are highly recommended.

11.3.2 Cell for proppant crush-resistance test, as described in Figure 7, or equivalent.

The piston length shall be 88,9 mm (3 1/2 in) and the piston diameter shall be 50,8 mm (2 in) and shall have a Rockwell C hardness of 43 or better (Rockwell C 60 preferred). Periodic inspection for internal diameter wear 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 % (–10 % increase in cross-sectional area), the cell shall be replaced;

11.3.3 Testing sieves, pan and lid, see Clause 6 for appropriate sieve numbers (sizes).

11.3.4 Balance, for weighing proppant sample and sieve fractions to 0,1 g precision or better.

11.3.5 Testing sieve shaker, see 6.3.2.

11.3.6 Timer, capable of measuring to ± 5 s.

11.3.7 Sample splitter, metal; see Figure 3.

11.3.8 Metal beaker or **weighing boat**. Plastic, glass or paper containers tend to generate static electrical charge and should be avoided.

11.4 Sample preparation

11.4.1 Using the sample splitter, reduce the sample size to 80 g to120 g.

11.4.2 Refer to Table 1 and select the first and last primary sieve sizes appropriate for the proppant being tested. Prepare the sieve stack consisting only of the first and the second primary sieves and place the sieve stack in the sieve shaker.

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

11.4.4 Properly secure the sieve stack in the shaker and shake for 10 min.

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 second primary sieve is used in the crush test.