INTERNATIONAL STANDARD

ISO 13503-2

First edition
2006-11-01

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
ISO 13503-2:2006(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword v

Introduction vi

1 Scope 1

2 Normative references 1

3 Abbreviations 1

4 Standard proppant sampling procedure 2
4.1 General 2
4.2 Particle segregation 2
4.3 Equipment 2
4.4 Number of required samples — Bulk 4
4.5 Sampling —Bulk material 5
4.6 Sampling — Bagged material 5

5 Sample handling and storage 5
5.1 Sample reduction 5
5.2 Sample splitting 5
5.3 Sample and record retention and storage 5

6 Sieve analysis 6
6.1 Purpose 6
6.2 Description 6
6.3 Equipment and materials 6
6.4 Procedure 6
6.5 Calculation of the mean diameter, median diameter and standard deviation 7
6.6 Sieve calibration 9

7 Proppant sphericity and roundness 11
7.1 Purpose 11
7.2 Description 12
7.3 Apparatus capability 12
7.4 Procedure 12
7.5 Alternate method for determining average sphericity and roundness 13

8 Acid solubility 13
8.1 Purpose 13
8.2 Description 13
8.3 Equipment and materials 14
8.4 Procedure 14

9 Turbidity test 15
9.1 Purpose 15
9.2 Description 16
9.3 Equipment and materials 16
9.4 Equipment calibration 16
9.5 Procedure 16

10 Procedures for determining proppant bulk density, apparent density and absolute density 17
10.1 Purpose 17
10.2 Description 17
10.3 Bulk density 17
10.4 Apparent density 19
10.5 Absolute density 21

11 Proppant crush-resistance test 21
11.1 Purpose 21
11.2 Description 21
11.3 Equipment and materials 22
11.4 Sample preparation 22
11.5 Crush-resistance procedure 23
12 Loss on ignition of resin-coated proppant 25
12.1 Objective 25
12.2 Apparatus and materials 25
12.3 Loss-on-ignition procedure for whole-grain proppant 25
Annex A (informative) **Formazin solution preparation 27**
Bibliography 28

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.

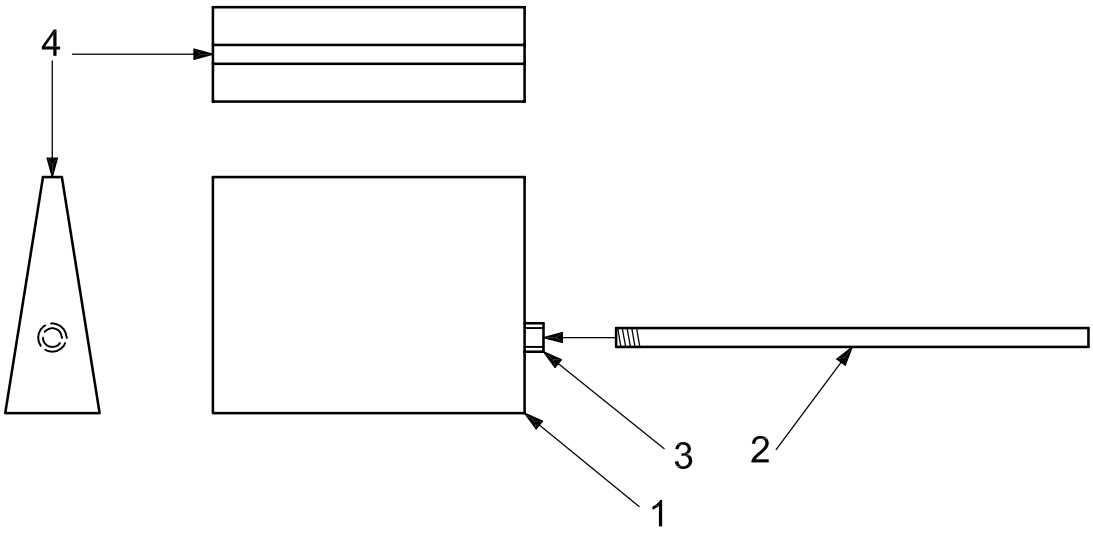
4.3.1 Box sampling device, with a 13 mm (0,50 in) slot opening.

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)

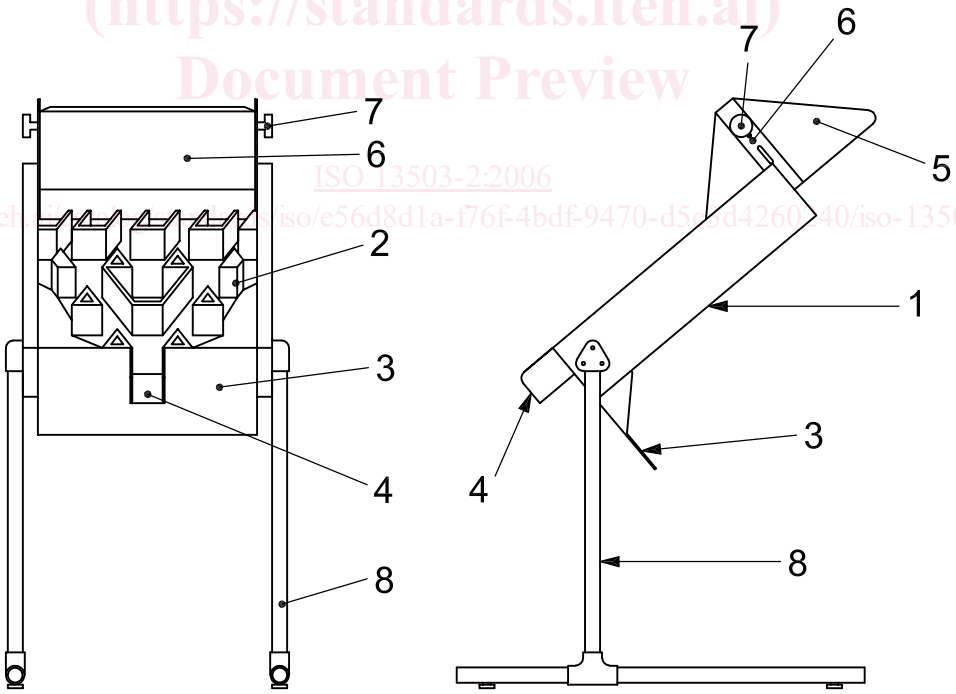


Key

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

Figure 1 — Box sampling device

Dimensions in centimetres (inches)

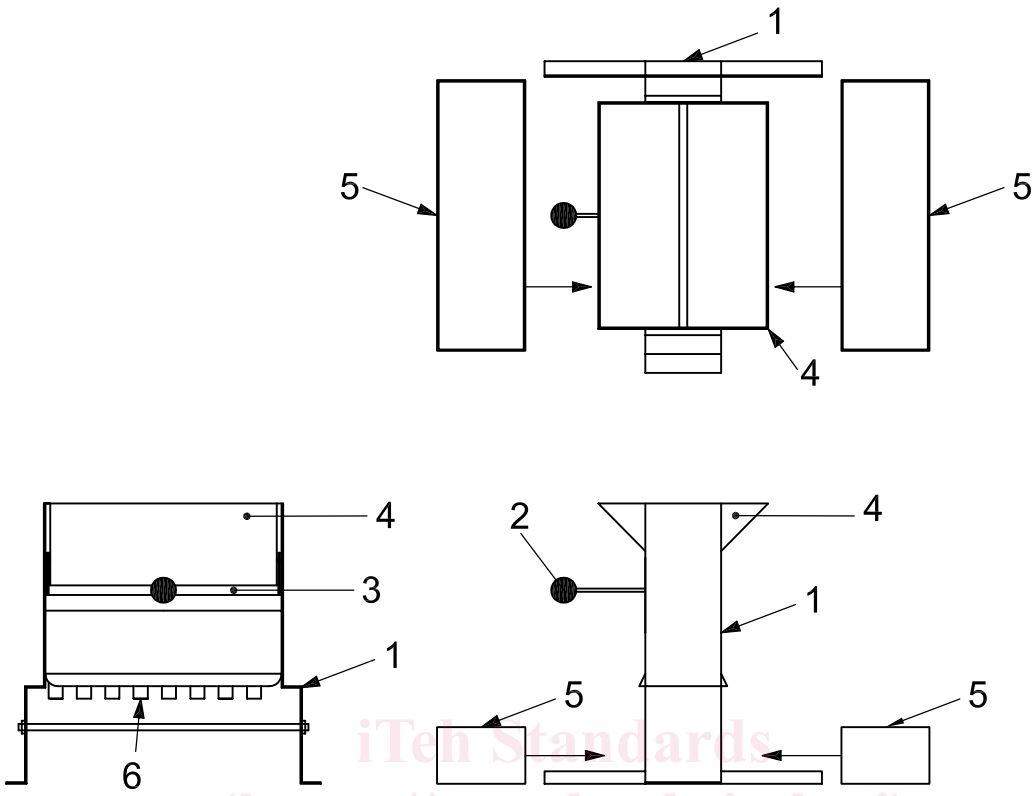


Key

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