

Measuring Conductivity of Proppants

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Introduction

This document is largely based on API Recommended Practice 61, “Recommended Practices for Evaluating Short Term Proppant Pack Conductivity.” Informative references are also included in the bibliography.

The tests and test apparatus herein have been developed to establish standard procedures and conditions for use in evaluating the conductivity of various hydraulic fracture proppant materials under laboratory conditions. This procedure enables users to compare the conductivity characteristics under the specifically described test conditions. The test results can aid users in comparing proppant materials for use in hydraulic fracturing operations.

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

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This document uses a format for numbers which follows the examples given in API *Document Format and Style Manual*, First Edition, June 2007 (Editorial Revision, November 2017). This numbering format is different than that used in API 19D, First 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 (USC) units are included in parentheses for information. The units do not necessarily represent a direct conversion of metric units (SI) to US Customary units, or USC units to SI units. Consideration has been given to the precision of the instrument making the measurement. Consideration has also been given to the nature of the measurement, is it critical or simply informational.

In this document, calibrating an instrument refers to assuring the accuracy of the measurement. Accuracy is the degree of conformity of a measurement of a quantity to its actual or true value. Accuracy is related to precision, or reproducibility, of a measurement. Precision is the degree to which further measurements or calculations will show the same or similar results. Precision is characterized in terms of the standard deviation of the measurement. The results of calculations or a measurement can be accurate, but not precise, precise but not accurate, neither or both. A result is valid if it is both accurate and precise.

Measuring Conductivity of Proppants

1 Scope

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

NOTE The “proppants” mentioned henceforth in this document refer to natural sand, ceramic media, resin-coated proppants, gravel packing media, and other materials used for hydraulic fracturing and gravel-packing operations.

The objective of the document is to provide consistent methodology for testing procedures used to measure performance of hydraulic-fracturing or gravel-packing proppants, or both. The testing procedures in this document are not designed to provide values of proppant conductivity under downhole reservoir conditions. Long-term test data have shown that time, elevated temperatures, fracturing fluid residues, cyclic stress loading, embedment, formation fines and other factors further reduce fracture proppant pack conductivity. Also, this reference test is designed to measure only the frictional energy losses corresponding to laminar flow within a pack. It is recognized that fluid velocity within an actual fracture can be significantly higher than in these laboratory tests and can be dominated by inertial effects.

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 standard (including any amendments) applies.

API Standard 19C, *Measurement of and Specifications for Proppants used in Hydraulic Fracturing and Gravel-packing Operations*

ISO ¹ 3506-1, *Mechanical properties of corrosion-resistant stainless-steel fasteners — Part 1: Bolts, screws, and studs*

3 Terms, Definitions, Symbols, and Abbreviations

3.1 Terms and Definitions

For the purposes of this document, the following terms and definitions apply.

3.1.1

conductivity

Width of the fracture multiplied by the permeability of the proppant pack

3.1.2

laminar flow

Darcy flow

Type of streamlined flow for single-phase fluids in which the fluid moves in parallel layers, or laminae, such that the layers flow smoothly over each other with instabilities being dampened by the viscosity.

3.1.3

permeability (k)

A measure of the ability of a porous medium to allow flow of fluid or gases.

¹ International Organization for Standardization, 8 chemin de Blandonnet, CP401, 1214 Vernier, Switzerland; www.iso.org

3.1.4

platen

The solid surfaces assembled adjacent to (above and below) the proppant pack in the conductivity cell that serves as the fracture faces. In the context of the current document, this material could comprise a quarried sandstone, formation core, or metal slab.

3.2 Symbols

A	is the cross-sectional area of test cell perpendicular to flow, expressed in square centimeters (square inches)
A_c	is the area of the cell, expressed in square millimeters (square inches)
C	is the proppant loading, expressed in kilograms per square meter (pounds per square foot)
k_{SI}	is the proppant pack permeability, expressed in square micrometers
k_{USC}	is the proppant pack permeability, expressed in darcys
$k W_{f-SI}$	is the proppant pack conductivity, expressed in micrometer•meters
$k W_{f-USC}$	is the proppant pack conductivity, expressed in darcy•feet
L	is the length between pressure ports, expressed in centimeters (inches)
M_p	is the mass of proppant to fill the cell, expressed in grams
Q	is the flow rate, expressed in milliliters per second
Q_c	is the test flow rate, expressed in milliliters per minute
W_f	is the proppant pack width, expressed in centimeters (inches)
w	is the cell width, expressed in centimeters (inches)
ΔP	is the pressure drop (pressure upstream minus pressure downstream), expressed in kilopascals (pounds-force per square inch)
μ	is the viscosity of test liquid at test temperature, expressed in millipascal•seconds (centipoise)

3.3 Abbreviations

EN	European standard
ISO	International Organization for Standardization
KCl	potassium chloride (chemical symbol)
NIST	National Institute of Standards and Technology
SAE	SAE International
RTV	room temperature vulcanizing
SS	stainless steel

4 Procedure for Evaluating Proppant Pack Conductivity

4.1 Objective

The objective is to establish a standard test procedure, using a standard apparatus, under standard test conditions to evaluate the conductivity of proppants under semi-steady state conditions and laboratory conditions. This procedure is not intended for use in obtaining values of proppant pack conductivities under downhole reservoir

conditions. The effects of fines, formation hardness, resident fluids, time, and/or other factors are beyond the scope of this procedure.

4.2 Discussion

In this procedure, a closure stress shall be applied across a test cell for 50 h $\pm$ 2 h to allow the proppant sample bed to reach a semi-steady state condition. As the fluid is forced through the proppant bed, the proppant pack width, differential pressure, temperature, and flow rates are measured at each stress level. Proppant pack conductivity parameters and width are measured, and permeability is calculated.

Multiple flow rates are used to verify the performance of the transducers, and to determine laminar (darcy) flow regime at each stress; an average of the data at these flow rates is reported. For highest data accuracy, a minimum pressure drop of 0.014 kPa (0.0020 lbf/in.²) shall be used; otherwise, flow rates should be increased to achieve the minimum pressure drop. Alternatively, for smaller proppants with lower conductivities, measurements shall use a pressure transducer with larger pressure span, to increase accuracy in measurements of higher pressure drop but which will reduce accuracy at smaller pressure drop values. At stipulated flow rates and temperature conditions, no appreciable non-darcy flow or inertial effects are encountered. After completing the rates at a closure stress level in all cells, the closure stress is increased to a new level; 50 h $\pm$ 2 h is allowed for the proppant bed to reach a semi-steady state condition, and multiple flow rates in all cells are introduced to gather data required to determine proppant pack conductivity at this stress level. The procedure is repeated until all desired closure stresses and flow rates have been evaluated. To achieve accurate conductivity measurements, it is essential that single-phase flow occurs.

Test condition parameters, such as test fluid, temperature, loading, platen material and time, at each stress are reported along with conductivity and permeability data. Other conditions can be used to evaluate different characteristics of proppants and, therefore, can be expected to produce differing results.

5 Reagents and Materials

5.1 Test Fluid

The test fluid shall be 2 % w/w (0.274 mol/kg) potassium chloride (KCl) in a deionized or distilled water solution filtered to 7 μ m (0.0003 in.) or smaller. The potassium chloride shall be at least 99 % by mass pure (Chemical Abstracts Service (CAS) No.7447-40-7).

5.2 Platen Material

5.2.1 Stone platens

Sandstone or naturally occurring stone platens shall have dimensions of 176.80 mm to 177.80 mm (6.960 in. to 7.000 in.) in length, 37.70 mm to 38.10 mm (1.485 in. to 1.500 in.) wide, and a minimum of 8.10 mm (0.320 in.) thickness. The ends of the platen shall be rounded to fit into the test cell (see 6.1). Platen parallelism shall be maintained to $\pm$ 0.03 mm ($\pm$ 0.001 in.) of platen length.

The preferred sandstone platen material should comprise Buena Vista sandstone² from Scioto County, Ohio (USA). Any other rock material used for these measurements shall be documented, including the physical properties of the material and place of origin. Representative properties for Buena Vista sandstone are summarized in Table 1.

NOTE Buena Vista sandstone is a homogeneous quartzose fine-grain rock.

² This information is given for the convenience of users of this part of API 19D and does not constitute an endorsement by API of this product.

Table 1—Representative Properties of Buena Vista Sandstone Platen Material

Representative Properties of Buena Vista Sandstone		
Percent Quartz	>78 %	
Percent Clays	<11 %	
Static Young Modulus	GPa 3.30 to 3.45	(10 ⁶ lbf/in. ²) (4.8 to 5.0)
Rockwell Hardness	56 HRE	
Relative Density	2.24	
Flexural Strength	kPa	(lbf/in. ²)
— Parallel to bedding	13,570	(1968)
— Perpendicular to bedding	10,880	(1578)

5.2.2 Stainless Steel 316 Platens (316 SS)

316 SS or equivalent material platens should be used as an alternative to sandstone platen. Alternative platens can produce substantially different results compared with sandstone. For example, 316 SS platens may produce higher conductivity/permeability values due to minimal proppant embedment. When used, test results shall be clearly marked as using a specific grade of 316 SS platens. 316 SS platens, in contact with the proppant bed, shall have dimensions and shape identical to stone platens as stated in 5.2.1 [length: 176.80 mm to 177.80 mm (6.960 in. to 7.000 in.), width: 37.70 mm to 38.10 mm (1.485 in. to 1.500 in.)]. As an alternative, a minimum of 1.40 mm (0.055 in.) thick disposable metal platen should be used in contact with the proppant supported by another 316 SS platen of a minimum of 6.70 mm (0.265 in.) thickness, contingent on the abilities of the 316 SS platen and piston to fit properly into the cell body. The ends of the 316 SS platens shall be rounded to fit into the test cell. Platen parallelism shall be maintained to ± 0.03 mm (± 0.001 in.) of platen length.

In the event of visible surface anomalies, 316 SS platens shall be returned to machine shop for resurfacing/repair to the original surface condition. Platens of 8.90 mm (0.350 in.) thickness should be desired for platens intended to be resurfaced and reused. Visible surface anomalies, such as pitting or machine marks, may be present on either new or reused metal platens and shall be repaired before use of those (new or reused) metal platens.

If any 316 SS equivalent platens were used material properties shall be reported.

5.3 Stainless Steel Shims (Backup Support)

316 SS or equivalent material shims should provide additional support for the stone platens and the seal ring at elevated stress where native stone platens tend to flex and crack or even break. Shims as noted in Figure C.1 shall have dimensions of 176.80 mm to 177.80 mm (6.960 in. to 7.000 in.) in length, 37.70 mm to 38.10 mm (1.485 in. to 1.50 in.) wide, and a minimum of 3.05 mm (0.120 in.) thick. The ends of the 316 SS shims shall be rounded to fit into the test cell. Shim parallelism shall be maintained to ± 0.03 mm (± 0.001 in.) of shim length.

If any 316 SS equivalent shims were used, material properties shall be reported.

6 Equipment

6.1 Conductivity Test Cell

6.1.1 General

The conductivity test cell shall be a linear flow design with a 64.5 cm² (10.0 in.²) proppant and bed area. Figure C.1 illustrates the details of the test cell assembly and an example of how cells can be stacked. The pistons and test chamber(s) shall be constructed of 316 stainless steel (316 SS as specified in ISO 3506-1, Grade A4), nickel-copper alloy, or nickel-chromium-molybdenum-tungsten high temperature corrosion resistant alloy.

6.1.2 Sealing Rings

Test chamber(s) sealing is provided by square rings size 247 [cross section 3.4 mm × 3.4 mm (0.134 in. × 0.134 in., Internal diameter 117.07 mm (4.609 in.)) or equivalent. Testing at all temperatures shall use rings made of higher temperature fluoro-rubber (FKM) such as Viton³.

6.1.3 Filters

Filters for the test cell shall be constructed using nickel-copper alloy wire cloth with an opening of 150 µm [0.0059 in. (100 U.S. mesh)] or equivalent. Nominal particle retention sizes shall be greater than 114 µm (0.0045 in.).

6.2 Hydraulic Load Frame

The hydraulic load frame shall have sufficient capacity to apply stress of 670 kN (150,000 lbf). To confirm uniform stress distribution, the platens shall be parallel to each other. The hydraulic load frame shall be of a four-post guided platen design that minimizes warping that can be transmitted to the test cell. Each post should have a minimum diameter of 64 mm (2.5 in.).

The hydraulic pressurization source shall be capable of holding any desired closure stress [at ±1.0 % or ±350 kPa (±50 lbf/in.²), whichever is greater] for 50 h. The hydraulic load frame shall be capable of loading rate changes of 4450 N/min (1000 lbf/min) or 690 kPa/min (100 lbf/in.²/min) on a 64.5 cm² (10.0 in.²) cell. A calibrated electronic load cell shall be used to calibrate the stress between the hydraulic ram and the opposing platen of the load frame.

6.3 Pack Width Measurement Device(s)

Pack width measurements shall be made at each end of the test cell. A measuring device capable of measuring to 0.03 mm (0.001 in.) accuracy or better shall be used. Figure C.5 shows an example of width slats allowing for the measurement of pack widths.

6.4 Test Fluid Drive System

Pulsation dampening may be accomplished by use of a piston, bladder accumulator or other effective means. Pressure fluctuations during differential pressure and flow rate measurements (for conductivity calculations) shall be maintained at less than 1.0 %. Each laboratory shall determine the technique for pulsation dampening. Large pressure spikes shall be corrected before recording data.

NOTE 1 Some constant-flow-rate pumps (e.g. chromatographic pumps) have been found satisfactory for this application.

NOTE 2 Large pressure spikes can be indicative of pump problems or trapped gas in the flow system.

6.5 Differential Pressure Transducers

Differential pressure transducers may be used with a maximum range of 0 kPa to 6.2 kPa (0 lbf/in.² to 0.9 lbf/in.²) for proppants bigger than 100 mesh and 0 kPa to 65 kPa (0 lbf/in.² to 9.0 lbf/in.²) for 100 mesh proppant. The transducer shall be capable of measuring the differential pressure to ±0.1 % of full scale of the transducer.

NOTE 100 mesh size: 150 µm (0.069 in.)

6.6 Back-Pressure Regulator

The back-pressure regulator shall be capable of maintaining a pressure of 2.0 MPa to 3.5 MPa (300 lbf/in.² to 500 lbf/in.²). The stress applied to the cells shall always be maintained greater than the cell back-pressure. For

³ Viton is an example of a suitable brand of FKM fluoro-rubber available commercially. This information is given for the convenience of users of API 19D and does not constitute an endorsement by API of these products.

example, if the back-pressure is 3.5 MPa (500 lbf/in.²), then the applied stress may be 3.5 MPa (500 lbf/in.²) greater to consider the pressure exerted outward from the pistons.

6.7 Balance

The balance shall be capable of accommodating a minimum capacity of 100 g. Balances shall have two (2) decimal place precision with a minimum accuracy of 0.01 g.

6.8 Oxygen Removal

The conductivity test fluid shall have the oxygen content reduced to simulate reservoir fluids and to minimize corrosion of test equipment. De-oxygenation can be accomplished with a two-reservoir system for the fluid.

The first reservoir that holds fluid for oxygen removal shall be connected to nitrogen gas. Gas bubbled through the fluid at a low rate shall be maintained at a pressure below 100 kPa (15 lbf/in.²). The nitrogen gas supply shall have oxygen content of less than 15 µg/L. To achieve deoxygenation, nitrogen gas shall first pass through an oxygen/moisture trap, such as current commercially available oxygen traps. An equivalent system may be made, which would allow nitrogen to pass through heated copper shavings at 370 °C (700 °F), where the copper reacts with the trace amounts of oxygen in the system forming copper oxide.

An indicating trap, which may include commercially available products, shall be installed after the oxygen-removal process. This indicating trap allows for visual confirmation that oxygen has been removed. When the visual indicating trap is oxygen-saturated, both traps shall be replaced to maintain the efficiency of oxygen removal.

The second reservoir holds the oxygen-free fluid; this is the supply reservoir for the pumping system. All fluids in each reservoir shall be held in sealed, oxygen free-gas pressurized containers to eliminate oxygen contamination from the air.

6.9 Temperature Control

The test cell and proppant pack shall be maintained at the desired temperature ± 1 °C (± 2 °F). The temperature for the test conditions shall be measured in the temperature port of the conductivity cell (see Figure C.1). This temperature shall be used to determine the 2 % KCl solution viscosity using Table A.1 or Table A.2. The thermocouple assembly is split into a temperature control device and a data acquisition system or equivalent. The temperature control devices shall be programmable proportional-integral-derivative (PID) controllers and capable of self-tuning for different temperature conditions and flow rates.

A temperature of 121 °C (250 °F) shall be employed in the test for ceramics and resin-coated proppants and 66 °C (150 °F) for naturally occurring proppants and low temperature resin-coated proppants. To ensure that the test fluid is saturated with silica prior to reaching the cell, the temperature for the silica-saturation vessel (see Annex B) should be 11 °C (20 °F) above testing temperature of 66 °C (150 °F) for naturally occurring proppants and low temperature resin-coated proppants. Further, the temperature shall be 20 °C (35 °F) above testing temperature of 121 °C (250 °F) for resin-coated and ceramic proppants. Care shall be taken to confirm that the fluid arriving to the cell is at the appropriate temperature.

NOTE Tests using other fluids or temperatures can be of value in evaluating proppant pack conductivity.

Test temperature for resin-coated proppant should be chosen based on resin properties, i.e. if working temperature of resin is lower than 121 °C (250 °F), such product should be tested at 66 °C (150 °F).

6.10 Silica Saturation and Monitoring

All tests shall use a silica-saturated solution flowing through the proppant pack to prevent dissolution of sandstone platens and proppant. A high-pressure vessel with a minimum volume of 300 mL and 10 mL/min flow rate capacity, that is equipped with 6.4 mm (0.25 in.) female pipe ends shall be included. Equipment setup shall be in accordance with Annex B.