Petroleum and natural gas industries — Completion fluids and materials —

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

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ICS 75.100

BSi
British Standards

National foreword

This British Standard is the official English language version of EN ISO 13503-5:2006. It is identical with ISO 13503-5:2006.

The UK participation in its preparation was entrusted by Technical Committee PSE/17, Materials and equipment for petroleum, petrochemical and natural gas industries, to Subcommittee PSE/17/-/3, Drilling and completion fluids and well cements, which has the responsibility to:

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Summary of pages

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Petroleum and natural gas industries - Completion fluids and materials - Part 5: Procedures for measuring the long-term conductivity of proppants (ISO 13503-5:2006)

Industries du pétrole et du gaz naturel - Fluides de complétion et matériaux - Partie 5: Modes opératoires pour mesurer la conductivité à long terme des agents de soutènement (ISO 13503-5:2006)

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Foreword

This document (EN ISO 13503-5:2006) has been prepared by Technical Committee ISO/TC 67 "Materials, equipment and offshore structures for petroleum and natural gas industries" in collaboration with Technical Committee CEN/TC 12 "Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2007, and conflicting national standards shall be withdrawn at the latest by January 2007.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of ISO 13503-5:2006 has been approved by CEN as EN ISO 13503-5:2006 without any modifications.

EN ISO 13503-5:2006

INTERNATIONAL STANDARD

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Petroleum and natural gas industries — Completion fluids and materials —

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

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

Partie 5: Modes opératoires pour mesurer la conductivité à long terme des agents de soutènement

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Contents

Page

Foreword

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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 13503-5 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 gravelpack fluid leakoff under static conditions*
- *Part 5: Procedures for measuring the long-term conductivity of proppants*

Introduction

This part of ISO 13503 is largely based on API RP 61[1]. Informative references are also included in the Biblography, References [2] to [15].

The tests and test apparatus herein have been developed to establish standard procedures and conditions for use in evaluating the long-term 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.

This part of ISO 13503 may be used by anyone desiring to do so. Every effort has been made by ISO and API to ensure the accuracy and reliability of the data contained in it. However, ISO and API make no representation, warranty, or guarantee in connection with this part of ISO 13503, and hereby expressly disclaim any liability or responsibility for loss or damage resulting from its use or for the violation of any federal, state, or municipal regulation with which this part of ISO may conflict.

In this part of ISO 13503, where practical, U.S. customary units are included in parentheses for information.

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Petroleum and natural gas industries — Completion fluids and materials —

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

CAUTION — The testing procedures in this part of ISO 13503 are not designed to provide absolute 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.

1 Scope

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

NOTE The “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 consistent methodology for testing performed on hydraulic-fracturing and/or gravel-packing proppants. It is not intended for use in obtaining absolute values of proppant pack conductivities under downhole reservoir conditions.

2 Normative reference

The following referenced document is 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.

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

3 Terms and definitions

3.1
conductivity
width of the fracture multiplied by the permeability of the proppant pack

3.2
laminar 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

3.3
Ohio sandstone
fine-grained sandstone found in the United States from the Scioto Formation in southern Ohio

3.4
permeability
a measure of the ability of media to transmit fluid through pore spaces

4 Abbreviations

API American Petroleum Institute

ASTM American Society for Testing and Materials

RTV Room temperature vulcanizing

ANSI American National Standards Institute

PID Proportional-integral device

5 Procedures for evaluating long-term proppant pack conductivity

5.1 Objective

The objective is to establish a standard test procedure, using a standard apparatus, under standard test conditions to evaluate the long-term conductivity of proppants under laboratory conditions. This procedure is used to evaluate the conductivity of proppants under laboratory conditions but is not intended for use in obtaining absolute 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.

5.2 Discussion

In this part of ISO 13503 procedure, a closure stress is applied across a test unit for 50 h ± 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 permeability and conductivity are calculated.

Multiple flow rates are used to verify the performance of the transducers, and to determine darcy flow regime at each stress; an average of the data at these flow rates is reported. A minimum pressure drop of 0,01 kPa (0,002 0 psi) is recommended; otherwise, flow rates shall be increased. 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 ± 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, sandstone and time, at each stress shall be reported along with long-term conductivity and permeability data. Other conditions can be used to evaluate different characteristics of proppants and, therefore, can be expected to produce differing results.

6 Reagents and materials

6.1 Test fluid

The test fluid is 2 % by mass potassium chloride (KCl) in a deionized or distilled-water solution filtered to at least 7 µm. The potassium chloride shall be at least 99,0 % by mass pure.

6.2 Sandstone

Ohio sandstone cores should have dimensions of 17,70 cm to 17,78 cm (6,96 in to 7,00 in) in length, 3,71 cm to 3,81 cm (1,46 in to 1,50 in) wide, and a minimum of 0,9 cm (0,35 in) thick. The ends of the sandstone cores shall be rounded to fit into the test unit (see 7.1). Parallel thickness shall be maintained within ± 0,008 cm (± 0,003 in).

7 Long-term conductivity test apparatus

7.1 Test unit

The test unit shall be a linear flow design with a 64,5 cm^2 (10 in^2) proppant and bed area. Figure C.1 illustrates the details of the test unit and an example of how cells can be stacked. The pistons and test chamber(s) shall be constructed of 316 stainless steel (e.g. ISO 3506-1, Grade A4), Monel[1)] or Hastalloy material. Filters for the test unit may be constructed using Monel wire cloth with an opening of 150 µm or equivalent (100 US mesh). Nominal particle retention sizes are greater than 114 µm.

7.2 Hydraulic load frame

The hydraulic load frame shall have sufficient capacity to develop 667 kN (150 000 lbf). To ensure uniform stress distribution, the platens shall be parallel to each other. It is recommended that the hydraulic load frame be of a four-post design that minimizes warping that can be transmitted to the test cell. Each post should have a minimum diameter of 6,35 cm (2,5 in).

The hydraulic pressurization source shall be capable of holding any desired closure stress [± 1,0 % or 345 kPa (50 psi), whichever is greater] for 50 h. The hydraulic load frame shall be capable of loading rate changes of 4 448 N/min (1 000 lbf/min) or 690 kPa/min (100 psi/min) on a 64,5 cm^2 (10 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.

7.3 Pack width measurement device(s)

Pack width measurements shall be made at each end of the test unit. A measuring device capable of measuring to 0,002 5 cm (0,001 in) accuracy or better shall be used. Figure C.4 shows an example of width slats allowing for the measurement of pack widths.

7.4 Test fluid drive system

Some constant-flow-rate pumps (e.g. chromatographic pumps) have been found satisfactory for this application. Pulsation dampening can be necessary and can 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 best technique for pulsation dampening. Large pressure spikes can be indicative of pump problems or trapped gas in the flow system and shall be corrected before recording data.

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1) Monel and Hastalloy are examples of suitable products 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.

7.5 Differential pressure transducers

Differential pressure transducers with a range of 0 kPa to 7 kPa (0 psi to 1,0 psi) are satisfactory. The transducer shall be capable of measuring the differential pressure to ± 0,1 % of full scale.

7.6 Back-pressure regulators

The back-pressure regulator shall be capable of maintaining a pressure of 2,07 MPa to 3,45 MPa (300 psi to 500 psi). The stress applied to the cells shall take into account the back-pressure. For example, if the back-pressure is 3,45 MPa (500 psi), then the applied stress shall be 3,45 MPa (500 psi) greater to take into account the pressure exerted outward from the pistons.

7.7 Balance

The balance shall be capable of accommodating a minimum capacity of 100 g with a precision greater than 0,01 g.

7.8 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 holds fluid for oxygen removal. This is connected to nitrogen gas that is bubbled through the fluid at low pressure below 103 kPa (15 psi) and at low rate. The nitrogen supply is first passed through an oxygen/moisture trap such as Agilent Model OT3-4[2)] that has an efficiency to remove oxygen to less than 15 µg/l. An equivalent system can be made; this system allows nitrogen to pass through heated copper shavings at 370 °C (698 °F), where the copper reacts with the trace amounts of oxygen in the system forming copper oxide. An indicating trap, such as the oxygen trap by Chrom Tech, Inc. part # 10T-4-HP[3)], after the oxygen-removal process 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 are held in sealed, inert-gas pressurized containers to eliminate oxygen contamination from the air.

7.9 Temperature control

The test cell and proppant pack shall be maintained at the desired temperature ± 1 °C (± 3 °F). The temperature for the test conditions is measured in the temperature port of the conductivity cell (Figure C.1). This temperature is used to determine the fluid viscosity from Table C.1. The thermocouple assembly is split into a temperature-control device and a data-acquisition system or equivalent. The temperature control devices shall be programmable PID controllers and capable of self-tuning for different temperature conditions and flow rates.

A temperature of 121 °C (250 °F) is employed in the test for ceramics and resin-coated proppants and 66 °C (150 °F) for naturally occurring sands. 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 sands. Sand 20 °C (35 °F) above 121 °C (250 °F) is used for resin-coated and ceramic proppants to ensure that the fluid is saturated with silica prior to reaching the cell. Care shall be taken to ensure that the fluid arriving to the cell is at the appropriate temperature. Tests using other fluids or temperatures can be of value in evaluating proppant pack conductivity.

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2) Agilent Model OT3-4 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.

3) Chrom Tech, Inc. part # 10T-4-HP 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.

7.10 Silica saturation and monitoring

It is critical to have a silica-saturated solution flowing through the proppant pack to prevent dissolution of the Ohio sandstone and proppant. To achieve this, a high-pressure cylinder with a minimum volume of 300 ml per 10 ml/min flow rate capacity, such as a Whitey sample cylinder 316L-HDF4[4)], or equivalent equipped with 0,635 cm (0,25 in) female pipe ends is needed. For equipment setup, see Annex B.

8 Equipment calibration

8.1 Pressure indicators and flow rates

Pressure indicators in the test fluid-flow stream with back-pressure applied shall be calibrated initially and rechecked before each test. Constant-flow-rate pumps shall be tested at several flow rates with back-pressure applied with suitable flow meters, or accurate balance, containers and timing device (stop watch). High- and low-pressure transducers shall be zeroed before each run. Use only that portion of the transducer range that is repeatable and linear.

8.2 Zero pack width measurement

8.2.1 Purpose

To accurately measure the width of the proppant pack, the variations in sandstone thickness, the compressibility of sandstone and the compression and thermal expansion of the metal shall be taken into account.

8.2.2 Procedure

8.2.2.1 Using callipers, measure and record the thickness of the cores and metal shims. Mark the width of the core on the face of the core with a pencil. Two cores are placed in each cell. Match the cores so that the combined thickness of the ends of the cores is the same. Cores that measure more than 0,008 cm (0,003 in) from parallel shall not be used. If the bottom core is different from end to end, then the top core shall offset this difference, so the total core thickness at each end is identical.

8.2.2.2 A width adjustment factor or zero pack width shall be calculated at each closure stress and at temperature to be tested for each cell and for each lot of Ohio sandstone and square rings. Measure the vertical dimension of the complete test unit [± 0,002 5 cm (± 0,001 in)] equipped with pistons, square rings, shims and sandstone cores, but without proppant, at each test closure stress level and temperature where the proppant will be tested. For each test, measure an initial zero width by measuring the vertical dimension of the pistons, shims and sandstone cores. This value is subtracted from the measured equipment and proppant values to obtain the actual width of the proppant pack.

8.2.2.2.3 Pistons for the baseline cell(s) shall be marked in the order in which they are stacked. Place the two matched sandstone cores in the cell and, if applicable, continue stacking the cells as in Figure C.1.

8.2.2.2.4 Heat the cells to the temperature at which the test will be run. Ramp closure stress at a rate of 689 kPa/min (100 psi/min).

8.2.2.2.5 Using telescoping gauges and digital callipers or equivalent, measure the piston from width slat to the bottom plate and from width slat to the top press plate or to the other width slat. All measurements shall be taken twice and both numbers shall be within ± 0,005 0 cm. Make another measurement 30 min after having made the first reading. Continue making measurements until the system reaches steady-state, e.g. the

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4) Whitey sample cylinder of 316L-HDF4 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.

measurements are within 1 % of each other. A minimum of three measurements shall be made. The last measurement shall be recorded. This procedure takes into account the compression of the sandstone cores and the expansion of metal at pressure and temperature. These values are used when calculating proppant pack widths (see Clause 12). Continue measurements at stress intervals indicated (see Clause 12) until maximum stress is reached.

8.3 Determination of cell width

Measure the inside of the cell at three places, two beside the high and low pressure ports and the third beside the middle port, using telescoping gauges and digital callipers. The three values are averaged. To determine the amount of proppant needed, multiply the average cell width by the amount of proppant desired divided by 38,1 mm. The following example is for a 9,76 kg/m^2 (2,00 lb/ft^2) loading (see 10.2.4).

EXAMPLE (38,35 mm + 38,40 mm + 38,37 mm)/3 = 38,37 mm.

A two pound per square foot loading requires (63,00 g/38,10) × 38,37 = 63,44 g of proppant.

8.4 Hydraulic load frame

Calibration of the load cell shall be done at a minimum annually or when long term conductivity results are questionable. This type of device is preferred over use of hydraulic pressure gauges as a method of determining closure stress applied to the test cell. In some cases, a load cell is part of the system and must be calibrated by an external source.

9 Leak tests

9.1 Hydraulic load frame

The hydraulic system, i.e. lines, fittings and pumps, shall be tested initially and periodically thereafter to ensure there are no leaks. This may be accomplished by placing an appropriate block of high strength material having at least 64,5 cm^2 (10,0 in^2) surface area between the platens at maximum load; shut in and observe to see if the pressure or load change is greater than ± 2 % of maximum reading during a 30 min period. If the pressure or load varies significantly, inspect all lines and fittings. If no line leaks are evident, there might be an internal leak in the control valve or the hydraulic ram.

9.2 Test fluid system

The initial complete test fluid system consisting of pump, lines, fittings and conductivity test unit shall be checked for leaks. To conduct a leak test, the conductivity test unit shall contain at least a monolayer of proppant material.

NOTE With no proppant between the platens, neither the square seal rings nor the downstream equipment can be tested.

Apply a closure stress of greater than 3,45 MPa (500 psi) to the conductivity unit and flow fluid through the system with a back-pressure 2,07 MPa to 3,45 MPa (300 psi to 500 psi). Close in the system and the pressure should not change more than 0,1 kPa (0,01 psi) in 5 min. Inspect all lines and fittings.

10 Procedure for loading the cells

10.1 Preparation of the test unit

10.1.1 Selecting the core

See 8.2.2

10.1.2 Presetting sandstone cores

10.1.2.1 After selecting the cores, apply transparent tape to both top and bottom of the core to prevent sealant from adhering. Use a sharp knife to trim all excess tape. Place transparent tape over all portholes inside the cells and the top of the bottom piston. Record the average width of the sandstone core.

10.1.2.2 With a spatula, apply a thin layer of high-temperature room temperature vulcanizing (RTV) silicone adhesive sealer around the sides of the cores. Allow the RTV to cure.

10.1.2.3 An alternative method of preparing the cores is to place the cores in the test unit. Level the bottom piston within 0,13 mm (0,005 in) from end to end and tighten the set screws. Lightly spray the inside of the cell with a silicone lubricant. Place a core that has been marked and taped in the cell. Up to four cores can be stacked in the cell at one time, as long as RTV is applied around the edges of each core before placement in the cell or equivalent mould device. Allow the RTV to cure.

10.1.2.4 Place the top piston in the conductivity cell. Place the cell in the press and apply between 0,3 MPa (50 psi) and 1 MPa (150 psi) closure. Attach the heat strips, and heat to 66 °C (150 °F) for one hour. Remove core slabs. Trim all excess RTV off the face of the preset core and be certain there are no chips or cracks in the core.

NOTE If no heat is applied, the RTV cures in about 24 h.

10.2 Cell setup

10.2.1 Setting bottom piston

The cells are stacked in the same order in which the zero pack widths were measured (see 8.2.2). Blocks can be used to hold the cell in place so the sandstone core is approximately 0,02 mm (0,000 8 in) or just below the differential pressure ports. This can be achieved by placing one metal shim and sandstone core with no RTV on piston in the cell. When the height of the piston is approximately in the right position, tighten the set screws to secure the position of the cell, and remove the shim and sandstone core to install the square ring. Protect the integrity of the square ring.

10.2.2 Setting the bottom core

Measure the metal shim and record its thickness. Differences between the shim thicknesses from 8.2.2.1 shall be taken in account. Place the shim in the bottom of the cell. Apply a thin film of RTV around the edge on the selected core (see 8.2.2.1). With a spatula, smooth the RTV to even the surface without getting RTV on the faces of the core. Remove the bottom tape from the core and slide the core into the cell until reaching the shim. Apply RTV around the edge of the core-to-cell interface and push RTV into the crack using a cotton swab. Remove the excess RTV and top tape.

10.2.3 Placement of screens

Screens are necessary to keep solids from flowing out of the proppant pack or occluding the ports. Place screens of 150 µm (100 US mesh) Monel or equivalent in all ports, including entry and exit ports and differential-pressure ports. Screens shall be replaced after every run as they can become plugged with crushed proppant.

10.2.4 Calculating the quantity of proppant

Conductivity may be tested on a volume equivalent to 0,64 cm (0,25 in) unstressed pack width or on a mass per unit cell surface area, such as 9,76 kg/m^2 (2,00 lbm/ft^2).

Calculate the desired amount of proppant material using one of the example calculations described below:

a) Mass per unit area, expressed in kilograms per square metre:

Load the desired amount of proppant, which can be calculated as given in Equation (1):

$$M_p = 6{,}452\ C \quad (1)$$

where

M_p is the proppant mass, expressed in grams;

C is the proppant loading, expressed in kilograms per square metre.

$6{,}452 = 0{,}006\ 452\ m^2 \times 1\ 000$ g/kg, where $0{,}006\ 452\ m^2$ is the surface area of the cell. The exact amount of proppant varies depending upon your cell width (see 8.3).

The unstressed proppant pack width can be approximated as given in Equation (2):

$$W_f = 0{,}100\ C/\rho \quad (2)$$

where

W_f is the proppant pack width, expressed in centimetres;

C is the proppant loading, expressed in kilograms per square metre;

ρ is the proppant bulk density, expressed in grams per cubic centimetre[16].

b) Unstressed proppant pack width equal to 6,35 mm (0,25 in)

Load the test cell with $41{,}0 \pm 0{,}1\ cm^3$ of proppant material. The approximate mass of the required proppant material can be calculated as given in Equation (3):

$$M_P = 41{,}0\ \rho \quad (3)$$

where

M_P is the proppant mass, expressed in grams;

ρ is the proppant bulk density, expressed in grams per cubic centimetre[16].

41,0 is $64{,}52\ cm^2$ ($10{,}0\ in^2$) times 0,635 cm (0,25 in) pack width. The exact amount of proppant varies depending upon your cell width (see 8.3).

10.2.5 Loading proppant in the cell(s)

10.2.5.1 Weigh a representative sample based on one of the above calculations.

10.2.5.2 Split the sample into four units. Pour one-forth of the sample as evenly as possible into the cell. Continue this procedure until all four split samples are added.

10.2.5.3 Level the proppant layer in the test unit with a levelling device (see Figure C.6) by making progressively deeper passes to level the proppant in the cell. The proppant shall not be packed by vibration or tamping, as this can cause segregation of material. Make certain that the proppant is level against the walls of the cell.

10.2.6 Setting the top core

10.2.6.1 Apply a thin film of RTV around the edge of the preset core (see 10.1.2). With a spatula (or equivalent), smooth the RTV to even the surface without getting RTV on the faces of the core.

10.2.6.2 Remove the bottom tape from the core and slide the core into the cell evenly. Apply RTV around the edge of the core-to-cell interface and push RTV into the crack using a cotton swab.

10.2.6.3 Measure, with digital callipers, the depth of the core at all four corners, confirming that core is level within 0,1 mm (0,004 in) and making any adjustments necessary. Remove tape from the top of core.

10.2.7 Stacking the cells

More than one cell can be placed in a press (see Figure C.1). Blocks and shims can be used to adjust the height of the cell to within 0,1 mm (0,004 in). Use the same procedure for loading the cells as described in 10.2.5. Tighten set screws.

11 Loading cell(s) in the press

11.1 Place the test unit between the platens of the load frame.

11.2 Apply 345 kPa (50 psi) to the stack of cells. A square angle is used to verify that the stack is perpendicular to the platens. Make necessary adjustments to ensure stack is perpendicular to the platens. Increase pressure from 345 kPa (50 psi) to 3,45 MPa (500 psi) at a rate of approximately 690 kPa/min (100 psi/min).

11.3 Begin flow through the cells. Check for leaks around the pistons, and at connections. If leakage around the pistons is observed, the test shall be terminated and the test unit reloaded with new material. Check uniformity of the proppant pack by measuring the pack bed width at each end of the test unit. If there is a difference of 5 % or more between these width measurements, the test shall be terminated and the cell reloaded with new material.

11.4 Remove air from the cells and transducer lines by flushing the cells and bleeding the lines. Flush the lines for at least 1 min after no air bubbles are visible. Zero the transducers according to manufacturer's specification with no flow.

12 Acquiring data

Initial absolute stress of 6,89 MPa (1 kpsi) is applied for a minimum of 12 h and a maximum of 24 h at required temperature (see 7.9). The back-pressure shall be maintained between 2,07 MPa (300 psi) and 3,45 MPa (500 psi). This value shall be taken into account when determining the stress being applied. After the initial stress of 6,89 MPa (1 kpsi) and time is achieved, raise the stress to 13,79 MPa (2 kpsi). Stresses applied to the proppant pack after the initial stress shall be held for 50 h ± 2 h; any time held less than 48 h at stress shall not be considered long-term conductivity. Stresses shall be increased in 13,79 MPa (2 kpsi) increments thereafter. All ramp rates shall be at 689 kPa/min ± 34 kPa/min (100 psi/min ± 5 psi/min).

Conductivity shall be measured at 13,79 MPa; 27,58 MPa and 41,37 MPa (2 kpsi, 4 kpsi and 6 kpsi) for naturally occurring sands. For ceramic and resin-coated proppants, the conductivity shall be measured at 13,79 MPa; 27,58 MPa; 41,37 MPa; 55,16 MPa and 68,95 MPa (2 kpsi, 4 kpsi, 6 kpsi, 8 kpsi and 10 kpsi). Additional stresses are optional.

Test flow rates are determined according to the pressure drop between the pressure ports. The first flow rate shall be 2 ml/min or a minimum of 0,01 kPa (0,002 psi). To ensure the data are within statistical limits, at least five data points shall be taken and an average permeability (see Clause 13) shall be reported over a range of 2 ml/min to 4 ml/min or at least 0,01 kPa to 0,03 kPa (0,002 psi to 0,004 psi). Report stresses.

Pack widths shall be measured at each stress and calculated to subtract out the compression of the sandstone cores and the expansion of the metal (see 8.2). Before each measurement, the differential pressure transducers shall be zeroed.

13 Calculation of permeability and conductivity

13.1 Equations (4) to (8) shall be used to calculate the permeability of proppant packs to liquid under laminar (darcy) flow conditions:

$$k = \mu QL / [100A(\Delta P)] \quad \text{(expressed in SI units)} \tag{4}$$

$$k = \mu QL / [A(\Delta P)] \quad \text{(expressed in US customary units)} \tag{5}$$

where

k is the proppant pack permeability, expressed in micron square metre (darcy);

μ is the viscosity of test liquid at test temperature, expressed in centipoise;

Q is the flow rate, expressed in cubic centimetres per second;

L is the length between pressure ports, expressed in centimetres;

A is the cross-sectional area of test unit perpendicular to flow, expressed in square centimetres;

ΔP is the pressure drop (pressure upstream minus pressure downstream), expressed in kilopascals (atmospheres).

For convenience, the conversion factors can be found in Annex A.

When the cross-sectional shape of the proppant bed is rectangular, as it is in a fracture, then the cross-sectional area can be given by Equation (6):

$$A = w \cdot W_f \tag{6}$$

where

A is the cross-sectional area perpendicular to flow, expressed in square centimetres;

w is the cell width, expressed in centimetres;

W_f is the pack width, expressed in centimetres.

Equations (4) and (5) can be rewritten so that either the permeability or the conductivity of the proppant pack can be calculated.

To calculate proppant pack permeability, use Equations (7) and (8):

$$k = \mu QL / [100w(\Delta P)W_f] \quad \text{(expressed in SI units)} \tag{7}$$

$$k = \mu QL / [w(\Delta P)W_f] \quad \text{(expressed in US customary units)} \tag{8}$$

13.2 To calculate proppant pack conductivity, use Equations (9) and (10):

$$kW_f = \mu QL / [100w(\Delta P)] \quad \text{(expressed in SI units)} \tag{9}$$

$$kW_f = \mu QL / [w(\Delta P)] \quad \text{(expressed in US customary units)} \tag{10}$$

10

13.3 The information and simplified equations in 13.3 a) and b) can be used when using constants (see Note below)

a) For proppant pack conductivity:

$$kW_f = 5{,}554\ \mu\ QL/(\Delta P) \qquad \text{(expressed in SI units)} \qquad (11)$$

$$kW_f = 26{,}78\mu\ Q/(\Delta P) \qquad \text{(expressed in US customary units)} \qquad (12)$$

where

kW_f is the proppant pack conductivity, expressed in micron square metre centimetre (millidarcy feet);

μ is the test liquid viscosity at test temperature, expressed in centipoise (refer to Table C.1);

Q is the flow rate, expressed in cubic centimetres per minute;

ΔP is the pressure drop (pressure upstream minus pressure downstream), expressed in kilopascals (pounds per square inch);

L is the length between pressure ports, expressed in centimetres.

b) For proppant pack permeability:

$$k = 100\ \mu\ Q/\left[\left[w(\Delta P)W_f\right]\right] \qquad \text{(expressed in SI Units)} \qquad (13)$$

$$k = 321{,}4\mu\ Q/\left[\left[(\Delta P)W_f\right]\right] \qquad \text{(expressed in US customary units)} \qquad (14)$$

where

k is the proppant pack permeability, expressed in micron square metres (millidarcy);

W_f is the pack width, expressed in centimetres (inches);

ΔP is the pressure drop (pressure upstream minus pressure downstream), expressed in kilopascals (pounds per square inch);

w is the cell width, expressed in centimetres.

NOTE The following dimensions were used in calculating the constants shown in Equations (11) through (14)

– test unit width, w = 3,81 cm (1,5 in);
– actual cell width is adjusted according to 8.3;
– length between pressure ports, L = 12,70 cm (5,000 in).

14 Data reporting

Data that are reported shall list have all parameters, such as sandstone type, temperature, time, test fluid, conductivity and permeability at each stress level, bulk density, sieve distribution, specific gravity and/or apparent specific gravity and proppant concentration.

Annex A
(informative)

Conversion factors

NOTE See Reference [17].

1 ft = 0,304 8 m

1 inch = 2,54 cm

1 darcy = 1 000 md = 0,986 9 μm^2

1 lbm = 453,6 g

1 lbf = 4,448 N

1 psi = 6,895 kPa

1 atm = 14,7 psi = 101,3 kPa

1 ml = 1 000 cm^3

°F = (1,80 × °C) + 32

1 cP = 1 mPa·s

Annex B
(normative)

Silica-saturation vessel setup

B.1 Background

Flowing fluid through proppant packs between sandstone core faces can result in silica dissolution and subsequently cause an abnormal failure of grains or increase embedment under closure stresses. For this reason and to mimic formation fluid, the fluid shall be saturated with silica to prevent degradation of the proppant or core material. The solubility of silica in water is primarily a function of temperature and pH, with ionic strength and pressure being of secondary importance.

B.2 Apparatus

B.2.1 High-pressure cylinder or silica-saturation vessel, with a minimum volume of 300 ml per 10 ml/min flow rate.

B.2.2 Monel or 316 stainless steel 150 µm opening (100 US mesh) screen, to place at entrance and exit of fittings of cylinder to prevent silica sand movement.

B.2.3 20/40 mesh or mixture of 50 % each 20/40 mesh and 12/20 mesh silica sand, washed, 50 ml.

B.2.4 70/140 mesh silica sand, washed and dried, 250 ml.

70/140 mesh silica sand that has a greater than 99,7 % silica content and less than 0,05 % iron content has been found satisfactory for this application.

B.2.5 Jacket heater, thermostatically controlled, with temperature control limits of ± 2 °C, that can surround the cylinder.

The temperature of the silica saturation vessel shall be 11 °C (20 °F) above testing temperature of 66 °C (150 °F) for naturally occurring sands and 20 °C (35 °F) above 121 °C (250 °F) for resin-coated and ceramic proppants to ensure that the fluid is saturated with silica prior to reaching the cell. Care shall be taken to ensure that the fluid arriving to the cell is at the appropriate temperature.

B.2.6 In-line stainless steel filter, containing a 7 µm screen to prevent solids from travelling into the test cell from the silica column.

B.3 Procedure

The pH of the fluid shall be adjusted between 6,4 to 6,8 with hydrochloric acid or potassium hydroxide to simulate reservoir fluids and to lower the rate of dissolution of silica from sand. The silica saturation column shall be placed inline prior to the entrance to the test cell. A maximum of two test cells shall be run from one sand column and the intermittent flow rate through the sand column shall not exceed 11 ml/min. A continuous flow rate shall not exceed 4 ml/min.

Place a screen in the fitting and attach the fitting to the bottom of the cylinder. Place a 25 ml layer of washed 20/40 mesh silica sand or mixture of 50 % each 20/40 mesh and 12/20 mesh silica sand into the column to prevent the 70/140 mesh from flowing out. Add approximately 250 ml of washed and dried 70/140 mesh silica sand on top of the 20/40 mesh silica sand or 50 % each 20/40 mesh and 12/20 mesh silica sand mixture. Vibrate the cylinder for a few seconds to pack the sand. Then add the rest of the 20/40 mesh silica sand or 50 % each 20/40 mesh and 12/20 mesh silica sand mixture or until the cylinder is full. Place the screen in the fittings, and attach fitting to cylinder.

Place the silica saturation vessel in line prior to the cells. Place the jacket on the cylinder. Monitoring of the levels of silica saturation can be accomplished by sampling the fluid media at three points:

– prior to the silica saturation column;

– prior to the entrance of the test cell;

– at the exit of the test cell.

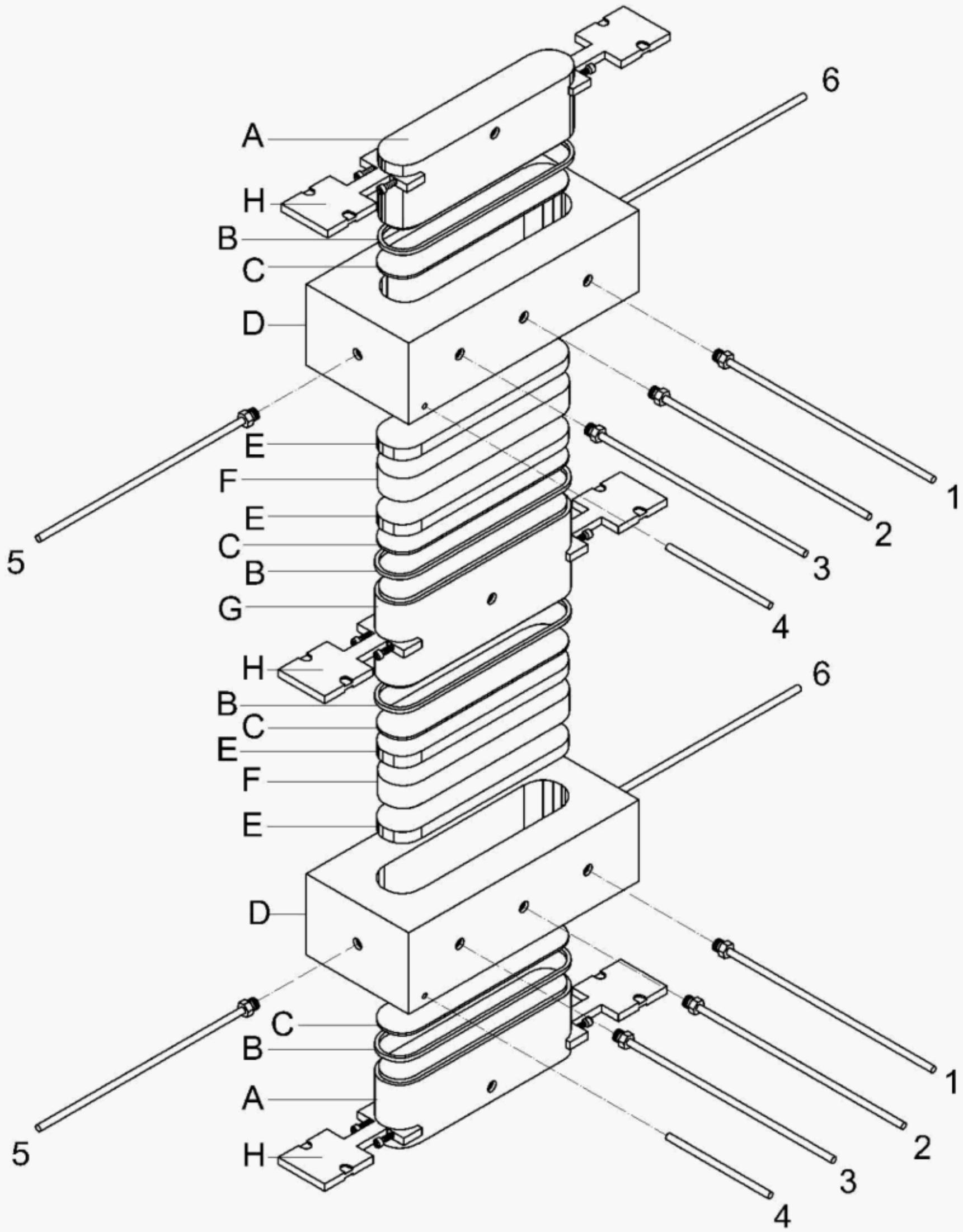
The silica content has been found to stay within the recommended concentration provided the pH, temperatures and flow rates are regulated. This allows running the test without continuous monitoring.

The collected samples are evaluated by an atomic absorption unit or by a wet-chemistry method such as the silicomolybdate colorimetric method to determine silica at the level of milligrams per litre. An increase in silica of 2 mg/l between the silica column and the test cell exit is an acceptable saturation limit.

Annex C
(informative)

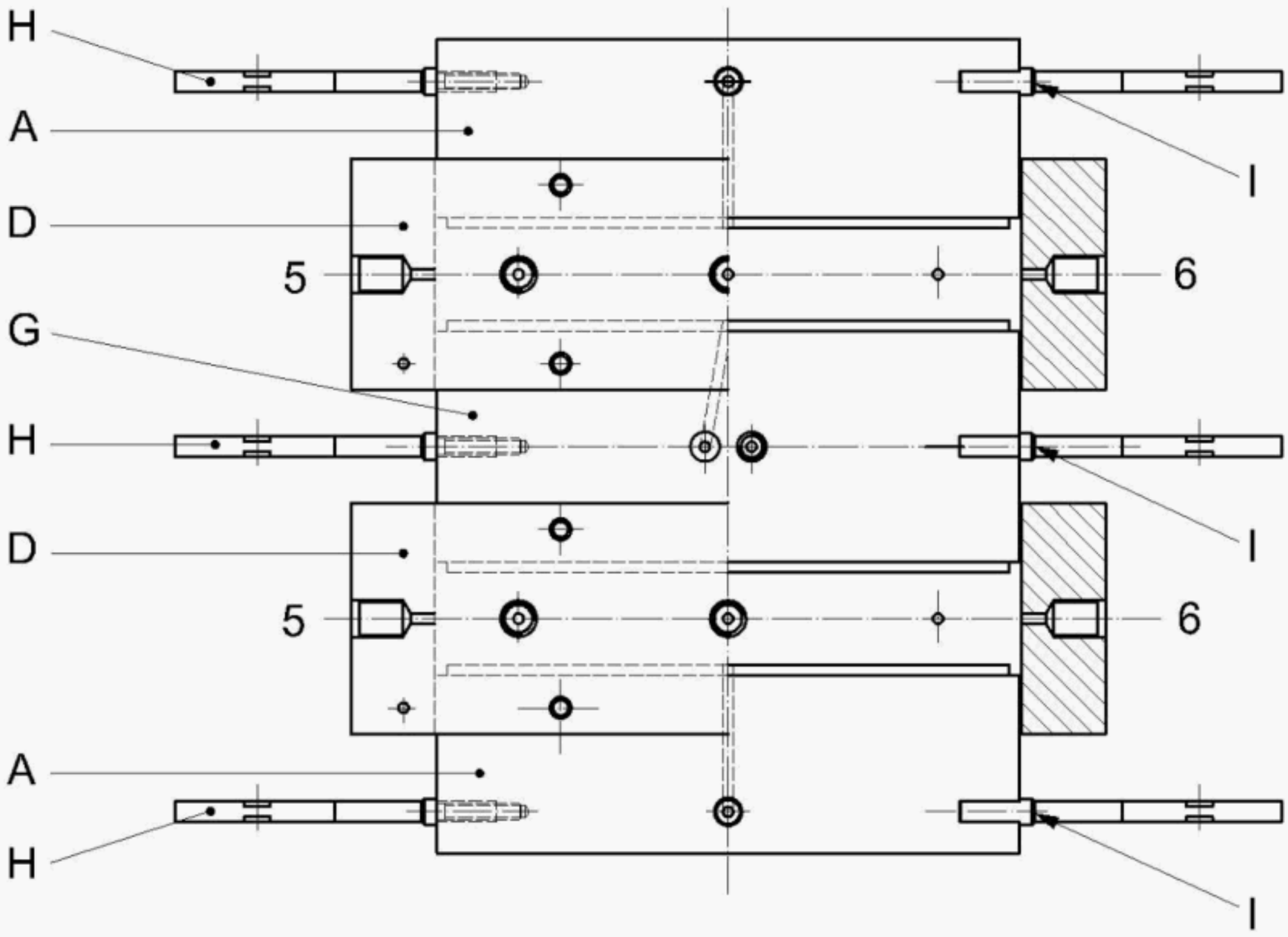
Figures

Dimensions in millimetres



a) Exploded view

Figure C.1 — Conductivity cell stack (see also Figures C.2 to C.5)



b) Assembled side view

Key

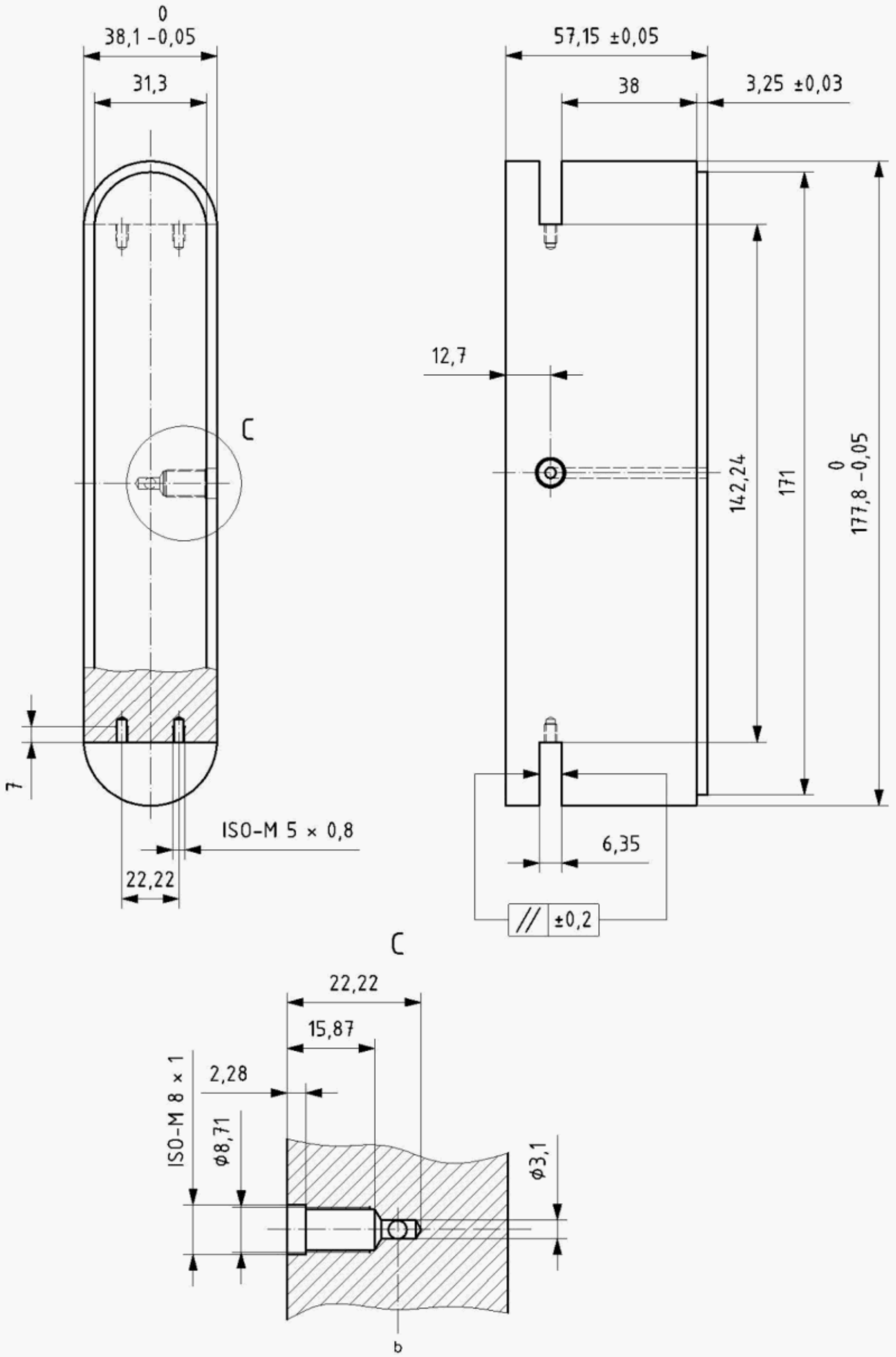
1 low pressure port
2 middle port
3 high pressure port
4 temperature control sensor
5 inlet of cell
6 outlet of cell

A upper and lower piston
B square ring seal
C metal shim
D cell body
E Ohio sandstone
F proppant
G central piston
H width slat
I set screws

NOTE The figures are not to scale.

Figure C.1 (*continued*)

Dimensions in millimetres



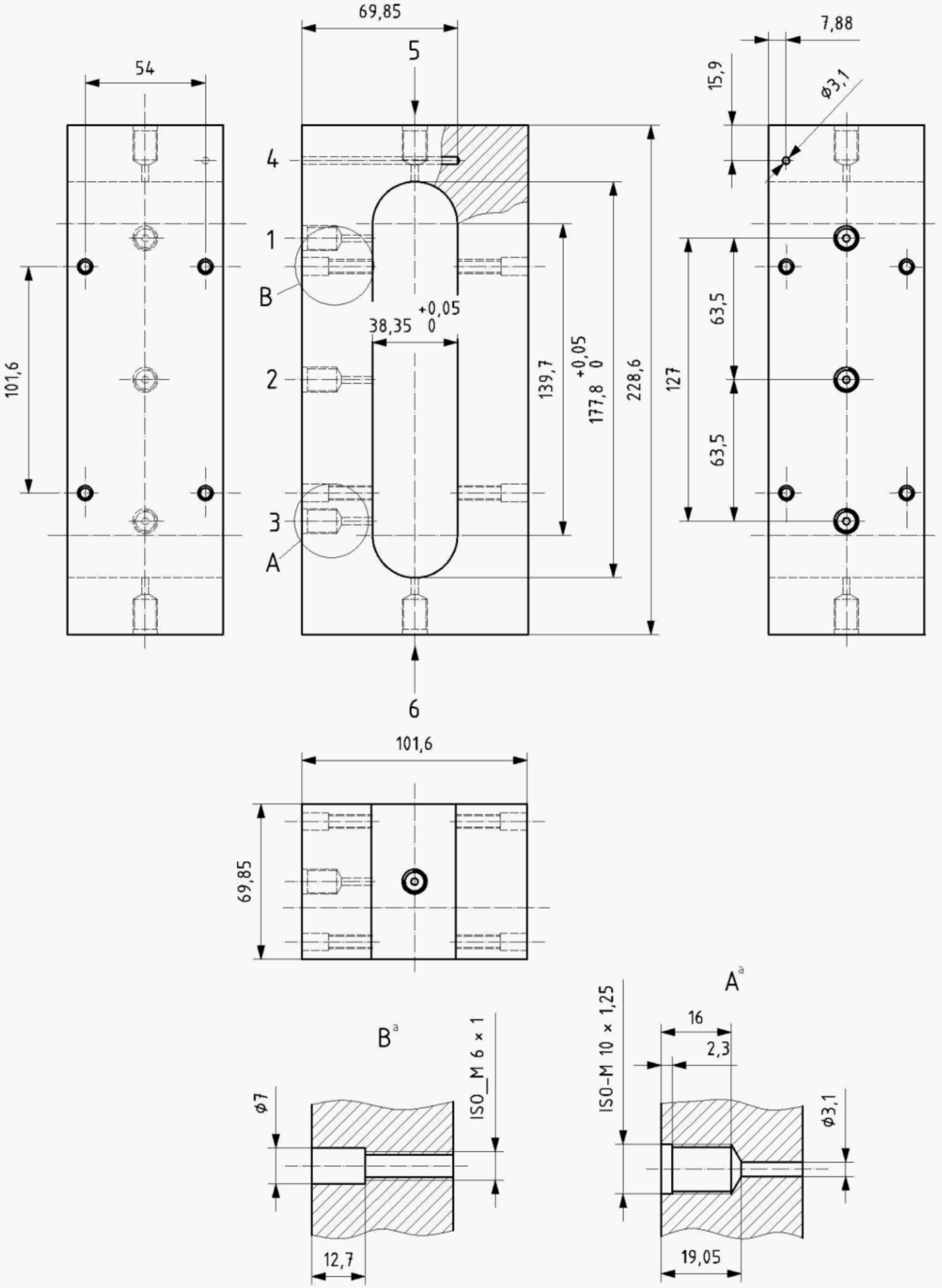
Surficial treatment of the inner matrix: 2,5 u Ra u 4

a Not to scale.

b L. centre.

Figure C.2 — Upper and lower piston

Dimensions in millimetres



Key

1 low-pressure port
2 middle port
3 high-pressure port
4 temperature control sensor
5 inlet of cell
6 outlet of cell

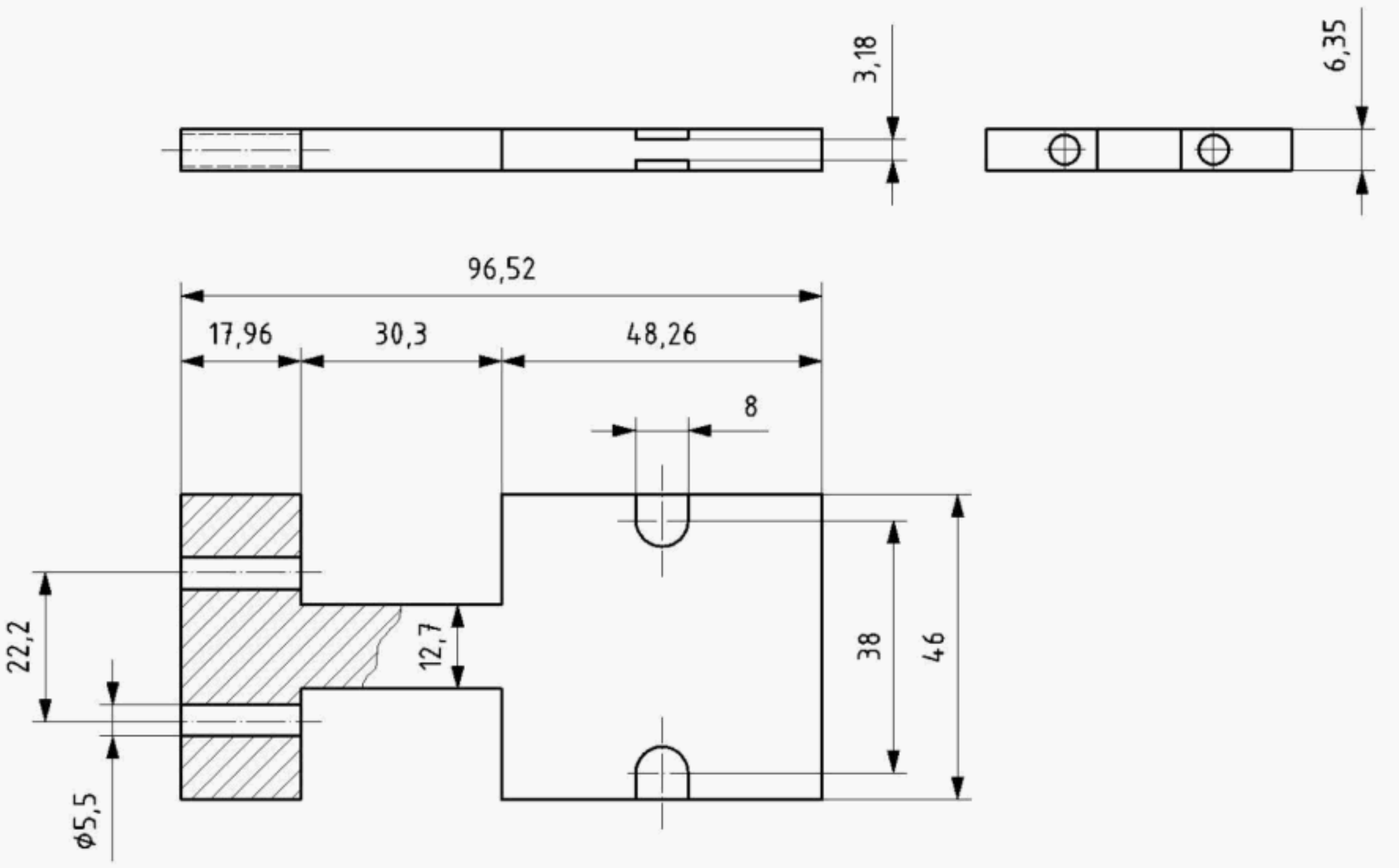
Surficial treatment of the inner matrix: 2,5 u Ra u 4.

Both sides of the cell shall be chamfered by 1,5 mm × 1,5 mm.

a Not to scale.

Figure C.3 — Cell body

Dimensions in millimetres

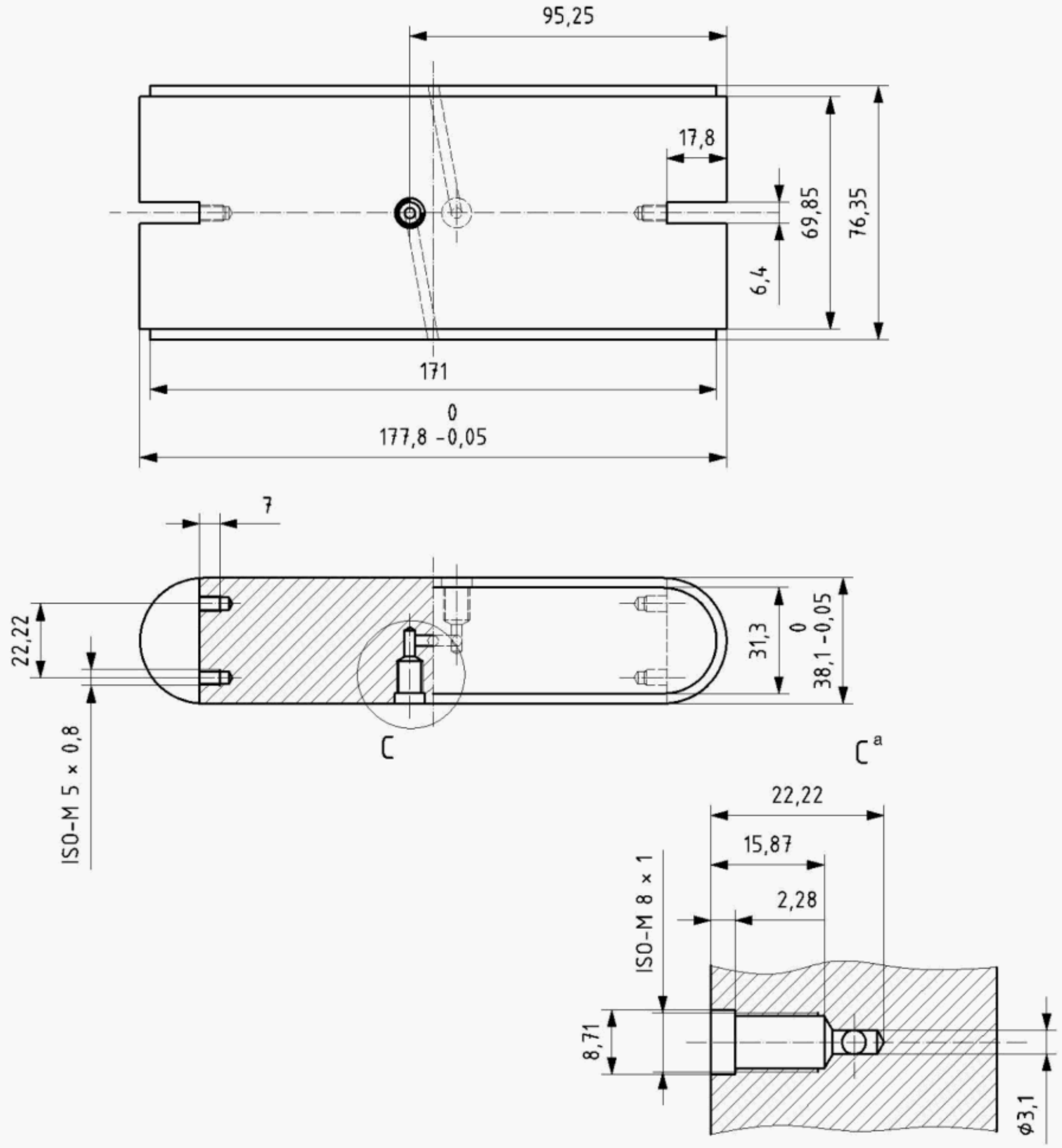


Surficial treatment of the inner matrix: 2,5 u Ra u 4.

No sharp edges.

Figure C.4 — Width slat

Dimensions in millimetres

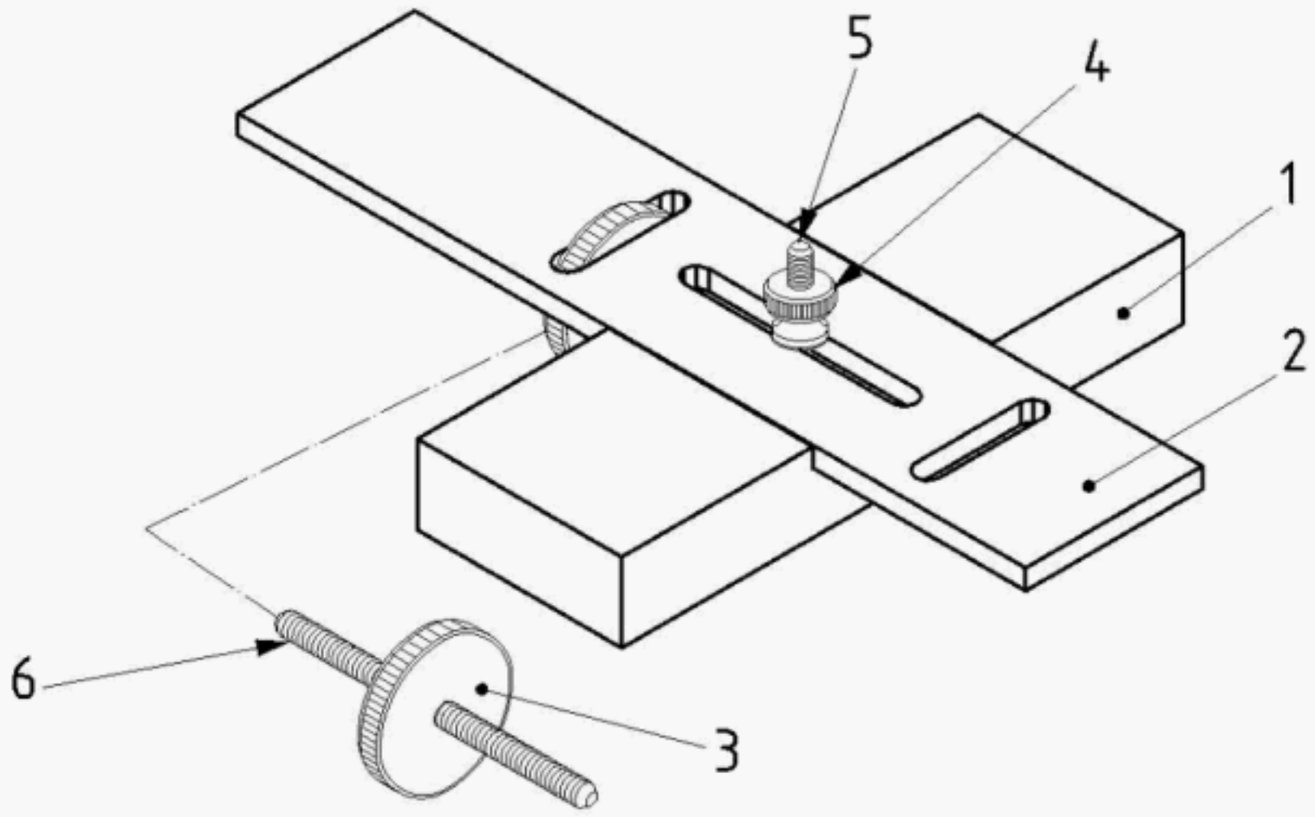


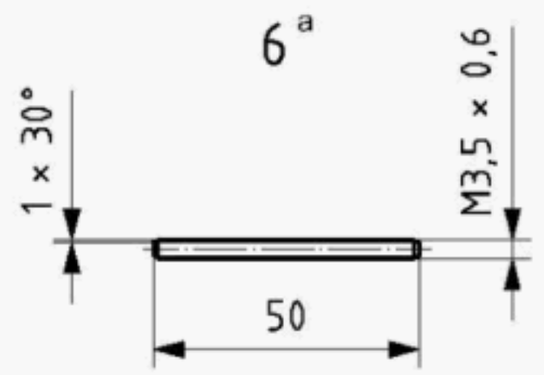
Surficical treatment of the inner matrix: 2,5 u Ra u 4.

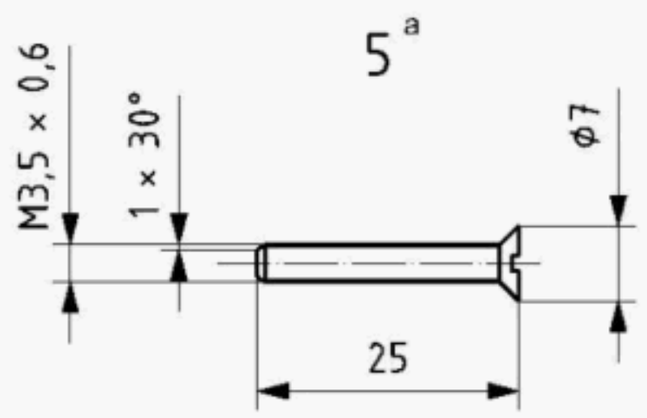
a Not to scale.

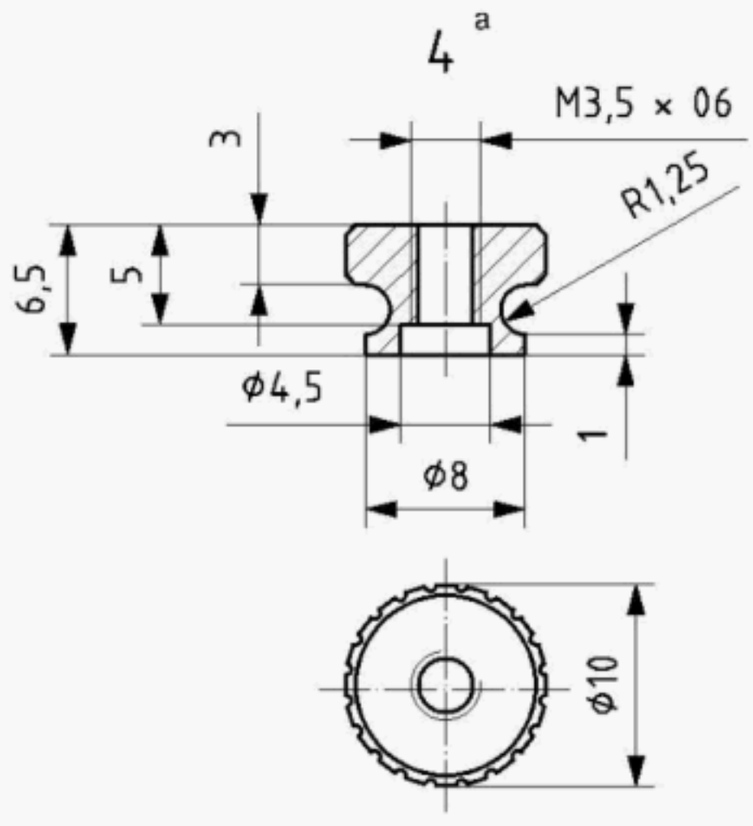
Figure C.5 — Central piston

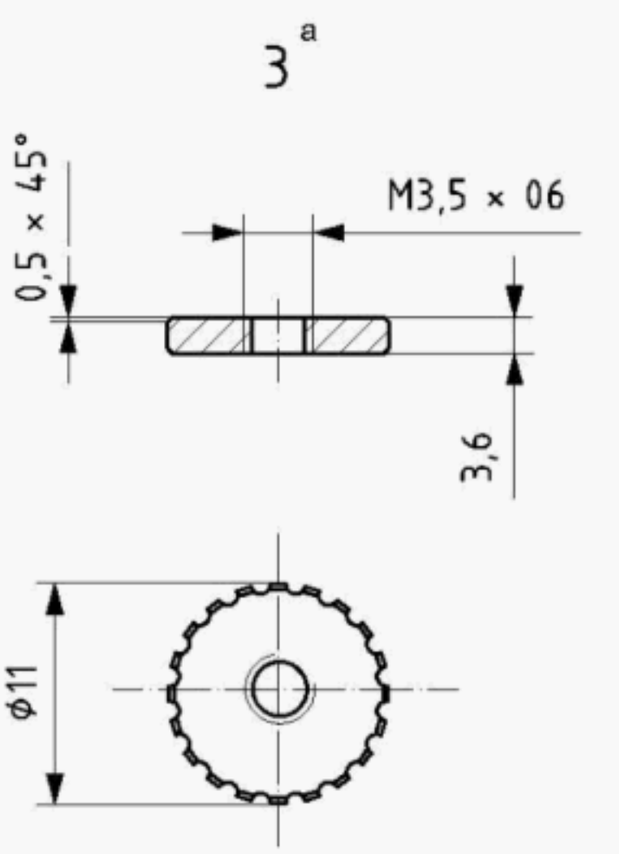
Dimensions in millimetres

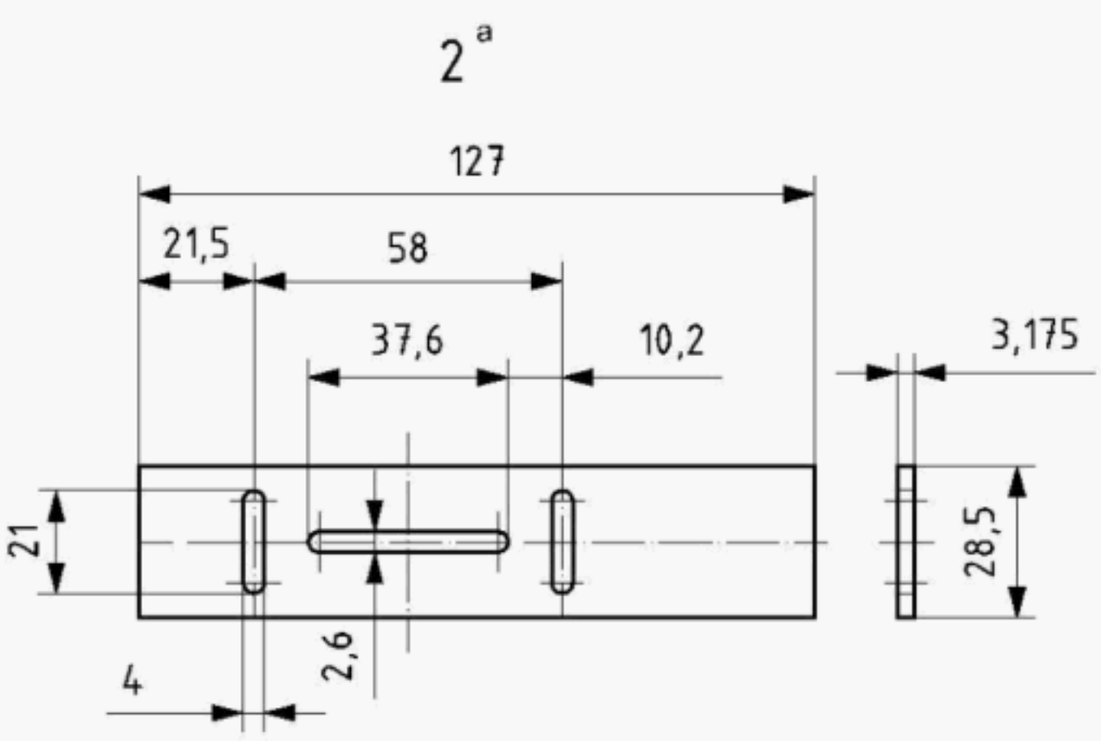


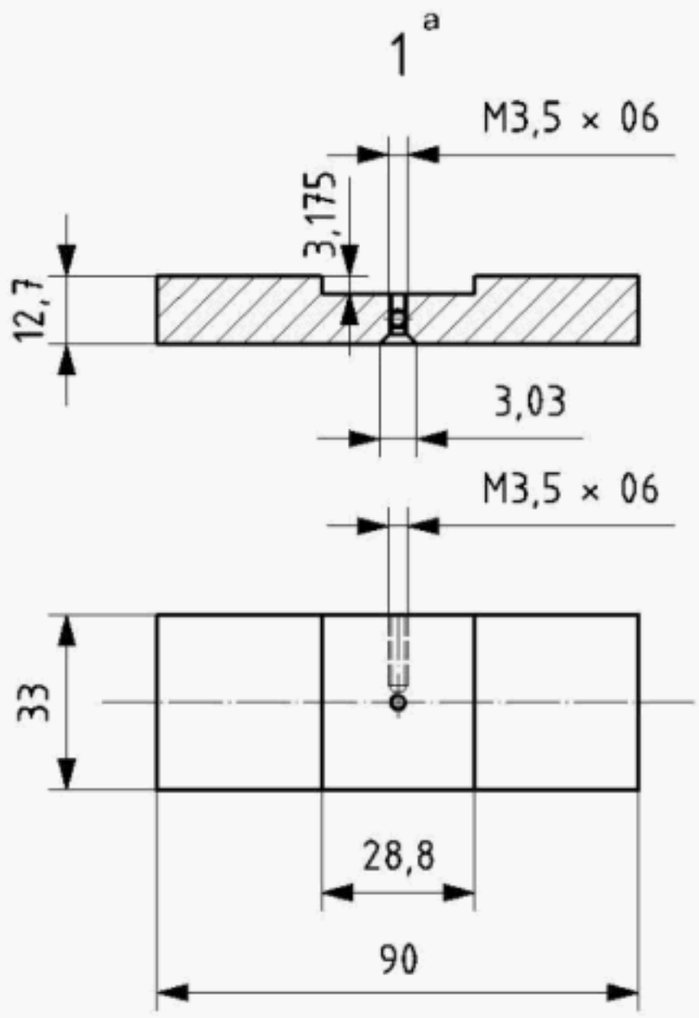












Key

1 straight edge support
2 straight edge
3 screw nut regulator
4 screw nut
5 screw R-3,5 × 0,6
6 screw M-3,5 × 0,6
a Not to scale.

Figure C.6 — Levelling device

Table C.1 — 2 % KCl viscosity vs. temperature

Temperature		Viscosity	Temperature		Viscosity	Temperature		Viscosity	Temperature		Viscosity
°C	°F	cP	°C	°F	cP	°C	°F	cP	°C	°F	cP
21,1	70	1,000 0	35,6	96	0,747 5	50,0	122	0,572 0	64,4	148	0,462 0
21,7	71	0,990 0	36,1	97	0,740 0	50,6	123	0,567 5	65,0	149	0,457 5
22,2	72	0,973 0	36,7	98	0,732 5	51,1	124	0,561 0	65,6	150	0,454 5
22,8	73	0,960 0	37,2	99	0,725 0	51,7	125	0,557 0	66,1	151	0,451 0
23,3	74	0,950 0	37,8	100	0,718 0	52,2	126	0,552 0	66,7	152	0,447 5
23,9	75	0,930 0	38,3	101	0,712 5	52,8	127	0,547 0	67,2	153	0,444 0
24,4	76	0,915 0	38,9	102	0,706 0	53,3	128	0,542 0	67,8	154	0,440 5
25,0	77	0,905 0	39,4	103	0,699 0	53,9	129	0,537 5	68,3	155	0,437 5
25,6	78	0,900 0	40,0	104	0,691 0	54,4	130	0,532 5	68,9	156	0,434 0
26,1	79	0,890 0	40,6	105	0,685 0	55,0	131	0,528 0	69,4	157	0,430 0
26,7	80	0,877 5	41,1	106	0,677 5	55,6	132	0,524 5	70,0	158	0,427 0
27,2	81	0,872 5	41,7	107	0,670 0	56,1	133	0,520 0	70,6	159	0,423 0
27,8	82	0,862 5	42,2	108	0,665 0	56,7	134	0,516 0	71,1	160	0,419 0
28,3	83	0,855 0	42,8	109	0,655 0	57,2	135	0,512 0	71,7	161	0,417 5
28,9	84	0,845 0	43,3	110	0,648 0	57,8	136	0,508 0	72,2	162	0,414 0
29,4	85	0,840 0	43,9	111	0,640 0	58,3	137	0,503 0	72,8	163	0,412 0
30,0	86	0,830 0	44,4	112	0,634 0	58,9	138	0,499 5	73,3	164	0,407 5
30,6	87	0,823 0	45,0	113	0,627 5	59,4	139	0,495 0	73,9	165	0,405 0
31,1	88	0,815 0	45,6	114	0,622 0	60,0	140	0,491 0	74,4	166	0,402 0
31,7	89	0,807 5	46,1	115	0,615 0	60,6	141	0,487 5	75,0	167	0,398 0
32,2	90	0,795 0	46,7	116	0,608 0	61,1	142	0,484 0	75,6	168	0,396 0
32,8	91	0,790 0	47,2	117	0,602 5	61,7	143	0,480 0	76,1	169	0,392 5
33,3	92	0,780 0	47,8	118	0,596 0	62,2	144	0,477 0	76,7	170	0,390 0
33,9	93	0,775 0	48,3	119	0,588 0	62,8	145	0,472 5	77,2	171	0,387 5
34,4	94	0,763 0	48,9	120	0,582 0	63,3	146	0,468 0	77,8	172	0,385 0
35,0	95	0,755 0	49,4	121	0,577 5	63,9	147	0,465 0	78,3	173	0,382 5

Temperature		Viscosity
°C	°F	cP
78,9	174	0,380 0
79,4	175	0,377 5
80,0	176	0,375 0
80,6	177	0,372 5
81,1	178	0,370 0
81,7	179	0,367 5
82,2	180	0,364 5
82,8	181	0,362 5
83,3	182	0,359 0
83,9	183	0,357 5
84,4	184	0,355 0
85,0	185	0,352 5
85,6	186	0,350 0
86,1	187	0,347 5
86,7	188	0,345 0
87,2	189	0,342 5
87,8	190	0,341 0
88,3	191	0,338 0
88,9	192	0,336 5
89,4	193	0,335 0
90,0	194	0,332 5
90,6	195	0,330 5
91,1	196	0,328 0
91,7	197	0,326 0
92,2	198	0,324 5
92,8	199	0,322 5
93,3	200	0,321 0
93,9	201	0,319 0
94,4	202	0,317 5
95,0	203	0,316 0
95,6	204	0,314 0
96,1	205	0,312 5
96,7	206	0,311 0
97,2	207	0,308 5
97,8	208	0,307 5
98,3	209	0,306 5
98,9	210	0,304 5
99,4	211	0,302 5

Temperature		Viscosity
°C	°F	cP
100,0	212	0,301 0
100,6	213	0,299 0
101,1	214	0,298 0
101,7	215	0,296 0
102,2	216	0,294 5
102,8	217	0,293 0
103,3	218	0,292 0
103,9	219	0,290 5
104,4	220	0,288 0
105,0	221	0,287 0
105,6	222	0,286 0
106,1	223	0,284 0
106,7	224	0,282 5
107,2	225	0,281 0
107,8	226	0,280 0
108,3	227	0,278 0
108,9	228	0,276 5
109,4	229	0,275 0
110,0	230	0,274 0
110,6	231	0,272 5
111,1	232	0,271 0
111,7	233	0,270 0
112,2	234	0,268 5
112,8	235	0,267 5
113,3	236	0,266 0
113,9	237	0,265 0
114,4	238	0,264 0
115,0	239	0,262 5
115,6	240	0,261 0
116,1	241	0,259 0
116,7	242	0,258 0
117,2	243	0,257 0
117,8	244	0,255 5
118,3	245	0,254 0
118,9	246	0,253 0
119,4	247	0,252 0
120,0	248	0,251 0
120,6	249	0,249 5

Temperature		Viscosity
°C	°F	cP
121,1	250	0,248 0
121,7	251	0,247 0
122,2	252	0,245 5
122,8	253	0,244 0
123,3	254	0,243 0
123,9	255	0,242 5
124,4	256	0,241 0
125,0	257	0,240 0
125,6	258	0,238 0
126,1	259	0,237 5
126,7	260	0,236 5
127,2	261	0,235 5
127,8	262	0,234 5
128,3	263	0,233 0
128,9	264	0,232 5
129,4	265	0,232 0
130,0	266	0,231 0
130,6	267	0,229 5
131,1	268	0,228 0
131,7	269	0,227 0
132,2	270	0,226 5
132,8	271	0,226 0
133,3	272	0,225 5
133,9	273	0,224 5
134,4	274	0,223 5
135,0	275	0,222 5
135,6	276	0,222 0
136,1	277	0,221 5
136,7	278	0,221 0
137,2	279	0,219 5
137,8	280	0,218 0
138,3	281	0,217 5
138,9	282	0,217 0
139,4	283	0,216 0
140,0	284	0,215 0
140,6	285	0,214 0
141,1	286	0,213 5
141,7	287	0,213 0

Temperature		Viscosity
°C	°F	cP
142,2	288	0,212 5
142,8	289	0,212 0
143,3	290	0,211 0
143,9	291	0,210 0
144,4	292	0,209 0
145,0	293	0,208 0
145,6	294	0,207 5
146,1	295	0,207 0
146,7	296	0,206 5
147,2	297	0,206 0
147,8	298	0,205 0
148,3	299	0,204 5
148,9	300	0,204 0
149,4	301	0,203 0
150,0	302	0,202 5
150,6	303	0,202 0
151,1	304	0,201 0
151,7	305	0,200 0
152,2	306	0,199 0
152,8	307	0,198 0
153,3	308	0,197 5
153,9	309	0,197 0
154,4	310	0,196 5
155,0	311	0,196 0
155,6	312	0,195 5
156,1	313	0,195 0
156,7	314	0,194 5
157,2	315	0,194 0
157,8	316	0,193 5
158,3	317	0,193 0
158,9	318	0,192 5
159,4	319	0,192 0

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24

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