FINAL
DRAFT

INTERNATIONAL
STANDARD

**ISO/FDIS
5119**

ISO/TC **131**/SC **7**

Secretariat: **JISC**

Voting begins on:
2023-08-29

Voting terminates on:
2023-10-24

Low temperature sealing capability of elastomeric seals — Test methods

Capacité d'étanchéité à basse température des joints en élastomère — Méthodes d'essai

Reference number
ISO/FDIS 5119:2023(E)

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Published in Switzerland

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Foreword

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This document was prepared by Technical Committee ISO/TC 131, *Fluid power systems*, Subcommittee SC 7, *Sealing devices*.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

There are several existing specifications to determine the low-temperature characteristics of elastomeric seal materials. There are also proprietary functional test procedures that aim to identify the minimum operating temperature for seals; however, all of these rely on the seal being energized by the pressure of the test fluid before being subjected to low temperature.

This specification gives details of a test procedure to act as a guide to the minimum operating temperature of elastomeric seals when used in static sealing applications when pressure is applied after cooling, the more commonly encountered situation.

Low temperature sealing capability of elastomeric seals — Test methods

1 Scope

This specification details a test method for static O-ring seals in elastomeric materials which are subject to pressurized gas media at low temperatures. It gives guidance on the design of test equipment, standard test parameters, and reporting criteria. It does not specify performance criteria that should be agreed upon between supplier and customer.

The test procedure may be used to test seals of alternate size and design or using alternative media but such deviations shall be detailed separately on the report form and the results shall not be used to determine the minimum operating temperature of seals of any other configuration than that tested.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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:

ISO 3601-1, *Fluid power systems — O-rings — Part 1: Inside diameters, cross-sections, tolerances and designation codes*

ISO 3601-2, *Fluid power systems — O-rings — Part 2: Housing dimensions for general applications*

ISO 3601-3, *Fluid power systems — O-rings — Part 3: Quality acceptance criteria*

ISO 5598, *Fluid power systems and components — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms, and definitions given in ISO 5598 apply and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
minimum seal temperature
minimum temperature at which the test seal holds the test pressure at the end of the test

3.2
zero leakage
gas leak rate considered to be negligible for the purposes of the test and equal to a displacement of less than 20 cm^3/h equivalents to no discernible bubbles

Note 1 to entry: The zero leakage is as defined as in ISO 10423.

3.3
room temperature
standard temperature of the test facility usually considered to be in the range of (20 ± 5) °C

3.4
surface roughness
surface roughness of metal parts refers to the finely spaced irregularities on the surface due to manufacturing processes or wear, influencing performance, durability, and aesthetics

Note 1 to entry: The surface roughness of the O-ring housing and any mating part has a significant impact on the life and sealing performance of the O-ring.

Note 2 to entry: Unless otherwise agreed, surface roughness values shall be in accordance with ISO 3601-2:2016, Table 1.

Note 3 to entry: All surfaces against which a seal operates should be free from scratches, burrs, gouges, scores, nicks, tool chatter, spiral machining marks (circumferential marks), or other defects along the operating axis of the seal as these may reduce sealing efficiency and the life of the seal.

Note 4 to entry: ISO 21920-2 for surface roughness measurement require new statements for roughness requirements. If due to the short measuring length, an exact roughness is not measurable, a visual inspection using master parts is permitted.

Note 5 to entry: Unless otherwise agreed, the material ratio, R_{mr}, should be 50 % to 80 % for surfaces of mating parts, determined at a cut depth of C = 0,25 Rz, relative to a reference profile line of C_o= 0,05 R_{mr}

3.5
housing material
selection based on availability, thermal conductivity, and corrosion resistance of the material.

Note 1 to entry: It is recommended to use: AISI 316L or EN X2CrNiMo17-12-2 (1.4404) or ISO 4404-316-03-I.

4 Test apparatus

4.1 The test apparatus shall be designed in accordance with the drawing shown using the housing material with the surface roughness defined following the Annex A and shall consist of a suitable test cell with 3 major components:

4.1.1 Solid cylindrical test plug, containing a groove on its outer diameter to suit a test O- ring size -316 (ISO 3601-1-316A-21.59x5.33-N)in accordance with ISO 3601-1 when used in a static piston sealing application.

4.1.2 Outer cylindrical test shroud with a bore to suit the test O-ring and an external means of sealing to retain the test fluid under pressure – normally an O-ring which will remain flexible at a temperature at least 10 °C below the minimum test temperature.

4.1.3 Cylindrical cap which fits around the test shroud and is sealed on its bore by the flexible O-ring and contains suitable fittings to allow the ingress of the test medium.

Means shall be provided to ensure centralization of the test plug within the test shroud such that the extrusion gap on the low-pressure side of the test seal does not exceed the requirements of ISO 3601-2

Seal sizes to ISO 3601-1-023A-26.70x1.78-N, ISO 3601-1-120A-25.07x2.62-N, or ISO 3601-1-213A-23.39x3.53-Ncan also be used but in those cases, the inside diameter of the seal housing shall be adjusted to suit the appropriate cross-section of the seal (see Annex C).

4.2 Test cell shall be provided with:

4.2.1 Means of cooling such that the temperature at the test cell can be reduced at a controlled rate of 60 °C/h (±10 °C/h).

4.2.2 Means of measuring the temperature of the test seal, positioned within (2 ± 0,5) mm of either the inner or outer diameter of the test seal.

4.2.3 Means of detecting leakage, bypassing the test seal by a mass flow meter with a minimum flow range lower than 20 cm^3/h – alternatively a leakage tube directly connected to the test cell and terminating within a water bath may be used where discernible bubbles of leakage can be observed. Leakage tube should have an inside diameter of (6 ± 0,5) mm.

4.2.4 Means of an arrangement by which the test medium may be applied under pressure to the test cell and the pressure within the cell measured.

4.2.5 Alternative sealing solution needed if the test temperature falls below the minimum seal temperature of the fixture dummy static O-ring.

NOTE The expected minimum seal temperature can be estimated by use of other material or functional tests e.g. ISO 812, ISO 815-2, ISO 1432, ISO 2921.

5 Test condition

5.1 Temperature

Tests shall be carried out at a range of temperatures from room temperature down to at least 10 °C below the expected minimum seal temperature.

NOTE The expected minimum seal temperature can be estimated by use of other material or functional tests e.g. ISO 812, ISO 815-2, ISO 1432, ISO 2921.

5.2 Test medium

The test medium shall be nitrogen gas.

5.3 Test pressure

The test pressure applied to the seals shall be selected based on the application, the predefined test pressures are:

— 5 MPa +0,25 /-0;

— 10 MPa +0,5/-0;

— 15 MPa +0,75/-0.

NOTE Alternatively, different test pressures can be used as an option in agreement with the customers' requirements.

6 Pre-test procedure

6.1 Inspect the test seals for conformity to their dimensional specification in accordance with ISO 3601-1 and visually in accordance with ISO 3601-3 Grade N and record their actual cross-section and inside diameter.

6.2 Install the dummy static O-ring and test seal in their respective grooves – the test seals shall not be lubricated.

6.3 Assemble the test cell and all relevant connections and monitoring devices.

6.4 Pressurize the cell with nitrogen to 1,5 MPa at ambient room temperature at a rate of approximately 0,5 MPa/min.

6.5 Hold the cell at 1,5 MPa for 2 min and check that there is zero leakage.

6.6 Apply the test pressure for 2 min and check that there is zero leakage.

6.7 Release the pressure.

7 Test procedure

7.1 Reduce the temperature of the test cell and seal (see 4.2.1) to a temperature 5 °C above the expected minimum seal temperature and hold for a minimum of 5 minutes after the fixture temperature has remained stable (±0,5 °C) for at least 5 min.

7.2 Apply the test pressure and check for leakage.

7.2.1 If leakage is observed release the test pressure and raise the temperature by 5 °C and hold for a minimum of 5 min after the temperature has remained stable (±0,5 °C) for at least 5 min then repeat the procedure from 7.2 onwards.

7.2.2 If zero leakage is observed hold pressure for 5 min.

7.3 If zero leakage is observed release the test pressure and reduce the temperature by a further 5° and hold for a minimum of 5 min after the temperature has remained stable (±0,5 °C) for at least 5 min.

7.4 Repeat the test procedure from 7.2 onwards until a temperature is reached where the seal fails to hold pressure.

7.5 Release the pressure and raise the temperature by 1 °C, hold for a minimum of 5 min after the temperature has remained stable (±0,5 °C) for at least 5 min, and then apply the pressure.

7.5.1 If leakage is observed release the test pressure and raise the temperature by 1 °C and hold for a minimum of 5 min after the temperature has remained stable (±0,5 °C) for at least 5 min then repeat the procedure from 7.5 onwards.

7.5.2 If zero leakage is observed hold pressure for 5 min.

7.6 Continue the process from clause 7.5 onwards until a temperature is reached at which the pressure can be held for 5 minutes with zero leakage – this is the minimum seal temperature.

7.7 The start point for each repeated test shall be 5 °C higher than the previous minimum seal temperature.

7.8 A new set of seals shall be used in every new test run.

7.9 A minimum of 5 test runs shall be carried out for each material. The final minimum seal temperature reported shall be the average of 3 of those 5 individual samples disregarding the highest and lowest sample value.

8 Test report

8.1 Record all test data on a seal test report form (an example is shown in Annex B).

8.2 Publishing of results.

When publishing results for consumption by potential users the following data shall be included:

— Standard reference (i.e. ISO 5119:—) and the issue number;

— Seal material;

— Test pressure;

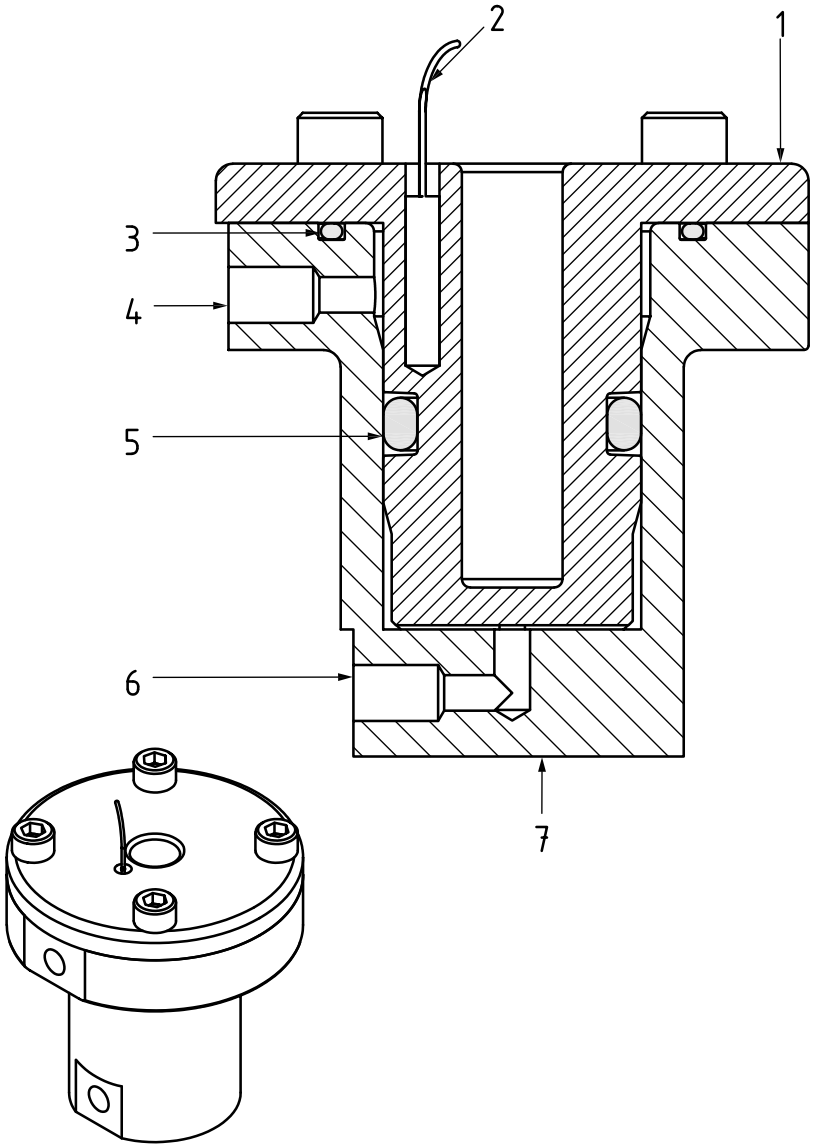
— Minimum seal temperature.

9 Precision

See Annex D.

Annex A
(informative)

Typical test assembly



Key

1 top view as shown Figure A.3
2 PT100 sensor fill volume around and above PT100 sensor with thermal paste.
3 static O-ring 40,95x2,62
4 leakage port
5 test specimen O-ring 21,59x5,33
6 pressure port
7 below view as shown Figure A.2

Figure A.1 — Isometric and cross-section of assembly

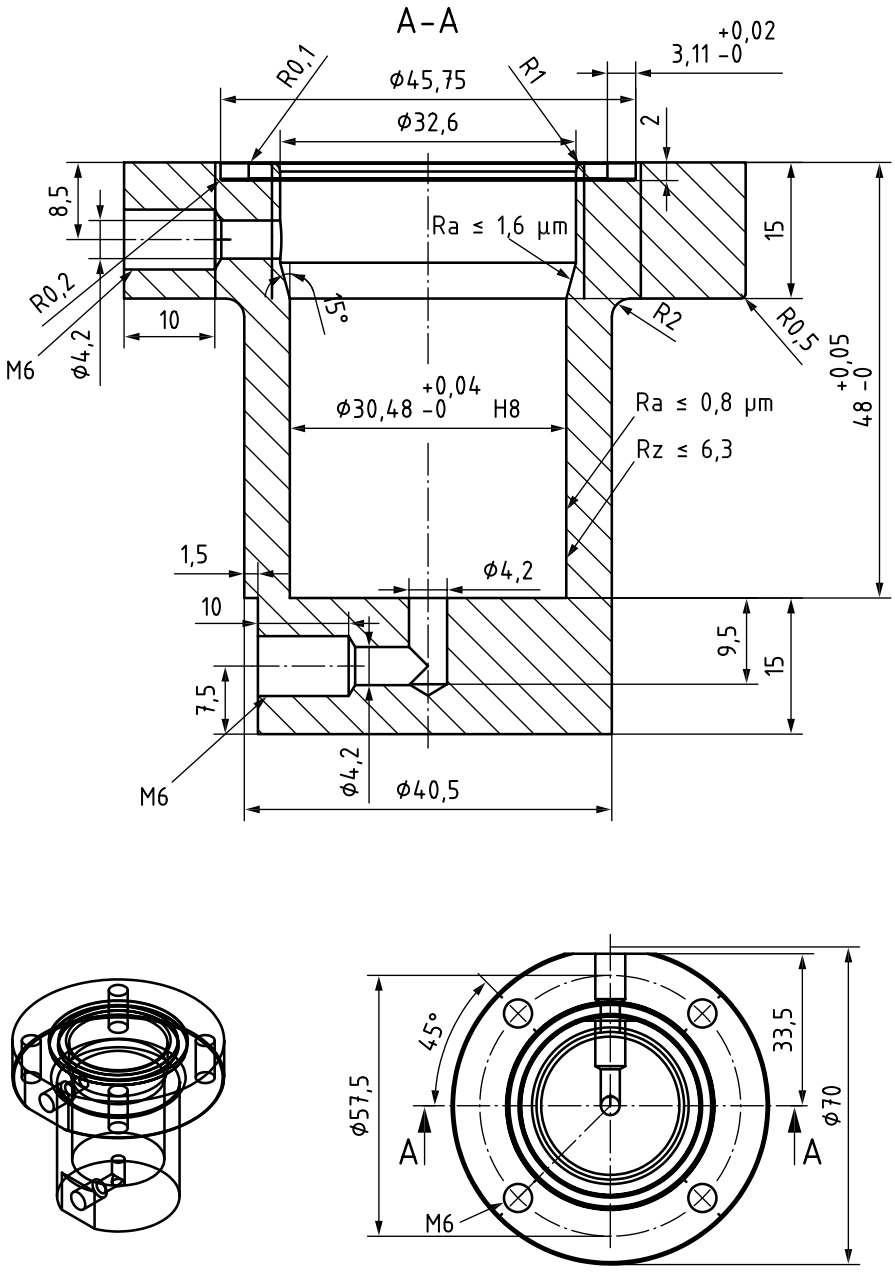


Figure A.2 — Isometric, cross-section and top view of female fixture

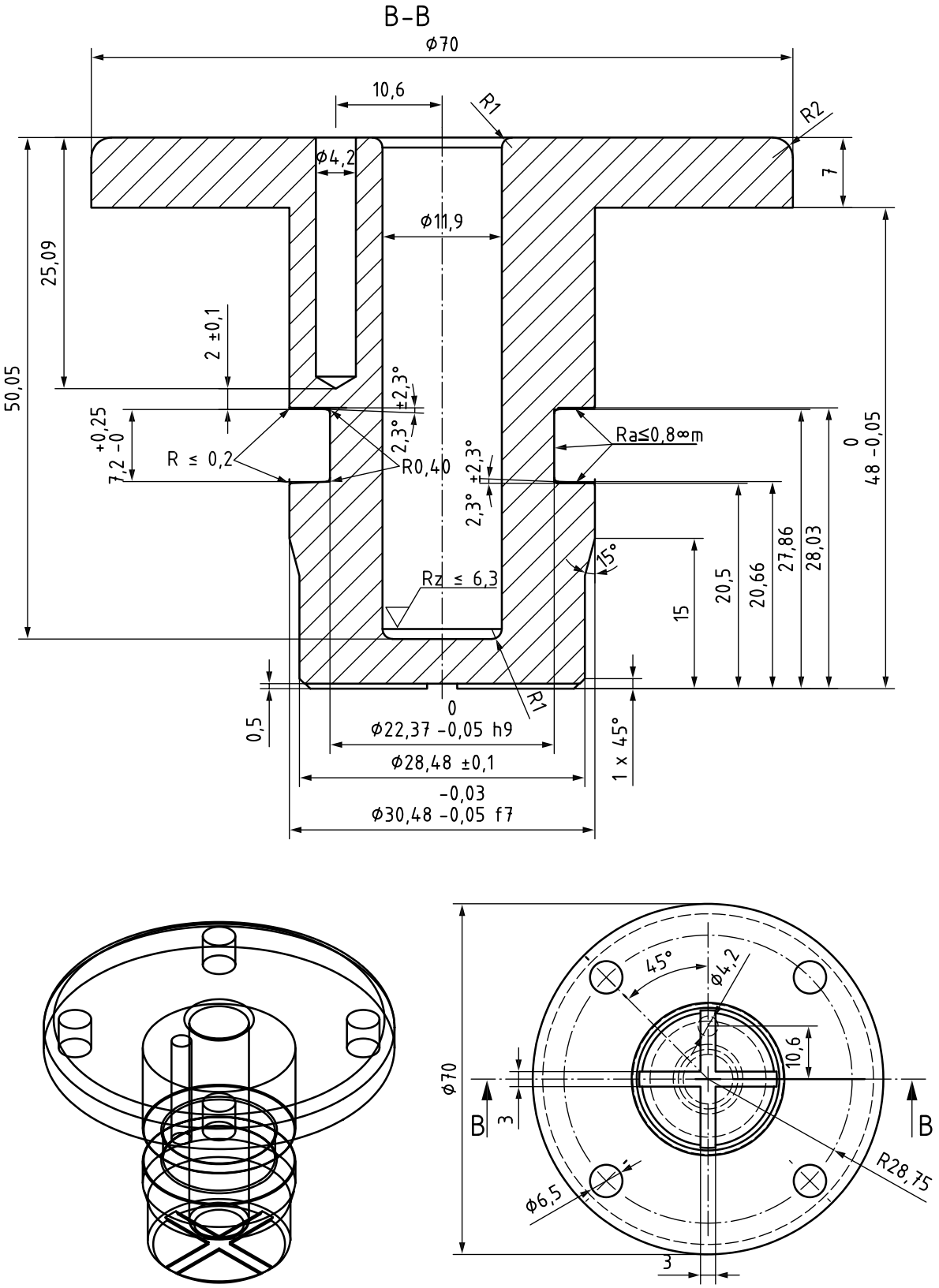


Figure A.3 — Isometric, cross-section and top view of male fixture

Annex B
(informative)

Test report form

SEAL DETAILS										
	Test 1		**Test 2**		**Test 3**		**Test 4**		**Test 5**	
Seal manufacturer										
Supplied by										
Compound name/no./ref.										
Elastomer type										
Lot/batch no.										
Seal type										
Manufacturing method										
Seal size (ISO 3601-1/AS 568)										
CSD(Cross-Sectional Diameter) (nominal, mm)										
Mean CSD(actual, radial, mm)										
SEAL HOUSING DETAILS										
Seal compression direction: Radial					Squeeze (average, %):					
Groove ID (mm) & Groove OD (mm):					Groove fill (area basis, %):					
Groove width (mm):					Lubrication (if applied):					
PRELIMINARY PRESSURE TEST										
Temperature (°C)										
Pressure (MPa)										
Pass/Fail										
TEST CONDITIONS										
Pressure (MPa):					Test media:					
	Temp °C	PASS/ FAIL	Temp °C	PASS/ FAIL	Temp °C	PASS/ FAIL	Temp °C	PASS/ FAIL	Temp °C	PASS/ FAIL

Temperature	Temperature 1										
	Temperature 2										
	Temperature 3										
	Temperature 4										
	Temperature 5										
	Temperature 6										
	Temperature 7										
	Temperature 8										
	Temperature 9										
	Temperature 10										
	Temperature 11										
	Temperature 12										
	Temperature 13										
	Temperature 14										

MINIMUM SEALING TEMPERATURE (°C)	
Test seal 1	Test seal 4
Test seal 2	Test seal 5
Test seal 3	
Minimum sealing temperature *(mean temperature, disregarding highest and lowest temperatures)*°C:	

GENERAL	
Test laboratory:	Test gas certified and available:
Test date(s):	Leakage detection method:
Leakage detection calibration available:	P/T reading available:
P/T calibration available:	Temperature sensor reading available:

ABOUT THE STANDARD		
the International Standard used (with year of publication)		
the method used (if the standard includes several)		
Any deviations from the procedure		
Any unusual features observed		
Reference to the clause which explains how the results were calculated		
Test operator	**Signed**	**Date**

Figure B.1 — Report form

Annex C
(informative)

Housing sizes for O rings of other cross-sections

When utilizing the test fixture body to test seals of alternative cross-section diameter (i.e. 3601-1 sizes 023, -120, -213), the inside diameter of the seal housing must be adjusted from that indicated in ISO 3601-2 in order to accommodate the slight differences in the bore diameter.

The amendments can be:

ISO 3601-1-023A-26.70x1.78-N – Increase by 0,40mm

ISO 3601-1-120A-25.07x2.62-N – Reduce by 0,65 mm

ISO 3601-1-213A-23.39x3.53-N - Reduce by 0,97 mm

Annex D
(informative)

Precision test results

Precision calculations to express repeatability and reproducibility were performed in accordance with ISO 19983. Outliers in original data were treated at the 5 % and 2 % significance level in accordance with the procedures described in ISO 19983.

Interlaboratory test programme

An interlaboratory test programme (ITP) was organized in 2021. ISO 3601-1-316A-21.59x5.33-N O-ring samples from the same batch made from two compounds of NBR and FKM were used for the low temperature testing. These compounds have different expected minimum seal temperatures.

A total of five laboratories participated in the ITP, from Türkiye, France, China and two from UK.

Fully prepared test O-rings were sent to each laboratory for evaluation in the ITP, a type 1 precision was determined.

The Precision Results of the ITP are given in Table 1.

p = number of measurements,

sr = repeatability standard deviation,

sR = reproducibility standard deviation,

m = mean for the level.

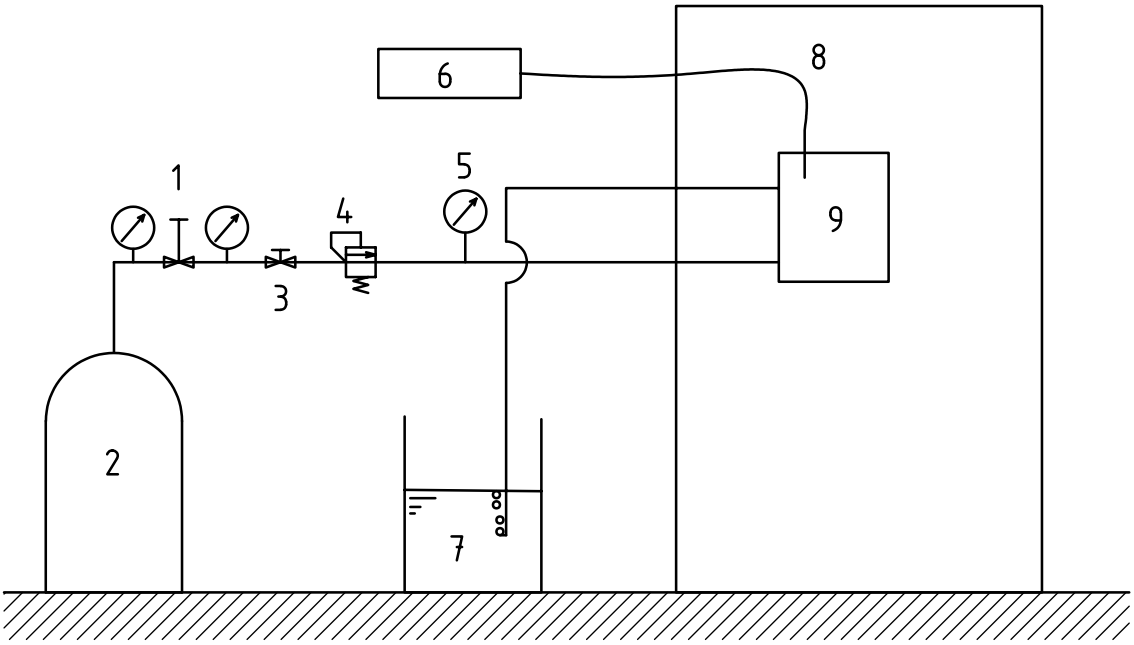
[a] RSD = denotes relative standard deviation; that is, the standard deviation s divided by the mean m for the level.

Table D.1 — Precision data

Material	p	m (°C)	sr (°C)	sR (°C)	RSDr[a]	RSDR[a]	No. of Laboratories
NBR	3	-26,93	0,93	2,11	-0,03	-0,08	5
FKM	3	-15,73	0,52	1,69	-0,03	-0,11	5

Annex E
(informative)

System diagram



Key

1 gas regulator
2 N2 gas cylinder
3 stop valve
4 pressure regulating valve
5 pressure gauge
6 thermometer
7 bubble bath
8 thermostatic
9 test fixture

Bibliography

[1] ISO 812, *Rubber, vulcanized or thermoplastic — Determination of low-temperature brittleness*

[2] ISO 815-2, *Rubber, vulcanized or thermoplastic — Determination of compression set — Part 2: At low temperatures*

[3] ISO 1432, *Rubber, vulcanized or thermoplastic — Determination of low-temperature stiffening (Gehman test)*

[4] ISO 2921, *Rubber, vulcanized — Determination of low-temperature characteristics — Temperature-retraction procedure (TR test)*

[5] ISO 10423, *Petroleum and natural gas industries — Drilling and production equipment — Wellhead and tree equipment*

[6] ISO 19983, *Rubber — Determination of precision of test methods*

ICS 83.140.50; 23.100.60

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