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Low Temperature Sealing Capability of Elastomeric Seals — Test Methods

Capacité d'étanchéité à basse température des joints élastomères — Méthodes d'essais

ICS: 83.140.50; 23.100.60

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

A list of all parts in the ISO 5119 series can be found on the ISO website.

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 or semi-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 O-ring seals in elastomeric materials which are subject to pressurized 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 utilized 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 812:2017, *Rubber, vulcanized or thermoplastic — Determination of low-temperature brittleness*

ISO 1432:2021, *Rubber, vulcanized or thermoplastic — Determination of low-temperature stiffening (Gehman test)*

ISO 815-2:2019, *Rubber, vulcanized or thermoplastic — Determination of compression set — Part 2: At low temperatures*

ISO 2921:2019, *Rubber, vulcanized — Determination of low-temperature characteristics — Temperature-retraction procedure (TR test)*

ISO 3601-1:2012, *Fluid power systems — O-rings — Part 1: Inside diameters, cross-sections, tolerances and designation codes*

ISO 3601-2:2016, *Fluid power systems — O-rings — Part 2: Housing dimensions for general applications*

ISO 3601-3:2005, *Fluid power systems — O-rings — Part 3: Quality acceptance criteria*

ISO 10423:2022, *Petroleum and natural gas industries — Drilling and production equipment — Wellhead and tree equipment*

ISO 5598:2020, *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. The following terms used in this specification have the meanings defined:

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

3.2
Zero leakage
A gas leak rate is 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 as defined in ISO 10423:2022.

3.3
Room Temperature
The standard temperature of the test facility is usually considered to be in the range of 20±5°C.

3.4
Surface Roughness
The surface roughness of the housing shall be in accordance with ISO 3601-2.

3.4.1 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.

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

*NOTE A*ll *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.*

3.4.3 ISO 21920-2:2021 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.

3.4.4 Unless otherwise agreed, the material ratio, Rmr, 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 Rmr

3.5
Housing Material
Housing material selection is based on availability, thermal conductivity, and corrosion resistance of the material, 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 in Annex A and shall consist of a suitable test cell with 3 major components:

4.1.1 A solid cylindrical test plug containing a groove on its outer diameter to suit a test O- ring in accordance with ISO 3601- 316 when used in a static piston sealing application.

4.1.2 An 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 A 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.

4.1.4 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

NOTE Seal sizes to ISO 3601-023, -120, -213 may also be used but in those cases, the inside diameter of the seal housing must be adjusted to suit the appropriate cross-section of the seal (see Annex C).

4.2 The test cell shall be provided with:

4.2.1 An external method of cooling such that the temperature at the test cell can be reduced at a controlled rate of 60 °C per hour (± 10 °C).

4.2.2 A 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 A means of detecting leakage bypassing the test seal by a mass flow meter with a minimum flow range lower than 20 cm^3/hour – 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 a diameter of 6 mm.

4.2.4 A means by which the test fluid may be applied under pressure to the test cell and the pressure within the cell measured.

4.2.5 An alternate seal arrangement is needed if the test temperature falls below the lowest sealing temperature of the fixture dummy static o-ring.

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 may be estimated by use of other material or functional tests e.g. ISO 812:2017, ISO 815-2:2019, ISO 1432:2021, ISO 2921:2019 etc.

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 end-user application, the predefined test pressures are;

— 5 MPa +5%/-0.

— 10 MPa +5%/-0.

— 15 MPa +5%/-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 leakage and test seals 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 per minute.

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

6.6 Apply the test pressure for 2 minutes 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 minutes.

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 minutes after the temperature has remained stable (±0,5°C) for at least 5 minutes then repeat the procedure from clause 7.2 onwards.

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

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

7.4 Repeat the test procedure from clause 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 minutes after the temperature has remained stable (±0,5°C) for at least 5 minutes, 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 minutes after the temperature has remained stable (±0,5°C) for at least 5 minutes then repeat the procedure from clause 7.5 onwards.

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

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 Reporting

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 must 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
(normative)

Typical Test Assembly

This drawing is provided for guidance only.

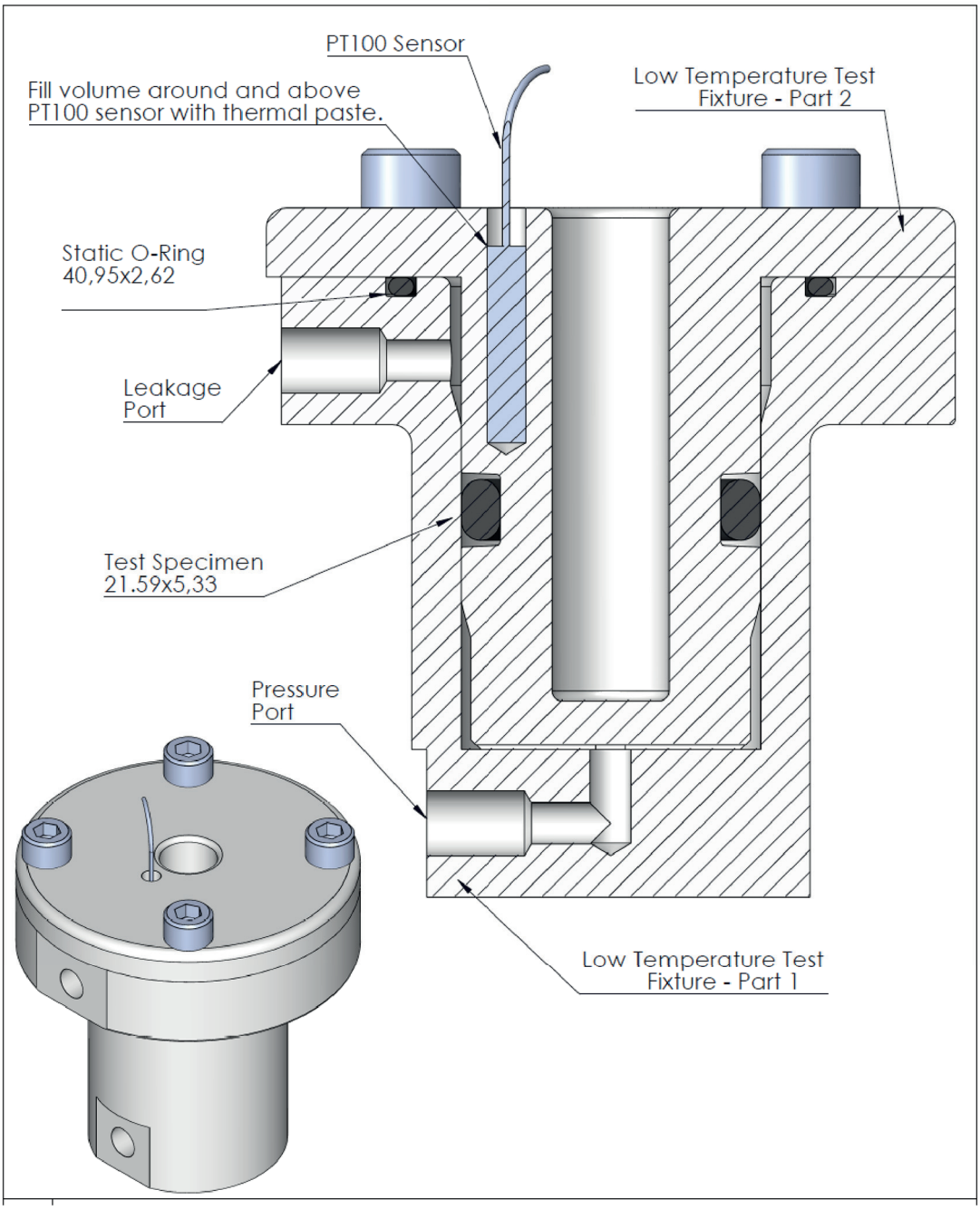

Figure A.1 — Assembly

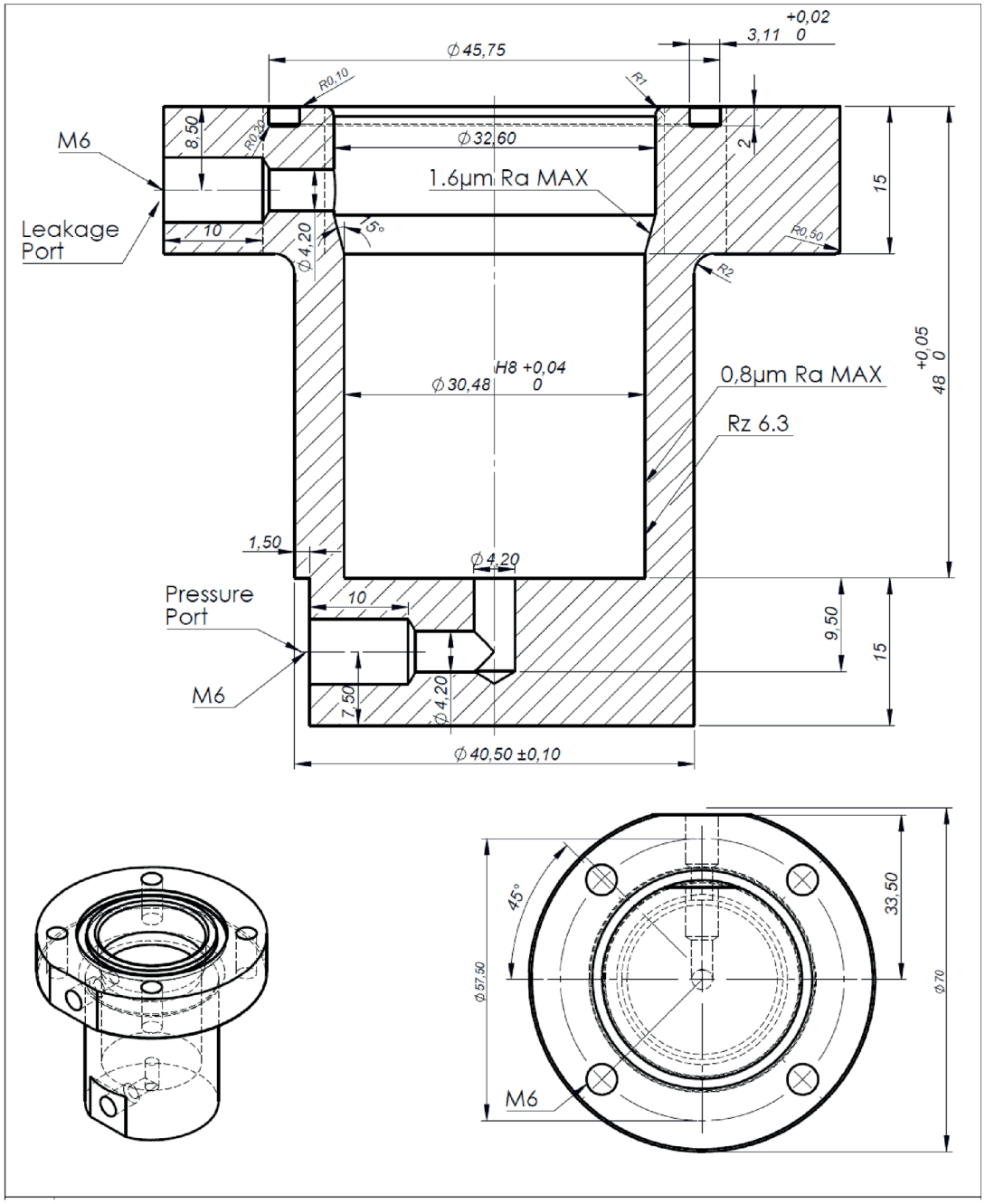

Figure A.2 — Low Temperature Fixture Part 1

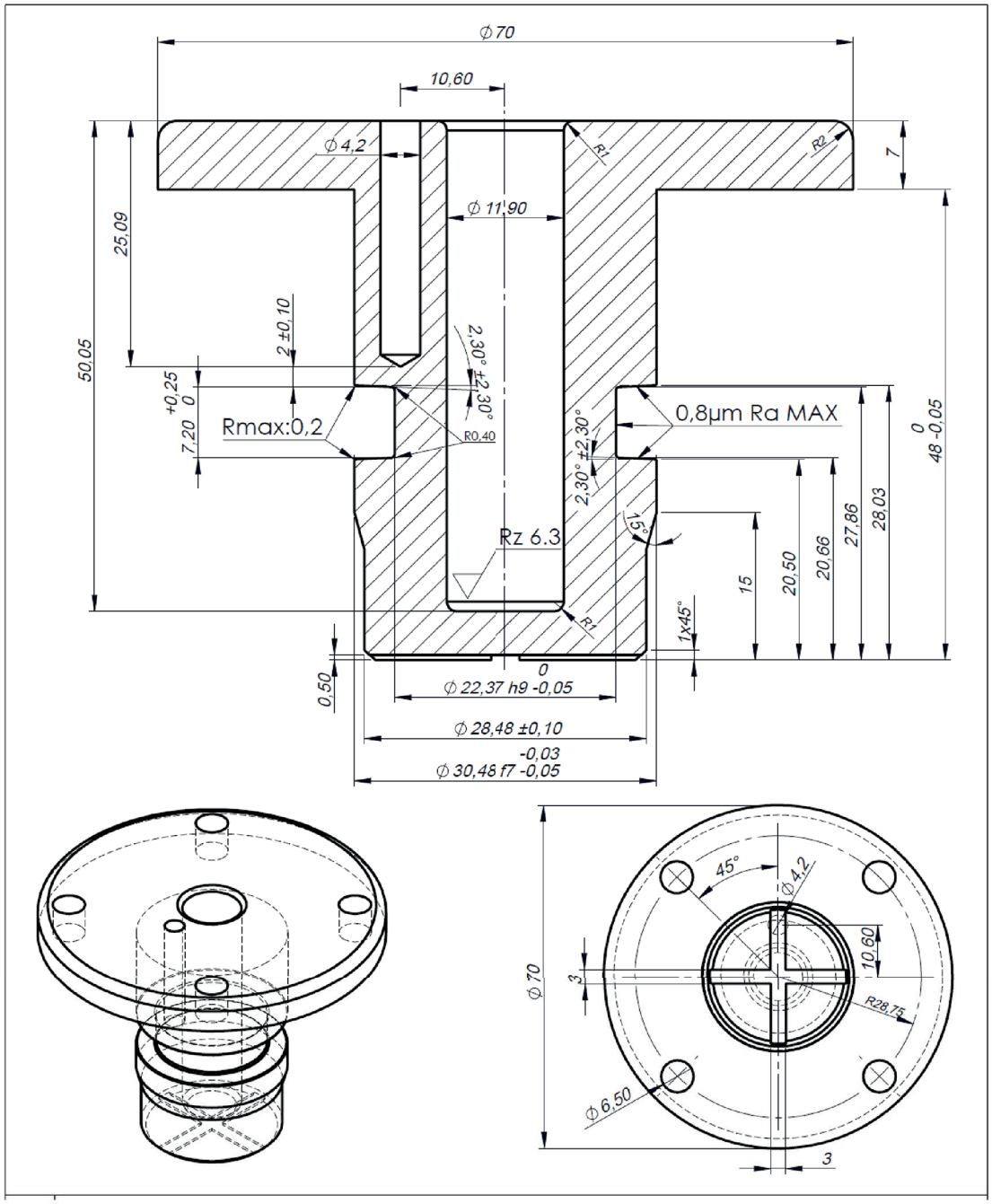

Figure A.3 — Low Temperature Fixture Part 2

Annex B
(informative)

Test Report Form

SEAL DETAILS											
		Test 1		Test 2		Test 3		Test 4		Test 5	
Seal manufacturer											
Supplied by											
Compound name/number/reference											
Elastomer type											
Lot/batch no.											
Seal type											
Manufacturing method											
Seal size (ISO 3601-1/AS 568)											
CSD (nominal)	mm										
Mean CSD (actual, radial)	mm										
SEAL HOUSING DETAILS											
Seal compression direction		Radial									
Groove ID	mm										
Groove OD	mm										
Groove width	mm										
Squeeze (average)	%										
Groove fill (area basis)	%										
Lubrication (if applied)											
PRELIMINARY PRESSURE TEST											
Temperature	°C										
Pressure	MPa										
Pass/Fail											
TEST CONDITIONS											
Pressure	MPa	15									
Test media (gas/liquid, type)		Gas Nitrogen									
		Temperature	PASS/FAIL	Temperature	PASS/FAIL	Temperature	PASS/FAIL	Temperature	PASS/FAIL	Temperature	PASS/FAIL
Temperature	Temperature 1	-15	Pass	-15	Pass	-15	Pass				
	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										
	Temperature 15										
	Temperature 16										
	Temperature 17										
	Temperature 18										
	Temperature 19										
	°C										
MINIMUM SEALING TEMPERATURE											
Test seal 1	°C										
Test seal 2	°C										
Test seal 3	°C										
Test seal 4	°C										
Test seal 5	°C										
Minimum sealing temperature	°C										
GENERAL											
Test laboratory											
Test date(s)											
Test gas certified and available											
Leakage detection method											
Leakage detection calibration available											
P/T calibration available											
P/T reading available											
Temperature sensor reading available											
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. 3601-023, -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 may be:

3601-023 – Increase by 0,40mm

3601-120 – Reduce by 0,65mm

3601-213 - Reduce by 0,97mm

Annex D
(informative)

Precision Test Results

Precision calculations to express repeatability and reproducibility were performed in accordance with ISO 19983:2017 Rubber — Determination of precision of test methods. Outliers in original data were treated at the 5% and 2% significance level in accordance with the procedures described in ISO 19983:2017.

Interlaboratory Test Programme

An interlaboratory test programme (ITP) was organized in 2021. ISO 316-316 size 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 Turkey, 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.

pj = number of measurements

sr = repeatability standard deviation,

sR = reproducibility standard deviation,

m = mean for the level.

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	pj	mj	srj	sRj	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