NORSOK STANDARD M-710

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Qualification of non-metallic sealing materials and manufacturers

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Foreword

The NORSOK standards are developed by the Norwegian petroleum industry to ensure adequate safety, value adding and cost effectiveness for existing and future petroleum industry developments.

The NORSOK standards are prepared to complement available international standards and fill the broad needs of the Norwegian petroleum industry. Where relevant NORSOK standards will be used to provide the Norwegian industry input to the international standardisation process. Subject to development and publication of international standards, the relevant NORSOK standard will be withdrawn.

These standards are developed according to the consensus principle generally applicable for most standards work and according to established procedures defined in NORSOK A-001

The preparation and publication of the NORSOK standards is supported by OLF (The Norwegian Oil Industry Association) and TBL (Federation of Norwegian Manufacturing Industries). NORSOK standards are administered and issued by NTS (Norwegian Technology Centre).

Annex A, B and C are normative.

Introduction

This revision replaces NORSOK standard M-CR-710, rev. 1.

The intent of this NORSOK standard and the qualification process described herein is to assure that the non-metallic sealing material manufacturer has sufficient understanding and experience with the applicable materials to provide them with acceptable performance in the specified environment. Further that the manufacturer has sufficient insight and experience in the manufacture of such materials and that they are supplied with stable quality to meet given specifications.

The aim is that a successful qualification of a manufacturer and a specific material shall be valid for a majority of future development projects and for different operators.

The consideration of qualification of a manufacturer is at the discretion and determination of the Purchaser, normally on the basis of documentation provided by the manufacturer as required in this standard. A qualification by one Purchaser may also be accepted by subsequent Purchasers, provided the requirements in this standard are still complied with.

The Purchaser is responsible for ensuring if necessary with external competence that the manufacturers selected is qualified.

There does neither exist, nor are there currently plans to introduce a NTS/NORSOK qualification or approval scheme or a public listing of qualified manufacturers in this regard.

1 Scope

This standard defines the requirements for critical non-metallic (polymer) sealing, seat and back up materials for permanent use subsea, including well completion, X-mas trees, control systems and valves. The standard also applies to topside valves in critical gas systems.

This standard covers the requirements and procedures for qualification of non-metallic (polymer) materials for use in such applications.

2 Normative references

The following standards include provisions, which through reference in this text constitute provisions of this NORSOK standard. Latest issue of the references shall be used unless otherwise agreed. Other recognised standards may be used provided it can be shown that they meet or exceed the requirements of the standards referenced below.

STANDARDS		THERMO-PLASTICS	ELASTOMETERS
ASTM D 395	Standard Test Method for Rubber Property – Compression Set (method B).		X
ASTM D 638	Test Method for Tensile Properties of Plastics.	X	
ASMT D 695	Test Method for Compressive Properties of Rigid Plastics.	X	
ASTM D 746	Test Method for Brittleness Temperature of Plastics and Elastomers by Impact.	X	X
ASTM D 790	Test method for Flexural Properties of Un-reinforced and Reinforced Plastics and Electrical Insulating Materials.	X	
ASTM D 792	Test Methods for Specific Gravity and Density of Plastics by Displacement.	X	X
ASTM D 1414	Methods of Testing Rubber O-rings.		X
ASTM D 1415	Standard Test Method for Rubber property – International hardness (IRHD).		X
ASTM D 1525	Test Method for Vicat Softening Point for Plastics.	X	
ASTM D 2240	Test Method for Rubber Property – Durometer Hardness (Shore A/ D).	X	X
ASTM D 2990	Test methods for Tensile, Compressive and Flexural Creep and Creep Rupture Test of Plastics.	X	
BS 1806	Standard Inch Sizes of O-rings.		X
BS 6442	Specification for limits of surface imperfections on elastomeric toroidal sealing rings (O-rings).		X
DIN 53453	Testing of Plastics, Impact Flexural Test.	X	
ISO 868	Determination of indentation hardness by means of a Durometer (Shore A/ D hardness).	X	X
ISO 1432	Rubber vulcanised – low temperature stiffening (Gehman test).		X
ISO 1817	Vulcanised rubbers – Resistance to liquids – methods of tests.		X
ISO D34	Tear resistance, method A.		X
ISO R 812	Method of test for temperature limit of brittleness for vulcanised rubbers.		X
Description of Arrhenius method: ASTM D 3032; Method of testing hook-up wire insulation.		X	X

3 Definitions and abbreviations

3.1 Definitions

Accelerated test	A test at temperatures and selected pressure values chosen to accelerate seal degradation effects.
Compression Set, %	100 x [(Original seal height – Post test seal height) / Interference].
Elastomer	A material compounded from polymers and other constituents, then cured to form a rubbery material.
Fluid (or medium)	A medium which flows without recovery such as a gas, liquid, supercritical gas, or a mixture of these.
Interference	[(Original seal cross section) – height of spacer bar (seal fixture recess)] each measured in same direction as direction of compression.
May	Verbal form used to indicate a course of action permissible within the limits of the standard.
Polymer	A high molecular weight organic compound, natural or synthetic, whose structure can be represented by a repeated small unit. Polymer includes thermoplastic and thermoset materials, of which elastomers are a sub-category.
Rapid Gas Decompression (RGD)	Rapid pressure drop in a gas containing system causes the fluid trapped inside an elastomer (polymer) to expand. The pressure drop rate must be faster than the diffusion rate of the fluid inside the polymer.
Room Temperature	20°C (+/- 2°C).
Seal cross-section	The free height of the seal at room temperature, measured normal to diameter in the direction of compression in the test. The measurement shall be taken at three circumferentially equidistributed positions.
Seal Type	A seal design of specified geometry, size and orientation.
Shall	Verbal form used to indicate requirements strictly to be followed in order to conform to the standard and from which no deviation is permitted, unless accepted by all involved parties.
Should	Verbal form used to indicate that among several possibilities one is recommended as particularly suitable, without mentioning or excluding others, or that a certain course of action is preferred but not necessarily required.
Thermoplastic	A material capable of being repeatedly softened by an increase in temperature and hardened by a decrease in temperature. Applicable to those materials whose change upon heat is substantially physical rather than chemical and that in the softened stage can be shaped by flow into articles by moulding or extrusion

3.2 Abbreviations

COC:	Certificate of Conformance
DSC:	Differential Scanning Calorimeter
DTMA/ TMA:	Differential Thermo Mechanical Analysis/ Thermo Mechanical Analysis
RGD:	Rapid Gas Decompression
QC:	Quality Control

4 Functional requirements

Material selection shall be based on evaluation of compatibility with service environment, functionality under service and the design lifetime. The following shall be considered as appropriate to the seal requirements and evaluated when selecting the material:

- Adequate physical and mechanical properties (hardness, tensile strength, elongation at break, modulus of elasticity, compression set, tear resistance, etc.)
- Resistance to high pressure extrusion or creep
- Resistance to thermal cycling, and dynamic movement
- Resistance against rapid gas decompression
- Long term behaviour
- Low temperature flexibility, ASTM D 746 and ASTM D 790

Clause 2 gives references to relevant standards for polymers, mainly thermoplastic materials and elastomeric materials. The standards describe test methodology for performing the tests. The test conditions and duration's shall be as described in this NORSOK standard in those cases where the NORSOK standard deviate from the referenced standards.

The polymers used shall be sourced from the same material manufacturers that performed the seal material qualification, using the same manufacturing route and procedures.

It is the responsibility of the equipment purchaser to provide all necessary information about service conditions and environment.

5 Documentation requirements

Required documentation of material properties is given in Table 1 and 2 for thermoplastic and elastomeric materials respectively. The requirements are valid both for seal materials as well as back-up materials when they are an integral part of the seal assembly. Requirements for documentation of properties and quality control are given. Each seal material used shall be traceable to the producer and his quality control documentation as required in Table 1 and 2 respectively. Each batch of material shall be supplied with a certificate of conformance (COC) and traceability as a minimum.

Tables 1 and 2 in this clause defines also the required minimum amount of production and quality control tests during manufacturing of seal material and back-up materials. The final procedures, with respect to key parameters and tolerances, shall be defined based on results from qualification testing performed according to this Standard. The purchaser shall define the necessary requirements with tolerances in the purchase specification.

Table 1 – Required documentation of thermoplastic material properties

Guidelines on selection of standards are given in parentheses. Characteristics, which are not relevant for expected service conditions and/or material type, may be omitted.

PROPERTIES	Documentation	QC test
• Softening point (ASTM D 1525).	D	
• Specific gravity (ASTM D 792).	D	B
• Hardness (ISO 868/ASTM D 2240, Shore D).	D	B
• Tensile properties and elongation (ASTM D 638).	D	B
• Compression properties (ASTM D 695).	D	
• Impact strength (DIN 53453).	D	
• Measurement of glass transition temperature by DSC.	D	
• Resistance to creep under permanent tensile and compressive loads (ASTM D 2990).	D	
• Ageing characteristics (Annex C).	D	

Table 2 – Required documentation of elastomeric material properties

Guidelines on selection of standards are given in parentheses. Characteristics, which are not relevant for expected service conditions and/or material type, may be omitted.

PROPERTIES	Documentation	QC tests
• Specific gravity (ASTM D 792)	**D**	**B**
• Hardness (Shore A/IRHD) (ISO 868, ASTM D 2240/ASTM 1415).	**D**	**B**
• Tensile and elongation properties (ASTM D 1414).	**D**	**B**
• Compression set (ASTM D 395/ASTM D1414) at 100°C for 7 days in Nitrogen atmosphere.	**D**	
• Low temperature characteristics by DSC or DTMA.	**D**	
• Tear resistance (ISO D 34).	**D**	
• Gehman plot (ISO 1432) +20°C to -20°C.	**D**	
• Ageing/RGD characteristics (Annex A/B).	**D**	

LEGEND for Table 1 and 2:

D: Properties to be documented for each supplier for each type of material.
Nominal values with tolerances shall be given (Data Sheet).

B: Properties to be documented on a batch-wise basis, minimum 3 samples per test per batch.
The acceptance criteria shall be established prior to the test and based on qualification test results.

6 Requirements for qualification of manufacturers

6.1 General requirements

In order to be qualified, the manufacturer shall document that he has manufactured materials and performed the testing required and that the material has met the relevant requirements in this standard.

The testing shall be performed on articles produced from specific polymer or rubber formulations and production procedures, made according to the normal production route and with regular production equipment.

This standard specifies the required minimum numbers of tests that must be performed in order to document the material suitability and compatibility with those test fluids specified in this standard, applicable to the intended seal application. The qualification testing shall apply for the polymer and elastomer materials on a one-off basis and the results shall be valid as long as the requirements stated in clause 6.2 are satisfied. For later supplies of identical material from the same manufacturer, a quality control of each batch of material shall be sufficient.

It will not be necessary to perform qualification testing, should well documented in-service experience with traceable production records and quality control documentation be available. Such documentation shall contain detailed information on service conditions such as time, temperature, pressure, fluid composition and chemicals added. An operating company can for example provide the documentation of flawless service. The service temperature must be in the same range as for the new application (maximum 10 °C below) and the service life shall be minimum 50% of design life.

6.2 Validity of qualification

The qualification shall apply for each specific seal material made of either polymer or elastomer materials and each specific manufacturer. The qualification shall be repeated if any changes have been made to the formulation of the product or the production route. This applies also for changes in raw materials or of sub-suppliers.
If production is carried out at different plants/locations, a separate qualification is required for each plant.

7 Qualification of elastomeric sealing materials

7.1 General

The technical requirements for testing of elastomeric seals are divided into two sections. The first section defines the ageing test requirements (Annex A) and the second (Annex B) defines the requirements for rapid gas decompression testing. The different test regimes shall be decided upon based on analysis of service requirements for the different equipment components and the material in question. Such assessment shall address all fluids coming into contact with the polymer and the nature of these fluids, both on the high pressure and low-pressure side. The service life of the seal material in the relevant service environment shall be evaluated using appropriate techniques.

7.2 Requirements for ageing tests

7.2.1 General

This standard defines test procedures for the prediction of the progressive degradation of elastomeric seals exposed to fluids at elevated temperatures and recommended pressure over an extended period of time. It is applicable where it is required to predict service life in a specific application or for the comparison of the performance of different seal materials. The prediction shall be based on tests performed at three different temperatures, all of which shall be above the defined service temperature.

An initial pre-check test shall be performed, if no previous knowledge about the behaviour of a seal material in a certain fluid exists. The test duration shall be sufficient to reach saturation of the fluid in the material at the test temperature. If no immediate changes in volume or weight occur, then the ageing test can start.

The supplier shall, during accelerated testing for obtaining results for extrapolation to service life, limit the test temperature so it can be ensured that the same chemical and/or physical processes will occur as during service.

The seal shall be tested in a constrained mode. The standard constraint shall be a flange or spigot/sleeve test arrangement whereby the seal is compressed by 20% of its original cross section. The flange or spigot/sleeve arrangement shall be submerged in the test fluid. No pressure difference over the seal is required. A standard O-ring seal shall be used, reference is made to Annex A, clause A.1.3.

When extrapolating data from the present procedures appropriate statistical techniques shall be applied. For example, if progressive degradation is dependent on a single chemical ageing process, a method based on Arrhenius equation/ method may be used as described in ASTM D 3032.

Test media, conditions, equipment, procedures and test report requirements are described in detail in Annex A.

7.2.2 Acceptance criteria

The acceptance criteria shall be established prior to commencing the ageing test. Based on previous experience the following criteria have been established as a baseline. The equipment manufacturer shall justify any relaxation of these requirements.

The following changes in properties are acceptable for elastomers:

- Hardness: + 10/-20 units (+5/-20 units when initial hardness is 90 Shore A)
- Swelling: +25 % / - 5 %
- Tensile strength, elongation and 50 % E-modulus: +/- 50 %

The tensile test results shall be used to extrapolate the service life according to Arrhenius equation.

7.3 Requirements for rapid gas decompression testing

7.3.1 General

This standard gives test procedures for measuring the effect on elastomeric materials of rapid de-pressurisation after periods at elevated temperature and high pressure in dry gaseous environments. Further, guidance notes for interpretation of the results are also given. The supplier shall evaluate those applications for which this failure mode is relevant.

The test media, conditions, procedure, test equipment, inspection procedure and test report requirements are described in Annex B.

7.3.2 Acceptance criteria

No seal cross section shall have a rating of more then three, reference is made to Annex B, clause B.4.

8 Qualification of thermoplastic materials

8.1 General

The technical requirements for testing of thermoplastic seals and back-up materials are described in Annex C. The different ageing test regimes shall be decided based on analysis of service requirements for the different equipment components and the material in question. Such assessment shall address all fluids that may come in contact with the polymer and the nature of these fluids, both on the high pressure and low-pressure side. The service life of the seal material in the relevant service environment shall be evaluated using appropriate techniques. For seal designs utilising metallic springs, only springs made of UNS R30035, R30003, Alloy 625, Alloy C276 or equivalent are acceptable.

8.2 Requirements for ageing tests

8.2.1 General

This standard defines test procedures for the prediction of the progressive degradation of thermoplastic seals and back-up rings exposed to fluids at elevated pressure and temperature over an extended period of time. It is applicable where it is required to predict service life in a specific application or for the comparison of the performance of different materials. This shall be achieved by testing at three different temperatures, all of which shall be above the defined service temperature.

An initial pre-check test shall be performed if no previous knowledge about the behaviour of a thermoplastic material in a certain fluid exists. The test duration shall be sufficient to reach saturation of the fluid in the material at the test temperature. If no immediate changes in volume or weight occur, then the ageing test can start.

The supplier shall, during accelerated testing for obtaining results for extrapolation to service life, limit the test temperature so that it can be ensured that the same chemical and/or physical processes will occur as during service.

When extrapolating data from the present procedures appropriate statistical techniques shall be applied. For example, if progressive degradation is dependent on a single chemical ageing process, a method based on Arrhenius equation/method may be used as described in ASTM D 3032.

Test media, conditions, equipment, procedures and test report requirements are described in detail in Annex C.

8.2.2 Acceptance criteria

The acceptance criteria shall be established prior to commencing the ageing test. Based on previous experience the following criteria have been established as a baseline. The equipment manufacturer shall justify any relaxation of these requirements.

The following changes in properties are acceptable for thermoplastic materials:

- Swelling: + 5 % / - 1 %
- Tensile strength, elongation and E-modulus: +/- 50 %
- Visual inspection: The materials shall show no tendency towards dissolution, cracking, blistering or physical deformation.

The tensile test results shall be used to extrapolate the service life according to Arrhenius equation.

Annex A
(normative)
Test media, conditions, equipment and procedures for ageing of elastomeric materials.

A.1 Test requirements

SAFETY PRECAUTIONS: The test procedures involve the use of pressurised fluids, which may be flammable and may have toxic effects. These media may be extremely hazardous if not handled correctly. The test operator shall ascertain and implement the appropriate safety precautions before commencing any test work.

A.1.1 Ageing test media

Tests fluids shall be representative of the seal application environment. The fluid exposures of the seal from both sides need to be considered.

A.1.1.1 Simulated production fluid

The production fluids are defined as either sour or sweet with composition as given in Table A.1 and A.2, respectively. Testing in sour service conditions will qualify the seal material for sweet service conditions as well. For wells with high H_2S levels (> 0,5 % of total), separate tests with higher H_2S level than required in table A.1 must be performed.

Table A.1 - Test condition for sour service conditions

Volume %	Composition
30	3% CO_2, 2% H_2S, 95% CH_4
10	Distilled water (conductivity < 5 μS)
60	70 % heptane, 20% cyclo-hexane, 10% toluene

Table A.2 - Test conditions for sweet service conditions.

Volume %	Composition
30	3% CO_2, 97% CH_4
10	Distilled water (conductivity < 5 μS)
60	70 % heptane, 20% cyclo-hexane, 10% toluene

The composition of all fluids to which the test seal is exposed shall be detailed in the Test Report.

A.1.1.2 Other test fluids

In addition to production fluids, it will be necessary in many instances to perform application specific testing of materials in contact with e.g. drilling fluids, scale inhibitors, hydrate inhibitors, well stimulation fluids and corrosion inhibitors. This will be specific for applications and will not be covered by the general test environment. Specific test procedures must be written detailing the exposure environment. The test methodology shall be according to this standard.

A.1.2 Ageing test conditions

A.1.2.1 Test temperatures

Since accelerated results are required for extrapolation, tests shall be run at a minimum of three test temperatures, all of which are above service temperature. Particular care is required to ensure that reactions occurring are representative of those which will occur at the service temperature. The limitations to extrapolation regarding temperature inherent in the Arrhenius method shall apply.

Test temperature versus time details shall be fully described in the Test Report.

A.1.2.2 Test pressure

A test pressure of 100 (+/- 10) bar or higher shall be used. This pressure shall be attained by pressurisation of the gas mixture after heating to the chosen temperature.

The pressure versus time details shall be fully described in the Test Report.

A.1.2.3 Exposure period

The exposure period for lifetime predictions shall take account of time to reach saturation of the test samples and be sufficiently long as to allow for reliable extrapolation according to requirements for methods such as Arrhenius plot.

A.1.3 Ageing test specimen

A.1.3.1 Standard O-ring size

A standard O-ring seal of size No. 325 (Cross section: 5,33 mm, ID: 37,47 mm) according to BS 1806 shall be used.

A.1.3.2 Constraint level

The elastomeric seal shall be tested in a constrained mode. The standard constraint shall be a flange or spigot/sleeve arrangement whereby the standard seal is compressed by 20% (+/-2%) of its original section diameter. The “spacer” height or seal-fixture recess height used to control the seal compression must be measured.

Fixtures used for compression of samples during testing shall be compatible with the test fluid.

A.2 Equipment

A.2.1 Test vessel

The test vessel shall be rated for use at the test temperatures and pressures and the metallic materials shall be compatible with the test fluid. The fluid capacity shall be such that the ratio of fluid to seal volume is greater than 25:1. The vessel shall be capable of being purged to remove air before testing. The seals shall be tested in a constraint condition and exposed to the test fluid from both sides.

A.2.2 Exposure in fluid mixtures

The samples shall be exposed in the oil phase of the prescribed production fluids.

A.3 Test procedure

A.3.1 Introduction

The following clauses describe the test procedure to be used in the qualification of elastomeric seals for critical applications.

A.3.1.1 Measurements

This test procedure provides for the determination of change or rate of change of various physical properties of the test seal. All property measurements shall be made in the free state at room temperature. The measurements in A.3.2 shall be made prior to exposure, whilst and those in A.3.4 – A.3.6 inclusive, shall be made after exposure. All measurements shall be recorded and reported in the Test Report.

A.3.1.2 Reproducibility

The intention is to extrapolate or interpolate performance and thus three test samples shall be run at each of a minimum of three exposure periods, for each of the three test temperatures. Altogether a minimum 30 samples shall be tested, including those necessary to obtain original material properties.

A.3.2 Measurements prior to test

A.3.2.1 Initial cross-sectional diameter, weight and volume of seal

All seal shall confirm to BS 6442, category N. Each seal shall be measured at three circumferentially equidistributed positions in the direction of compression, with an accuracy of ± 0,05 mm. The seal fixture recess height or "spacer" used for controlling the compression shall be measured to the same accuracy. The volume shall be measured by weighing the seal dry and submerged in a fluid of known density, of which has no effect on the material. The individual and mean measured values shall be reported together with mean interference of the seal installed in its test fixture.

A.3.2.2 Initial hardness, tensile strength, elongation and E-modulus

The initial hardness, tensile strength, elongation and E-modulus (50%) of virgin material shall be measured according to the referenced procedures. The initial hardness of each seal shall be measured at three equidistributed positions on the seal before mounting in the test fixture.

The individual and mean measured values shall be reported.

A.3.3 Main test sequence

- Clean test vessel and housings.
- Install test seals in the vessel.
- Fill vessel with test liquid.
- Purge vessel with nitrogen gas (< 5 ppm O_2) through the test liquid.
- Fill vessel with specified test gas mixture.
- Heat vessel to the specified test temperature
- Pressurise the specified gas mixture to the specified test pressure.
- Maintain test pressure and temperature for the specified test duration.

To reduce the risk of decompression damage, the pressure should ideally be released as slow as possible over weeks, but for practical reasons the following procedure is recommended:

- If test pressure higher then 100 bars, reduce to minimum 100 bar at an average rate of 0,5 bar pr. minute and leave the system at temperature for 24 hours.
- Cool down the autoclave to ambient temperature (recorded as the final date of ageing).
- Reduce remaining pressure at an average rate of less then 0,5 bar pr. minute and leave seals undisturbed until the next day.
- Remove fixture from vessel without disturbing seals.
- Carry out post-test procedures as described in A.3.4 – A.3.6.

A.3.4 Visual inspection for physical damage

The test specimen shall be visually inspected for external damage. The nature of any physical damage, set, embattlement, swell, blistering etc. and its location shall be recorded and reported in the Test Report.

A photographic record (10-x magnification) of specific features shall be included in the Test Report.

A.3.5 Compression set, weight and volume after test

The seal cross section height after test in the direction of compression, shall be measured 24 hours after removal from the test fixture, as in A.3.2.1. The measurement shall be reported as a "% Compression Set":

% Compression Set = 100 x [(Original seal height – Post test seal height)/ Interference]
(For definition of interference, see clause 3.1)

Measurement of weight and volume of the exposed seals shall be conducted immediately following the seal thickness determination. Percentage weight change as a result of exposure shall be calculated for each sample from:

% Weight change = [(Weight prior to exposure - Weight after exposure)/ Weight prior to exposure] x 100

Percentage volume change as a result of exposure shall be calculated for each sample from:

% Volume change = [(Volume prior to exposure - volume after exposure)/ volume prior to exposure] x 100

The weight and volume changes shall be averaged for each batch and the average reported in the Test Report along with the standard deviation.

A.3.6 Mechanical properties after test

The hardness, tensile strength, elongation and E-modulus shall be measured 24 hours after removal from the test vessel. The hardness shall be the average of the 3 highest readings of 6 readings, made at equidistant points on the seal.

A.4 Ageing test report

The report shall clearly state the following:

A.4.1 Test seal details

a. Manufacturer, seal type, manufacturer's compound reference number and size.
b. Seal material identification: generic polymer type (according to ASTM definition), batch number and cure date.

A.4.2 Test conditions

a. Test medium identification with detailed composition
b. Test temperature (°C) and temperature history
c. Test pressure (bar) and pressure history
d. Test duration (hours)
e. Date and time for start and end of test

A.4.3 Pre-test measurements

a. Seal dimensions, (mm), weight (g) and volume (cm^3).
b. Hardness, Shore A.
c. Tensile strength, elongation at break and E-modulus (50%).
d. Initial linear interference, (mm).

A.4.4 Post-test examination

a. Visual condition of the test specimens after test:
   Physical damage, set, embrittlement, etc.
b. Mean cross-section, (mm), weight (g) and volume (cm3).
c. Hardness, Shore A/ IRHD.
d. Compression set, %.
e. Tensile strength, elongation at break and E-modulus (50%).

A.4.5 Trends

Measurements from A.4.4 shall also, where possible, be presented graphically as a plot against a logarithmic time scale. Further, a graphical presentation according to Arrhenius method [logarithmic time against 1/T (absolute temperature (K))] based on trends for tensile properties shall be made. A best-fit line should be drawn to permit interpolation or extrapolation.

Annex B
(normative)
Test media, conditions, equipment and procedures for rapid gas decompression testing of elastomeric materials

B.1 Test requirements

SAFETY PRECAUTIONS: The test procedures involve the use of pressurised fluids, which may be flammable and may have toxic effects. These media may be extremely hazardous if not handled correctly. The test operator shall ascertain and implement the appropriate safety precautions before commencing any test work.

B.1.1 RGD test conditions

B.1.1.1 Introduction

In this standard, three alternatives are prescribed for the three variables, medium, temperature and pressure. These are given below in the following sections. The applicability of each of the alternatives will depend on the intended application for the seal. Successful testing at higher values of temperature and pressure will automatically qualify the material for use in applications where the service temperature and pressure lie below these values.

B.1.1.2 Test media

In most cases selection of dry gas media is considered to provide adequate indication of the resistance of the elastomer to RGD.

In the case of fluids, testing in pure CO_2 will qualify only for applications where CO_2 is the major component. Testing in the prescribed (CO_2/CH_4) mixtures will qualify for RGD applications in both sour well as well as sweet well conditions according to selected CO_2 level. Seal material ageing characteristics must be qualified according to relevant environment and requirements of Annex A in this Standard.

The three test media are defined in table B.1 below:

Table B.1 - Test media for RGD testing.

Sweet/ Sour Wells/ low CO_2	Sweet/ Sour Wells/ high CO_2	CO_2 Injection Wells
3 % CO_2, 97 % CH_4	10 % CO_2, 90 % CH_4	100 % CO_2

B.1.1.3 Test temperature

The test shall be conducted at one of the following temperatures; 100 °C, 150 °C or 200 °C. The test temperature shall be measured with calibrated temperature measurement equipment throughout the test and the measured temperature recorded and reported.

When performing the test, the temperature shall be increased to the test temperature and held for 10 minutes before applying the gas pressure. The temperature shall be maintained during the decompression stage of the test as far as possible and cooling only started after ambient pressure is reached.

B.1.1.4 Test pressure

The test shall be conducted at one of the following pressures: 150, 200 or 300 bar. The test pressure shall be measured with calibrated pressure measurement equipment through out the test and the measured test pressure reported at the end of the test.

B.1.1.5 Exposure period

The standard initial exposure period shall be 72 hrs. (+4/-0hrs) and is linked to the recommended seal cross section. Should the standard exposure period not be used, then justification for this shall be presented in the

test report together with supporting evidence that the initial exposure period has been sufficient to ensure gas saturation.

B.1.1.6 Decompression rate

The standard decompression rate shall be 20 – 40 bar per minute. The decompression rate shall be measured and the data included in the test report.

B.1.1.7 Number of decompression cycles

Each decompression cycle shall consist in reducing the gas pressure at the rate specified in B.1.1.6, while maintaining the test temperature as constant as possible. Following a hold period of 1 hour at ambient pressure, the vessel shall be re-pressurised to the test pressure and held at this pressure for 23 hrs. (+1/-0 hr) The decompression cycle shall then be repeated. The pressure cycling shall be continued for 10 cycles. Assessment of RGD damages according to B.4 shall be performed afterwards.

B.1.2 RGD test specimens

The standard test specimen shall be a O-ring seal of size No. 325 (Cross section: 5,33 mm, ID: 37,47 mm) according to BS 1806. A minimum of 3 seals shall be tested.

B.2 Test equipment

The test vessel shall be rated for use at the test pressure and temperature and shall be capable of being purged to remove air before testing is initiated.

Testing shall be performed on constrained O-rings. The standard constraint shall be between parallel plates whereby the standard O-ring is compressed by 20% (±2%) of its original section diameter.
Only RGD-resistant seals (spring energised lip seals) shall be used to seal the pressure vessel since they will be subjected to the same conditions as the test seals. The use of secondary seals is recommended. These should be situated close to the primary seals.

B.3 Test procedure

Measure the initial dimensions of the test seal and inspect and record any initial defects. All seals shall confirm to BS 6442, category N.

All tests shall be conducted with a minimum of three replicate seals

a) Mount the test seals on the test fixture.
b) Place the fixture inside the test vessel and seal the test vessel.
c) Purge the vessel with the test gas to remove any air.
d) Charge the vessel with the specified gas mixture to 10 bar before heating.
e) Heat the vessel to the test temperature. Record the initial temperature.
f) Charge the vessel with the specified gas mixture to the test pressure.
g) Maintain the test pressure and temperature for the exposure period, recording temperature and pressure at regular intervals.
h) The multiple decompression tests shall be performed according to B.1.1.7.
i) At the end of the test period, check and record temperature. Reduce the test pressure according to B.1.1.6.
j) Cool the vessel to ambient temperature.
k) Allow the vessel to stand with exit ports open for 24 +4/-0 hrs prior to dismantling.
l) Open the vessel and remove the test fixture.
m) Remove the test seals from the fixture. Record the visual appearance of the seals within 30 minutes.
n) Cut each test seal into 4 equal radial sections as shown in Fig. B.1 and examine the cross sections for internal cracks with a microscope or other visual means providing at least 10x magnification. Record the observations in accordance with the rating system described in B.4.1. Photographic documentation of appearance shall be included in the test report.

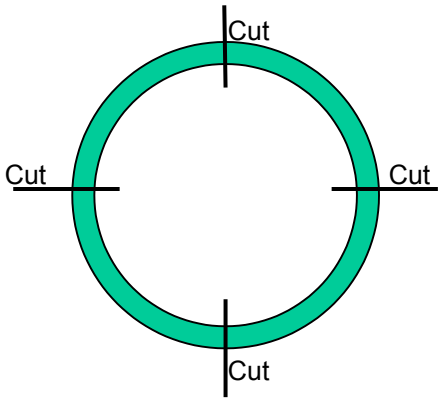

Figure B.1 - Sectioning of test O-rings in four.

B.4 Rating procedure for rapid gas decompression damage

The rating system is derived from internal crack measurements and although not quantitative, it is nevertheless objective and not dependent on the time of the observation.

B.4.1 Rating system

Examine four cut sections by microscopy using a magnification of at least 10x. For each section record a rating between 0 and 5 according to Table B.2 below.

Table B.2 - Description of rating number system for each seal cross section surface

Description	Rating #
No internal cracks, holes or blisters of any size	0
Less than 4 internal cracks, each shorter than 50% of cross section with a total crack length less than the cross section.	1
Less than 6 internal cracks, each shorter than 50% of the cross section, with a total crack length of less than 2,5 times the cross section.	2
Less than 9 internal cracks of which max. 2 cracks can have a length between 50% and 80 % of the cross section.	3
More than 8 internal cracks or one or more cracks longer than 80 % of the cross section.	4 *)
Crack(s) going through cross section or complete separation of the seal into fragments.	5 *)

*): Seals with rating 4 or 5 are not acceptable.

Record the rating of each seal by listing the individual ratings for each cut section in the order of the highest first to the lowest last. Thus a rating for a whole seal of 1000 means that one cut section had a few small cracks of rating #1 but no other cut section had any crack at all. Rating of 5422 would mean that one section had one or more cracks going through seal cross section, one section had more than 8 cracks or at least one longer then 80% of seal cross section and the other two sections had less than 6 cracks of which each were shorter then 50% of seal cross section.

The "overall rating" for a set of three replicate seals is defined on a worst case basis as the highest rating for each cross section over the three replicates. Thus, if the rating for 3 seals were 1110, 3110, 2220, the overall rating would be 3220 and the seal had passed the test.

Comparisons between materials shall be based on overall ratings made on the same basis and with the same number of replicate seals.

Note
The photograph in figure B.2 illustrate typical RGD damage and application of the rating system. Tensile tests and hardness tests on O-rings with internal RGD damage are not considered meaningful and so have been intentionally excluded.

Fig. B.2 - Photograph of typical cross section with RGD damage (Rating # 4)
(Reproduced by courtesy of MERL, UK).

B.5 RGD Test report

The test report shall state the following:

a) Date of tests.
b) Seal reference information, batch number, polymer type, trade name, manufacturer, date of curing, etc.
c) Composition of test medium.
d) Initial observations.
e) Temperature records, including heating and cooling.
f) Test pressure records, including decompression and re-pressurisation.
g) Rapid gas decompression damage by rating system in B.4.1.
h) Any other pertinent observations or records, e.g. photographs of seal sections and any non-conformances from the described test procedure.

Annex C
(normative)
Test media, conditions, equipment and procedures for ageing of thermoplastic materials and components

C.1 Test requirements

SAFETY PRECAUTIONS: The test procedures involve the use of pressurised fluids, which may be flammable and may have toxic effects. These media may be extremely hazardous if not handled correctly. The test operator shall ascertain and implement the appropriate safety precautions before commencing any test work.

C.1.1 Ageing test media

C.1.1.1 Simulated production fluid

Tests fluids shall be representative of the seal application environment. The fluid exposure of the seal from both sides needs to be considered. The production fluids are defined as either sour or sweet with composition as given in Table C.1 and C.2, respectively. Testing in sour service conditions will qualify the polymer material for sweet service conditions as well. For wells with high H_2S levels (> 0,5 % of total), separate tests with higher H_2S level than required in table C.1 must be performed.

Table C.1 - Test condition for sour service conditions.

Volume %	Composition
30	3% CO_2, 2% H_2S, 95% CH_4
10	Distilled water (conductivity < 5 μS)
60	70 % heptane, 20% cyclo-hexane, 10% toluene

Table C.2 - Test conditions for sweet service conditions.

Volume %	Composition
30	3% CO_2, 97% CH_4
10	Distilled water (conductivity < 5 μS)
60	70 % heptane, 20% cyclo-hexane, 10% toluene

C.1.1.2 Other test fluids

In addition to production fluids, it will be necessary in many instances to perform application specific testing of materials in contact with e.g. drilling fluids, scale-, hydrate-, wax-, asphalthene inhibitors, well stimulation fluids and corrosion inhibitors. This will be specific for applications and will not be covered by the general test environment. Specific test procedures must be written detailing the exposure environment. The test methodology shall be according to this standard.
The composition of all fluids to which the test seal is exposed shall be detailed in the Test Report.

C.1.2 Ageing test conditions

C.1.2.1 Test temperatures

Since accelerated results are required for extrapolation, tests shall be run at a minimum of three test temperatures, all of which are above service temperature. Particular care is required to ensure that reactions occurring are representative of those which will occur at the service temperature. The limitations to extrapolation regarding temperature inherent in the Arrhenius method shall apply

Test temperature versus time details shall be fully described in the Test Report.

C.1.2.2 Test pressure

A test pressure of 100 (+/- 10) bar shall be used. This pressure shall be attained by pressurisation of the gas mixture after heating to the chosen temperature.

The pressure versus time details shall be fully described in the Test Report.

C.1.2.3 Exposure period

The exposure period for lifetime predictions shall take account of time to reach saturation of the test samples and be sufficiently long as to allow for reliable extrapolation according to requirements for methods such as Arrhenius plot.

C.1.3 Ageing specimens

The sample materials shall be of the same material quality as the finished component, and be obtained from the same manufacturer. Where possible these shall be in the form of test specimens for tensile properties and E-modulus measurements, as described in the referenced standards in clause 2.

C.2 Equipment

C.2.1 Test vessel

The test vessel shall be rated for the appropriate test pressures and temperatures and the metallic materials shall be compatible with the test fluid. The fluid capacity shall be such that the ratio of the fluid to the total sample volume is grater that 25:1. The vessel shall be capable of being purged to remove air prior to testing. Samples shall be exposed from all sides.

C.2.2 Exposure in fluid mixtures

The material shall be exposed in the oil phase of the prescribed production fluids.

C.3 Test procedure

C.3.1 Introduction

The following clauses describe the test procedure to be used in the qualification of thermoplastic seals for critical applications.

C.3.1.1 Measurements

This test procedure provides for the determination of change of rate of various physical properties of the test seal. All property measurements shall be made in the free state at room temperature. The measurements described in C.3.2 shall be performed prior to exposure, whilst those in C.3.4 – C.3.6 inclusive shall be made after exposure. All measurements/observations shall be recorded in the Test Report.

C.3.1.2 Reproducibility

The intention is to extrapolate or interpolate performance and thus three tests samples shall be run at each of a minimum of three time periods for each of three test temperatures. Altogether a minimum of 30 samples shall be tested, including those necessary to obtain original material properties.

C.3.2 Measurements prior to test

C.3.2.1 Sample dimensions, volume and weight

The cross section dimensions shall be measured for all samples with an accuracy of ± 0,05mm. The volume and weight of all exposure samples shall be determined according to the displacement method in a suitable fluid prior to exposure. A balance with an accuracy of 1 mg shall be used.

C.3.2.2 Tensile properties

The tensile properties including E-modulus of test material shall be determined according to the referenced standards in clause 2. The individual and mean measured values shall be reported.

C.3.3 Main test sequence

- Clean test vessel.
- Install test samples in the vessel.
- Fill vessel with the test liquid.
- Purge vessel with an inert gas through the test liquid.
- Fill vessel with the specified gas mixture.
- Heat vessel to the specified temperature.
- Pressurise vessel to the specified test pressure with the specified gas mixture.
- Maintain temperature and pressure for the required test duration.
- Cool down vessel to ambient temperature (recorded as the final date of ageing).
- Depressurise vessel at a rate of 10 bar min-1 to atmospheric pressure.
- Retrieve the samples.
- Carry out the test procedures in C.3.4 – C.3.6.

C.3.4 Visual inspection for physical damage

The test specimens shall be visually inspected for external damage. The nature of any physical damage, internal de-lamination, swell, blistering etc., and its location shall be included in the Test Report.

A photographic record (10-x magnification) of the specific features shall be included in the test report.

C.3.5 Sample volume and weight

The volume and weight of each sample in the tested batch shall be measured in a suitable liquid by the displacement method. A balance with an accuracy of 1 mg shall be used. Percentage weight changes as a result of exposure shall be calculated for each sample from:

% Weight change = [(Weight prior to exposure - Weight after exposure)/ Weight prior to exposure] x 100

Percentage volume change as a result of exposure shall be calculated for each sample from:

% Volume change = [(Volume prior to exposure - volume after exposure)/ volume prior to exposure] x 100

The weight and volume changes shall be averaged for each batch and the average reported in the Test Report along with the standard deviation.

C.3.6 Tensile properties

The tensile properties including E-modulus shall be measured for 5 replicates, 24 hours after retrieval of each batch. The measured values shall be averaged for each batch and the average reported in the Test Report along with the standard deviation.

C.4 Ageing test report

C.4.1 Test seal details

a) Manufacturer, Seal/ Component type and sample dimensions.
b) Seal/ Component material identification: manufacturer designation, polymer type, polymer quality, batch/ lot number and production date.

C.4.2 Test conditions

a) Test medium identification with detailed composition
b) Test temperature (°C) and temperature history
c) Test pressure (bar) and pressure history
d) Test duration (hours)
e) Date and time for start and end of test

C.4.3 Pre-test measurement

a) Volume (cm^3) and weight (g) for all samples
b) E-Modulus (MPa)
c) Tensile strength (MPa) and elongation (%).

C.4.4 Post-test examination

a) Visual condition of the test piece after test, dissolution, cracking or physical deformation.
b) Volume and weight change (%) for each sample and mean value for all batches.
c) E-Modulus (MPa) mean and standard deviation for each sample and mean value for all batches.
d) Tensile strength (MPa) and elongation (%) mean and standard deviation for all batches.

C.4.5 Trends

Measurements from C.4.4 shall also, where possible, be presented graphically as a plot against a logarithmic time scale. Further, a graphical presentation according to Arrhenius method {logarithmic time against 1/T [absolute temperature (K)]} based on trends for tensile properties shall be made. A best-fit line shall be drawn in order to allow extrapolation or interpolation.