
**Oil and gas industries including
lower carbon energy — Non-metallic
materials in contact with media
related to oil and gas production —**

**Part 1:
Thermoplastics**

*Industries du pétrole et du gaz y compris les énergies à faible teneur
en carbone — Matériaux non-métalliques en contact avec les fluides
relatifs à la production pétrole et de gaz —*

Partie 1: Matières thermoplastiques



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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

This document was prepared by Technical Committee ISO/TC 67, *Oil and gas industries including lower carbon energy*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 12, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 23936-1:2009), which has been technically revised.

The main changes are as follows:

- added a short-term, single temperature 28-day non-H₂S material stability evaluation as Level 2;
- added a 56-day total duration target for the traditional three temperature Arrhenius material degradation evaluation as Level 3 and this is very similar to the previous edition;
- moved the life estimation analysis requirement to Level 4 and this new section has a 180-day total duration target for the Arrhenius material degradation evaluation;
- added life estimation analysis examples for plastics.

A list of all parts in the ISO 23936 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

Non-metallic materials are used in the petroleum, petrochemical and natural gas industries for a wide range of components. The purpose of this document is to establish requirements and guidelines for systematic and effective planning, for non-metallic material selection to achieve cost effective technical solutions, taking into account possible constraints due to safety and/or environmental issues.

This document will be of benefit to a broad industry group ranging from operators and suppliers to engineers and authorities. It covers relevant generic types of non-metallic material (e.g. thermoplastics, elastomers, thermosetting plastics) and includes the widest range of existing technical experience. Coatings are excluded from the scope of this document.

This document complements the ISO 15156 series on metallic materials in sour service. It differs in the form of guidance provided to the user related to the potential degradation of desired properties when used in equipment for oil and gas production environments. The ISO 15156 series provides application limits and qualification requirements for metallic materials in H₂S-containing environments, which are related solely to relevant environmentally assisted cracking mechanisms.

This document recognizes that a wider range of compounds and parameters influence the degradation of non-metallic materials and thus provides guidance to permit selection of materials for hydrocarbon exploration and production applications based upon stability in appropriate test conditions.

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Oil and gas industries including lower carbon energy — Non-metallic materials in contact with media related to oil and gas production —

Part 1: Thermoplastics

CAUTION — The non-metallic materials selected using this document are resistant to the given environments in the petroleum and natural gas industries, but not necessarily immune under all service conditions. This document allocates responsibility for suitability for the intended service in all cases to the equipment user.

1 Scope

This document gives general principles, specifies requirements and gives recommendations for the assessment of the stability of non-metallic materials for service in equipment used in oil and gas exploration and production environments. This information aids in material selection. It can be applied to help avoid costly degradation failures of the equipment itself, which could pose a risk to the health and safety of the public and personnel or the environment. This document also provides guidance for quality assurance. It supplements but does not replace, the material requirements given in the appropriate design codes, standards or regulations.

This document addresses the resistance of thermoplastics to the deterioration in properties that can be caused by physical or chemical interaction with produced and injected oil and gas-field media, and with chemical treatment. Interaction with sunlight and ionizing radiation are excluded from the scope of this document.

This document is not necessarily suitable for application to equipment used in refining or downstream processes and equipment.

The equipment considered includes, but is not limited to, non-metallic pipelines, piping, liners, seals, gaskets and washers.

Blistering by rapid gas decompression is not included in the scope of this document.

This document applies to the assessment of the stability of non-metallic materials in simulated hydrocarbon production conditions to aid the selection of materials for equipment designed and constructed using conventional design criteria. Designs utilizing other criteria are excluded from its scope.

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 75-1, *Plastics — Determination of temperature of deflection under load — Part 1: General test method*

ISO 75-2, *Plastics — Determination of temperature of deflection under load — Part 2: Plastics and ebonite*

ISO 178, *Plastics — Determination of flexural properties*

ISO 306, *Plastics — Thermoplastic materials — Determination of Vicat softening temperature (VST)*

ISO 527-1, *Plastics — Determination of tensile properties — Part 1: General principles*

ISO 604, *Plastics — Determination of compressive properties*

ISO 868, *Plastics and ebonite — Determination of indentation hardness by means of a durometer (Shore hardness)*

ISO 1183-1, *Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pycnometer method and titration method*

ISO 2039-2, *Plastics — Determination of hardness — Part 2: Rockwell hardness*

ISO 3451-1, *Plastics — Determination of ash — Part 1: General methods*

ISO 6721-11, *Plastics — Determination of dynamic mechanical properties — Part 11: Glass transition temperature*

ISO 11357-2, *Plastics — Differential scanning calorimetry (DSC) — Part 2: Determination of glass transition temperature and step height*

ASTM D638, *Standard Test Method for Tensile Properties of Plastics*

ASTM D648, *Standard Test Method for Deflection Temperature of Plastics Under Flexural Load in the Edgewise Position*

ASTM D695, *Standard Test Method for Compressive Properties of Rigid Plastics*

ASTM D785, *Standard Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials*

ASTM D790, *Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials*

ASTM D792, *Standard Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement*

ASTM E1640, *Standard Test Method for Assignment of the Glass Transition Temperature By Dynamic Mechanical Analysis*

ASTM D1708, *Standard Test Method for Tensile Properties of Plastics by Use of Microtensile Specimens*

ASTM D2240, *Standard Test Method for Rubber Property-Durometer Hardness*

ASTM D5630, *Standard Test Method for Ash Content in Plastics*

3 Terms, definitions and abbreviated terms

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions 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/>

3.1.1

batch

specified quantity of raw material, packaging material or product issued from one process or series of processes so that it could be expected to be homogeneous

[SOURCE: ISO 22716:2007, 2.3 with modification: “defined” changed into “specified”]

3.1.2**certificate of conformance**

document issued by the manufacturer in accordance with specific requirements

Note 1 to entry: The specific requirements shall be the requirement stated in this document or in the purchase order.

3.1.3**component**

individual, finished thermoplastic shape

3.1.4**compound**

intimate mixture of a polymer or polymers with other ingredients such as fillers, plasticizers, catalysts and colorants

[SOURCE: ISO 472:2013, 2.184]

3.1.5**conversion process**

manufacturing process that converts a compound into a plastic shape or component

3.1.6**end user**

oil and/or gas operating company

3.1.7**fluid**

liquid or gas

3.1.8**gasket**

sealing component compressed in a joint

3.1.9**glass transition temperature**

temperature of a thermoplastic material at which its mechanical properties change from elastic (glassy) to viscous (rubbery)

3.1.10**liner**

thermoplastic material for protection of medium-contacted surfaces of pipes, piping, pipelines or equipment

3.1.11**lot**

part of a batch or part of a continuously manufactured thermoplastic material

3.1.12**maximum operating temperature**

maximum temperature to which a component is subjected, including deviations from normal operations, such as start-up/shutdown

3.1.13**maximum rated temperature**

upper limit temperature that the material can be used regardless the environment/fluid

3.1.14**neat resin**

thermoplastic resin without additives

3.1.15

operating temperature

temperature to which a component is subjected during normal operation

3.1.16

pipeline

components of a pipeline system connected together to convey fluids between stations and/or plants, including pipe, pig traps, components, appurtenances, spools, risers, isolating valves, and sectionalizing valves

[SOURCE: ISO 13623:2017, 3.1.15, modified — Note 1 to entry has been deleted.]

3.1.17

piping

pipe or system of pipes for the transport of fluids and gases

Note 1 to entry: Interruption by different components such as pumps, machines, vessels, does not preclude integration into one single piping system.

3.1.18

preconditioning

exposure to specified conditions in relevant fluids prior to ageing

3.1.19

room temperature

temperature of $(23 \pm 2) ^\circ\text{C}$

3.1.20

seal

deformable polymeric device designed to separate different environments

3.1.21

swelling

increase in volume due to absorption of fluids

3.1.22

thermoplastics

plastics that are capable of being repeatedly softened by heating and hardened by cooling through a temperature range characteristic of the plastics and, in the softened state, of being repeatedly shaped by flow into articles by moulding, extrusion or forming

[SOURCE: ISO 15750-3:2022, 3.3]

3.1.23

washer

flat plate of a material with a centralized hole used to seat bolt heads and nuts, among others

3.2 Abbreviated terms

Af	acceleration factor
CDF	critical degradation factor
COC	certificate of conformance
COV	coefficient of variation
DMA	dynamic mechanical analysis
DSC	differential scanning calorimetry

FEP	fluorinated ethylene propylene
HDT	heat distortion temperature
KCl	potassium chloride
PA	polyamides
PFA	perfluoroalkoxy
PEEK	polyether-ether ketone
PTFE	polytetrafluoro-ethylene
PTFEm	polytetrafluoro-ethylene modified
PVDF	polyvinylidene fluoride
QC	quality control
ST	softening temperature

4 Technical requirements

4.1 General requirements

Thermoplastic selection depends upon material property characteristics and fluid ageing behaviour. This document establishes four levels of testing for the purpose of comparing the properties of various thermoplastic materials. Material property data will be generated at the four levels to allow consistent comparison of the subject materials. Generic data shall be derived per Level 1 and Level 2 including threshold criteria, solely for the purpose of producing information for preselection. Where the user requires accelerated ageing material stability data in a multi-phase H₂S containing fluid, Level 3 shall apply. Where the user requires the material stability data beyond 56 days and an attempted long-term life estimation, Level 4 shall apply.

Level 1 conformance consists of the characterization and documentation of material properties in a material data report. It includes a COC for batch quality control testing. See [5.1](#) and [Table 1](#) for a list of the required material properties to be documented. Physical and mechanical properties shall be characterized on materials in their unaged condition. These standard properties assist with the selection of materials that meet a design specification. Some property tests are also used for quality assurance and control. Level 1 testing establishes a baseline for higher level testing.

Level 2 conformance pertains to material stability (ageing) behaviour and shall be accompanied by a report. [Clause 6](#) provides requirements for Level 2 conformance. The effect of the first three fluids listed in [6.2.4](#) on material properties shall be investigated with real-time ageing studies. A material's resistance to chemical/physical/mechanical change is determined.

Level 3 conformance pertains to material stability (accelerated ageing) behaviour and shall be accompanied by a report. [Clause 7](#) provides requirements for Level 3 conformance. The effects on material properties of three temperature aging evaluations shall be investigated. The intent of Level 3 evaluations is to accelerate material property changes specifically in multi-phase H₂S-fluids.

Level 4 conformance pertains to a material stability (long-term) assessment of 180 days or longer following the methodology as shown by [Annex B](#). Level 4 attempts life estimation and shall be accompanied by a report. [Clause 8](#) provides requirements for Level 4 conformance. The intent of Level 4 assessment is to predict the material's progressive degradation, hence conformance threshold recommendations are offered for life estimation purposes. The report shall include a thorough account of data analysis, extrapolation, life estimation, and statistical confidence. Users shall evaluate the

threshold criteria, life estimation results and all methodology to determine the suitability of materials for application.

All reports shall detail the testing and analysis that was performed as well as a reference to this document, i.e. ISO 23936-1:2022.

Laboratory studies using standard test conditions may not derive data that can be used for design purposes. The user may require fit-for-purpose testing or alternative testing to simulate production conditions to allow materials selection for final application. Component functional testing is not detailed in this document.

For some highly resistant polymers, the chemicals used for ageing in Level 3 and Level 4 will not have any significant thermal-chemical effect on the polymer even at higher temperatures. In such cases, the first observable change in property would be related to fluid absorption or melting phase change rather than a degradation mechanism induced by the chemical. Fully fluorinated polymers (e.g. PTFE, PTFEm, PFA, FEP) either unfilled or filled exclusively with carbon-based fillers (e.g. graphite, carbon black, carbon fibre) are known to behave as such and shall be exempt from Level 3 or Level 4 evaluation. Performance of Level 3 or Level 4 testing may reveal other polymers e.g. PVDF in fluid 3.1 and fluid 3.2 in [7.4](#) also falling into this category.

If blistering by rapid gas decompression is a concern, a test should be performed according to API 17J 4th edition, section 6.2.3.3.

4.2 Cautionary remarks

Designers should not assume that properties provided in a material data report as explained in [Clause 5](#) will accurately represent those properties found in finished product geometries. The method of conversion is known to have an impact on these properties and that impact should be accounted for during design.

Life estimation usefulness and certainty can increase when longer term data are used to establish the degradation trend. Level 3 testing at durations up to 56 days are most useful for shorter term (up to 1 year) life estimations and can have reduced certainty for long-term (greater than 1 year) life estimations. Level 4 testing requires up to 180 day or longer data in an effort to create higher certainty in long-term life estimation.

In some cases, progressive degradation of thermoplastics over long periods of time at temperatures well above the target service temperature is not observed. The data and the attempted life estimation are still valuable because they demonstrate material stability in that test environment.

4.3 Traceability

For a final component to maintain its conformance, it shall be made from a thermoplastic material that conforms with this document. The entire compound manufacturing process shall be fully traceable. Conformance records shall include a reference to this document, i.e. ISO 23936-1:2022.

Each compound and accompanying COC shall be traceable back to the compound manufacturer. Each company that participates in the manufacture of a compound that conforms with this document shall maintain traceability records for a minimum of 10 years that include its own manufacturing procedures, locations, and dates.

Further requirements on conformance and traceability over the supply chain can be found in relevant product standards and agreed between interested parties.

4.4 Test specimen identification

4.4.1 Coding overview

The specimen fabrication details shall be reported using the following identification code system:

- moulding (for individual codes see [4.4.2](#));
- orientation (for individual codes see [4.4.3](#));
- form (for individual codes see [4.4.4](#));
- post treat (for individual codes see [4.4.5](#));
- shaping (for individual codes see [4.4.6](#)).

The test specimen identification shall give the following information:

- a) test standard;
- b) specimen type;
- c) test speed;
- d) identification code.

EXAMPLE Sample test call out for an ISO 527-1 or ASTM D638 tensile test with injection moulded Type 1BA and Type V specimens respectively:

- 1) ISO 527-1, 1BA, 50 mm/min (MI/OA/FN/PA/SN);
- 2) ASTM D638 – TV, 2"/min (MI/OA/FN/PA/SN);

4.4.2 Moulding

Process used to convert a pellet, flake, powder, resin, etc. into a shape and is the final forming step:

- a) injection (MI): process of moulding a material by injection under pressure from a heated cylinder through a sprue into the cavity of a closed mould;
- b) compression (MC): load/pour material into mould, heat, and then consolidate melted polymer under uniaxial or isostatic compression;
- c) transfer (MT): process of moulding a material by passing it from a heated pot into the cavity of a closed, heated mould;
- d) extrusion (ME): transfer melted material into a shape using a die in a continuous process;
- e) rotational moulding (MR): load material in mould, heat and rotate, where inertial forces are used to consolidate the thermoplastic;
- f) casting (MS): transfer melted material into a mould with only the force of gravity acting on the thermoplastic;
- g) additive (MA): manufacturing methods that add layers of material by a melt process;
- h) new methods (MZ).

4.4.3 Orientation

Orientation pertains to the alignment of molecules or fillers compared to the longest dimension of the test specimen.

- a) none (ON): isotropic with insignificant x, y, z variation of properties;
- b) axial or flow direction (OA): longest dimension of the specimen is parallel to the orientation direction (i.e. injection moulded tensile bar);
- c) hoop or transverse (OT): longest dimension of the specimen is transverse to the orientation direction (i.e. flex specimen cut from hoop plane of an extruded tube).

4.4.4 Form

Form describes the source of the test specimen.

- a) final part (FN): the finished test specimen;
- b) rod (FR): solid cylinder;
- c) tube (FT): hollow cylinder;
- d) plate/sheet (FP): greater than or equal to 1,27 mm (0,050 inch) thick;
- e) film (FF): less than 1,27 mm (0,050 inch) thick;
- f) other finished (FO): part that the specimen is cut from.

4.4.5 Post treatment

Annealing or post cure comprising temperature cycles that alter the physical properties of the moulded form.

- a) none (PN): has not undergone a post-moulding heat cycle process and in which conditioning “dry as moulded” according to material standards, e.g. for PA-U 12 see ISO 16486-2;
- b) annealed (PA): has undergone a post-moulding heat cycle process, e.g. annealing for 48 h according to ISO 2578 or according to material standards;
- c) sintered (PS): hot sintering of a cold moulded precursor (or green parts);
- d) environmentally conditioned (PE), e.g. 98 % humidity at 50 °C for 48 h.

4.4.6 Shaping

Cite the process used to shape the specimen.

- a) net shape (SN): moulded into test specimen with no post process;
- b) machined (SM): material removed with cutting tool;
- c) stamped (SS): specimen die cut from a formed sheet or a sheet machined from a different form.

4.4.7 Test specimen fabrication for Level 2, Level 3 and Level 4 ageing experiments

Test specimens shall be produced using a single fabrication process suitable for test specimens.

4.5 Validation of conformance

A compound loses its conformance if changes are made to the raw material supply, the compound formulation or the compound manufacturing process. New testing is required for each desired level of conformance.

If Level 4 conformance is complete prior to change, new Level 4 testing is not required if Level 1, Level 2 and Level 3 test results are equal or improved compared to previous Level 1, Level 2 and Level 3 test results.

If compounding is carried out at different plants/locations, a separate Level 1 conformance is required for each plant.

Level 1 to Level 4 testing is not required on the component if no compositional changes have been made to the compound during the conversion process, regardless of the conversion process being used. The influence of the conversion process on the physical properties and fluid ageing behaviour of the component is outside the scope of this document. To evaluate this influence testing should be performed for the level of information needed, as described by [Clause 5](#) to [Clause 8](#), using test specimens that are manufactured from the conversion process used to produce the component.

5 Level 1 – Material property characterization

5.1 General

[Table 1](#) gives the required documentation of material properties. Requirements pertaining both to initial documentation of properties and batch QC are given.

[Table 1](#) documentation properties shall be presented as a material data report. This material data report shall be made available to all companies in the supply chain.

[Table 1](#) QC properties for each batch of thermoplastic shall be presented as a COC. This COC shall then be passed through each company within the supply chain to the end user. Each company in the supply chain shall ensure that the COC of the compound is available if requested.

The material's QC threshold criteria shall be based on batch testing of the compound. The material's QC threshold criteria shall be noted in the COC and in the material data report. The user can further specify the necessary requirements with tolerances in the purchase specification.

Table 1 — Required documentation for thermoplastic material properties

Properties	Documentation ^a	Minimum quality control tests ^b
Specific gravity: ISO 1183-1 or ASTM D792	D	B ^f g
Ash content for filled materials: ISO 3451-1 or ASTM D5630	D	-
Hardness IRHD/Shore D: ISO 868 or ASTM D2240 or Rockwell: ISO 2039-2 or ASTM D785	D	-
Tensile yield stress, maximum stress, modulus, strain at break at (23 ± 2) °C: ISO 527-1 or ASTM D638	D	B ^c
Tensile yield stress, maximum stress, modulus, strain at break: ISO 527-1 or ASTM D638 at maximum rated temperature.	D	-
Tensile strength, elongation at break: ASTM D1708 for sintered PTFE materials at (23 ± 2) °C	D	B ^c
Tensile strength, elongation at break: ASTM D1708 for sintered PTFE materials at 150 °C	D	-
Flexural modulus and strength at (23 ± 2) °C: ISO 178 or ASTM D790	D	B ^c
Flexural modulus and strength: ISO 178 or ASTM D790 at maximum rated temperature.	D	-
Glass transition temperature by DSC: ISO 11357-2	D ^d	-
Glass transition temperature by DMA: ISO 6721-11 or ASTM E1640	D ^d	-
Compressive strength at (23 ± 2) °C: ISO 604 or ASTM D695	D	-
Compressive strength: ISO 604 or ASTM D695 at maximum rated temperature.	D	-
HDT: ISO 75-1, ISO 75-2 or ASTM D648	D ^e	-
ST: ISO 306	D ^e	-
^a D: Properties to be documented for each manufacturer for each compound (material data report). ^b B: Properties to be documented on a batch-wise basis, minimum 5 specimens. ^c Either tensile or flexural shall be used for QC purposes. ^d Either DMA or DSC shall be used as appropriate for material. DMA full form should be provided. ^e Either HDT or ST shall be used as appropriate for material. ^f For specific gravity one specimen only. ^g Optional for PVDF unfilled.		

5.2 Reporting

5.2.1 Material data report

The material data report for each compound shall include as a minimum the following:

- manufacturer contact information;
- values for the documentation tests in [Table 1](#);
- test standards options such as method, specimen type, test speed;
- specimen identification call out for each test method;
- conformance statement with reference to the Level 2 fluid ageing report and a reference to this document, i.e. ISO 23936-1:2022;
- QC threshold criteria.

5.2.2 Certificate of conformance

The COC for each batch shall include as a minimum the following:

- a) manufacturer contact information;
- b) batch results including for averages, standard deviations, and threshold criteria of the QC tests in [Table 1](#);
- c) test standards options such as method, specimen type, test speed;
- d) specimen identification call out for each test method;
- e) batch production date and unique identification;
- f) signature approval by an authorized quality representative;
- g) conformance statement with reference to the material data report and Level 2 fluid ageing report, including a reference to this document, i.e. ISO 23936-1:2022.

6 Level 2 – Material stability (short-term)

6.1 General

This clause specifies the tests that shall be performed in order to document the 28-day maximum material stability temperature in the reported test fluid (see [6.2.4](#)). Test procedure is listed in [Annex A](#).

6.2 Test criteria

6.2.1 General

The conformance requires the following:

- a) exposure shall be performed in Fluid 2.1 through Fluid 2.3 listed according to [6.2.4](#).
- b) Fluid 2.4 may also be tested and reported as bespoke;
- c) results of physical and mechanical tests shall meet threshold criteria according to [6.2.6](#);
- d) Level 2 report shall be made available.

6.2.2 Exposure temperature

The upper temperature limit and knowledge of fluid compatibility shall be considered to select an exposure temperature. Exposures can be run at different temperatures for the different fluids. Exposures can be run multiple times in an attempt to find the high temperature limit. The reported temperature represents the highest exposure temperature that fulfils all specified requirements according to [6.2.6](#) at each duration according to [6.2.3](#).

6.2.3 Exposure durations

The exposure durations for Level 2 testing shall be 2 days, 7 days, 14 days, 28 days. Exposure can be longer as bespoke. Tolerances shall be in accordance with [Table A.2](#).

6.2.4 Test fluids

Air ageing is significant because many components and seals are exposed to air on the external surfaces of equipment while exposed to oilfield fluids on the inner surface. KCl brine is a common light-weight well fluid that identifies a material's resistance to swelling, polymer hydrolysis, and filler interface

degradation in an aqueous solution. Pure water is not common in service. Aromatic hydrocarbon exposure will identify a material's resistance to swelling and polymer softening. Bespoke fluids, consistent with a particular application environment or product specification may be used as Fluid 2.4.

Test fluids are:

- a) Fluid 2.1: air;
- b) Fluid 2.2: aqueous: 3 wt-% KCl in de-ionized or distilled water;
- c) Fluid 2.3: aromatic hydrocarbon: 70 % heptane, 20 % cyclohexane, 10 % toluene;
- d) Fluid 2.4: bespoke fluid such as field stimulation: any fluid including liquid/gas combinations can be used with the same test methodology.

6.2.5 Property test methods

The following properties shall be measured in unaged conditions and after each exposure duration so that changes in properties at each duration can be calculated:

- a) mass;
- b) volume;
- c) specific gravity;
- d) tensile maximum stress, yield stress, strain at break, and modulus; if material exhibits no yield stress, then stress at break shall be reported.

The properties specified by the tensile test standard used (e.g. ASTM D638, ISO 527-1, ASTM D1708) shall be reported. ASTM D1708, used for PTFE, does not include yield properties or modulus.

The tensile properties shall be calculated using the pre-aged dimensions.

For materials that yield in the unaged condition: if a zero-slope yield point is difficult to generate, then an offset yield point shall be used. The method adopted shall be reported and used consistently throughout the program. The 1 % offset yield point is given as a common method. If a different percent offset generates more consistent results, then that value shall be used throughout the entire test program and shall be reported. ASTM D638 describes the method for determining the offset yield point. ISO 527-1 describes the determination of the yield point of plastic tensile.

For materials that do not yield in the unaged condition but yield in the exposed condition, then yield stress changes shall be incorporated into the analysis. In this case, the initial yield stress shall be generated using a 1 % offset yield stress. If no initial 1 % offset yield stress exists, then the maximum stress shall be determined and documented. When gathering yield stress of the exposed specimens, the 1 % offset yield stress method shall be used.

When generating modulus data, multiple methods can be used. The same modulus calculation method shall be used for the entire test program.

NOTE A "Young's Modulus" or "Automatic Young's Modulus" is sometimes automatically generated by the tensile machine software and the software uses a least-squares fit to determine the initial linear part of the stress/strain curve. These can have variation, because there is rarely a "linear region" and this region location can change because of the immersions. Another method is the "Secant Modulus", which uses the zero point as the start value and a user selected value for the end point. Common methods are the 1 % or 2 % "Secant Modulus" and these typically have lower standard deviation than the "Young's Modulus" methods making the "Secant Modulus" values more consistent for judging percent changes in modulus properties after immersion.

Additional properties, such as Tg and hardness, may be gathered for information. It shall be ensured that the method is appropriate for the specimen in the wet condition.

6.2.6 Threshold criteria

To aid the evaluation of changes in measured material properties, the following threshold criteria shall be used:

- a) volume change: -1 % to +10 %;
- b) tensile maximum stress and yield stress change: ± 50 %;
- c) tensile strain at break change: ± 50 %;
- d) tensile modulus change: ± 50 %.

The percent change calculations are based on as-received samples.

If the standard deviation is larger than 50 % of the median value, the property shall be reported but not used for the threshold criteria.

For filled thermoplastics, the tensile strain at break change can be extended greater than +50 % change. In this case, the property shall be reported but not used for the threshold criteria.

For materials typically tested according to ASTM D1708 (PTFE type materials) tensile modulus and yield properties are not required. For the threshold criteria, the tensile properties called out for the particular tensile test standard applied shall be used.

NOTE Different examples show that a change of 50 % strain at break (or elongation at break) is established as threshold (e.g. at PPI TR-19 as option to specify a level of chemical resistance of thermoplastics including those for oilfield piping systems).

6.3 Preconditioning considerations

Preconditioning time and temperature for use in Level 3 and Level 4 exposures can be estimated from Level 2 data. Level 2 data reveal the mass change over time and 28 days is a sufficient amount of time to characterize saturation.

Preconditioning shall not be performed in liquids prior to Level 2 exposure. If additional work is done to further investigate preconditioning using Level 2 methods, this work should be reported.

Preconditioning of materials in air at a particular temperature and humidity level is allowed prior to Level 2, Level 3, or Level 4 exposure and, if performed, shall be reported (e.g. PA is typically preconditioned prior to fluid exposure testing according to ISO 16396-2 or ISO 1110).

6.4 Reporting

The Level 2 test report shall be done according to [Annex A](#). Level 2 conformance statements shall have a structure given below in the report citation example.

EXAMPLE Company XYZ Material ABC

ISO 23936-1:2022 Conformance to Level 2, Fluid 2.1 (180 °C, air)

ISO 23936-1:2022 Conformance to Level 2, Fluid 2.2 (240 °C, 3 % KCl)

ISO 23936-1:2022 Conformance to Level 2, Fluid 2.3 (240 °C, aromatic hydrocarbon)

ISO 23936-1:2022 Conformance to Level 2, Fluid 2.4 (220 °C, bespoke fluid description)

7 Level 3 – Material stability (accelerated)

7.1 General

Level 3 characterizes progressive degradation of materials by comparing physical and mechanical property changes after conditioning at three temperatures according to 7.2. This clause specifies the required tests and exposures that shall be performed to document the accelerated ageing of thermoplastic materials exposed to the fluids according to 7.4. Level 3 allows direct comparison of the changes in properties of candidate thermoplastic materials.

Ageing at higher temperatures thermally accelerates chemical reactions (if this occurs) between the fluids and the thermoplastic material, causing property values to change. Pre-determined threshold limits are used to evaluate the severity of the changes when comparing materials.

The Level 3 report shall provide property change data that allows the user to analyse the results as they see fit. A test procedure shall be followed according to Annex A.

Level 3 evaluations provide data sets at three different temperatures. Three different opportunities to reach the thresholds for each threshold property can be possible, due to three different temperatures. The highest test temperature is expected to produce the shortest time to the threshold limit. Often the data curves do not cross the threshold and the most important part of the analysis involves proper extrapolation of the data curves to the time that they cross the threshold criteria. The analyst shall use their judgement based on documented experience to isolate important data and choose the best curve fitting method.

7.2 Exposure temperatures

Exposures shall be run at three temperatures (T1, T2 and T3). The temperature difference shall be at least 10 °C and preferably 15 °C.

T1 temperature should be high enough for the property changes to cross at least one threshold criteria within 56 days or within the longest duration. A T1 temperature that results in the crossing of a threshold criteria in less than 7 days can be too high and shall either be lowered or well justified in the final report. Some materials might not cross any threshold criteria within 56 days.

7.3 Exposure durations

Level 3 exposures shall run for 56 days if no threshold criteria are met. Longer exposure durations are allowed.

Property tests shall be performed at a minimum of 4 durations.

Exposure at a temperature can be stopped before 56 days if three of the following four thresholds are met: changes in maximum stress, yield stress, modulus, and strain at break.

In the example below at T1, three property threshold criteria would have been met by day 21 to allow stoppage at that time. At T2, thresholds can or cannot have been met and the exposure was allowed to run the full 56 days. The T3 immersion study was also allowed to run the full 56 days. The 30-day time period is within the 28 ± 3 day tolerance.

EXAMPLE

T1	High temperature	225 °C	2 days, 7 days, 14 days, 21 days
T2	Medium temperature	215 °C	2 days, 7 days, 14 days, 30 days, 56 days
T3	Low temperature	200 °C	2 days, 7 days, 14 days, 30 days, 56 days

7.4 Exposure fluids

The multiphase H₂S fluid combines the effects observed in water and hydrocarbon exposures with an addition of H₂S. These multiphase fluids can be a combination seen in oil and gas environments and are consistent with existing material ageing standards.

Exposure fluids are:

- Fluid 3.1: multiphase 2 % sour gas:
 - water phase: 10 volume %, de-ionized water or distilled water;
 - hydrocarbon phase: 60 volume %, aromatic hydrocarbon (70 % heptane, 20 % cyclohexane, 10 % toluene);
 - gas phase: 30 volume %, sour gas (5 % CO₂, 2 % H₂S, 93 % CH₄ at 6 MPa at room temperature).
- Fluid 3.2: multiphase 10 % sour gas:
 - water phase: 10 volume %, de-ionized water or distilled water;
 - hydrocarbon phase: 60 volume %, aromatic hydrocarbon (70 % heptane, 20 % cyclohexane, 10 % toluene);
 - gas phase: 30 volume %, sour gas (5 % CO₂, 10 % H₂S, 85 % CH₄ at 6 MPa at room temperature).
- Fluid 3.3: as bespoke.

7.5 Initial swelling

In order to provide information on the physical effects of the liquid phase, the gas phase specified in Fluid 3.1 and Fluid 3.2 according to 7.4 shall only be applied after the seven-day data is acquired. Data at two and seven days shall be liquid only exposures with a nitrogen purge and a nitrogen gas cap.

7.6 Property test methods

The property test methods from Level 2 according to 6.2.5 shall apply to Level 3.

7.7 Threshold criteria

The threshold criteria for potentially reducing test durations are given in Table 2.

Test conditions shall be such that threshold criteria are exceeded for at least one Table 2 property, for at least one exposure temperature. If threshold criteria are not exceeded when ageing and increasing the exposure temperature is not feasible due to material temperature limit, this fact shall be documented and the data shall be reported.

Table 2 — Threshold criteria

Yield stress	Tensile property change			Maximum stress
	Strain at break	Modulus		
%	%	%		%
-50	-50	-50		-50

7.8 Preconditioning considerations

Preconditioning is exposure to specified conditions in relevant fluids prior to ageing. Preconditioning time and temperature for use in Level 3 exposures can be estimated from Level 2 data. Level 2 data

reveal the mass change over time and 28 days is a sufficient amount of time to characterize saturation. Preconditioning may be performed upon agreement between all interested parties.

Preconditioning of specimens such that every specimen experiences a liquid only exposure prior to a Level 3 multiphase fluid exposure is not common. If this preconditioning effort is performed, it shall be reported along with the data to justify a preconditioning effort. Preconditioning of materials in air at a particular temperature and humidity level is allowed and shall be reported if performed.

NOTE 1 For example, a material can be preconditioned for 7 days in a liquid prior to Level 3 exposure such that the 56-day specimens would have seen a total of 63 days of exposure.

NOTE 2 For example, PA is typically preconditioned prior to fluid exposure testing according to ISO 16396-2 or ISO 1110.

7.9 Reporting

The Level 3 test report shall be done according to [Annex A](#). Level 3 conformance statements shall have a structure given below in the report citation example.

EXAMPLE Company XYZ Material ABC

ISO 23936-1:2022 Conformance to Level 3, Fluid 3.1 (multiphase 2 % sour gas)

ISO 23936-1:2022 Conformance to Level 3, Fluid 3.2 (multiphase 10 % sour gas)

ISO 23936-1:2022 Conformance to Level 3, Fluid 3.3 (bespoke fluid description)

8 Level 4 – Material stability (long-term)

8.1 General requirements for Level 4 evaluation

This clause specifies test procedures for the assessment, evaluation and prediction of progressive degradation of thermoplastic materials exposed to fluids at elevated temperatures over extended periods of time. Level 4 exposure is applicable where it is necessary to forecast material life in a specific long-term application and for directly comparing the changes in properties of candidate thermoplastic materials.

Level 4 exposure can build upon the short-term material stability data from Level 2 and the thermally accelerated ageing data from Level 3.

Level 4 conformance pertains to a material stability (long-term) assessment of 180 days or longer following the methodology as shown in [Annex B](#).

This clause specifies the required types of tests that shall be performed in order to document the potential life estimation in Fluid 3.1, Fluid 3.2 or bespoke exposure environments. A test procedure shall be followed according to [Annex A](#).

8.2 Exposure temperatures

Exposures shall be run at three temperatures (T1, T2 and T3). The temperature difference shall be at least 10 °C.

T1 temperature shall be high enough for the property changes to cross at least one threshold criteria within 180 days within the longest duration. A T1 temperature that results in the crossing of a threshold criteria in less than 7 days can be too high and shall either be lowered or well justified in the final report.

8.3 Exposure durations

Level 4 exposures shall run for 180 days if no threshold criteria are met. Longer exposure durations are allowed.

The exposure durations for Level 4 testing should be 2 days, 7 days, 14 days, 28 days, 56 days, 90 days, 120 days, 180 days or more as appropriate. Tolerances shall be in accordance with [Table A.2](#).

Exposure at a temperature can be stopped before 180 days if three of the following four thresholds are met: changes in maximum stress, yield stress, modulus, and strain at break.

8.4 Exposure fluids

The Level 4 test fluid requirements shall apply according to [7.4](#).

8.5 Initial swelling

The Level 4 test fluid requirements shall apply according to [7.5](#).

8.6 Property test methods

The Level 4 property test methods shall apply according to [6.2.5](#).

8.7 Guidance for selection of Level 4 test methods

Because Level 4 evaluation is in place to allow forecasting of material property change beyond the time frame of the testing duration, a predictive assessment method is required. [Annex B](#) provides recommendations for selection of life estimation methods, but bespoke methods may be utilized where agreed with the end user. In any case, the selected methods shall allow for acceleration of the effects of the test fluids on the test specimens without changing the mode of degradation.

8.8 Preconditioning considerations

Preconditioning is exposure to specified conditions in relevant fluids prior to aging. Preconditioning time and temperature for use in Level 4 exposures can be estimated from Level 2 data. Level 2 data reveal the mass change over time and 28 days is a sufficient amount of time to characterize saturation. Preconditioning may be performed upon agreement between all interested parties.

Preconditioning of specimens such that every specimen experiences a liquid only exposure prior to a Level 4 multiphase fluid exposure is not common. If this preconditioning effort is performed, it shall be reported along with the data to justify a preconditioning effort. Preconditioning of materials in air at a particular temperature and humidity level is allowed and shall be reported if performed.

NOTE 1 For example, a material can be preconditioned for 7 days in a liquid prior to Level 4 exposure such that the 180-day specimens would have seen a total of 187 days of exposure.

NOTE 2 For example, PA is typically preconditioned prior to fluid exposure testing according to ISO 16396-2 or ISO 1110.

8.9 Evaluation of data for Level 4

This document acknowledges that due to variety of materials responses as a result of fluid exposure, an extrapolation for service life prediction might not be feasible. In the following subclauses, guidance rules are provided to assist in the selection of materials based upon their response in short-term tests. The reporting of data shall include a reference to this document, i.e. ISO 23936-1:2022. The basis of the analysis of data shall be clearly stated. Guidance procedures are given in [Annex B](#) to assist in the interpretation of data for each material response category.

By running exposure tests with test fluids at least three different elevated temperatures, three different times to reach the threshold boundary will result, with the highest test temperature producing the shortest time to threshold limit.

In some cases, the data do not cross the threshold boundary. The most important part of the analysis involves proper interpolation or extrapolation of the raw data curves to the time where they cross the threshold. Approaches to derive such analysis may use linear, polynomial or logarithmic curve fitting methods. The analyst shall use their best judgement to choose the best curve fitting method. Initial data points that were generated may also be ignored. The most care should be given to extrapolation of the raw data. Examples of data interpolation and extrapolation are given in [Annex B](#).

Plotting the log of time to threshold against the reciprocal of the test temperature should result in a linear trend, enabling an estimate of reliability at the operating temperature.

If progressive degradation is apparently dependent upon a single chemical ageing process, a method based on the Arrhenius relationship may be used as described in [Annex B](#). It is recommended that results always first be assessed on an Arrhenius basis. If an Arrhenius relationship does not exist or ageing does not occur, see [Annex B](#) for further guidance.

Test media, conditions, equipment, procedures and test report requirements are described in detail in [Annex A](#).

8.10 Threshold baseline

The additional requirements of Level 4 primarily involve the measurement of longer-term exposures and an attempt at life estimation. Life estimation requires a threshold baseline to be established for tensile properties in order to calculate the threshold criteria values. The analysis can be done with one baseline and the rationale for this choice shall be included in the test report. The same baseline shall be used for all tensile properties throughout a given analysis. The following four options can be used to calculate the percent change of aged data for the Level 4 analysis:

- a) as-received tensile properties;
- b) 2-day liquid exposure properties;
- c) 7-day liquid exposure properties;
- d) preconditioned properties.

8.11 Threshold criteria

The threshold criteria for Level 4 exposure are given in [Table 3](#). Other properties and other threshold criteria may be used by agreement between all parties.

Table 3 — Threshold criteria

Tensile property change			
Yield stress	Strain at break	Modulus	Maximum stress
%	%	%	%
-50	-50	-50	-50

NOTE Different examples show, that a change of 50 % strain at break (or elongation at break) is established as threshold (e.g. at PPI TR-19 as option to specify a level of chemical resistance of thermoplastics incl. those for oilfield piping systems or at API TR 17TR2 as option to predict lifetime of plasticized Polyamide for unbonded flexible risers).

Annex A (normative)

Test media, conditions, equipment and procedures for ageing of thermoplastic materials

SAFETY PRECAUTIONS — Test procedures involve the use of pressurized fluids, which can be flammable and can have toxic effects. These media can be extremely hazardous if not handled correctly. Multiphase mixtures give particular problems. Hydrogen sulphide is extremely toxic. The testing organization shall ascertain and implement the appropriate safety precautions before commencing any test work.

To avoid vessel burst, the maximum filling with liquid is 75 % of the total volume.

A.1 Test requirements

A.1.1 Test vessel

A.1.1.1 General

The test vessel shall be rated for use at the test temperature and pressure. The metallic materials shall be resistant to the test fluid. The vessel shall be capable of being purged to remove air before testing.

A.1.1.2 Vessel capacity

In multiphase liquid and gas containing exposures, such as Fluid 3.1 and Fluid 3.2, the vessel capacity shall be such that the ratio of the vessel volume to test specimen volume is at least 10:1.

In liquid only tests with an inert gas purge, the liquid to specimen volume ratio shall be at least 7:1.

The volume per cent of the fluids shall be presented.

A.1.1.3 Specimen location

The test specimens shall be immersed such that test liquid contacts all surfaces.

In multiphase liquid and gas containing exposures, such as Fluid 3.1 and Fluid 3.2, the specimens shall be immersed in the hydrocarbon liquid phase and shall not touch the water phase.

In liquid only tests, the specimens shall be fully immersed in the liquid phase.

In bespoke fluids, the location of the specimens in the vessel relative to the liquid and gas phases shall be documented.

A.1.2 Exposure media

A.1.2.1 General

Exposure fluids shall be stable at the exposure temperature. Changes in the fluid during the test, such as visual observations and the pH of aqueous solutions, can be monitored.

In addition to production fluids, it will be necessary in many instances to perform application-specific testing of materials in contact with other types, e.g. drilling fluids, scale inhibitors, hydrate inhibitors,

well stimulation fluids and corrosion inhibitors. Specific test procedures shall be written detailing the exposure environment. The test methodology shall be according to this document

A.1.2.2 Level 2 media replacement

Exposure durations of 28 days and less can be run without replacing liquid or gas media. If a vessel is opened for an intermediate duration, the gases shall be replaced. For exposures longer than 28 days, the liquid and gas media shall be replaced at 28 days and then at least every 56 days after that.

The liquid and gas replacement schedule shall be reported in the final report.

A.1.2.3 Level 3 and Level 4 media replacement

Exposure durations of 56 days and less can be run without replacing liquid or gas media. If a vessel is opened for an intermediate duration, the gases shall be replaced. For exposures longer than 56 days, the liquid and gas media shall be replaced at 56 days and then at least every 56 days after that.

The liquid and gas replacement schedule shall be reported in the final report.

A.1.3 Exposure temperatures

Since data from accelerated tests are required in Level 3, exposures shall be run at a minimum of three temperatures, all of which are above the temperature of interest. In addition, exposure at relevant temperature may be added. Suggested elevated exposure temperatures are given in [Table A.1](#); other temperature regimes may be chosen. The temperature difference between each of the three temperatures shall be at least 10 °C and preferably 15 °C. The tolerance on temperature is ± 2 °C. The device for temperature measurement shall be calibrated.

Table A.1 — Exposure temperature

Temperature of interest °C	Suggested elevated temperatures (T1, T2, T3) °C	Temperature of interest °F	Suggested elevated temperatures (T1, T2, T3) °F
22	37, 52, 67	72	99, 126, 153
60	75, 90, 105	140	167, 194, 221
75	90, 105, 120	167	194, 221, 248
90	105, 120, 135	194	221, 248, 275
105	120, 135, 150	221	248, 275, 302
120	135, 150, 165	248	275, 302, 329
135	150, 165, 180	275	302, 329, 356
150	165, 180, 195	302	329, 356, 383
180	195, 210, 225	356	383, 410, 437
195	210, 225, 240	383	410, 437, 464

A.1.4 Exposure pressure

A.1.4.1 General

If pressure will be logged during the exposure, the device for pressure measurement shall be calibrated. Temperatures and pressure should be monitored with 1-minute intervals.

In an unstirred liquid medium, it takes time to reach the solubility equilibrium of test gas components in the liquid. Therefore, stirring is recommended. In case of non-stirred autoclaves, it is recommended to let the equilibrium be reached over night.

A.1.4.2 Level 2 ageing

The vessel pressure will rise to match the vapour pressure of the fluids as a function of temperature. This applies to Fluid 2.2, Fluid 2.3, and bespoke fluids within 2.4. [Figure A.1](#) and [Figure A.2](#) show pressure versus temperature curves so that the operator can become familiar with the expected pressure for a particular temperature.

Reporting of the vessel pressure for liquid only exposures with a simple inert gas purge is optional.

The pressure history shall be reported if the exposure media includes a gas composition that is critical to the execution of the test program.

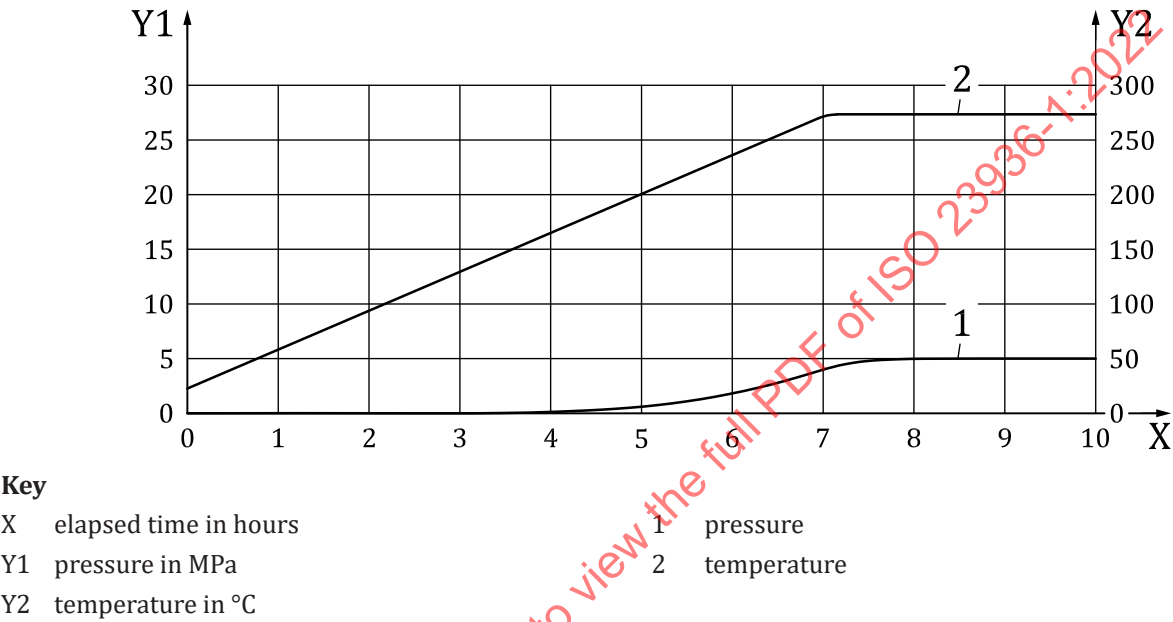


Figure A.1 — Pressure versus temperature curve with KCl Level 2.2

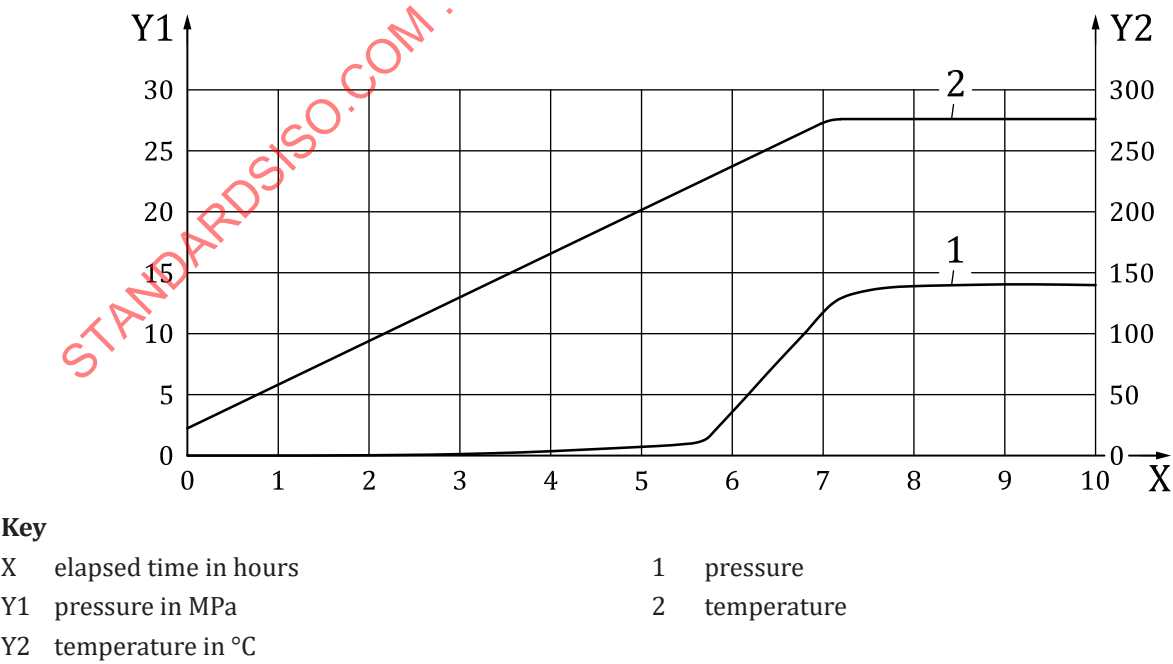


Figure A.2 — Pressure versus temperature curve with hydrocarbon Level 2.3

A.1.4.3 Level 3 and Level 4 ageing

Sour multi-phase exposures in Fluid 3.1 and Fluid 3.2 shall be pressurized to $(6 \pm 0,5)$ MPa at room temperature and sealed. The vessel pressure will rise as the temperature increases and will reach a new equilibrium pressure typically different than the initial 6 MPa charge pressure. Figure A.3 shows pressure versus temperature curve for the Fluid 3.2 multiphase sour mixture.

The entire pressure history shall be reported.



Key

X elapsed time in hours

1 pressure

Y1 pressure in MPa

2 temperature

Y2 temperature in °C

Figure A.3 — Pressure versus temperature curve with Fluid 3.2 multiphase sour aromatic

A.1.5 Exposure durations

Exposure duration tolerances are given in Table A.2.

Table A.2 — Exposure duration tolerances

Test duration	Tolerance
2 days to 10 days	± 1 day
11 days to 20 days	± 2 days
>20 days	± 3 days

Exposures may comprise individual test vessels which are locked for the period required, or run in one or more test vessels which are opened to allow specimen removal. The protocol used shall be reported accordingly.

The exposure time is the time the material was subjected to test pressure and test temperature. Time spent heating and cooling the test-cell shall not be included in the calculation.

Exposures will have extra time added between each duration to allow for cooling, gas depressurization, testing, and reheating. So, the total test program time will be longer than the sum of the exposure durations. Significant deviations of test temperature and/or pressure during an exposure shall be reported and justified technically.

A.1.6 Test specimens

Test specimens shall be fabricated with the same compound batch and methods throughout the test program. The fabrication method shall be specified by the identification code. The number of specimens shall be a minimum of five.

Mass and volume change specimens should be cut from the same sample used for the tensile specimens and can be either a tensile bar or a 25 mm × 25 mm specimen with the same thickness as the tensile specimens. The fabrication method shall be specified by the identification code. The number of specimens shall be a minimum of three.

The ageing of a stock shape, for subsequent fabrication of tensile test pieces, is not allowed. The thermoplastic material shall be exposed in unconstrained mode (i.e. free-standing, with fluid able to freely access all surfaces).

A.1.7 Tensile property measurements

Tensile properties of un-aged and aged specimens shall be measured at room temperature. All tensile properties shall be based on dimensions taken before specimens are aged. All tensile property measurements of a given material shall be based on the same test speed and specimen type. The test speed and specimen type used shall be reported.

Mass and volume properties of un-aged and aged specimens shall be measured at room temperature. The volume change shall be determined according to the displacement method according to ASTM D792. A balance with an accuracy of 1 mg shall be used.

A.1.8 Leaching considerations

Mass and volume properties after exposure are measured and can also be evaluated after a drying event to determine how much mass a material has lost during the exposures. Since a high-temperature fluid immersion/ageing test is extreme to most of the polymer resins, the lower-molecular-weight monomers, unbound substances, or the hydrolysis products inside the neat resin or filled specimens can be extracted by the surrounding ageing fluid. So, the mass change (the net weight change) of a specimen after the high-temperature fluid ageing should be considered to be a combination of the fluid absorption and the substance leaching. To identify these two processes quantitatively, an additional drying process of the aged wet specimen may be conducted at material specific temperature (e.g. 90 °C for PEEK) in an oven until its weight is at equilibrium. The difference between the weight of the aged wet specimen and the final dry weight of the aged specimen is the amount of the liquid absorbed, and the difference between the final dry weight of the aged specimen and the initial dry weight of the un-aged specimen is the amount of leaching. Therefore, the per cent absorption and the per cent leaching of the test specimen can be calculated by using [Formulae \(A.1\)](#) and [\(A.2\)](#):

$$\%A = \frac{(m_{aw} - m_{fd})}{m_{id}} \times 100 \quad (A.1)$$

$$\%L = \frac{(m_{fd} - m_{id})}{m_{id}} \times 100 \quad (A.2)$$

where

%A is the per cent absorption;

%L is the per cent leaching;

m_{aw} is the aged wet weight which is the weight of the specimen after the fluid ageing;

m_{fd} is the final dry weight which is the weight of the aged specimen after drying in an oven at material specific temperature until its weight is in equilibrium;

m_{id} is the initial dry weight, which is the weight of the un-aged specimen after drying in an oven at material specific temperature to an equilibrium in its weight.

This absorption and leaching property measurement procedure should be conducted with the un-aged and the aged specimens at room temperature.

If leaching measurements are performed, they shall be reported.

A.1.9 Visual inspection

The test specimens shall be visually inspected for salient features directly after removal from the vessels. The nature of any salient features shall be recorded including but not limited to:

- a) colour change of the surface after immersion in the medium;
- b) opacity after immersion;
- c) blister formation;
- d) crazing;
- e) crack formation.

A photographic record of specimens before exposure and after the final exposure shall be included in the test report. Representative salient features (visual changes) at intermediate exposures shall also be included in the test report.

A.1.10 Sample handling and storage procedures

For exposure fluids that are liquid at ambient pressure, test specimens from aged liquid shall be retrieved and shall be stored in fresh liquid (using the same neat liquid as used in the immersion test) until required for measurement. When weighing, sample shall be removed from the liquid, patted dry and weighed in air and then in water within 15 min to minimize evaporative losses. The sample shall be returned to liquid immediately until put back into the test vessel. When removing samples for tensile testing, each sample shall be removed individually from the storage liquid and tensile test, all within 30 min. This entire process shall be completed within one week of ageing completion.

Tensile tested material shall be stored in re-sealable polyethylene bags, appropriately labelled for 60 days without disposal after test report provided by laboratory. Mass and volume change specimens shall be added to the tensile storage bag.

A.2 Test procedure

A.2.1 General

The following subclauses describe the procedures to be employed to determine the chemical ageing characteristics of thermoplastic materials.

A recommended sequence of actions is given in this clause. The procedure used shall be documented in the test report. Operators shall perform appropriate nitrogen leak tests on the vessel system prior to running ageing tests with samples.

A.2.2 Sequence for liquid immersion test (vapour pressure)

The sequence for liquid immersion shall be as follows:

- a) add liquid(s) to vessel;
- b) immerse test specimens in a way to avoid specimen to specimen contact in the liquid;

- c) close vessel;
- d) commence monitoring the pressure and temperature with a sampling rate of once per minute except for exposure durations where rapid changes of parameters are expected (such as heating or cooling periods or pressure increase or decrease);
- e) purge vessel to 5 bar 5 times with nitrogen (inert) gas having oxygen content <5 ppm O₂ to remove most of the oxygen;
- f) raise temperature to exposure set point and hold the temperature for a predetermined time;
- g) cool the vessel to room temperature;
- h) open vessel, retrieve test specimens and keep them wet according to the procedure in [A.1.10](#);
- i) carry out post-exposure procedures of inspection (see [A.1.9](#)) and measurement (see [A.1.7](#));
- j) take pictures of tested tensile specimens including a scale.

A.2.3 Sequence for dual phase or multiphase fluid immersion test

In the case of two non-completely miscible liquids, the liquid mixture should be stirred. The sequence for multiphase fluid immersion test shall be as follows:

- a) add liquid(s) to vessel;
- b) immerse test specimens in a way to avoid specimen to specimen contact in the liquid;
- c) close vessel;
- d) commence monitoring the pressure and temperature with a sampling rate of once per minute except for exposure durations where rapid changes of parameters are expected (such as heating or cooling periods or pressure increase or decrease);
- e) purge vessel to 5 bar 5 times with nitrogen (inert) gas having oxygen content <5 ppm O₂ to remove most of the oxygen;
- f) add test gas mixture at room temperature to a total pressure of (6 ± 0,5) MPa; if individual gases are used, fill to correct partial pressures in the order of lowest liquefaction pressure;
- g) raise temperature to exposure set point and hold the temperature for a predetermined time;
- h) cool the vessel to room temperature;
- i) reduce remaining pressure safely at a maximum rate of 0,1 MPa/min, neutralizing H₂S, if used, with a scrubber;
- j) flush vessel with nitrogen to remove remaining H₂S, if used, via a scrubber;
- k) open vessel, retrieve test specimens and keep them wet according to the procedure in [A.1.10](#);
- l) carry out post-test procedures of inspection ([A.1.9](#)) and measurement (see [A.1.7](#));
- m) take pictures of tested tensile specimens including a scale.

A.3 Test report for Level 2, Level 3, and Level 4

A.3.1 General

The following items shall be addressed in the final test report for all Level 2, Level 3 and Level 4 reports:

- a) conformance statement that details which clause of this document has been satisfied by the testing and a reference to this document, i.e. ISO 23936-1:2022;
- b) a reference to the test standards used including publication year and additional details required according to [5.1](#);
- c) compound manufacturer, contact information, manufacturer's compound name/number or other reference, recipe revision level, batch/lot number and moulding date;
- d) specimen identification call-out for each test method using the coding system in [4.4](#);
- e) exposure media (liquids and gas) identification with classification, sub-classification where appropriate, and detailed composition;
- f) fluid replacement durations and results of fluid monitoring, if performed;
- g) plot of test temperature (°C) and pressure (MPa) versus time for multi-phase test fluids; liquid only exposure plots are not required;
- h) dates for start and end of test;
- i) test procedure based on [Clause A.2](#);
- j) median material properties values for each duration;
- k) specific gravity, mass and volume with descriptive test method and sample identification code;
- l) maximum stress, yield stress, strain at break, modulus, with descriptive test method and sample identification code;
- m) table of median results for physical property measurements from [6.2.5](#) and threshold criteria;
- n) table of property percent change data;
- o) graphical percent change representation using dotted lines showing the threshold criteria;
- p) description of visual inspection and condition at the final duration;
- q) pictures of specimens before exposure and after final duration;
- r) a photographic record of representative damage features;
- s) raw data, standard deviation, COV, median and intermediate duration pictures upon request;
- t) pressure and temperature log for all test durations, if the bespoke fluid contains a gas composition that contributes to the material property changes. The application of a nitrogen gas cap to a bespoke hydrocarbon fluid does not need the log presented;
- u) preconditioning details;
- v) leaching results.

A.3.2 Test report for Level 2

A single thermoplastic material can have multiple Level 2 conformance statements and test reports. Test reports can contain numerous data sets and resulting conformance statements. [Table A.3](#) provides an example of a test report that covers a single data set.

Compound manufacturers should list conformance statements on their material data report, if the supporting report is available.

Table A.3 — Example test report for Level 2

AcmeManf PhenDura XTRA-6880 conforms to ISO 23936-1:2022		
Conformance statement	-	ISO 23936-1:2022 Conformance to Level 2, Fluid 2.2 (250 °C, 3 % KCl)
Client address	-	ToolCo Inc. Energy Dr, Denver, CO, USA
Compound manufacturer	-	AcmeManf,
Compound name/number	-	PhenDura XTRA-6880
Material / reinforcement	-	PEKK / unfilled
Recipe revision level	-	n/a
Lot/batch no.	-	FF2344rw4r/07
Moulding date	-	Q4 2015
Ageing test conditions		
Fluid	Classification	ISO 23936-1:2022 Level 2.2
	Description	3 wt-% KCl
	Liquid	30 grams KCl in 970 ml of distilled water
	Gas Pressure	Nitrogen purge 20 psi to 50 psi gas cap
Volume ratio minimum	-	21:1
Exposure temperature	°C (°F)	250 (482)
Pressure at temperature	MPa (psi)	4,5 (700)
Exposure durations	days	2, 6, 14, 30
Specimen preconditioning	-	None
Leaching	%	None
Test lab	-	Ace Test Lab
Address	-	123 Sample Rd, Testing, LA, USA
Test date	-	10 June 2015 - 25 October 2015
Test gas mixture or individual gases certified	YES/NO	YES
Pressure calibration available	YES/NO	NO
P/T logs available	YES/NO	NO

Test description	Standard	Specimen identification
Tensile properties	ASTM D638 (T1, 50 mm/min)	(MI/OA/FN/PA/SN)
Mass and volume of tensile bars	ASTM D792	(MI/OA/FN/PA/SN)
Hardness of tensile bars	ASTM D2240 Shore D	(MI/OA/FN/PA/SN)

General test details						
Ageing days	0	2	6	14	30	Threshold criteria
Planned days and tolerance	0	2 ± 1	7 ± 1	14 ± 2	28 ± 3	
Fresh liquids	yes	no	no	no	n/r	
Fresh gas	yes	yes	yes	yes	n/r	
Median values for each test duration						
Tensile strength (MPa) ^a	95,0	92,7	93,0	90,1	89,3	n/r
Yield stress (MPa) ^a	95,0	89,0	88,8	87,0	85,2	n/r
Nominal strain at break (%) ^a	42,0	29,0	26,4	24,8	23,3	n/r
Modulus (specified 2 % secant method) (GPa) ^a	4,11	3,90	3,73	3,60	3,57	n/r
Mass (grams) ^b	8,506	8,621	8,644	8,652	8,641	n/r
Specific gravity ^b	1,356	1,388	1,401	1,405	1,408	n/r
Percent change in properties						
Tensile strength Δ (%) ^c	0 %	-2 %	-2 %	-5 %	-6 %	±50 %
Yield stress Δ (%) ^c	0 %	-6 %	-7 %	-8 %	-10 %	±50 %
Nominal strain at break Δ (%) ^c	0 %	-31 %	-37 %	-41 %	-45 %	-50 %
Modulus (specified 2 % secant method) Δ (%) ^c	0 %	-5 %	-9 %	-12 %	-13 %	±50 %
Mass Δ (%) ^c	0 %	1 %	2 %	2 %	2 %	n/r
Volume Δ (%) ^c	0 %	2 %	2 %	3 %	3 %	-1 % to +10 %
^a Measured data shall be presented with at least three significant figures.						
^b Measured data shall be presented with at least three decimal places.						
^c Calculated percent change data shall be presented with no decimal places.						

Property percent changes charts are shown in [Figure A.4](#) and [Figure A.5](#).

The pressure and temperature chart is shown in [Figure A.6](#).

The appearance of the specimens before and after ageing is shown in the [Figure A.7](#).

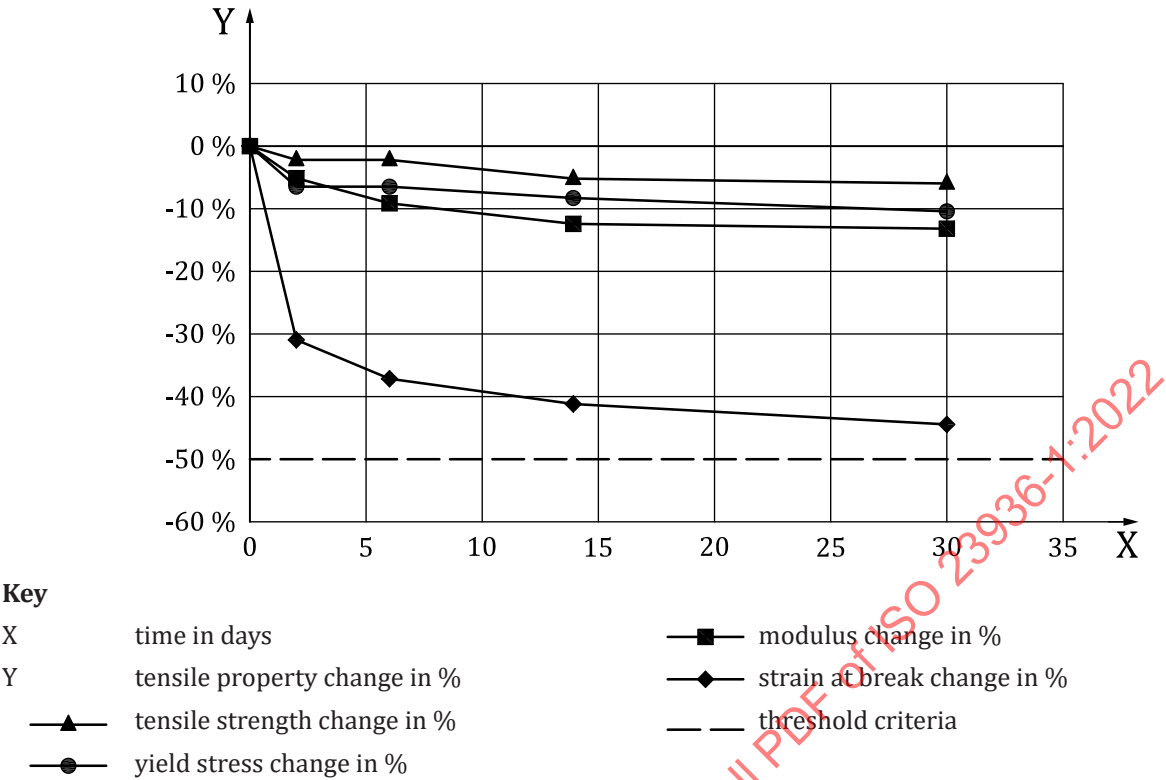


Figure A.4 — Tensile property change percentages

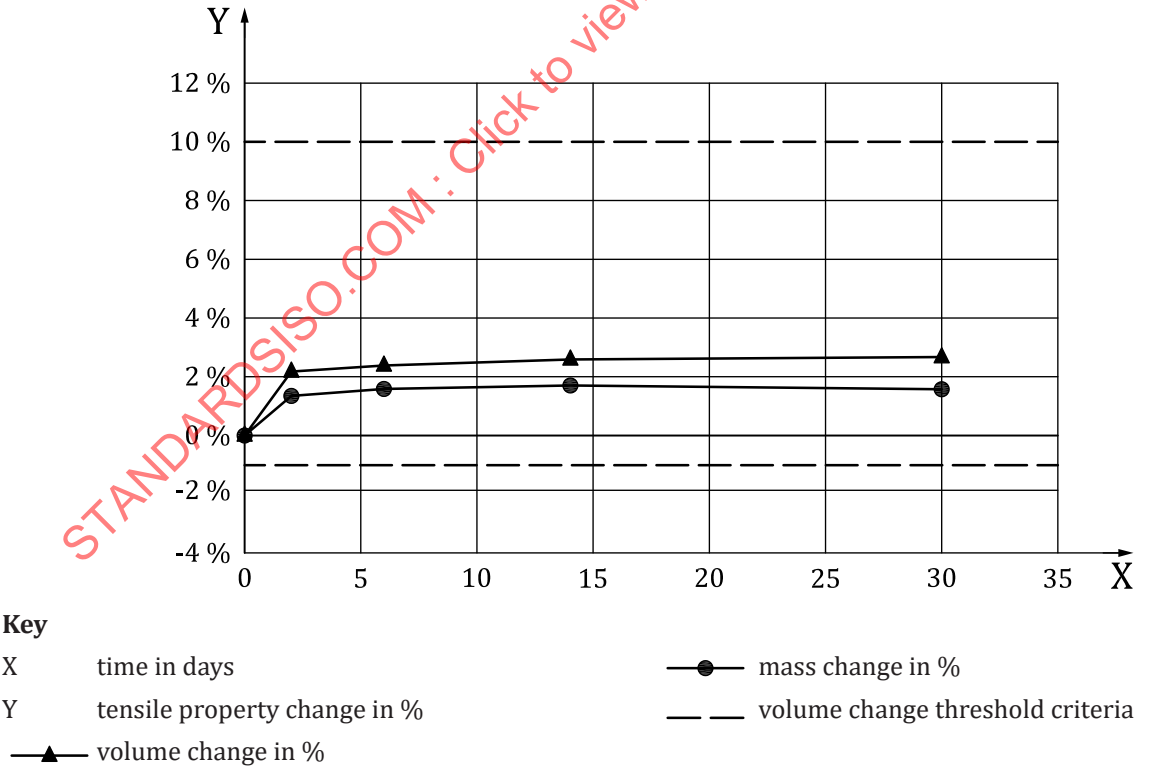


Figure A.5 — Volume and mass property change percentages

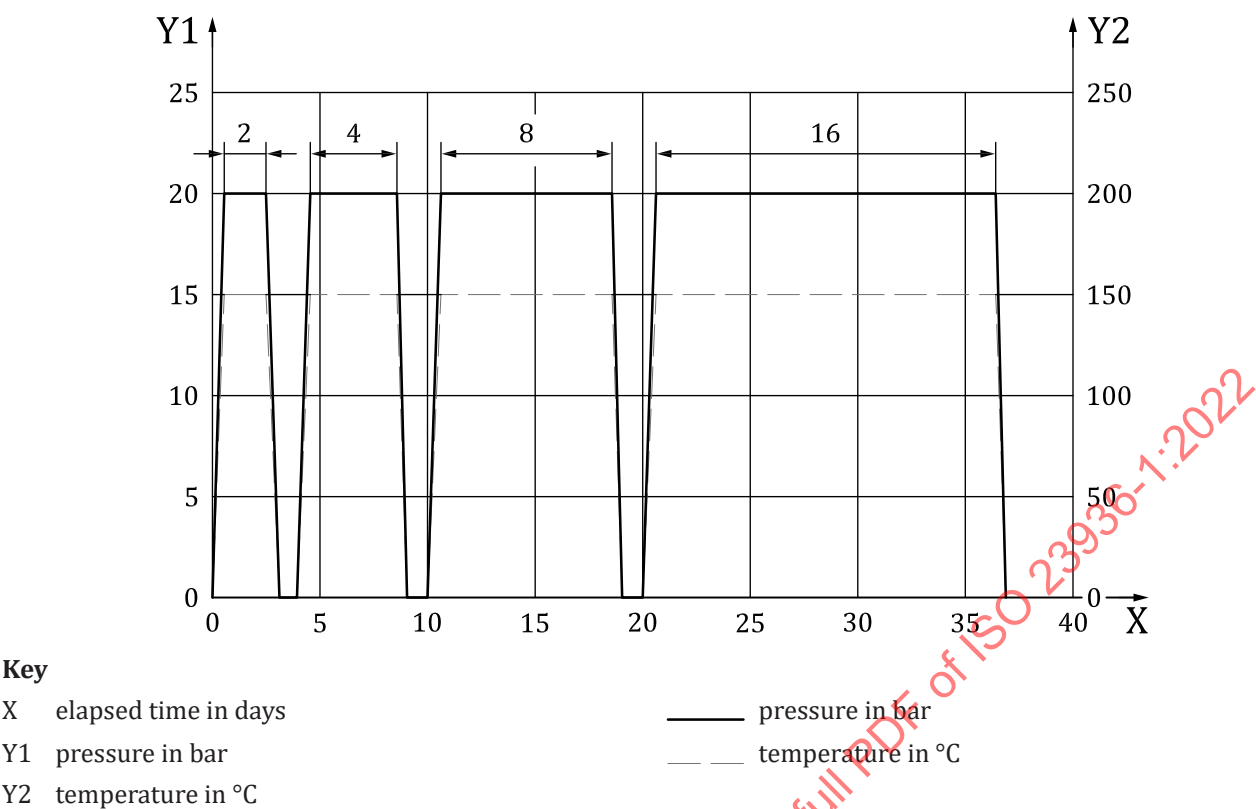


Figure A.6 — Pressure temperature chart of multiphase fluid

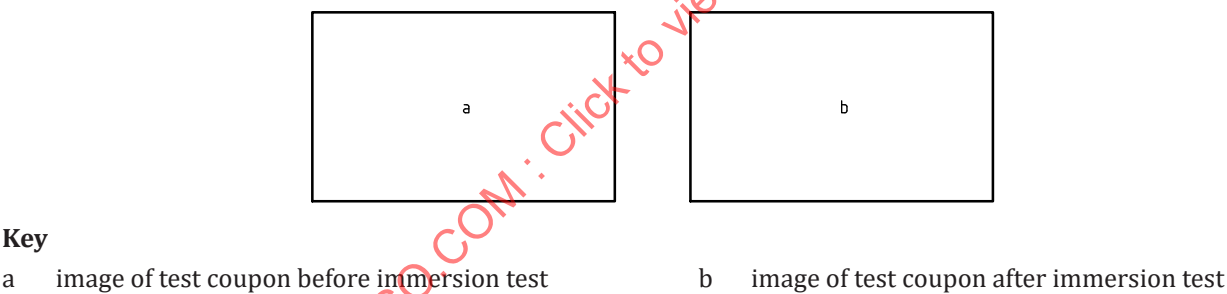


Figure A.7 — Before and after aging test specimen images

A.3.3 Test report for Level 3

The Level 3 test report shall include all the requirements of the Level 2 test report according to [A.3.2](#) and the following:

- a) the three exposure temperatures and the durations selected for each temperature;
- b) expansion of the data, tables, and charts to include all exposure durations;
- c) three sets of tables with results, percent changes, and visual observations;
- d) three sets of final duration specimen images;
- e) report data sets for 2-day and 7-day liquid only exposures with a nitrogen purge and gas cap at all three temperatures.

[Table A.4](#) provides an example of a test report that covers a single data set.

Table A.4 — Example test report for Level 3

AcmeManf PhenDura XTRA-6880 conforms to ISO 23936-1:2022		
Conformance statement	-	ISO 23936-1:2022 Conformance to Level 3, Fluid 3.2 (multiphase 10 % sour gas)
Client address	-	ToolCo Inc. Energy Dr, Denver, CO, USA
Compound manufacturer Compound name/number	-	AcmeManf, PhenDura XTRA-6880
Material / reinforcement	-	PEKK / unfilled
Recipe revision level	-	n/a
Lot/batch no.	-	FF2344rw4r/07
Moulding date	-	Q4 2015
Ageing test conditions		
Fluid	Classification	ISO 23936-1:2022 Fluid 3.2
	Description	10 % Sour aromatic
	Liquid	60 vol %: 70 % heptane, 20 % cyclohexane, 10 % toluene 10 vol %: distilled water
	Gas Pressure	30 vol %: 10 % H ₂ S, 5 % CO ₂ , 85 % CH ₄ (6 ± 0,5) MPa
Volume ratio minimum	-	10:1
T1 Exposure temperature	°C (°F)	250 (482)
T1 Pressure at temperature	MPa (psi)	16,5 (2 400)
T1 Exposure durations	days	2, 7, 14, 27, 42
T2 Exposure temperature	°C (°F)	240 (464)
T2 Pressure at temperature	MPa (psi)	14,5 (2 100)
T2 Exposure durations	days	7, 14, 30, 42, 56
T3 Exposure temperature	°C (°F)	225 (437)
T3 Pressure at temperature	MPa (psi)	10,3 (1 500)
T3 Exposure durations	days	7, 14, 28, 41, 57
Specimen preconditioning	-	None
Leaching	%	None
Test lab address	-	Ace Test Lab 123 Sample Rd, Testing, LA, USA
Test date	-	10 June 2015 - 25 October 2015
Test gas mixture or individual gases certified	YES/NO	YES
Pressure calibration available	YES/NO	YES
P/T logs available	YES/NO	YES

The example in [Table A.5](#) shows the T2 data table. The T1 and T3 data table shall also be presented in the same format within the final report.

Table A.5 — Example T2 test data

General test details									
Ageing days at 240 °C	0	pre-conditioning	2	7	7	14	30	42	56
Fluid description	none	N/A	3,2 Liquids only	3,2 Liquids only	3,2	3,2	3,2	3,2	3,2
Days and tolerance	0	N/A	2 ± 1	7 ± 1	7 ± 1	14 ± 2	28 ± 3	42 ± 3	56 ± 3
Fresh liquids	yes	N/A	no	no	no	no	yes	no	no
Fresh gas	yes	N/A	yes	yes	yes	yes	yes	yes	no
Median values for each test duration									
Tensile strength (MPa) ^a	95,0	N/A	92,7	92,5	93,0	90,1	89,3	88,0	87,0
Yield stress (MPa) ^a	95,0	N/A	89,0	89,5	88,8	87,0	85,2	84,0	83,0
Nominal strain at break (%) ^a	42,0	N/A	29,0	27,0	25,4	23,8	22,3	21,3	20,3
Modulus (specified 2% secant method) (GPa) ^a	4,11	N/A	3,90	3,81	3,73	3,60	3,57	3,54	3,50
Mass (grams) ^b	8 506	N/A	8 721	8 730	8 744	8 852	8 851	8 862	8 858
Specific gravity ^b	1,356	N/A	1,388	1,390	1,401	1,405	1,408	1,408	1,408
Percent change in tensile properties using 2 day liquid saturation as the baseline									
Tensile strength Δ (%) ^c	-	N/A	0 %	-	0 %	-3 %	-4 %	-5 %	-6 %
Yield stress Δ (%) ^c	-	N/A	0 %	-	0 %	-2 %	-4 %	-6 %	-7 %
Nominal strain at break Δ (%) ^c	-	N/A	0 %	-	-12 %	-18 %	-23 %	-27 %	-30 %
Modulus (specified 2% secant method) Δ (%) ^c	-	N/A	0 %	-	-4 %	-8 %	-8 %	-9 %	-10 %
Percent change in tensile properties using 7 day liquid saturation as the baseline									
Tensile strength Δ (%) ^c	-	N/A	-	0 %	-2 %	-4 %	-5 %	-5 %	-7 %
Yield stress Δ (%) ^c	-	N/A	-	0 %	-1 %	-3 %	-4 %	-6 %	-8 %
Nominal strain at break Δ (%) ^c	-	N/A	-	0 %	-13 %	-19 %	-26 %	-28 %	-31 %
Modulus (specified 2% secant method) Δ (%) ^c	-	N/A	-	0 %	-5 %	-10 %	-9 %	-10 %	-10 %
Percent change in tensile properties using preconditioning as the baseline									
Tensile strength Δ (%) ^c	-	N/A	-	-	N/A	N/A	N/A	N/A	N/A
Yield stress Δ (%) ^c	-	N/A	-	-	N/A	N/A	N/A	N/A	N/A
Nominal strain at break Δ (%) ^c	-	N/A	-	-	N/A	N/A	N/A	N/A	N/A
Modulus (specified 2% secant method) Δ (%) ^c	-	N/A	-	-	N/A	N/A	N/A	N/A	N/A
^a Measured data shall be presented with at least three significant figures.									
^b Measured data shall be presented with at least three decimal places.									
^c Calculated percent change data shall be presented with no decimal places.									

Table A.5 (continued)

General test details									
Percent change in physical properties using as-received data as the baseline									
Mass Δ (%) ^c	-	N/A	-3 %	-3 %	-3 %	-4 %	-4 %	-4 %	-4 %
Volume Δ (%) ^c	-	N/A	5 %	5 %	5 %	6 %	6 %	7 %	6 %
^a Measured data shall be presented with at least three significant figures.									
^b Measured data shall be presented with at least three decimal places.									
^c Calculated percent change data shall be presented with no decimal places.									

The examples in [Figure A.8](#) and [Figure A.9](#) show the T1 data charts. The T2 and T3 data charts shall also be presented in the same format within the final report. The baseline selection shall be identified in the data charts.

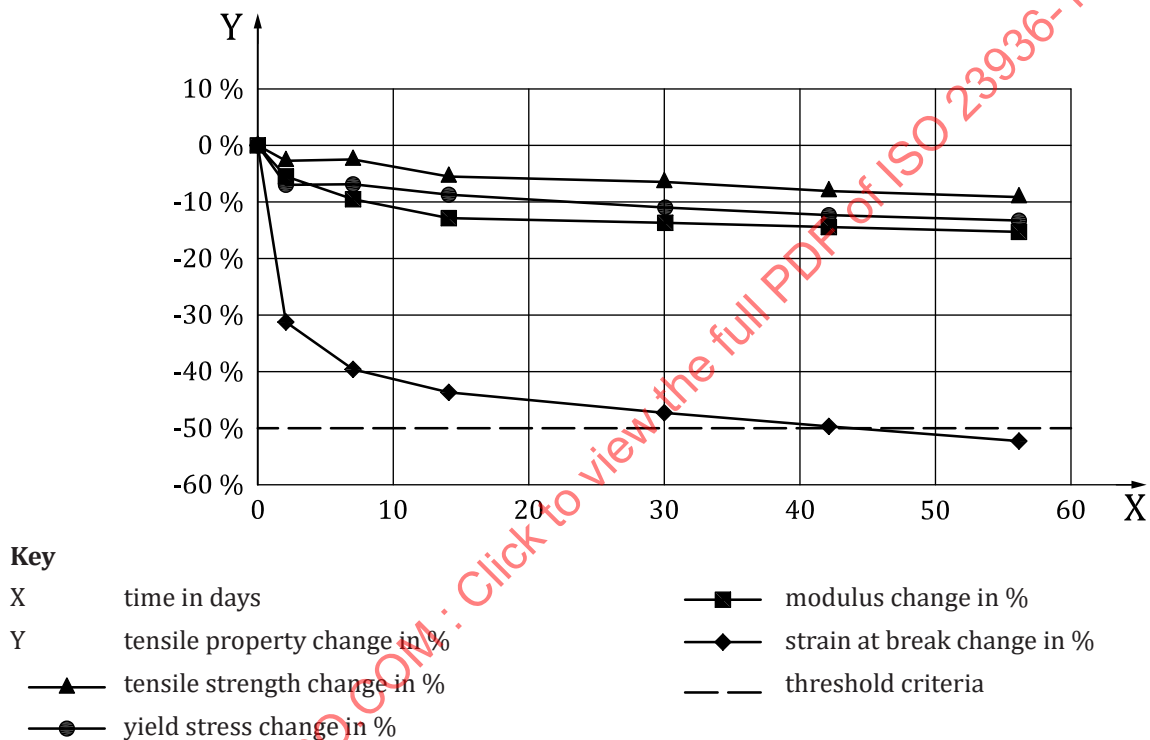


Figure A.8 — Example T1 tensile property change percentages using 2-day baseline data

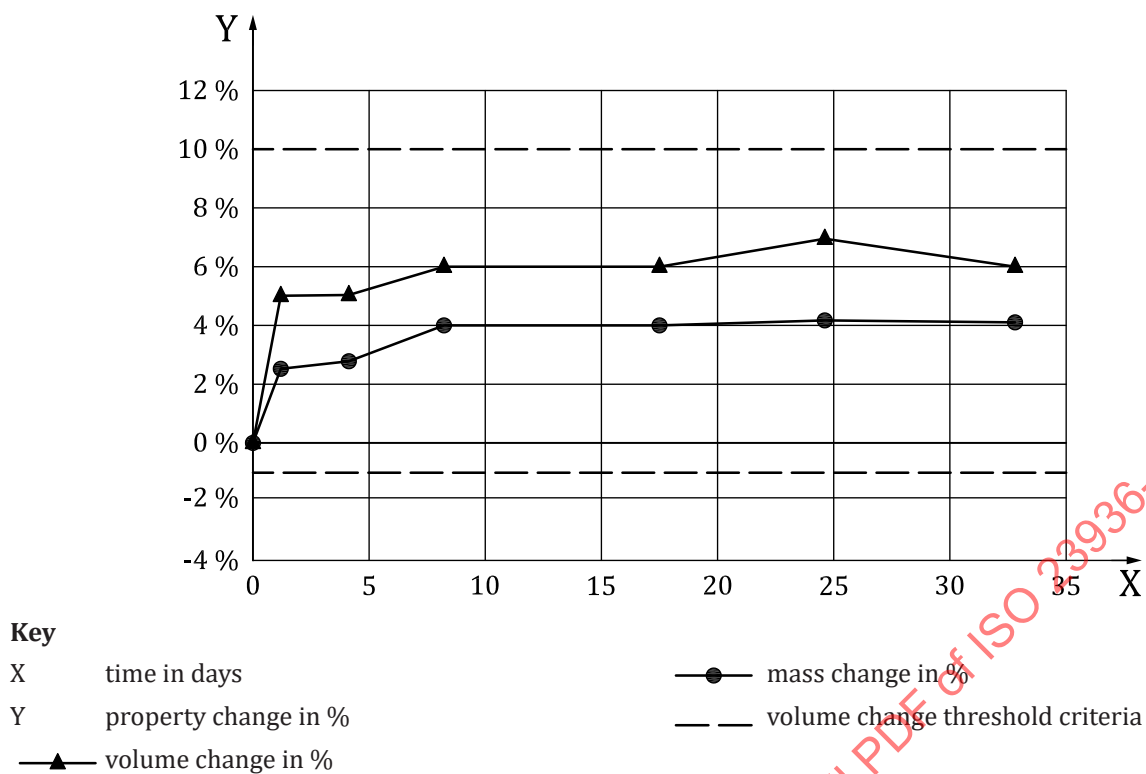


Figure A.9 — Example T1 volume and mass property change percentages

A.3.4 Level 4 reporting

The Level 4 test report shall include all the requirements of the Level 3 test report according to [A.3.3](#) and the following additional requirements:

- all three exposure temperatures and the durations selected for each temperature;
- threshold baseline selection and justification;
- life estimation analysis and results (for examples, see [Annex B](#)).

Annex B (informative)

Long-term life estimation methodology

B.1 Arrhenius approach

The Arrhenius approach to material life estimation requires that:

- chemical reactions occur in the material during laboratory testing;
- the chemistry is mirrored by the change of at least one material property;
- failure in service would occur as a result of the same chemistry and material property changes probed during testing.

The approach is often challenged by a number of highly ageing-resistant polymers (e.g. PTFE, PEEK) for which little discernible degradation is often visible upon laboratory testing.

A material can be fit for service even if an Arrhenius-based lifetime estimate is not possible. In this case, a statement of suitability can be made if no significant chemical reaction is observed. The following conditions should be met:

- a) test temperatures are high enough to expect chemical reaction;
- b) test duration at least as specified in Level 3 or Level 4, as applicable;
- c) application temperature is below the lowest test temperature.

[Clause B.2](#) introduces a methodology for Arrhenius life estimation.

[Clause B.3](#) provides a guideline for the implementation of the methodology described in [Clause B.2](#).

[Clause B.4](#) shows examples of different curve fits used to model the degradation behaviour that affects life time estimations.

B.2 Example for life estimation

B.2.1 Evaluation of material stability under given specific environmental conditions

One of the aims of this document is to provide a procedure to estimate the useful life of a material in a particular test fluid. The latter may be identical to the service situation (e.g. inhibited brine), or a representation of it (e.g. synthetic hydrocarbon oil). This clause describes how data obtained from a laboratory ageing test can be used to predict how long a material will retain 50 % of its tensile property levels. The procedure is only applicable if the polymer chemically ages in the test fluid. Background information can be found in ISO 11346 and ASTM D3032.

Ageing data have been obtained for a thermoplastic immersed in a test fluid with the temperature and durations listed in [Table B.1](#).

Table B.1 — Test conditions

Temperature °C	Sampling intervals d
100	30, 60, 100, 200, 400, 700, 1 100
110	14, 28, 56, 100, 150, 250
130	7, 14, 20, 42, 60, 100, 150, 200

Table B.2 lists the average results for the strain at break values recorded as a result of the exposure testing. The samples were tested at room temperature while still soaked with fluid. Also included in Table B.2 are strain at break values obtained from the original dry unaged samples. The data in Table B.2 are shown together as exposure duration versus strain at break in Figure B.1.

Table B.2 — Test results for a thermoplastic strain at break when aged in test fluid

Exposure conditions		Strain at break %
Unaged		250
100 °C	30 days	270
	60 days	265
	100 days	250
	200 days	210
	400 days	90
	700 days	15
	1 100 days	10
110 °C	14 days	255
	28 days	270
	56 days	190
	100 days	120
	150 days	80
	250 days	15
130 °C	7 days	270
	14 days	210
	20 days	110
	42 days	20
	60 days	15
	100 days	15
	150 days	10
	200 days	10

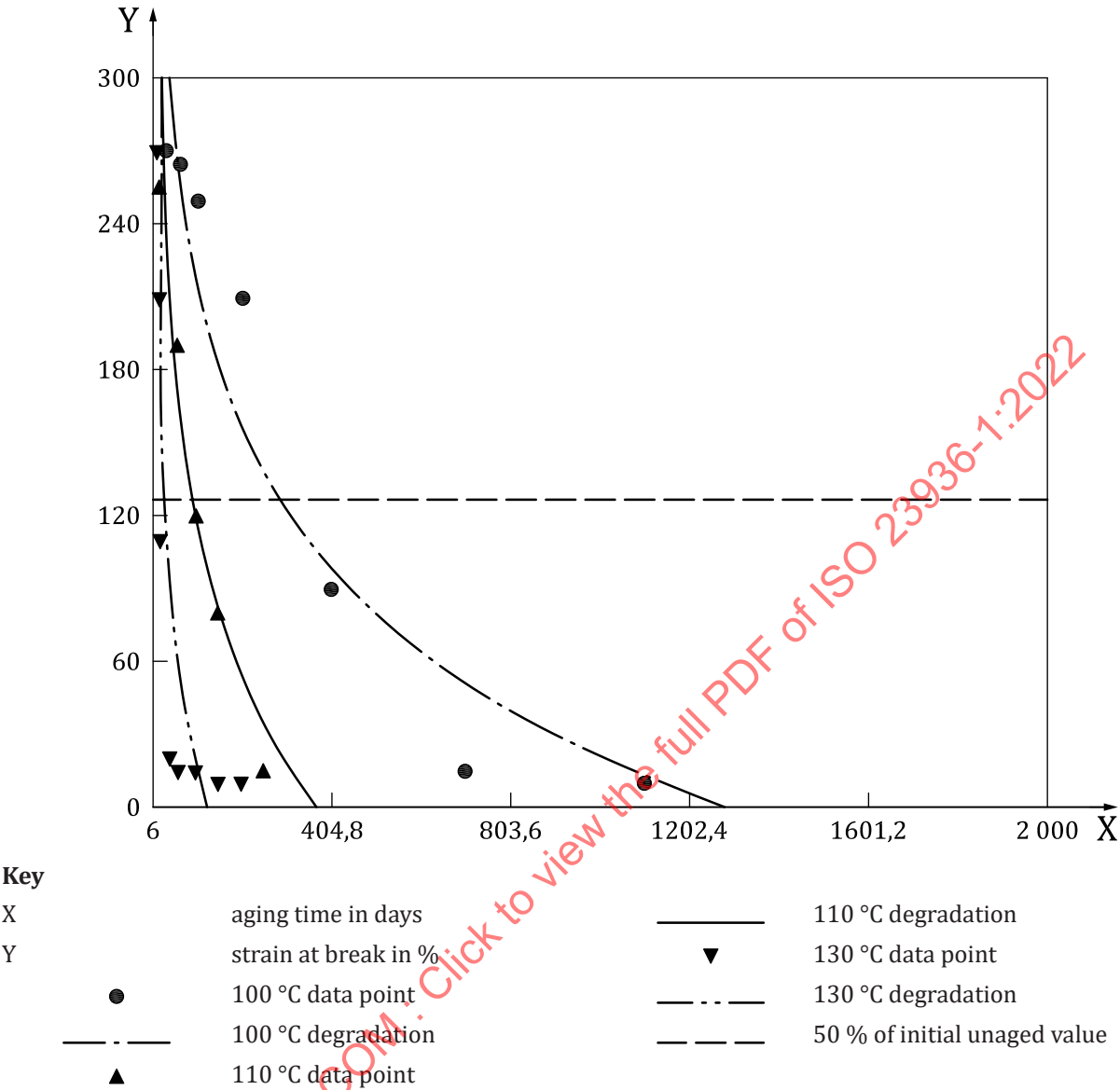


Figure B.1 — Change in property level with exposure duration and temperature

Figure B.1 shows how increasing temperature accelerates the decrease in strain at break. Also shown on the graph is a line corresponding to a 50 % drop in the original unaged strain at break value. Commonly time to generate a 50 % drop in properties is considered as the "threshold time" for the specific temperature point.

Multiple curve fits can be used to model the data like linear, power, logarithmic or exponential. The best curve fit used in this example is logarithmic.

A 2-day or 7-day saturated data can be used as the baseline for the 50 % change threshold. In this example, the unaged (as-received) properties are used as the baseline.

B.2.2 Arrhenius approach, trends, calculation and interpretation

Time to failure (or time to 50 % drop in strain at break in this example) can be found graphically for each temperature by determining the cross-points of the 50 % drop line and the property plot for the specific temperature point. Alternatively, a linear interpolation can be made between the two data points on each side of the cross-point. Using the graphical approach, the time to failure (t_{50}) levels are determined in Table B.3. These times are then re-calculated as the natural logarithm of the rate $[\ln(1/t_{50})]$ and plotted against the inverse of the test temperature ($1/T_K$), expressed in K^{-1} , which is