International Standard

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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 4: Fiber-reinforced composite materials

*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 de pétrole et de gaz —*

*Partie 4: Matériaux composites renforcés de fibres*

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# Foreword

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

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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, *Oil and gas industries including lower carbon energy,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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.

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# 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 is 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.

This information aids in material selection. It can be applied to help avoid costly degradation failures of the equipment itself, which can pose a risk to the health and safety of the public and personnel or the environment. This document complements the document for metallic materials in sour service (the ISO 15156 series). 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 $H_2S$-containing environments which are related solely to relevant environmentally assisted cracking mechanisms.

Mechanical properties and the environmental stability of composite materials depend on the properties and environmental stability of matrix resins, fibres and fibre/resin bonding interfaces. This document focuses on the overall composite properties and their environmental stability. To permit this assessment this document utilizes flat plates and/or tubular shapes made specifically for these tests.

The 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 oil and gas 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 4: Fiber-reinforced composite materials

**CAUTION — Non-metallic materials selected using the ISO 23936 series 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.**

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## 1 Scope

This document provides general principles, requirements and recommendations for the assessment of stability of fibre-reinforced composite materials for service in equipment used in oil and gas production environments.

This document describes the procedures for comparative testing of composite materials consisting of polymers (thermoplastics and thermosets) and re-enforcing materials e.g. glass, carbon, aramid and metals as continuous fibres or woven fabric used in equipment for oil and gas production.

Testing and characterization of neat resins and fibre products are beyond the scope of this document.

The equipment considered includes, but is not limited to, non-metallic pipelines, piping, liners and downhole tool components.

Blistering by rapid gas decompression, coatings and compounded particulate- and short fibre-reinforced composites are excluded from the scope of this document.

## 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 175, *Plastics — Methods of test for the determination of the effects of immersion in liquid chemicals*

ISO 527-4, *Plastics — Determination of tensile properties — Part 4: Test conditions for isotropic and orthotropic fibre-reinforced plastic composites*

ISO 527-5, *Plastics — Determination of tensile properties — Part 5: Test conditions for unidirectional fibre-reinforced plastic composites*

ISO 1172, *Textile-glass-reinforced plastics — Prepregs, moulding compounds and laminates — Determination of the textile-glass and mineral-filler content using calcination methods*

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

ISO 1268-1, *Fibre-reinforced plastics — Methods of producing test plates — Part 1: General conditions*

ISO 1268-3, *Fibre-reinforced plastics — Methods of producing test plates — Part 3: Wet compression moulding*

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ISO 1268-4, *Fibre-reinforced plastics — Methods of producing test plates — Part 4: Moulding of prepregs*

ISO 1268-5, *Fibre-reinforced plastics — Methods of producing test plates — Part 5: Filament winding*

ISO 1268-7, *Fibre-reinforced plastics — Methods of producing test plates — Part 7: Resin transfer moulding*

ISO 1268-9, *Fibre-reinforced plastics — Methods of producing test plates — Part 9: Moulding of GMT/STC*

ISO 2781, *Rubber, vulcanized or thermoplastic — Determination of density*

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

ISO 7822, *Textile glass reinforced plastics — Determination of void content — Loss on ignition, mechanical disintegration and statistical counting methods*

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

ISO 14126, *Fibre-reinforced plastic composites — Determination of compressive properties in the in-plane direction*

ISO 14127, *Carbon-fibre-reinforced composites — Determination of the resin, fibre and void contents*

ISO 14129, *Fibre-reinforced plastic composites — Determination of the in-plane shear stress/shear strain response, including the in-plane shear modulus and strength, by the plus or minus 45 degree tension test method*

ISO 14130, *Fibre-reinforced plastic composites — Determination of apparent interlaminar shear strength by short-beam method*

ISO 15024, *Fibre-reinforced plastic composites — Determination of mode I interlaminar fracture toughness, $G_{IC}$, for unidirectionally reinforced materials*

ISO 15114, *Fibre-reinforced plastic composites — Determination of the mode II fracture resistance for unidirectionally reinforced materials using the calibrated end-loaded split (C-ELS) test and an effective crack length approach*

EN 2564, *Aerospace series – Carbon fibre laminates – Determination of the fibre, resin and void contents*

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

ASTM E1131, *Standard Test Method for Compositional Analysis by Thermogravimetry*

ASTM D2290, *Standard Test Method for Apparent Hoop Tensile Strength of Plastic or Reinforced Plastic Pipe*

ASTM D2344, *Standard Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates*

ASTM D2412, *Standard Test Method for Determination of External Loading Characteristics of Plastic Pipe by Parallel-Plate Loading*

ASTM D3039, *Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials*

ASTM D3171, *Standard Test Methods for Constituent Content of Composite Materials*

ASTM D3410, *Standard Test Method for Compressive Properties of Polymer Matrix Composite Materials with Unsupported Gage Section by Shear Loading*

ASTM D3418, *Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry*

ASTM D3518, *Standard Test Method for In-Plane Shear Response of Polymer Matrix Composite Materials by Tensile Test of a ±45° Laminate*

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ASTM D5229, *Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials*

ASTM D5379, *Standard Test Method for Shear Properties of Composite Materials by the V-Notched Beam Method*

ASTM D5448, *Standard Test Method for Inplane Shear Properties of Hoop Wound Polymer Matrix Composite Cylinders*

ASTM D5449, *Standard Test Method for Transverse Compressive Properties of Hoop Wound Polymer Matrix Composite Cylinders*

ASTM D5450, *Standard Test Method for Transverse Tensile Properties of Hoop Wound Polymer Matrix Composite Cylinders*

ASTM D5528, *Standard Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites*

ASTM D6641, *Standard Test Method for Compressive Properties of Polymer Matrix Composite Materials Using a Combined Loading Compression (CLC) Test Fixture*

ASTM D7028, *Standard Test Method for Glass Transition Temperature (DMA Tg) of Polymer Matrix Composites by Dynamic Mechanical Analysis (DMA)*

ASTM D7078, *Standard Test Method for Shear Properties of Composite Materials by V-Notched Rail Shear Method*

ASTM D7905, *Standard Test Method for Shear Properties of Composite Materials by V-Notched Rail Shear Method Standard Test Method for Determination of the Mode II Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites*

NPL (2020) Measurement Good Practice Guide No. 38, *Fibre Reinforced Plastic Composites – Machining of Composites and Specimen Preparation; National Physical Laboratory (UK)*[1)]

# 3 Terms and 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**
**composite material**
fibre-reinforced material system consisting of thermoplastic or thermoset polymers and reinforcing materials in the form of long and continuous glass, carbon and/or aramid fibres or woven fabric

**3.1.2**
**fabricator**
producer of test plates and specimens

1) Available at www.npl.co.uk.

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**3.1.3**
**glass transition temperature**
$T_g$
characteristic value of the temperature range over which the glass transition takes place and at which the *composite material's* (3.1.1) mechanical properties change from elastic (glassy) state to viscous (rubbery) state

Note 1 to entry: The assigned glass transition temperature ($T_g$) may vary, depending on the specific property and on the method and conditions selected to measure it [for instance, by differential scanning calorimetry (DSC) or by dynamic mechanical analysis (DMA)].

**3.1.4**
**lamina**
ply
thin, single sheet of long and continuous reinforcing fibres sharing the same orientation in a polymeric resin matrix built up into a flat or curved arrangement

**3.1.5**
**laminate**
combination of *laminas* (3.1.4)

**3.1.6**
**manufacturer**
producer of the materials used for creation of semi-finished and/or finished products

**3.1.7**
**sizing**
optional treatments usually applied to yarn by *manufactures* (3.1.6) for reasons that can include increasing fibre-matrix compatibility, as well as facilitating handling during manufacture

**3.1.8**
**wet $T_g$**
*glass transition temperature* (3.1.3) of the fluid saturated material

## 3.2 Abbreviated terms

| | |
|---|---|
| ATM | accelerated testing method |
| CA | autoclave cured |
| CH | hot-press cured |
| CNC | computerized numerical control |
| CO | oven cured |
| COA | certificate of analysis |
| COC | certificate of conformity |
| COV | coefficient of variation |
| CU | UV cured |
| DMA | dynamic mechanical analysis |
| DSC | differential scanning calorimetry |
| GFRP | glass fibre reinforced polymer |
| GMT | glass fibre mat reinforced thermoplastic |

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| | |
|---|---|
| HDT | heat deflection temperature |
| HPHT | high-pressure high-temperature |
| IPS | in plane shear |
| LF | filament winding lamination |
| LP | prepreg lamination |
| LR | vacuum-assisted resin-transfer moulding |
| LW | wet layup lamination |
| MOL | material operational limit |
| NDT | non-destructive testing |
| PA | Polyamide |
| ppm (vol) | volume parts per million volume parts |
| QC | quality control |
| QD | quality documentation |
| SEM | scanning electron microscopy |
| STC | sheet thermoplastic composite |
| TA | autoclave consolidation |
| TI | isothermal consolidation |
| TP | hot pressing |
| TGA | thermogravimetric analysis |
| UD | unidirectional |
| UV | ultraviolet light |

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# 4 Technical requirements

## 4.1 General requirements

Composite 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 composite materials. The testing methods at the material level shall focus upon the laminate and specimen geometry and do not represent a functional application test which is beyond the scope of this document. The specimen layup described is to provide a consistent, common basis for generating comparable data for different composite materials. Specific testing shall be required for the actual layup for samples representative of the final product form. Material property data are 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

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material stability data in a multi-phase $H_2S$ 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.

NOTE Ageing of composites faces combined challenges. Polymer or thermoset ageing alone deals in most cases with quasi-isotropic material properties. Composites of any configuration furthermore deal with polymeric challenges as well as with highly anisotropic properties resulting from fibre and interface properties, which can age very differently than the matrix material. All effects can overlay and become apparent in different failure modes or shifts thereof over the ageing period.

Ageing experiments can be designed in the following way to extract meaningful information, especially with regards to establishing lifetime models with these complex effects in mind:

a) identify possible ageing mechanisms for matrix material;

b) identify possible ageing mechanisms for fibre material;

c) identify possible ageing mechanisms for interface;

d) differentiate between physical and chemical ageing;

e) differentiate between reversible and irreversible ageing;

f) identify probabilities for the above-mentioned mechanisms to occur simultaneously during test periods;

g) rank mechanisms for severity;

h) rank for material characterization or application related testing;

i) exclude any unwanted ageing mechanism by physical/chemical exclusion of other ageing influences (monitored and protocolled), choosing variations in layup to promote specific failure, or some combination of both.

Level 1 conformity 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 2 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 conformity pertains to material stability (ageing) behaviour and shall be accompanied by a report. Clause 6 provides requirements for Level 2 conformity. 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 conformity pertains to material stability (accelerated ageing) behaviour and shall be accompanied by a report. Clause 7 provides requirements for Level 3 conformity. 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 $H_2S$-fluids.

Level 4 conformity pertains to a material stability (long-term) assessment of 180 days or longer. Level 4 attempts life estimation and shall be accompanied by a report. Clause 8 provides requirements for Level 4 conformity. The intent of Level 4 assessment is to predict the material's progressive degradation; hence conformity 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 the edition of this document utilized at time of testing.

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.

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If there is scientific evidence on resistance of the material to the chemicals at the intended pressure and temperatures then such material may be exempt from Level 3 and Level 4.

## 4.2 Cautionary remarks

Designers should not assume that properties provided in a material data report as defined in Clause 5 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 composites 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 ISO 23936-4 material conformity, it shall be made from a composite material that conforms with this document. The entire compound manufacturing process shall be fully traceable. conformity records shall state the edition of this document used in the assessment. Reference to conformity with the ISO 23936 series shall include the part and edition (year) of the standard used e.g. ISO 23936-4:2024.

Each component 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 conformity and traceability over the supply chain can be found in relevant product standards and agreed between interested parties.

## 4.4 Test specimen identification

The specimen fabrication details shall be reported using the following identification code system. If the type of fibre or resin is a new class not covered below, the full designation shall be added in the datasheet.

a) Material system:

A.1 Type of reinforcement fibre (e.g. carbon: CF, E-glass: EG, ECR-glass: ECR, S-glass: SG, aramid: AR, information regarding sizing and any additives, fibre modulus and precursor, bundle type, linear weight, tow size and type of weave, whenever applicable) and its grade should be included if made available. Designations for carbon, glass and aramid fibres can be provided according to ISO 13002, ASTM D578/D578M-23 and the EN 13003 series, respectively.

NOTE Discontinuous (short) fibre reinforced composites like veils composites are covered in ISO 23936-1.

A.2 Type of resin (e.g. epoxy: EP, Phenolic: PH, Vinyl ester: VE, unsaturated polyester: UP, Polyethylene: PE, bismaleimide: BMI, PEEK: PK, Polyphenylen sulfide: PPS, polyamide: PA, polyvinylidene fluoride: PVDF, polypropylene: PP) and its grade.

A.3 Type of fabric arrangement [e.g. woven roving: WR, stitch-bonded: SB, unidirectional (prepreg): UD, filament roving: FR, Braiding: BR].

b) Laminate lamina orientation and stacking sequence in test specimen preparation:

B.1 Cross-lamina laminate 0/90 orientation, $[0/90]_{ns,}$ where $n$ represents the number of repeats and s stands for symmetrical stacking;

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B.2 UD laminate $[0]_n$;

B.99 other cross winded orientations like + $\alpha$ /- $\alpha$.

c) Lamination method:

C.1 Prepreg lamination (LP);

C.2 Vacuum-assisted resin transfer moulding (LR);

C.3 Wet layup lamination (LW);

C.4 Filament winding lamination (LF);

C.5 Tape placement welding (LT);

C.99 New lamination technology (method name).

d) Consolidation method for thermoplastic composites:

D.1 Hot pressing (TP);

D.2 Isothermal consolidation (TI);

D.3 Autoclave consolidation (TA);

D.99 New consolidation technology (method name).

e) Curing method for thermoset composites:

E.1 Oven cured (CO);

E.2 Hot-press cured (CH);

E.3 Autoclave cured (CA);

E.4 UV cured (CU);

E.99 New curing technology (method name).

f) Specimen extraction orientation from $[0/90]_{ns}$ and $[0]_n$ laminate or arbitrary pipe layup:

F.1 Longitudinal (OL);

F.2 Transversal (OT);

F.3 In ±45° direction (OD);

F.4 Perpendicular (hoop) to pipe axis (OP);

F.5 Along pipe axis (OA).

g) Extraction method:

G.1 Sawing (ES);

G.2 Milling/Turning (EM);

G.3 Water jet (EW);

G.4 Laser (EL);

G.99 New extraction technology (method name).

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h) Tabbing method (if required):

H.1 Tabbing before extraction (BB);

H.2 Tabbing of individual specimen (BI);

H.3 Un-tabbed specimen (BU);

H.4 Tabbing before ageing (BF);

H.5 Tabbing after ageing (BA);

H.99 New tabbing technology (method name).

The test specimen identification shall give the following information: test standard, specimen type, test speed and identification code.

EXAMPLE Sample test call out for an ISO 527-5 or ASTM D3039 tensile specimen of $[0/90]_{5s}$ laminate in zero direction:

a) ISO 527-5: 2009, Type A, 2,0 mm/min (EG/EP/$[0/90]_{5s}$/LP/TP/CO/OL/ES/BB);

b) ASTM D3039, Type A, 2,0 mm/min (EG/EP/$[0/90]_{5s}$/LP/TP/CO/OL/ES/BB).

## 4.5 Validation of conformity

A composite loses its conformity if changes are made to the raw material supply, the composite matrix material formulation, reinforcement material, or the composite manufacturing process. New testing shall be done for each desired level of conformity.

If Level 4 conformity is complete prior to change, new Level 4 testing shall not be done 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 composite material manufacturing process is carried out at different plants/locations, a separate Level 1 conformity shall be done for each plant.

Level 1 to Level 4 testing shall not be done on the component if no compositional changes (other than due to cross-linking reactions) have been made to the matrix material 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.

Level 1 conformity pertains to material property characterization of laminate and shall be documented by a material datasheet that details material properties, see Clause 5, and documented by COCs for batch quality control testing in accordance with Annex D.

Physical and mechanical properties shall be characterized in as-received 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 2 conformity pertains to material fluid stability (ageing) behaviour and shall be accompanied by a report that details the testing that was performed, and the edition of this standard utilized at time of testing. This document provides requirements for Level 2 conformity, see Clause 6.

The effect on material properties of Fluid 2.1 to Fluid 2.4 listed in 6.2.4 shall be investigated with real time ageing studies. A material's resistance to chemical/physical change is determined.

Level 3 evaluations include additional fluid stability (ageing) assessments such as fit-for-purpose tests or other assessments related to service conditions, see Clause 7.

The intent of Level 3 assessment is to predict the material progressive degradation, in one of three specified fluids, Fluid 3.1 to Fluid 3.3, which includes a bespoke fluid. Properties to be measured are taken from Level 2, 6.2.5. There are no conformity requirements for Level 3 testing. Users shall determine acceptance criteria and suitability of materials for application.

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All Level 3 activities should be accompanied by a report that details the approach undertaken in line with the application; including the test methods and results obtained.

## 4.6 Quality control for fabrication of laminated test plates

### 4.6.1 General

QC in this document focuses upon controlling the quality and processing consistency of the fabricated laminates and the test specimens extracted from them. This shall be achieved by:

a) ensuring the laminate fabricator is given a specification for test plate manufacture listing the constituent materials and desired minimum laminate properties, in accordance with Annex B;

b) using methods of test specimen preparation as described in Clause 5.

As the quality control assessment can be undertaken by various members of the supply chain, it is essential to collate the results in QD; this ensures that material comparisons by testing according to this document are made without influence from fabrication variability.

The consolidation process has a significant influence on interlaminar or interface properties of the plates. The identification codes i.e. the time, temperature and pressure cycles of the test plate manufacture, volume fraction of fibres and stacking sequence, shall be reported.

Fabrication of plates shall be performed in accordance with Annex B. Extraction of test specimen shall be performed in accordance with Annex C.

### 4.6.2 Quality control for fabrication of plates

#### 4.6.2.1 General

In the fabrication of plates from which specimens are extracted, the governing quality control requirements shall be invoked for the constituent materials in accordance with Annex B. Test plates shall be fabricated in accordance with the ISO 1268 series. ISO 1268-1 shall apply to all composites.

For thermoplastic composite ISO 1268-4 and ISO 1268-9 shall apply.

For thermoset composite ISO 1268-3, ISO 1268-4, ISO 1268-5, and ISO 1268-7 shall apply.

#### 4.6.2.2 Material and test plate specification

A material and test plate specification shall be agreed between the fabricator and the user, for the requested laminated test plates, in accordance with Annex B. The specification shall detail the selected constituent materials, the processing method, the required plate dimensions, density, fibre content, discontinuity features (voids overall content, shape, size and distribution) and the glass-transition temperature ($T_g$) of the fabricated laminated test plates. The specification shall also define the stacking sequence of the lamina and the QC acceptance criteria for the laminated test plates.

#### 4.6.2.3 Certificates of constituent materials

##### 4.6.2.3.1 General

COA and/or COC documents shall be provided with each constituent material.

##### 4.6.2.3.2 Certificates for UD-tapes for thermoplastic composites

The fabricator of thermoplastic plates for testing in accordance with this document shall only process UD-tapes which meet the minimal requirements of Annex D. This can be guaranteed by the tape supplier based on acceptable QC/QD. Any missing QC/QD items shall be generated by the plate fabricator before processing of the UD-tapes.

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#### 4.6.2.4 QC testing and analysis of composite

The inspection should follow the applicable part of the ISO 1268 series.

Laminates/tubes should be manufactured according to the following QC requirements during manufacturing:

a) COA and/or COC for constituent materials;

b) overlapping of tapes;

c) gaps between tapes with acceptable tolerances;

d) ensuring alignment of tape with those already laid;

e) no wrapping of tapes;

f) no dry fibres;

g) no visible inclusions or foreign objects;

h) no broken fibres or fibre wrinkles.

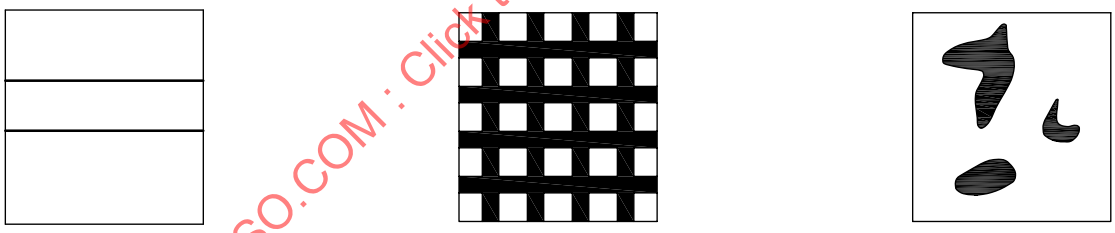
Laminates/tubes should be manufactured according to Figure 1 and the following QC requirements for the finished product:

— no dry patches/fibres;

— no visible defects (fibre misalignment, holes, dips, wrinkles, warping, bubbles);

— check the through thickness quality by Non-destructive testing [e.g. C-scan, X-ray, microtomography, shearography, thermography, microwave, acoustic emission and their variations/combination].

NOTE QC guidance and requirements can be found in addition to the general requirements in ISO 1268-1 and the more specific preparation guidance in ISO 1268-2 to ISO 1268-9. These additional items aim to ensure production of quality laminates suitable for subsequent test specimen preparation for oil and gas exposure testing, see Annex B.



**a) Lines of high porosity** **b) Damping too high / missing calibration** **c) Areas of high porosity**

**Figure 1 — Examples of defects detected by NDT test methods**

Further information for defect types and NDT can be taken from the NCN (2007) Best-Practice Guide as well as other reference standards, such as ASTM D2563, ASTM D4385 and the ISO 14692 series and ISO 20144.

The produced test plates shall be flat; both surfaces shall be smooth, free of cracks, voids, dry fibres, fibre wrinkles or undulation, non-uniform fibre distribution, visible impurities, and visible delamination.

Thickness, width, and length shall be measured and reported. Thickness of the plate shall be uniform and shall be measured in multiple distributed locations, being representative for the composite design concept.

For the examination of porosity, internal delamination and damage, methods like microscopy on polished cross-sections (see ISO 7822), ultrasonic, X-ray tomography, thermography, their variations/combination, or alternative methods with comparable qualitative examination level shall be used.

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Where the above fibre content and void content testing methods are inappropriate due to the fibre matrix combinations, alternative methods may be utilized.

The certified properties in Table 1 of the laminated plate and/or tubes shall be determined per corresponding test standards and shall meet the requirements specified in the material specification. Frequency of this testing shall be agreed between the manufacturer and the purchaser based upon the criticality of the delivered components.

**Table 1 — QC testing and analysis of composite**

| Certified property | Density | Fibre content | Void content[a] | Cured composite $T_g$ |
|---|---|---|---|---|
| Test standard | ISO 1183-1; ISO 2781 or ASTM D792 | ISO 1172 for glass fibre or EN 2564 or ISO 14127 for carbon fibre or ASTM D3171 or ASTM E1131 (TGA) | ISO 7822 or ASTM D3171 | ISO 6721-11 for DMA or ISO 11357-2 for DSC or ASTM D7028 DMA or ASTM D3418 for DSC |

[a] Other reference/complementary standards are ASTM D2563, ASTM D4385 and ISO 14692-4:2017, Annex A and ISO 20144.

#### 4.6.2.5 Documentation to be supplied with the test plates/tubes

The test part fabrication report should follow the part of the ISO 1268 series that is applicable for the tested material.

The identification code system in accordance with 4.4 shall be used in any report.

### 4.7 Test specimen preparation, quality control and identification

#### 4.7.1 Preparation of test specimens

Test specimens shall be prepared using repeatable methods that meet requirements of test standards given by Table 2 for the flat specimen from panels or by Table 3 for the curved specimen from pipes. The composite material supplied should be a true representative for final products. If it is not true representative, the final product result can vary.

#### 4.7.2 Quality control of specimen preparation

Test specimen extraction processes shall follow the requirements of the corresponding mechanical test standards. Special attention shall be paid to the direction of cutting (with respect to the principal fibre direction) in test plates and pipes. Dimensions and geometric requirements shall be measured and inspected for the extracted specimens. The surface finish of the machined or polished specimen edge surfaces shall be free of delamination and any machining damage in accordance with NPL (2020) Measurement Good Practice Guide No. 38.

## 5 Level 1 – Material property characterization

### 5.1 General

Standard tensile, compression, in-plane shear, interlaminar shear and fracture toughness tests are the tests for mechanical characterization of laminated composite materials (see Table 2). The tests in Table 2 and Table 3 shall be performed for the Level 1 mechanical property characterisation. All mechanical testing shall be performed at room temperature in unaged condition.

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**Table 2 — Mechanical tests on the flat specimen from panels**

| Test denomination | Standard | Stacking sequence | Test direction with respect to fibre direction |
|---|---|---|---|
| Tensile properties | ISO 527-5 or ASTM D3039 | $[0]_n$ | 0° and 90° |
| | ISO 527-4 or ASTM D3039 | $[0/90]_{ns}$ | 0° |
| Compression properties in the in-plane direction | ISO 14126 or ASTM D3410 or ASTM D6641 | $[0/90]_{ns}$ | 0° |
| In-plane shear stress/shear strain response | ISO 14129 or ASTM D3518 or ASTM D5379 or ASTM D7078 | $[0/90]_{ns}$ | ±45° |
| Apparent interlaminar shear strength[a] | ISO 14130 or ASTM D2344 | $[0]_n$ | 0° |
| Mode I interlaminar fracture toughness | ISO 15024 or ASTM D5528 | $[0]_n$ | - |
| Mode II interlaminar fracture toughness | ISO 15114 or ASTM D7905 | $[0]_n$ | - |

[a] Specimen size may require modifications from ISO 14130 recommended values. It is acknowledged that it may be difficult to obtain valid failure modes for thermoplastic matrix composites.

To compare composite pipes without additional protection layer (thermoplastic liner, chemical barrier layer or topcoat) oversized pipe segments can be aged before extraction of the test specimen, see Table 3.

**Table 3 — Mechanical tests of curved specimen from pipes or pipe section/ring**

| Test denomination | Standard | Stacking sequence | Test direction with respect to fibre direction |
|---|---|---|---|
| Apparent hoop tensile strength[a] | ASTM D2290 | $[0]_n$ | 0° for hoop winding |
| In-plane shear stress/shear strain response[a] | ASTM D5448 | $[0]_n$ | 0° for hoop winding |
| Apparent interlaminar shear strength[a] | ASTM D2344 | $[0]_n$ | 0° |
| Transverse tensile properties of hoop wound | ASTM D5450 | $[90]_n$ | hoop winding |
| Transverse compressive properties of hoop wound | ASTM D5449 | $[90]_n$ | hoop winding |
| Crush test | ASTM D2412 | $[-\alpha°/+\alpha°]_n$ | cross winding |

[a] For most pipes 0° and 90° layers cannot be produced, due to the helical angle during winding. Therefore small angles are considered axial and angles close to 90°, but lower, are considered with hoop.

The tests listed in Table 2 and Table 3 consider only initial characterization of materials. Whenever deemed pertinent for composite equipment application, long-term properties like fatigue and creep (also called time to rupture or stress rupture) and their combination shall be considered, taking into account representative layouts ($\pm\alpha$ laminates, fibre and transverse direction, through-thickness), loads (tensile, compression, shear) and duration (number of cycles or hours). Documents like DNV-ST-C501 and ISO/TS 18226 can be consulted for additional information.

NOTE The tests listed in Table 2 and Table 3 can be used to characterise the effect of the exposure to the different chemical environments on the mechanical performance of the material. This can include, but not limited to, effect degradation of reinforcement, resin matrix and the reinforcement / matrix interfaces.

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### 5.2 Reporting

The ISO 23936-4 material data report for each composite shall include as a minimum the following:

a) manufacturer contact information;

b) values for the documentation tests in Table 2;

c) test standards options such as method, specimen type, test speed;

d) specimen identification call out for each test method.

## 6 Level 2 - Material stability (short-term)

### 6.1 General

One or more of the tests in accordance with Table 2 or Table 3 shall be performed in order to document the 28 day maximum material stability temperature in the reported test fluid (see 6.2.4). The test procedure is listed in Annex A.

In addition, real-time short-term thermomechanical evaluation and qualification may be performed according to Annex E if user values capturing properties measured at elevated temperatures.

When smaller samples are cut from larger panels, it is not unusual that those edges may represent a preferable way for fluid to diffuse into material core. The use of adhesives in the edges in order to try to prevent or at least minimize such phenomena should be carefully considered as, depending on sealing material used to cover these edges, fluid absorption of this material can be sometimes higher than the composite matrix itself, so, careful choice is recommended.

For the purposes of fluid exposure, it should be taken into account that material takes some time to saturate (wet, conditioning) and only when saturation is completed (or at a previously bespoke agreed level between interested parties), material ageing period starts. For the purposes of tracking material saturation, standards such as ISO 62, ISO 175, ISO 4433 series, ASTM D792, ASTM D5229, ASTM D1505 and ASTM D543 may be used as references. According to ASTM D5229, variations as small as 0,02 % in weight change for consecutive same-interval measurements can be used as references to determine that equilibrium has been attained.

Reference values (unaged dry and/or fluid saturated) shall be agreed between interested parties for the purposes of comparison between aged and un-aged samples.

### 6.2 Test criteria

#### 6.2.1 General

The conformity requires the following:

a) exposure shall be performed in Fluid 2.1 through Fluid 2.3 listed in accordance with 6.2.4;

b) Fluid 2.4 may also be tested and reported as bespoke;

c) testing shall be performed on samples after fluid exposure in accordance with 6.2.3;

d) Level 2 report shall be made available.

The test piece geometry shall be machined before exposure. Great care should be taken to ensure that test geometry is appropriate to get valid failures in post-exposure testing.

The composite material shall be exposed in unconstrained mode; that is, free-standing, with fluid able to freely access all surfaces.

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### 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.

The exposure duration shall start after the material is saturated.

### 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 which 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 can 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: 30 g/kg KCl in de-ionized or distilled water;

c) Fluid 2.3: aromatic hydrocarbon: 70 vol % heptane, 20 vol % cyclohexane, 10 vol % 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 in accordance with ASTM D5229 or ISO 175;

b) dimensions in accordance with ISO 175;

c) one or more mechanical tests selected from Table 2 or Table 3, based upon the failure modes of the application.

It shall be reported whether mechanical test results were based on pre or post aged dimensions.

Additional properties, such as $T_g$ 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

Because of the complexity of the materials a clear definition of threshold criteria encompassing current and potential future applications is not currently possible.

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### 6.3 Preconditioning considerations

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 in accordance with ISO 16396-2 or ISO 1110).

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 shall be reported.

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. Typically, up to 28 day is a sufficient amount of time to characterize a levelling of saturation. If performed, this work shall be reported.

### 6.4 Reporting

The 23936-4 Level 2 test report for each material shall include as a minimum the following in accordance with Annex A:

a) material supplier contact information;

b) test standard details such as method, edition, specimen type, test speed, etc.;

c) specimen identification call out for each test method;

d) exposure conditions (i.e. temperature, pressure and fluids);

e) ISO 23936-4 conformity statement with reference to the data sheet including referencing the version of ISO 23936-4 utilized;

f) table of results with mean, standard deviation and COV (%) at each time duration;

h) description of visual inspection and photographs of specimens after the longest duration regarding:

   1) colour change of the surface after immersion in the medium;

   2) delamination;

   3) appearance of bare fibres and resin loss;

i) a photographic record of representative damage features shall be included in the test report.

EXAMPLE

A report citation example is:

Company XYZ Material ABC

ISO 23936-4 Level 2.1 (at MOL) conformity

ISO 23936-4 Level 2.2 (at MOL) conformity

ISO 23936-4 Level 2.3 (at MOL) conformity

ISO 23936-4 Level 2.4 (at MOL) conformity

## 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. One or more of the tests in accordance with Table 2 or Table 3 shall be performed in order to document the accelerated ageing of composite

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materials exposed to the fluids in accordance with 7.4. Level 3 allows direct comparison of the changes in properties of candidate composite materials.

Ageing at higher temperatures thermally accelerates chemical reactions (if this occurs) between the fluids and the composite material, causing property values to change. Pre-defined 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. The test procedure specified in Annex A shall be followed.

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.

To attempt service life prediction, test plates should be similar in quality to that produced by the processing method to be used for the final part.

The test piece geometry shall be machined before exposure. Great care should be taken to ensure that test geometry is appropriate to get valid failures in post-exposure testing.

The composite material shall be exposed in unconstrained mode; that is, free-standing, with fluid able to freely access all surfaces.

For considerations for short-term thermomechanical evaluation and qualification, see Annex E.

## 7.2 Exposure temperatures

Exposures shall be run at three temperatures ($T_1$, $T_2$ and $T_3$). The temperature difference shall be at least 10 °C and preferably 15 °C. The temperatures chosen should remain in the same regime in relation to critical transition temperatures as the application temperatures reside (i.e. if application uses materials above $T_g$, then choose temperatures also above $T_g$).

$T_1$ should be high enough for the property changes to cross at least one threshold criteria within 56 days or within the longest duration.

A $T_1$ 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 test report. Some materials may 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 may be chosen.

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 three thresholds are met: changes in maximum stress, modulus, and strain at break.

In the example below at $T_1$, three property threshold criteria would have been met by day 21 to allow stoppage at that time. At $T_2$, thresholds can or cannot have been met and the exposure was allowed to run the full 56 days. The $T_3$ immersion study was also allowed to run the full 56 days.

EXAMPLE

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| | | | |
|---|---|---|---|
| $T_1$ | High temperature | 225 °C | 2 days, 7 days, 14 days, 21 days |
| $T_2$ | Medium temperature | 215 °C | 2 days, 7 days, 14 days, 30 days, 56 days |
| $T_3$ | Low temperature | 200 °C | 2 days, 7 days, 14 days, 30 days, 56 days |

## 7.4 Exposure fluids

The multiphase $H_2S$ fluid combines the effects observed in water and hydrocarbon exposures with an addition of $H_2S$. These multiphase fluids can be a combination seen in oil and gas environments and are consistent with existing material ageing standards.

The quantities of exposure fluids are related to an autoclave free volume of 1 000 ml (free volume is the volume of the vessel minus the volume of the specimens and the specimen stands):

a) Fluid 3.1: multiphase 2 mol % sour gas:

   1) water phase: 100 ml de-ionized water or distilled water;

   2) hydrocarbon phase: 600 ml aromatic hydrocarbon (70 vol % heptane, 20 vol % cyclohexane, 10 vol % toluene);

   3) gas phase: 300 ml sour gas (5 mol % $CO_2$, 2 mol % $H_2S$, 93 mol % $CH_4$ at 6 MPa at room temperature).

b) Fluid 3.2: multiphase 10 mol % sour gas:

   1) water phase: 100 ml de-ionized water or distilled water;

   2) hydrocarbon phase: 600 ml aromatic hydrocarbon (70 vol % heptane, 20 vol % cyclohexane, 10 vol % toluene);

   3) gas phase: 300 ml sour gas (5 mol % $CO_2$, 10 mol % $H_2S$, 85 mol % $CH_4$ at 6 MPa at room temperature).

c) Fluid 3.3: as bespoke.

## 7.5 Initial swelling

If it is desired to gather information solely on the physical effects of the liquid phase, the gas phase defined in Fluid 3.1 and Fluid 3.2 according to 7.7 should only be applied to durations greater than 7 days. Durations shorter than or equal to 7 days should 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 6.2.5 shall apply to Level 3.

## 7.7 Threshold criteria

Because of the complexity of the materials a clear definition of threshold criteria encompassing current and potential future applications is not currently possible.

## 7.8 Preconditioning considerations

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 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. Typically, up to 28 day is a sufficient amount of time to characterize saturation. If performed, this work shall be reported.

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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 in accordance with ISO 16396-2 or ISO 1110).

NOTE 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.

## 7.9 Evaluation of data for Level 3

This document acknowledges that due to variety of materials responses to elevated temperature and fluid exposure, an Arrhenius extrapolation for service life prediction may not be feasible. In the following, guidance rules are provided to assist in the selection of materials based upon their response in short-term tests. A dated reference to this document shall be included in the reporting of data and the basis of the analysis of data shall also be clearly stated.

By running exposure tests with test fluids at three different elevated temperatures above the temperature of interest, results in three different times to reach the threshold boundary, 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 at the point where they cross the acceptance criteria limit where defined by user. Approaches to derive such analysis may use linear, polynomial or logarithmic curve fitting methods. The analyst should model the data using different methods and shall use their best judgement to choose the best curve fitting method. One may also choose to ignore initial data points that were generated. The most care should be given to extrapolation of the raw data.

Plotting the logarithm 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. The results should first be assessed on an Arrhenius basis.

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

A lower bound confidence interval approach (e.g. 95 % lower bound) can be used so that a safety criterion can at least partially account for data scatter.

## 7.10 Reporting

The ISO 23936-4 Level 3 test report shall be done in accordance with Annex A. ISO 23936-4 Level 3 conformity statements shall have a structure given below in the report citation example.

EXAMPLE

Company XYZ Material ABC

ISO 23936-4: YYYY Conformity to Level 3, Fluid 3.1 (multiphase 2 mol % sour gas)

ISO 23936-4: YYYY Conformity to Level 3, Fluid 3.2 (multiphase 10 mol % sour gas)

ISO 23936-4: YYYY Conformity to Level 3, Fluid 3.3 (bespoke fluid description)

Guidance procedures are also given to assist in the interpretation of data for each material response category, see Annex A.

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# 8 Level 4 – Material stability (long-term)

## 8.1 General requirements for Level 4 evaluation

This clause defines test procedures for the assessment, evaluation, and prediction of progressive degradation of composite 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 composite 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 conformity pertains to a material stability (long-term) assessment of 180 days or longer.

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. The test procedure specified in Annex A shall be followed.

## 8.2 Exposure temperatures

Exposures shall be run at three temperatures ($T_1$, $T_2$ and $T_3$). The temperature difference shall be at least 10 °C.

$T_1$ shall be high enough for the property changes to cross at least one threshold criteria within 180 days within the longest duration.

A $T_1$ 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 test report.

## 8.3 Exposure durations

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

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 three thresholds are met: changes in maximum stress, modulus, and strain at break.

If, for any chosen temperature, material does not reach threshold criteria within exposure period, curve extrapolation may be allowed and reported if property drop slope is already clearly stablished considering available experimental data and robust statistical analysis is used within data treatment (e.g. adoption of lower bound confidence interval).

## 8.4 Exposure fluids

The Level 4 test fluid requirements shall apply in accordance with 7.4.

## 8.5 Initial swelling

The Level 4 test fluid requirements shall apply in accordance with 7.5.

## 8.6 Property test methods

The Level 4 property test methods shall apply in accordance with 6.2.5.

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## 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 shall be required.

## 8.8 Preconditioning considerations

Preconditioning is exposure to defined 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 may be 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 may not be feasible. In the following, guidance rules are provided to assist in the selection of materials based upon their response in short-term tests. A dated reference to this document shall be included in the reporting of data. The basis of the analysis of data shall be clearly stated.

By running exposure tests with test fluids at least three different elevated temperatures, results in three different times to reach the threshold boundary, 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.

Plotting the logarithm 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.

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 change percentage 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;

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d) preconditioned properties.

## 8.11 Threshold criteria for composites

The threshold value should be left to the application designer to define.

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# Annex A
(normative)

# Test media, conditions, equipment and procedures for ageing of composite materials

## A.1 Test requirements

### A.1.1 General

**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 sulfide 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 test material shall provide a minimum gas cap of 25 vol % of the autoclave volume.

### A.1.2 Test vessel

#### A.1.2.1 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.

However, for hydrocarbon-based fluids, purging before testing should be carefully evaluated as purging itself may lead to extraction of lighter oil fractions from base fluid, jeopardizing fluid original characteristics.

#### A.1.2.2 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 fraction of the fluids shall be presented.

#### A.1.2.3 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.

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### A.1.3 Exposure media

#### A.1.3.1 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. For multiphase fluids where gases ($CO_2$, $H_2S$) can strongly influence overall pH, experimental procedures should be carefully evaluated, and aqueous solution saturation is jeopardized during vessel opening. Software and models can be of use for the purpose of pH prediction.

In addition to production fluids, it may be necessary in many instances to perform application-specific testing of materials in contact with other types of chemicals, 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 in accordance with this document.

For chemical fluids that are not used in a continuous base, tests can be performed considering the total amount of time (total of operations involving the chemical product under evaluation) of the fluid being in contact with the material during the equipment lifespan.

#### A.1.3.2 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, unless signs of fluid degradation (especially hydrocarbon-based ones) require more frequent fluid renewal.

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

#### A.1.3.3 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. Exceptions apply when signs of fluid degradation (especially hydrocarbon-based ones) require more frequent fluid renewal.

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

In case of hydrocarbon-based fluids, it shall be guaranteed that fluid remains representative during sample exposure period. For such, periodic fluid renewal shall be carefully considered whenever necessary.

### A.1.4 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 but 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.

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**Table A.1 — Exposure temperature**

| Temperature of interest °C | Suggested elevated temperatures °C | Temperature of interest °F | Suggested elevated temperatures °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 |

Choice of temperature shall carefully avoid crossing critical temperature values for the material, see Annex F for additional information. Important points are:

a) for thermoset matrix resins, wet $T_g$ of the laminate; maximum test temperature should be 15 °C below wet $T_g$;

b) for thermoplastic materials, user to be aware of typical transitions, for instance, but not limited to, Vicat/HDT temperatures and/or $T_g$ when choosing test temperatures so that representativeness is guaranteed;

c) required service life;

d) fluid types and their maximum temperature limits;

e) pressure.

Wet $T_g$ is difficult to measure if not done in situ due to driving off the fluid during measurements. Testing above wet $T_g$ can provide results showing different degradation mechanisms than are meant to be studied. Conducting test at lower temperatures can be beneficial, see Annex F for more details.

### A.1.5 Exposure pressure

#### A.1.5.1 General

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

In an unstirred liquid medium it takes time to reach the solubility equilibrium of test gas components in the liquid. Therefore, stirring should be done. Non-stirred autoclaves should be left at least overnight to reach an equilibrium.

#### A.1.5.2 Level 2 ageing

The vessel pressure may change 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 Fluid 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.

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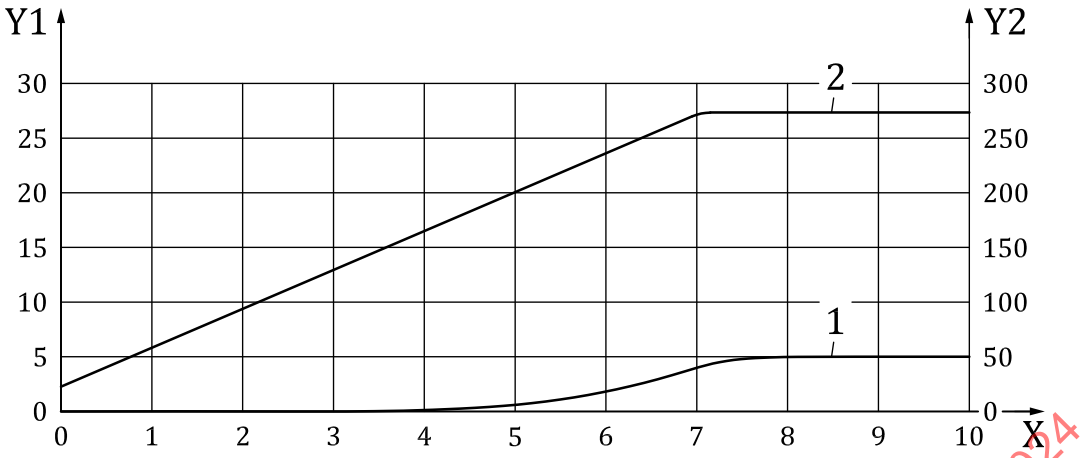
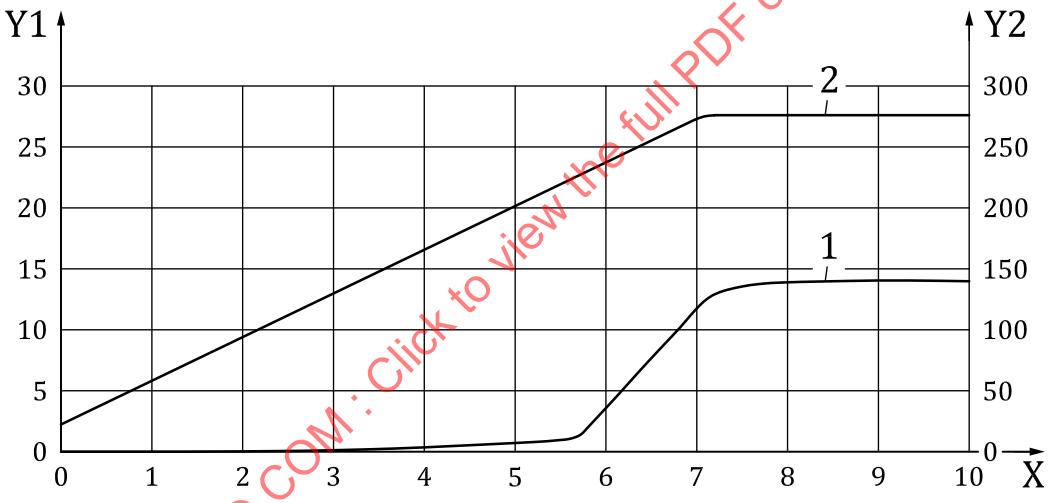



**Key**

| | | | |
|---|---|---|---|
| X | elapsed time in hours | 1 | pressure |
| Y1 | pressure in MPa | 2 | temperature |
| Y2 | temperature in °C | | |

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




**Key**

| | | | |
|---|---|---|---|
| X | elapsed time in hours | 1 | pressure |
| Y1 | pressure in MPa | 2 | temperature |
| Y2 | temperature in °C | | |

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

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### A.1.5.3 Level 3 and Level 4 ageing

Sour multi-phase exposures in Fluid 3.1 and Fluid 3.2 shall be pressurized to (6 ± 0,5) MPa at room temperature and sealed. The vessel pressure rises as the temperature increases and reaches 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.

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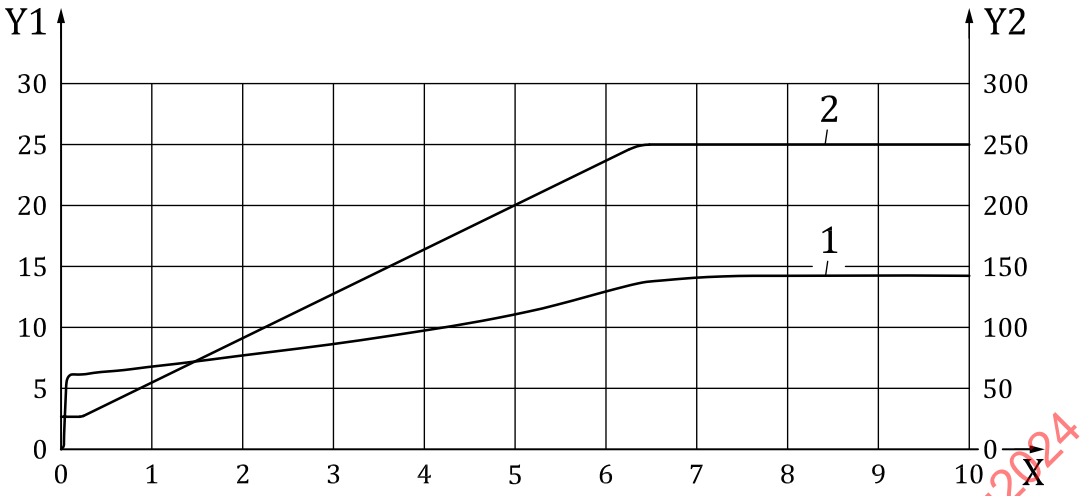


**Key**

| | | | |
|---|---|---|---|
| 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.1.6 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.

The total project time includes the extra time between each duration to allow for cooling, gas depressurization, testing, and reheating. Significant deviations of test temperature and/or pressure during an exposure shall be reported and justified technically.

### A.1.7 Test specimens

Test specimens shall be fabricated with the same compound batch and methods throughout the test program. If samples for different batches or methods are proposed, similarity shall be proven in a traceable manner. DNV-ST-F119 and DNV-ST-C501 can be used as references for sample representativeness. 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.

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

### A.1.8 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 in accordance with the displacement method specified in ISO 1183-1, ISO 2781 or ASTM D792. A balance with an accuracy of 1 mg shall be used.

### A.1.9 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, plasticizers 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 epoxy) in an oven until its weight is at equilibrium. The difference between the mass of the aged wet specimen and the final dry mass of the aged specimen is the amount of the liquid absorbed, and the difference between the final dry mass of the aged specimen and the initial dry mass of the un-aged specimen is the amount of leaching. Therefore, the absorption (mass fraction in %) and the leaching (mass fraction in %) of the test specimen can be calculated by using Formulae (A.1) and (A.2):

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

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

where

$A$ is the absorption as a mass fraction in per cent;

$L$ is the leaching as a mass fraction in per cent;

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

$m_{fd}$ is the final dry mass 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 mass, 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.

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### A.1.10 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;

f) delamination;

g) exposed fibres (or resin poor areas).

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. ASTM D2563 can be used as reference.

### A.1.11 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.

Special care shall be taken in what regards sample conditioning between retrieval from vessel and testing, so that saturation level in the moment of characterization is in accordance with a) bespoke conditioning previously agreed between parts and b) reference data (unaged dry or wet).

## A.2 Test procedure

### A.2.1 General

The sequence of actions as given in this subclause should be followed. The procedure used shall be documented in the test report. Operator 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 should 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 (like heating or cooling periods or pressure increase or decrease);

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e) purge vessel to 5 bar[2)] 5 times with nitrogen (inert) gas having oxygen content < 5 ppm (vol) $O_2$ 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 should 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 (like 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 (vol) $O_2$ 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 $H_2S$, if used, with a scrubber;

j) flush vessel with nitrogen to remove remaining $H_2S$, 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 (see 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 test report for all Level 2, Level 3 and Level 4 reports:

a) conformity statement that details which clause of this document has been satisfied by the testing and a reference to this document, i.e. ISO 23936-4:2024;

b) test standard details such as method, edition, specimen type, test speed;

2) 1 bar = 0,1 MPa = 105 Pa; 1 MPa = 1 N/mm$^2$.

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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) pressure and temperature recording for all test durations;

h) dates for start and end of test;

i) test procedure based on 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, 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 change percentages of properties;

o) graph of change percentages of properties 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, mean, standard deviation, 95 % confidence interval, COV, median and intermediate duration pictures upon request;

t) preconditioning details;

u) leaching results.

### A.3.2 Test report for Level 2

A single composite material can have multiple Level 2 conformity statements and test reports. Test reports can contain numerous data sets and resulting conformity statements. Table A.3, Table A.4 and Table A.5 provide an example of a test report that covers a single data set.

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

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**Table A.3 — Example test report for Level 2 general**

| AcmeManf PhenDura XTRA-6880 conforms to ISO 23936-4:2024 | | |
|---|---|---|
| Conformity statement | - | ISO 23936-4:2024<br>Conformity to Level 2, Fluid 2.2 (250 °C, 30 g/kg KCl) |
| Client address | - | ToolCo Inc.<br>Energy Dr, Denver, CO, USA |
| Compound manufacturer<br>Compound name/number | - | AcmeManf,<br>PhenDura XTRA-6880 |
| Material / reinforcement | - | PEKK / unfilled |
| Recipe revision level | - | n/a |

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**Table A.3** *(continued)*

| AcmeManf PhenDura XTRA-6880 conforms to ISO 23936-4:2024 | | |
|---|---|---|
| Lot/batch no. | - | FF2344rw4r/07 |
| Moulding date | - | Q4 2024 |
| **Ageing test conditions** | | |
| Fluid | Classification | ISO 23936-4:2024 Level 2.2 |
| | Description | 30 g/kg KCl |
| | Liquid | 30 grams KCl in 970 ml of distilled water |
| | Gas<br>Pressure | Nitrogen purge<br>0,138 MPa to 0,345 MPa (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 | Mass fraction (%) | None |
| Test lab<br>Address | - | Ace Test Lab<br>123 Sample Rd, Testing, LA, USA |
| Test date | - | 10 June 2024 - 25 October 2024 |
| Test gas mixture or individual gases certified | YES/NO | YES |
| Pressure calibration available | YES/NO | NO |
| P/T recordings available | YES/NO | NO |

**Table A.4 — Example test report for Level 2 test description**

| Test description | Standard | Specimen identification |
|---|---|---|
| Tensile properties | ISO 527-4 ISO 527-5 or<br>ASTM D3039 | (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) |

**Table A.5 — Example test report for Level 2 single data set**

| 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 |
| 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 |

[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 change percentage data shall be presented with no decimal places.

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**Table A.5** *(continued)*

| Change percentages in properties | | | | | | |
|---|---|---|---|---|---|---|
| Tensile strength Δ (%)[c] | 0 % | -2 % | -2 % | -5 % | -6 % | ±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.

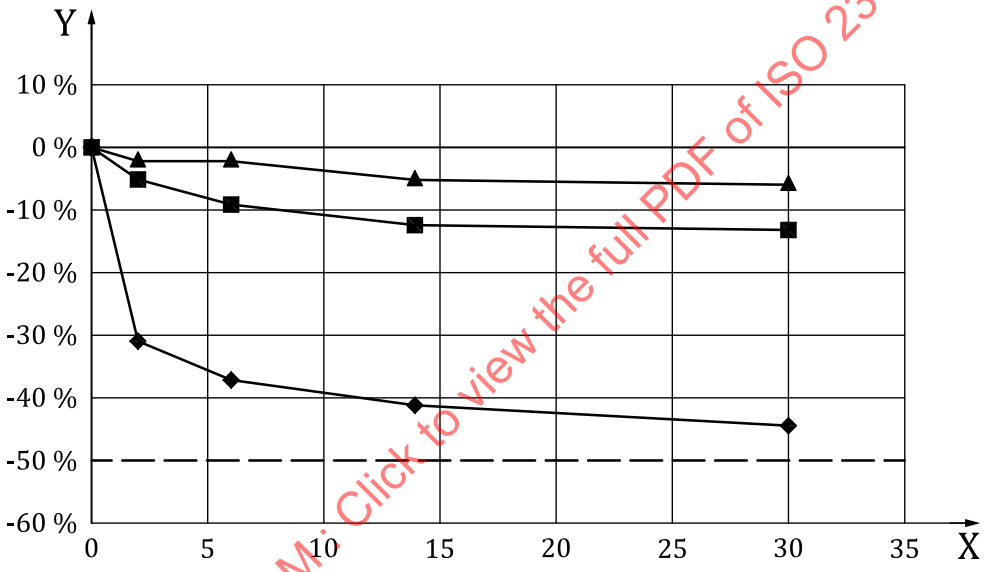
[b] Measured data shall be presented with at least three decimal places.

[c] Calculated change percentage data shall be presented with no decimal places.

The charts for property change percentages 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.




**Key**

X time in days

Y tensile property change in %

tensile strength change in %

modulus change in %

strain at break change in %

threshold criteria

**Figure A.4 — Tensile property change percentages**

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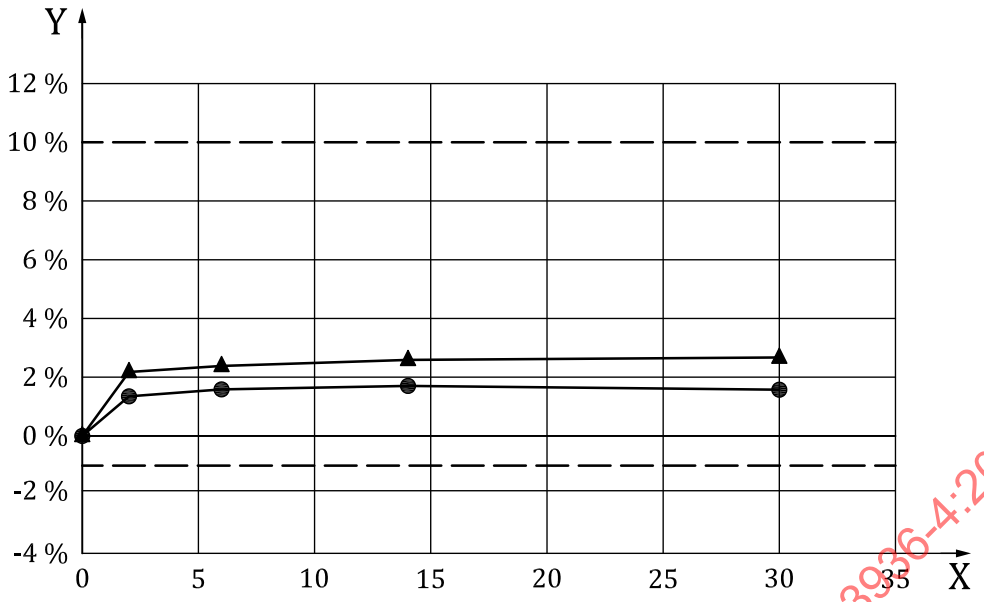
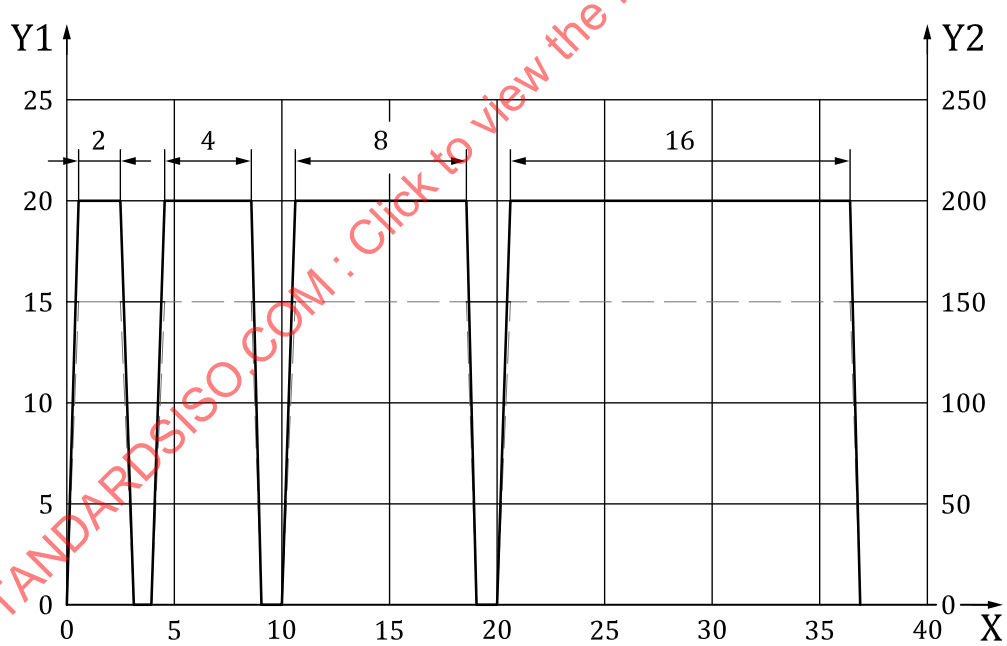



**Key**

| | | | |
|---|---|---|---|
| X | time in days | —●— | mass change in % |
| Y | tensile property change in % | — — | volume change threshold criteria |
| —▲— | volume change in % | | |

**Figure A.5 — Volume and mass property change percentages**




**Key**

| | | | |
|---|---|---|---|
| X | elapsed time in days | ——— | pressure in bar |
| Y1 | pressure in bar | — — | temperature in °C |
| Y2 | temperature in °C | | |

**Figure A.6 — Pressure temperature chart of multiphase fluid**

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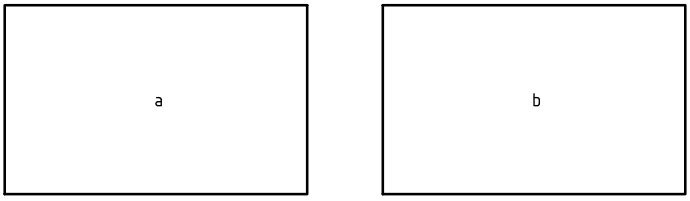


**Key**

a image of test coupon before immersion test b image of test coupon after immersion test

**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 in accordance with A.3.2 and the following additional requirements:

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, change percentages, and visual observations;

d) three sets of final duration specimen images;

e) report data sets for liquid only exposures with a nitrogen purge and gas cap at all three temperatures, if performed.

Table A.6 provides an example of a test report that covers a single data set.

**Table A.6 — Example test report for Level 3**

| **AcmeManf PhenDura XTRA-6880 conforms to ISO 23936-4:2024** | | |
|---|---|---|
| Conformity statement | - | ISO 23936-4:2024<br>Conformity to Level 3, Fluid 3.2 (multiphase 10 mol % sour gas) |
| Client address | - | ToolCo Inc.<br>Energy Dr, Denver, CO, USA |
| Compound manufacturer<br>Compound name/number | - | AcmeManf,<br>PhenDura XTRA-6880 |
| Material / reinforcement | - | PEKK / unfilled |
| Lot/batch no. | - | FF2344rw4r/07 |
| Manufacturing date | - | Q4 2024 |
| **Ageing test conditions** | | |
| Fluid | Classification | ISO 23936-4:2024 Fluid 3.2 |
| | Description | 10 vol % sour aromatic |
| | Liquid | 60 vol % of the autoclave volume, the liquid is composed of 70 vol % heptane, 20 vol % cyclohexane, 10 vol % toluene<br>10 vol % of the autoclave volume: distilled water |
| | Gas<br>Pressure | 30 mol %: 10 mol % $H_2S$, 5 mol % $CO_2$, 85 mol % $CH_4$<br>(6 ± 0,5) MPa |
| Volume ratio minimum | - | 10:1 |
| $T_1$ Exposure temperature | °C (°F) | 232 (450) |
| $T_1$ Pressure at temperature | MPa (psi) | 16,5 (2 400) |
| $T_1$ Exposure durations | days | 2, 7, 14, 27, 42 |

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**Table A.6** *(continued)*

| AcmeManf PhenDura XTRA-6880 conforms to ISO 23936-4:2024 | | |
|---|---|---|
| $T_2$ Exposure temperature | °C (°F) | 200 (392) |
| $T_2$ Pressure at temperature | MPa (psi) | 14,5 (2 100) |
| $T_2$ Exposure durations | days | 7, 14, 30, 42, 56 |
| $T_3$ Exposure temperature | °C (°F) | 180 (356) |
| $T_3$ Pressure at temperature | MPa (psi) | 10,3 (1 500) |
| $T_3$ Exposure durations | days | 7, 14, 28, 41, 57 |
| Specimen preconditioning | - | None |
| Leaching | Mass fraction (%) | None |
| Test lab address | - | Ace Test Lab<br>123 Sample Rd, Testing, LA, USA |
| Test date | - | 10 June 2024 - 25 October 2024 |
| Test gas mixture or individual gases certified | YES/NO | YES |
| Pressure calibration available | YES/NO | YES |
| P/T recordings available | YES/NO | YES |

The example in Table A.7 shows the $T_2$ test data. The $T_1$ and $T_3$ data table shall also be presented in the same format within the test report.

**Table A.7 — Example $T_2$ test data**

| General test details | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Ageing days at 240 °C | 0 | pre-condition-ing | 2 | 7 | 7 | 14 | 30 | 42 | 56 |
| Fluid descrip-tion | none | N/A | 3,2 Liquids only | 3,2 Liquids only | 3,2 | 3,2 | 3,2 | 3,2 | 3,2 |
| Days and toler-ance | 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 |
| Nominal strain at break (%)[a] | 42,0 | N/A | 29,0 | 27,0 | 25,4 | 23,8 | 22,3 | 21,3 | 20,3 |
| Modulus (spec-ified 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 grav-ity[b] | 1,356 | N/A | 1,388 | 1,390 | 1,401 | 1,405 | 1,408 | 1,408 | 1,408 |
| **Change percentage in tensile properties using 2 day liquid saturation as the baseline** | | | | | | | | | |
| Tensile strength Δ (%)[c] | - | N/A | 0 % | - | 0 % | -3 % | -4 % | -5 % | -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 change percentage data shall be presented with no decimal places.

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**Table A.7** *(continued)*

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| 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 % |
| **Change percentage in tensile properties using 7 day liquid saturation as the baseline** | | | | | | | | | |
| Tensile strength Δ (%)[c] | - | N/A | - | 0 % | -2 % | -4 % | -5 % | -5 % | -7 % |
| 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 % |
| **Change percentage in tensile properties using preconditioning as the baseline** | | | | | | | | | |
| Tensile strength 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 |
| **Change percentage 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 change percentage data shall be presented with no decimal places.

The examples in Figure A.8 and Figure A.9 show the $T_1$ data charts. The $T_2$ and $T_3$ data charts shall also be presented in the same format within the test report. The baseline selection shall be identified in the data charts.

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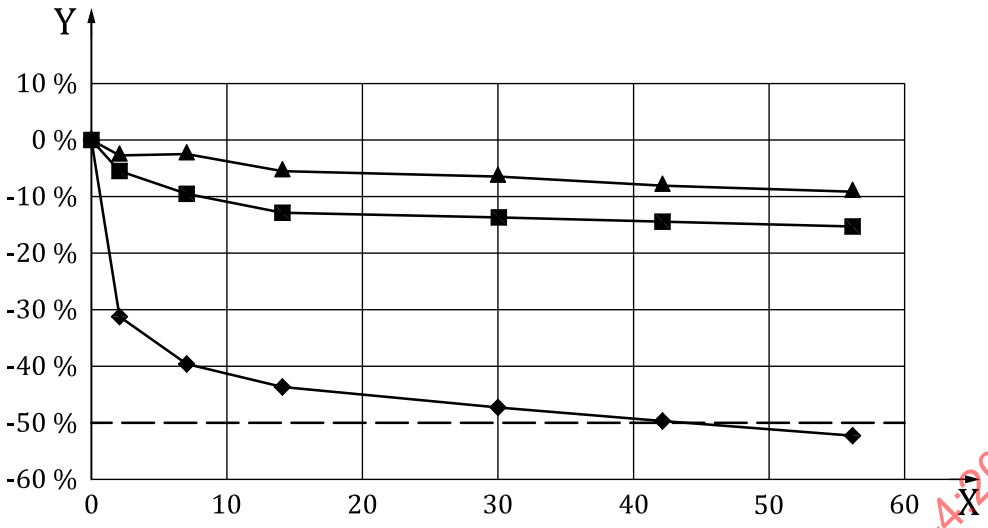
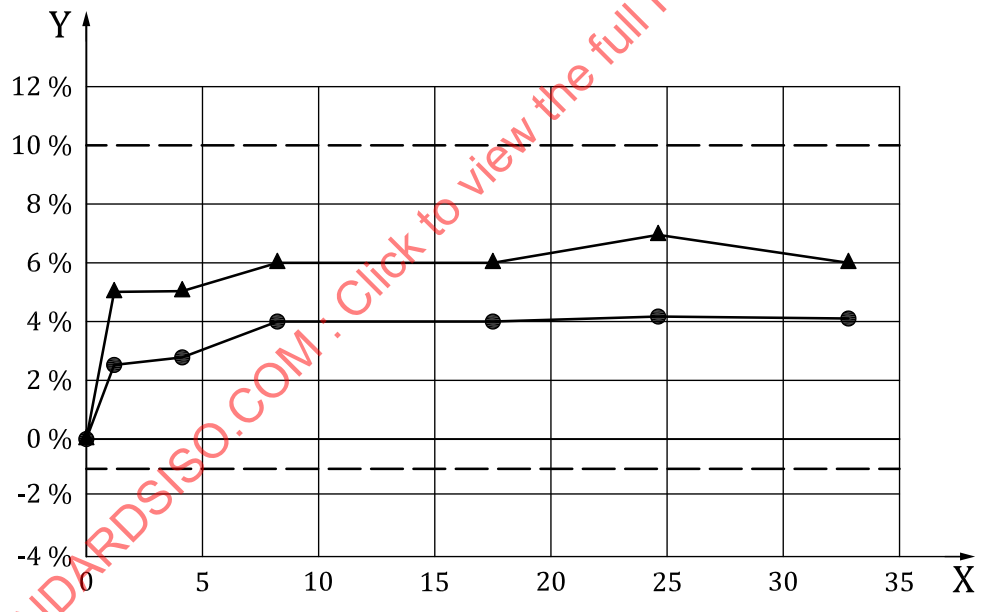



**Key**

| | | | |
|---|---|---|---|
| X | time in days | ■ | modulus change in % |
| Y | tensile property change in % | ◆ | strain at break change in % |
| ▲ | tensile strength change in % | — — | threshold criteria |

**Figure A.8 — Example $T_1$ tensile property change percentages using 2 day baseline data**




**Key**

| | | | |
|---|---|---|---|
| X | time in days | ● | mass change in % |
| Y | property change in % | — — | volume change threshold criteria |
| ▲ | volume change in % | | |

**Figure A.9 — Example $T_1$ 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 in accordance with A.3.3 and the following additional requirements:

a) all three exposure temperatures and the durations selected for each temperature;

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b) threshold baseline selection and justification;

c) life estimation analysis and results.

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# Annex B
(normative)

# Fabrication of laminated plates

## B.1 Fabrication of laminated plates with thermoplastic matrices

### B.1.1 General

The method for producing test plates by molding layers of prepregs (preimpregnated unidirectional fibres or fabrics) should be used according to ISO 1268-4.

General conditions like verification of the characteristics of the plates should be done according to ISO 1268-1.

### B.1.2 Hot pressing

The pressing mould is used for heating and cooling. The variotherm process leads to long cycle times of pressing. Often this become more than 1,5 h for minimum 4 mm thick plates.

On a certain level hot pressing cures poor prepreg qualities. For example, for tapes such defects can be poor fibre impregnation or axial fractures within the tapes.

### B.1.3 Isothermal consolidation

The TI process describes a pressureless contact heating, followed by isothermal cooling and so it is also called hot transfer pressing. The separation of heating and cooling enables high speed press cycles. So this process is much more efficient especially for high demands of thin plates or for process parameter driven studies.

Isothermal consolidation should be done for the thermoplastics materials of this document according to ISO 1268-9, which specifies the compression molding of glass fibre mat reinforced thermoplastic (GMT) and sheet thermoplastic composite (STC), including plates moulded from UD-tapes. The demolding shall take place below glass transition temperature, which can influence the mould temperature as defined at ISO 1268-9.

The undulation of fibres can have a bigger effect on the properties of plates or extracted specimen.

### B.1.4 Autoclave consolidation

During the autoclave process, consolidation of prepreg laminas through simultaneous elevated pressure and temperature results in a uniform high-end material system. A controlled pressure vessel (autoclave) applies pressure during the consolidation process while the temperature is being applied locally by heat blankets. This vessel gives the ability to design manufacturing processes with different pressures while applying temperature at desired regions of the composite.

### B.1.5 Automated tape laying or automated fibre placement

Automated tape laying (ATL) consists of placing composite tape on flat or moderately curved surfaces to fabricate multi-layered composite structures. The placement of tape by ATL machines is precisely controlled by a computer to create layers with specific alternating fibre orientations aligned with the expected loads for high mechanical strength.

AFP is designed for composite tape placement on curved or complex surfaces to fabricate multi-layered composite structures.

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The most unique feature of both processes is the use of fabric or unidirectional fibre prepregs.

The heating systems currently found are:

— hot gas torches;

— laser heaters;

— pulsed light heaters.

## B.2 Fabrication of laminated plates with thermoset based matrices

### B.2.1 General

Recommended methods for fabrication of the continuous fibre-reinforced, laminated composite test plates with thermoset based matrices include wet layup lamination, prepreg lamination, filament winding lamination and vacuum-assisted resin transfer moulding. ISO 1268-3, ISO 1268-4, ISO 1268-5 and ISO 1268-7, and ASTM D5687/D5687M-95, do provide general guideline and detailed procedures for these fabrication methods, and should be used as the guidance for fabrication of the laminated test plates with thermoset based matrices in a reproducible manner in this document, so that it is possible to compare the test results carried out from the test plates fabricated by different producers or different batches, or tested at different times or in different places.

### B.2.2 Wet layup lamination

LW is a contact moulding method for fabricating thermoset-matrix laminates by a wet lamination process using woven or non-woven fabrics with a liquid thermosetting resin, and consolidated and cured under a controlled press with two heated parallel mould plates. Glass- or carbon-fibre, woven or non-woven, unidirectional or 0/90 fabrics are used as the reinforcement, and polyester, vinyl ester, epoxy or other liquid thermosetting resins are used as matrices depending on the application requirements. The standard size of the test plates shall be 300 mm × 300 mm × 3,20 mm, and a set of spacers is used to control the laminate thickness. Number of fabric layers needed to produce the required laminate thickness can be calculated from the fibre density, resin density, fibre content by mass and the area mass of the fabric per ISO 1268-3.

For fabric wet layup, resin viscosity should be a minimum of 1 000 mPa·s at 23 °C. Moulding pressure shall be 5 bar to 10 bar. Curing temperature is determined by the type of resin system. After consolidation and cure, the four sides of the moulded laminate should be cut initially along the principal fibre direction and at least 15 mm from the moulded edges. Before the initial cutting, the principal fibre direction of the laminate should be marked on the plate clearly. ISO 1268-3 and the specific resin technical data sheets provide information on detailed processing procedure and parameters. Dimensions and flatness, fibre content and, void content for the laminated test plates produced by this method shall be done in accordance with Table B.1.

### B.2.3 Prepreg lamination

LP is a more advanced contact moulding method for fabricating thermoset-matrix composite laminates by using pre-impregnated unidirectional tapes or pre-impregnated fabrics, the prepregs, and more advanced consolidation and cure process. With this lamination method, the produced laminated plates exhibit better and more consistent quality. Continuous fibre-reinforced prepreg products include glass or carbon fibre unidirectional tapes pre-impregnated with latent and partially cured epoxy or other advanced thermosetting resins, and glass- or carbon-fibre fabrics pre-impregnated with latent and partially cured epoxy or phenolic resins. Lamina thickness, fibre content and tack of the prepreg products are accurately controlled. The standard sizes of the test plates in this document are 300 mm × 300 mm × 3,20 mm. Unidirectional $[0]_n$ and symmetric cross-lamina $[0/90]_{ns}$ laminate plates should be produced from unidirectional prepreg tapes or unidirectional and [0/90] prepreg fabrics. Number of prepreg layers needed for producing the required laminates can be calculated from lamina thickness and the required laminate thickness.

The prepreg lamination process involves:

a) contact mould or mould plate preparation;

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b) prepreg cutting and laminate layup per the required orientation and stacking sequence;

c) debulking, air breather and vacuum bagging (for autoclave consolidation);

d) laminate consolidation and cure by using a controlled hot-press or an autoclave;

e) initial cutting to the four sides of the moulded laminate along the principal fibre direction, and at least 15 mm from the moulded edges. Before the initial cutting, the principal fibre direction of the laminate should be marked on the plate clearly.

ISO 1268-4, ASTM D5687/D5687M-95 provide information on specific prepreg technical data sheets for detailed processing procedure and parameters. Dimensions and flatness, fibre content and, void content for the laminated test plates produced by this method shall be done in accordance with Table B.1.

### B.2.4 Filament winding lamination

LF is a contact moulding method with continuous fibre placement by wet filament winding process to produce unidirectional composite laminate plates. ISO 1268-5 described a filament winding method by using a specially designed winding former with two parallel contact moulding surfaces. The wound two unidirectional composite plates can be consolidated and cured under a controlled hot-press or in an oven with two out-mould contact moulding plates under clamping pressure. The specified dimensions of the wound unidirectional composite plate are 300 mm × 220 mm × 3 mm. Various glass roving or carbon fibre tow products are used with polyester or epoxy liquid resins in this winding process. ISO 1268-5 and the specific roving/tow and resin technical data sheets provide information on detailed winding parameters and the processing procedure. Dimensions and flatness, fibre content and, void content for wound unidirectional laminated test plates produced by this method shall be done in accordance with Table B.1.

### B.2.5 Vacuum-assisted resin transfer moulding

The LR is a closed mould moulding method, where a fibre preform is placed in the mould cavity and is infused by resin flow injected from a resin pump under vacuum extraction. This method can be used to produce fabric or mat reinforced laminate plates with moulded surfaces on the both sides of the laminates. A resin transfer mould, a controlled air-circulated oven, a resin pump and a vacuum pump or vacuum source are required in this process. Glass- or carbon-fibre woven fabric, or non-woven or non-crimp fabric can be used in stacking of the preform and produce unidirectional or [0/90] laminated test plates.

The resin transfer moulding process involves:

a) assemble the mould and place the preform in the mould cavity;

b) connect the mould to the vacuum pump and the resin pump;

c) apply vacuum to extract the air out and pump the resin from the resin tank in to the preform in the mould cavity;

d) cure the laminates in the mould in the controlled oven;

e) open mould and apply post cure if needed;

f) cut the four sides of the moulded laminate along the principal fibre direction and at least 15 mm from the moulded edges. Before the initial cutting, the principal fibre direction of the laminate should be marked on the plate clearly.

ISO 1268-7 and the specific resin technical data sheets provide information on detailed processing procedure and parameters. Dimensions and flatness, fibre content and, void content for wound unidirectional laminated test plates produced by this method shall be done in accordance with Table B.1.

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**Table B.1 — Required dimensions, flatness, fibre content and void content of cured laminated test plates produced by various fabrication methods**

| Fabrication method | Test plate dimensions mm | | | Flatness[a] mm/plate | Fibre content vol % | | Void content vol % |
|---|---|---|---|---|---|---|---|
| | *L* | *W* | *t* | | [0] | [0/90] | |
| Wet compression moulding | 300 | 300 | 2,5 to 3,5 ± 0,050 | ≤ 1,5 | 50 to 60 | 50 to 58 | ≤ 4 |
| Prepreg moulding | 300 | 300 | 2,5 to 3,5 ± 0,050 | ≤ 1,5 | 52 to 64 | 52 to 62 | ≤ 2 |
| Filament winding | 300 | 220 | 2,5 to 3,5 ± 0,050 | ≤ 1,5 | 50 to 55 | - | ≤ 4 |
| Resin-transfer moulding | 300 | 300 | 2,5 to 3,5 ± 0,050 | ≤ 1,5 | 50 to 62 | 50 to 62 | ≤ 2 |

[a] For bending and twisting control.

## B.3 Curing method for laminated plates with thermoset based matrices

### B.3.1 General

Curing is an irreversible chemical process or a polymerization process of a thermosetting resin. It is commonly accomplished by addition of a curing or cross-linking agent, with or without heat and pressure. For advanced composite materials, especially high-temperature composite materials, a proper curing and consolidation process should be carried out with a designed time-temperature-pressure cycle in certain curing equipment.

### B.3.2 Oven cured

Curing process of the laminated plates is carried out in a controlled, air-circulated oven. For initial cure, the wet processed laminates should be cured with the contact moulding plates, filament winding former, or resin-transfer mould. Vacuum bagging can be used with open mould for consolidation and removal of trapped air or gasses, but the applied pressure is limited to the difference to the atmospheric pressure. The oven can be also used for free standing post cure of the initially cured laminated plates. The oven should be properly calibrated in temperature and its distribution, and equipped with the capabilities of temperature control, monitoring and recording.

### B.3.3 Hot-press cured

Curing process of the laminated plates is carried out in a controlled hot press. The hot press is equipped with two heated, parallel moulding plates, and the wet-layup or prepreg processed laminated plate is placed between the two moulding plates. Pressure, temperature and the cycle time can be readily controlled. Good thickness tolerance can be achieved.

### B.3.4 Autoclave cured

Curing process of the laminated plates is carried out in an autoclave. An autoclave system allows a well-controlled, complex curing process to occur in a pressure vessel according to a specified time, temperature and pressure profile. The major elements of an autoclave system include a pressure vessel, heating and circulation source, pressure source, vacuum bagging system, operating control system and mould loading system. In this process, the uncured laminate plate is first loaded in to the autoclave with the open moulding plate, then vacuum bagging is used to remove trapped air or gasses, and then an optimized heating and pressurization cycle is applied to consolidate and cure the processed laminate plate. Multiple laminated plates can be cured simultaneously if multiple moulds are available and the autoclave is big enough with multiple vacuum ports. Autoclave curing is a more expensive curing process, but with more precisely controlled curing cycle and can produce laminated plates with more consistent top quality.

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### B.3.5 UV cured

UV curing is an emerging speed curing process in which high-intensity ultraviolet light is used to create a photochemical reaction that instantly cures coatings, adhesives and even fast cures certain composite materials. This is a low-temperature, high-speed, solventless polymerization process. UV light can be generated by mercury vapour lamps, fluorescent lamps and emerging UV LED devices. Specially formulated UV cure resins available in the market include UV cure polyester, vinyl ester and epoxy resins.

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# Annex C
(normative)

# Specimen extraction from laminated plates

## C.1 Extraction of test specimen from laminated plates with thermoplastic matrices

### C.1.1 General

The different machining methods for specimen extraction are described by NPL (2020) Measurement Good Practice Guide No. 38.

Irrespective of the method used for extraction of specimens the presence of water or other fluids can influence the specimen performance and therefore if some water uptake is foreseen then the specimen shall be dried immediately after extraction. Test plates shall not be reduced in thickness through sample extraction. Table C.1 is not claiming to be a result of a direct comparison test. It only gives a tentative overview about the strength and weakness of different extraction methods based on some practical experience.

Table C.1 shows a tentative comparison of different specimen extraction methods.

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