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Development of the requirements on fibre-reinforced plastics (FRP) tanks

Transmitted by the Government of France

Introduction

1. Following document ECE/TRANS/WP.15/AC.1/2021/5, we propose to adapt the existing requirements of RID/ADR Chapter 6.9 to technical developments in this area on the basis of the requirements adopted for the Model Regulations.

2. With the introduction of the new Chapter 6.9 for FRP portable tanks, the existing RID/ADR Chapter 6.9 would become Chapter 6.13.

3. The aim of the following proposal is to present how such development should be done.

Proposal

4. Replace Chapter 6.9 by Chapter 6.13 as follows. Comparing to the existing Chapter 6.9, the additional text appears in blue and the deleted text is stricken through.

CHAPTER 6.139

REQUIREMENTS FOR THE DESIGN, CONSTRUCTION, EQUIPMENT, TYPE APPROVAL, TESTING AND MARKING OF FIBRE-REINFORCED PLASTICS (FRP) FIXED TANKS (TANK-VEHICLES), DEMOUNTABLE TANKS[, TANK-CONTAINERS AND TANK SWAP BODIES]

NOTE: For portable tanks and UN multiple-element gas containers (MEGCs) see Chapter 6.7; for FRP portable tanks see Chapter 6.9, for fixed tanks (tank-vehicles), demountable tanks and tank-containers and tank swap bodies, with shells made of metallic materials, and battery-vehicles and multiple element gas containers (MEGCs) other than UN MEGCs see Chapter 6.8; for vacuum operated waste tanks see Chapter 6.10.

6.139.1 General

- 6.139.1.1 FRP tanks shall be designed, manufactured and tested in accordance with a quality assurance programme in accordance with 6.13.1.2 recognized by the competent authority; in particular, lamination work and welding of thermoplastic liners shall only be carried out by qualified personnel in accordance with a procedure recognized by the competent authority.
- 6.13.1.2 *Manufacturer's quality system*
- 6.13.1.2.1 The quality system shall contain all the elements, requirements, and provisions adopted by the manufacturer. It shall be documented in a systematic and orderly manner in the form of written policies, procedures, and instructions.
- 6.13.1.2.2 The contents shall in particular include adequate descriptions of:
 - (a) The organizational structure and responsibilities of personnel with regard to design and product quality;
 - (b) The design control and design verification techniques, processes, and procedures that will be used when designing the portable tanks;
 - (c) The relevant manufacturing, quality control, quality assurance and process operation instructions that will be used;
 - (d) Quality records, such as inspection reports, test data and calibration data;
 - (e) Management reviews to ensure the effective operation of the quality system arising from the audits in accordance with 6.13.1.2.4;
 - (f) The process describing how customer requirements are met;
 - (g) The process for control of documents and their revision;
 - (h) The means for control of non-conforming portable tanks, purchased components, in-process and final materials; and
 - (i) Training programmes and qualification procedures for relevant personnel.
- 6.13.1.2.3 Under the quality system, the following minimum requirements shall be met for each FRP portable tank manufactured:
 - (a) Use of an inspection and test plan (ITP);
 - (b) Visual inspections;
 - (c) Verification of fibre orientation and mass fraction by means of documented control process;

- (d) Verification of fibre and resin quality and characteristics by means of certificates or other documentation;
- (e) Verification of liner quality and characteristics by means of certificates or other documentation;
- (f) Verification of whichever is applicable of formed thermoplastic resin characteristic or degree of cure of thermoset resin, by direct or indirect means (e.g. Barcol test or differential scanning calorimetry) to be determined in accordance with 6.13.4.2.2 (h), or by creep testing of a representative sample or parallel-shell specimen in accordance with 6.13.4.2.2 (e) for a period of 100 hours;
- (g) Documentation of whichever is applicable of thermoplastic resin forming processes or thermoset resin cure and post-cure processes; and
- (h) Retention and archiving of shell samples for future inspection and shell verification (e.g. from manhole cut out) for a period of 5 years.
- 6.13.1.2.4 Audit of the quality system

The quality system shall be initially assessed to determine whether it meets the requirements in 6.13.1.2.1 to 6.13.1.2.3 to the satisfaction of the competent authority.

The manufacturer shall be notified of the results of the audit. The notification shall contain the conclusions of the audit and any corrective actions required.

Periodic audits shall be carried out, to the satisfaction of the competent authority, to ensure that the manufacturer maintains and applies the quality system. Reports of the periodic audits shall be provided to the manufacturer.

6.13.1.2.5 Maintenance of the quality system

The manufacturer shall maintain the quality system as approved in order that it remains adequate and efficient.

The manufacturer shall notify the competent authority that approved the quality system of any intended changes. The proposed changes shall be evaluated to determine whether the amended quality system will still satisfy the requirements in 6.13.1.2.1 to 6.13.1.2.3.

- 6.139.1.2 For the design and testing of FRP tanks, the provisions of 6.8.2.1.1, 6.8.2.1.7, 6.8.2.1.13, 6.8.2.1.14 (a) and (b), 6.8.2.1.25, 6.8.2.1.27, 6.8.2.1.28 and 6.8.2.2.3 shall also apply.
- 6.139.1.3 Heating elements shall not be used for FRP tanks If a heating system is installed, it shall comply the following requirements:
 - (a) The heating system shall be designed or controlled so that a substance cannot reach a temperature at which the pressure in the tank exceeds its maximum working pressure or causes other hazards (e.g. dangerous thermal decomposition);
 - (b) The maximum operating temperature of the heating elements integrated or connected to the shell shall not exceed the maximum design temperature of the tank;
 - (c) The heating elements shall be designed, controlled, and utilized so that the temperature of the carried substance cannot exceed the maximum design temperature of the tank or a value at which the internal pressure exceeds the maximum working pressure;
 - (d) The heating system shall be designed or controlled so that power for internal heating elements shall not be available unless the heating elements are completely submerged. The temperature at the surface of the heating elements for internal heating equipment, or the temperature at the shell for external heating equipment

shall, in no case, exceed 80% of the autoignition temperature (in $^{\circ}$ C) of the substance carried;

- (e) If an electrical heating system is installed inside the tank, it shall be equipped with an earth leakage circuit breaker with a releasing current of less than 100 mA;
- (f) Electrical switch cabinets mounted to tanks shall not have a direct connection to the tank interior and shall provide protection of at least the equivalent of type IP56 according to IEC 144 or IEC 529:
- (g) Structures of the tank and its heating elements shall allow examination of the shell with respect to possible effects of overheating.
- 6.139.1.4 For the stability of tank-vehicles, the requirements of 9.7.5.1 shall apply.

6.139.2 Construction

- 6.139.2.1 Shells shall be made of suitable materials, which shall be compatible with the substances to be carried in a service temperature range of between -40°C and +50°C, unless temperature ranges are specified for specific climatic conditions by the competent authority of the country where the transport operation is performed.
- 6.139.2.2 Shells shall consist of the following three elements :
 - internal liner,
 - structural layer,
 - external layer.

NOTE: The layers may be combined if all applicable functional criteria are met.

6.139.2.2.1 The internal liner is the inner shell wall zone designed as the primary barrier to provide for the long-term chemical resistance in relation to the substances to be carried, to prevent any dangerous reaction with the contents or the formation of dangerous compounds and any substantial weakening of the structural layer owing to the diffusion of products through the internal liner. Chemical compatibility shall be verified in accordance with 6.13.4.2.3.

The internal liner may either be a FRP liner or a thermoplastic liner.

- 6.139.2.2.2 FRP liners shall consist of:
 - (a) surface layer ("gel-coat"): adequate resin rich surface layer, reinforced with a veil, compatible with the resin and contents. This layer shall have a fibre mass content of not more than 30% and have a thickness between 0.25 and 0.60 mm;
 - (b) strengthening layer(s): layer or several layers with a minimum thickness of 2 mm, containing a minimum of 900 g/m² of glass mat or chopped fibres with a mass content in glass of not less than 30% unless equivalent safety is demonstrated for a lower glass content.
- 6.139.2.2.3 Thermoplastic liners shall consist of thermoplastic sheet material as referred to in 6.139.2.3.4, welded together in the required shape, to which the structural layers are bonded. Durable bonding between liners and the structural layer shall be achieved by the use of an appropriate method adhesive.

NOTE: For the carriage of flammable liquids the internal layer may require additional measures in accordance with 6.139.2.14, in order to prevent the accumulation of electrical charges.

6.139.2.2.4 The structural layer of the shell is the zone specially designed according to 6.139.2.4 and to 6.139.2.56 to withstand the mechanical stresses. This part normally consists of several fibre reinforced layers in determined orientations. 6.139.2.2.5 The external layer of resin or paint is the part of the shell which is directly exposed to the atmosphere. It shall eonsist of a resin rich layer with a thickness of at least 0.2 mm. For a thickness larger than 0.5 mm, a mat shall be used. This layer shall have a mass content in glass of less than 30% and shall be capable of withstanding exterior conditions, in particular the occasional contact with the substance to be carried. The resin shall contain fillers or additives to provide protection against deterioration of the structural layer of the shell by ultra-violet radiation.

6.139.2.3 Raw materials

6.139.2.3.1 All materials used for the manufacture of FRP tanks shall be of known origin and specifications.

6.139.2.3.2 Resins

The processing of the resin mixture shall be carried out in strict compliance with the recommendations of the supplier. This concerns mainly the use of hardeners, initiators and accelerators. These resins can be:

- unsaturated polyester resins;
- vinyl ester resins;
- epoxy resins;
- phenolic resins;
- thermoplastic resins.

The heat distortion temperature (HDT) of the resin, determined in accordance with EN ISO 75-1:20202013 shall be at least 20°C higher than the maximum service temperature of the tank, but shall in any case not be lower than 70 °C.

6.139.2.3.3 *Reinforcement fibres*

The reinforcement material of the structural layers shall be a suitable grade of fibres such as glass fibres of type E or ECR according to ISO 2078:1993 + Amd1:2013. For the internal surface liner, glass fibres of type C according to ISO 2078:1993 + Amd1:2013 may be used. Thermoplastic veils may only be used for the internal liner when their compatibility with the intended contents has been demonstrated.

6.139.2.3.4 *Thermoplastic liner material*

Thermoplastic liners, such as unplastified polyvinyl chloride (PVC-U), polypropylene (PP), polyvinylidene fluoride (PVDF), polytetrafluoroethylene (PTFE), etc. may be used as lining materials.

6.139.2.3.5 *Additives*

Additives necessary for the treatment of the resin, such as catalysts, accelerators, hardeners and thixotropic substances as well as materials used to improve the tank, such as fillers, colours, pigments etc. shall not cause weakening of the material, taking into account lifetime and temperature expectancy of the design.

- 6.139.2.4 Shells, their attachments and their service and structural equipment shall be designed to withstand without loss of contents (other than quantities of gas escaping through any degassing vents) during the design lifetime:
 - the static and dynamic loads in normal conditions of carriage;
 - the prescribed minimum loads as defined in 6.139.2.5 to 6.139.2.10.

6.139.2.5 At the pressures as indicated in 6.8.2.1.14 (a) and (b), and under the static gravity forces caused by the contents with maximum density specified for the design and at maximum filling degree, the design stress σ in longitudinal and circumferential direction of any layer of the shell failure criteria (FC) in the longitudinal direction, circumferential direction, and any other in-plane direction of the composite layup shall not exceed the following value:

$$\sigma \leq \frac{R_{m}}{K}$$

$$FC \leq \frac{1}{K}$$

where:

R_m = the value of tensile strength given by taking the mean value of the test results minus twice the standard deviation of the test results. The tests shall be carried out, in accordance with the requirements of EN ISO 527 4:1997 and EN ISO 527 5:2009, on not less than six samples representative of the design type and construction method;

$$\mathbf{K} = \mathbf{S} \times \mathbf{K}_0 \times \mathbf{K}_1 \times \mathbf{K}_2 \times \mathbf{K}_3$$

where

K shall have a minimum value of 4, and

- S = the safety coefficient. For the general design, if the tanks are referred to in Column (12) of Table A of Chapter 3.2 by a tank code including the letter "G" in its second part (see 4.3.4.1.1), the value for S shall be equal to or more than 1.5. For tanks intended for the carriage of substances which require an increased safety level, i.e. if the tanks are referred to in Column (12) of Table A of Chapter 3.2 by a tank code including the number "4" in its second part (see 4.3.4.1.1), the value of S shall be multiplied by a factor of two, unless the shell is provided with protection against damage consisting of a complete metal skeleton including longitudinal and transverse structural members;
- K₀ = a factor related to the deterioration in the material properties due to creep and ageing and as a result of the chemical action of the substances to be carried. It shall be determined by the formula:

$$K_o = \frac{1}{\alpha\beta}$$

where " α " is the creep factor and " β " is the ageing factor determined in accordance with 6.13.4.2.2 (e) and (f) EN 978:1997 after performance of the test according to EN 977:1997. Alternatively, a conservative value of K₀ = 2 may be applied. In order to determine α and β the initial deflection shall correspond to 2σ When used in calculation, factors α and β shall be between 0 and 1;

 K_1 = a factor related to the service temperature and the thermal properties of the resin, determined by the following equation, with a minimum value of 1:

$$K_1 = 1.25 - 0.0125 (HDT - 70)$$

where HDT is the heat distortion temperature of the resin, in °C;

 $K_2 =$ a factor related to the fatigue of the material; the value of $K_2 = 1.75$ shall be used unless otherwise agreed with the competent authority. For the dynamic design as outlined in 6.8.2.1.2 6.9.2.6 the value of $K_2 = 1.1$ shall be used;

- $K_3 =$ a factor related to resine curing and has the following values:
 - 1.04 where curing is carried out in accordance with an approved and documented process, and the quality system described under 6.13.1.2 includes verification of degree of cure for each FRP portable tank using a direct measurement approach, such as differential scanning calorimetry (DSC) determined via ISO 11357-2:2016, as per 6.13.4.2.2 (h) (i);
 - 1.1 where thermoplastic resin forming or thermoset resin curing is carried out in accordance with an approved and documented process, and the quality system described under 6.13.1.2 includes verification of whichever is applicable formed thermoplastic resin characteristics or degree of cure of thermoset resin, for each FRP portable tank using an indirect measurement approach as per 6.13.4.2.2 (h) (ii), such as Barcol testing via ASTM D2583:2013-03 or EN 59:2016, HDT via ISO 75-1:2020, thermo-mechanical analysis (TMA) via ISO 11359-1:2014, or dynamic thermo-mechanical analysis (DMA) via ISO 6721-11:2019;
 - 1.5 in other cases.

A design validation exercise using numerical analysis and a suitable composite failure criterion is to be undertaken to verify that the plies in the shell are below the allowables. Suitable composite failure criteria include, but are not limited to, Tsai-Wu, Tsai-Hill, Hashin, Yamada-Sun, Strain Invariant Failure Theory, Maximum Strain, or Maximum Stress. Other relations for the strength criteria are allowed upon agreement with the competent authority. The method and results of this design validation exercise are to be submitted to the competent authority.

The allowables are to be determined using experiments to derive parameters required by the chosen failure criteria combined with factor of safety K, the strength values measured as per 6.13.4.2.2 (c), and the maximum elongation strain criteria prescribed in 6.13.2.6. The analysis of joints is to be undertaken in accordance with the allowables determined in 6.13.2.10 and the strength values measured as per 6.13.4.2.2 (g). Buckling is to be considered in accordance with 6.13.2.7. Design of openings and metallic inclusions is to be considered in accordance with 6.13.2.11.

6.139.2.6 At the dynamic stresses, as indicated in 6.8.2.1.2 the design stress shall not exceed the value specified in 6.9.2.5, divided by the factor α .

At any of the stresses as defined in 6.8.2.1.2 and 6.13.2.5, the resulting elongation in any direction shall not exceed the value indicated in the following table or one tenth of the elongation at fracture of the resin determined by ISO 527-2:2012, whichever is lower.

Type of resin	Maximum strain in tension (%)
Unsaturated polyester or phenolic	0.2
Vinylester	0.25
Ероху	0.3
Thermoplastic	See 6.13.2.8

Examples of known limits are presented in the table below.

6.139.2.7 At any of the stresses as defined in 6.9.2.5 and 6.9.2.6, the resulting elongation in any direction shall not exceed 0.2% or one tenth of the elongation at fracture of the resin, whichever is lower.

For the external design pressure the minimum safety factor for linear buckling analysis of the shell shall not be less than three.

- 6.139.2.8 At the specified test pressure, which shall not be less than the relevant calculation pressure as specified in 6.8.2.1.14 (a) and (b) the maximum strain in the shell shall not be greater than the elongation at fracture of the resin.
- 6.139.2.9 The shell shall be capable of withstanding the ball drop test according to 6.9.4.3.3 without any visible internal or external defects.
- 6.139.2.10 The adhesive bondlines and/or overlay laminates used in the joints, including the end joints, the joints of the surge plates and the partitions with the shell shall be capable of withstanding the static and dynamic stresses mentioned above. In order to avoid concentrations of stresses in the overlay lamination, the applied tapper shall not be steeper than 1:6.

The shear strength between the overlay laminate and the tank components to which it is bonded shall not be less than:

$$\tau = \gamma \frac{Q}{l} \le \frac{\tau_R}{K}$$

where:

- τ_R is the bending interlaminar shear strength according to ISO 14130:1997 and Cor 1:2003 EN ISO 14125:1998 + AC:2002 + A1:2011 (three points method) with a minimum of $\tau_R = 10 \text{ N/mm}^2$, if no measured values are available;
- Q is the load per unit width that the joint shall carry under the static and dynamic loads;
- K is the factor calculated in accordance with 6.139.2.5 for the static and dynamic stresses;
- l is the length of the overlay laminate;
- γ is the notch factor relating average joint stress to peak joint stress at failure initiation location.
- 6.139.2.11 Metallic flanges and their closures are permitted to be used in FRP shells, under design requirements of 6.8.2. Openings in the shell shall be reinforced to provide at least the same safety factors against the static and dynamic stresses as specified in 6.139.2.5 and 6.9.2.6 as that for the shell itself. The number of openings shall be minimized. The axis ratio of oval-shaped openings shall be not more than 2.

If metallic flanges or componentry are integrated into the FRP shell using bonding, then the characterisation method stated in 6.13.2.10 shall apply to the joint between the metal and FRP. If the metallic flanges or componentry are fixed in an alternative fashion, e.g. threaded fastener connections, then the appropriate provisions of the relevant pressure vessel standard shall apply.

- 6.139.2.12 For the design of flanges and pipework attached to the shell, handling forces and the fastening of bolts shall also be taken into account.
- 6.13.2.x Check calculations of the strength of the shell shall be performed by finite element method simulating the shell layups, joints within FRP shell, joints between the FRP shell, the attachments and the structure equipment, and openings.
- 6.139.2.13 The tank shall be designed to withstand, without significant leakage, the effects of a full engulfment in fire for 30 minutes as specified by the test requirements in 6.139.4.3.4. Testing may be waived with the agreement of the competent authority, where sufficient proof can be provided by tests with comparable tank designs.
- 6.9139.2.14 Special requirements for the carriage of substances with a flash-point of not more than 60 °C

FRP tanks used for the carriage of substances with a flash-point of not more than 60°C shall be constructed so as to ensure the elimination of static electricity from the various component parts so as to avoid the accumulation of dangerous charges.

- 6.139.2.14.1 The electrical surface resistance of the inside and outside of the shell as established by measurements shall not be higher than 10⁹ ohms. This may be achieved by the use of additives in the resin or interlaminate conducting sheets, such as metal or carbon network.
- 6.139.2.14.2 The discharge resistance to earth as established by measurements shall not be higher than 10^7 ohms.
- 6.139.2.14.3 All components of the shell shall be electrically connected to each other and to the metal parts of the service and structural equipment of the tank and to the vehicle. The electrical resistance between components and equipment in contact with each other shall not exceed 10 ohms.
- 6.139.2.14.4 The electrical surface-resistance and discharge resistance shall be measured initially on each manufactured tank or a specimen of the shell in accordance with a procedure recognized by the competent authority.
- 6.139.2.14.5 The discharge resistance to earth of each tank shall be measured as part of the periodic inspection in accordance with a procedure recognized by the competent authority.
- 6.139.3 Items of equipment
- 6.139.3.1 The requirements of 6.8.2.2.1, 6.8.2.2.2, 6.8.2.2.4 and 6.8.2.2.6 to 6.8.2.2.8 shall apply.
- 6.139.3.2 In addition, when they are shown under an entry in Column (13) of Table A of Chapter 3.2, the special provisions of 6.8.4 (b) (TE) shall also apply.
- 6.139.4 Type testing and approval
- 6.139.4.1 For any design of a FRP tank type, its materials and a representative prototype shall be subjected to the design type testing as outlined below.
- 6.139.4.2 *Material testing*
- 6.139.4.2.1 The elongation at fracture according to EN ISO 527-2:2012 4:1997 or EN ISO 527-5:2009 and the heat distortion temperature according to EN ISO 75-1:20202013 shall be determined for the resins to be used.
- 6.139.4.2.2 The following characteristics shall be determined for samples cut out of the shell. Samples manufactured in parallel may only be used, if it is not possible to use cutouts from the shell. Prior to testing, any liner shall be removed.

The tests shall cover:

- (a) Thickness of the laminates of the central shell wall and the ends;
- (b) Mass content and composition of glass composite reinforcement by ISO 1172:1996 or ISO 14127:2008, orientation and arrangement of reinforcement layers;
- (c) Tensile strength, elongation at fracture and modulus of elasticity according to EN ISO 527-4:1997 or EN ISO 527-5:2009 in the direction of stresses for the circumferential and longitudinal directions of the shell. In addition, the elongation at fracture of the resin shall be established by means of ultrasound. For areas of the FRP shell, tests shall be performed on representative laminates in accordance with ISO 527-4:1997 or ISO 527-5:2009, to permit evaluation of the suitability of safety factor (K). A minimum of six specimens per measure of tensile strength shall be used, and the tensile strength shall be taken as the average minus two standard deviations;
- (d) Bending strength and deflection established by the bending creep test according to EN ISO 14125:1998 + AC:2002 + A1:2011 for a period of 1000 hours using a

sample with a minimum width of 50 mm and a support distance of at least 20 times the wall thickness.

- In addition, tThe creep factor α determined by taking the average result of at least (e) two specimens with the configuration described in (d), subject to creep in threepoint or four-point bending, at the maximum design temperature nominated under 6.13.2.1, for a period of 1 000 hours. The following test is to be undertaken for each specimen:
 - Place specimen into bending apparatus, unloaded, in oven set to maximum (i) design temperature and allow to acclimatise for a period of not less than 60 minutes:
 - (ii) Load specimen bending in accordance with ISO 14125:1998 + Amd 1:2011 at flexural stress equal to the strength determined in (d) divided by four. Maintain mechanical load at maximum design temperature without interruption for not less than 1 000 hours;
 - (iii) Measure the initial deflection six minutes after full load application in (e) (ii). Specimen shall remain loaded in test rig;
 - (iv) Measure the final deflection 1 000 hours after full load application in (e) (ii); and
 - Calculate the creep factor α by dividing the initial deflection from (e) (iii) (v) by the final deflection from (e) (iv).
- and tThe ageing factor β determined by taking the average result of at least two (f) specimens with the configuration described in (d), subject to loading in static three-point or four-point bending, in conjunction with immersion in water at the maximum design temperature nominated under 6.13.2.1 for a period of 1 000 hours. The following test is to be undertaken for each specimen:
 - Prior to testing or conditioning, specimens shall be dried in an oven at (i) 80 °C for a period of 24 hours;
 - The specimen shall be loaded in three-point or four-point bending at (ii) ambient temperature, in accordance with to ISO 14125:1998 + Amd 1:2011, at the flexural stress level equal to the strength determined in (d) divided by four. Measure the initial deflection 6 minutes after full load application. Remove specimen from test rig;
 - (iii) Immerse unloaded specimen in water at the maximum design temperature for a period of not less than 1 000 hours without interruption to the water conditioning period. When conditioning period has lapsed, remove specimens, keep damp at ambient temperature, and complete (f) (iv) within three days;
 - (iv) The specimen shall be subject to second round of static loading, in a manner identical to (f) (ii). Measure the final deflection six minutes after full load application. Remove specimen from test rig; and
 - Calculate the ageing factor β by dividing the initial deflection from (f) (ii) (v) by the final deflection from (f) (iv).

shall be determined by this test and according to EN 978:1997.

- 69423 The interlaminarte shear strength of the joints shall be measured by testing (g) representative samples in the tensile test according to in accordance with EN ISO 14130:1997.
 - (h) The efficiency of whichever is applicable of thermoplastic resin forming characteristics or thermoset resin cure and post-cure processes for laminates are to be determined using one or more of the following methods:
 - Direct measurement formed thermoplastic resin characteristics or (i) thermoset resin degree of cure: glass transition temperature (Tg) or melting

temperature (Tm) determined using differential scanning calorimetry (DSC) via ISO 11357-2:2016; or

- (ii) Indirect measurement of formed thermoplastic resin or thermoset resin degree of cure:
 - HDT via ISO 75-1:2020;
 - Tg or T_m using thermo-mechanical analysis (TMA) via ISO 11359-1:2014;
 - Dynamic thermo-mechanical analysis (DMA) via ISO 6721-11:2019:
 - Barcol testing via ASTM D2583:2013-03 or EN 59:2016.
- 6.139.4.2.34 The chemical compatibility of the liner and chemical contact surfaces of service equipment shell with the substances to be carried shall be demonstrated by one of the following methods with the agreement of the competent authority. This demonstration shall account for all aspects of the compatibility of the materials of the shell and its equipment with the substances to be carried, including chemical deterioration of the shell, initiation of critical reactions of the contents and dangerous reactions between both.
 - In order to establish any deterioration of the shell, representative samples taken from the shell, including any internal liners with welds, shall be subjected to the chemical compatibility test according to EN 977:1997 for a period of 1 000 hours at 50°C. Compared with a virgin sample, the loss of strength and elasticity modulus measured by the bending test according to EN 978:1997 shall not exceed 25%. Cracks, bubbles, pitting effects as well as separation of layers and liners and roughness shall not be acceptable.
 - Certified and documented data of positive experiences on the compatibility of the filling substances in question with the materials of the shell with which they come into contact at given temperatures, times and any other relevant service conditions.
 - Technical data published in relevant literature, standards or other sources, acceptable to the competent authority.
 - Upon agreement with the competent authority other methods of chemical compatibility verification may be used.

6.139.4.3 *Type testing*

A representative prototype tank shall be subjected to tests as specified below. For this purpose service equipment may be replaced by other items if necessary.

- 6.139.4.3.1 The prototype shall be inspected for compliance with the design type specification. This shall include an internal and external visual inspection and measurement of the main dimensions.
- 6.139.4.3.2 The prototype, equipped with strain gauges at all locations where a comparison with the design calculation is required, shall be subjected to the following loads and the strains shall be recorded:
 - Filled with water to the maximum filling degree. The measuring results shall be used to calibrate the design calculation according to 6.139.2.5;
 - Filled with water to the maximum filling degree and subjected to accelerations in all three directions by means of driving and braking exercises with the prototype attached to a vehicle. For comparison with the design calculation according to 6.139.2.56 the strains recorded shall be extrapolated in relation to the quotient of the accelerations required in 6.8.2.1.2 and measured;
 - Filled with water and subjected to the specified test pressure. Under this load, the shell shall exhibit no visual damage or leakage.

- 6.139.4.3.3 The prototype shall be subjected to the ball drop test according to EN 976-1:1997, No. 6.6. No visible damage inside or outside the tank shall occur.
- 6.139.4.3.4 The prototype with its service and structural equipment in place and filled to 80% of its maximum capacity with water, shall be exposed to a full engulfment in fire for 30 minutes, caused by an open heating oil pool fire or any other type of fire with the same effect. The fire shall be equivalent to a theoretical fire with a flame temperature of 800 °C, emissivity of 0.9 and to the tank a heat transfer coefficient of 10 W/(m²K) and surface absorptivity of 0.8. A minimum net heat flux of 75 kW/m² shall be calibrated according to ISO 21843:2018. The dimensions of the pool shall exceed those of the tank by at least 50 cm to each side and the distance between fuel level and tank shall be between 50 cm and 80 cm. The rest of the tank below liquid level, including openings and closures, shall remain leakproof except for drips.
- 6.139.4.4 *Type approval*
- 6.139.4.4.1 The competent authority or a body designated by that authority shall issue in respect of each new type of tank an approval attesting that the design is suitable for the purpose for which it is intended and meets the construction and equipment requirements of this chapter as well as the special provisions applicable to the substances to be carried.
- 6.139.4.4.2 The approval shall be based on the calculation and the test report, including all material and prototype test results and its comparison with the design calculation, and shall refer to the design type specification and the quality assurance programme.
- 6.139.4.4.3 The approval shall include the substances or group of substances for which compatibility with the shell is provided. Their chemical names or the corresponding collective entry (see 2.1.1.2), and their class and classification code shall be indicated.
- 6.139.4.4.4 In addition, it shall include design and threshold values (such as life-time, service temperature range, working and test pressures, material data) specified and all precautions to be taken for the manufacture, testing, type approval, marking and use of any tank, manufactured in accordance with the approved design type.
- 6.9139.5 Inspections
- 6.9139.5.1 For every tank, manufactured in conformity with the approved design, material tests and inspections shall be performed as specified below.
- 6.139.5.1.1 The material tests according to 6.139.4.2.2, except for the tensile test and for a reduction of the testing time for the bending creep test to 100 hours shall be performed with samples taken from the shell. Samples manufactured in parallel may only be used, if no cutouts from the shell are possible. The approved design values shall be met.
- 6.139.5.1.2 Shells and their equipment shall either together or separately undergo an initial inspection before being put into service. This inspection shall include:
 - a check of conformity to the approved design;
 - a check of the design characteristics;
 - an internal and external examination;
 - a hydraulic pressure test at the test pressure indicated on the plate prescribed in 6.8.2.5.1;
 - a check of operation of the equipment;
 - a leakproofness test, if the shell and its equipment have been pressure tested separately.
- 6.139.5.2 For the periodic inspection of tanks the requirements of 6.8.2.4.2 to 6.8.2.4.4 shall apply. In addition, the inspection in accordance with 6.8.2.4.3 shall include an examination of the internal condition of the shell.

6.139.5.3 The inspections and tests in accordance with 6.139.5.1 and 6.139.5.2 shall be carried out by the expert approved by the competent authority. Certificates shall be issued showing the results of these operations. These certificates shall refer to the list of the substances permitted for carriage in this shell in accordance with 6.139.4.4.

6.139.6 Marking

- 6.139.6.1 The requirements of 6.8.2.5 shall apply to the marking of FRP tanks, with the following amendments:
 - the tank plate may also be laminated to the shell or be made of suitable plastics materials;
 - the design temperature range shall always be marked.
- 6.13.6.2 The information required on materials shall be "Shell structural material: Fibre-reinforced plastic", the reinforcement fibre e.g. "Reinforcement: E-glass", and resin e.g. "Resin: Vinyl Ester".
- 6.139.6.32 In addition, when they are shown under an entry in Column (13) of Table A of Chapter 3.2, the special provisions of 6.8.4 (e) (TM) shall also apply.