

Distr.
GENERAL

TRANS/WP.29/GRPE/2002/1
29 October 2001

Original: ENGLISH

ECONOMIC COMMISSION FOR EUROPE

INLAND TRANSPORT COMMITTEE

World Forum for Harmonization of Vehicle Regulations (WP.29)

Working Party on Pollution and Energy (GRPE)
(Forty-third session, 15-18 January 2002,
agenda item 4.1.)

PROPOSAL FOR DRAFT SUPPLEMENT 3
TO THE 01 SERIES OF AMENDMENTS TO REGULATION No. 67

(Equipment for liquefied petroleum gas)

Transmitted by the Expert from the Netherlands

Note: The text reproduced below was prepared by the expert from the Netherlands and distributed without a symbol (informal document No. 24) during the forty-second session of GRPE (TRANS/WP.29/GRPE/42, para. 29).

Note: This document is distributed to the Experts on Pollution and Energy only.

A. PROPOSAL

Insert new paragraphs 2.3.2. and 2.3.3., to read:

"2.3.2. "All-composite container" means a container made only of composite materials with a non metallic liner.

"Batch of containers" means a maximum of 200 containers of the same type produced consecutively on the same production line."

Paragraph 2.4., items (e) and (f) amend to read ,

"...

- (e) the welding process (in case of metal containers),
- (f) the heat treatment (in case of metal containers),

..."

Annex 10,

Insert a new paragraph 1.1., to read:

"1.1. **Cylinders covered by this annex are as follows:**

LPG-1 Metal containers
LPG-4 All-Composite containers."

Paragraph 1.1. (former), renumber as paragraph 1.2.

Paragraphs 1.2. to 1.2.3., renumber as paragraphs 1.3. to 1.3.3.

Paragraph 1.2.4., renumber as paragraph 1.3.4., and amend to read:

"1.3.4. The filler materials must be compatible with the parent material so as to form welds with properties equivalent to those specified for the parent material (EN 288 - 3)."

Paragraph 1.2.5., renumber as paragraph 1.3.5., and amend to read:

"1.3.5. The container manufacturer must obtain and provide:

- (a) for metal containers: chemical cast analysis certificates;
- (a) for all-composite containers: chemical resistance analysis certificates related to tests performed according to the requirements of appendix 6;
- (c) mechanical properties of the material in respect of the steels or other materials applied for the construction of the parts subject to pressure."

Paragraphs 1.2.6. to 1.3.2., renumber as paragraphs 1.3.6. and 1.4.2.

Paragraph 1.4., renumber as paragraph 1.5., and amend to read:

"1.5. **The heat treatment procedures, on metal containers only, shall be according to the following requirements:"**

Paragraphs 1.4.1. to 1.5., renumber as paragraphs 1.5.1. to 1.6.

Insert a new paragraph 1.6.1., to read:

"1.6.1. Calculation of the parts under pressure for metal containers."

Paragraph 1.5.1. (former), renumber as paragraph 1.6.1.1.

Paragraphs 1.5.1.1. and 1.5.1.2., renumber as paragraphs 1.6.1.2. and 1.6.1.3.

Paragraph 1.5.2., renumber as paragraph 1.6.1.2.

Paragraphs 1.5.2.1. to 1.5.2.3., renumber as paragraph 1.6.1.2.1. to 1.6.1.2.3.

Paragraphs 1.5.3. and 1.5.4., renumber as paragraph 1.6.1.3. and 1.6.1.4.

Insert a new paragraph 1.6.2. , to read:

"1.6.2. Calculation of the parts under pressure for all-composite containers

The stresses in the container shall be calculated for each container type. The pressures used for these calculations shall be the design pressure and burst test pressure. The calculations shall use suitable analysis techniques to establish stress distribution throughout the container."

Paragraph 1.6. to 1.6.1.1., renumber as paragraphs 1.7. to 1.7.1.1.

Paragraph 1.6.1.2., renumber as paragraph 1.7.1.2., and amend to read:

"1.7.1.2. The manufacturer must ensure through adequate supervision that the parent materials and pressed parts used to manufacture the containers are free from defects likely to jeopardize the safe use of the containers."

Paragraphs 1.6.2. to 1.6.3., renumber as paragraphs 1.7.2 to 1.7.3.

Paragraph 1.6.3.1., renumber as paragraph 1.7.3.1., and amend to read:

"1.7.3.1. The supports must be manufactured and attached to the container body in such a way as not to cause dangerous concentrations of stresses or be conducive to the collection of water."

Paragraphs 1.6.3.2. to 1.6.4.1., renumber as paragraphs 1.7.3.2 to 1.7.4.1.

Paragraph 2., amend to read:

"2. **TESTS**

Tables 1 and 2 below give an overview of the tests to be performed on the LPG containers on prototypes as well as during the production process according to their nature. All tests shall be performed at ambient temperature of $20\text{ °C} \pm 5\text{ °C}$, unless otherwise stated.

Table 1 – Overview of tests to be performed on metal containers

Test to be performed	Production Batch tests	Number of containers to be tested for type approval	Test description
Tensile test	1 per batch	2 1/	See para. 2.1.2.2.
Bend test	1 per batch	2 1/	See para. 2.1.2.3.
Burst test		2	See para. 2.2.
Hydraulic test	Each container	100 %	See para. 2.3.
Bonfire test		1	See para. 2.6.
Radiographic examination	1 per batch	100 %	See para. 2.6.1.
Macroscopic examination	1 per batch	2 1/	See para. 2.6.2.
Inspection of welds	1 per batch	100 %	See para. 1.6.2.3.
Visual inspection of the parts of the container	1 per batch	100 %	

1/: These test pieces can be taken from one container

Note 1: 6 containers shall be submitted for type approval.

Note 2: On one of these prototypes the volume of the container and the wall thickness of each part of the container shall be determined.

Table 2 – Overview of tests to be performed on all-composite containers

Test to be performed	Production Batch tests	Number of containers to be tested for type approval	Test description
Burst test	1 per batch	3	See para. 2.2.
Hydraulic test	Each container	All containers	See para. 2.3.6.
Ambient temperature pressure cycling test	1 per 5 batches	3	See para. 2.4.1.
High temperature pressure cycling test		1	See para. 2.4.2.
External leak test		1	See para. 2.4.3.
Permeation test		1	See para. 2.4.4.
LPG cycling test		1	See para. 2.4.5.
High temperature creep test		1	See para. 2.4.6.
Bonfire test		1	See para. 2.6.
Impact test		1	See para. 2.7.
Drop test		1	See para. 2.8.
Boss torque test		1	See para. 2.9.
Acid environment test		1	See para. 2.10.
Ultra-violet radiation test		1	See para. 2.11.

Paragraph 2.1.1.1., amend to read:

"2.1.1.1. The frequency of the tests for metal containers shall be: 1 container from each batch during production and for type testing, see Table 1.

Test pieces which are not flat shall be flattened by a cold process.

In test pieces containing a weld, the weld shall be machined to trim the surplus.

Metal containers shall be subjected to the tests as described in Table 1.

Test pieces from containers with one circumferential weld only (two sections) shall be taken from the places shown in appendix 2, Figure 1.

Test pieces from containers with longitudinal and circumferential welds (three or more sections) shall be taken from the places shown in appendix 2, Figure 2."

Paragraphs 2.1.2.2.1.1. and 2.1.2.2.1.2., amend to read:

"2.1.2.2.1.1. The tensile test shall be carried out in accordance with Euronorms EN 876, EN 895 and EN 10002-1.

2.1.2.2.1.2. The values determined for yield stress, tensile strength and elongation after break must comply with the characteristics of the metal as required in paragraph 1.3 of this annex."

Paragraph 2.1.2.3.1. , amend to read:

"2.1.2.3.1. The bend test shall be carried out in accordance with standards ISO 7438:2000 and ISO 7799:2000 and Euronorm EN 910 for welded parts.
The bend tests shall be carried out on the inner surface in tension and the outer surface in tension."

Insert a new paragraph 2.1.2.4., to read:

"2.1.2.4. Retesting for the tensile and bend tests."

Paragraph 2.1.2.4. (former), renumber as paragraph 2.1.2.4.1.

Paragraphs 2.2.2. to 2.2.2.1.2., replace by the following text (paragraphs 2.2.2. to 2.2.2.1.3.):

"2.2.2. **Interpretation of test**

2.2.2.1. The criteria adopted for the interpretation of the burst test are as follows:

2.2.2.1.1. Volumetric expansion of the metal container; it equals: volume of water used between the time when the pressure starts to rise and the time of bursting;

2.2.2.1.2. Examination of the tear and the shape of its edges, for the metal containers and of the failure mode for the all-composite containers;

2.2.2.1.3. The value of the bursting pressure."

Paragraph 2.2.3.2., amend the word "container" to read "metal container" (4-times).

Paragraph 2.2.3.3.2. amend to read:

"2.2.3.3.2. For metal containers the fracture must not reveal an inherent defect in the metal. The weld must be at least as strong as the original metal but preferably stronger.

For all-composite containers, the fracture shall not reveal any defects in the structure."

Insert new paragraphs 2.3.6. to 2.3.6.6.4., to read:

"2.3.6. **Additional hydraulic tests to be performed on all-composite containers**

2.3.6.1. Ambient temperature pressure cycling test

2.3.6.1.1. Test procedure

The finished container shall be pressure cycled to a maximum of 20,000 cycles, according to the following procedure:

- (a) fill the container to be tested with a non-corrosive fluid such as oil, inhibited water or glycol;
- (b) cycle the pressure in the container between not more than 300 kPa and not less than 3,000 kPa at a rate not to exceed 10 cycles per minutes.
This cycle shall be performed at least 10,000 times and continued until 20,000 times unless a leak before break appears;
- (c) the number of cycles to failure shall be reported, along with the location and description of the failure initiation.

2.3.6.1.2. Test interpretation

Before reaching 10,000 cycles, the container shall not fail or leak.
After completing 10,000 cycles, the container may leak before break.

2.3.6.1.3. Retesting

Retesting is permitted for the ambient temperature pressure cycling test.

A second test shall be performed on two containers which have been produced successively to the first container within the same batch.

If the results of these tests are satisfactory, the first test shall be ignored.

In the event where one or both of the retests fail to meet the requirements, the batch shall be rejected.

2.3.6.2. High temperature pressure cycling test

2.3.6.2.1. Test procedure

Finished containers shall be cycle tested, without showing evidence of rupture, leakage, or fibre unravelling, as follows:

- (a) Fill the container to be tested with a non-corrosive fluid such as oil, inhibited water or glycol;
- (b) Condition for 48 hours at 0 kPa, 65 °C, and 95 % or greater relative humidity.
- (c) Hydrostatically pressurize for 3,600 cycles not exceeding 10 cycles per minute, between not more than 300 kPa and not less than 3,000 kPa at 65 °C and 95 % humidity;

Following the pressure cycling at high temperature, containers shall be submitted to the external leak test and then hydrostatically pressurized to failure in accordance with the burst test procedure.

2.3.6.2.2. Retesting

Retesting is permitted for the high temperature pressure cycling test.

A second test shall be performed on two containers which have been produced successively to the first container within the same batch.

If the results of these tests are satisfactory, the first test shall be ignored.

In the event where one or both of the retests fail to meet the requirements, the batch shall be rejected.

2.3.6.3. External leak test

2.3.6.3.1. Test procedure

While under 3,000 kPa pressure, the container without any valves shall be submerged in soapy water to detect leakage (bubble test).

2.3.6.3.2. Test interpretation

The container shall not show any leakage.

2.3.6.3.3. Retesting

Retesting is permitted for the external leak test.

A second test shall be performed on two containers which have been produced successively to the first container within the same batch.

If the results of these tests are satisfactory, the first test shall be ignored. In the event where one or both of the retests fail to meet the requirements, the batch shall be rejected.

2.3.6.4. Permeation test

2.3.6.4.1. Test procedure

The test shall be performed at 40 °C on a container fuelled with commercial propane at 80 % of its water capacity.

The test shall be held during at least 8 weeks until the steady state permeation of the structure is observed during at least 500 hours.

The graph of mass change per number of days shall be recorded.

2.3.6.4.2. Test interpretation

The rate of mass loss shall be less than 0,15 g/hour.

2.3.6.4.3. Retesting

Retesting is permitted for the permeation test.

A second test shall be performed on two containers which have been produced successively to the first container within the same batch.

If the results of these tests are satisfactory, the first test shall be ignored. In the event where one or both of the retests fail to meet the requirements, the batch shall be rejected.

2.3.6.5. LPG cycling test

2.3.6.5.1. Test procedure

A container having successfully passed the permeation test shall be submitted to an ambient temperature pressure cycling test according to the requirements of paragraph 2.4.1. of this annex.

The container shall be sectioned and the liner / end boss interface shall be inspected.

2.3.6.5.2. Test interpretation

The container shall comply with the ambient temperature pressure cycling test requirements.

Inspection of the liner / end boss interface of the container shall not reveal any evidence of deterioration, such as fatigue cracking or electrostatic discharge.

2.3.6.5.3. Retesting

Retesting is permitted for the LPG cycling test.

A second test shall be performed on two containers which have been produced successively to the first container within the same batch.

If the results of these tests are satisfactory, the first test shall be ignored.

In the event where one or both of the retests fail to meet the requirements, the batch shall be rejected.

2.3.6.6. High temperature creep test

2.3.6.6.1. General

This test shall only be performed on all-composite containers with a resin matrix having a glass transition temperature (T_G) below the design temperature + 50 °C.

2.3.6.6.2. Test procedure

One finished container shall be tested as follows:

- (a) The container shall be pressurised to 3,000 kPa and held at a temperature defined according the table on the basis of the test period duration :

Table 3: Test temperature relating to the High temperature creep test duration

T (°C)	Exposure time (h)
100	200
95	350
90	600
85	1000
80	1800
75	3200
70	5900
65	11000
60	21000

- (b) The container shall be submitted to an external leak test.

2.3.6.6.3. Test interpretation

The maximum allowed volume increase is 5 %. The container shall meet the requirements of the external leak test as defined in paragraph 2.4.3. of this annex and the burst test as defined in paragraph 2.2. of this annex.

2.3.6.6.4. Retesting

Retesting is permitted for the high temperature creep test. A second test shall be performed on two containers which have been produced successively to the first container within the same batch.

If the results of these tests are satisfactory, the first test shall be ignored.

In the event where one or both of the retests fail to meet the requirements, the batch shall be rejected."

Paragraph 2.5., amend to read:

"2.5. **Examination on the outside of the weld for metal containers.**"

Paragraph 2.6.4., insert new item (e) to read:

"

(e) The pressure inside the container.

Metallic shielding shall be used"

Paragraph 2.6.7., amend to read:

"2.6.7. Acceptable results:

The LPG in metal containers shall vent through a pressure relief device, and no burst shall occur.

The LPG in all-composite containers may vent through a pressure relief device and/or may vent through the container wall or other surfaces, and no burst shall occur."

Insert paragraphs 2.7. to 2.11.3., to read:

"2.7. **Impact test**

2.7.1. General

At the choice of the manufacturer, all the impact tests may be carried out on one container or each may be carried out on a different container.

2.7.2. Test procedure

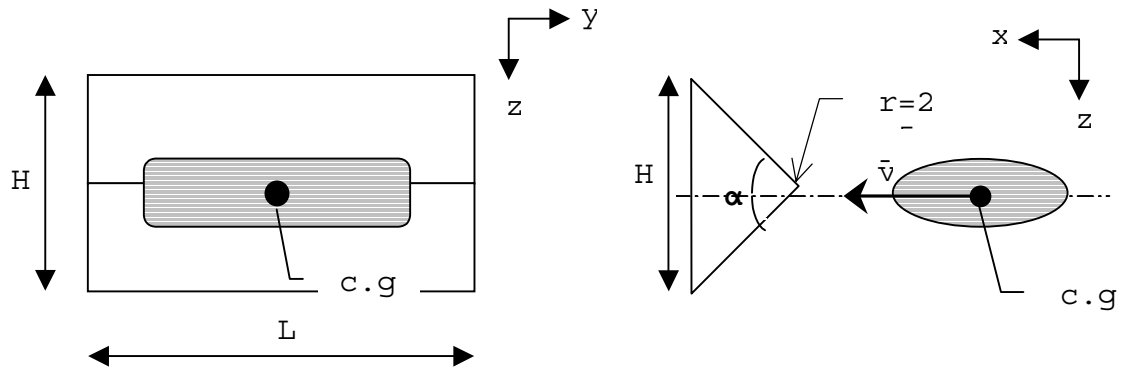
For this test, the fluid medium shall be water/glycol mixture or another liquid having a low freezing point which does not change the properties of the container material.

A container filled with the fluid medium to the weight that equals the filling with 80 % of LPG with a reference mass of 0.568 kg/l, is projected, parallel to the length axle (x-axis in Figure 1) of the vehicle in which it is intended to be fitted at a velocity, V of 50 km/h, against a solid wedge, fixed horizontally, perpendicular to the movement of the container.

The wedge shall be installed so that the centre of gravity (c.g.) of the container hits the centre of the wedge.

The wedge shall have an angle α of 90 degrees and the point of impact shall be rounded with a maximum radius of 3 mm. The length of the wedge L , shall be at least equal to the width of the container in respect to its movement during the test. The height H , of the container shall be at least 600 millimetres

Figure 1 - description of the impact test procedure:



Note: c.g. = center of gravity

In the case where a container can be installed in more than one position in the vehicle, each position shall be tested.

After this test, the container shall be submitted to an external leak test as defined in paragraph 2.4.3. of this annex.

2.7.3. Test interpretation

The container shall comply with the external leak test requirements as defined in paragraph 2.4.3. of this annex.

2.7.4. Retesting

Retesting is permitted for the impact test.

A second test shall be performed on two containers which have been produced successively to the first container within the same batch.

If the results of these tests are satisfactory, the first test shall be ignored.

In the event where one or both of the retests fail to meet the requirements, the batch shall be rejected.

2.8. Drop Test

2.8.1. Test procedure

One finished container shall be drop tested at ambient temperature without internal pressurization or attached valves. The surface onto which the containers are dropped shall be a smooth, horizontal concrete pad or flooring. The drop height (H_d) shall be 2 m.

The same empty container shall be dropped:

- in a horizontal position,
- vertically on each end,
- at an angle of 45°.

Following the drop test, the containers shall be submitted to an ambient temperature pressure cycling test according the requirements of paragraph 2.4.1. of this annex.

2.8.2. Test interpretation

The containers shall comply with the requirements of the ambient temperature pressure cycling test according the requirements of paragraph 2.4.1. of this annex.

2.8.3. Retesting

Retesting is permitted for the drop test.

A second test shall be performed on two containers which have been produced successively to the first container within the same batch.

If the results of these tests are satisfactory, the first test shall be ignored.

In the event where one or both of the retests fail to meet the requirements, the batch shall be rejected.

2.9. **Boss torque test**

2.9.1. Test procedure

The body of the container shall be restrained against rotation and a torque of 2 times the valve or PRD installation torque specified by the manufacturer shall be applied to each end boss of the container, first in the direction to tighten a threaded connection, then in the untightening direction, and finally again in the tightening direction.

The container shall then be subjected to an external leak test in accordance with the requirements shown in paragraph 2.4.3 of this annex.

2.9.2. Test interpretation

The container shall comply with the requirements of the external leak test as shown in paragraph 2.4.3. of this annex.

2.9.3. Retesting

Retesting is permitted for the boss torque test.

A second test shall be performed on two containers which have been produced successively to the first container within the same batch.

If the results of these tests are satisfactory, the first test shall be ignored.

In the event where one or both of the retests fail to meet the requirements, the batch shall be rejected.

2.10. **Acid environment test**

2.10.1. Test procedure

A finished container shall be exposed for 100 hours to a 30 % sulphuric acid solution (battery acid with specific gravity of 1.219) while pressurized to 3,000 kPa. During the test, a minimum of 20 % of the total area of the container has to be covered by the sulphuric acid solution.

Then, the container shall be submitted to a burst test as defined in paragraph 2.2. of this annex.

2.10.2. Test interpretation

The burst pressure measured shall be at least 85 % of the container burst pressure.

2.10.3. Retesting

Retesting is permitted for the acid environment test.

A second test shall be performed on two containers which have been produced successively to the first container within the same batch.

If the results of these tests are satisfactory, the first test shall be ignored.

In the event where one or both of the retests fail to meet the requirements, the batch shall be rejected.

2.11. **Ultra-violet (UV) test**

2.11.1. Test procedure

When the container is directly subjected to sunlight (also behind glass), UV-radiation might degrade polymeric materials. Therefore, the manufacturer has to prove the ability of the material to withstand the UV-radiation during his lifetime of 20 years.

- (a) If the outer layer has a mechanical (load carrying) function, the container shall be burst tested according to the requirements of paragraph 2.2 of this annex, after exposing to a representative UV-radiation;
- (b) If the outer layer has a protective function, the manufacturer has to prove that the coating remains integer within 20 years, in order to protect the under-laying structural layers from a representative UV-radiation.

2.11.2. Test interpretation

When the outer layer has a mechanical function, the container shall comply with the burst test requirements as defined in paragraph 2.2 of this annex.

2.11.3. Retesting

Retesting is permitted for the ultra-violet test.

A second test shall be performed on two containers which have been produced successively to the first container within the same batch.

If the results of these tests are satisfactory, the first test shall be ignored.

In the event where one or both of the retests fail to meet the requirements, the batch shall be rejected."

Insert new Annex 10 - Appendix 6, to read:

"Annex 10 - Appendix 6

MATERIAL TEST METHODS

1. Chemical resistance

Materials used in an all-composite container must be tested according to standard ISO 175 for 72 hours at room temperature. Approving the chemical resistance by adequate literature is also allowed.

Compatibility with the following media shall be checked:

- (a) brake fluid;
- (b) window cleaner;
- (c) cooling liquid;
- (d) non-leaded petrol;
- (e) solution of deionized water, sodium chloride (2.5 % by mass ± 0.1 %), (calcium chloride (2.5 % by mass ± 0.1 %) and sulphuric acid sufficient to achieve a solution of pH 4.0 ± 0.2 .

Test acceptance criteria:

- (a) Elongation:
The elongation of a thermoplastic material, after testing, must be at least 85 % of the initial elongation. The elongation of an elastomer, after testing, must be at least larger than 100 %.
- (b) For structural components (e.g., fibres):
The residual strength for a structural component after testing must be at least 80 % of the original tensile strength.
- (c) Non-structural components (e.g., coating):
There are no visual cracks allowed.

2. Composite structure

(a) Fibres embedded in a matrix

Tensile properties:	ASTM 3039	Fiber-resin composites
	ASTM D2343	Glass, Aramid (tens.prop.yarns glass)
	ASTM D4018.81	Carbon (tens.prop.continuous filament) with special remark for the matrix
Shear properties:	ASTM D2344	(Interlaminar shear strength of parallel fibre composite by short beam method)

(b) Dry fibres on an isotensoid shape

Tensile properties:	ASTM D4018.81	Carbon (continuous filament), other fibres.
---------------------	---------------	---

3. Protective coating

UV-radiation degrades polymeric material when directly subjected to the sunlight. Depending on the installation, the manufacturer has to proof a "safe life" for the coating.

4. Thermoplastic components

The Vicat softening temperature of a thermoplastic component must be above 70 °C. For structural components, the Vicat softening temperature must be at least 75 °C.

5. Thermoset components

The Vicat softening temperature of a thermo-set component must be above 70 °C.

6. Elastomeric components

The glass transition temperature (Tg) of an elastomer component must be lower than -40 °C. The glass transition temperature shall be tested according ISO 6721 "Plastics - Determination of dynamic mechanical properties". The Tg-onset is derived from the plotted diagram storage modulus versus temperature by determining the temperature, where the two tangents, which are representing the slopes of the diagram before and after the dramatic loss of stiffness, intersects."

* * *

B. JUSTIFICATION

Re. paras. 2.3.2. and 2.3.3.

Definitions of an all-composite container and container batch are added in paragraph 2.3. related to container definitions.

In the definition of a type of container in paragraph 2.4. the items (e) welding process and (f) heat treatment must only be applicable for steel containers.

Re. Annex 10

Corrections and supplementary paragraphs in Annex 10 concerning all-composite LPG containers: "Provisions regarding the approval of LPG containers".

Re. Annex 10, para. 1.1.

A new paragraph 1.1. is introduced to precise the scope of this annex.

Re. Annex 10, para. 1.2.4. (renumbered as 1.3.4.)

A reference to EN 288-3 is added in the renumbered paragraph 1.3.4.

Re. Annex 10, para. 2.

A new paragraph is added in the paragraph 2: "Tests" to make an overview of the tests to be performed on the different types of containers.

Re. Annex 10, para. 2.1.1.1.

To improve the reading of the text, current provisions of paragraph 2.1.1.1. describing the test methods to be applied for the tensile and bend test shall be deleted and its provisions moved to the specific paragraphs describing these tests: paragraphs 2.1.2.2. and 2.1.2.3.

Re. Annex 10, para. 2.1.2.2.1.2.

The target values to be achieved by the parent metal when performing the tensile test shall be changed.

Re. Annex 10, paras. 2.2.3.2. and 2.2.3.3.2.

Specific provisions applying to all-composite containers shall be added in the paragraph 2.2.3. defining the test acceptance conditions for the burst test.

Re. Annex 10, para. 2.6.4.

The pressure measurements during the bonfire test are introduced, which is already practice.

Re. Annex 10, para. 2.6.7.

The behaviour of a all composite is different in a bonfire test than a steel container. After a period of time during the bonfire test the container shall leak through the container wall. This behaviour is safe because during leakage of the container the pressure drops, the LPG is vented and the container shall not burst.

Re. Annex 10, paras. 2.7. to 2.11.3.

New paragraphs are added to introduce specific tests requirements to safeguard the good construction and design of all-composite containers:

Re. Annex 10, Appendix 6 (new)

A new appendix is added in the Annex 10 to introduce all the provisions regarding the materials requirements for all-composite containers:
