Informal Document No. 24
(42nd GRPE, 29-30 May 2001,
(agenda item 6.)

Proposal for a Supplement to UN/ECE Regulation 67- 01 (Equipment for LPG) with a view to introduce specific requirements for the type-approval of all-composite LPG containers.

Transmitted by the Expert from the Netherlands

Part 1

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

Insert new paragraph 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.

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

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.

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

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

Part 1, Annex 10 , *Provisions regarding the approval of LPG containers.*

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

A new paragraph 1.1 shall be introduced to precise the scope of this annex :

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."

Renumber paragraph 1.1. into 1.2.

Renumber paragraph 1.2. to 1.2.7. into 1.3. to 1.3.7.

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

Renumbered paragraph 1.3.4 , 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)."

Renumbered paragraph 1.3.5 , amend to read::

"1.3.5 The container manufacturer must obtain and provide :

(a) for metal containers : chemical cast analysis certificates,
(b) for all-composite containers : chemical resistance analysis certificates related to tests performed according 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."

Renumber paragraph 1.3 "Design temperatures and pressures" and paragraphs 1.3.1 to 1.3.2. , into paragraphs 1.4 to 1.4.2.

Renumber paragraphs 1.4. to 1.4.5. , into paragraphs 1.5. to 1.5.5.

Renumbered paragraph 1.5., amend to read :

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

Renumber paragraphs 1.5. into paragraphs 1.6.

Renumber paragraph 1.5.1. , into paragraph 1.6.1.1.

Insert a new paragraph 1.6.1, to read:

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

Renumber paragraph 1.5.1.1 and 1.5.1.2. , into paragraph 1.6.1.2. and 1.6.1.3.

Renumber paragraph 1.5.2. into paragraph 1.6.1.2.

Renumber paragraph 1.5.2.1 to 1.5.2.3. , into paragraph 1.6.1.2.1. to 1.6.1.2.3.

Renumber paragraph 1.5.3. and 1.5.4. , into 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."

Renumber paragraph 1.6. , into paragraph 1.7.

Paragraphs 1.6.1 and 1.6.1.1 shall be renumbered in respectively paragraphs 1.7.1 and 1.7.1.1.

Renumber paragraphs 1.6.1.2. , into 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."

Renumber paragraphs 1.6.2. to 1.6.2.4. , into 1.7.2 and 1.7.2.4.

Renumber paragraph 1.6.3., into 1.7.3.

Renumber paragraph 1.6.3.1., into 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."

Renumber paragraphs 1.6.3.2. to 1.6.3.6. , into 1.7.3.2 and 1.7.3.6.

Renumber paragraphs 1.6.4. and 1.6.4.1., into paragraphs 1.7.4 and 1.7.4.1.

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

Paragraph 2. , amend to read:

"2. **TESTS**

The tables 1 & 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°C ± 5°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 2.1.2.2
Bend test	1 per batch	2[1)]	See 2.1.2.3
Burst test		2	See 2.2
Hydraulic test	Each container	100 %	See 2.3
Bonfire test		1	See 2.6
Radiographic examination	1 per batch	100 %	See 2.6.1
Macroscopic examination	1 per batch	2[1)]	See 2.6.2
Inspection of welds	1 per batch	100 %	See 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 2.2
Hydraulic test	Each container	All containers	See 2.3.6.
Ambient temperature pressure cycling test	1 per 5 batches	3	See 2.4.1
High temperature pressure cycling test		1	See 2.4.2
External leak test		1	See 2.4.3
Permeation test		1	See 2.4.4
LPG cycling test		1	See 2.4.5
High temperature creep test		1	See 2.4.6
Bonfire test		1	See 2.6
Impact test		1	See 2.7
Drop test		1	See 2.8
Boss torque test		1	See 2.9
Acid environment test		1	See 2.10
Ultra-violet radiation test		1	See 2.11

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.

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.

Paragraph 2.1.2.2.1.1. , amend to read:

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

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

Paragraph 2.1.2.2.1.2. , amend to read:

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

Renumber paragraph 2.1.2.4. into 2.1.2.4.1, and insert new paragraph 2.1.2.4., to read:

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

A type-writing error shall be corrected in the paragraph 2.2.2 showing the provisions for the interpretation of the burst test. In addition specific criteria shall be added for the tests on all-composite containers :

Paragraph 2.2.2. to 2.2.2.1.3. , amend to read:

" 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."

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.

Insert in the text of paragraph 2.2.3.2. , the word "metal" before "container"

Insert in the beginning of the text of paragraph 2.2.3.3.2., the words: "For metal containers, ..."

Paragraph 2.2.3.3.2. amend to read:

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

New hydraulic tests are introduced in paragraph 2.3. to check the behaviour of all-composite containers :

Insert new paragraph 2.3.6 , 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 3000 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 10 000 cycles, the container shall not fail or leak.
After 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, 65ºC, and 95% or greater relative humidity.

c. Hydrostatically pressurize for 3600 cycles not exceeding 10 cycles per minute, between not more than 300 kPa and not less than 3000 kPa at 65ºC and 95% humidity;

Following the pressure cycling at high temperature, containers shall be submitted to the external leak test and then hydrostatically pressurised 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 3000 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 weight change per number of days shall be recorded.

2.3.6.4.2. **Test interpretation**

The rate of weight 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 the 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 3000 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 a 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 the paragraph 2.4.3 of this Annex and the burst test as defined in the 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."**

Introduce the pressure measurements during the bonfire test, which is already practice.

Paragraph 2.6.4. , insert new (e) in the list :

2.6.4. ……..
(e) The pressure inside the container.

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.

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."

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

New paragraph 2.7. and 2.8 , 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**

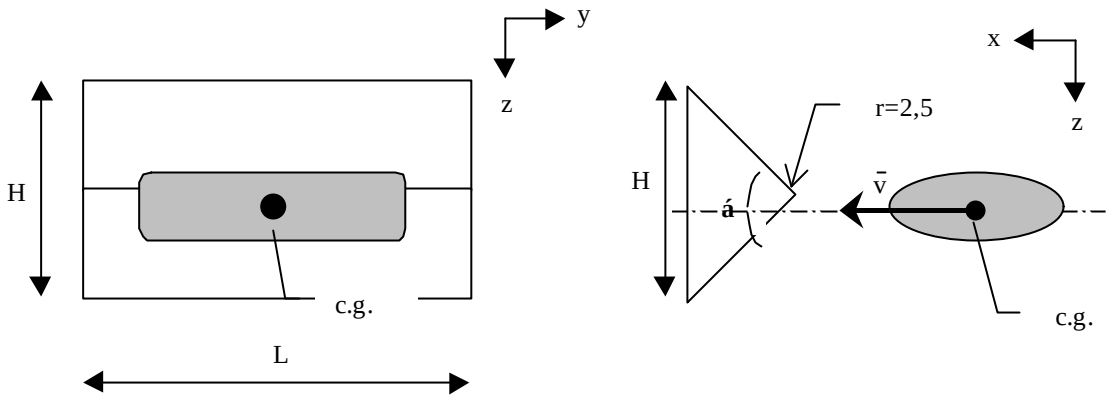
For this test, the fluid medium shall be water-glycol mixture or another liquid having a low freezing point which does not change the properties of the container material.

A container filled with the fluid medium to the weight that equals the filling with 80% of LPG with a reference mass of 0.568kg/l, is projected, parallel to the length axle (x-axes in Figure 1) of the vehicle in which it is intended to be fitted at a velocity, V of 50km/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 á 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 the 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 the 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 pressurisation or attached valves. The surface onto which the containers are dropped shall be a smooth, horizontal concrete pad or flooring.
The drop height (Hd) 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 the 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 the 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 the 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 the 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 pressurised to 3000 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 the 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 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.

- If the outer layer has a mechanical (load carrying) function, the container shall be burst tested according to the requirements of the paragraph 2.2 of this Annex, after exposing to a representative UV-radiation;

- 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 the 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."

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

New appendix add to Annex 10 , to read:

" Annex 10 – Appendix 6 –

Material Test Methods

1. Chemical resistance

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

Compatibility with the following mediums shall be checked:

- brake fluid,
- window cleaner,
- cooling liquid,
- gasoline (98/70/EEC),
- solution of deionized water, Sodium Chloride 2.5 % by weight ±0.1%, Calcium Chloride 2.5 % by weight ±0.1% and Sulphuric Acid sufficient to achieve a solution of pH 4.0 ± 0.2

Test acceptance criteria :

- 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%.

- 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.

- Non-structural components (e.g., coating):
  There are no visual cracks allowed.

2. Composite structure

- 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)

- 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 ºC. For structural components, the Vicat softening temperature must be at least 75 ºC.

5. Thermoset components

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

6. Elastomeric components

The glass transition temperature (Tg) of an elastomer component must be lower than -40ºC. 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.”
