Submitted by the expert from ISO (TC 58/SC 3)

Informal document GRSG-113-02
(113th GRSG, 10–13 October 2017,
Agenda item 6.(b))

Rationale for the proposed amendments to Regulation No. 110 (Specific components for CNG)

Submitted by the expert from ISO (TC 58/SC 3)

The text reproduced below was prepared by the expert from the International Organization for Standardization (ISO). The expert is the Convener of the Working Group (ISO TC 58/SC 3/WG 17) responsible for the ISO 11439 standard *High pressure cylinders for the on- board storage of natural gas as a fuel for automotive vehicles*. The proposed changes are for the purpose of harmonizing certain CNG cylinder requirements in Regulation No. 110 with certain requirements in the ISO 11439: 2013 standard.

The proposed changes are a follow-up to the discussion that started during the 106th session of the Working Party on General Safety Provisions (see report ECE/TRANS/WP.29/GRSG/85, para. 31). The modifications to the current text of UN Regulation No. 110 are marked by strikethroughs and in bold characters.

Justification

The justification for this proposal was previously presented in the document *ECE R110 Annex 3 & ISO 11439 "High pressure cylinders for the onboard storage of natural gas as a fuel for automotive vehicles"*, Informal document GRSG-106-29 (106th GRSG, 5-9 May 2014, agenda item 8). As a result of comments from Committee members, the proposed harmonization changes are limited to those items that either eliminate certain tests required in the ECE R110 from being performed, or are changes required to improve the safety of the cylinder designs. The rationale for these changes are provided in red italics under each proposed change.

Proposal - Alignment of certain items with ISO 11439:2013 - Proposed Revisions to:

E/ECE/324/Rev.2/Add.109/Rev.3
E/ECE/TRANS/505/Rev.2/Add.109/Rev.3

Regulation No. 110

Uniform provisions concerning the approval of:

I. Specific components of motor vehicles using compressed natural gas (CNG) and/or liquefied natural gas (LNG) in their propulsion system

II. Vehicles with regard to the installation of specific components of an approved type for the use of compressed natural gas (CNG) and/or liquefied natural gas (LNG) in their propulsion system

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2. References

ASTM D4814 Standard Specification for Automotive Spark-Ignition Engine Fuel

Standard added for Environmental test chemical to be specified in A.14.

~~ASTM G53-93 Standard Practice for Operating Light and Water – Exposure Apparatus (Fluorescent UVCondensation Type) for Exposure of nonmetallic Materials~~

ASTM G154-12a Standard Practice for Operating Fluorescent Light Apparatus for UV Exposure of Nonmetallic Materials

ASTM replacement for ASTM G53.

ISO 527~~-Pt 1-93 Plastics - Determination of Tensile Properties – Part I: General principles~~**-2, Plastics – Determination of tensile properties – Part 2: Test conditions for moulding and extrusion plastics**

Replacement reference for ISO 3628 (see below).

~~ISO 3628-78 Glass Reinforced Materials – Determination of Tensile Properties~~

ISO 3628 is for photography processing chemicals. The correct reference is ISO 527-2 Plastics -- Determination of tensile properties -- Part 2: Test conditions for moulding and extrusion plastics.

ISO ~~6982-84~~ **6892** Metallic Materials – Tensile Testing

Wrong designation was previously used (numbers reversed).

ISO~~/DIS~~ 7866~~-1992~~ Refillable ~~Transportable~~ ~~S~~**s**eamless ~~A~~**a**luminium ~~A~~**a**lloy **gas** ~~C~~**c**ylinders ~~for Worldwide Usage~~ – Design, **construction and testing** ~~Manufacture and Acceptance~~

Updated reference as is no longer a DIS.

~~ISO/DIS 9809 Transportable Seamless Steel Gas Cylinders Design, Construction and Testing – Part I: Quenched and Tempered Steel Cylinders with Tensile Strength < 1,100 MPa~~
ISO 9809-1, Gas cylinders — Refillable seamless steel gas cylinders — Design, construction and testing — Part 1: Quenched and tempered steel cylinders with tensile strength less than 1 100 MPa

Updated reference as is no longer a DIS.

6.3.6 Plastic liners

The tensile yield strength and ultimate elongation shall be determined in accordance with paragraph A.22. (Appendix A to this annex). Tests shall demonstrate the ductile properties of the plastic liner material at temperatures of -50 °C or lower by meeting the values specified by the manufacturer; the polymeric

material shall be compatible with the service conditions specified in paragraph 4. of this annex. In accordance with the method described in paragraph A.23. (Appendix A to this annex), the softening temperature shall be at least ~~90 °C, and the melting temperature at least~~ 100 °C.

The increased softening temperature requirement to 100°C is in consideration of the high temperatures generated in Type 4 cylinders during fast filling. The melting temperature was eliminated as the ISO 306 standard "Plastics -- Thermoplastic materials -- Determination of Vicat softening temperature (VST)", as required in A.23, does not determine the melting point.

6.12. Exterior environmental protection

The exterior of cylinders shall meet the requirements of the environmental test conditions of paragraph A.14. (Appendix A to this annex). Exterior protection may be provided by using any of the following:

(a) A surface finish giving adequate protection (e.g. metal sprayed on aluminium, anodizing); or

(b) The use of a suitable fibre and matrix material (e.g. carbon fibre in resin); or

(c) A protective coating (e.g. organic coating, paint) that shall meet the requirements of paragraph A.9. (Appendix A to this annex).

Any coatings applied to cylinders shall be such that the application process does not adversely affect the mechanical properties of the cylinder. The coating shall be designed to facilitate subsequent in service inspection and the manufacturer shall provide guidance on coating treatment during such inspection to ensure the continued integrity of the cylinder. ~~Manufacturers are advised that an environmental performance test that evaluates the suitability of coating systems is provided in the informative Appendix H to this annex.~~

The environmental test in Annex C - Appendix H has been moved into A.14 as a mandatory test. The mandatory use of the Appendix H test is primarily the result of in-service stress corrosion cracking failures of CNG cylinders reinforced with glass fibre composites.

The environmental test method in Appendix H has been performed by every manufacturer of composite-reinforced cylinders since the early 2000's, as it has been in the ISO 11439 and North American NGV2 standards since that time. This test has been in Appendix H since the Regulation was first published. As a result of the widespread use of the Environmental test as provided in Appendix H, there have not been any environmental stress corrosion cracking failures involving cylinder designs since multiple failures occurred in the 1990's. The proposed change to make the Environmental test in Appendix H mandatory under A.14 is for the purpose of enhancing safety based on in-service experience. A version of this test has been adopted by automotive OEMs into the UN GTR No. 13, the Global Technical Regulation on Hydrogen and Fuel Cell Powered Vehicles. As such, there is no need seen by the industry itself to provide further data related to the scope or test setups.

A.14. ~~Acid e~~**E**nvironment**al** test

~~On a finished cylinder the following test procedure should be applied:~~

~~(a) Exposing a 150 mm diameter area on the cylinder surface for 100 hours to a 30 per cent sulfuric acid solution (battery acid with a specific gravity of 1.219) while the cylinder is held at 26 MPa;~~

~~(b) The cylinder shall then be burst in accordance with the procedure defined in paragraph A.12. above and provide a burst pressure that exceeds 85 per cent of the minimum design burst pressure.~~

The Environmental test in Annex C – Appendix H (and modified below to harmonize with ISO 11439) is a far more comprehensive test of road environments compared to the Acid environment test that is currently in the Regulation. The Environmental test in Appendix H was developed by the automotive industry due to ruptures of glass reinforced composite cylinders in CNG service by stress corrosion cracking.

The Environmental test method as described below has been performed by every manufacturer of composite-reinforced cylinders since the early 2000's, as it has been in the ISO 11439 and North American NGV2 standards since that time. This test has been in Appendix H since the Regulation was first published. As a result of the widespread use of the Environmental test as provided in Appendix H, there have not been any environmental stress corrosion cracking failures involving cylinder designs since multiple failures occurred in the 1990's. The proposed change to make the Environmental test in Appendix H mandatory under A.14 is for the purpose of enhancing safety based on in-service experience. A version of this test has been adopted by automotive OEMs into the UN GTR No. 13, the Global Technical Regulation on Hydrogen and Fuel Cell Powered Vehicles. As such, there is no need seen by the industry itself to provide further data related to the scope or test setups.

~~H.1.~~ **A.14.1** Scope

~~The environmental test is intended to demonstrate that NGV cylinders can withstand exposure to the automotive underbody environment and occasional exposure to other fluids. This test was developed by the United States of America (USA) automotive industry in response to cylinder failures initiated by stress corrosion cracking of the composite wrap.~~ **This test is applicable to type CNG-2, CNG-3 and CNG-4 designs only.**

The preamble about the development of the test is not needed in a regulation. It is a test of composite reinforcement~~,~~ and, therefore, is not applicable to Type CNG-1 (all-metal) designs (it has been shown elsewhere that the chemicals and exposure times involved have no effect on steel or aluminum alloys).

~~H.2. Summary of test method~~

~~A cylinder is first preconditioned by a combination of pendulum and gravel impacts to simulate potential underbody conditions. The cylinder is then subjected to a sequence of immersion in simulated road salt/acid rain, exposure to other fluids, *and* pressure cycles and high and low temperature exposures. At the conclusion of the test sequence the cylinder will be hydraulically pressured to destruction. The remaining residual burst strength of the cylinder shall be not less than 85 per cent of the minimum design burst strength.~~

Unnecessary summary of a test.

~~H.3.~~ **A.14.2** Cylinder set-up and preparation

~~The cylinder shall be tested in a condition representative of installed geometry including coating (if applicable), brackets and gaskets, and pressure fittings using the same sealing configuration (i.e. O-rings)~~

~~as that used in service. Brackets may be painted or coated prior to installation in the immersion test if they are painted or coated prior to vehicle installation.~~

~~Cylinders will be tested horizontally and nominally divided along their horizontal centreline into "upper" and "lower" sections. The lower section of the cylinder will be alternatively immersed in road salt/acid rain environment and in heated or cooled air.~~

The immersion part of the test was eliminated in ISO 11439 as the concentrated exposures to the 5 chemicals would provide a more severe test condition.

The upper section **of the cylinder** will be divided into 5 distinct areas and marked for preconditioning and fluid exposure (see Figure ~~H~~**A.**1). The areas will be nominally 100 mm in diameter. The areas shall not overlap on the cylinder surface. While convenient for testing, the areas need not be oriented along a single line, but shall not overlap the immersed section of the cylinder.

Although preconditioning and fluid exposure is performed on the cylindrical section of the cylinder, all of the cylinder, including the domed sections, should be as resistant to the exposure environments as are the exposed areas.

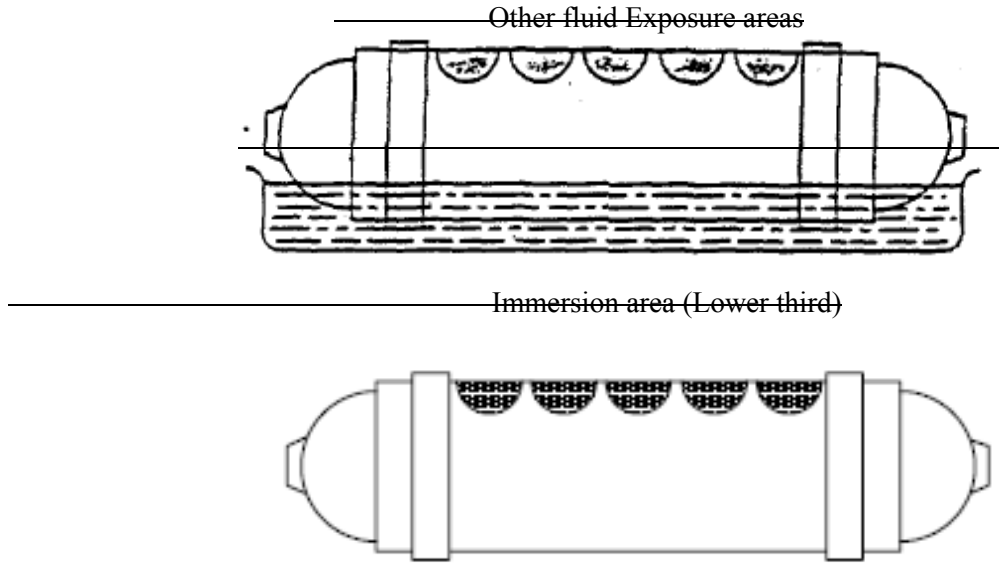


Figure ~~H~~ **A**.1 - Cylinder orientation and layout of exposure areas

~~H.4.~~ **A.14.3** ~~Preconditioning apparatus~~ **Pendulum impact preconditioning**

~~The following apparatus are needed for preconditioning the test cylinder by pendulum and gravel impact.~~

~~(a) Pendulum impact preconditioning~~

The impact body shall be of steel and have the shape of a pyramid with equilateral triangle faces and a square base, the summit and the edges being rounded to a radius of 3 mm. The centre of percussion of the pendulum shall coincide with the centre of gravity of the pyramid; its distance from the axis of rotation of the pendulum shall be 1 m. The total mass of the pendulum referred to its centre of percussion shall be 15 kg. The energy of the pendulum at the moment of impact shall be not less than 30 Nm and as close to that value as possible.

During pendulum impact, the cylinder shall be held in position by the end bosses or by the intended mounting brackets. **The cylinder shall be un-pressurized during preconditioning.**

Pressurization increases the resistance of the cylinder wall to impact damage. Thus the most susceptibility to damage is when the cylinder is un-pressurized during the pendulum impact.

~~(b) Gravel impact~~

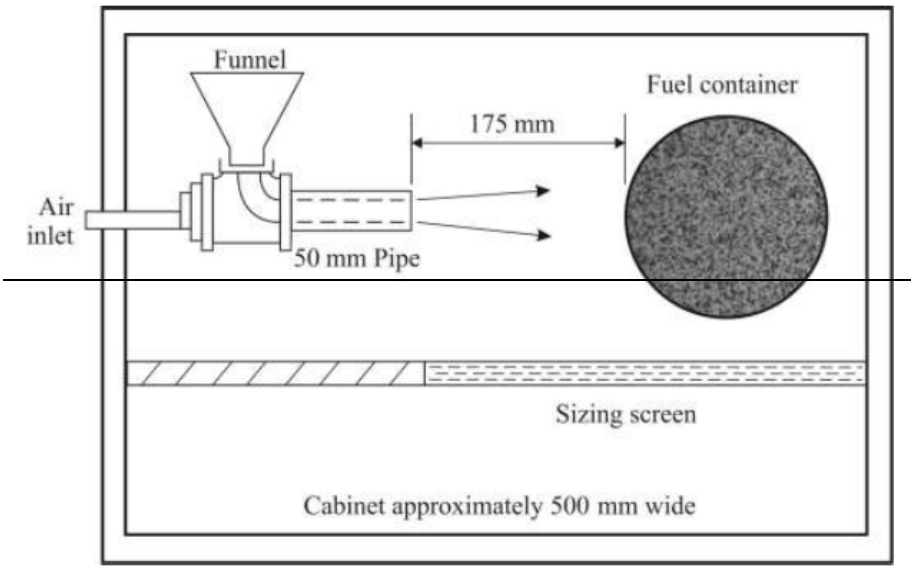
~~Machine constructed according to the design specifications shown in Figure H.2. This procedure for operation of the equipment shall follow that described in ASTM D3170, Standard Test Method for Chip Resistance of Coatings with the exception that the cylinder may be at ambient temperature during gravel impact;~~

~~(c) Gravel~~

~~Alluvial road gravel passing through a 16 mm space screen but retained on a 9.5 mm space screen. Each application is to consist of 550 ml of graded gravel (approximately 250 to 300 stones).~~

~~Figure H.2~~

~~Gravel impact test~~



It was found in testing that the gravel impact had no apparent effect on the integrity of cylinders, or any protective coating – the pendulum impact was a much more severe test of a coating and its ability to prevent chemicals from reaching the underlying composite reinforcement.

~~H.5.~~ **A.14.4** E**nvironmental fluids for e**xposure ~~environments~~

~~(a) Immersion environment~~

~~At the specified stage in the test sequence (Table 1) the cylinder will be oriented horizontally. with the lower third of the cylinder diameter immersed in a simulated acid rain/road salt water solution. The solution will consist of the following compounds:~~

~~Deionized water;~~

~~Sodium chloride: 2.5 per cent by weight ± 0.1 per cent;~~

~~Calcium chloride: 2.5 per cent by weight ± 0.1 per cent;~~

~~Sulphuric acid: Sufficient to achieve a solution pH of 4.0 ± 0.2;~~

~~Solution level and pH are to be adjusted prior to each test step which uses this liquid. The temperature of the bath shall be 21 ± 5 °C. During immersion, the unsubmerged section of the cylinder shall be in ambient air.~~

The use of an "immersion environment" in the Environmental test was discarded as the use of concentrated chemicals was considered more severe.

~~(b) Other fluid exposure~~

~~At the appropriate stage in test sequence (Table 1)~~ ~~e~~**E**ach marked area is to be exposed to one of five solutions for 30 minutes. The same environment shall be used for each location throughout the test. The solutions are:

Sulphuric acid: 19 per cent solution by volume in water;

Sodium hydroxide: 25 per cent solution by weight in water;

5% Methanol/**95%** gasoline: **gasoline concentration of M5 fuel meeting the requirements of ASTM D4814** ~~30/70 per cent concentrations~~;

There was a need to define "gasoline", thus ASTM D4814 was added.

Ammonium nitrate: 28 per cent by weight in water;

Windshield washer fluid **(50%by volume solution of methyl alcohol and water)**

There was a need to define "windshield washer fluid".

When exposed, the test sample will be oriented with the exposure area uppermost. A pad of glass wool ~~one layer thick~~ (approximately 0.5 mm **thick**~~)~~ and ~~trimmed to the appropriate dimensions~~ **between 90 and 100 mm in diameter shall** ~~is to~~ be placed on the exposure area. ~~Using a pipet, apply 5 ml of the test fluid to the exposure area~~ **Apply an amount of the test fluid to the glass wool sufficient to ensure that the pad is wetted evenly across its surface and through its thickness for the duration of the test, and that the concentration of the fluid is not changed significantly during the duration of the test.** ~~Remove the gauze pad after pressurization of the cylinder for 30 minutes.~~

There was a need to define how the chemicals were held onto the surface of the cylinder.

~~H.6.~~ **A.14.5** ~~Test conditions~~ **Pressure cycle and hold**

~~(a) Pressure cycle~~

~~As defined in the test sequence,~~ **The** cylinder shall be hydraulically pressure cycled between not less than 2 MPa and not more than 26 MPa **for a total of 3 000 cycles. The maximum pressurization rate shall be 2.75 MPa per second. After pressure cycling, the cylinder shall**

be pressurized to 26 MPa and held at that pressure a minimum of 24 hours and until the elapsed exposure time (pressure cycling and pressure hold) to the environmental fluids equals 48 hours. ~~The total cycle shall be not less than 66 seconds and will include a 60 second minimum hold at 26 MPa. The nominal cycle process will be:~~

~~Ramp up from ≤ 20 MPa to ≥ 26 MPa;~~

~~Hold at ≥ 26 MPa for 60 seconds minimum;~~

~~Ramp down from ≥ 26 MPa to ≤ 2 MPa;~~

~~Total minimum cycle time to be 66 seconds.~~

~~(b) Pressure during other fluid exposure~~

~~Following application of the other fluids, the cylinder shall be pressured to not less than 26 MPa for a minimum of 30 minutes;~~

~~(c) High and low temperature exposure~~

~~As defined in the test sequence, the entire cylinder shall be exposed to high or low temperature air in contact with external surface. The low temperature air shall be -40 °C or lower and the high temperature air shall be 82 °C ± 5 °C. For the low temperature exposure, the fluid temperature of type CNG-1 cylinders shall be monitored using a thermocouple installed within the cylinder to ensure it remains at -40 °C or lower.~~

~~H.7. Test procedure~~

~~(a) Preconditioning of the cylinder~~

~~Each of the five areas marked for other fluid exposure on the upper section of the cylinder shall be reconditioned by a single impact of the pendulum body summit at their geometric centre. Following impact, the five areas shall be further conditioned by a gravel impact application. The central section of the bottom portion of the cylinder that will be submerged shall be preconditioned by an impact of the pendulum body summit at three locations spaced approximately 150 mm apart.~~

~~Following impact, the same central section that was impacted shall be further conditioned by a gravel impact application. The cylinder shall be unpressured during preconditioning.~~

~~(b) Test sequence and cycles~~

~~The sequence of the environment exposure, pressure cycles, and temperature to be used are defined in Table 1.~~

~~The cylinder surface is not to be washed or wiped between stages.~~

~~H.8.~~ **A.14.6** Acceptable results

~~Following the above test sequence, t~~**T**he cylinder shall be hydraulically tested to destruction in accordance with the procedure in paragraph A.12. ~~of Appendix A to this annex.~~ The burst pressure of the cylinder shall be not less than 8**0**~~5~~ per cent of the minimum design burst pressure.

~~Table 1~~

~~Test conditions and sequence~~

Test steps	*Exposure environments*	*Number of pressure cycles*	*Temperature*
1	Other fluids	-	Ambient
2	Immersion	1,875	Ambient
3	Air	1,875	High
4	Other fluids	-	Ambient
5	Immersion	1,875	Ambient
6	Air	3,750	Low
7	Other fluids	-	Ambient
8	Immersion	1,875	Ambient
9	Air	1,875	High
10	Other fluids	-	Ambient
11	Immersion	1,875	Ambient

A.16. Penetration tests

A cylinder pressurised to 20 MPa ± 1 MPa with compressed gas shall be penetrated by an armour piercing bullet with a diameter of 7**,**62 mm or greater. The bullet shall completely penetrate at least one side wall of the cylinder. **For type CNG-1 designs, the projectile shall impact the side wall at 90°.** For type CNG-2, CNG-3 and CNG-4 designs, the projectile shall impact the side wall at an approximate angle of 45°. The cylinder shall reveal no evidence of fragmentation failure. Loss of small pieces of material, each not weighing more than 45 grams, shall not constitute failure of the test. The approximate size of entrance and exit openings and their locations shall be recorded.

Changed impact angle for Type CNG-1 designs to 90 degrees (perpendicular), as it was found that armour-piercing bullets may ricochet (not penetrate) when impacting steel cylinders at 45 degree angle.

A.22. Tensile properties of plastics

The tensile yield strength and ultimate elongation of plastic liner material shall be determined at -50 °C using ISO **527-2** ~~3628~~, and meet the requirements of paragraph 6.3.6. of Annex 3A.

ISO 3628 is for photography processing chemicals. The correct reference is ISO 527-2 Plastics -- Determination of tensile properties -- Part 2: Test conditions for moulding and extrusion plastics

A.23. ~~Melting~~ **Softening** temperature of plastics

Polymeric materials from finished liners shall be tested in accordance with the method described in ISO 306~~,~~**.** **The softening temperature shall be at least 100°C** ~~and meet the requirements of paragraph 6.3.6. of Annex 3A~~.

The ISO 306 standard "Plastics -- Thermoplastic materials -- Determination of Vicat softening temperature (VST)", does not determine the melting point, so this value has been eliminated. The softening temperature was increased to the former melting point value in consideration of the higher temperatures that can be obtained in Type 4 cylinder designs during fast filling.

~~Annex 3A - Appendix H~~
~~Environmental test~~

Delete Annex H in its entirety, as it is now inserted into A.14 (with modifications to harmonize with ISO 11439).