

**Economic Commission for Europe**

Inland Transport Committee

**Working Party on the Transport of Dangerous Goods**

**Joint Meeting of the RID Committee of Experts and the**

**Working Party on the Transport of Dangerous Goods**

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Item 5 (a) of the provisional agenda

**Proposals for amendments to RID/ADR/ADN:**

**pending issues**

**Periodic inspection and test of some transportable refillable  
LPG steel cylinders in RID/ADR**

**Transmitted by the European Liquefied Petroleum Gas Association  
(AEGPL)**

## **Annex 1**

### **Annex F of prEN 16728 LPG equipment and accessories - Transportable refillable LPG cylinders other than welded and brazed steel cylinders: periodic inspection.**

**(sent to Public Enquiry in May 2014)**

#### **Annex F**

**(normative)**

#### **Periodic inspection procedure for over-moulded cylinders**

##### **F.1 General**

This annex is applicable to over-moulded cylinders. An example of an over-moulded cylinder is given in Figure F.1.

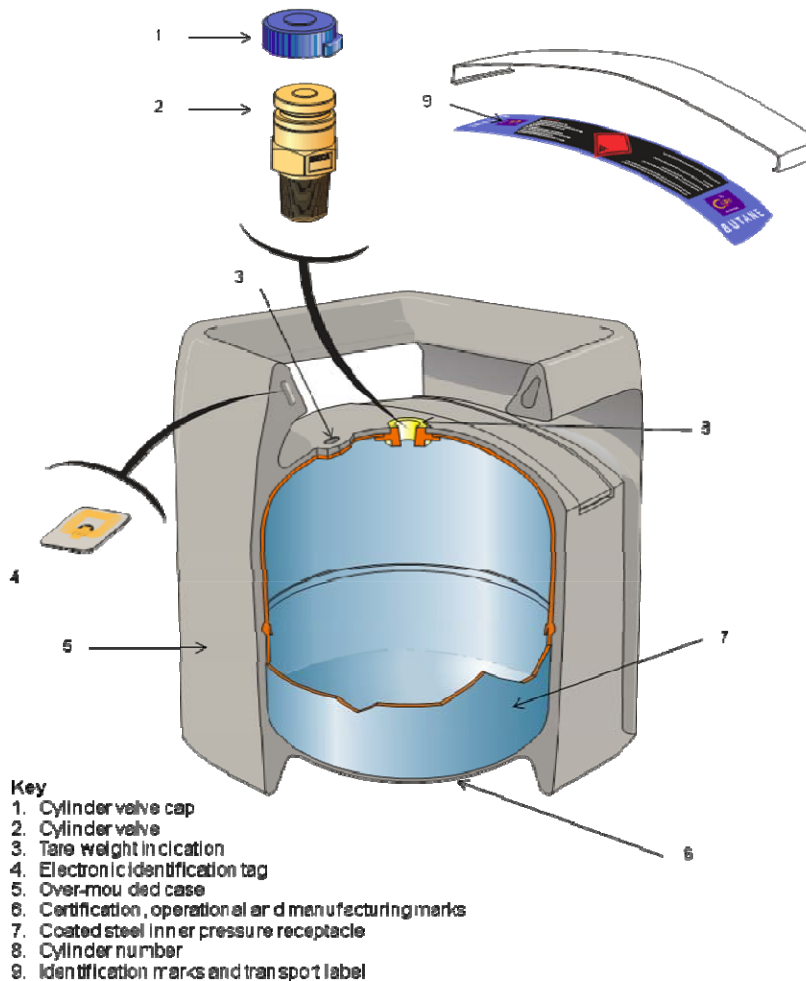


Figure F.1 —Example of an over-moulded cylinder

## F.2 Cylinders design and manufacturing requirements

### F.2.1 Over-moulded cylinder

The over-moulded cylinder shall be designed and manufactured in accordance with prEN 1442 or prEN 14140.

Note – RID/ADR defines an over-moulded cylinder as -

“Over-moulded cylinder, means a cylinder intended for the carriage of LPG of a water capacity not exceeding 13 litres made of a coated steel inner pressure receptacle with an over-moulded protective case made from cellular plastic which is non removable and bonded to the outer surface of the inner receptacle wall”.

An over-moulded batch is equivalent to a group defined in RID/ADR in 6.2.3.5.4.

### **F.2.2 Marking and recording requirements**

Each cylinder shall be fitted with an individual resilient identification electronic tag or any equivalent device linked to an electronic database as defined in prEN 1442 and prEN 14140.

This electronic database allows:

- To automatically withdraw a batch of cylinders to perform tests and / or manage the periodic inspection of test date;
- In case of an issue with a cylinder (detected at filling plant, at customer's, during periodic tests..), the electronic tag linked to the database allows cylinders from for the same batch to be automatically withdrawn to perform relevant tests and to assess if it is a batch issue or not. If necessary, the whole batch or sub-batch can be automatically withdrawn and disposed;
- To carry out the marking which indicates the successful completion of the periodic inspection.

Additional database recording requirements are listed in prEN 1442 and prEN 14140.

### **F.3 Inspection at filling**

Cylinders shall be individually checked before, during and after each filling in accordance with EN 1439.

### **F.4 Periodic destructive tests on batch sampling**

#### **F.4.1 Testing procedure**

Testing shall occur:

- after 3 years of service, and  
every 5 years after the first tests.

#### **F.4.2 Destructive tests**

The destructive test shall include at least the following:

- burst test in accordance with EN 1442 or EN 14140,
- peeling and corrosion test in accordance with EN ISO 4628-3, and
- adhesion tests of the polyurethane material. The number of cylinders to be tested is set by ISO 2859-1:1999 (single sampling for normal inspection, inspection level 1) applied to one thousandth of the annual production. The table 1 below gives the correspondence between the number of cylinders in the batch divided by one thousand and the sample size according to ISO 2859-1:1999. 5 adhesion test are done per cylinder. The minimum adhesion value is set to 0,5 N/mm<sup>2</sup>. If the result does not comply with this criteria for at least one test, a second sampling of the same size is made. If at least one cylinder of the second sampling does not comply with the minimum value of the adhesion criteria, tests are re-done considering sub-batches to define the sub-batch with a manufacturing defect.
- Adhesion test procedure is described in prEN14140:2013 in 7.3.7.3.2.

**Table 1 – Assessment of the sample size and compliance criteria**

<i>Number of OMC in the batch</i>	<i>Number of OMC considered to determine the sampling size</i>	<i>Sample size</i>
< 9 000	2 to 8	2
Between 9 000 and 16 000	9 to 15	2
Between 16 000 and 25 000	16 to 25	3
Between 25 000 and 51 000	25 to 50	5
Between 51 000 and 91 000	51 to 90	5
Between 91 000 and 150 000	91 to 150	8

**F.4.3 Rejection criteria and batch sampling**

Rejection criteria and sampling levels shall be in accordance with Table F.1.

Table F.1 — Batch sampling

Test interval (years)	Test type	Standard	Rejection criteria	Batch sampling level	Test results
After 3 years in service	Burst test	EN 1442	Burst pressure (*) <70 bar in propane service or 50 bar in butane service  Volumetric expansion (*) < 15 or 9 % (**)	$3\sqrt[3]{Q}$ or $Q/200$ whichever is lower,  and with a minimum of 20 per batch (Q)	If any test fails, repeat tests replacing Q with monthly production q of representative sub-batches
	Peeling and corrosion	EN ISO 462 8-3	Max corrosion grade :  Ri2	Q/1 000	
Every 5 years	Burst test	EN 1442	Burst pressure (*) <70 bar in propane service or 50 bar in butane service  Volumetric expansion (*) < 13, 12 or 9% (**)	$6\sqrt[3]{Q}$ or $Q/100$ whichever is lower,  and with a minimum of 40 per batch (Q)	
	Peeling and corrosion	EN ISO 462 8-3	Max corrosion grade :  Ri2	Q/1 000	
<p>Q Represents the total number of cylinders made by manufacturer in the same year.</p> <p>q Represents a continuous production batch.</p>					

(\*)

For each of the two groups of figures (burst pressure and volumetric expansion), the “right” unilateral statistical tolerance interval is calculated for a confidence level of 95% and a fraction of population equal to 99%. The calculation is made in accordance with the standard ISO 16269-6:2005 (Statistical interpretation of data – Part 6: Determination of statistical tolerance intervals) admitting, for each of the groups of figures, the normality of the population and that the variance is unknown.

Which means, considering:

- p, proportion of the population selected for the tolerance interval (99%),
- $1 - \alpha$ , confidence level (95%),
- n, sample size,
- $k_3(n; p; 1 - \alpha)$ , tabulated factor. This value can be read from the table D.4 given in Annex D,
- $\bar{x}$ , sample mean,

- s, sample standard deviation.

The tolerance interval with coverage  $p$  at confidence level  $1 - \alpha$  has lower limit  $x_L$  defined by this equation:

$$x_L = \bar{x} - k_3(n; p; 1 - \alpha) \cdot s$$

(\*\*)

For the cylinders manufactured according to Directive 84/527/EEC, the volumetric expansion cannot be lower than:

- 15% for the tests done 3 years after manufacturing
- 13% for the tests done 8 years after manufacturing
- 12% for the following tests.

For the cylinders manufactured according to Directive 1999/36/EC or Directive 2010/35/EU and according to EN1442, the volumetric expansion cannot be lower than 9%.

For the cylinders manufactured according to Directive 1999/36/EC or Directive 2010/35/EU according to EN14140, it is necessary to have at least 8 years of experience related to the cylinder type to determine the criteria for the volumetric expansion.

If the burst test or peeling test fails the tests are re-done considering sub-batches to define the sub-batch with a manufacturing defect or if it is an isolated defect. The production batch or sub-batch with defect has to be withdrawn immediately after detection using the electronic tag.

## F.5 Periodic inspection tests reports and records

Periodic inspection reports shall be made available to the competent authority upon request. At the end of the tests, the database is updated for the cylinders of the batch or sub-batch.

When the cylinders return to a filling plant, the cylinders from the relevant batch are:

- Marked provided the successful completion of the valve control or the valve replacement;
- Or, if the batch or sub-batch fails, withdrawn.

The test results have to be monitored and kept available by the owner for 30 years to the competent authority upon request.

## F.6 Lifetime

The design lifetime of the over-moulded cylinder is set at present to 30 years. However, this lifetime can then be extended every 5 years, as long as the tests undertaken at the periodic inspection demonstrate that the polyurethane adhesion to the inner receptacle has retained its properties.

The electronic tag linked to the database enables a batch of cylinders to be withdrawn when it has reached its lifetime.

## Annex 2

### **Extracts from pr EN 14140 : [2014], LPG equipment and accessories. Transportable refillable welded steel cylinders for LPG - Alternative design and construction**

*(Formal vote for summer 2014)*

#### **3 Terms, definitions and symbols**

##### 3.1.3 over-moulded cylinder

a cylinder intended for the carriage of LPG of a water capacity not exceeding 13 l made of a coated steel inner pressure receptacle with an over-moulded protective case made from cellular plastic which is non removable and bonded to the outer surface of the inner receptacle wall

##### 3.1.10 over-moulded casing

layer of over-moulded material which gives mechanical protection which, either cannot be removed without destroying it or is only removable with special tools or is bonded to the cylinder wall

Note 1 to entry: This definition can be applied to cylinders with over-moulded layers or with separate casings.

##### 3.1.14 production batch (over-moulded cylinders)

12 months production of cylinders from a single over-moulding company, using inner cylinders manufactured by one manufacturer

#### **5 Design**

##### 5.10 Over-moulded cylinders

5.10.1 The over-moulded case shall be polyurethane or a material which provides the same protection. The over-moulding shall be applied to a coated cylinder which meets the requirements of 7.3.7.1. Over-moulded case shall protect the coating and provide protection to the metal cylinder against corrosion as required in 7.3.7 and shall be designed to withstand the drop test requirements of 7.5.4.

5.10.2 The design of over-moulded cases shall facilitate manual handling, stowage and stacking without impeding access to the valve for the purposes of filling, emptying, maintenance and replacement.

5.10.3 The material used for the over-moulding shall be of the dense semi-rigid cellular plastic type, with an integral skin of mostly closed cells.

5.10.4 The design life of the cellular plastic over-moulding shall be not less than the life time of the design type of the over-moulded cylinder.

5.10.5 The over moulding shall be applied in a thermosetting casting process.



5.10.6 The over-moulding shall not cover the cylinder serial number and if the other permanent marks are covered by the over-moulding they shall be moulded into the over-moulding or on a plate entrained within the over-moulding. Where a separate plate is used it shall also be marked with the cylinder serial number.

5.10.7 Additional details on the design of over-moulded cylinders are given in Annex B.

NOTE Figure B.1 displays an example of an over-moulded cylinder.

5.10.8 The material used for the over-moulding shall be of the dense semi-rigid cellular plastic type, with an integral skin of mostly closed cells. 5.10.9 The design life of the cellular plastic over-moulding shall be not less than the life time of the design type of the over-moulded cylinder.

5.10.11 The over moulding shall be applied in a thermosetting casting process.

## **7 Tests and examinations**

### 7.3.7.3 Over-moulded cylinders

#### 7.3.7.3.1 Over-moulded protective case design

The over-moulding shall be tested for adhesion.

#### 7.3.7.3.2 Adhesion test procedure

##### 7.3.7.3.2.1 General

This additional test shall be carried out on one finished over-moulded cylinder.

##### 7.3.7.3.2.2 Preparation

The epoxy adhesive components used in this test procedure shall be stored according to the manufacturer instructions.

The adhesion test block shall be aluminium with a diameter of 20 mm and of suitable length for testing.

The over-moulded cylinder to be tested shall be stored at  $(23 \pm 2)$  °C for a minimum of 24 h before preparation and curing of the adhesive.

The test shall be carried out at  $(23 \pm 2)$  °C and at a relative humidity of  $(50 \pm 5)$  %.

The ambient conditions shall be recorded on the test report.

The test area shall be prepared by lightly sanding with an abrasive material.

Any paint on the over-moulded case shall be removed with an abrasive material.

The adhesion test block shall be glued to the over-moulded case using an epoxy type adhesive. The epoxy adhesive shall be allowed to cure for a minimum period of 48 h after application and as required by the manufacturer's instructions.

The over-moulded case around the adhesion test block shall be cut through to the steel surface of the cylinder.

##### 7.3.7.3.2.3 Breaking strength

The adhesion test block shall be placed in the pull-off adhesion tester, taking care to align the adhesion test block so that the tensile force is applied uniformly across the over-moulded cylinder wall.

A tensile stress, increasing at a rate not greater than 1 MPa/s, perpendicular to the plane of the substrate shall be applied.

7.3.7.3.2.4 Results

The acceptance values shall be in accordance with Table 8 (type test) and Table 10 (production test).

The following data shall be recorded:

- a) the breaking strength measured by the pull-off adhesion tester; and
- b) the type of fracture and the percentage of pulled-off area.

7.3.7.3.2.5 Test report

The test report shall contain at least the following information:

- a) all details necessary to identify the product(s) tested;
- b) the results of the test as per 7.3.7.3.2.4;
- c) the type of the cutting tool employed to cut around the adhesion test block; and
- d) the date of the test.

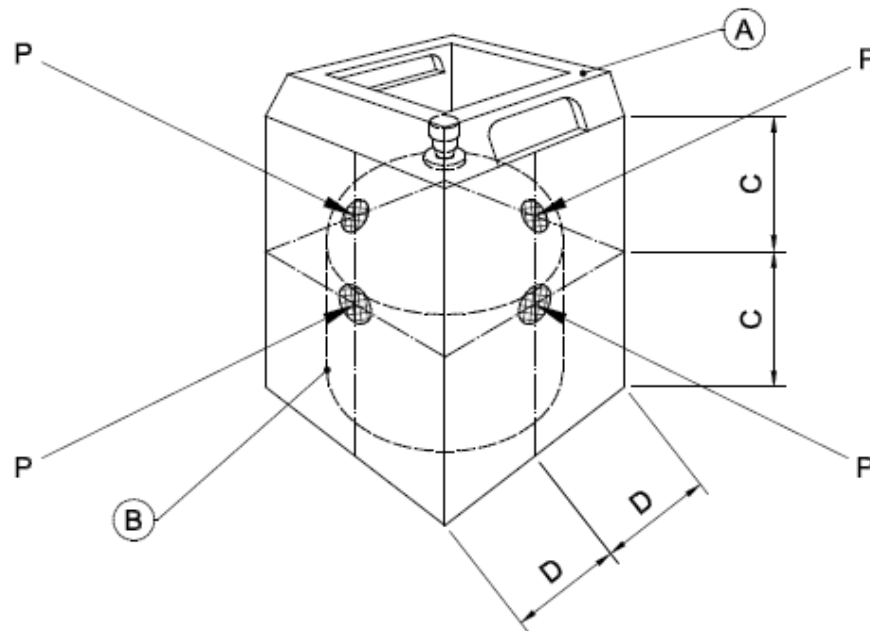
7.3.7.3.3 Requirements

The type test requirements and acceptance values are detailed in Table 8.

The production test requirements and acceptance values are detailed in Table 10.

**Table 8 — Resistance to external corrosion type test for over-moulded cylinders**

<i>Test type</i>	<i>Test details</i>	<i>Acceptance value</i>
Adhesion of protecting material on coating test	5 pull-off tests at mid height (on the sides) every 90° and on the bottom as illustrated in Figure 9. See procedure in 7.3.7.3.2.	Breaking strength > 2,5 MPa per test. Breaking shall occur into the protecting material layer or between the protecting material and the adhesive.



**Key**

- A over-moulded case
- B steel cylinder
- C equidistant dimension (4 places)
- D equidistant dimension (4 places)
- P location of adhesion test block for pull off test

**Figure 9 — Pull-off test locations**

7.3.7.3.4 Over-moulding material requirements

The over-moulding material shall meet the following requirements:

7.3.7.3.4.1 The applied cellular plastic shall be resistant to water absorption. The cellular plastic shall not absorb more than 0,05 grams of water per gram (5 % by mass) of cellular plastic coating material when the finished cylinder is totally immersed in a water bath for a period of 72 h (water temperature of 25 °C). This test shall be undertaken on the over-moulded cylinder before any paint is applied after the moulding operation.

7.3.7.3.4.2 In addition to the drop test requirements of 7.5.4.1, the cellular plastic material shall be subjected to a load resistance test. A sample 16 mm in diameter and 10 mm high shall be subjected to an equally distributed load of 70 N, placed on one of the flat faces of the sample. The sample shall be subjected to this load for a period of 72 h at a temperature of 50 °C. Following the completion of the test the load shall be removed and the sample shall return to within 1 mm of the original height within 5 min of the load being removed.

7.3.7.3.4.3 If the over-moulding is to be painted, the properties of the cellular plastic shall not be degraded by the paint.

## 9 Initial inspection and tests

### 9.10 Production adhesion test for over-moulded cylinders

Production testing for the adhesion of the over-moulded case shall be undertaken by the manufacturer that applies the over-moulded protective case.

The testing shall be carried out in accordance with 7.3.7.3.2 and the test acceptance values shall be in accordance with Table 10.

The production test for adhesion of protecting material on coating shall be carried out on one over-moulded cylinder every 1 000 cylinders produced.

In the event of the cylinder failing the test, the test shall be repeated on two additional cylinders.

In the event of one or more of the cylinders failing the retest the production batch shall be rejected.

**Table 10 — Resistance to external corrosion production test for over-moulded cylinders**

<i>Test type</i>	<i>Test details</i>	<i>Acceptance value</i>
Adhesion of protecting material on coating test	5 pull-off tests at mid height (on the sides) every 90° and on the bottom as illustrated in Figure 11 See procedure in 7.3.7.3.2	Breaking strength > 1 MPa per test. Breaking shall occur into the protecting material layer or between the protecting material and the adhesive.

### 9.11 Production water absorption test for over-moulded cylinders

9.11.1 The production testing for the water absorption of the over-moulding shall be undertaken by the manufacturer that applies the over-moulding.

9.11.2 The testing shall be carried out in accordance with 7.3.7.3.4.4 and the test acceptance values shall be in accordance with Table 11.

9.11.3 The production test shall be carried out on one over-moulded cylinder for every 1 000 cylinders produced.

9.11.4 In the event of the cylinder failing the test, the test shall be repeated on two additional cylinders.

In the event of one or more of these cylinders failing the retest the production batch shall be rejected.

**Table 11 — Production water absorption test for over-moulded cylinders**

<i>Test type</i>	<i>Test details</i>	<i>Acceptance value</i>
Water absorption	See 7.3.7.3.4.4	Shall not absorb more than 0,05 grams of water per gram (5 % by mass) of the cellular plastic coating material

## 10 Marking

10.1 Each cylinder shall be marked clearly and legibly with certification, manufacturing and operational information in accordance with EN 14894 and Annex A.

10.2 The over-moulding case shall not cover the cylinder serial number and if the other permanent marks are covered by the over-moulding they shall be moulded into the over-moulding or on a plate entrained within the over-moulding. Where a separate plate is used it shall also be marked with the cylinder serial number.

10.3 Each over-moulded cylinder shall be fitted with an individual resilient identification electronic tag or an equivalent device linked to an electronic database.

10.4 For over-moulded cylinders a technology database shall be established and maintained by the owner or operating company of the cylinders with shall allow:

- To trace the specific technical characteristics of the cylinders;
- To accurately operate each cylinder (including tare mass);
- To ensure the mandatory monitoring of the cylinders;
- To automatically withdraw a batch of cylinders to perform tests and/or to manage the periodic inspection test date;
- To carry out the marking which indicates the successful completion of the periodic inspection;
- To look for the history of all the events during the cylinders life.

## Annex 3

### **Annex B from prEN 1442 : LPG equipment and accessories - Transportable refillable welded steel cylinders for LPG - Design and construction**

**(2014 April draft document for the public enquiry)**

#### **Annex B**

**(Normative)**

#### **Over-moulded cylinders**

##### **B.1. Over-moulded cylinder case design**

The over-moulded protective case shall be polyurethane or a material which provided the same protection. The

over-moulded protective case shall be applied to a coated cylinder which meets the requirements of B.1.3. It

shall have mechanical properties and adequate adhesion to the coating to prevent water ingress between the

coating and the over-moulded case to guarantee the metallic cylinder properties during the life of the cylinder.

The over-moulding shall not cover the cylinder serial number and if the other permanent marks are covered by

the over-moulding they shall be moulded into the over-moulding or on a plate entrained within the over-moulding.

Where a separate plate is used it shall also be marked with the cylinder serial number.

##### **B.2 Tests and examinations**

###### **B.2.1 General**

Clause B.1.1 sets out the additional tests and examinations that shall be undertaken on over-moulded cylinders

for both ‘type approval’ and production testing. All tests and examinations shall be documented; production tests

shall be referenced to the ‘type approval’ documents.

Cylinders in accordance with this European Standard are subject to the conformity assessment system outlined

in clause 8, clause 9, clause B.1.6 and clause B.1.7 consisting of the testing and approval of the design type, the

recognition of quality assurance systems for the production and the initial inspection and testing of cylinders

manufactured according to the design type.

## B.2.2 Types of additional test and evaluation of results

**Table A.B 1 — Applicability of tests/examinations**

<i>Test/examination</i>	<i>Clause</i>	<i>Type tests</i>	<i>Production tests</i>		
			<b>Production batch tests</b>	<b>All cylinders</b>	
			<b>Specified in sub-clause</b>	<b>Specified in sub-clause</b>	<b>Specified in sub-clause</b>
Corrosion	B.1.3	X B.1.6	Z B.1.7	-	-
Over-moulded material properties	B.1.5	X B.1.6	-	-	-
Over-moulding adhesion	B.1.4	Z B.1.4	Z B.1.7.1	-	-
Water absorption	B.1.5.6	X B.1.6	Z B.1.7.2	-	-

X No option permitted – test shall be performed  
Z Carried out by the organisation which applies the over moulding

**B.3 Coated cylinder - resistance to external corrosion**

## B.3.1 Procedure

B.3.1.1 The test requirements and the relevant standards are detailed in Table B 2.

B.3.1.2 The tests specified in Table B 2 shall be carried out on standard test plates in accordance with the

specified standards except the adhesion tests, which shall be carried out on test pieces taken from one finished cylinder.

B.3.1.3 On completion of the climatic test, the salt spray test and the water immersion test, the test plates used shall then be subject to a cross-cut and a pull-off adhesion test.

## B.3.2 Requirements

The requirements are detailed in the column —Acceptance values‡ in Table B 2.



**Table A.B 2 — External corrosion tests for coated cylinders**

<i>Test type</i>	<i>Test details</i>	<i>Standard</i>	<i>Acceptance values</i>
Adhesion	Cross-cut test, with adhesive tape	ISO 2409:2013	Before climatic/salt spray/water test:- Table 1 – Classification □ 1 After climatic/salt spray/water test:- Table 1 – Classification □ □ 3
	Pull-off test	EN ISO 4624	Values listed in Table B.3
Climatic test	Resistance to cyclic corrosion conditions – wet (salt fog)/dry/humidity/UV light, using scribed panels	EN ISO 11997-2	Surface blistering density = 0 degree of rusting = Ri0 cracking to substrate, type (c) = 0 flaking to substrate, type (b) = 0 Adjacent to scratch extent of rusting □ □ 3mm blistering = size 4
	Resistance to humid atmospheres containing SO <sub>2</sub> , including environmental cabinet exposure. 0,2 l SO <sub>2</sub> , 28 cycles	EN ISO 3231:1997, utilising 9.3 b)	blistering = 0 degree of rusting = 0
Salt spray	720 h exposure on scratched surfaces. (2 scratches)	EN ISO 9227	Surface blistering density = 0 degree of rusting = Ri0 cracking to substrate, type (c) = 0 flaking to substrate, type (b) = 0 Adjacent to scratch : extent of rusting □ □ 3mm blistering = size 4
Water resistance	400 h exposure	EN ISO 2812-2	blistering = 0 no other signs of deterioration

**Table A.B 3 — Pull-off test values for Table B 2**

<i>Conditions test</i>	<i>Painting with hot zinc spraying</i>	<i>Painting applied directly on substrate</i>
New paint – Type A break (until metal substrate)	2 MPa	8 MPa
New paint – Type B break (cohesive break in paint layer)	Not required	6 MPa
After aging test – Type A break (until metal substrate)	1 MPa	4 MPa
After aging test – Type B break (cohesive break in paint layer)	Not required	3 MPa

**B.4 Over-moulding adhesion test procedure****B.4.1 General**

This test shall be carried out on one finished over-moulded cylinder.

**B.4.2 Preparation**

The epoxy adhesive components used in this test procedure shall be stored according to the manufacturer

instructions.

The adhesion test block shall be aluminium with a diameter of 20 mm and of suitable length for testing.

The over-moulded cylinder to be tested shall be stored at  $(23 \pm 2) ^\circ\text{C}$  for a minimum of 24 h before reparation and curing of the adhesive.

The test shall be carried out at  $(23 \pm 2) ^\circ\text{C}$  and at a relative humidity of  $(50 \pm 5) \%$ .

The ambient conditions shall be recorded on the test report.

The test area shall be prepared by lightly sanding with an abrasive material.

Any paint on the over-moulded protective case shall be removed with an abrasive material.

The adhesion test block shall be glued to the over-moulded protective case using an epoxy type adhesive. The epoxy adhesive shall be allowed to cure for a minimum period of 48 h after application and as required by the manufacturer's instructions.

The over-moulded protective case around the adhesion test block shall be cut through to the steel surface of the cylinder.

**B.4.3 Breaking strength**

The adhesion test block shall be placed in the pull-off adhesion tester, taking care to align the adhesion test

block so that the tensile force is applied uniformly across the over-moulded cylinder wall.

A tensile stress, increasing at a rate not greater than 1 MPa/s, perpendicular to the plane of the substrate shall be applied.

**B.4.4 Results**

The acceptance values shall be in accordance with Table B 4 (type test) and Table B 5 (production test).

The following data shall be recorded:

- a) the breaking strength measured by the pull-off adhesion tester; and
- b) the type of fracture and the percentage of pulled-off area.

#### B.4.5 Test report

The test report shall contain at least the following information:

- a) all details necessary to identify the product(s) tested;
- b) the results of the test as per B.1.4.4;
- c) the type of the cutting tool employed to cut around the adhesion test block; and
- d) the date of the test.

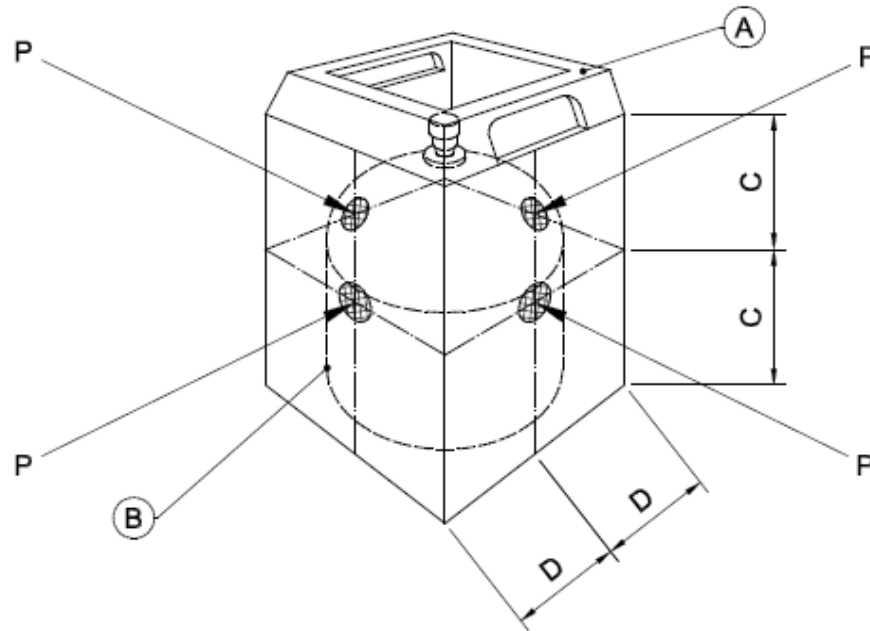
#### B.4.6 Requirements

The type test requirements and acceptance values are detailed in Table B 4.

The production test requirements and acceptance values are detailed in Table B 5.

**Table A.B 4 — Resistance to external corrosion type test for over-moulded cylinders**

<i>Test type</i>	<i>Test details</i>	<i>Acceptance value</i>
Adhesion of protecting material on coating test	5 pull-off tests at mid height (on the sides) every 90° and on the bottom as illustrated in figure B.1. See procedure in B.1.4.	Breaking strength > 2,5 MPa per test. Breaking shall occur into the protecting material layer or between the protecting material and the adhesive.

**Key**

- A Over-moulded protective case
- B Steel cylinder
- C Equidistant dimension (4 places)
- D Equidistant dimension (4 places)
- P Location of adhesion test block for pull off test

**Figure B 1 — Pull-off test locations**

### **B.5 Over-Moulding Material Requirements**

The over-moulding material shall meet the following requirements

B.5.1 The material used for the over-moulding shall be of the dense semi-rigid cellular plastic (foam) type,

with an integral skin of mostly closed cells.

B.5.2 The design life of the over-moulding material shall be a minimum of 10 years.

B.5.3 The over moulding shall be applied in a thermosetting casting process.

B.5.4 When the over-moulding has been completed the complete outer surface shall be painted with a

compatible paint to provide both water ingress and ultra violet protection.

B.5.5 The density of the over-moulding material shall be such that the finished cylinder can withstand all loads that it is expected to be exposed to during service and use.

B.5.6 The applied over-moulding material shall be resistant to water absorption and the foam shall not absorb more than 0.04 grams of water per gram (4% by mass) of coating

material when the finished cylinder is totally immersed in a water bath for a period of 168 hours (minimum water temperature 50C).

B.5.7 A sample of the over-moulding material shall be subjected to a load resistance test. A sample 16mm

diameter and 10mm high shall be subjected to an equally distributed load of 70N, placed on one of the flat faces of the sample. The sample shall be subjected to this load for a period of 72 hours, at a temperature of 50C. Following this test the load shall be removed and the sample shall return to within 1mm of the original height within 5 minutes of the load being removed.

### **B.6 Additional technical requirements for type approval**

B.6.1 For cylinders with over-moulding, the design and materials of the over-moulding shall be recorded on

the type approval certificate.

B.6.2 The resistance to external corrosion tests on the coated cylinder shall be carried out in accordance with B.4.

### **B.7 Additional initial inspection and tests**

B.1.7.1 Production adhesion test for over-moulded cylinders

Production testing for the adhesion of the over-moulding shall be undertaken by the manufacturer that applies the over-moulding.

The testing shall be carried out in accordance with B.1.4 and the test acceptance values shall be in accordance with Table B 5.

The production test for adhesion of protecting material on coating shall be carried out on one over-moulded

cylinder every 1 000 cylinders produced.

In the event of the cylinder failing the test, the test shall be repeated on two additional cylinders.

In the event of one or more of the cylinders failing the retest the production batch shall be rejected.

**Table A.B 5 — Resistance to external corrosion production test for over-moulded cylinders**

<i>Test type</i>	<i>Test details</i>	<i>Acceptance value</i>
Adhesion of protecting material on coating test	5 pull-off tests at mid height (on the sides) every 90° and on the bottom as illustrated in Figure B 1 See procedure in B 1.4	Breaking strength > 1 MPa per test. Breaking shall occur into the protecting material layer or between the protecting material and the adhesive.

B.1.7.2 Production water absorption test

B.1.7.2.1 The production testing for the water absorption of the over-moulding shall be undertaken by the

manufacturer that applies the over-moulding.

B.1.7.2.2 The testing shall be carried out in accordance with B.1.5.6 and the test acceptance values shall be

in accordance with Table B 6.

B.1.7.2.3 The production test shall be carried out on one over-moulded cylinder for every 1 000 cylinders

produced.

B.1.7.2.4 In the event of the cylinder failing the test, the test shall be repeated on two additional cylinders.

In the event of one or more of these cylinders failing the retest the production batch shall be rejected.

**Table B 6 — Production water absorption test**

<i>Test details</i>	<i>Acceptance value</i>	<i>Test details</i>
Water absorption	Shall not absorb more than 0,05 grams of water per gram (5 % by mass) of the cellular plastic coating material	See B.1.5

**B.1.8 Over moulded cylinder Identification system**

**B.1.8.1 Requirements**

Each over-moulded cylinder shall be fitted with an individual resilient identification electronic tag or an equivalent device linked to an electronic database.

B.1.8.2 For over-moulded cylinders a technology database shall be established and maintained by the owner or operating company of the cylinders with shall allow:

- To trace the specific technical characteristics of the cylinders;
- To accurately operate each cylinder (including tare mass);
- To ensure the mandatory monitoring of the cylinders;
- To automatically withdraw a batch of cylinders to perform tests and/or to manage the periodic inspection test date;
- To carry out the marking which indicates the successful completion of the periodic inspection;
- To look for the history of all the events during the cylinders life.

B.1.8.3 The database shall be operated by the cylinder owner or operating company and should be capable of recording the following:

- (a) the identification details of each cylinder;
- (b) the manufacturing information of each cylinder;
- (c) the status of each cylinder regarding periodic inspection;
- (d) the tare mass of each cylinder; and
- (e) information to allow identified cylinders or production batches to be segregated for any purposes (e.g. periodic inspection, sampling).

#### **B.1.9 Certification**

Each batch of cylinders shall be covered by a certificate stating that the cylinders meet the requirements of this European Standard and the approved design type specification in all respects.

The design and materials of the over-moulding shall be recorded on the type approval certificate.

If the over-moulding is painted it should be recorded in the technical specification and in the type approval certificate.

#### **B.10 Environmental Considerations**

The design of the over-moulded cylinder shall minimise the waste of materials.

Over-moulded protective casings manufactured from recyclable plastic materials shall display the appropriate recycling symbol.

The manufacturer should endeavour to minimise wastage of material by selecting appropriately sized materials related to the finished parts required for manufacture.

The process should be designed to minimise VOC emissions.

## **Annex 4**

### **Annex G from EN1439 :2008 (LPG equipment and accessories. Procedure for checking LPG cylinders before, during and after filling)**

#### **Annex G (informative)**

#### **Inspection procedure for a particular design of protected cylinder**

##### G.1 Application and cylinder description

This annex is applicable for protected cylinders such as polyurethane protected cylinders (see Figure G. 1) or other cylinders with the same protection.

All of the requirements of this standard shall apply except where specific reference is made to this annex. These requirements are listed below.

##### G.2 Cylinders suitable for filling

The age of the protected cylinder shall be less than the periodic inspection interval for this design of cylinder or the protected cylinder shall be from a batch that has been tested in accordance with EN 1440 (batch testing).

##### G.3 Cylinders for periodic inspection

The selection of protected cylinders for periodic inspection shall be done by sampling in accordance with EN 1440.

##### G.4 Cylinders requiring further assessment

Damaged protected cylinders shall be reassessed in accordance with G.5

##### G.5 Reassessment of cylinders

Rejection criteria for physical and material defects or heat damage on the cylinder shell are contained in Table G.1.



**Table G.1 — Defects of protected cylinders and rejection criteria before filling**

<i>Defect on</i>	<i>Description</i>	<i>Rejection limit</i>
Casing	Visible material gouge, cut or crack	Depth > 4 mm Length > 10 mm
Casing	Visible depression	All
Casing	Visible swelling	All
Feet and handles	Visible material gouge, cut or crack	Depth > 20 mm Length > 50 mm Missing feet
Handles	Broken handles	For each handle if more than one crack. Parts of handle missing.
Electronic tag	Illegible tag	All
Fire damage	Excessive general or localized heating of a cylinder usually indicated by: <ul style="list-style-type: none"> <li>– surface charring or burning;</li> <li>– distortion of the cylinder;</li> <li>– melting of metallic valve parts;</li> <li>– melting of any plastic components</li> </ul>	All
Depressed bung	Damage to the bung, which has altered the profile of the cylinder.	All, except a limited level of depression/alignment deviation, as agreed by the competent body.
Valves	Broken valves	All

## Annex 5

### Example on application of periodic inspection method

We consider a batch of 30 000 over-moulded cylinders (OMC) in propane service produced in 2008 as per EN1442 and tested 3 years after: in 2011. The batch is defined as cylinders with the steel inner pressure receptacle manufactured within a calendar year by single manufacturer and over-moulded by a single over-moulding company. The sub-batch is the monthly production of the OMC.

*Adhesion test:*

5 OMC has taken off to perform adhesion tests:

quantity of OMC batch = 30 000 / 1 000 = 30

=> letter C considered in ISO 2859-1 (Sampling procedures for inspection by attributes Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection) table 1 for a general control level I

=> size of 5 considered in ISO 2859-1 table 2.A

Then 5 sampling are done on each OMC.

If all the 25 values are above 0.5 N/mm<sup>2</sup>, the sample passes the test. In case of one value below 0.5N/mm<sup>2</sup>, a second sampling is done to determine if it is an isolated defect or not. In case a second value is below 0.5N/mm<sup>2</sup>, additional samplings are done for each sub-batch to determine the sub-batch(es) with the defect. The sub-batch(es) are immediately withdrawn.

*Burst test*

94 OMC are taken off to perform burst test:

$3 \cdot (29\,836)^{1/3} = 93.2 < 30\,000 / 200 = 150$

The 94 OMC are burst and the burst pressure and the volumetric expansion are recorded below.

For the burst pressure, the tolerance interval with coverage p (99%) at confidence level 1 -  $\alpha$  (95%) has lower limit Lip defined by this equation:

$$Lip = \bar{x} - k_3(n; p; 1-\alpha) \cdot s$$

$\bar{x}$  (mean) and s (standard deviation) are calculated:

$$\bar{x} = 129.77 \text{ bar}$$

$$S = 2.61$$

$k_3(n; p; 1-\alpha)$ , is given by ISO 16269-6 table D.4 given in Annex D for p=0.99:

n=94 and  $k_3$  values are only given for n=90 and n=100 but as  $k_3$  is a linear function of n,  $k_3$  can be calculated

$$k_3 = 2.7$$

$$Lip = 122.74 \text{ bar}$$

Which means that with confidence level of 0.95 (95%), at least 0.99 (99%) of the cylinders in the batch will have a burst pressure above 122.74 bar.

For the volumetric expansion, the tolerance interval with coverage  $p$  (99%) at confidence level  $1 - \alpha$  (95%) has lower limit  $Li(ex)$  defined by this equation:

$$Li(p) = \bar{x} - k_3(n; p; 1 - \alpha) \cdot s$$

$\bar{x}$  and  $s$  are calculated:

$$\bar{x} = 20.041$$

$$S = 1.75$$

$k_3$  is the same than above:  $k_3 = 2.7$

$$Li(Ex) = 15.31\%$$

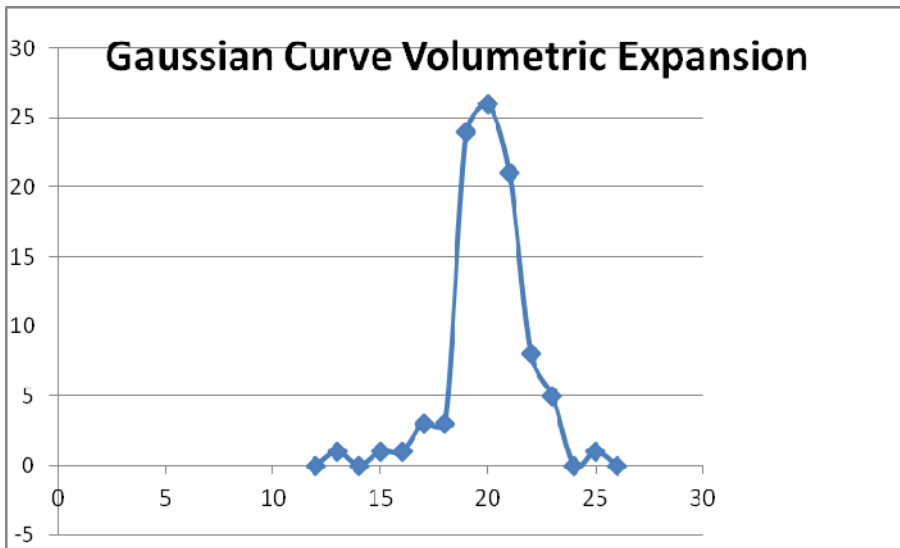
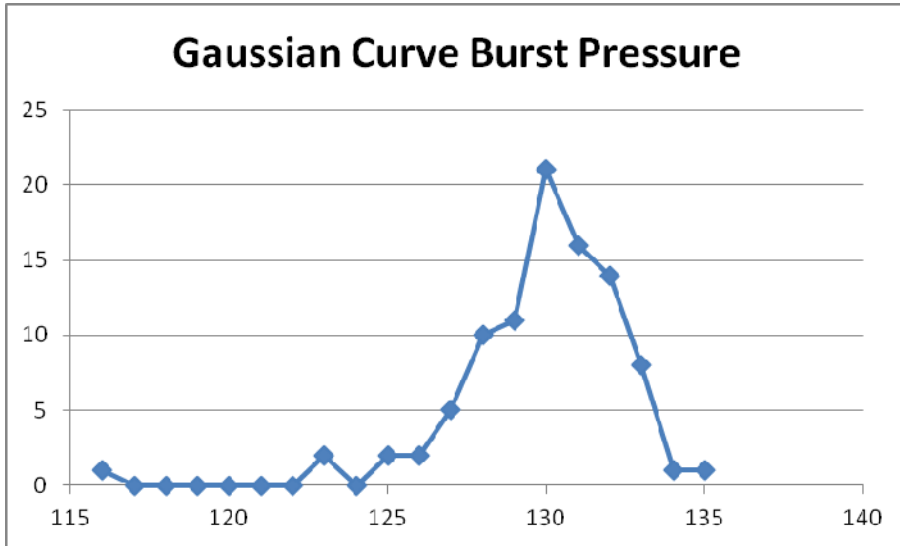
Which means that with confidence level of 0.95 (95%), at least 0.99 (99%) of the cylinders in the batch will have a volumetric expansion above 15.31%.

These values are above the criteria of 70 bar (criteria for propane service) and 9% (criteria for EN1442 design) so the sample passes the burst test. In case the one of the criteria would not have met, the same methodology is applied for each sub-batch to determine the sub-batch(es) with defect and withdraw it.

OMC number	Burst pressure, bar	Volumetric expansion, %	OMC number	Burst pressure, bar	Volumetric expansion, %	OMC number	Burst pressure, bar	Volumetric expansion, %	OMC number	Burst pressure, bar	Volumetric expansion, %
1	132,8	25,10	26	125,6	17,05	51	116,4	12,50	76	132,8	21,79
2	129,0	21,88	27	127,3	19,38	52	130,5	17,90	77	127,4	18,75
3	130,0	22,75	28	131,0	19,38	53	127,5	18,68	78	128,4	20,31
4	130,5	21,01	29	128,5	21,88	54	130,5	21,01	79	129,7	18,68
5	130,3	21,01	30	127,0	18,82	55	129,7	19,46	80	132,9	21,01
6	130,9	21,01	31	130,9	21,09	56	128,1	19,46	81	130,4	19,53
7	131,1	21,01	32	134,7	20,16	57	132,2	19,53	82	131,4	20,23
8	128,8	20,23	33	125,0	18,82	58	131,8	19,53	83	132,5	22,66
9	130,1	20,93	34	127,9	20,47	59	129,7	19,38	84	128,6	19,46
10	133,0	22,66	35	129,0	18,90	60	130,2	21,09	85	131,3	21,09
11	130,3	22,57	36	126,9	20,39	61	131,4	20,31	86	132,4	21,09
12	129,5	21,01	37	127,3	20,31	62	130,5	19,46	87	129,8	19,53
13	129,5	21,79	38	128,3	19,53	63	129,7	19,46	88	131,7	21,96
14	132,1	21,71	39	127,8	20,31	64	129,6	21,09	89	129,8	18,68
15	128,9	20,31	40	129,2	20,31	65	129,6	19,53	90	133,4	20,23
16	129,8	21,09	41	128,2	20,31	66	131,0	18,68	91	131,5	19,53
17	124,6	17,25	42	123,1	15,56	67	129,3	20,31	92	132,4	20,23
18	125,8	17,12	43	130,4	17,90	68	129,2	18,91	93	131,8	21,01
19	131,9	18,60	44	128,2	18,68	69	129,2	19,46	94	127,6	17,97
20	134,2	20,93	45	131,3	19,46	70	132,3	18,75			
21	130,9	19,38	46	129,5	19,53	71	131,6	21,01			
22	129,8	21,01	47	129,1	20,23	72	123,4	14,84			
23	129,7	20,93	48	133,0	23,35	73	131,1	21,01			
24	131,6	18,60	49	130,5	21,79	74	132,5	21,79			
25	131,5	21,01	50	127,7	20,23	75	131,7	21,79			

We can check in the data above that there is one value below 122.74 bar and two values below 15.31%: OMC n°51 and OMC n°72. So approximately 99% (93 / 94) of the sampling population is indeed above the calculated values of 122.74 bar and 15.31%.

We can also check that the distribution is normal by drawing the Gaussian curves.



*Peeling tests*

The number of cylinders to be peeled off is 30 (=30 000 / 1 000).

These cylinders are taken from the burst cylinders sampling.

The aspect of the outer wall of steel inner receptacle is compared with the picture below to determine if the degree of corrosion is above Re1. In case no corrosion is detected or in case the degree of rusting is equal to Re1, the sampling passes the test. In case one cylinder has a degree of rusting above Re1, the same methodology is applied for each sub-batch to determine the sub-batch(es) with defect and withdraw it.



*Figure 2 of ISO 4628-3 (Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 3: Assessment of degree of rusting) Degree of rusting Re1*

**In case these 3 tests are passed, the whole batch is considered to pass the periodic inspection.**

## Annex 6

### **Document presented in 2004, during ESOPE symposium, on specific periodic inspection procedure for protected over-moulded cylinders**

*ESOPE is a symposium done every 3 years in France to bring together experts and actors in fixed and transportable pressure equipment “community”, to meet up and discuss all the developments in their field.*

TRANSPORTABLE REFILLABLE WELDED STEEL CYLINDER “protected over-moulded cylinder” FOR LIQUID PETROLEUM GAS (LPG) – PERIODIC INSPECTION AND TEST BY INDIVIDUAL CONTROLS AT FILLING, BATCH SAMPLING AND DESTRUCTIVE TESTS

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### **Summary**

This method has been developed like an alternative to individual hydraulic pressure test for periodic inspection.

It is meant for reduced capacity welded steel cylinders totally covered with a protective plastic case in polyurethane (PU) acting as a protection against impacts and corrosion. It has been used for the last 4 years for “the Cube” cylinder by Butagaz.

Members having taken part in developing this step-by-step progressing method are:

- Ministry of Industry (gas and pressurized equipment dept),
- Ecole des Mines in Douai (Polymer and Composites dept),
- Butagaz: a LPG operating company in charge of distribution, filling and cylinders maintenance.

It has been developed on the following basis: preliminary tests protocol showing amongst other properties, the link between PU adhesion on the inner receptacle and the non-appearance of corrosion the pressure receptacle wall during the cylinder’s life.

Periodic inspection of such cylinders is composed of:

- Individual inspection and controls before, during and after filling,
- Periodic destructive tests by batch sampling.

### **Introduction**

The BUTAGAZ Company, since 1997 markets a LPG cylinder called “The Cube” ranging from 5 to 6 kg (butane-propane).

First of all, it is a pressure steel device which manufacturing meets transportable pressure equipment directives 84/527/CE or 1999/36/CE.

This pressure receptacle is coated with a polyurethane case which ensures two primary functions: resistance to outside attacks (drops, impacts, corrosion) and marketing (meeting customer's needs).

Prior to commercialisation tests have been conducted during 3 years between 1995 and 1997 in the Shell and Butagaz R&D laboratories leading to the design of the cylinder and the approval to operate the "Cube". These tests have shown amongst other things the Cube's properties in its resistance to impacts and corrosion.

Partly based on those tests, the Cube periodic inspection method by sampling has been developed step by step taking in account experience feedback between Butagaz and the Ministry of Industry (DGAP, DRIRE) with the support of the Ecole des Mines de Douai, particularly the Polymer and Composite department. A preliminary test phase has been led in order to define periodic inspection method and to precise and determine content of the tests by sampling as well as acceptance criteria. Checking of the good conservation of the PU case adhesion properties was the main item of this communication.

Until now, the first four years of production (year  $n$ ): 1997, 1998, 1999 and 2000 destructive tests by sampling have been led in year  $n + 3$ . Destructive tests in year  $n + 3$  have also been led on production 2001 ; in 2005 destructive tests on year  $N + 3 + 5$  will be led on production 1997, with double bursting tests.

## Description of the cube

The Cube is made of a steel vessel covered with an anti-corrosion coating, the whole unit being protected by a non-removable polyurethane (PU) foam. The steel part of the cylinder guarantees leakproofness and a good pressure in cylinder. In addition, the PU foam coating closely glued on the steel vessel must ensure two functions: protection of vessel against outside attacks (corrosion, drops, impacts, fire resistance, chemical resistance) and marketing (esthetical appearance, stability, prehension, comfort...)

The design of this pressure vessel is the result of research for the best material for the functions required at the best cost. This approach has led to choosing hybridization between steel and polymer material. It offers many possibilities of conception as already proven by the design of composite vessels fitted with a steel liner reinforced with an organic matrix composite external layer. By analogy with those vessels, the Cube could be considered here as a transportable composite appliance which structural integrity is ensured by the liner realised by the steel vessel.

## Periodic inspection methodology

By definition, the PU layer masks the external surface of the steel vessel and does not allow giving information on corrosion state through direct observation. An original and indirect method has been proposed and accepted. It is based on adhesion test results from PU foam pulling and also on checking the merit of the relation "**good adhesion of PU foam on the inner receptacle = non corrosion**".

The general structure of the Cube is ensured by the steel liner in association with the PU coating acting as a protection against environmental and mechanical aggressions of any type and allows it to maintain its mechanical properties in service. The periodical inspection method must take into account the synergy of material. Many destructive tests have been proposed to determine the Cube ability to safely fulfil its functions.

**Method Purpose and Development: characterization of interface PU / cylinder**

The difficulty of controls lies in controlling whether there is or not corrosion on the interface steel/PU since the PU coating is not removable. Corrosion developing at the surface of steel vessel would highlight presence of humidity through deteriorated PU foam. Different types of ageing like mechanical or physicochemical have been applied to the Cubes in order to induce failure in the PU layer barrier and to alter either adhesion properties on vessel or even its real mechanical performances. Definition of those tests relies on NF EN 12245 standard relative to fully wrapped composite cylinders (transportable gas cylinders).

In addition, in order to establish a link between PU adhesion and presence of corrosion, different tests have been carried out like PU foam adhesion test on vessels with corrosion and also on vessels coated, before foaming, with the product used to remove the over-moulded cylinder from the mould.

Main justification for choosing adhesion test relies on the fact that the PU foam has very good natural adhesion properties. Therefore, testing its adhesion on steel is like testing one of its main characteristic from its chemical nature and not manufacturing conditions. It keeps the reproducibility of adhesion measures and reliability of interpretations resulting from it.

**Preliminary adhesion measure tests of PU foam on vessel****Accelerated ageing with cycling (“VA+C”) according to EN 1442**

The tests have been realised on 3 Cubes which have been stored approximately 2 years under external atmospheric conditions (in Sologne, non sheltered).

The 3 Cubes are tested under the following conditions:

- Salt spray > 200 hours,
- Climatic room: 5 cycles (rain, -20°C, hygrometry 0-100%, UV) on 5 days,
- Cycling tests 2-30-2 bars (12 000 cycles at 20°)

Assessing cylinders and adhesion measures have led to:

- No visible cracks on Cube surface,
- No burst has occurred along the interface steel/PU, burst values indicating an average 4,4 N/mm<sup>2</sup>. Adhesion steel/PU is at least 3,6 N/mm<sup>2</sup>.

**Cycling to extreme temperatures (“CTE”) according to EN 12245**

Cycling tests to extreme temperatures according to NF EN 12245 are normally meant for transportable gas cylinders, fully wrapped in composite material. Here, the Cube is not seen as a steel vessel coated with polymer but as a polymer vessel with steel liner. These tests are meant to thermo mechanically test the polymer protection of the Cube.

3 Cubes, stored approximately 2 years outside, have been tested under the following conditions :

- 5000 cycles 2-20 bars at 50°C
- 5000 cycles 2-20 bars at -20°C
- 30 cycles 2-30 bars at 20°C

Results are as follows :



- No visible cracks on Cube surface
- No burst has occurred along the interface steel/PU, burst values indicating an average 4,4 N/mm<sup>2</sup>. Adhesion steel/PU is at least 4,4 N/mm<sup>2</sup>.

#### **Accelerated ageing including cycling (“VA+C+CTE”) according to EN 1442 and EN 12245**

This test is particularly severe for the vessel since it accumulates the two previous tests. Three Cubes stored roughly two years in external conditions have been tested in the conditions previously quoted.

Results are as follows :

- No visible cracks on Cube surface
- No burst has occurred along the interface steel/PU, burst values indicating an average 4 N/mm<sup>2</sup>. Adhesion steel/PU is at least 4 N/mm<sup>2</sup>.

A slight weakening of mechanical properties of pulled PU foam can be seen which shows the high interest of adhesion test used as a method of PU foam ageing close following.

#### **Vessels with corrosion (“xxx H Corrosion”)**

3 exposed steel vessels (without protective coating against corrosion) have been packed in a salt spray room during respectively 50, 120 and 200 hours before being sent for PU foaming. A fourth vessel without has also been foamed, as a reference.

The adhesion measured values are presented in chart 1

	<i>REFERENCE</i>	<i>50 HOUR of salt spray</i>	<i>120 HOUR of salt spray</i>	<i>200 HOUR of salt spray</i>
<b>TYPE OF RUPTURE</b>	Cohesive in the PU foam	Interface steel with corrosion/PU foam	Interface steel with corrosion /PU foam	Interface steel with corrosion /PU foam
<b>ADHESION VALUE (N/mm<sup>2</sup>)</b>	4,4	0,5	0,2	0

**Chart 1: adhesion value and burst types**

These measures clearly show that presence of corrosion strongly affects characteristics of PU adhesion on the exposed steel vessel. This drop of properties is compared in the following test to the one possibly appearing after accidental contamination of steel by an anti-grip agent.

#### **Vessels coated, before foaming, with the product used to remove the over-moulded cylinder from the mould (“agent démoulant”)**

Foaming the Cube requires coating the PU foam injection mould with a product. If it accidentally slips on steel before foaming, adhesion steel/PU would be affected or even inexistent. In such a case, there could be a gap of air, with humidity slowly progressing, given the time, through PU foam which would in the end contaminate vessel’s surface however coated with protection against corrosion.

Two vessels from current production, protected with a paint to protect against corrosion are half coated with the product, then foamed.

Measured adhesion values are for:

- The halves of Cubes non coated with release product are an average of 4,2 N/mm<sup>2</sup>. All bursts have been observed at foam heart. The PU/steel adhesion is at least 4,2 N/mm<sup>2</sup>.
- The halves of Cubes coated with release product are an average of 2,8 N/mm<sup>2</sup>. All bursts have been observed along interface steel/PU where the release product has been put, without bifurcation in foam. The PU/steel cohesion of foam is superior to 2,8 N/mm<sup>2</sup>.

The 2,8 N/mm<sup>2</sup> value obtained represents two thirds of nominal characteristic, it is anyway quite superior to adhesion values measured on Cubes with corrosion.

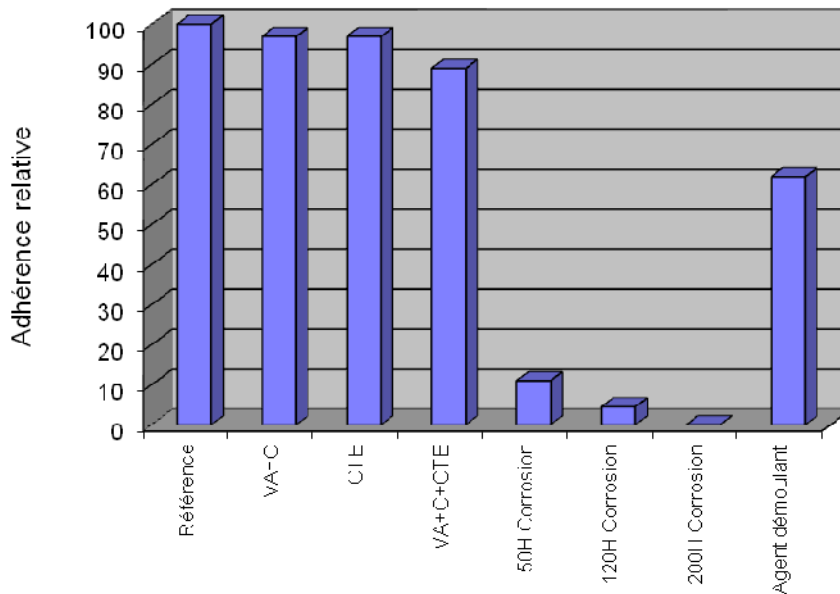


Figure 2 : Adhesion tests results chart

All adhesion measures results are presented in figure 2. This summary shows that only vessel with corrosion can lead to very weak adhesion values (inferior to 10% of nominal value). In comparison, Thermo mechanical ageing in severe environment and polluting products introduced along interface steel/PU has only led to minor adhesion weakening.

#### Periodical destructive test by proposed batch sampling

On a preliminary work basis, and in order to take into account presence of both material of different nature participating to the Cube structural integrity, it has been suggested to periodically sample Cubes for the following destructive tests :

- Burst under hydraulic pressure according to EN 1442 standard
- Peeling and corrosion according to ISO 4628/3 standard
- Adhesion according to EN 24264 standard
- Hydraulic burst test

In order to check the steel vessel is still able to stand the pressure, the hydraulic burst test is carried out according to prescription of EN 1442 standard.

- Peeling test

In order to check there is no corrosion along the weld, to ensure PU case remains indivisible from the welded vessel and to guaranty its protection function, destructive peeling tests will be carried out according to ISO 4628/3.

- Description of adhesion test by pulling

First of all the test consists in covering the 4 sides and both bottoms of the Cube with steel pull-off blocks glued on the PU. PU surface preparation is needed in order to guaranty optimal adhesion. Blocks are placed in traction through an adhesion dynamometer.

Traction adhesion tests done on the Cubes before ageing indicate that adhesion steel/PU is at least 4,5 N/mm<sup>2</sup>.

### **Experience feedback on destructive tests**

Since this procedure has been set up, experience feedback on destructive tests on a 5 year production after n+3 confirms the interest of the periodical inspection method including destructive tests by batch sampling among which PU/steel adhesion.

### **Electronic identification and database management system**

Each “Le Cube” cylinder is equipped with an electronic indestructible identification tag relating it to a database.

Maintenance and exploitation of this data base are both ensured by Butagaz

The system guaranties for each cylinder, among others :

- identification
- manufacturing data
- periodical inspection status
- tare value

And allows then segregation before filling of any cylinder or batch of cylinders whatever the reason (example : periodical inspection tests sampling, periodical inspection failure, complementary sampling, ...)

Moreover, it allows periodical inspection status marking for each cylinder.

### **Individual control before, during and after filling**

The Cube cylinder is under a specialised organisation control, responsible for its distribution, its filling and its supervision when in service but also its maintenance: Butagaz.

Before, during and after filling, each cylinder is individually checked according to EN 1439. Additional specific criteria indicated below do consider physical defaults but also specifications of PU foam.

- External visual inspection: any cylinder that does not comply with the criteria is isolated before filling for treatment or disposal.
- When periodical status control is not known before filling, the cylinder is isolated for complementary investigation, treatment or disposal.
- Any cylinder isolated for treatment or disposal is examined by competent staff who will decide whether it can be filled, repaired or disposed according to EN 12816.

## Conclusions

### On the method

The main target for those adhesion tests by pulling PU coating is qualification of adhesion level between vessel and PU coat, but it also allows following PU mechanical performances on pulling in the long run.

When burst occurs on interface PU/steel, adhesion between those two materials is directly quantified. A burst in the middle of PU foam means cohesion forces of the foam are inferior to adhesion forces of steel/PU.

In the second case the mechanical characteristic of coating when being pulled is measured which allows by comparison with nominal values a follow up of PU foam's ageing.

A procedure for each test has been developed in order to practice them in optimal conditions i.e. ensuring maximum reproducibility.

It has been proved that good adhesion of PU foam on vessel means the vessel has no corrosion. A minimum adhesion criteria has been defined in order to evaluate PU capacity to ensure his protective function against corrosion in the long run.

On the other hand grip test by pulling PU coating led with all precaution described in this procedure allows to set up a close follow up of PU foam ageing state.

### Requirements and limits of these methods

The periodic inspection method by sampling developed for the Cube vessel could be widened to other cylinder's periodical control problems or on an even wider scale, to transportable pressure device provided all following prescriptions are compliant:

- Large scale production through automatic procedures
- Reduced unit capacity
- Non corrosive content
- Individual visual check at filling
- Automatic control of periodical state control
- Automatic individual segregation before filling
- Computerized and automatized registration of each event of the cylinder's life (its initial test, first filling... until its disposal).

For "the Cube", Butagaz has the responsibility for exploiting its cylinders: distribution, filling, periodical inspection, maintenance, disposal. The process for "providing the Cube cylinder for filling" is certified in accordance with ISO 9001-2000.

The experience feedback after a 5 year experience successfully tested, confirms for the Cube validity of the periodical inspection method by associating controls and unit tests when filling with destructive periodical tests on batch samples.

### Regulatory framework

This method is now applicable on Cubes manufactured according to the pre-existing French relementation : January 18<sup>th</sup> 1943 modified. It has been confirmed by the decision DM-T/P n°32327 dated 9<sup>th</sup> December 2002.

According to experience feedback, evolutions of the method can be proposed to French administration.

Similar provisions are not today for transportable pressure equipments in international and European regulation texts: DESPT, RID/ADR, U.N. model regulation.

Adjustments to those texts must then be proposed for a similar periodical inspection method to be applicable to Cubes marked  $\pi$ , some of them also being exploited in Belgium and Portugal.

As a first approach, work has been carried within CEN TC 286 (equipment and accessories for LPG) to propose and introduce periodical inspection method, as it is carried on the Cube with review of standards relative to welded steel gas cylinders :

- EN 1439 : Before, during and after filling controls,
- EN 1440 : Periodical controls.

A similar approach will have to be taken within ISO TC 58 (gas cylinders).

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