# **Economic Commission for Europe**

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# Working Party on the Transport of Dangerous Goods

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# 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 1440 (WI 00286156), LPG equipment and accessories - Transportable refillable LPG cylinders other than welded and brazed steel cylinders: periodic inspection -**December 2013 draft** 

# 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tests..), the electronic tag linked to the database allows cylinders from for the same batch to be automatically withdrawn to perform relevant tests and to assess if it is a batch issue or not. If necessary, the whole batch or sub-batch can be automatically withdrawn and disposed;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 subbatches 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
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Number of OMC in the batch	Number of OMC considered to determine the sampling size	Sample size
< 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		5
	25 to 50	
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.

Test	Test type	Standard	Rejection	Batch sampling level	Test results		
interval	• •		criteria				
(years)							
After 3 yers 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 (*)	$6\sqrt[3]{Q}$ or $Q/100$ whichever is lower, and with a minimum of 40 per batch (Q)			
	Peeling and corrosion	EN ISO 462 8-3	< 13, 12 or 9% (**) Max corrosion grade : Ri2	Q/1 000			
Q Represents the total number of cylinders made by manufacturer in the same year.   q Represents a continuous production batch.							

Table F.1 — Batch sampling

(\*)

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,
- k3 (n; p; 1- $\alpha$ ), tabulated factor. This value can be read from the table D.4 given in Annex D,
- $\overline{\mathbf{x}}$ , 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:

$$x_{L} = x - k 3 (n; p; 1 - \alpha)$$
. 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.

# 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 2859-1table 2.A

Then 5 sampling are done on each OMC.

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

Bust test

94 OMC are taken off to perform burst test:

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

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 =  $\bar{x} - k 3 (n; p; 1-\alpha)$ . s

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

 $\bar{x} = 129.77$  bar

S = 2.61

k3 (n; p;  $1-\alpha$ ), is given by ISO 16269-6 table D.4 given in Annex D for p=0.99:

n=94 and k3 values are only given for n=90 and n=100 but as k3 is a linear function of n, k3 can be calculated

k3 = 2.7

Lip = 122.74 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:

Lip =  $\overline{x} - k 3$  (n; p;  $1-\alpha$ ). s  $\overline{x}$  and s are calculated:

 $\bar{x} = 20.041$ 

S = 1.75

k3 is the same than above: k3 = 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 compare 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 a 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 inappearance — 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.