RECOMMENDATIONS FOR FILLING OF CYLINDERS AND BUNDLES WITH CARBON DIOXIDE

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Amendments from 83/08

Section	Change
	Editorial to align style with EIGA Style Manual
All	Major re-write and update to reflect current practices

NOTE Technical changes from the previous edition are underlined

1 Introduction

Although very few carbon dioxide cylinder incidents are reported by EIGA Members, approximately 30% of these incidents are the result of either:

- internal corrosion, due to ingress of water or other liquids into carbon dioxide cylinders or bundles; or
- overfilling and subsequent failure of carbon dioxide cylinders.

The number of failures represents a fractional percentage compared to the overall number of cylinders in use.

This publication aims to give recommendations and an overview of the gas industry's current practices to prevent and detect internal corrosion and to avoid overfilling carbon dioxide cylinders and bundles. Filling of carbon dioxide cylinders and bundles differ from other industrial gases in that they are filled with liquefied carbon dioxide rather than gas, as a result they are filled by weight rather than pressure.

2 Scope and purpose

2.1 Scope

This publication applies to the filling of gas cylinders and bundles containing carbon dioxide as a single product. The recommendations given in this publication do not replace national regulations, where they exist. This publication does not cover the entire filling process.

This publication does not address the additional requirements resulting from the implementation of Good Manufacturing Practices for medical or for food gases, see EIGA Doc 125, *Guide to the Supply of Gases for Use in Foods*, EIGA Doc 99 Part 1, *Good Manufacturing Practice Guide Part I for Medical Gases* and EIGA Doc 99 Part 2, *Good Manufacturing Practice Guide Part II for Medical Gases: Basic Requirements for Active Substances Used as Starting Materials* [1, 2, 3].[1]

Other EIGA publications relating to carbon dioxide filled in cylinders and bundles include, EIGA Doc 62, *Methods to Avoid and Detect Internal Gas Cylinder Corrosion*, EIGA Doc 67, *Carbon dioxide cylinders at users' premises* and EIGA Doc 95, *Avoidance of Failure of CO and of CO/CO2 Mixtures Cylinders* [4, 5, 6].

2.2 Purpose

To provide specific guidelines for the safe filling of carbon dioxide in either single cylinders or bundles. It is intended to be followed by the industrial gases industry to eliminate incidents involving both cylinders and bundles containing carbon dioxide.

3 Definitions

For the purpose of this publication, the following definitions apply.

3.1 Publication terminology

3.1.1 Shall

Indicates that the procedure is mandatory. It is used wherever the criterion for conformance to specific recommendations allows no deviation.

[1] References are shown by bracketed numbers and are listed in order of appearance in the reference section.

3.1.2 Should

Indicates that a procedure is recommended.

3.1.3 May

Indicate that the procedure is optional.

3.1.4 Will

Is used only to indicate the future, not a degree of requirement.

3.1.5 Can

Indicates a possibility or ability.

3.2 Technical definitions

3.2.1 Blowdown

A process of emptying cylinder contents by opening the cylinder valve and venting down to the set pressure of the RPV or emptying the cylinder to atmospheric pressure in the absence of an RPV. This process does not involve evacuating the cylinder below atmospheric pressure by vacuum.

3.2.2 Bundles

Assembly of cylinders that are fastened together and which are interconnected by a manifold and carried as a unit. The total water capacity shall not exceed 3000 litres.

3.2.3 Corrosion

Deterioration of the cylinder material by an electro-chemical reaction, for carbon dioxide cylinders typically when in contact with carbon dioxide and water.

3.2.4 Empty weight of the cylinder

Mass of the cylinder including all permanent attachments (for example neckring, footring), but excluding the mass of valve, valve cap or valve guard and any coating.

3.2.5 Total weight

Tare weight of the cylinder plus the maximum permissible filling weight.

3.2.6 Filling ratio

Ratio of the mass of gas to the mass of water at 15 °C that would fill completely a cylinder fitted ready for use.

3.2.7 Maximum permissible filling weight

Maximum mass of gas in kg which is allowed in a filled cylinder, product of the minimum guaranteed water capacity of the cylinder and the filling ratio of the gas. For bundles this is the product of the total cylinder water capacity of bundle and the filling ratio.

3.2.8 Pressure

In this publication bar shall indicate gauge pressure unless otherwise noted i.e., (bar, abs) for absolute pressure and (bar, dif) for differential pressure.

3.2.9 Overfilling

When the total weight marked on the cylinder (tare weight and maximum permissible filling weight) is exceeded.

3.2.10 Tare weight – cylinder

Weight of the cylinder when empty, including accessories fitted, including valves, guards and coatings etc. as presented for filling.

3.2.11 Tare weight – bundles

Weight of a bundle includes the items in 3.2.9 and the support frame and manifold piping.

3.2.12 Top filling

A process where a cylinder containing residual positive pressure is filled without blowdown of the contents.

4 Sources of moisture contamination in carbon dioxide cylinders and bundles

Several sources of possible moisture contamination can be found during the life of a carbon dioxide cylinder, including from manufacture, use and maintenance.

4.1 Water from manufacturer's or gas companies hydraulic testing procedure

As part of a cylinder's acceptance procedure, a mandatory hydraulic test is performed. It is essential that subsequent emptying and drying of the cylinder is undertaken such that there is no free moisture left in the cylinder. Once achieved, it is essential that this internal condition is maintained, see 5.2.

4.2 Water from product and prefill treatment

Liquid carbon dioxide used can contain some water, a minimum dew point shall be established to prevent gross moisture contamination (see 5.2.1).

Some prefill operations can also introduce moisture into cylinders, for example if water-ring vacuum pumps are used without adequate precautions to prevent water carry over.

4.3 Water backflow during use

Water or liquid backflow into cylinders can occur whenever the cylinder is connected to a system that is at a higher pressure than the cylinder. Many carbon dioxide uses are found in the beverage or food industry, so that backflow of beer and soft drink syrups or other liquids is a constant risk. For more detailed information see EIGA Doc 62 and EIGA Doc 67 [4, 5].

The risk of moisture contamination is greatest if the cylinder contents and pressure is emptied.

4.4 Customer misuse

Water or liquid backflow into cylinders can occur as result of customer misuse, for example the removal of a residual pressure valve (RPV).

5 Avoidance of corrosion of carbon dioxide cylinders and bundles

Several methods are currently in use to reduce the risk of incidents due to cylinder corrosion. The different methods include:

- material selection;

- prevention of moisture / water ingress; and
- corrosion detection methods.

These methods can be applied as either single measures or in combination depending on the application in carbon dioxide service.

5.1 Materials selection

5.1.1 Aluminium alloy cylinders

Aluminium alloy cylinders are widely used in the gas industry. Their high corrosion resistance makes them suitable for carbon dioxide and its mixtures even in the presence of water. However, care shall be taken to avoid ingress of fluids into the cylinder, for example, beer and soft drinks syrups, as it shall not be assumed that the alloy will protect entirely against all corrosion mechanisms.

5.1.2 Carbon and low alloy seamless steel cylinders

Cylinders made from low alloy or carbon steels are very widely used for carbon dioxide and its mixtures. In the presence of water, internal corrosion can occur and the rate of corrosion will depend on the amounts of contaminants present in the water. Corrosion rates of about 1 mm per month can be experienced.

5.2 Prevention of water or liquid ingress

5.2.1 Dew point

For cylinder filling, the carbon dioxide shall have a dewpoint below –50 °C.

5.2.2 Use of residual pressure valves

For single cylinders, valve design can minimise the ingress of water during the cylinder use, especially when cylinder valves are left open after use, contrary to the practice recommended by gas suppliers.

A Residual Pressure Valve (RPV) incorporates a device which retains a residual, positive gas pressure inside the cylinder, see EIGA Doc 64, *Guidelines on the Use of Residual Pressure Valves* for more detail [7]. This pressure prevents possible ingress of humid air into the cylinder. Non-Return Valves (NRV) are designed to prevent backflow from the customer's process. Figure 1 shows the differences between standard cylinder valves and cylinder valves with residual pressure valves. Residual pressure valves can be identified by the presence of a pin in the centre of the valve outlet.

Figure 1 – Standard cylinder valve, inline RPV, off-centre RPV comparison

Type 1 RPVs combine the function of a residual pressure valve and a non-return valve (see EIGA Doc 64), thus both of the above advantages are gained [7]. Type 2 RPVs, without an NRV, do not provide the same level of protection against backflow and should be treated as a non-RPV cylinder. Where RPVs are mentioned in this publication, it refers to type 1 RPVs.

5.2.3 Customer installations

Many customer installations are equipped with non-return devices. However, it should not be assumed that these alone provide adequate protection. Therefore, precautions shall be taken for those applications where a risk of backflow contamination exists, see EIGA Doc 67 [5].

5.2.4 Purge wash

For some types of cylinders, a gaseous purge wash can be sufficient to prevent moisture build up in the cylinder. This is filling the cylinder with low pressure carbon dioxide and venting the cylinder for a number of times.

5.3 Moisture and corrosion detection methods

Though several corrosion detection methods are available, such as Ultrasonic Test (UT), Acoustic Emission Test (AET), internal visual inspection (see 5.3.6), tare weight checks (see 5.3.2) and the hammer test. None of these methods is entirely satisfactory for cylinder filling applications.

UT and AET are sophisticated methods usually restricted to periodic inspection as an alternative, or as a supplement to the hydraulic test.

Internal examination is not practical as an prefill inspection but is normally used when other methods indicate suspicion of corrosion. Weight checks and hammer test are relatively simple and inexpensive methods which detect heavy generalised corrosion but will not detect the frequently encountered localised corrosion such as line, pit or crevice corrosion.

Water and liquids are the main reason for corrosion. This section indicates the methods available to detect the presence of water or liquids in carbon dioxide cylinders and bundles or tests that can be performed to ensure no water ingress has occurred.

5.3.1 Residual pressure check

Presence of residual pressure in the cylinder before filling indicates that water ingress is unlikely to have occurred under normal service conditions.

Cylinders or bundles found without residual pressure and when the previous service is not known, should be submitted to one or more prefill procedures, for example, weight check, internal visual inspection, moisture check, evacuation, drying or purging.

5.3.2 Weight check

Water or liquid ingress can be detected by a cylinder weight check. This method is appropriate for carbon dioxide, when the tare weight of the clean empty cylinder is checked before filling.

The sensitivity of this method depends on the size of the cylinder, the accuracy of the scale used and of the stamped tare weight. The maximum weight deviation is 25 - 400 g depending on cylinder size according to ISO 24431, *Gas cylinders - Seamless, welded and composite cylinders for compressed and liquefied gases (excluding acetylene) - Inspection at time of filling* [8].

Similar considerations also apply to bundles, see EN 13365, *Transportable gas cylinders - Cylinder bundles for permanent and liquefied gases (excluding acetylene) - Inspection at time of filling* [9].

5.3.3 Inverting cylinder

Water contamination may be detected by inverting the cylinder, opening the cylinder valve and checking for free water. This method is not suitable for cylinders fitted with dip tubes.

5.3.4 Moisture analysis

Gas withdrawn from the cylinder may be analysed for moisture content (dew point).

5.3.5 Evacuation of cylinders or bundles

The evacuation of single cylinders or bundles before filling may be performed for quality and safety reasons.

When an expected vacuum level is not achievable in a given time, this can be an indication there is free water in one or more of the connected cylinders.

5.3.6 Internal visual inspection

An internal visual inspection checks directly for corrosion. This inspection is normally only performed during the periodic inspection and test cycle for gas cylinders and shall be performed whenever the cylinder valve is removed, such as for repair or change of gas service.

5.4 Guidance for corrosion prevention for carbon dioxide cylinders used in food applications

Each gas company should have a programme to identify cylinders where there is a possibility of internal corrosion, for example, carbon dioxide cylinders and bundles used in food applications.

Carbon dioxide cylinders and bundles used in food applications should be clearly identified when they are returned to distributors or filling stations. Such cylinders shall either be equipped with an RPV or be subjected to an additional prefill check (additional to ISO 24431) such as in 5.3.2, 5.3.3, 5.3.4 or 5.3.5 [8].

If there are indications of contamination, for example, water, the cylinders shall not be filled until they have been internally visually inspected.

6 Avoidance of overfilling single cylinders with carbon dioxide

In order to avoid overfilling, a systematic procedure shall be followed by the personnel carrying out the filling operation. Personnel shall be trained and assessed for competency. The filling process for single cylinders is outlined in Section 6, the filling process for bundles is outlined in Section 7.

6.1 Prefill checks

Prefill inspection requirements shall be in accordance with ISO 24431 [8]. This is a mandatory requirement for the transport of dangerous goods in Europe.

Before a cylinder can be filled, the following steps are necessary:

- external visual inspection of each cylinder to identify cylinders due for testing, defective cylinders, cylinders with defective accessories, or cylinders without tare weight indication; and

- if necessary, clean the cylinder and its accessories of contamination.

6.1.1 Residual pressure

All cylinders shall be checked for the presence of a residual pressure.

During blow down (venting of cylinder), dry ice can be formed in a cylinder and this will be indicated by frost. No inspection or filling shall be performed until all frosting has gone and there is no flow of gas from the cylinder valve.

6.1.1.1 RPV equipped cylinders

The presence of residual pressure shall be checked, this may require using a specifically designed tool. If a positive pressure is detected, the cylinder may be filled. If no residual pressure can be detected the cylinder shall be subject to additional checks (see 5.3) and where moisture backflow is suspected, the cylinder de-valved, the interior condition inspected and if satisfactory a new or refurbished valve fitted.

6.1.1.2 Non RPV equipped cylinders

The residual pressure in the cylinder shall be vented down slowly and safely. All cylinders without an initial residual pressure shall be set aside for further inspection, such as 5.3.3. 5.3.4 and / or 5.3.5.

6.2 Cylinder filling

There are three cases that can be considered for filling:

- cylinders fitted with an RPV returned with positive pressure see 6.2.1;
- cylinders fitted with an RPV returned without positive pressure, see 6.2.2; and
- cylinders not fitted with an RPV returned with or without positive pressure, see 6.2.3.

6.2.1 Filling procedure for cylinders with RPV returned with positive pressure

All food and medical carbon dioxide cylinders, including beverage applications, shall not be top filled i.e. they shall be blown down prior to refill.

Industrial carbon dioxide gas cylinders fitted with RPVs may only be top filled if particular procedures and precautions are in place to ensure that no impurities, for example, or other liquids are present in the cylinder, such that no overfilling of the cylinders can occur.

Before a cylinder can be filled, the following steps are necessary:

1. Complete prefill checks as per ISO 24431 [8].
2. Check for positive pressure and blowdown if required.
3. If positive pressure is detected, fill the appropriate quantity of carbon dioxide, so as not to overfill, see Section 7.
4. If no positive pressure is detected see 6.2.2.

6.2.2 Filling procedure for cylinders fitted with RPV returned without positive pressure

Cylinders fitted with an RPV but returned without positive pressure (typically less than 1.5-2 bar), shall be set aside for separate inspection prior to refill.

Before a cylinder can be filled, the following steps are necessary:

1. Complete prefill checks as per ISO 24431 [8].
2. Check for positive pressure, if no positive pressure can be determined, the cylinder should be subject to a weight check (see 5.3.2) and other additional checks (such as 5.3.3, 5.3.4 and / or 5.3.5) if required.
3. If the tare weight is not correct or any residues are shown the cylinder should have the valve removed and the cylinder internally inspected. If necessary, clean the cylinder and its accessories of dirt, then the normal procedure for the internal cleaning and drying should be followed before the cylinder can be filled.

4. If the tare weight is correct and no residues shown, fill the appropriate quantity of carbon dioxide, so as not to overfill, see Section 7.

6.2.3 Cylinders not fitted with an RPV returned with or without positive pressure

Cylinders not fitted with an RPV returned with or without positive pressure shall have the empty weight checked, see 6.1.1.2.

Before a cylinder can be filled, the following steps are necessary:

1. Complete prefill checks as per ISO 24431 [8].

2. Vent down slowly and safely the residual pressure. Cylinders without residual pressure should be set aside for further inspection such as 5.3.3, 5.3.4 and / or 5.3.5.

3. Check tare weight, see 5.3.2.

4. If the tare weight is not correct or any residues are shown the cylinder should have the valve removed and the cylinder internally inspected. If necessary, clean the cylinder and its accessories of dirt, then the normal procedure for the internal cleaning and drying should be followed before the cylinder can be filled.

5. If the tare weight is correct and no residues shown, fill the appropriate quantity of carbon dioxide, so as not to overfill, see Section 7.

7 Filling procedure for cylinders

It is recommended that the total weight check is performed according to a quality assurance system. Where the entire filling process has been validated to avoid overfilling, random weight checks shall be carried out.

When an empty cylinder is placed on the filling scale, the scale tare weight reading shall be compared to the marked tare weight.

Tolerances for the tare weight, depending on cylinder size, shall be taken from the relevant EN or ISO standards (see ISO 24431 or EN 13365). Total fill weight tolerance shall be taken from local regulations where they exist otherwise relevant EN or ISO standard tolerance may be used [8, 9]. If cylinder weight deviate from the given tolerances, the cylinder shall be put on one side for further inspections.

When the cylinder is connected to the filling line, the desired filling weight shall be set on the filling scale before opening the cylinder valve and then starting the filling process.

Usually, the filling scale will terminate the filling process automatically when the maximum permissible filling weight is reached. After closing the cylinder valve and draining the liquid carbon dioxide trapped between cylinder valve and shut off valve, the maximum permissible filling weight of the cylinder shall be checked.

In cases where overfilling occurs, the excess product shall be vented to a safe location until the desired filling weight is reached.

8 Avoidance of overfilling of bundles with carbon dioxide

The procedure to prevent overfilling of bundles with carbon dioxide fitted with or without an RPV is similar to single cylinders filled with carbon dioxide and equally important. The relevant parts of Section 6 shall apply as well as EN 13365 [9].

8.1 Preparation for filling bundles

During blow down, dry ice can form in a bundle and this will be indicated by frost. No inspection or filling should be performed until all frosting has gone and there is no flow of gas from the main bundle isolation valve.

8.2 Filling procedure for carbon dioxide bundles

At the beginning and the end of the filling process, a leak test on all valves and joints in the piping system of the bundle is recommended, see EIGA Doc 78, *Leak Detection Fluids Cylinder Packages* [10].

Warning: *Where bundles are equipped with a main filling / discharge valve and valves on each cylinder, ensure that all cylinder valves are in the open position prior to, and after filling.*

9 Weigh scales

9.1 Accuracy of the weigh scales

Only scales that meet the appropriate accuracy requirements, (see 5.3.2) shall be used.

This applies to all types of scales for filling cylinders with carbon dioxide including:

- manually operated scales;
- semi-automatic scales; and
- automatic filling with integrated weight control.

The filling scales should be checked daily prior to the filling operation. Suitable control weights shall be used, and the result should be recorded in a log.

Only scales calibrated to a recognised standard should be used.

NOTE There may be local legislation requirements for calibration of weigh scales.

WARNING: *The potential interference of the filling hose with respect to the final weighing result shall be considered to avoid overfilling.*

9.2 Maintenance and inspection of the weigh scales

To ensure that the scales are working correctly at required sensitivity levels, maintenance, inspection and calibration of the scales by the manufacturer or recognised body on a regular basis, for example every year, is recommended. Each maintenance work or inspection should be documented.

NOTE Some countries legally require period calibration of weighing equipment.

10 Bursting discs

One of the most important measures to prevent any incidents or damage due to overfilling is the use of bursting discs on all valves fitted to single cylinders and bundles filled with carbon dioxide.

The burst pressure is dependent on the filling ratio and test pressure of the cylinder.

For more details about the use of bursting discs see EIGA Doc 64 and EN 14513 *Transportable gas cylinders - Bursting disc pressure relief devices (excluding acetylene gas cylinders)* [7, 11]

11 References

Unless otherwise stated the latest edition shall apply.

[1] EIGA Doc 125, *Guide to the Supply of Gases for Use in Foods*, www.eiga.eu.

[2] EIGA Doc 99 *Part 1, Good Manufacturing Practice Guide Part I for Medical Gases*, www.eiga.eu

[3] EIGA Doc 99 *Part 2, Good Manufacturing Practice Guide Part II for Medical Gases: Basic Requirements for Active Substances Used as Starting Materials*, www.eiga.eu.

[4] EIGA Doc 62, *Methods to Avoid and Detect Internal Gas Cylinder Corrosion*, www.eiga.eu.

[5] EIGA Doc 67, *Carbon Dioxide Cylinders at Users' Premises*, www.eiga.eu.

[6] EIGA Doc 95, *Avoidance of Failure of CO and of CO/CO2 Mixtures Cylinders*, www.eiga.eu.

[7] EIGA Doc 64, *Guidelines on the Use of Residual Pressure Valves* www.eiga.eu.

[8] EN ISO 24431, *Gas cylinders - Seamless, welded and composite cylinders for compressed and liquefied gases (excluding acetylene) - Inspection at time of filling,* www.cen.eu.

[9] EN 13365, *Transportable gas cylinders - Cylinder bundles for permanent and liquefied gases (excluding acetylene) - Inspection at time of filling,* www.cen.eu.

[10] EIGA Doc 78, *Leak Detection Fluids Cylinder Packages* www.eiga.eu.

[11] EN 14513, *Transportable gas cylinders - Bursting disc pressure relief devices (excluding acetylene gas cylinders)* www.cen.eu.