RECOMMENDATIONS FOR SAFE FILLING OF CO_2 CYLINDERS AND BUNDLES

AIGA 069/10

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Revised by the members of Ad Hoc Group 8.1

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Acknowledgement

This document is adopted from the European Industrial Gases Association's (EIGA) document IGC 83/08 'Recommendations for safe filling of CO2 cylinders and bundles'. Acknowledgement and thanks are hereby given to EIGA for permission granted for the use of their document.

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1 Introduction

Although very few CO_2 cylinder incidents are reported by the Industrial Gas Producers approximately 30% of these are the result of either:

- Internal corrosion, due to ingress of water or other liquids into CO_2 cylinders or bundles
or
- Overfilling and subsequent failure of CO_2 cylinders

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

This document aims to give recommendations and an overview of the gas industry's current practices to prevent and detect internal corrosion and to avoid overfilling CO_2 cylinders and bundles.

The objective is to provide specific guidelines for the safe filling of carbon dioxide in either single cylinders or cylinder bundles. It is intended to be followed by the industrial gases industry to eliminate/reduce the number of accidents involving both cylinders and bundles containing carbon dioxide, due to the above-mentioned reasons.

2 Scope

This document applies to the safe filling of gas cylinders and bundles containing carbon dioxide as a single product. The instructions/recommendations given in this document are not intended to replace current regulations in EIGA-member countries. Also it does not cover the entire filling process. This paper does not address the additional requirements resulting from the implementation of Good Manufacturing Practices for medical or for food gases, see EIGA doc 125/06

Other Documents apply to CO/CO_2 cylinders (see Doc 95/07), Methods to avoid and detect internal gas cylinder corrosion. (Doc. 62/08) and “CO_2 cylinders at users’ premises”. (Doc. 67/08).

3 Definitions

3.1 Corrosion

Deterioration of the cylinder material by an electro-chemical reaction, when in contact with water or other liquids (e. g. CO_2 and water).

3.2 Overfilling

A CO_2 cylinder is overfilled, when the total weight marked on the cylinder (tare weight + maximum permissible filling weight) is exceeded. Permissible deviations from the total weight are given under clause 5.3.2.

3.3 Cylinder Bundles

A portable assembly, which is designed for being routinely lifted and which comprises of a frame and two or more cylinders each of capacity up to 150 litres connected to a manifold by cylinder valves or fittings such that the cylinders are filled, transported and emptied without disassembly. The term "cylinder bundle" is synonymous with the term "bundle".

3.4 Empty weight

3.4.1 Empty weight of the Cylinder

The weight of the cylinder including all integral parts (for example neck ring, foot ring, etc.), followed by the letters "KG". This weight does not include the weight of valve, valve cap or valve guard, any coating.

3.4.2 Empty weight of the Bundle

The empty weight of the bundle is equal to the tare weight.

3.5 Tare weight

3.5.1 Tare Weight - Cylinder

The tare weight of the cylinder is the sum of the empty weight, the mass of the valve including dip tube where fitted, any fixed valve guard and the mass of all other parts which are permanently attached to the cylinder (for example by clamping or bolting) when presented for filling.

3.5.2 Tare Weight - Bundle

The tare weight of a bundle includes the items in 3.5.1 and the support frame and manifold piping.

3.6 Maximum permissible filling weight

The product of the minimum guaranteed water capacity of the cylinder and the filling ratio of the gas.

3.7 Total weight

For CO_2 cylinders the total weight comprising the tare weight plus the maximum permissible filling weight.

4 Sources of moisture contamination in CO2 cylinders and bundles

Several sources of possible free moisture contamination may be found during the life of a CO_2 cylinder, i.e. 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 absolutely 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 also clause 5.2)

4.2 Water from product and prefill treatment

Though not a current problem in modern filling facilities, it is possible to fill cylinders with products containing moisture. Additionally some prefill operations may introduce moisture into cylinders, e.g. if water-ring vacuum pumps are used, without adequate precautions to prevent water carry over.

4.3 Water feedback during use

Water/liquid feedback into cylinders may occur whenever the cylinder is at a lower pressure than the application involving a fluid to which it is connected. Many CO_2 users are found in the beverage/ food industry, so that feedback of beer and soft drink syrups and other liquids is a constant risk. For more

detailed information see IGC-Doc 62/08 or AIGA 062/09 “Methods to avoid and detect internal gas cylinders corrosion” and IGC-Doc 67/08 “CO_2 cylinders at users’ premises”.

5 Avoidance of CO_2 cylinder and cylinder bundles corrosion

To reduce the risk of accidents due to cylinder corrosion several methods are presently in use. The different methods are based upon material selection, prevention and detection methods. These methods can be applied as either single measures or in combination depending on the application in CO_2 service.

5.1 Materials selection

5.1.1 Aluminium Alloys

Aluminium alloy cylinders are widely used in the gas industry. Their high corrosion resistance makes them suitable for CO_2 and its mixtures even in the presence of water. However, care shall be taken to avoid ingress of fluids into the cylinder e. g. beer, soft drinks syrups, as it must not be assumed that the alloy will protect entirely against all corrosion mechanisms.

5.1.2 Carbon steels and low alloy steels

Cylinders made from low alloy or carbon steels are very widely used for CO_2 and its mixtures. In the presence of water, internal corrosion will occur and the rate of corrosion will depend on the amount of contaminants present in the water. If the contamination level is at the cylindrical wall, corrosion rates of about 1 mm per month can be experienced.

5.2 Avoidance of water or liquid ingress

For cylinder filling the CO2 shall have a minimum dew point below – 50 °C.

5.2.1 Use of residual Pressure Valves

For single cylinders, valve design can help 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 IGC-Doc.-No. 64/05 “Guidelines on the use of residual pressure valves” for more detail). 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.

Some valves combine the function of a residual pressure and a non-return valve, thus both of the above advantages are gained.

To gain full confidence in these various valving approaches, adequate experience needs to be acquired prior to widespread use.

5.2.2 Customer installation

Many customer installations are equipped with non-return devices. However, it should not be assumed that these alone provide adequate protection. Therefore, special precautions shall be taken for those applications where a risk of backflow contamination exists (see clause 4.3).

5.3 Moisture detection methods

Water and liquids are the main reason for corrosion. The aim of this clause is to indicate the methods available to detect the presence of water or liquids in CO_2 cylinders and bundles.

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/bundles found with no residual pressure and when the previous service is not known, should be submitted to one or more special prefill procedures e.g. weight check, internal visual inspection, moisture check, evacuation, drying, purging etc.

5.3.2 Weight check

If a significant amount of water or liquid is present, this can be detected by a cylinder weight check. This method is appropriate for CO_2, when the tare weight of the empty cylinder is checked.

The sensitivity of this method depends on the size of the cylinder, the accuracy of the scale used and of the stamped tare weight (permissible tolerance +/- 50-400 g according to EN 1968/EN 1802).
Similar considerations as above also apply to cylinder bundles. The permissible tolerance range is +/- 0,5 % (see EN 13365) of the bundle's total weight.

5.3.3 Internal visual inspection

This inspection is normally performed during the periodic inspection and test cycle for gas cylinders, and shall be performed whenever the valve is removed, e. g. for repair or change of gas service.

5.3.4 Cylinder/bundle evacuation

The evacuation of single cylinders or bundles before filling is a common procedure for quality and for safety reasons.

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

5.3.5 Purge wash

For some types of cylinders a gaseous purge wash may be sufficient.

5.4 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), tare weight checks, hammer test etc., none of them is entirely satisfactory for cylinder filling applications.

UT and AET are sophisticated methods involving expensive and time-consuming procedures, and are applicable to single cylinders and bundles. (AET only).

For this reason their use is restricted to periodic inspection as an alternative or as a supplement to the hydraulic test.

Internal examination is not practical as an “in line” 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.

5.5 Guidance for corrosion prevention

Each gas company should have or be implementing a programme to identify cylinders where there is a possibility of internal corrosion, e.g. carbon dioxide cylinders and bundles used in “food” applications.

Prevention measures:

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 a RPV/NRV or be subjected to a special “prefill check” .

The special “prefill check” shall include:
- Emptying of gas and check weighing of the tare weight (see clauses 6.1.4.1 and 6.1.3).

or alternatively

- Inverting the cylinder (not applicable for dip tube cylinders)

If there are indications of contamination, e.g. water, the cylinders shall not be filled until internally visually inspected.

5.6 Bundle Design

Bundles shall have at least one main valve. Individual cylinders should not be valved due to the risk of overfilling in the event one of the valves remaining closed. The valve outlet shall be horizontal or facing down. The exact nature of the valve will be dependent on technical issues revolving around filling/emptying rates.

In most applications the incorporation of a residual pressure/ non-return valve will reduce the risk of backflow.

6 Avoidance of overfilling CO_2 cylinders and bundles

In order to avoid overfilling, a systematic procedure is necessary to be followed by the operators. Special care shall be taken at every step. Experienced and well-trained people are required to follow the filling instructions.

6.1 Single cylinders

In order to ensure the avoidance of overfilling CO_2 cylinders, it is essential to describe the preparation and filling in detail. Each of the steps explained below, collectively will ensure absolute safety during filling, transportation and customer use.

6.1.1 Prefill Inspection (see also EN 1919….)

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

- external visual inspection of each cylinder to sort out cylinders due for testing, defective cylinders or cylinders with defective accessories, cylinders without tare weight indicator.
- if necessary, clean the cylinder and its accessories of contamination.

6.1.2 Residual Pressure

All cylinders shall be checked for residual pressure.

RPV/NRV equipped cylinders

These cylinders shall be “prod checked” using a specially designed probe. If a positive pressure is given, the cylinder may be filled. If no residual pressure can be determined the cylinder shall be devalved, the interior condition inspected and if satisfactory a new/refurbished valve fitted.

Non-RPV/NRV equipped cylinders

Vent down slowly and safely the residual pressure. All cylinders without an initial residual pressure shall be set aside for further inspection, (see clause 5.3)

During blow down, dry ice may form 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 trickle of gas from the cylinder valve.

Three cases can now be considered for filling:

Case 1) Cylinders fitted with a Residual Pressure Valve (RPV)/Non Return Valve (NRV) returned with positive pressure may be “top filled”, subject to the note below, (see 6.1.3).

Note : Top filling using RPV-valves with a non return function can only be accepted, when it can be ensured by a safe and reliable method, that no impurities (water or other liquids) are present in the cylinders (bundles) and no overfilling of the cylinders (bundles) can occur.
This filling procedure shall not be allowed for CO2, intended for use in food processes, including beverages, and medical applications.

Case 2) Cylinders fitted with a Residual Pressure Valve (RPV)/Non Return Valve (NRV), but returned with no positive pressure, shall be set aside for separate inspection prior to refill.

Case 3) Cylinders not fitted with either a RPV or a NRV returned with or without positive pressure should have the empty weight checked (see 6.1.5).

6.1.3 Filling procedure for CO_2 cylinders with RPV/NRV returned with positive pressure (Case 1)

- Blowdown carefully and check for positive pressure
- If positive pressure then fill the appropriate quantity of CO2, so as not to overfill
- If no positive pressure then subject to further checks - such as tare weight check and inverting or internal inspection

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 CO_2, trapped between cylinder valve and shut off valve, the maximum permissible filling weight of the cylinder shall be checked.

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.

Suitable tolerances for the tare weight shall be taken from the relevant EN Standards (see under clause 9 ‘References’) and equivalent tolerance shall be used for the total weight.

In cases, where overfilling occurs, the excess gas product shall be released safely, until the desired filling weight is reached.

6.1.4 Filling procedure (Case 2)

6.1.4.1 Cylinder preparation for filling

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

- check the cylinder for residual pressure. If no residual pressure can be determined, the cylinder should be checked by tare weight or by turning upside down for any liquid content. If the tare weight is correct or no residues shown, the cylinder can be filled.
  If the tare weight is not correct or any residues are shown it 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 follow before the cylinder can be filled.

- external visual inspection shall be performed as described under clause 6.1.1.

6.1.4.2 Filling procedure for CO_2 cylinders with RPV and NRV returned without positive pressure.

The same filling procedure as stated under clause 6.1.4.1 applies.

6.1.5 Filling procedure (Case 3)

6.1.5.1 Cylinder preparation for filling

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

- Vent down slowly and safely the residual pressure

- Cylinders without residual pressure should be set aside for further inspection (see clause 5.3)

- External visual inspection of each cylinder to sort out cylinders due for testing, defective cylinders or cylinders with defective accessories, cylinders without tare weight indicator

- if necessary, clean the cylinder of dirt.

6.1.6 Filling Procedure for other CO_2 Cylinders (Cases 2 and 3)

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

Different data applies, depending on the cylinder size; suitable tolerances for the tare weight shall be taken from the relevant EN Standards (see under clause 9 ‘Reference’).
If cylinders 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 has to be set on the filling scale and the cylinder valve can be opened. Then the filling process can begin.

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 CO_2, trapped between cylinder valve and shut off valve, the maximum permissible filling weight of the cylinder shall be checked.

It is recommended, that the total weight check be 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.

In cases, where overfilling occurs, the excess gas product shall be released safely, until the desired filling weight is reached.

6.2 CO_2 bundles

The procedure to prevent overfilling CO_2 bundles fitted with or without an RPV/NRV is similar to CO_2 cylinders and equally important. The relevant parts of 6.1 shall apply.

6.2.1 Bundle preparation for filling

During blow down, dry ice may 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 trickle of gas from the master valve.

6.2.2 Filling procedure for CO_2 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.

Warning note :
In case of cylinder bundles equipped with a main filling/discharge valve and individual valves on each cylinder, ensure that all individual valves are in the open position prior and after filling.

7 Weigh Scales

7.1 Accuracy of the weigh scales

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

This applies to all different types of scales for filling CO_2 cylinders used in the gas industry.

- Manually operated scales
- Semi automatic scales
- 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 approved and marked by a recognized body or authority should be employed. In order to make continuous safety improvements, when replacing weighing scales, as tight a tolerance as practical should be specified.

Warning note:
Special consideration shall be given to potential interference of the filling hose with respect to the final weighing result.

7.2 Maintenance and inspection of the weigh scales

To make sure, that the scales are properly working at such high sensitivity levels as mentioned above, maintenance and inspection of the scales by the manufacturer on a regularly basis (e. g. every six months) is recommended. Each maintenance work or inspection should be documented.

8 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 CO_2 cylinders and CO_2 bundles.

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 IGC-Doc 64/05 “Use of pressure relief devices for gas cylinders” and CEN-Standard EN 1268-3 “Bursting discs/safety devices”.

9 References

- IGC-Doc 62/08 (AIGA 062/09) "Methods to avoid and detect internal gas cylinder corrosion"
- IGC-Doc 64/05 "Guidelines on the use of Residual Pressure Valves"
- IGC-Doc 67/08 “CO_2 cylinders at users’ premises
- IGC-Doc 91/03 “Use of pressure relief devices for gas cylinders”
- IGC-Doc 95/07 (AIGA 65/09) "Recommendations for the avoidance of failure of CO and CO/ CO_2 mixtures cylinders”
- IGC-Doc 125/06 “Guide to the supply of gases for use in foods”
- EN 13365 "Cylinder bundles for permanent and liquefied gases (excluding acetylene). Inspection at time of filling"
- EN 1089-1 "Gas cylinder identification (excluding LPG) Part 1: Stamp marking"
- EN 1919 "Gas cylinders for liquefied gases (excluding acetylene and LPG) - Inspection at time of filling"
- EN 1968 “Periodic inspection and testing of seamless steel gas cylinders”
- EN 1802 “Periodic inspection and testing of seamless aluminium gas cylinders”
- EN 1268-3 “Bursting discs/safety devices”
- CGA G-6-3 Carbon Dioxide Cylinder filling and Handling Procedures