INTERNATIONAL STANDARD 3427

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Gaseous halogenated hydrocarbons (liquefied gases) — Taking of a sample

Hydrocarbures halogénés gazeux (gaz liquéfiés) — Prélèvement d'un échantillon

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3427 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in February 1974.

It has been approved by the Member Bodies of the following countries :

Austria
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Bulgaria
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No Member Body expressed disapproval of the document.

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Gaseous halogenated hydrocarbons (liquefied gases) — Taking of a sample

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the apparatus and the procedures to be used when taking a laboratory sample of a gaseous halogenated hydrocarbon (as a liquefied gas) from a container (cylinder, drum, tank, etc.).

2 PRINCIPLE

Taking of a sample of halogenated hydrocarbon in a stainless steel cylinder, previously cleaned, dried and evacuated.

The sample cylinder is filled to the filling ratio allowed by safety considerations : relevant details will be given in the individual International Standards for the products concerned.

This sample shall be taken only from the liquefied phase.

3 APPARATUS

3.1 Sampling assembly (see figure 2).

3.1.1 Sample cylinder, constructed of stainless steel, capable of withstanding an internal pressure of at least 3 MPa* or as prescribed for a particular product**, and of one of the two types shown in figure 1 :

a) sample cylinder fitted with two needle valves, connected respectively to an internal dip-tube extending to near the bottom of the cylinder and to a short internal tube (length 30 mm);

b) sample cylinder with only one valve and without internal tubes.

The sample cylinder shall be designed so as to be easily cleaned and dried and its valve outlet(s) shall be fitted with sealing cap(s) for use during transport. Its capacity depends on the quantity of the product required to carry out all of the intended analyses. This quantity shall be small in relation to the total quantity contained in the cylinder in order to avoid any variation in the composition of the liquid phase.

NOTE — The interior of the sample cylinder shall be examined periodically. If the surface is not clean, wash it with water and an appropriate solvent, for example ethanol or acetone.

Check its pressure-tightness periodically, for example by immersing it in water and filling it with nitrogen to a pressure of about 3 MPa*.

3.1.2 Connecting tube, preferably made of stainless steel, of suitable length and having threaded connectors (unions) fitting respectively the valve of the liquefied halogenated hydrocarbon container and the valve of the sample cylinder (3.1.1).

All connectors shall be fitted with washers made of a material which is not attacked by the product to be analysed.

3.2 Electric oven, capable of being controlled at 105 to 110 °C.

3.3 Balance, accurate to ± 1 g.

3.4 Vacuum pump, capable of reducing the pressure in the cylinder (3.1.1) rapidly to about 100 Pa***.

3.5 Desiccator, capable of containing the cylinder (3.1.1), with an efficient desiccant.

3.6 Compressed nitrogen, clean and dry.

3.7 Cooling bath, at a temperature appropriate to the product to be sampled.

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* 3 MPa = 30 bar

** If there exists a national or international regulation on this subject, it should be respected.

*** 100 Pa = 1 mbar

4 PROCEDURE

4.1 Preparation of the cylinder (3.1.1) and connecting tube (3.1.2).

4.1.1 In the case of a cylinder of type (a) — with two valves — carry out the three following operations in succession :

- purge the cylinder and connecting tube with the nitrogen (3.6) at ambient temperature;
- continue this purging in the oven (3.2), controlled at 105 to 110 °C, for at least 30 min by means of a thick-walled flexible tube passing through an opening in the side of the oven;
- reduce the pressure in the connecting tube/cylinder assembly, after its removal from the oven and while still hot, to about 100 Pa or lower, and maintain at this pressure for at least 30 min.

4.1.2 In the case of a cylinder of type (b) — with only one valve — carry out the five following operations in sucession :

- remove the valve of the cylinder, join a suitable flexible tube to the connecting tube (3.1.2) and pass a stream of the nitrogen (3.6) through these and then, by means of a further length of flexible tube, into the cylinder emerging inside near the bottom;
- continue this purging with the nitrogen (3.6) in the oven (3.2), controlled at 105 to 110 °C, for at least 30 min by means of a thick-walled flexible tube passing through an opening in the side of the oven;
- cool the valve, the cylinder and the connecting tube in the desiccator (3.5);
- reassemble the valve and cylinder, and fit the connecting tube;
- reduce the pressure in the connecting tube/valve/cylinder assembly to about 100 Pa or lower, and maintain at this pressure for at least 30 min.

NOTE — When the sample cylinder is used only for one product of practically constant composition, the procedure specified above may be simplified as follows. Empty the cylinder of the remainder of the previous sample. Fit the connecting tube (3.1.2), evacuate to a pressure of about 100 Pa and then allow to refill with the nitrogen (3.6) at atmospheric pressure. Repeat the evacuation and refilling twice and place the cylinder and connecting tube, with the valve fully open, in the oven (3.2), controlled at 105 to 110 °C, for at least 1 h. Reduce the pressure in the still-hot cylinder and connecting tube to about 100 Pa or lower, and maintain it at this pressure for at least 30 min.

4.1.3 In both cases, close the valve(s) of the cylinder to retain the vacuum. Disconnect the cylinder and connecting tube from the vacuum pump (3.4) and from each other and weigh the cylinder, using the balance (3.3).

4.2 Taking of the sample

Cool the cylinder (3.1.1) by immersing it in the cooling bath (3.7) for 10 to 15 min, taking care to keep the valve(s) free from condensation.

Clean the mouth of the liquid-phase valve on the container from which the sample is to be taken, using a jet of the nitrogen (3.6).

When the sample is to be taken from a container fitted with only one valve, position the container so that the sample will be taken from the liquid phase.

Join the connecting tube (3.1.2) hermetically to the liquid phase valve of the container. Fit the other end of the tube (3.1.2) to the valve of the sample cylinder (3.1.1) (valve A for cylinder type (a)), but without tightening the connection (see figure 2).

Cautiously open the valve of the container and allow the liquid to flow so that the air present in the connecting tube is forced out through the non-tightened connection. Then tighten the connection on the valve of the sample cylinder to make a hermetic seal.

Open the valve of the cylinder (3.1.1) and allow it to fill with liquid. Close the valves and disconnect the cylinder. Check the filling of the cylinder immediately after taking the sample.

NOTE — It is recommended that, if suitable apparatus (spring balance and flexible connecting tube) is available, the cylinder be weighed during filling.

4.3 Checking of filling

Using the balance (3.3), reweigh the full cylinder (3.1.1) to check on the filling operation, taking into account the total capacity of the cylinder and the density of the liquid phase halogenated hydrocarbon concerned.

If the recommended filling ratio of the sample cylinder has been exceeded, discharge the excess as follows. For a cylinder of type (a), hold it with the valves uppermost, open valve B and close it when the discharge consists of vapour only. For a cylinder of type (b), hold it with the valve underneath, open the valve carefully and allow the necessary amount of liquid to escape. Fit the valve outlet caps.

Weigh the cylinder again to check the filling and check its pressure-tightness by reweighing later.

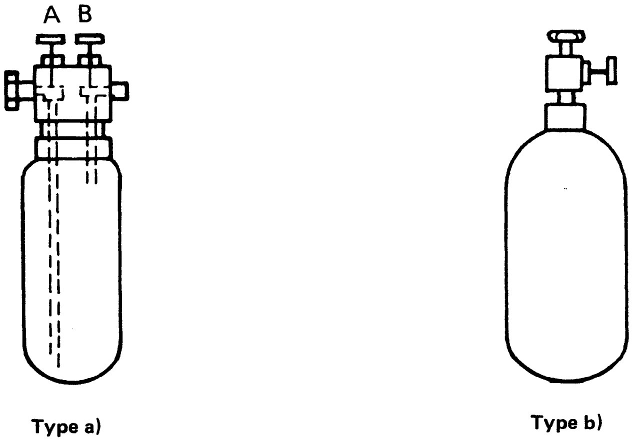

FIGURE 1 — **Sample cylinder (3.1.1)**

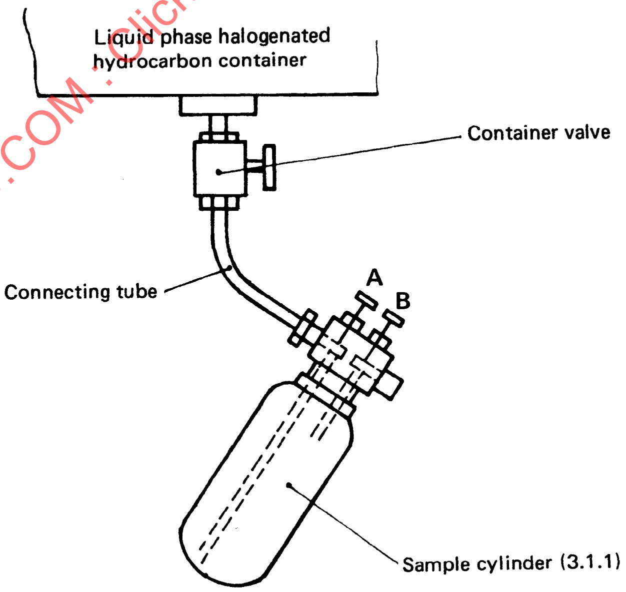

FIGURE 2 — **Assembly using a type (a) sample cylinder**

ANNEX

OTHER ISO PUBLICATIONS RELATING TO HALOGENATED HYDROCARBONS FOR INDUSTRIAL USE

ISO/R 1393 – Determination of the acidity [liquids].

ISO/R 1394 – Determination of the cloud point [liquids].

ISO 2209 – Sampling [liquids].

ISO 2210 – Determination of residue on evaporation [liquids].