International Standard 7103

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Liquefied anhydrous ammonia for industrial use — Sampling — Taking a laboratory sample

Ammoniac anhydre liquéfié à usage industriel — Échantillonnage — Prélèvement d'un échantillon pour laboratoire

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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 7103 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in July 1981.

It has been approved by the member bodies of the following countries :

Austria	India	Poland
Belgium	Italy	Portugal
Brazil	Korea, Dem. P. Rep. of	Romania
Czechoslovakia	Korea, Rep. of	South Africa, Rep. of
Egypt, Arab Rep. of	Mexico	Switzerland
France	Netherlands	Thailand
Germany, F. R.	New Zealand	USSR
Hungary	Philippines	

The member body of the following country expressed disapproval of the document on technical grounds :

United Kingdom

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Liquefied anhydrous ammonia for industrial use — Sampling — Taking a laboratory sample

WARNING — Liquefied anhydrous ammonia is a highly corrosive, toxic substance, which boils at −33,3 °C at standard atmospheric pressure. Its action on the skin and eyes is strongly corrosive, producing severe and painful burns.

Its vapour is strongly irritant to the mucous membrane and eyes, and produces a suffocating effect on the respiratory tract.

In concentrations of 16 to 25 % (*V*/*V*), gaseous anhydrous ammonia forms explosive mixtures with air.

Personnel responsible for handling the product shall be fully informed as to its dangerous character and the precautions to be taken.

Operators shall wear rubber gloves a rubber apron and full face and head protection, and shall be provided with a protective gas-mask fitted with a filter for ammonia.

Samples shall be handled only inside a well-ventilated fume-cupboard.

For further information, see the appropriate sections of ISO 3165.

1 Scope and field of application

This International Standard specifies the apparatus and the procedure to be used for taking a representative laboratory sample* of liquefied anhydrous ammonia for industrial use, from a container (barrel, cylinder, tank, etc.).

2 Reference

ISO 3165, *Sampling of chemical products for industrial use — Safety in sampling.*

3 Principle

Removal of a liquified anhydrous ammonia sample into a stainless steel cylinder, which has previously been cleaned, dried and evacuated. Filling the sampling cylinder with the stipulated quantity that is permissible with regard to safety.

The filling rate shall never exceed 75 % of the capacity of the cylinder at ambient temperature.

4 Apparatus

4.1 Equipment for taking the sample, including the following items :

4.1.1 Sampling cylinder (see typical cylinder in figure 1), made of stainless steel, effective capacity not less than 1 litre, capable of withstanding an internal pressure of not less than 3 MPa**.

Check the volume of the cylinder by filling with water. The sampling cylinder shall have two needle valves, A and B, joined to two tubes inside the cylinder, the first reaching almost to the bottom and the other of a length which makes it possible to guarantee the degree of safe filling (see clause 3 and 5.3).

The cylinder shall be designed in such a way as to be easily cleaned and dried, and the valve outlets shall have caps for transportation.

NOTES

1 The capacity of the cylinder depends on the quantity of product required for carrying out all the required tests.

* In this specific case, the laboratory sample is the same as the test sample.

** 3 MPa = 30 bar

2 The interior of the cylinder shall be inspected from time to time. If the surface is not clean, wash it with running water, dry it, wash it several times with pure acetone and finally flush the cylinder with the nitrogen (4.5). At the same time, check the air-tightness of the cylinder, for example by immersion in water and filling it with nitrogen to a pressure of about 3 MPa.

4.1.2 Connecting tube (see figure 2), preferably of stainless steel, of a suitable length, internal diameter 5 mm in which is inserted tightly and permanently, the three-way valve (4.1.3). The connecting tube is fitted with threaded joints one of which connects to the valve on the liquified anhydrous ammonia container and the other to valve A of the cylinder (4.1.1).

All the connections shall be of a material that is resistant to ammonia (for example, hard rubber, metal alloys with a high lead content, excluding materials containing copper).

This connecting tube shall be designed to withstand a pressure of 3 MPa.

4.1.3 Three-way valve (see figure 2) inserted in the connecting tube (4.1.2). The valve can be adjusted to allow liquid ammonia to flow from the container, either to atmosphere or to the inlet of the sample cylinder, or to enable the inlet of the sample cylinder to be opened to atmosphere with the valve from the container closed.

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

4.3 Balance, capable of weighing to an accuracy of 1 g.

4.4 Vacuum pump, capable of rapidly reducing the pressure in the cylinder (4.1.1) to about 100 Pa*.

4.5 Cylinder of compressed nitrogen, clean and dry.

4.6 Freezing bath, a mixture of solid carbon dioxide (dry ice) and trichloroethane. The temperature may fall to −35 °C. The bath is fitted with an appropriate thermometer.

5 Procedure

5.1 Preparation of the cylinder and the connecting tube

Carry out the following operations in the given order :

a) Connect the connecting tube (4.1.2) to the cylinder (4.1.1) with valves A and B open. Turn the three-way valve (4.1.3) so as to allow passage of the nitrogen (4.5) through the assembled apparatus. Clean the assembled apparatus at ambient temperature by flushing with the nitrogen.

b) Continue the flushing of the assembled apparatus for not less than 30 min in the oven (4.2), controlled at 105 to 110 °C, via a flexible thick-walled tube passing through an opening in the wall of the oven; connected to the connecting tube (4.1.2).

c) Disconnect the connecting tube (4.1.2) from the flexible thick-walled tube which allowed nitrogen to pass through the apparatus and turn the three-way valve so as to isolate the apparatus from the atmosphere, leaving valves A and B of the cylinder open. Connect the vacuum pump (4.4) on the outlet of the cylinder (B) and lower the internal pressure to about 100 Pa or less. Maintain the apparatus with this pressure for not less than 30 min.

d) Close the two valves A and B on the cylinder.

e) Disconnect the cylinder from the connecting tube and weigh it empty, to the nearest 1 g, on the balance (4.3).

5.2 Taking the sample

Cool the cylinder (4.1.1) by immersing it in the freezing bath (4.6), regulated between −30 and −35 °C, for 10 to 15 min, avoiding any condensation on the valves.

Fit the connecting tube (4.1.2) hermetically on the liquid phase valve of the container and connect the other end of the connecting tube to the valve A of the cylinder, tightening the joint (see figure 2).

It is essential to open the three-way valve to the atmosphere before opening the container valve.

Carefully open the valve on the container and let the liquid flow so that the air present in the connecting tube discharges through the three-way valve. Allow the liquid to flow freely to clear the sample line and valve completely. Turn the three-way valve to connect the container to the sample cylinder, open valve A on the cylinder and allow it to fill with the liquid at a safe level. Close valve A and the container valve and disconnect the cylinder.

Immediately after taking the sample, check the amount in the cylinder as specified in 5.3.

NOTE — It is recommended that if suitable apparatus is available, the cylinder be weighed during filling so as to eliminate the need for the filling check (5.3).

5.3 Filling check

Using the balance (4.3), weigh, to the nearest 1 g, the full cylinder (4.1.1), to establish its contents, taking into account the total capacity and the density of the liquid phase of the liquefied anhydrous ammonia (ϱ 0,68 g/ml). If the quantity recorded exceeds the recommended quantity, discharge the excess as follows :

Connect the outlet of valve B of the cylinder to a flexible thick-walled tube, carefully open valve B while holding the cylinder in a vertical position, valves uppermost. Allow a certain quantity of the product to flow out and close valve B when only vapour appears.

Disconnect the flexible tube and weigh the cylinder again to the nearest 1 g.

NOTE — Check periodically the air-tightness of the cylinder. The two control weighings should be carried out at a suitable interval.

* 100 Pa = 1 mbar

Figure 1 — Typical sampling cylinder (4.1.1)

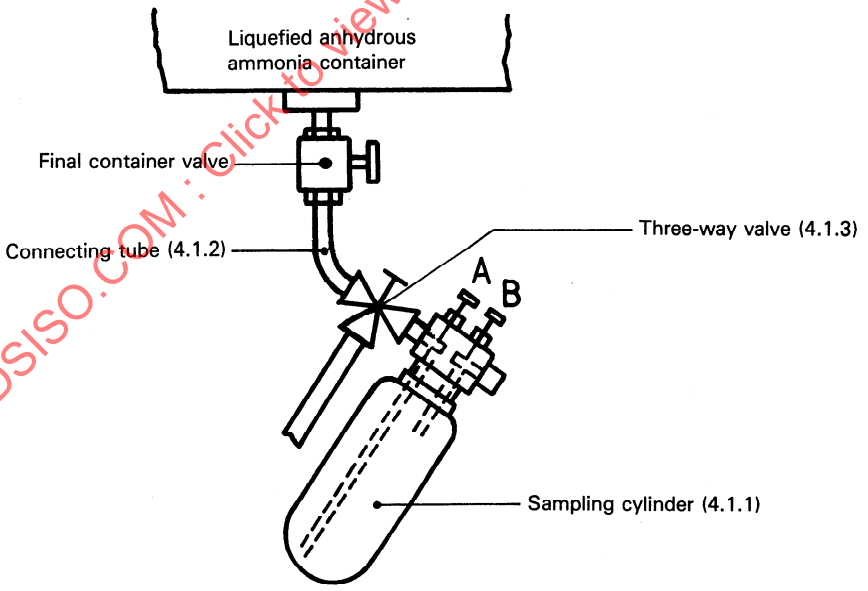

Figure 2 — Sketch of assembly

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