

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 1552

LIQUID CHLORINE FOR INDUSTRIAL USE

METHOD OF SAMPLING

(FOR DETERMINING ONLY THE VOLUMETRIC CHLORINE CONTENT)

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1552, *Liquid chlorine for industrial use – Method of sampling (for determining only the volumetric chlorine content)*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1552 which was circulated to all the ISO Member Bodies for enquiry in July 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Iran	South Africa, Rep. of
Belgium	Israel	Spain
Colombia	Italy	Switzerland
Cuba	Korea, Rep. of	Thailand
Czechoslovakia	Netherlands	Turkey
France	New Zealand	U.A.R.
Germany	Poland	United Kingdom
Hungary	Portugal	U.S.S.R.
India	Romania	

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

LIQUID CHLORINE FOR INDUSTRIAL USE

METHOD OF SAMPLING

(FOR DETERMINING ONLY THE VOLUMETRIC CHLORINE CONTENT)

1. SCOPE

This ISO Recommendation describes a method of sampling liquid chlorine for industrial use, with a view to determining only the volumetric chlorine content.

2. FIELD OF APPLICATION

The method can be applied to the sampling of liquid chlorine for industrial use, with a view to determining only the volumetric chlorine content, when an agreement has previously been reached between the parties concerned (removal of two representative samples when the tank-wagon is filled at the premises of the manufacturer). It cannot be applied when it is intended to determine the water content.

2.1 Special case

Method to be applied when no agreement has been made, and it is therefore a question of taking one or more representative samples (when the tank-wagon is emptied at the customer's premises), with a view to determining only the volumetric chlorine content.

3. PRECAUTIONS

The sampling of liquid chlorine involves dangerous and difficult handling operations which can only be carried out by specialist staff using suitable equipment and operating in the open air or in well ventilated places. Direct sampling from the glass containers used for the titration processes (gas burettes in the case of gaseous chlorine, and insulated vessels in the case of liquid chlorine) seems at first sight to be a satisfactory solution, since it prevents any pollution of the sample resulting from intermediate operations.

However, it can only rarely be carried out under sufficiently safe conditions, since the testing laboratory is not usually near the place where the sample is taken. That is why it is recommended to take the representative sample in the small steel bottles which are commonly available, and the use of which entails no deterioration of the product.

4. PRINCIPLE

A check that the equipment used is gas-tight.

The collection of two representative samples in steel bottles during the filling of the delivery tank-wagon.

One bottle is intended for the customer and the other is retained by the manufacturer in case an analysis is required for checking purposes.

5. APPARATUS

The installation, which is shown in diagrammatic form in Figure 4, consists of

- (a) *a liquid chlorine bottle* made of steel, having a usable capacity of from 1 to 10 litres (see example in Fig. 1) and provided with a two-valve head having two dipping tubes (see example in Fig. 2), the shorter tube having a length so calculated as to keep below its lower end a residual volume of 12 to 15 % of the total capacity of the bottle;
- (b) *connection pipes* of copper or steel having a diameter of approximately 10 mm and suitable needle valves of stainless steel.

6. PROCEDURE

6.1 Preliminary operations

Clean the piping and valves of the installation with steam and dry them with dry air.

Clean the chlorine bottle and bottle head in the same way with a jet of steam and dry them with dry air.

Fix the head on the bottle after making a reference mark on the outer wall to indicate the lower level of the shortest tube and weigh the complete assembly.

Check that there is no blockage in the plunger tubes. For this purpose, connect the bottle as indicated in Figure 3 to a vacuum pump and open carefully and progressively both valves. The rapid passage of air observed in the sulphuric acid bubbler indicates the absence of any blockage.

Then check that the valves are functioning correctly by closing them alternately. The passage of air should then cease.

6.2 Sampling liquid chlorine while the tank-wagon is being filled

- 6.2.1 *Connection of the bottle and verification of gas-tightness.* Once the chlorine bottle has been cleaned, weighed and checked, connect it to the installation illustrated in Figure 4, with all the valves closed.

Slightly open the valve A connecting the storage tank containing the liquid chlorine to the tank-wagon.

Open valves 1 and 4 in succession and check the gas-tightness of the bottle connections with a little soapy water (do not use ammonia).

Close valves 1 and 4.

- 6.2.2 *Filling of the bottle.* Successively open valves 2 and 3 and then 1 and 4 in such a way that the liquid chlorine flows in the direction indicated by the arrows in Figure 4.

Regulate the flow by operating valve 1 so as to take two representative samples over the whole period during which the tank-wagon is being filled. Operate in such a way that the flow can still continue for at least 10 minutes after the level has reached the lowest point of the shortest plunger tube, which has been previously marked as described in clause 6.1. Finally, a compressed gaseous phase is thus left in the top part of the bottle, which prevents complete filling of the latter.

When these conditions have been fulfilled, close valves 1, 2, 3 and 4 in succession and immediately open valves 5 and 6.

- 6.2.3 *Disconnection of the bottle.* Allow the chlorine contained in the piping to evaporate. If necessary, heat the pipes with steam.

Then close valves 5 and 6 and disconnect the bottle.

6.3 Checking the filling by weighing the bottle

Wipe away the condensation water on the outer wall and then weigh the bottle.

The mass of the chlorine taken is obtained by the difference

$$m_2 - m_1$$

where

m_1 is the mass, in kilogrammes, of the empty bottle;

m_2 is the mass, in kilogrammes, of the bottle after filling.

This difference should not be greater than the product

$$V \times 1.25 \text{ (to be labeled on each bottle)}$$

where V is the total capacity, in litres, of the bottle (previously measured by weighing with water at 20 °C).

If this value is exceeded, eliminate the excess quantity of the liquid by inclining the head of the bottle downward.

7. SPECIAL CASE

The taking of one or more representative samples, during unloading of a tank-wagon, when there has been no previous agreement between the parties concerned.

7.1 Precautions

As in section 3.

7.2 Principle

A check that the equipment is gas-tight.

Purging of the tank-wagon.

The collection of one or more representative samples in steel bottles when the tank-wagon is being emptied.

7.3 Apparatus

As in section 5, but in this case the installation is diagrammatically illustrated in Figure 5.

7.4 Procedure

7.4.1 Purging of the tank-wagon. Allow approximately 100 kg of liquid chlorine to flow from the tank-wagon towards the chlorine tank, by slightly opening the valve A, and then close it again.

7.4.2 Preliminary operations. As in clause 6.1.

7.4.3 Collection of a sample

7.4.3.1 CONNECTION OF THE BOTTLE AND VERIFICATION OF GAS-TIGHTNESS. Connect the chlorine bottle which has just been cleaned, weighed and checked to the installation as illustrated in Figure 5, with all the valves closed.

Slightly open valve A connecting the tank-wagon to the liquid chlorine tank.

Open valves 4 and 1 in succession and check the gas-tightness of the bottle connections with a little soapy water (do not use ammonia).

Close valves 4 and 1.

7.4.3.2 FILLING THE BOTTLE. Successively open valves 3 and 2 and then 4 and 1 so that the liquid chlorine flows in the direction indicated by the arrows in Figure 5.

Regulate the flow by operating the valve 4.

When the level reaches the point below the shortest plunger tube which has been previously marked, let the liquid chlorine continue to flow for at least 10 minutes. Finally, a compressed gaseous phase thus remains in the upper part of the bottle, which prevents total filling.

When these conditions have been fulfilled, close valves 4, 3, 2 and 1 in succession and immediately open valves 5 and 6.

7.4.3.3 DISCONNECTION OF THE BOTTLE. As in clause 6.2.3.

7.4.4 Checking the filling by weighing the bottle. As in clause 6.3.

8. SAMPLING REPORT

Give the following particulars :

- (a) the reference of the method used;
- (b) any unusual features noted during the sampling;
- (c) any operation not included in this ISO Recommendation or regarded as optional.

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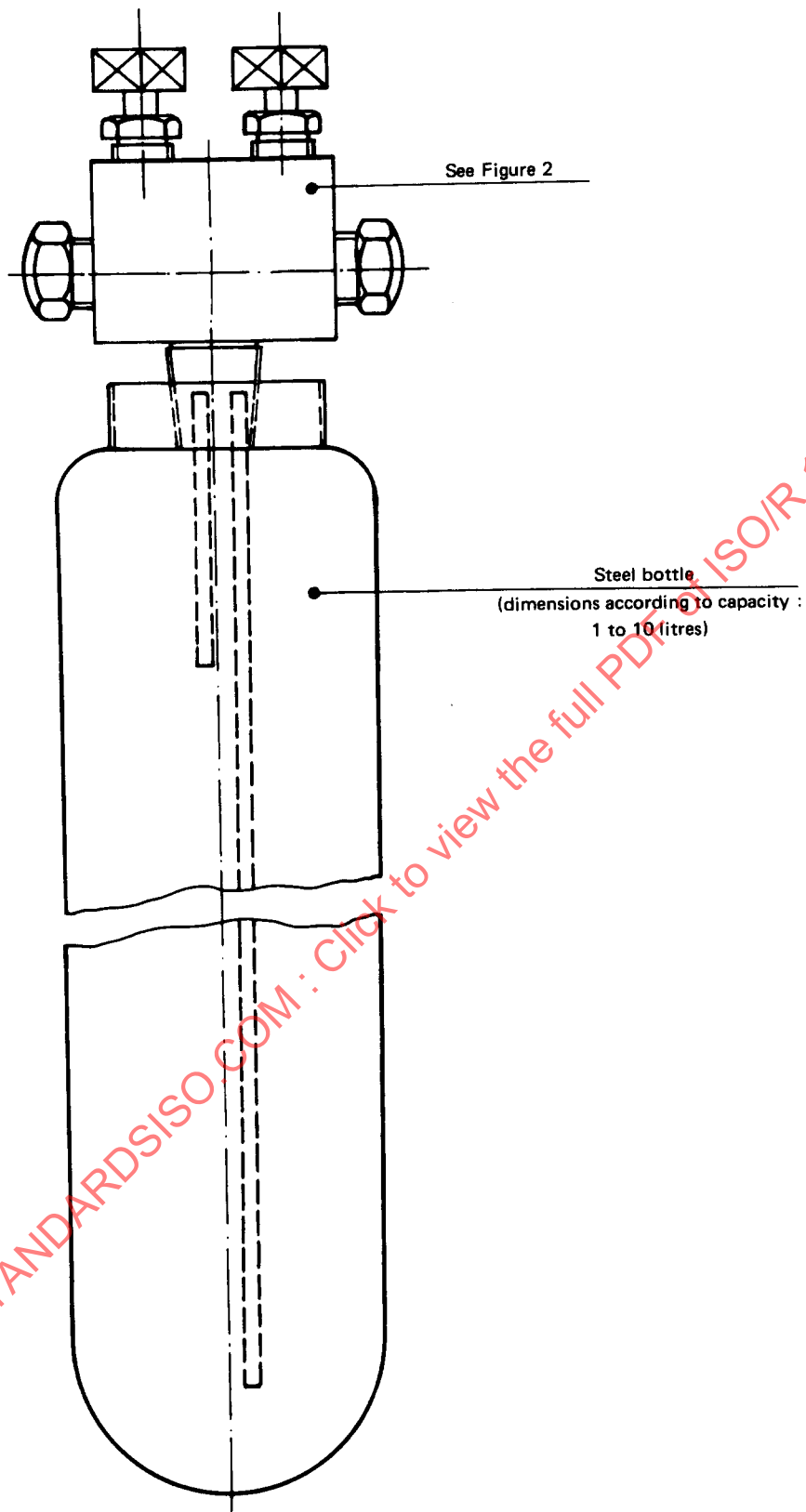


FIG. 1 – Liquid chlorine bottle

Dimensions in millimetres

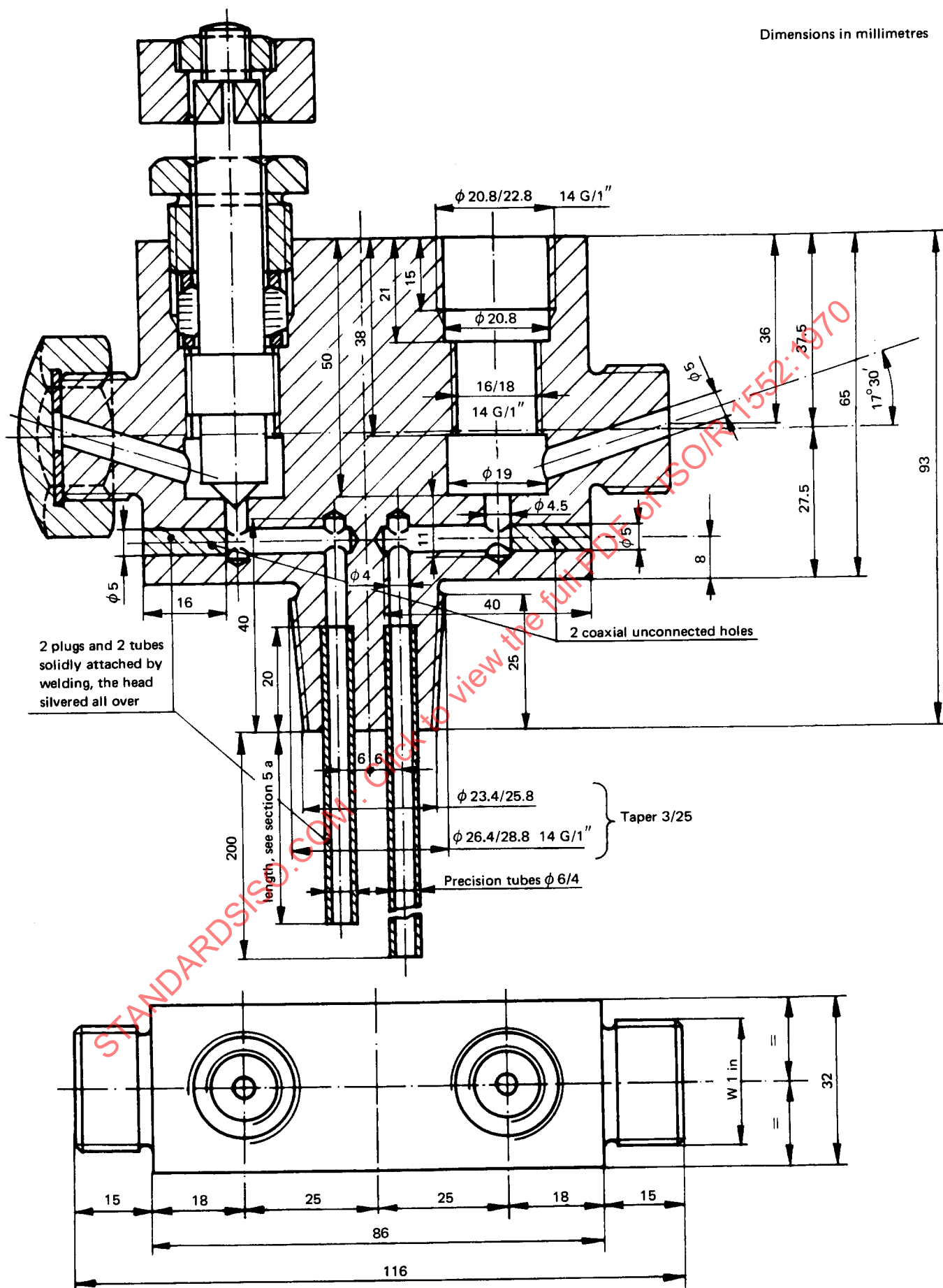


FIG. 2 — Head of liquid chlorine bottle

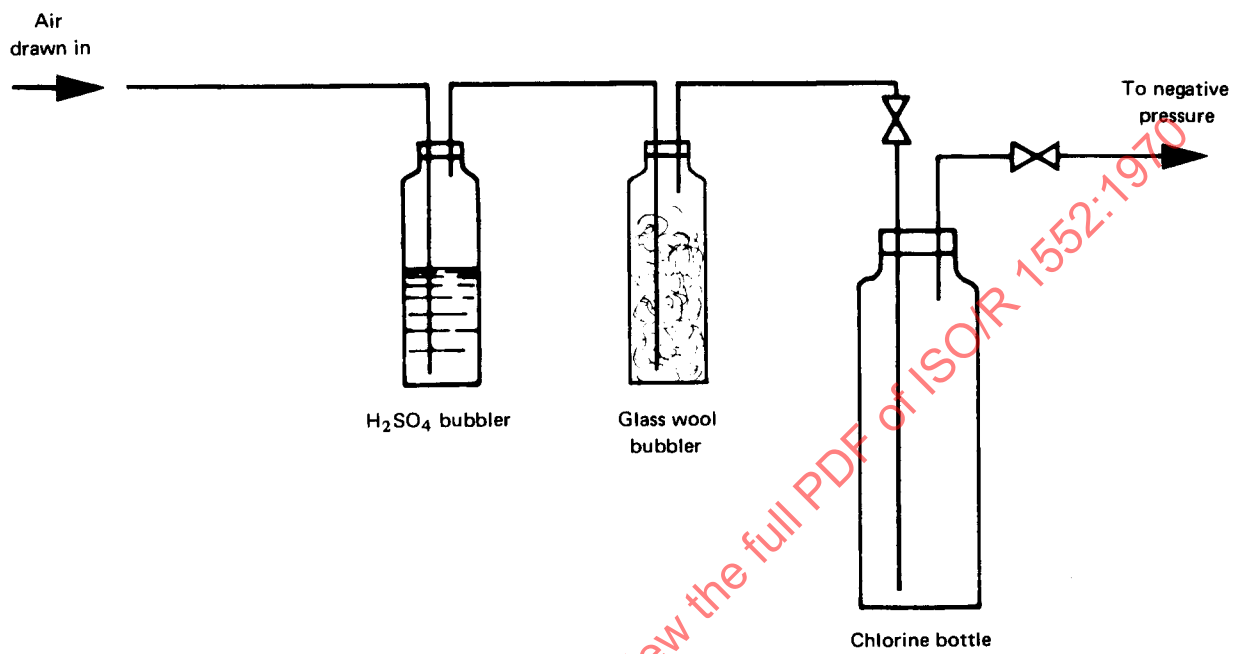


FIG. 3 Checking of chlorine bottle before the removal of the sample – Diagram of assembly