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# INTERNATIONAL STANDARD



# 1552

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION · МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ · ORGANISATION INTERNATIONALE DE NORMALISATION

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## Liquid chlorine for industrial use — Method of sampling (for determining only the volumetric chlorine content)

*Chlore liquide à usage industriel — Méthode d'échantillonnage (en vue uniquement de la détermination du titre volumétrique en chlore)*

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**Descriptors** : liquefied gases, chlorine, sampling, determination of content, volumetric analysis.

## FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47 has reviewed ISO Recommendation R 1552 and found it technically suitable for transformation. International Standard ISO 1552 therefore replaces ISO Recommendation R 1552-1970 to which it is technically identical.

ISO Recommendation R 1552 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1552 into an International Standard.

# Liquid chlorine for industrial use – Method of sampling (for determining only the volumetric chlorine content)

## 1 SCOPE

This International Standard specifies 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 is applicable 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 interested parties (removal of two representative samples during filling of the tank-wagon at the manufacturer's premises). 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 (during emptying of the tank-wagon at the customer's premises), with a view to determining only the volumetric chlorine content.

## 3 WARNING

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. For this reason, it is recommended that the representative sample be taken 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

**5.1 Liquid chlorine bottle**, made of steel, having a usable capacity of from 1 to 10 l (see example in figure 1) and provided with a two-valve head having two dipping tubes (see example in figure 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.

**5.2 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 a jet of steam and dry them with dry air.

Clean the liquid 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 shorter 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 during filling of the tank-wagon

### 6.2.1 Connection of the bottle and verification of gas-tightness.

Once the liquid 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 ammonium hydroxide).

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 of filling of the tank-wagon. Operate in such a way that the flow can still continue for at least 10 min after the level has reached the lowest point of the shorter plunger tube, which has been previously marked as specified in 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 a jet of 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 chlorine taken, expressed in kilograms, is given by the formula :

$$m_1 - m_0$$

where

$m_0$  is the mass, in kilograms, of the empty bottle;

$m_1$  is the mass, in kilograms, of the bottle after filling.

This difference shall be not greater than the product  $V \times 1,25$  (to be labelled 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 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, in the absence of a previous agreement between the interested parties.

### 7.1 Warning

See clause 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 during emptying of the tank-wagon.

### 7.3 Apparatus

See clause 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 liquid chlorine tank, by slightly opening the valve A, and then close it again.

#### 7.4.2 Preliminary operations

See 6.1.

#### 7.4.3 Collection of a sample

##### 7.4.3.1 CONNECTION OF THE BOTTLE AND VERIFICATION OF GAS-TIGHTNESS

Connect the liquid 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 ammonium hydroxide).

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 valve 4.

When the level reaches the point below the shorter plunger tube which has been previously marked as specified in 7.4.2, let the liquid chlorine continue to flow for at least 10 min. 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

See 6.2.3.

#### 7.4.4 Checking the filling by weighing the bottle

See 6.3.

### 8 SAMPLING REPORT

The sampling report shall include 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 International Standard or regarded as optional.

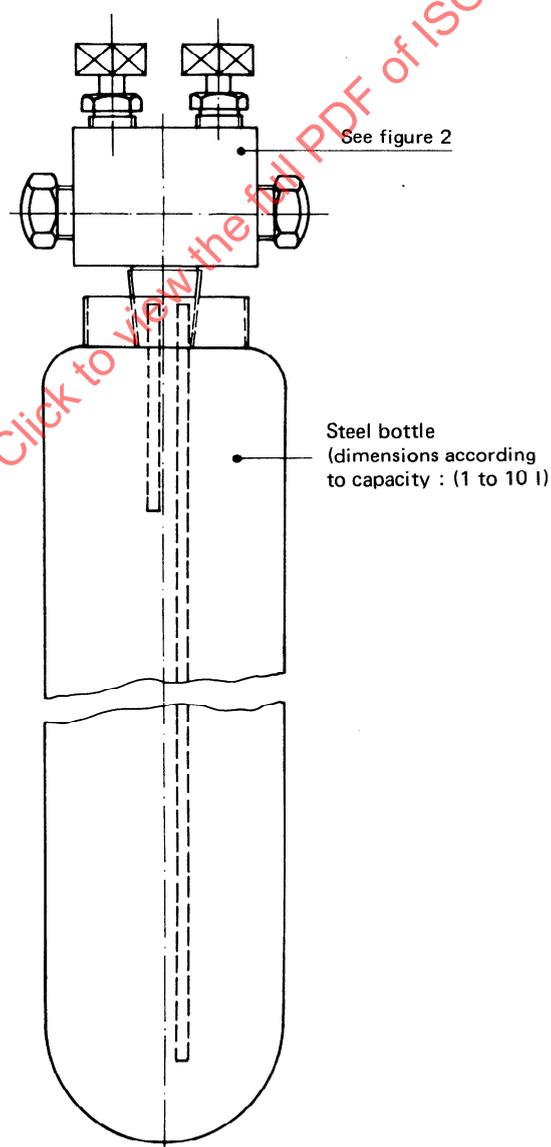


FIGURE 1 – Liquid chlorine bottle

Dimensions in millimetres

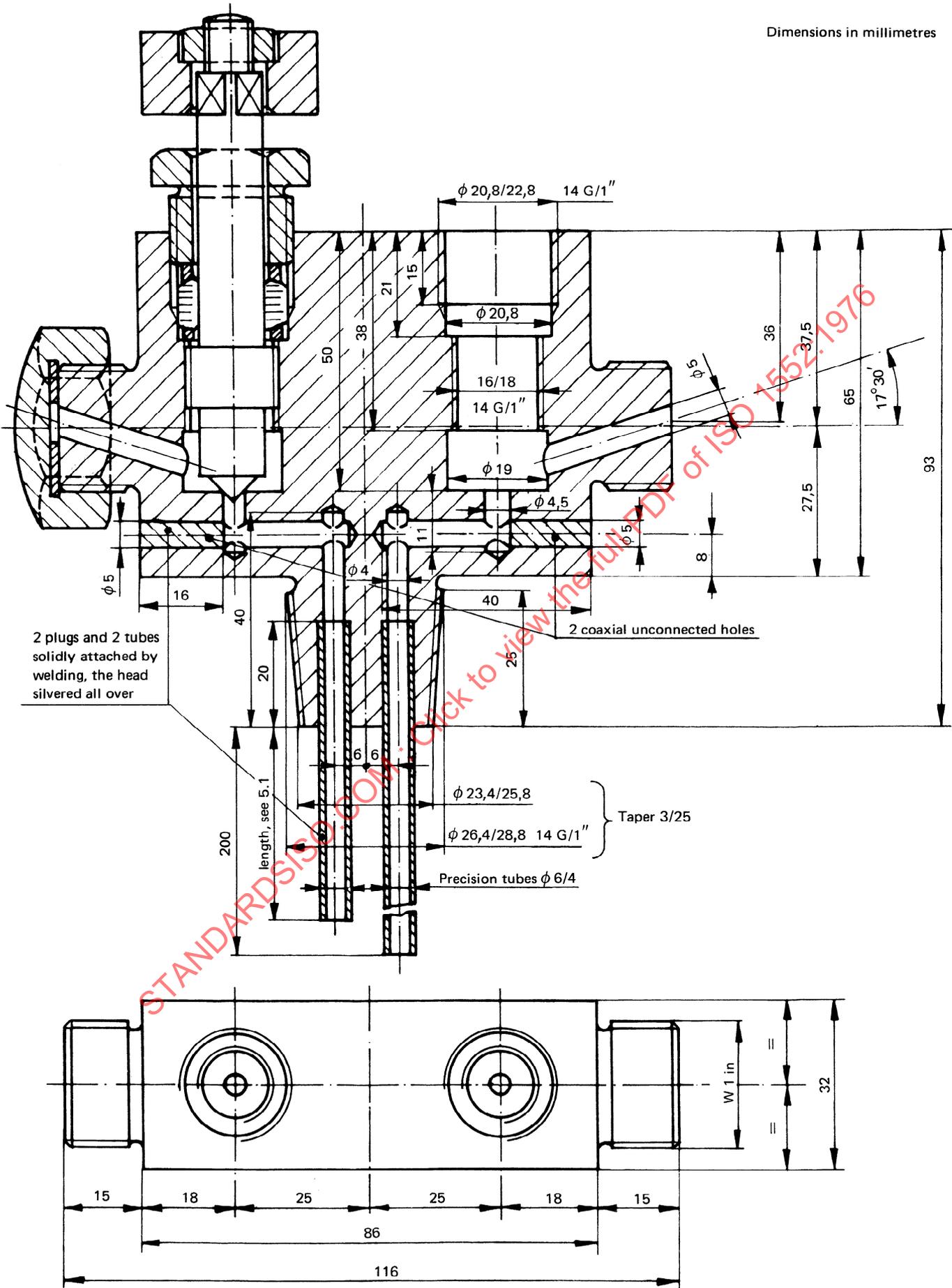


FIGURE 2 – Head of liquid chlorine bottle