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INTERNATIONAL STANDARD ISO 2096

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Glycerols for industrial use — Methods of sampling

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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.

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Austria	India	Thailand
Belgium	Italy	Turkey
Czechoslovakia	Netherlands	United Kingdom
Egypt, Arab Rep. of	New Zealand	U.S.A.
France	Portugal	U.S.S.R.
Germany	South Africa, Rep. of	
Hungary	Switzerland	

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INTERNATIONAL STANDARD ISO 2096-1972 (E)

Glycerols for industrial use — Methods of sampling

1 SCOPE AND FIELD OF APPLICATION

This International Standard describes methods for sampling quantities of glycerol, either crude or refined, for industrial use, in the course of filling (see section 4), or already contained in drums (see section 5) or in transportable or fixed tanks (see section 6).

2 GENERAL PRINCIPLES

2.1 The preparation of a representative sample presents particular difficulties owing to the frequent heterogeneity of the lots. This can lead either to sedimentation of the solids in suspension in the case of crude glycerols, or the stratification in layers of different densities in the course of storage, particularly if the glycerol has frozen after filling into drums or tanks and has then thawed.

2.2 It is consequently recommended to adopt, as far as possible, a procedure of sampling during filling (section 4) or, at least, to carry out the sampling of the lots immediately after this operation, while the product is still homogeneous.

2.3 If glycerol that has been stored for some time is to be sampled, one of the methods described in this International Standard shall be chosen, according to the container in which the product has been stored and the details supplied as to its condition by several samples taken from different locations.

2.4 It is customary to divide the composite sample into at least three portions, each being stoppered and sealed by the sampler. One portion is for the purchaser, one for the vendor and one is kept for an independent analysis in case of dispute.

3 PARTICULAR PRECAUTIONS

3.1 In view of the very hygroscopic nature of glycerol, it is essential that samples shall at all times be protected from humidity and humid air. The containers used for mixing and storage shall be airtight and kept closed between filling and between the taking of various samples. As far as possible, sampling shall be carried out under cover, the containers particularly being protected from rain and all other accidental pollution.

3.2 The laboratory samples shall be poured into flasks of chemically resistant glass closed with ground glass stoppers. In certain cases these flasks may be closed with a screw cap closure having a polyethylene or aluminium liner. The flasks intended for laboratories shall be sealed by the responsible sampler with the aid of sealing wax.

3.3 In the case of samples containing a deposit or suspended material the containers shall be filled to only two-thirds of their capacity in order to aid mixing. Otherwise the containers shall be completely filled.

3.4 All the apparatus and containers shall be clean and dry at the time of use.

4 METHODS OF SAMPLING GLYCEROLS DURING FILLING

4.1 Intermittent sampling method

4.1.1 *Principle*

Successive samplings, at regular intervals, of equal quantities throughout filling. Mixing of these successive samples, homogenization and then taking the laboratory samples.

4.1.2 *Apparatus*

4.1.2.1 Sampling can, with handle, capacity 300 to 500 ml, of brass or, for refined glycerols, of stainless steel.

4.1.2.2 Cylindrical container, of suitable capacity, of the same material as the can (4.1.2.1) or, preferably, of glass and fitted with a lid making it airtight. This container is used for holding and mixing the successive samples taken.

4.1.3 *Procedure*

Determine the number of samples to be taken and the time interval between successive samplings, according to the quantity of product and the rate of flow in filling.

Take the product at the discharge end of the filling pipe by means of the can (4.1.2.1), spacing the samplings as uniformly as possible.

Before each sampling rinse the can (4.1.2.1), after it has been inverted and allowed to drain, with the glycerol to be

sampled. Place the successive samples together in the container (4.1.2.2), keeping it closed between each addition.

Mix the whole sample rapidly with the aid of a stainless steel palette knife or, **preferably**, by rotating the **closed** container on its side, about its axis. Take the laboratory samples and immediately bottle them in quantities of about 500 g, taking the precautions described in 3.2, 3.3 and 3.4.

4.2 Continuous sampling method

4.2.1 *Principle and field of application*

Taking an aliquot part by allowing a constant proportion of the product to flow through a special device fitted to the filling pipe. Mixing and taking the laboratory samples.

This procedure is applicable only to glycerols free from materials in suspension.

4.2.2 *Apparatus*

4.2.2.1 Stainless steel continuous tapping sampler comprising the following parts :

a) tubular element bent at 90°, or bevelled at 45°, inserted and fixed in a vertical part of the pipe with upward flow under pressure of the pump, so that the inner end is in the middle of this pipe and pointing downwards (see Figure 1);

b) flow control tap fixed to the tubular element (see above);

c) flexible discharge tube linking the control tap to the collecting vessel (4.2.2.2), in which it descends to the bottom through an inlet pipe.

4.2.2.2 Cylindrical collecting vessel, of suitable capacity, of stainless steel or glass, which can be closed hermetically and is fitted with an inlet pipe and a ventilation hole so that the air can escape from the vessel as filling proceeds, without risk to the sample of pollution by moisture.

4.2.3 *Procedure*

Choose the setting of the sampler tap (4.2.2.1) and the capacity of the mixing vessel (4.2.2.2) according to the quantity of product to be sampled. It is recommended that a total sample of about 1 kg should be taken per tonne of glycerol subject to a maximum of 50 kg.

Allow the glycerol to flow throughout the whole filling period without altering the setting of the tap.

Mix the whole sample and take the laboratory samples as described in the last paragraph of 4.1.3.

5 METHOD OF SAMPLING GLYCEROLS IN DRUMS

5.1 Field of application

This method is applicable in its general form only to glycerols containing no compact deposit of solids at the bottom of the drums.

5.1.1 *Special case*

Sampling of crude glycerols having a compact deposit of solids at the bottom of the drums (see 5.4.2).

5.2 Principle

Taking one sample per drum, submitted to sampling throughout the whole of its height, with the aid of a sampling tube inserted through the bung hole.

Mixing of the samples, homogenization, and taking the laboratory samples.

It is recommended that no more than 100 drums should be grouped together for each global sample.

5.3 Apparatus

5.3.1 *Sampling tube* (Figure 2), consisting of two cylinders of brass or, for refined glycerols, of stainless steel or of any other chemically resistant material. The inner cylinder fits exactly into the outer and both have alternating longitudinal slots spread over a quarter of their circumference and their complete length. These slots may either coincide or be sealed by rotation of the inner cylinder by means of a handle fitted with a pointer. The pointer indicates the relative positions of the slots on a scale fitted on the outer cylinder.

In the filling position, the slots form two discontinuous lines of openings, which allow the sample to enter the tube at all levels of the drum simultaneously.

The two cylinders are pierced at the base, providing openings which coincide when the pointer is in the emptying position, while the longitudinal slots remain closed.

The length of the sampling tube should be proportional to the depth of the material to be sampled and its effective volume should be about 0.1 % of that of the drum.

5.3.2 *Wiper plug* to fit the bung hole of the drums to be sampled.

5.3.3 *Cylindrical collecting vessel* of the same material as the sampling tube or, preferably, of glass, fitted with a cover forming a hermetic seal, having a capacity of about 1.5 l per tonne of product to be sampled.

5.4 Procedure

5.4.1 *General case*

If the drums are not freshly filled, ensure by means of a flat-ended probe that no solid deposit has formed at the bottom of the drum. If a deposit is present, and as far as

this is still possible, bring this deposit back into suspension by heating the material and mixing it by repeated rotations of the drum while it is lying on its axis.

Introduce the sampling tube (5.3.1), closed, through the wiper plug (5.3.2) to the bottom of the drum. Open the longitudinal slots by placing the pointer at the filling position. Wait until the tube is full, then close the slots and withdraw the apparatus, the exterior of which is cleansed by rubbing against the wiper plug (5.3.2).

NOTE — The time required for filling varies with the viscosity of the product, influenced by the ambient temperature. It should be determined by a preliminary test.

Introduce the filled sampling tube (5.3.1) into the collecting vessel (5.3.3) and place the pointer at the position "emptying". Empty the tube and operate in this way for all the samples in turn, keeping the vessel (5.3.3) closed between each emptying operation. Mix and take the laboratory samples as described in 4.1.3.

5.4.2 *Special case*

If the solids present remain in the form of a compact deposit after the treatment described in 5.4.1, it is not possible to obtain an aggregate representative sample by means of the tubular sampler (5.3.1).

In this case, take separate samples of the liquid phase on the one hand, as described in 5.4.1, and on the other hand, of the solid deposit. In order to sample the latter, empty out the liquid phase, weigh the drum with the deposit of solids, dissolve the latter, if possible, in a known quantity of water and take samples from the resultant solution. In addition, estimate the extent of the deposit from the difference between the mass taken previously and that of the drum only, after completely emptying.

NOTE — Record in the report, if appropriate, the presence of any insoluble material.

6 METHODS OF SAMPLING GLYCEROLS IN TANKS

6.1 General remarks

It is difficult to define one single standardized method applicable to all cases of sampling glycerol in tanks. The form of the latter and the arrangement of the premises will determine the choice among the three following methods:

- taking localized amounts by means of a sampler;
- localized sampling with a weighted bottle;
- continuous sampling.

Localized sampling takes more time but gives more representative samples than those obtained by continuous sampling. If the tank contains a solid deposit, it is not possible to take completely representative samples; the sampling applies only to the liquid phase. However, the thickness of the deposit is estimated, with the aid of a rod sampler or band sampler fitted with a flat weight at the end, and this shall be mentioned in the report.

6.2 Method of localized sampling, with the aid of a sampler

6.2.1 *Principle*

Successive samplings, at the surface of the liquid, at different levels and at the bottom of the tank, of localized quantities representative of the level in question, with the aid for example, of an open-ended cylindrical sampler.

Mixing of these successive samples, homogenization and taking the laboratory samples.

6.2.2 *Apparatus*

6.2.2.1 Open-ended cylindrical sampler (Figure 3), of suitable capacity, made of brass or, for refined glycerols, of stainless steel, consisting of the following parts:

a) a vertical cylindrical shell, open at both ends, the base of which can be closed by operation from another point by means of a set of levers acting on a rubber valve;

b) two lines or small chains provided with regularly spaced guiding marks serving to manoeuvre and control the position of the sampler.

This must be capable of removing a sample above about 10 mm from the bottom of the tank.

6.2.2.2 Collecting vessel, identical to that described in 5.3.3.

6.2.3 *Procedure*

Determine the number of samples and the different levels at which they are to be taken, distributed throughout the height of the tank, depending on its shape and the quantity to be sampled. It is recommended that a sample of 1 kg be taken at a depth corresponding to each successive layer of one tonne of glycerol, subject to a maximum of 50 kg.

The following procedure shall be followed in taking these samples.

Using the line or small chain attached to the cylindrical body of the sampler (6.2.2.1), allow it to descend vertically into the contents of the tank at such a speed as to allow the glycerol to flow through the cylinder from the base upwards during the descent, so that at any moment the contents of the apparatus are exactly representative of the level reached.

At the selected depth, close the valve by pulling firmly on the line or small chain controlling the levers, then withdraw the sampler, using this same line or small chain. Carefully wipe the outer walls of the sampler.

Empty the successive samples into the collecting vessel

(6.2.2.2), keeping it closed between additions. Mix the whole sample and take the laboratory samples as described in the last paragraph of 4.1.3.

6.3 Method of localized sampling, with the aid of a weighted bottle

6.3.1 *Principle*

Identical to that of the method using the sampler (6.2.1), the latter being replaced by a weighted bottle or any other simple device which can be filled at a certain level of the tank.

6.3.2 *Apparatus*

6.3.2.1 Weighted bottle (Figure 4), made of brass or, for refined glycerols, of stainless steel or glass, fitted with a stopper which is removable by operation at another point, the bottle being manoeuvred with the aid of a line, small chain or rod.

6.3.2.2 Collecting vessel, identical to that described in 5.3.3, of the same material as the weighted bottle, or of glass.

6.3.3 *Procedure*

Determine the number of portions and the quantity of sample to be taken as described in 6.2.3 and proceed as follows.

Allow the weighted bottle (6.3.2.1), with the stopper in position, to descend to the selected depth, remove the stopper, wait until the time necessary for filling has elapsed, then withdraw the bottle. Wipe the outer walls carefully.

Take samples also at the bottom of the tank, either in a bottle manoeuvred into a horizontal position by means of a rod, or in a small weighted bottle having a flat bottom, of a height of only a few centimetres and of capacity in proportion to the depth of the layer of sediment. Empty the successive samples into the collecting vessel (6.3.2.2) keeping it closed between additions. Mix the whole sample and take the laboratory samples as described in the last paragraph of 4.1.3.

6.4 Method of continuous sampling during the movement of an open sampling container

6.4.1 *Principle*

Repetitive collection of representative samples of the product by continuous removal using a device which is progressively filled while being moved to different levels of the tank.

Mixture of these quantities and removal of the laboratory samples.

6.4.2 *Apparatus*

6.4.2.1 Weighted bottle, similar to that described in 6.3.2.1.

6.4.2.2 Stopper for continuous sampling (Figure 4), to fit the weighted bottle (6.4.2.1), bored with a hole of such a diameter as to allow the glycerol to enter the container only slowly while it is being moved in the tank.

6.4.2.3 Collecting vessel, identical to that described in 5.3.3, of the same material as the weighted bottle, or of glass.

6.4.3. *Procedure*

Take a number of samples sufficient to make up the quantity required. It is recommended that a total sample of about 1 kg should be taken per tonne of glycerol, subject to a maximum of 50 kg. Allow the weighted bottle (6.4.2.1) fitted with the stopper (6.4.2.2) to descend slowly until it rests on the bottom of the tank, then raise it slowly, at the same uniform rate, so that the bottle is not full on emerging. Wipe the outer walls carefully. Also sample the bottom of the tank, as in 6.3.3, following the operations described therein, in order finally to obtain the laboratory sample.

7 SAMPLING REPORT

In view of the relative freedom allowed to the sampler responsible, as to the choice of procedure to use, and the methods, depending on local circumstances, it is essential that sampling shall form the subject of a report.

This report shall include the following particulars:

a) the reference of the method used;

b) the proportion of the lot constituting the total sample, the number of separate samples taken in making it up and, if appropriate, the mass of solid deposit noted, the presence of insoluble material, etc.;

c) the number of laboratory samples prepared, with an indication of the characteristics defining them (packing, mass, destination, etc.), mentioning the existence, if any, of separate samples of salt deposit with, in this case, an indication of the operations carried out;

d) all special details arising in the course of sampling;

e) all operations not covered by this International Standard or regarded as optional.

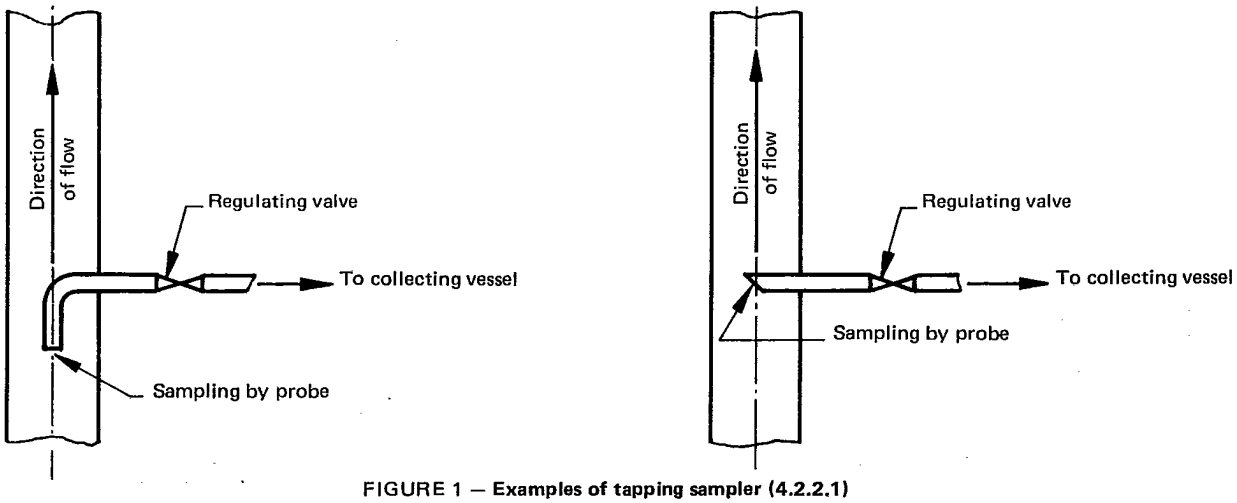

FIGURE 1 — **Examples of tapping sampler (4.2.2.1)**

Dimensions in millimetres

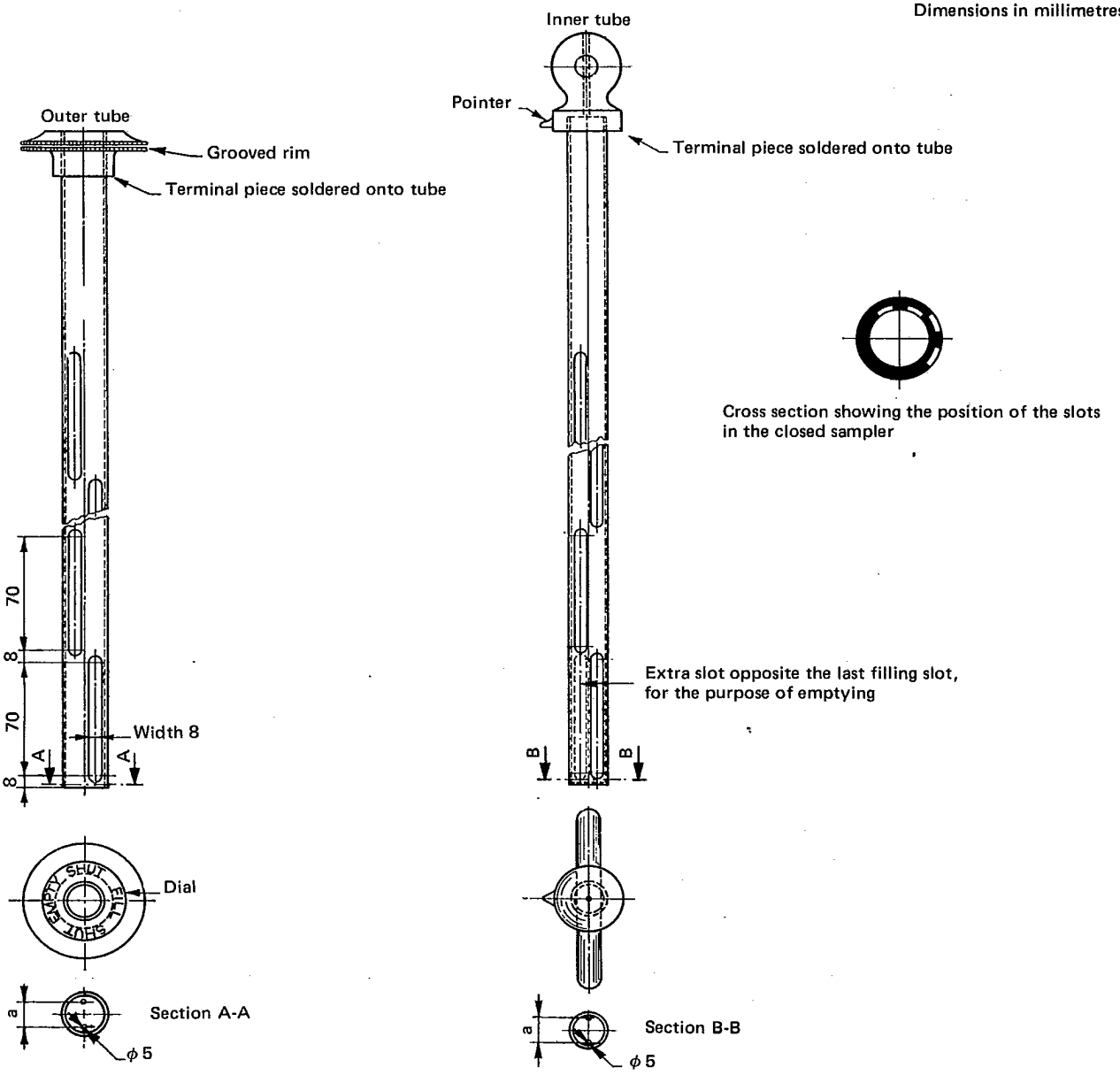

FIGURE 2 — **Example of sampling tube (5.3.1) (Dimensions for guidance only)**

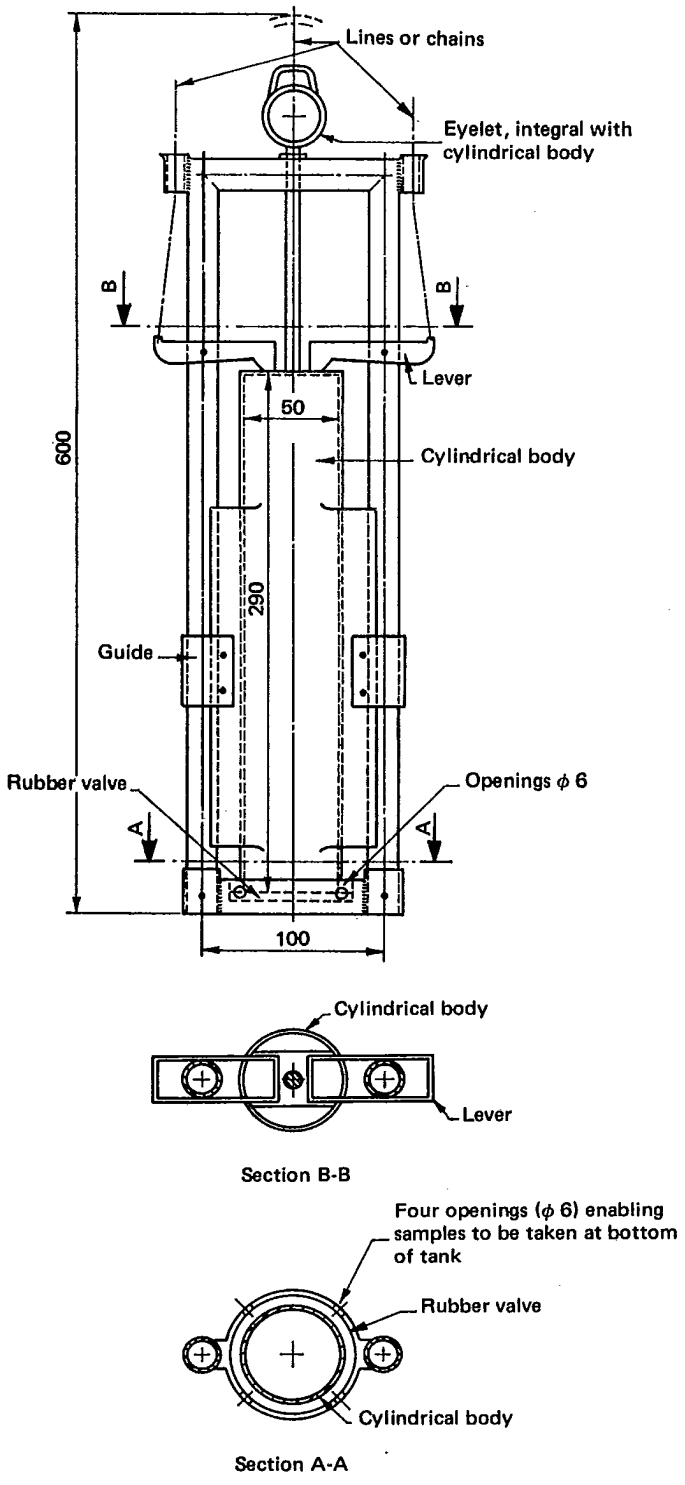

FIGURE 3 — **Example of open-ended cylindrical sampler (6.2.2.1) (Dimensions for guidance only)**

Dimensions in millimetres

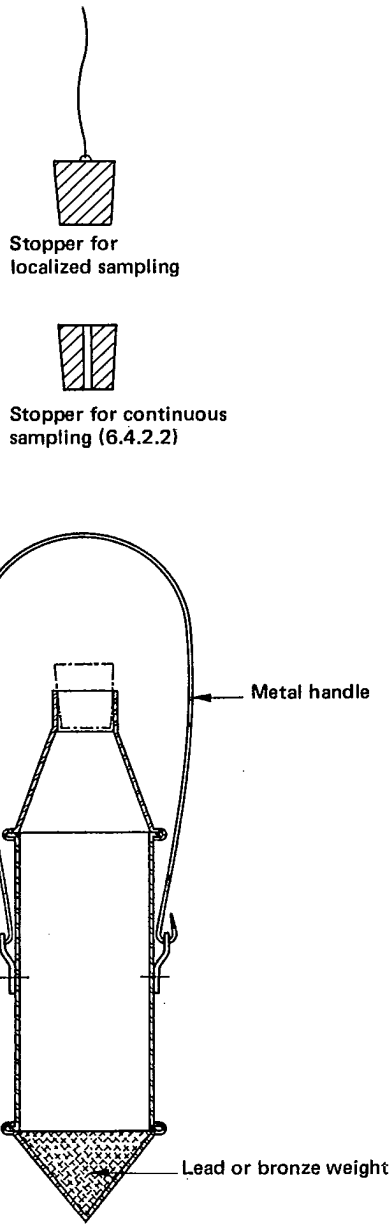

FIGURE 4 — **Example of weighted bottle for sampling from tanks (6.3.2.1)**