BRITISH STANDARD

BS 5598-1:
1978
ISO 2209:1973

Methods of sampling and test for halogenated hydrocarbons —

Part 1: Sampling of liquid products

UDC 661.723:547.411:543.05 – 14

Cooperating organizations

The Chemicals Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

Association of Fatty Acid Distillers
British Tar Industry Association
Chemical Industries Association*
Chemical Society, Analytical Division
Department of Health and Social Security
Department of Industry, Laboratory of the Government Chemist
Fertiliser Manufacturers' Association Ltd.
Hydrocarbon Solvents Association
Ministry of Agriculture, Fisheries and Food
Ministry of Defence*
National Sulphuric Acid Association
Paintmakers Association of Great Britain Ltd.
Royal Institute of Public Health and Hygiene
Soap and Detergent Industry Association
Standardization of Tar Products Tests Committee

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

British Plastics Federation
Fabric Care Research Association
Fire Extinguishing Trades Association
Imperial Chemical Industries Limited
Imperial Smelting Corporation
Institute of Metal Finishing
Institute of Refrigeration
Oil and Colour Chemists Association
Pharmaceutical Society of Great Britain
Royal Institute of Chemistry
Society of Chemical Industry

This British Standard, having been prepared under the direction of the Chemical Standards Committee, was published under the authority of the Executive Board on 30 June 1978

The following BSI references relate to the work on this standard:
Committee reference CIC/4
Draft for comment 74/50328 DC

ISBN 0 580 10237 8

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

National foreword

This Part of this British Standard is identical with ISO 2209 "*Liquid halogenated hydrocarbons for industrial use — Sampling*". It has been prepared under the direction of the Chemicals Standards Committee in order to provide methods of sampling and test for halogenated hydrocarbons.

For some years the United Kingdom has participated in the work of preparing methods of sampling and test applicable to halogenated hydrocarbons for industrial use, organized by Sub-committee 12 "Halogenated hydrocarbons and amines" of Technical Committee 47 "Chemistry" of the International Organization for Standardization (ISO). As international agreement is reached on the methods, it is proposed to publish them as Parts of this British Standard.

Terminology and conventions. The text of the International Standard has been approved as suitable for publication, without deviation, as a British Standard. Some terminology and certain conventions are not identical with those used in British Standards; attention is especially drawn to the following.

Wherever the words "International Standard" appear, referring to this standard, they should be interpreted as "British Standard".

Cross-references. The following International Standards are referred to in the text and for each there is a corresponding British Standard; these are as listed below:

International Standard	Corresponding British Standard
ISO 842	BS 4726 *Methods for sampling raw materials for paints and varnishes* (technically equivalent)
ISO 3165[a]	BS 5309 *Methods for sampling chemical products* Part 1 *Introduction and general principles* (related)

[a] ISO 3165 is no longer at the stage of draft, as indicated in the text; it was published in 1976.

This standard specifies methods of sampling only and should not be used as a specification defining limits of purity. Reference to this standard should indicate that the methods of sampling used comply with the requirements of BS 5598-1.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 6, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope and field of application

This International Standard specifies sampling procedures for liquid halogenated hydrocarbons for industrial use, with the exception of liquefied gases.

NOTE For the sampling of liquid halogenated hydrocarbons intended to be used as raw materials for paints and varnishes, see ISO 842, *Raw materials for paints and varnishes — Sampling*.

The methods are applicable to products not contaminated by an extraneous phase (water, solid deposits, etc). However, accidental pollution is also considered and an initial examination is provided for checking phase homogeneity.

Three cases are considered, namely:

— small containers (cans, drums);

— large containers (cylinders, tanks);

— continuous sampling.

2 Principle

Formation of a blended bulk sample, representing the whole of the batch, by mixing several elementary samples. The number and the method of taking of the elementary samples will depend on the number and capacity of vessels containing the product.

3 Apparatus

Three main types of apparatus can be used for sampling, as appropriate:

3.1 *Usual apparatus*, of steel or glass, with a capacity of 250 to 500 ml (for example, a pipette, as in Figure 1) for sampling from a small vessel.

3.2 *Closeable device*, with a capacity of 500 to 1 000 ml (for example, a steel sampler with a ground closure, as in Figure 2) used mainly for sampling from large containers.

A ballast bottle with a cork stopper can also be used, but not for bottom sampling (for example, a bottle as in Figure 3).

3.3 *Continuous samplers* (for example as in Figure 4), connected to the flowline of the product, such as when delivering the batch to the tank.

4 Procedure

WARNING

A number of hazards may arise in the sampling of volatile solvents. They include flammability and toxicity.[1)]

Flammability

The lower-boiling solvents are flammable and the following precautions are advised:

1) Care must be taken to see that all sampling equipment used for these substances is made of low-energy spark generating material such as beryllium-copper alloys or glass. If an earth connection can be made to large containers, this should be done.

2) All regulations regarding “controlled” or “flammable” areas in which the samples are being drawn must be strictly adhered to.

Toxicity

Vapour from lower-boiling solvents is toxic and precautions should be taken to avoid its inhalation.

It is recommended that two persons should be present when samples are drawn from large containers such as storage tanks, road tanks or rail tanks.

Before sampling from rail tanks it should be ensured that no shunting operations are likely.

In order to allow for the high coefficient of expansion of certain products and to allow for the need ultimately to mix the samples thoroughly to obtain representative test samples the containers should be filled to between about 80 and 90 % of their total capacity.

Contacts with the skin, spillage on clothing, etc. should be avoided as far as possible during sampling. The correct treatment for any harmful material should be known beforehand and the appropriate treatment antidote should be at hand.

4.1 General

All sampling operations shall be carried out carefully and with a due regard for cleanliness.

It is essential in the case of some products to work away from moisture, dust, smoke, etc.

For a given level of sampling, the number of sampling vessels depends on the degree of homogeneity of the product, and not on the tonnage. However, the larger the batch to be sampled, the greater is the need to increase the probability that the blended bulk sample is representative, resulting in a larger number of samples being taken.

4.2 Small containers

Cans, drums and, in general, containers with a capacity less than or equal to 300 litres.

[1)] With regard to safety precautions, see also ISO 3165, *Sampling of chemical products — Safety* (at present at the stage of draft).

4.2.1 *Number and selection of containers for sampling*

In the absence of any agreement to the contrary between the parties, use Table 1 to give reasonable values for the number of vessels to be sampled for various numbers of containers making up the batch.

Table 1

Number of containers in the batch	Number of containers to be sampled
1 to 4	all
5 to 10	5
11 to 20	6
21 to 30	7
31 to 50	8
51 to 75	9
76 to 100	10
101 to 125	11
126 to 150	12
151 to 200	13
201 to 250	14
etc.	

The containers to be sampled shall be chosen at random.

4.2.2 *Taking of elementary samples*

Carry out the sampling using the usual apparatus (**3.1**). Always take the same quantity of liquid.

In order to take a sample, introduce the sampler vertically into the container, down to the bottom. Immersion shall be carried out sufficiently slowly for the level of the liquid inside the sampler to be near the level in the container. Close the sampler with a finger, then withdraw it from the container, and allow the quantity thus sampled to run into a completely clean and dry flask.

NOTE If sampling has to be carried out at a certain level, for example for checking homogeneity (see **4.2.3**), introduce the sampler, *closed at the top*, down to the level to be examined, open and then, after the liquid has been introduced, re-close and withdraw from the container.

4.2.3 *Initial examination for assessing phase homogeneity in the container*

If confirmation is required that the contents of the vessels are homogeneous, or can be made homogeneous readily by stirring, an initial visual examination is recommended on a number of elementary samples.

These samples shall be taken from a certain number of containers for sampling. (See Table 1.)

Take three samples from each of the containers selected: one at the *top*, another at the *middle*, and a third as near as possible to the *bottom*, in accordance with the instructions in **4.2.2**.

First examine the appearance of the three samples (homogeneous phase, suspended particles, etc.), then mix them and stir vigorously. Leave to stand and examine the appearance of the mixture.

Three cases are possible according to the observations:

a) the product appears *homogeneous* in each container examined; continue as indicated in **4.2.5**;

b) the product appears slightly heterogeneous but *can be made homogeneous* by stirring (fine suspended particles, for example); continue as indicated in **4.2.4**;

c) the product *cannot be made homogeneous* by stirring (layer of water or solid deposit which cannot be dispersed, for example); continue the initial examination on other containers, in order to reach a closer assessment of the phase homogeneity in the container.

In each of the containers taken for the initial examination, separate the two phases present as well as possible, in order to determine approximately the percentage of extraneous phase.

Carry out sampling as indicated in **4.2.5**, but take care to sample only the halogenated hydrocarbon.

4.2.4 *Homogenisation*

If it is first necessary to make the contents of the containers to be sampled homogeneous [**4.2.3** b)], stir the contents of each of the containers mechanically for several minutes, using a stirrer.

Stirring shall be more vigorous the less homogeneous is the product.

4.2.5 *Sampling proper — Blended bulk sample and laboratory sample*

Take the elementary samples as described in **4.2.2**, taking samples from the container to be sampled.

NOTE If the product is homogeneous, the samples taken in the course of the initial examination can be used if less than one third of the total volume is taken.

Pour all the elementary samples into a clean and dry container, and stir vigorously in order to make homogeneous. The *blended bulk sample* is thus obtained.

If this sample is too large, take a suitable volume, after stirring if necessary. A *laboratory sample* is thus obtained.

In all cases the sample shall be kept in a clean, dry, perfectly stoppered glass flask away from light.

NOTE If a closer knowledge is required of the batch to be sampled, it may be advantageous to prepare several partial samples, obtained by blending several elementary samples, and not a single blended bulk sample which will provide only one test result corresponding to the average of the batch. Each partial sample shall be the subject of a test, and several results will thus be obtained providing a knowledge not only of the mean value of the batch, but also of the deviations from this mean value.

4.3 Large containers (capacity over 300 litres)

Large drums, cylinders, tanks, fixed or transportable.

4.3.1 ***Number and choice of containers for sampling***

Follow exactly the instructions given in **4.2.1**.

4.3.2 ***Taking of elementary samples***

Use a closeable device (**3.2**) or preferably a sampling tube. Always take the same amount of liquid, between 500 and 1 000 ml.

In order to take a sample, lower the closed device into the liquid down to the necessary depth (see **4.3.4**). Open the device to allow it to fill and then reclose, in the case of the sampling tube, and raise to the surface.

Transfer the sample thus obtained into a clean, dry flask.

Continuous sampling can also be carried out when transferring the liquid (see **4.3.5**).

4.3.3 ***Initial examination for assessing phase homogeneity in the container***

If confirmation is required that the contents of the containers are homogeneous, an initial visual examination of a number of elementary samples is recommended. These samples shall be taken from a certain number (see Table 1) of containers for sampling.

For containers with a capacity less than or equal to 1 000 litres proceed as for small containers (see **4.2.3**).

For larger containers, take one or, if possible, two series of samples along one or two vertical lines, selected as a function of the form and location of the openings (vertical lines located near the two ends of the tank for example).

Take three samples, one at the top, another at the middle and a third as near as possible to the bottom.

Take the samples as described in **4.3.2** and place them in different containers.

Examine the appearance of all the elementary samples taken from the same container (homogeneous phase, suspended particles, etc.).

Two cases are possible according to the observations:

a) the product appears homogeneous (but perhaps turbid) for each container examined; continue as described in **4.3.4**;

b) the product is not homogeneous (layer of water or solid deposit, for example); continue the initial examination on other containers, in order to obtain a closer assessment of the phase homogeneity in the container.

Try to determine the approximate percentage and nature of the extraneous phase and indicate this in the sampling test report.

Carry out the sampling as described in **4.3.4** but take care to sample only the halogenated hydrocarbon, or better, carry out continuous sampling when transferring the liquid (see **4.3.5**).

4.3.4 ***Sampling proper — Blended bulk sample and laboratory sample***

Take the elementary samples on each container to be sampled and proceed as described in **4.3.2** and, in addition, as follows:

4.3.4.1 *Parallelepiped or vertical cylindrical tanks*

Carry out sampling at depths, measured from the bottom of the tank, of 5 h/6, h/2 and h/6, h being the depth of the liquid.

Depth of the liquid as a percentage of the diameter	**Sampling level as a percentage of the diameter, measured from the bottom**			**Proportions of different samples to be mixed, to obtain a sample representative of the container**		
	Upper sample	Middle sample	Lower sample	Upper sample	Middle sample	Lower sample
90	75	50	20	3	4	3
80	70	50	20	2	5	3
70		50	20		6	4
60		50	20		5	5
50		40	20		4	6
40			20			10
30			15			10
20			10			10
10			5			10

Mix the three elementary samples of equal volume. A *sample representative of the container* is thus obtained.

4.3.4.2 *Horizontal cylindrical containers*

Take the elementary samples on the containers as indicated in Table 2, as a function of the depth of the liquid.

Mix the elementary samples in the proportions indicated in Table 2, to obtain a *sample representative of the container*.

Whatever the form of the container, mix all the samples representative of the containers, in volumes approximately proportional to the content of each of the containers.

Stir vigorously in order to make homogeneous. The *blended bulk sample* is thus obtained.

If this sample is too large, take a suitable volume, after stirring if necessary. The *laboratory sample* is thus obtained.

In all cases, the sample shall be kept in a clean, dry, perfectly stoppered glass flask away from the light.

NOTE If a closer knowledge is required of the contents of each container, it is possible to obtain this from the representative samples of the containers by testing these separately.

4.3.5 ***Continuous sampling***

There may be some advantage in carrying out continuous sampling while transferring from one tank to another.

For this purpose use a continuous sampling device (**3.3**).

The speed of the liquid flowing through the cock of the sampler must be the same as that of the principal stream (isokinetic sampling) *throughout the time of the transfer*.

The quantity of liquid thus sampled shall be between 20 and 200 litres and the capacity of the sampling container shall be selected accordingly.

Note, however, that, in the case of a product containing an extraneous phase (water, suspended particles, etc.), it is essential that the sampler does not have a segregating effect.

A sample is thus obtained directly which, after stirring, comprises the representative sample of the container or even the blended bulk sample from which the laboratory sample can be drawn.

5 Sampling report

The sampling report shall include the following particulars:

a) all normal commercial information (name of the product, supplier, place and date of sampling, number and specifications of the vessels, tonnage, etc.);

b) type of sampling carried out (by container or continuous);

c) number of containers sampled;

d) number of elementary samples per container sampled;

e) number and quality of samples prepared (blended bulk samples, laboratory sample, representative sample of the container);

f) any special information, such as: heterogeneous product, turbid product, presence of an aqueous phase, sludge and, if possible, a quantitative assessment.

Dimensions in millimetres

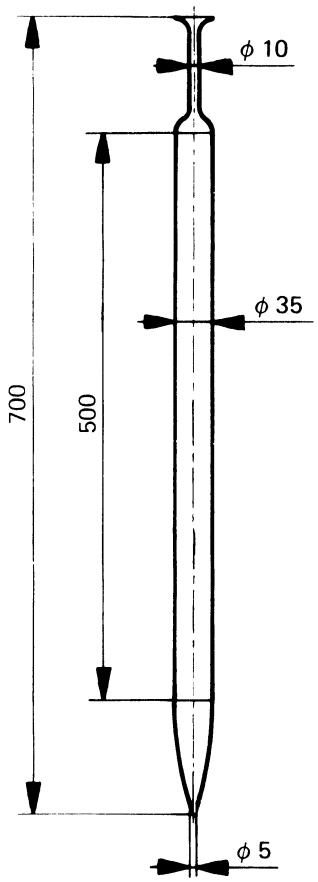

Figure 1 — Pipette

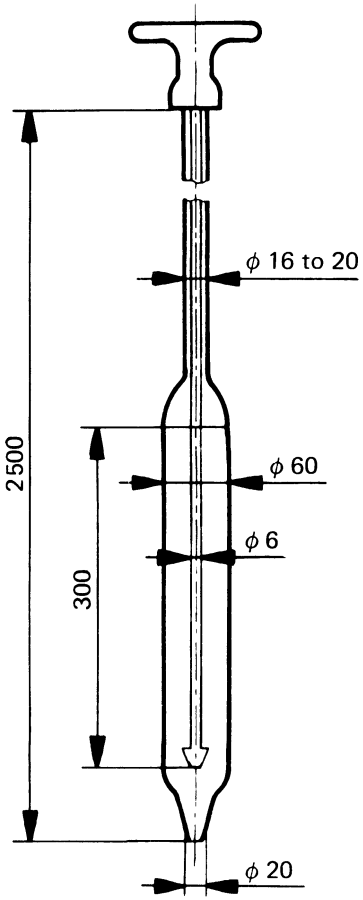

Figure 2 — Steel sampler with ground closure

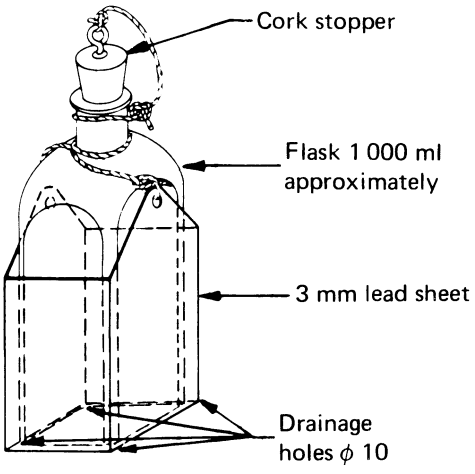

Figure 3 — Bottle

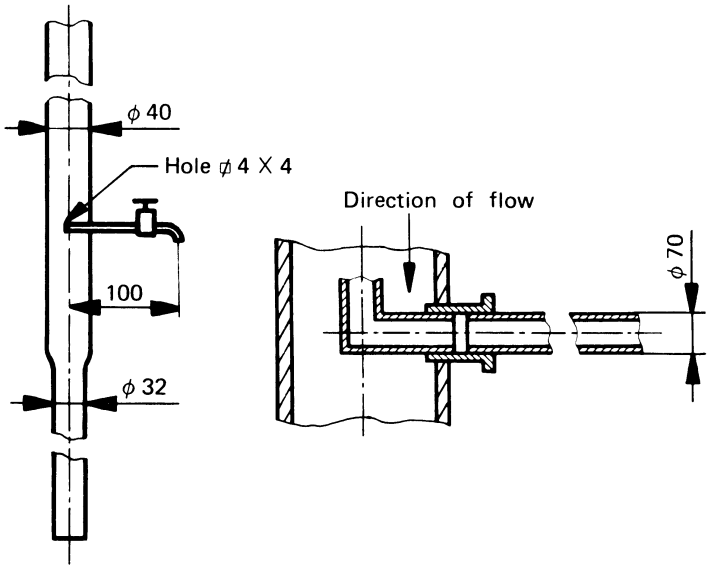

Figure 4 — Continuous sampler

Figure 1 to Figure 4 — Examples of sampling devices

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Publications referred to

See national foreword

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