

Methods for

Sampling chemical products —

Part 3: Sampling of liquids

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Co-operating organizations

The Technical Committee which has been working under the supervision of the Chemical Divisional Council, and which is responsible for the preparation of this British Standard, consists of representatives from the following Government departments and scientific and industrial organizations:

- British Colour Makers' Association
- British Tar Industry Association*
- Chemical Industries Association*
- Department of Industry, Laboratory of the Government Chemist*
- Fertiliser Manufacturers' Association
- Institute of Petroleum*
- Institute of Trading Standards Administration*
- Institution of Gas Engineers*
- Ministry of Defence*
- Society for Analytical Chemistry
- Standardization of Tar Products Tests Committee
- United Kingdom Atomic Energy Authority

The Government departments and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the sub-committee entrusted with the preparation of this Part of this British Standard:

Individual experts

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Contents

	Page
Co-operating organizations	Inside front cover
Foreword	iii
Section 1. General	
1 Scope	1
2 References	1
3 General considerations	1
Section 2. Liquids normally mobile at ambient temperatures	
4 Sampling from a drum or a cask	3
5 Sampling from a small container	4
6 Sampling from a tank having a uniform horizontal cross-section throughout its depth	4
7 Sampling from a horizontal cylindrical tank	6
8 Sampling from a tank of irregular shape	8
9 Sampling from a flexible walled container	8
10 Sampling from a pipeline	8
11 Sampling from a channel	11
12 Sampling automatically	13
Section 3. Liquids and solids rendered mobile	
13 Introduction	13
14 General precautions	14
15 Sampling from a container	14
16 Sampling from a pipeline	14
Section 4. Viscous liquids	
17 Introduction	14
18 Methods of sampling	14
Section 5. Multi-phase liquids (including slurries)	
19 Introduction	20
20 Methods of sampling	20
Section 6. Liquefied gases	
21 Introduction	20
22 General precautions	20
23 Gases liquefied by pressure at ambient temperatures	21
24 Gases liquefied by low temperature	22
Section 7. Liquids requiring special handling	
25 Introduction	26
26 Liquids that react with the environment	26
27 Reactive or unstable liquids	31
28 Flammable liquids	31
29 Methods	31
Section 8. Reduction of sample quantity and preparation of laboratory sample	
30 Introduction	35
31 Mobile liquids	35
32 Liquids and solids rendered mobile	35
33 Viscous liquids	35
34 Multi-phase liquids	35
35 Liquids requiring special handling	35

	Page
Appendix A Sampling apparatus	36
Appendix B Methods of mixing	37
Appendix C Static electricity	45
Figure 1 — Sampling scheme for a full cylindrical tank and a partially filled elliptical tank	9
Figure 2 — Typical bypass sampling apparatus	10
Figure 3 — Pipeline probes	12
Figure 4 — Typical continuous channel sampling apparatus	15
Figure 5 — Typical intermittent channel sampling apparatus	16
Figure 6 — Typical automatic sampling apparatus	17
Figure 7 — Typical water cooled dip rod	18
Figure 8 — Typical piston sampling apparatus	19
Figure 9 — Arrangement for sampling gas liquefied by pressure at ambient temperature	22
Figure 10 — Sampling bombs	23
Figure 11 — Typical sampling point for installation in transfer line	27
Figure 12 — Typical sampling point installed in storage tank	28
Figure 13 — Typical metal Dewar apparatus	29
Figure 14 — Sampling apparatus for gases liquefied by low temperature	30
Figure 15 — Typical pressure sampling apparatus	32
Figure 16 — Two types of typical vacuum sampling apparatus	33
Figure 17 — Surface sampling ladle	34
Figure 18 — Typical open sampling tube	37
Figure 19 — Typical bottom-valved sampling tube	38
Figure 20 — Typical weighted sampling can	39
Figure 21 — Typical sampling cage	40
Figure 22 — Typical bottom sampling apparatus	41
Figure 23 — Typical mechanical mixer with fixed links	42
Figure 24 — Typical mechanical mixer	43
Figure 25 — Typical hand mixers	44
Publications referred to	Inside back cover

Foreword

This British Standard has been prepared under the authority of the Chemical Divisional Council following a conference convened by the British Standards Institution at which agreement was reached on the importance of standardizing techniques and apparatus for obtaining samples.

The other Parts of this British Standard are as follows:

- *Part 1: Introduction and general principles;*
- *Part 2: Sampling of gases;*
- *Part 4: Sampling of solids;*
- *Part 5: Probability theory applied to sampling (under consideration).*

The Parts of this British Standard are intended to provide a basis for specifying methods of sampling in other British Standards, and also to act as a guide to manufacturers and users of materials not covered by British Standards.

Therefore this standard is not intended to be used as a working instruction for the person who actually takes the sample. It is essential that users of Part 3 shall first read Part 1 in which the principles of sampling are discussed so that they will be competent to develop a detailed procedure for sampling any quantity of a liquid chemical.

Terms used in the separate Parts are defined in section 1 of Part 1:1976. General safety precautions to be followed by the person taking the sample are dealt with in section 6 of Part 1:1976. The sampling of liquids that require special handling is dealt with in section seven of this Part.

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 to iv, pages 1 to 46, 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.

Section 1. General

1 Scope

This Part of this British Standard specifies methods for the sampling of chemical products in the form of mobile liquids, liquids and solids rendered mobile, viscous liquids, multi-phase liquids (including slurries) and liquefied gases.

It does not cover liquids as contaminants of gases (for which see Part 2) or solids fluidized by gas or by liquid (for which see Part 4).

2 References

The titles of the British Standards referred to in this standard are listed on the inside back cover.

3 General considerations

3.1 Variability

3.1.1 Sampling from a single container. It cannot be assumed that variability does not exist in liquids. Layers may be formed because of differences in temperature or viscosity, or the liquid may contain more than one phase of uneven distribution. Variability of a liquid in a container can be reduced by thorough mixing and the effect of any residual variability can also be reduced by the sampling method.

3.1.2 Sampling from more than one container. The methods that follow apply to the sampling of a single container. If the material under consideration is in more than one container a decision should be taken, dependent on the variability between the containers, as to the number of containers to be sampled. Reference should be made to Part 1 of this standard for details of the other factors that affect the decision.

Examine the containers for any obvious difference between them, e.g. different batch numbers or order of filling, as indicated by a running number etc.

Examine the contents of the selected containers for any obvious signs of variability between them, e.g. differences in colour, etc. Consider the history of the material, i.e. how it was made, how it was handled, etc. Confirm any apparent or suspected variability by testing spot samples from the appropriate containers.

For a liquid that is being regularly handled it is probably only necessary to investigate the nature of the variability of the first few consignments.

Thereafter a brief inspection of each consignment to detect anything abnormal should suffice. It also helps if it is known when any changes are made in production or handling methods. A check can then be carried out to confirm that the nature of the variability has not altered.

3.2 Mixing the liquid

3.2.1 Reference should be made to Appendix B which describes the methods that may be employed for mixing liquids. It is recommended that the effectiveness of a given method of mixing should be assessed as outlined in Appendix B before being used on a particular type of container or with a particular product.

3.2.2 The sampling of the contents of drums and other small packages which can be easily agitated or stirred is based on the assumption that the contents can be rendered uniform in this way. The method given for obtaining a representative sample is designed to reduce the effect of any residual variability after mixing.

3.2.3 Transport tanks and some large storage containers are not fitted with mixing devices and special schemes should be used for sampling the contents. These are based on the principles given in Part 1 of this standard. Any variability known to be present or detected in the liquid by the normal sampling procedure should be taken into account. If necessary, additional samples shall be drawn for further testing.

3.2.4 The use of mixers on large tanks, when these are fitted, or, where it is practicable, circulation of the contents by pumping from and returning to the tank through submerged jets, will improve the precision of sampling. However, no change in the methods of sampling such containers should, in general, be made unless the sampling precision is improved since it is often difficult to ensure complete homogeneity even after prolonged circulation of the liquid or operation of a mixing device. The degree of variability will dictate the detailed sampling procedure.

3.3 Multi-phase liquids

Multi-phase liquids that can be made homogeneous satisfactorily by mixing and that do not separate quickly again may be sampled as single-phase liquids.

If the phases are not easily mixed it is often preferable not to mix but to sample each phase separately. In such cases the quantity of each phase present should be measured using standard procedures. Otherwise the material is sampled as described in section 5.

3.4 Preliminary examination

3.4.1 General. In the sampling of liquids, advantage may be taken of the tendency of some contaminants to sink or float, by making a preliminary examination to verify the identity of the material under examination and to detect any visible abnormalities. Such preliminary examination should be performed before mixing is carried out. If the contents are already mixed a representative sample should be taken first and the contents of the container should then be allowed to stand undisturbed to permit detection of visible abnormalities. **Any abnormalities noted during preliminary examination should be reported.**

3.4.2 Suspect consignment. A consignment shall be considered suspect if any of the following abnormalities is noted:

- a) a container is damaged or defective in a way that may have caused a change of composition of the contents;
- b) the presence of an old label, doubtful markings, absence of seals, incorrect masses or variation in container type;
- c) evidence of an unexpected lack of uniformity or of deterioration of the contents within a container;
- d) unusual variations between the contents of containers in the consignment.

Samples drawn in any of these circumstances should be fully reported upon and should not be regarded as acceptable without mutual agreement between the parties concerned.

3.5 Grouping of liquids. Liquids are grouped as follows and for each group the appropriate sampling procedure is described in subsequent sections.

- a) *Mobile liquids.* Materials normally mobile at ambient temperatures including volatile liquids and emulsions.
- b) *Liquids and solids rendered mobile.* Materials in this category are those that can be rendered mobile by heat without deterioration, or have been converted into mobile liquids for ease of handling. They include viscous liquids, partly solidified liquids and liquefiable solids, which may have been brought into these physical states by low ambient temperatures.
- c) *Viscous liquids.* Viscous materials that cannot be rendered mobile.

d) *Multi-phase liquids.* Materials in this category are liquids containing separate solid or liquid phases, the whole of which is under consideration. The separate phase may be suspended or may settle quickly (see 3.3).

e) *Liquefied gases.* Gases in the liquid phase that are sampled as liquids. These are normally held under pressure at ambient temperature, or at low temperature and at atmospheric or near-atmospheric pressure.

f) *Liquids requiring special handling.* These are dealt with separately in section 7. **For these materials the need to adhere strictly to safety precautions is stressed.**

3.6 General precautions. It cannot be over-emphasized that the most careful work in the laboratory or in quantity measurement may be rendered useless if care is not taken when drawing the samples upon which such work is based. It is essential that a sample shall not include material other than that to be sampled and shall not be altered, e.g. by evaporation or by oxidation, in the process of sampling. It is recommended that the person responsible for sampling makes himself familiar with the theory of sampling outlined in Part 1 of this standard as well as with the detailed procedures described in this Part.

3.6.1 It is essential that nothing in the sampling procedure should lead to contamination of the sample or of the material being sampled.

3.6.2 The sampling apparatus including operating cords or other ancillary equipment and sample containers should be free from any contaminating substance.

3.6.3 The operator engaged in sampling should have clean hands. Clean gloves may be worn but only when essential to protect the operator, or to protect the sample from contamination.

3.6.4 During sampling operations the material being sampled should be protected from the effects of wind and weather, and the sample containers shall be closed immediately the sample has been taken.

3.6.5 Normally no sample container shall ever be completely filled with liquid and sufficient ullage shall be allowed for expansion through any expected temperature changes, except when complete exclusion of the gaseous phase is required (e.g. in samples for biological oxygen demand tests). For those liquids where variations in the ullage allowed may affect the results of tests, e.g. tests of vapour pressure, the ullage provided shall be fixed.

3.6.6 Neither the material of which the sampling equipment and sample containers are made nor the product being sampled should adversely affect the other.

3.6.7 Samples of materials that may be affected by light should be stored in a dark place; the use of amber or dark glass bottles may be required (see **26.4**). Those affected by change of temperature should be stored under appropriate temperature conditions (see **26.5**). Containers holding volatile materials, the composition of which may be affected by loss of lighter components, may need their containers to be stored upside down, in which case periodical examination should be made for leakage.

3.6.8 When the sampling procedure requires separate samples to be drawn from varying levels in a tank or bulk container, the samples shall be drawn in order from the surface downwards so that each sample is obtained from previously undisturbed liquid.

Section 2. Liquids normally mobile at ambient temperatures

4 Sampling from a drum or a cask

4.1 Introduction. There are two purposes in sampling from a drum or a cask. The first, a preliminary examination, is to detect the presence of, or to sample, undissolved matter which normally will be found, after allowing to stand undisturbed, at the surface or on the bottom. The second is to obtain a representative sample. This order may be reversed if the contents are known to be mixed at the time of sampling.

4.2 Preliminary examination

4.2.1 General. Examine the exterior of the drum or cask to be sampled for damage, corrosion and leakage, and check the identification marks. If sealed, inspect the seal on each bung to ensure that it is intact. Record any abnormalities noted (see **3.4.2**).

Place the drum on its side with the bung hole uppermost, or if the drum only has an end bung stand it upright. If the drum has been disturbed allow time, known or found to be required, for any contaminants in the drum to separate. Clean round the bung, remove it with care and place it with the inner side uppermost beside the bung hole.

If the nature or quantity of any contaminant found by the following procedures departs from the normal the consignment should be considered suspect (see **3.4.2**).

Examine the surface of the liquid. If the presence of a surface contaminant is suspected take a surface sample, if not proceed with the examination by taking a bottom sample.

4.2.2 Surface sample. Remove a sample by using the ladle described in **A.1**. Lower the ladle into the drum until the upper points of the serrated edge are just above the surface of the liquid so that the surface layer runs into it. **Remove the ladle before it fills completely** and allow any liquid adhering to the ladle to drain off.

Examine the contents of the ladle for the presence of any contaminant, if necessary transferring the sample to a suitable transparent container for examination.

Record the probable nature and approximate quantity of any contaminant. If a sample of any contaminant is required transfer the contents of the ladle to a sample container and repeat the procedure until sufficient sample is obtained. If a sample is not required return the contents of the ladle to the drum.

4.2.3 Bottom sample. Choose an open sampling tube (see **A.2**) to suit the viscosity of the liquid to be sampled, close it and lower it to the bottom of the drum. Open the tube and move it rapidly so that the bottom of the tube traverses the lower inside surface of the drum before the tube is filled. Close the tube, withdraw it from the container and allow any liquid adhering to the outside of the tube to drain off. Alternatively, use a bottom-valved sampling tube (see **A.3**) in a similar manner, lowering it with the valve closed.

Examine the contents of the tube for the presence of any contaminant. If the tube is opaque run the liquid out into a suitable transparent container for examination.

Record the probable nature and approximate quantity of any contaminant. If a sample of any contaminant is required, transfer the contents of the sampling tube to a sample container and repeat the procedure until sufficient sample is obtained. If a sample is not required return the contents of the tube to the drum.

NOTE If any contaminant or discontinuous phase present has a density greater than that of the liquid being sampled, it may be advantageous to tilt the drum on its bottom chine on the opposite side to the bung hole and insert the sampling tube diagonally to the bottom corner.

4.3 Representative sample. The following procedure will give a representative sample when no preliminary examination is made, or when in the preliminary examination no samples were removed. If samples were removed the procedure described may be used but will only give a representative sample of the remaining material.

Unless the contents of the drum or cask are known to be mixed, mix them before or after it is opened, using one of the methods described in Appendix B, as appropriate. In all cases, before the bung is removed, clean the exterior of the bung and the area around the bung hole. Then remove the bung, clean it and place it with the inner side uppermost beside the bung hole.

Choose an open sampling tube (see A.2) to suit the viscosity of the liquid to be sampled and lower it to the bottom sufficiently slowly to ensure that the liquid level in the tube does not fall below that of the outside liquid. Close the tube, withdraw it from the container and allow any liquid adhering to the outside of the tube to drain off. Open the tube and allow the whole of the contents to drain into a sample container. Repeat the operation if necessary; do not obtain a larger sample by a “pumping” action. Alternatively, use a bottom-valved sampling tube (see A.3) in a similar manner, lowering it with the valve open.

In the case of volatile liquids use the contents of the tube on the first withdrawal of sample to rinse out the sample container so as to saturate the air in it with vapour of the material. Drain the sampling tube thoroughly and reject the rinsings. Ensure that the bottom of the sampling tube is placed near the bottom of the sample container whilst it is being emptied.

5 Sampling from a small container

Small containers are those that may be easily carried and shaken by hand.

Sampling may be by the same methods as described in clause 4, using (if necessary) smaller versions of the sampling apparatus described for these methods.

A preliminary examination of material held in an opaque container may be made after transferring the whole of the contents after shaking to a transparent container. Allow time, known or found to be required, for any contaminants to settle. After transferring the contents to a transparent container examine the interior of the original container to ensure that no sediment has been left behind.

A representative sample, in many cases, may be taken by thoroughly shaking the container and pouring an appropriate quantity of liquid from it.

In the case of volatile liquids the sample container should be rinsed out with the liquid being sampled so as to saturate the air in it with the vapour of the material.

6 Sampling from a tank having a uniform horizontal cross section throughout its depth

6.1 Introduction. This clause covers road, rail and static tanks. Such tanks, for the convenience of sampling methods, are divided into two classes, shallow tanks of up to about 1.8 m deep and deep tanks.

If it is found that the methods for obtaining a representative sample are not satisfactory then trials should be carried out to ascertain whether sampling on discharge is satisfactory (see clause 10).

6.2 Shallow tank

6.2.1 Introduction. There are two purposes in sampling from a shallow tank. The first, a preliminary examination, is to detect the presence of, or to sample, undissolved matter which normally will be found, after allowing to stand undisturbed, at the surface or on the bottom. The second is to obtain a representative sample. This order may be reversed if the contents are known to be mixed at the time of sampling.

6.2.2 Preliminary examination

6.2.2.1 If a preliminary examination is required and the tank has just been filled allow time, known or found to be required, for any contaminants in the tank to separate.

If the nature or quantity of any contaminant found by the following procedures departs from the normal the consignment should be considered suspect (see 3.4.2).

Examine the surface of the liquid. If the presence of a surface contaminant is suspected take a surface sample, if not proceed with the examination by taking a bottom sample.

6.2.2.2 Surface sample. Remove a sample by using the ladle described in A.1. Lower the ladle into the tank until the upper points of the serrated edge are just above the surface of the liquid so that the surface layer runs into it. **Remove the ladle before it fills completely** and allow any liquid adhering to the ladle to drain off.

Examine the contents of the ladle for the presence of any contaminant, if necessary transferring the sample to a suitable transparent container for examination.

Record the probably nature and approximate quantity of any contaminant. If a sample of any contaminant is required transfer the contents of the ladle to a sample container and repeat the procedure until sufficient sample is obtained. If a sample is not required return the contents of the ladle to the tank.

6.2.2.3 Bottom sample. Choose an open sampling tube (see A.2) to suit the viscosity of the liquid to be sampled, close it and lower it to the bottom of the tank. Open the tube and move it rapidly so that the bottom of the tube traverses the lower inside surface of the tank before the tube is filled. Close the tube, withdraw it from the tank and allow any liquid adhering to the outside of the tube to drain off. Alternatively, use a bottom-valved sampling tube (see A.3) in a similar manner, lowering it with the valve closed.

Examine the contents of the tube for the presence of any contaminant. If the tube is opaque run the liquid out into a suitable transparent container for examination.

Record the probable nature and approximate quantity of any contaminant. If a sample of any contaminant is required transfer the contents of the sampling tube to a sample container and repeat the procedure until sufficient sample is obtained. If a sample is not required return the contents of the tube to the tank.

6.2.3 Representative sample

6.2.3.1 General. The procedure depends on whether or not the contents of the tank have been mixed by one of the methods described in Appendix B or are known to have been mixed already.

In the case of volatile liquids use the contents of the tube on the first withdrawal of sample to rinse out the sample container so as to saturate the air in it with vapour of the material. Drain the sampling tube thoroughly and reject the rinsings. Ensure that the bottom of the sampling tube is placed near the bottom of the sample container whilst it is being emptied.

6.2.3.2 Contents mixed. Choose an open sampling tube (see A.2) to suit the viscosity of the liquid to be sampled and lower it to the bottom sufficiently slowly to ensure that the liquid level in the tube does not fall below that of the outside liquid. Close the tube, withdraw it from the tank and allow any liquid adhering to the outside of the tube to drain off. Open the tube and allow the whole of the contents to drain into a sample container. Repeat the operation if necessary; do not obtain a larger sample by a “pumping” action. Alternatively, use a bottom-valved sampling tube (see A.3) in a similar manner, lowering it with the valve open.

6.2.3.3 Contents not mixed. A representative sample may be obtained either by means of spot samples as described in a) and b), or by sampling on discharge by means of the methods given in clause 10.

Alternatively, take spot samples at evenly spaced intervals measured from the surface of the liquid. The intervals should be not greater than 500 mm and should be smaller if it is suspected that layering is present. The interval should be not less than 80 mm for the sampling tube, and not less than 300 mm for the sampling can or sampling cage.

a) *Sampling tube.* Choose an open sampling tube (see A.2) to suit the viscosity of the liquid to be sampled, close it and lower it into the liquid to the required depth. Open the tube for a short period, close it, withdraw it from the tank and allow any liquid adhering to the outside of the tube to drain off. Open the tube and run the required volume of liquid into the sample container. Alternatively, use a bottom-valved sampling tube (see A.3) in a similar manner, lowering it with the valve closed.

b) *Sampling can or cage.* Close the can or cage (see Appendix A) and lower it to the required depth, open it by sharply jerking the chain and lift it out when air bubbles cease to rise. (Experience may show the time needed if the bubbles are difficult to see.) When the apparatus is raised some contamination of the contents by the upper layers takes place and it is therefore recommended that, when the liquid on the outside has drained off, part of the sample contained in the neck of the bottle (see 3.6.5) should be carefully poured off. If the weighted sampling can has been used transfer part or all of the remainder of liquid in the can to a sample container. If the sampling cage has been used close the bottle tightly and remove it from the cage.

The spot samples obtained may be examined individually or combined in correct proportions to give a representative sample (see 3.1).

6.3 Deep tank

6.3.1 Introduction. A preliminary examination is only needed if it is necessary to ascertain the extent of contamination of the material in the tank or the nature of the total contents.

6.3.2 Preliminary examination

6.3.2.1 General. If a preliminary examination is required and the tank has just been filled allow time, known or found to be required, for any contaminants in the tank to separate.

If the nature or quantity of any contaminant found by the following procedures departs from the normal, the consignment should be considered suspect (see 3.4.2).

Examine the surface of the liquid. If the presence of a surface contaminant is suspected take a surface sample, if not proceed with the examination by taking a middle spot sample and a bottom sample.

6.3.2.2 Surface sample. Obtain a surface sample by means of a weighted sampling can (see A.4) or a sampling cage (see A.5). Use the apparatus unstoppered and lower it carefully until the neck of the can or bottle is just below the surface of the liquid. Withdraw the can or bottle before it is filled completely.

6.3.2.3 Spot sample. Use a weighted sampling can (see A.4) or a sampling cage (see A.5); use the latter if the liquid is volatile.

Close the can or cage and lower it to the required depth, open it by sharply jerking the chain and lift it out when air bubbles cease to rise. (Experience may show the time needed if the bubbles are difficult to see.) When the apparatus is raised some contamination of the contents by the upper layers takes place and it is therefore recommended that, when the liquid on the outside has drained off, part of the sample contained in the neck of the bottle (see 3.6.5) should be carefully poured off. If the weighted sampling can has been used transfer part or all of the remainder of liquid in the can to a sample container. If the sampling cage has been used close the bottle tightly and remove it from the cage.

6.3.2.4 Bottom sample. Obtain a bottom sample by means of the bottom sampling apparatus (see A.6). Lower the apparatus slowly until it just rests in an upright position on the bottom of the tank. When bubbles cease to rise lift the apparatus out and, after the adhering liquid has drained off, transfer the whole of the contents of the apparatus to a sample container, shaking to ensure that any solid material present may be carried out by the liquid. (Experience may show the time needed if the bubbles are difficult to see.)

6.3.3 Representative sample. If the contents of the tank have been mixed by one of the methods described in Appendix B, or are known to have been mixed already, take upper, middle and lower spot samples in that order in the manner described in 6.3.2.3. In all other circumstances take spot samples at regular intervals (to a minimum interval of 300 mm) throughout the depth of the liquid. If a known volume of the liquid in the tank is to be discharged sample the tank in a similar manner throughout the volume of liquid to be discharged, measured upwards from the discharge point.

Large tanks may have more than one sampling point. If necessary, in such cases the sampling procedure decided upon may be repeated at all these points.

The spot samples obtained may be examined individually or combined in the correct proportions to give a representative sample.

NOTE The use of an open bottle raised through the liquid at a uniform rate is deprecated as, unless the liquid is homogeneous, it is unlikely that a representative sample will be obtained.

7 Sampling from a horizontal cylindrical tank

7.1 Introduction. This clause covers road, rail and static tanks. They may be circular or elliptical in cross section and may be horizontal or slightly sloped. Steeply sloped cylindrical tanks can only be sampled satisfactorily on discharge by one of the methods described in clause 10. If a tank has separate compartments each compartment should be sampled separately. If there is more than one sampling point in the compartment the sampling procedure may be repeated, if necessary, at all these points. In all cases, if it is required to take more than one sample from a tank, the order of taking the samples shall be from the top downwards so that each sample is obtained from undisturbed material.

There are two purposes in sampling from these containers. The first, a preliminary examination, is to detect the presence of, or to sample, undissolved matter which normally will be found, after allowing the liquid to stand undisturbed, at the surface or on the bottom. The second is to obtain a representative sample. This order may be reversed if the contents are known to be mixed at the time of sampling.

7.2 Preliminary examination

NOTE Certain tanks are fitted with a fill pipe and a surface sample taken therefrom will only represent the last material entering the tank. If the fill pipe is the only means of access a representative sample can only be obtained on discharge by one of the methods described in clause 10.

7.2.1 General. If the nature or quantity of any contaminant found by the following procedure departs from the normal, the consignment should be considered suspect (see 3.4.2).

If possible examine the surface of the liquid. If the presence of a surface contaminant is suspected, or if it is impossible to examine the surface, take a surface sample, then proceed with the examination by taking a bottom sample.

7.2.2 Surface sample. Remove a sample by using the ladle described in A.1. Lower the ladle into the tank until the upper points of the serrated edge are just above the surface of the liquid so that the surface layer runs into it. **Remove the ladle before it fills completely** and allow any liquid adhering to the ladle to drain off.

Examine the contents of the ladle for the presence of any contaminant, if necessary transferring the sample to a suitable glass container for examination.

Record the probable nature and approximate quantity of any contaminant. If a sample of any contaminant is required transfer the contents of the ladle to a sample container and repeat the procedure until sufficient sample is obtained. If a sample is not required return the contents of the ladle to the tank.

7.2.3 Bottom sample. The sample may be removed either through the hatch or manhole or from the bottom outlet, whichever is more convenient. The latter method should always be used with sloped tanks.

a) *Hatch or manhole.* Choose an open sampling tube (see A.2) to suit the viscosity of the liquid to be sampled, close it and lower it to the bottom of the tank. Open the tube and move it rapidly so that the bottom of the tube traverses as much as possible of the lower inside surface of the tank before the tube is filled. Close the tube, withdraw it from the tank and allow any liquid adhering to the outside of the tube to drain off. Alternatively, use a bottom-valved sampling tube (see A.3) in a similar manner, lowering it with the valve closed.

b) *Bottom outlet.* Open the valve slightly so that a small flow is obtained and collect sufficient sample in a suitable vessel. Ensure that the sample is taken directly from the valve and not through ancillary pipework, etc.

In either case examine the sample obtained, if necessary transferring it to a glass container. Record the probable nature and approximate quantity of any contaminant. If a sample of any contaminant is required repeat the procedure if necessary until sufficient sample is obtained, transferring the material to a sample container. If a sample is not required return the sample to the tank.

7.3 Representative sample

7.3.1 Introduction. The sampling procedure depends on whether or not the contents of the tank are known to have been mixed at the time of sampling (see Appendix B).

In the case of volatile liquids use the contents of the tube on the first withdrawal of sample to rinse out the sample container so as to saturate the air in it with vapour of the material. Drain the sampling tube thoroughly and reject the rinsings. Ensure that the bottom of the sampling tube is placed near the bottom of the sample container whilst it is being emptied.

7.3.2 Contents mixed. Choose an open sampling tube (see A.2) to suit the viscosity of the liquid to be sampled and lower it to the bottom sufficiently slowly to ensure that the liquid level in the tube does not fall below that of the outside liquid. Close the tube, withdraw it from the tank and allow any liquid adhering to the outside of the tube to drain off. Open the tube and allow the whole of the contents to drain into a sample container. Repeat the operation if necessary; do not obtain a larger sample by a “pumping” action. Alternatively, use a bottom-valved sampling tube (see A.3) in a similar manner, lowering it with the valve open.

7.3.3 Contents not mixed. A representative sample may be obtained either by sampling on discharge by one of the methods described in clause 10, or by combining a number of spot samples taken as follows (but also see 3.1).

Take a bottom-valved sampling tube (see A.3), close it, lower it to the required depth in the liquid and open it for a short period. Close the tube, withdraw it and allow any adhering liquid to drain off.

Discharge the required volume into the sample container. Repeat the sampling at the same depth if sufficient sample is not obtained.

If tank calibration tables are available ascertain the levels corresponding to $\frac{1}{6}$, $\frac{1}{2}$ and $\frac{5}{6}$ of the total volume contained in the tank at the time of sampling. Take spot samples at these depths as described in the previous paragraph and combine them in equal proportions. If the tank is full take the samples at standard depths of $\frac{1}{6}$, $\frac{1}{2}$ and $\frac{5}{6}$ of the total depth and combine them in the proportions 3 : 4 : 3.

If, however, tank calibration tables are not available, take spot samples at evenly spaced intervals commencing at the surface of the liquid. The intervals should be not greater than 300 mm and should be smaller if it is suspected that layering is present, but the intervals should be not less than 80 mm so as to obtain material undisturbed by the previous sampling procedures.

Combine the spot samples in proportion to the horizontal volume interval of the tank about the point at which the sample was taken. The proportions in which the spot samples should be combined may readily be obtained by first determining the position of the liquid surface in the tank, then plotting on squared paper an outline of the tank, the liquid surface, and drawing horizontal lines representing the boundaries of each layer to be spot sampled. Counting the squares in each area representing a liquid layer gives the proportions of the volumes and hence the proportions in which the spot samples should be combined. Examples are shown in Figure 1 representing a full cylindrical tank and a partially filled elliptical tank.

The volume of each spot sample should be checked as adequate to provide the required proportion for the combining scheme before taking the next lower sample. Each spot sample should be placed in its own separate container and well mixed before withdrawing the portion required for the representative sample.

Alternatively, use a weighted sampling can (see A.4) or a sampling cage (see A.5); use the latter if the liquid is volatile, as described in 6.3.2.3.

8 Sampling from a tank of irregular shape

Sample as described in 6.2 (shallow tank) or 6.3 (deep tank) but if the contents are not mixed the depth : volume characteristic should be known, e.g. from tank calibration tables or by taking spot samples at evenly spaced intervals as described in 7.3.3 (if the tank has parallel sides) in order that the examination and combining of the spot samples may be carried out properly (see 3.1).

9 Sampling from a flexible walled container

NOTE It is essential when sampling from a flexible walled container that a rigid platform be provided across the tank from which the operator may work safely.

This clause covers flexible walled containers, i.e. collapsible tanks and dracones.

The container is normally sampled on loading or discharge by the methods described in clause 10, but spot samples and, if the material is known to be mixed, representative samples may be obtained by the methods described in clause 7.

10 Sampling from a pipeline

10.1 Introduction. The methods given in 10.2 may fail to give a representative sample if the liquid contains appreciable quantities of an immiscible contaminant or if there is incomplete mixing of materials of very different densities. If this is suspected the variability of the materials and/or the accuracy of the sampling method should be assessed and a special sampling scheme set up (see Part 1 of this standard).

The methods available are:

- a) sampling from the end of a pipeline;
- b) sampling by diversion of the whole of the stream;
- c) sampling by means of a bypass fitted to a centrifugal pump;
- d) sampling by means of a probe fitted in a pipeline.

10.2 Methods

10.2.1 End sampling. If the flow from the end of a pipe is accessible, and it is possible to collect the whole of the discharge from the pipe in a container for a short period, the following method is used.

Place a funnel in a sample container and hold it under the exit stream for a short period, ensuring that the whole of the stream is caught by the funnel.

10.2.1.1 Spot sample. This is one sample collected as described in 10.2.1.

10.2.1.2 Representative sample. An intermittent sample is obtained by taking spot samples, as described in 10.2.1.1, at regular intervals during the whole of the transfer of the liquid from the end of the pipeline. The spot samples should be taken at time intervals inversely proportional to the rate of flow and combined in equal volumes, or be taken at regular time intervals and combined in volumes proportional to the rate of flow.

10.2.2 Diversion sampling. If the pipeline has no open end allowing the use of the method described in 10.2.1 and the pipeline diameter is small so that the probe (see 10.2.4) cannot be fitted, a three-way cock should be fitted in the pipeline so that the whole of the flow can be diverted for a set period into the sample container. The sampling apparatus used is as described in 10.2.1

10.2.3 Bypass sampling. If the liquid to be sampled is pumped through the pipeline by means of a centrifugal pump, a sampling point can be provided by means of a bypass round the pump, a typical arrangement of which is shown in Figure 2. A centrifugal pump is shown in this arrangement; the procedure always works satisfactorily with a centrifugal pump but not always with a positive displacement pump. The valves A and B are provided to test that there is a flow of liquid round the bypass; it is necessary to ensure this flow otherwise a sample may be obtained that will not be a representative one.

The bypass is used as follows.

With all the valves closed, open S and alternately open A and B to ensure that liquid is flowing through the bypass. Close S, open A and B, open S and flush the lines.

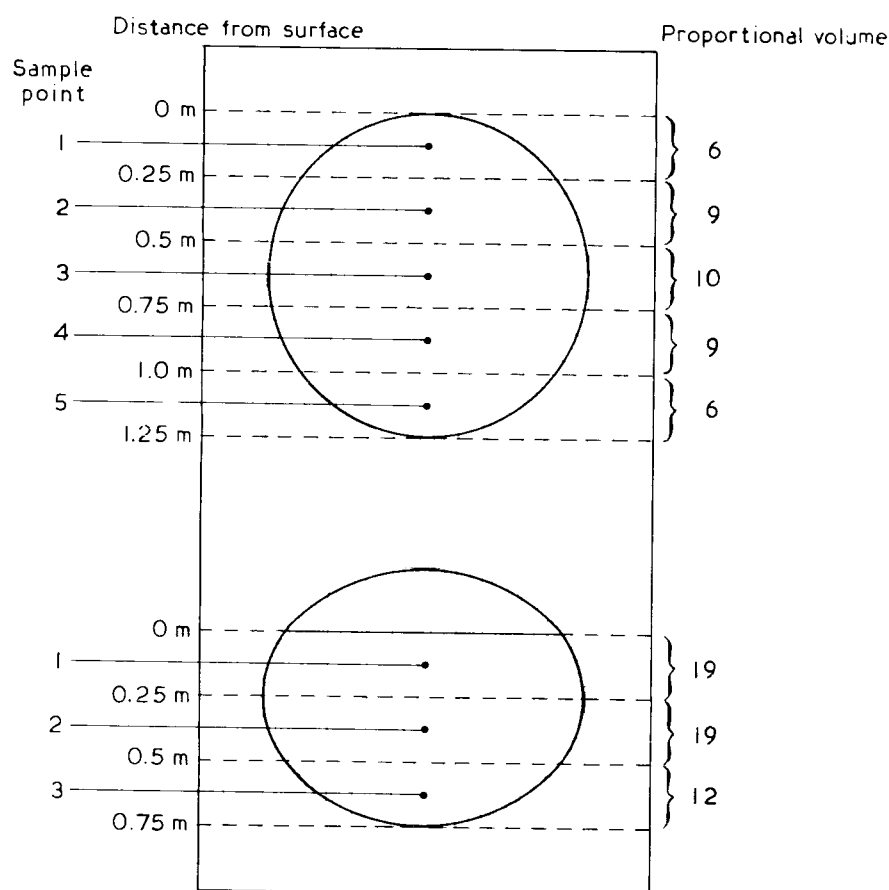


Figure 1 — Sampling scheme for a full cylindrical tank and a partially filled elliptical tank

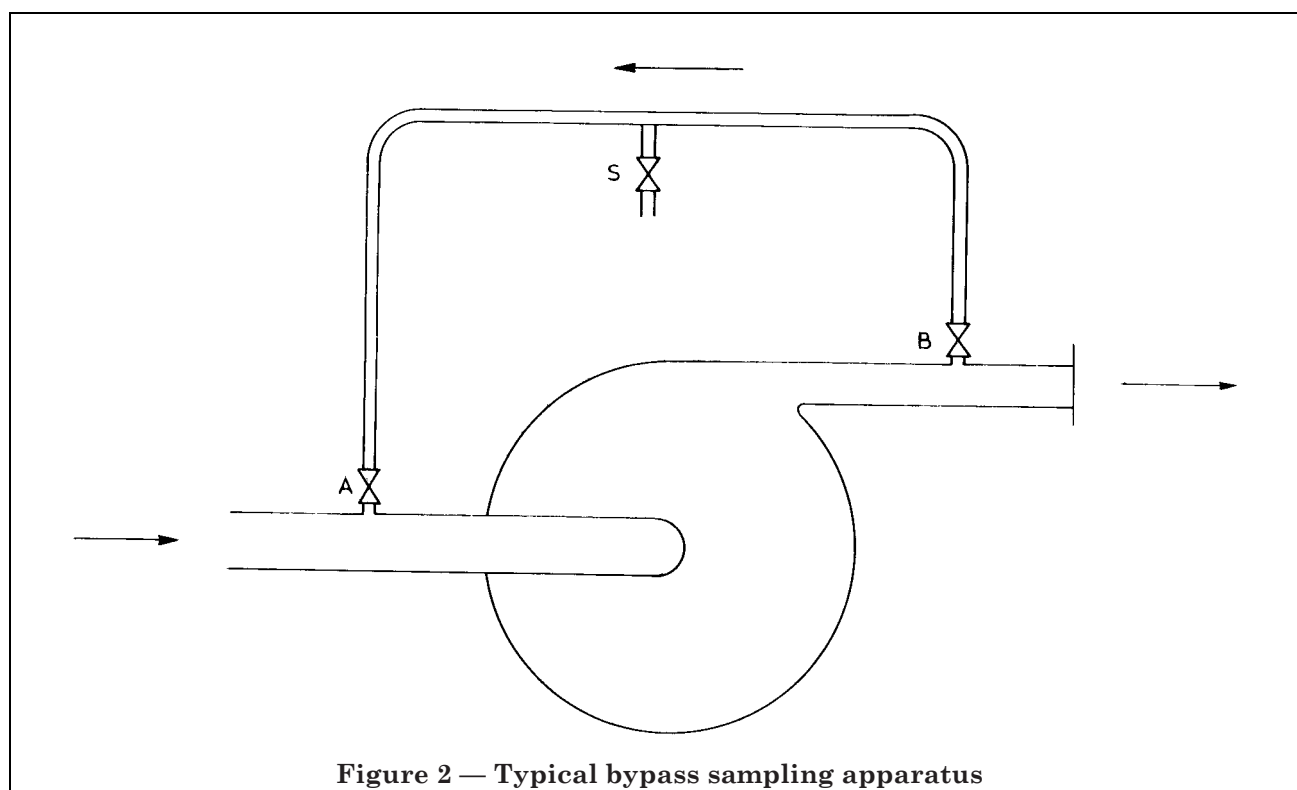


Figure 2 — Typical bypass sampling apparatus

10.2.3.1 Spot sample. Open valve S and collect the required quantity in the sampling container.

10.2.3.2 Representative sample. This is a continuous sample obtained by opening valve S to such an extent that sufficient liquid is collected in a sample container during the whole of the transfer of the liquid through the pipeline. If the liquid flow from the pump varies by more than 10 % of the mean adjust the flow from valve S so that it remains proportional to the liquid flow.

If the flow rate from the pump is very variable manual adjustment of the rate of sample withdrawal may be difficult or impracticable in which case the use of an automatic sampler is a possible alternative (see clause 12).

10.2.4 Probe sampling. For a theoretical consideration of probe sampling see clause 22 of Part 1:1976 of this standard.

10.2.4.1 If the diameter of the pipeline is large enough a suitable sample probe can be installed. The probe should be such that it will minimize the effect of stratification or the dropping out of heavier particles or droplets in the liquid being sampled.

The following conditions are necessary for satisfactory probe sampling according to the type of liquid.

a) *Homogeneous and random heterogeneous liquids.* No special conditions are necessary and the design of the probe is not critical.

b) *Non-uniform and heterogeneous liquids.* To obtain representative samples the probe should be placed in an area of turbulent flow, i.e. the Reynolds number would be well above 2 000. This turbulence may be achieved, where flow is normally streamline, by placing a specially designed restrictor/mixer device up-stream of the probe.

c) *Multi-phase liquids.* No firm recommendation can be given for these liquids because probe sampling can be affected by pipeline size, configuration and flow rate.

Two types of probe, in general use, are as follows.

1) A pipe of about 12 mm bore, fitted into the side of the pipeline, that extends into the centre of the pipeline with its inner end bevelled at 45° [see Figure 3(a)] or its inner end bent at 90° and extended for a very short distance. Alternatively, a probe incorporating a hole or aperture at the centre of the pipeline can be used. In all cases the orifice faces upstream.

2) A probe extending the full height of an internal square section installed in the pipeline. The probe is so designed that a representative sample is obtained automatically across the whole vertical cross section (see Figure 3(b) and clause 12).

In all cases the length of the probe outside the pipeline should be as short as possible and should terminate in a suitable valve capable of controlling the rate of flow from the probe.

10.2.4.2 During probe sampling knowledge is required of the velocities of flow, as follows.

- a) The mean velocity of the liquid in the pipeline at the orifice of the probe during the period over which the sample is taken.
- b) The mean velocity of the sample flowing from the probe. This is adjusted, using the valve, so that it is approximately equal to the centre line velocity of the liquid in the pipeline (i.e. it is isokinetic). This centre line velocity can be taken as approximately 1.2 times the mean velocity in the pipeline as in a).

If the velocity of flow in the pipeline is variable, manual adjustment of the velocity of flow from the probe to achieve isokinetic conditions may be difficult or impracticable, in which case the use of an automatic sampler is a possible alternative (see clause 12).

10.2.4.3 Before taking a sample the probe line should be flushed. This can be conveniently done while setting up the required velocity of flow, as described in 10.2.4.2. The probe is used as follows.

10.2.4.3.1 *Spot sample.* This is obtained by operating the probe under the conditions described in 10.2.4.2 and running the required quantity of liquid into a sample container.

10.2.4.3.2 *Representative sample.* Either a continuous sample is taken by operating the probe continuously, running the sample into a sample container, or, if a continuous sample would be too large, an intermittent sample is obtained by taking spot samples, as described in 10.2.4.3.1, at intervals during the whole of the transfer of the liquid from the end of the pipeline. The spot samples should be taken at time intervals inversely proportional to the rate of flow and combined in equal volumes, or be taken at regular time intervals and combined in volumes proportional to the rate of flow. In both cases the probe should be operated under the conditions described in 10.2.4.2 and the sampling should take place during the whole of the transfer of the liquid through the pipeline.

11 Sampling from a channel

11.1 General. Sampling from channels is made difficult by the possibility of wide fluctuations in the flow of the liquid; by variability in the composition of the liquid, particularly of its solid content; by irregularity in channel shape; by limited access, particularly to closed channels.

As far as possible, therefore, sampling should be carried out at a point of free fall of the liquid, for example at a step, weir or pipe end. The sampling methods described in 10.2.1 may be used but with broad or large flows it may only be possible to sample part of the stream and a representative sample may be difficult to obtain even if spot samples are taken from different parts of the falling stream.

In open channels or closed channels that are not too deep, surface and bottom samples may be taken by the methods described in 6.2 but the method to obtain a representative sample using a closable sampling tube will only produce a sectional sample that is dependent on the flow characteristics.

In small scale operations it may be possible to block the channel at a manhole and, by means of a pump, transfer the whole of the liquid flowing to a holding drum or tank the contents of which can then be sampled as described earlier.

11.2 Sampling automatically. A number of automatic systems are available commercially that can obtain samples at a fixed point in a channel. Two systems that can be made are described in 11.2.1 and 11.2.2. The first takes a continuous sample of up to 5 litres over a period of up to 12 h; the second takes an intermittent sample, the sampling interval being determined by a timer. In both cases a representative sample will only be taken if the stream is steady and well mixed.

11.2.1 Continuous channel sampling apparatus. A typical device capable of extracting liquid continuously from channels is shown in Figure 4. It can draw amounts up to 5 litres in 12 h but does not give a weighted average since it works at a constant rate irrespective of the rate of flow of liquid in the channel. The apparatus requires no power supply, is unaffected by suspended solids and should only be used just under the surface of the liquid as shown in Figure 4. A shallow upper vessel, A, of capacity about 6 litres is filled initially with clean water which can leak out of an orifice, B, at a rate of about one drop per second. The orifice is made of a dimensionally stable plastics material; alternatively, a screw clip on the rubber outlet tube may be used. The space above the water in A is connected by a tube to a lower vessel, C, of capacity about 5 litres which is connected at its lower end by a pipe to the channel. As water runs out of the upper vessel the reduced atmospheric pressure within the system draws liquid at the same rate into the lower vessel.