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Third edition
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Petroleum liquids — Manual sampling

Produits pétroliers liquides — Échantillonnage manuel

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Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3170 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, Subcommittee SC 3, *Static petroleum measurement*.

This third edition cancels and replaces the second edition (ISO 3170:1988), which has been technically revised.

The principal technical changes include the addition of

— procedures for tank sampling under restricted and closed system conditions, and

— procedures for the taking of manual spot samples from pipelines containing high vapour pressure liquids.

Introduction

This International Standard may be applied in combination with ISO 3171.

The purpose of this International Standard is to standardize conditions for obtaining a sample of liquid/semi-liquid hydrocarbons from a tank, drum or pipeline by manual means. If the hydrocarbon to be sampled is of non-homogeneous character showing significant variations in composition or containing sediments and water, samples taken manually should not be expected to be representative, but may enable the degree of non-homogeneity to be assessed and estimates of quality and quantity to be made.

Procedures are specified which minimize or eliminate losses of light ends from samples. Such losses can occur during handling or transfer of samples, thereby making them non-representative of the bulk.

The procedures specified are intended to provide samples for the following purposes:

a) the determination of the liquid/hydrocarbon quality;

b) the determination of the water content;

c) the determination of other contaminants that are not considered to be part of the liquid transferred.

If the sampling conditions for purposes a), b) and c) are in conflict, separate samples are required.

Sampling procedures for tank contents that are not homogeneous are specified that enable the degree of non-homogeneity to be assessed and estimates of quality and quantity to be made.

Procedures for the tank sampling of liquid hydrocarbons under inert gas pressure are included, together with techniques for sampling from tanks which are equipped with vapour emission control systems.

It is recognized that, in many countries, some or all of the items covered by this International Standard are the subject of mandatory regulations imposed by the laws of those countries. In cases of conflict between such mandatory regulations and this International Standard, the former prevail.

Petroleum liquids — Manual sampling

1 Scope

This International Standard specifies the manual methods to be used for obtaining samples of liquid or semi-liquid hydrocarbons, tank residues and deposits from fixed tanks, railcars, road vehicles, ships and barges, drums and cans, or from liquids being pumped in pipelines.

It applies to the sampling of petroleum products, crude oils and intermediate products, which are stored in tanks at or near atmospheric pressure, or transferred by pipelines, and are handled as liquids at temperatures from near ambient up to 200 °C.

The sampling procedures specified are not intended for the sampling of special petroleum products which are the subject of other International Standards, such as electrical insulating oils (IEC 60475), liquefied petroleum gases (ISO 4257), liquefied natural gases (ISO 8943) and gaseous natural gases (ISO 10715).

This International Standard refers to existing methods of sampling and the type of equipment presently in use. It is, however, not intended that it should exclude the use of new equipment not yet developed for commercial use, provided that such equipment enables samples to be obtained in accordance with the requirements and procedures of this International Standard.

NOTE For the purposes of this International Standard, the term "% (m/m)" is used to represent the mass fraction.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1998 (all parts), *Petroleum industry — Terminology*

ISO 2859-1:1999, *Sampling procedures for inspection by attributes — Part 1: Sampling plans indexed by acceptable quality limit (AQL) for lot-by-lot inspection*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1998 and the following apply.

3.1
acceptable quality level
AQL
maximum per cent defective (or the maximum number of defects per hundred units) that, for purposes of sampling inspection, can be considered satisfactory as a process average

3.2
all-level sample
sample obtained with an apparatus which accumulates the sample while passing in one direction only through the total liquid height, excluding any free water

3.3
automatic sampler
device used to extract a representative sample from the liquid flowing in a pipe

NOTE The automatic sampler generally consists of a probe, a sample extractor, an associated controller, a flow measuring device, and a sample receiver.

3.4
batch
collection of packages containing a product of a single type and composition and of a single manufactured lot, or of a single delivery

3.5
bottom sample
spot sample taken from the product at or close to the bottom of a tank or container

See Figure 1.

3.6
bottom water sample
spot sample of free water taken from beneath the petroleum in a tank

3.7
closed sampling
process of taking samples within a tank under closed conditions, which does not permit the release of any tank contents or vapour to the atmosphere

3.8
composite sample
sample obtained by combining a number of spot samples in defined proportions so as to obtain a sample representative of the bulk of the product

3.9
dipper sample
sample obtained by placing a dipper or other collecting vessel in the path of a free-flowing stream to collect a definite volume from the full cross-section of the stream at regular time intervals for a constant time rate of flow, or at time intervals varied in proportion to the flow rate

NOTE This method is normally restricted to sampling petroleum coke from conveyor belts.

3.10
drain sample
sample obtained from the water draw-off valve on a storage tank

NOTE Occasionally, a drain sample may be the same as a bottom sample, for example, in the case of a tank car.

3.11
floating roof sample
spot sample taken just below the surface to determine the density of the liquid on which the roof is floating

3.12
grease sample
spot sample obtained by scooping or dipping a quantity of soft or semi-liquid material from a container

3.13
integrity of the sample
condition of being complete and unaltered, i.e. the sample being preserved with the same composition as when it was taken from the bulk of the liquid

3.14
lower sample
spot sample taken at a level of five-sixths of the depth of liquid below the top surface

See Figure 1.

3.15
middle sample
spot sample taken at a level of one-half of the depth of liquid below the top surface

See Figure 1.

3.16
mixer
device which provides a homogeneous mixture of the liquid within a pipeline or container in order to obtain a representative sample

3.17
open sampling
process of taking traditional samples within a tank via an open gauge hatch or gauging access point

NOTE If the tank ullage space is pressurized, it will normally be necessary to use other (closed or restricted) procedures to avoid de-pressurizing the tank and the consequent loss of volatile organic compounds (VOCs).

3.18
per cent defective
one hundred times the number of defective units of product contained in any given quantity of units of product divided by the total number of units of product inspected, i.e.:

$$\text{per cent defective} = \frac{\text{number of defectives}}{\text{number of units inspected}} \times 100$$

3.19
portable sampling device
PSD
housing designed to provide a gas-tight connection to a vapour-lock valve, which contains a restricted or closed system sampler and is fitted with a tape or cable winding mechanism for lowering and retrieving the sampler

3.20
representative sample
sample having its physical or chemical characteristics identical to the volumetric average characteristics of the total volume being sampled

3.21
residues and deposits
organic and inorganic matter, together with any water dispersed within it, which has separated from the liquid and either fallen to the bottom of the tank containing the liquid, or been left in the tank after the liquid has been pumped out

3.22
restricted sampling
process of taking samples within a tank using equipment which is designed to substantially reduce or minimize the vapour losses that would occur during open sampling, but where the equipment is not completely gas-tight

3.23
running sample
sample obtained with an apparatus which accumulates the sample while passing in both directions through the total liquid height, excluding any free water

3.24
sample conditioning
mixing necessary to homogenize the sample during sample handling in preparation for subsampling and/or analysis

3.25
sample handling
any conditioning, transferring, dividing and transporting of the sample

NOTE Sample handling includes transferring the sample from the primary sampling device to any secondary container, and the transferring of subsamples to the laboratory apparatus in which it is to be analyzed.

3.26
sample size
number of samples to be drawn from a batch to determine its acceptability as given in sampling plans

3.27
skim sample
surface sample
spot sample taken from the surface of the liquid

See Figure 1.

3.28
spot sample
sample taken at a specific location in a tank or from a flowing stream in a pipe at a specific time

3.29
static mixer
mixing device having no moving parts and located within a pipe or tube

NOTE The effectiveness of the static mixer depends on the kinetic energy of the moving liquid for the energy required to mix the liquid.

3.30
still-well
guide pole
still-pipe
sounding-pipe
stand pipe
vertical cylindrical pipe built into a tank to permit gauging operations while reducing errors arising from turbulence or agitation of the liquid

NOTE 1 Samples taken from unperforated or unslotted still-wells should not be used for custody transfer applications, see 7.2.1.3.

NOTE 2 Still-wells may also be found on ships and barges.

3.31
suction-level sample
outlet sample
sample taken at the lowest level from which liquid hydrocarbon is pumped from the tank

See Figure 1.

NOTE In determining this level, appropriate allowance is made for any fittings within the tank such as swing-arm, suction baffle or internal bend.

3.32
sump sample
spot sample taken from within a sump

3.33
suspended water
water within the oil that is finely dispersed as small droplets

NOTE It may, over a period of time, either collect as free water or become dissolved water, depending on the conditions of temperature and pressure prevailing.

3.34
tap sample
tank-side sample
spot sample taken from a sample tap on the side of a tank

3.35
test portion
portion of a sample or subsample that is introduced into the analytical test apparatus

3.36
top sample
spot sample obtained 150 mm below the top surface of the liquid

See Figure 1.

3.37
total water
sum of all the dissolved, suspended and free water in a cargo or parcel of oil

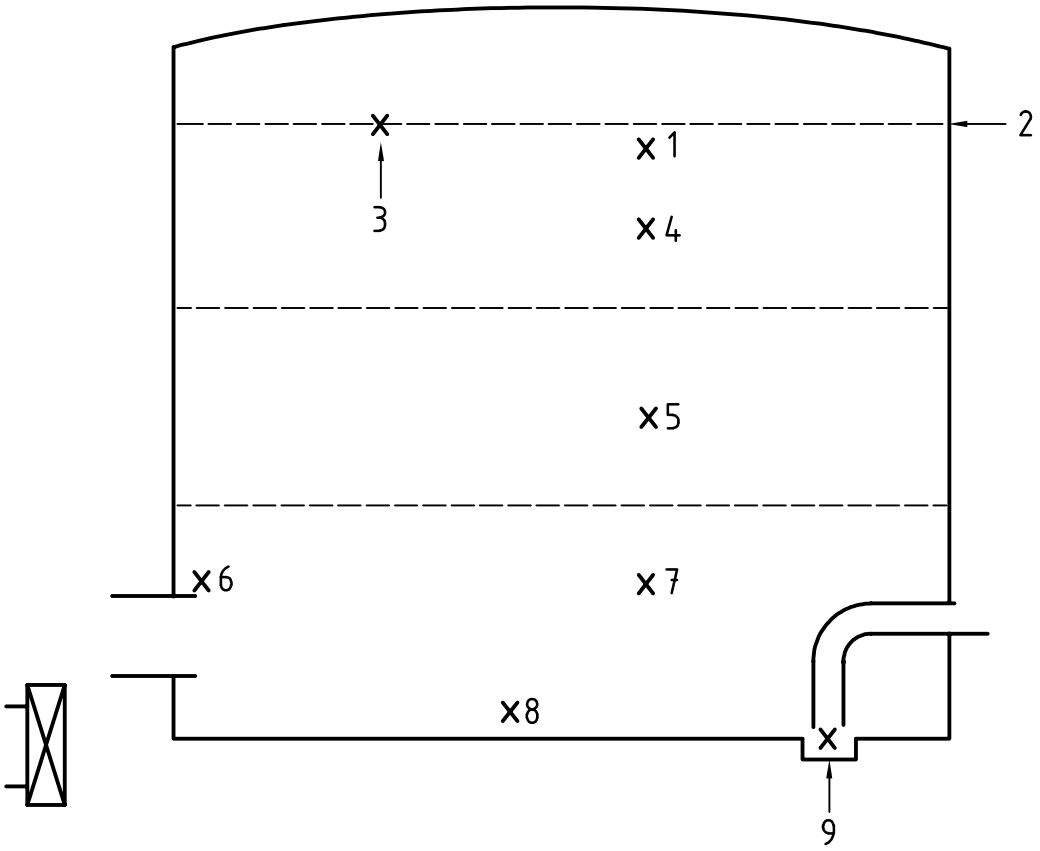
3.38
ullage
empty capacity left in a fixed volume sample receiver/container above the liquid surface

3.39
upper sample
spot sample taken at a level of one-sixth of the depth of liquid below the top surface

See Figure 1.

3.40
vapour-lock valve
vapour control valve
device fitted to the top of vapour-tight or pressure tanks to permit manual measurement and/or sampling operations to be carried out without loss of pressure

3.41
zone sample
core sample
flow-through sample
sample taken as that part of the liquid column which is trapped within the whole height of a sampler when it is sealed at a single spot location within a tank, after having been fully flushed as it was lowered to that position



Key

1 top sample
2 surface of oil
3 skim sample
4 upper sample
5 middle sample
6 suction level or outlet sample
7 lower sample
8 bottom sample
9 sump sample

Figure 1 — Examples of spot sample positions

4 Principles

4.1 To ensure that samples submitted for examination are as representative as possible of the liquid being sampled, the necessary precautions are given. These depend on the characteristics of the liquid, the tank, container or pipeline from which the sample is being obtained, and the nature of the tests to be carried out on the sample.

Two basic manual sampling methods are available:

— tank sampling (static sampling);

— pipeline sampling (dynamic sampling).

When a batch is received or consigned, either tank or pipeline sampling, or both, may be possible. However, if both methods are used, the two sets of samples shall not be mixed.

4.2 Tank sampling is commenced when the contents of the tank are at rest. The following types of samples are normally taken for analysis:

a) upper, middle and lower samples, or

b) upper, middle and suction-level (outlet) samples.

If tests on these samples show that the contents of the tank are homogeneous, they may be combined, in proportion to the volume that each sample represents, for further tests.

If the tests on these samples show that the contents of the tank are non-homogeneous, it may be necessary to draw spot samples from more than three levels and either a composite sample is prepared for analysis or, if blending would impair the integrity of the sample, each sample is analysed separately and the composition corresponding to the composite sample is calculated. In this calculation, allowance is made for the proportion of the oil represented by each sample. Examples of spot sample positions are shown in Figure 1.

Other methods are a running sample or an all-level sample.

As both these methods only result in a single sample, they cannot be used to assess the homogeneity (or otherwise) of a tank's contents. Running and all-level samples are commonly taken and used to determine the average quality of a tank's contents.

NOTE 1 Safety and environmental regulations may restrict tank sampling operations which can result in the release of hydrocarbons or other volatile organic compounds (VOCs) into the atmosphere. In these circumstances, it will not normally be feasible to use traditional open sampling procedures via an open gauge hatch or gauging access point. If the tank ullage space is pressurized, and/or the tank forms part of a vapour balancing/recovery system, it will normally be necessary to use closed or restricted sampling procedures to avoid de-pressurizing the tank and minimize the consequent loss of VOCs. If the vapour from the tank contents is hazardous, it will also normally be necessary to use closed or restricted sampling procedures to minimize the risk of environmental impact.

NOTE 2 Closed sampling is the process of taking samples within a tank using closed sampling devices under closed system conditions. A closed system exists when the operations do not permit the direct exposure and/or release of any tank contents to atmosphere. Manual closed sampling is therefore normally carried out via a vapour-lock valve, using a closed sampling device that provides a gas-tight seal when in use. In order to ensure that no residual vapour is released from a closed system, special facilities may be provided to displace any vapour held up within the device prior to disconnecting the sampling device from the vapour-lock valve.

NOTE 3 Restricted sampling is the process of taking samples within a tank using a restricted sampling device that is operated via a vapour-lock valve. Restricted equipment is designed to substantially reduce or minimize the vapour losses that would occur during open sampling, but may still allow a small quantity of vapour to escape because the equipment is not completely gas-tight.

4.3 To obtain a representative sample of a batch/parcel transfer quantity being pumped in a pipeline, the sample is drawn using an automatic sampling device in accordance with ISO 3171. On occasions, it may be necessary to take dynamic pipeline samples manually. These are spot samples and may not be representative of the bulk (see 7.4).

5 Apparatus

5.1 General

All sampling devices shall be designed to be leak-tight, and constructed so as to assure the function for which they are intended in order to maintain the initial characteristics of the oil. They shall be of sufficient strength and externally protected to withstand normal internal pressures likely to be generated, and sufficiently robust to withstand any handling that may be encountered. Their cleanliness shall be confirmed before use.

NOTE 1 In some cases, it may be desirable to rinse the sample receiver/equipment with the fluid that is to be sampled, prior to taking the actual sample (although this will normally only be practicable with liquid hydrocarbons).

NOTE 2 Various sampling devices are described in 5.2 to 5.7 and any essential aspects are specified. Detailed specifications have not been given for these items because any suitable device of the type described may be used.

5.2 Tank samplers

5.2.1 General

Tank samplers are classified according to the type of sample to be drawn, these are

— spot sample,

— zone/core sample,

— running sample, and

— all-level sample.

Tank samplers are also classified according to the mode of tank operation and sampling access, these are

— open (traditional) sampling,

— restricted sampling, and

— closed sampling.

Synthetic fibre cords shall not be used for lowering or raising tank samplers through the tank contents as they can generate electrostatic sparks.

NOTE Chains are not recommended for suspending samplers because earth continuity cannot be guaranteed.

5.2.2 Spot and zone samplers

5.2.2.1 General

Spot and zone samplers shall be constructed so that a sample can be taken at any specific level in a tank. The equipment described in 5.2.2.2 to 5.2.2.4 is suitable.

NOTE Other spot sampling devices are available and may be used. Some have special opening facilities, for example, having valves opened or closed at the desired level by a weight falling down alongside and guided by the suspending cable, or having wing or flap valves which are closed upon initiation of upward movement. Some are designed to be operated when deployed via a vapour-lock valve (restricted and closed system samplers).

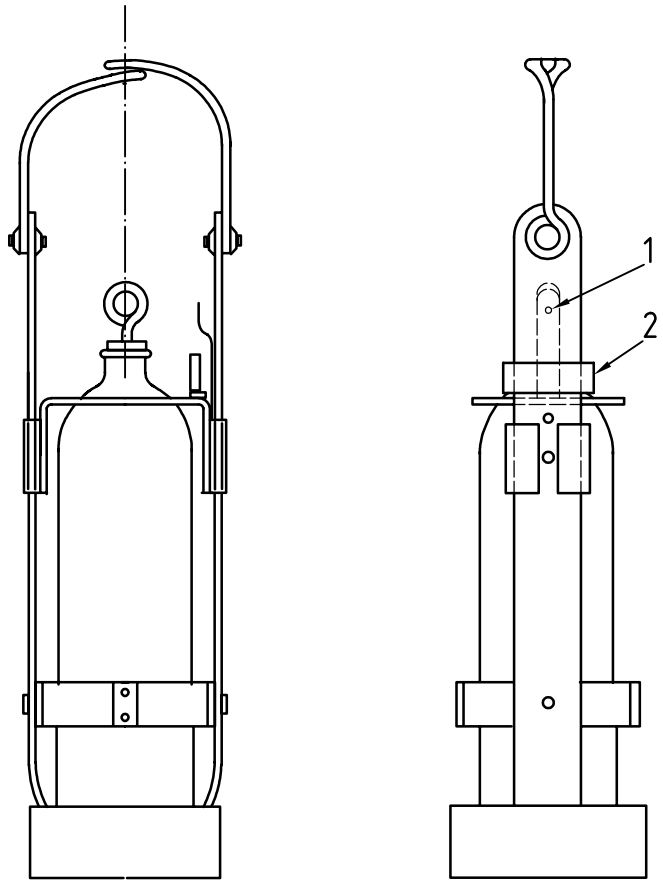
5.2.2.2 Sampling cage

A sampling cage shall be a metal or plastics holder or cage, suitably constructed to hold the appropriate container, typically a bottle or can. The combined apparatus shall be weighted so as to sink readily in the product to be sampled, and provision shall be made to fill the container at any desired level (see Figure 2).

Sampling cages should be sized to fit the desired sample bottle size. Some designs of bottle cage can accept a variety of different neck size (and volume) bottles, and incorporate a floating ball system to seal the bottle once it has been filled.

NOTE 1 The use of a sampling bottle cage is preferred to other spot sampling methods for volatile products, since it avoids the loss of light ends that is likely to occur when transferring the sample to another container.

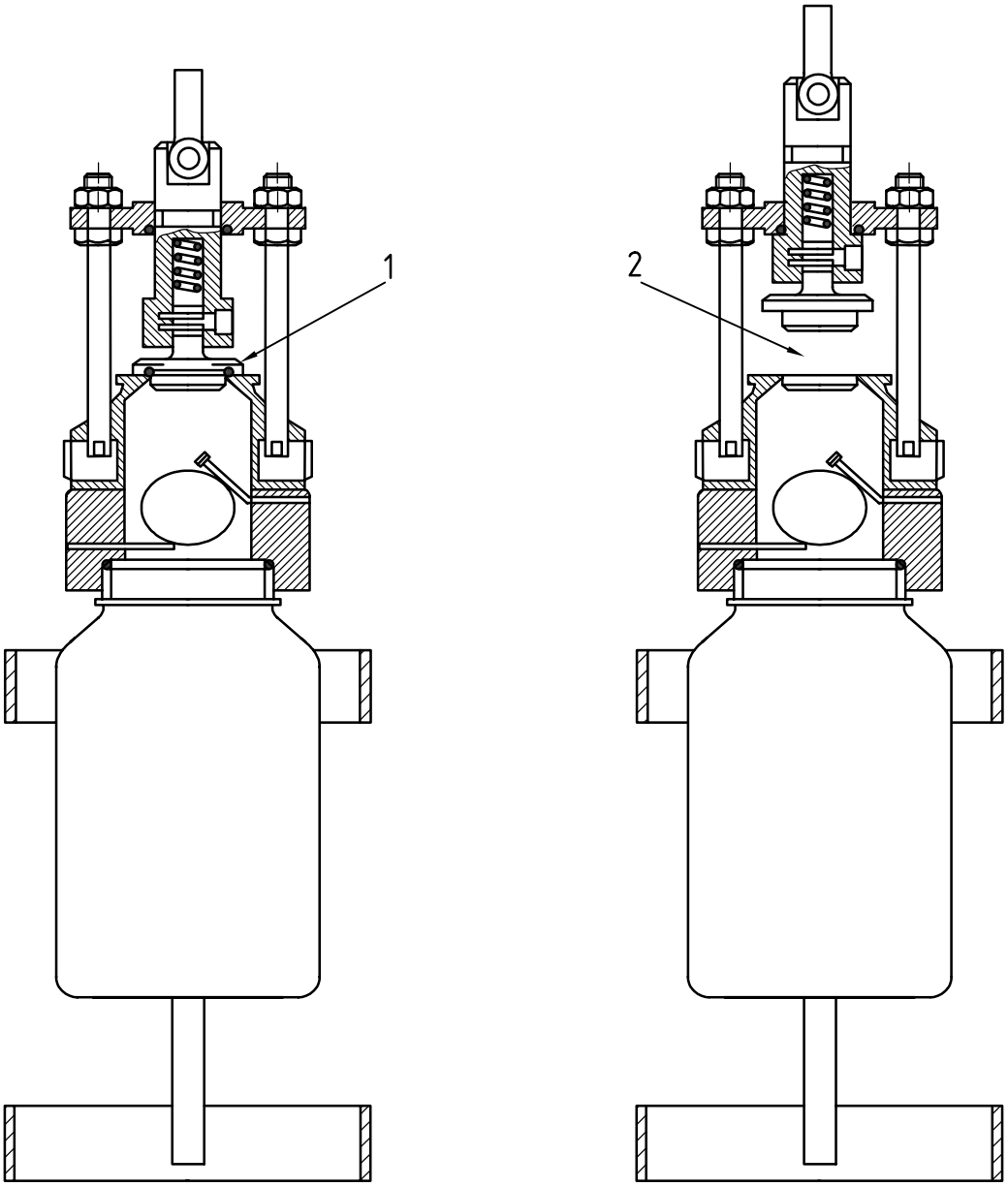
NOTE 2 The sampling cage may be omitted if the sample bottle is securely attached to a weighted cord. The cork is also tied to the line about 150 m from the neck of the bottle.



Key

1 swivel point
2 locking piece

Figure 2 — Example of a sample-bottle cage



Key

1 sample inlet closed

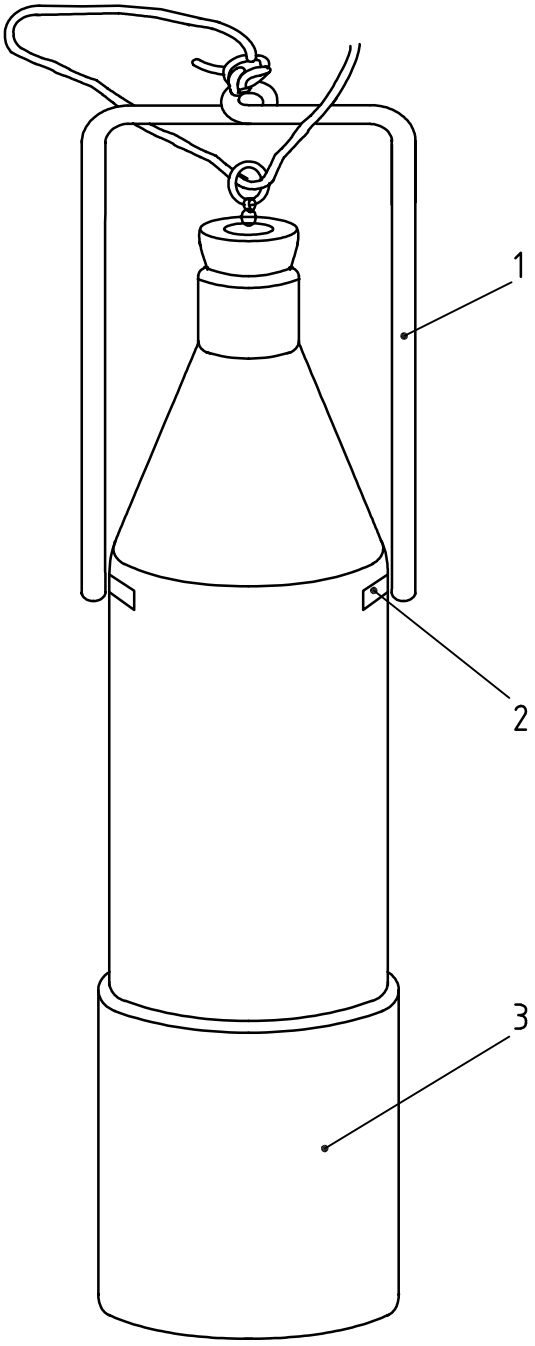
2 sample inlet open

NOTE This example of a proprietary bottle cage sampler incorporates a self-sealing mechanism to close the inlet once the bottle is full.

Figure 2 — Example of a sample-bottle cage (*continued*)

5.2.2.3 Weighted sampling can (beaker)

The sampling can or beaker (see Figure 3) shall be weighted so as to sink readily in the liquid to be sampled. The lowering device shall be attached to the can in such a manner that the stopper can be opened by means of a sharp jerk. In order to avoid problems in cleaning the can and/or possible contamination of sensitive samples, any weighting material shall be fixed to the can in such a way that it does not come into contact with the sample.



Key

1 wire handle
2 wire lugs
3 weighting material

Figure 3 — Example of a weighted can or beaker

5.2.2.4 Zone/core sampler

A zone/core sampler shall consist of a tube made of glass, metal or plastics material, open at both ends to allow a free flow of liquid during lowering through the fluid. The closing of the lower end at the desired level may be achieved by various devices:

a) a closure mechanism actuated by upward movement of the sampler;

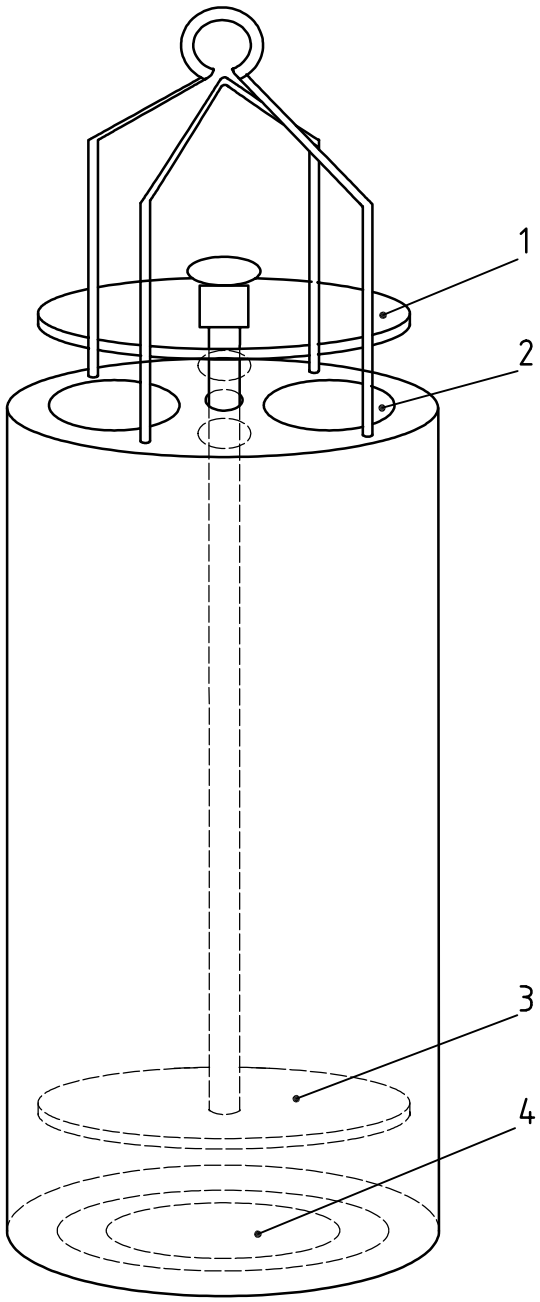
b) a weight falling down the suspending cable (drop messenger), so as to actuate the closure mechanism;

c) a float-operated trigger closure mechanism;

d) a closure mechanism actuated by an extension rod or by a sharp tug of the cord.

A zone/core sampler shall be designed and constructed such that, if lowered slowly, it can be used to trap a vertical column of liquid at any selected level, including the bottom of the tank. (See Figures 4 and 5.)

NOTE An interface sampler is a specific type of zone/core sampler designed to trap a vertical column of liquid at the oil/water interface at the bottom of a tank, or at any other selected level, such as the interface of oil floating on ballast water in a ship's tank, but may also be used to withdraw zone samples from any selected level within a tank (see Figure 6).



Key

1 top valve opens as sampler is lowered through the liquid

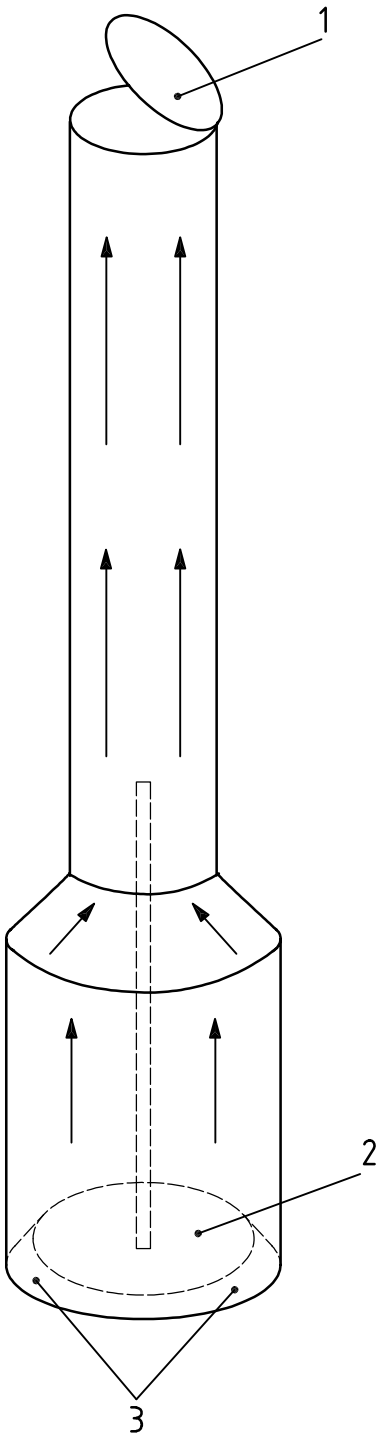
2 liquid outlet

3 bottom valve opens as sampler is lowered through the liquid

4 liquid inlet

NOTE In this example, both valves close when the sampler is raised. Other samplers may have only one valve (see Figure 6).

Figure 4 — Example of a zone/core sampler

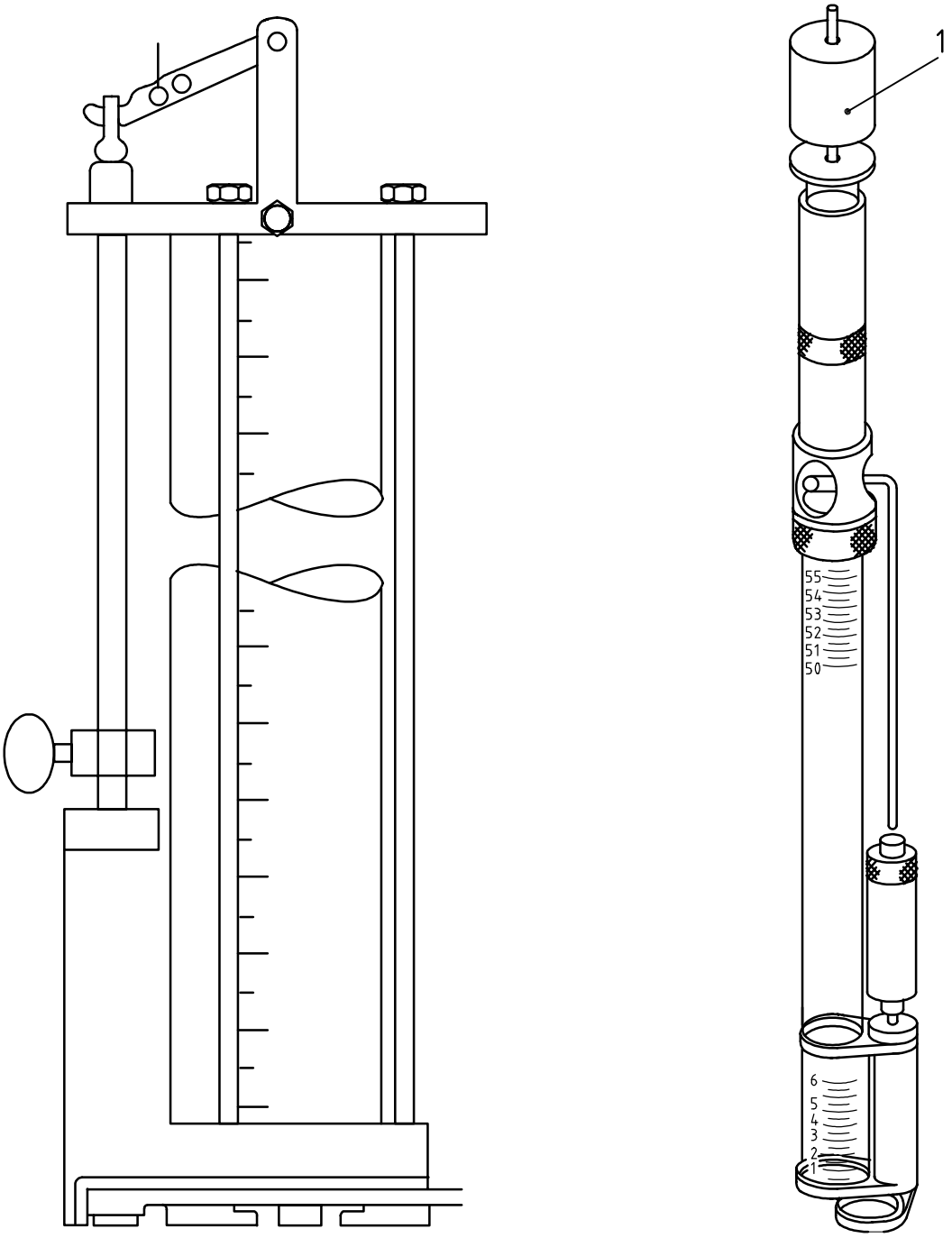


Key

1 upper flap valve
2 bottom valve
3 product flows through as sampler is lowered through the liquid

NOTE Both valves close when the sampler is raised

Figure 5 — Example of a zone/core sampler



Key

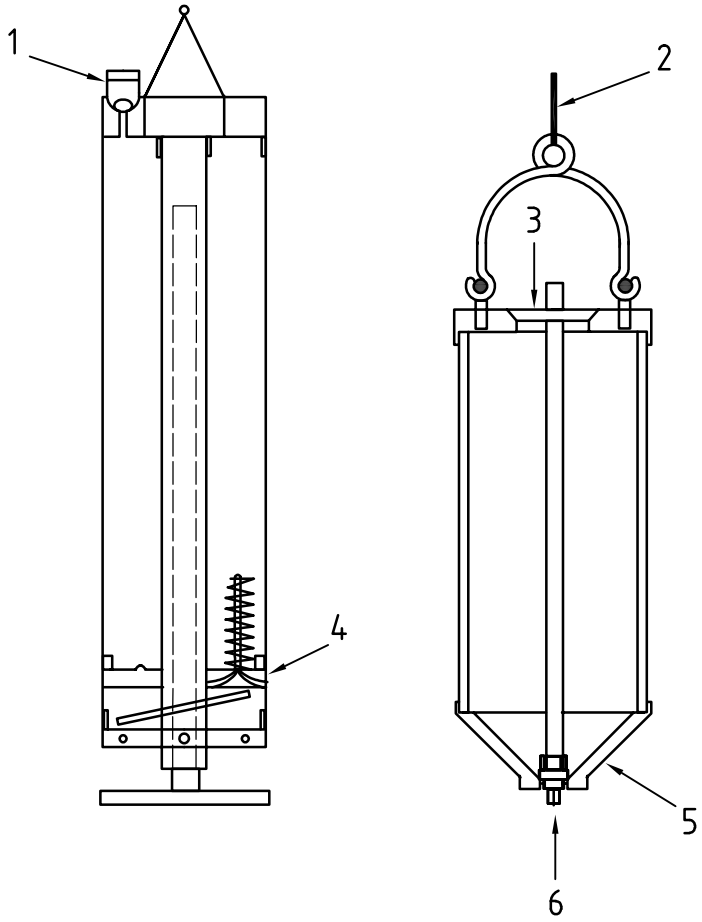
1 weight for triggering closing mechanism

Figure 6 — Examples of core/interface samplers

5.2.3 Bottom samplers

Bottom samplers are receptacles which can be lowered to the bottom of a tank where a valve or similar closure is opened by contact with the floor of the tank and closed on lifting (see Figure 7). Some designs of bottom samplers are available with an extendible "foot", to allow sampling just above a layer of sediment (that might otherwise prevent the leak-tight closure of the sampler).

NOTE Some designs of zone/core or interface sampler may also be used as bottom samplers.

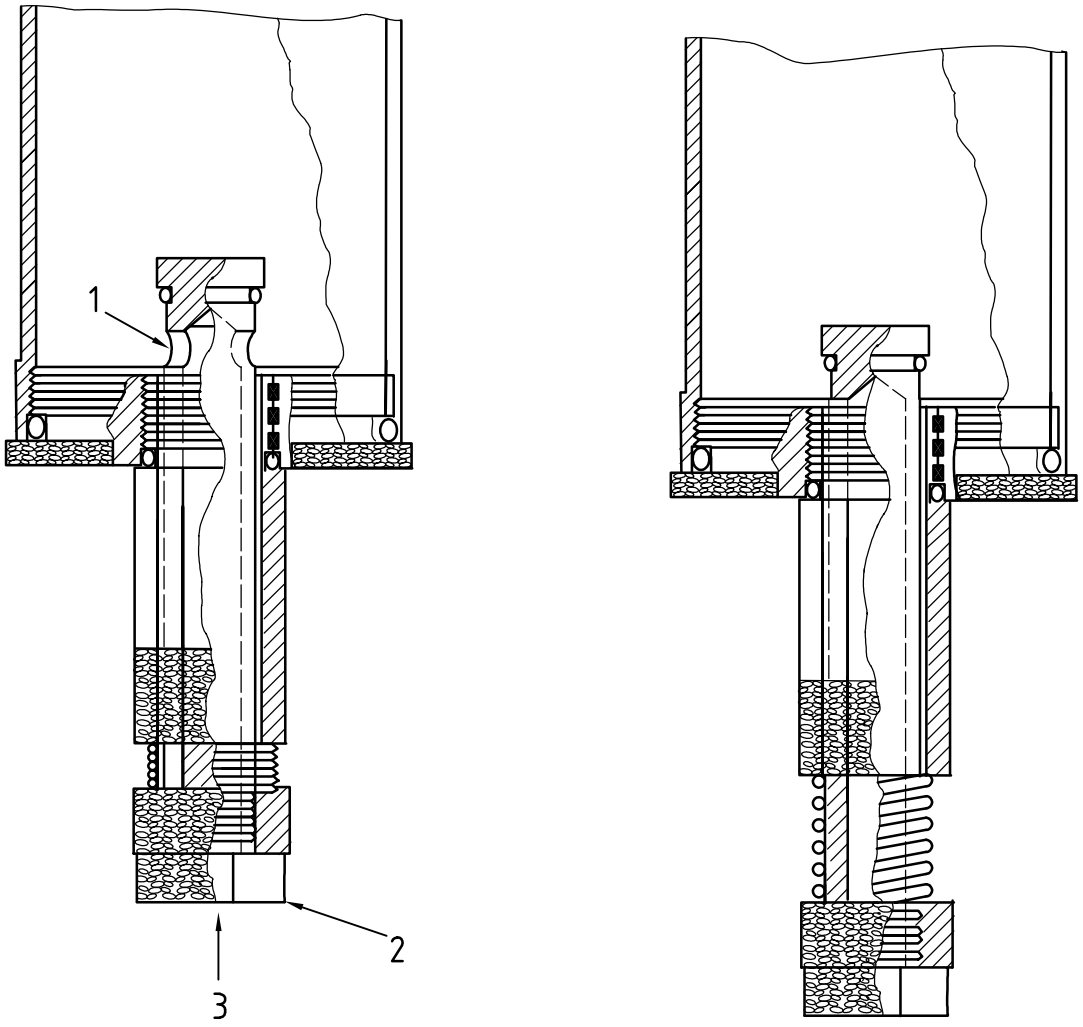


Key

1 ball valve/air outlet
2 line for lowering
3 air outlet
4 spring-loaded inlet valve
5 four lugs
6 weighted inlet valve

NOTE The foot of adjustable length triggers the opening and closing of the inlet valve.

Figure 7 — Examples of bottom samplers



a) Open position **b) Closed position**

Key

1 hole, allowing sample to enter sampler
2 bottom line
3 filling hole

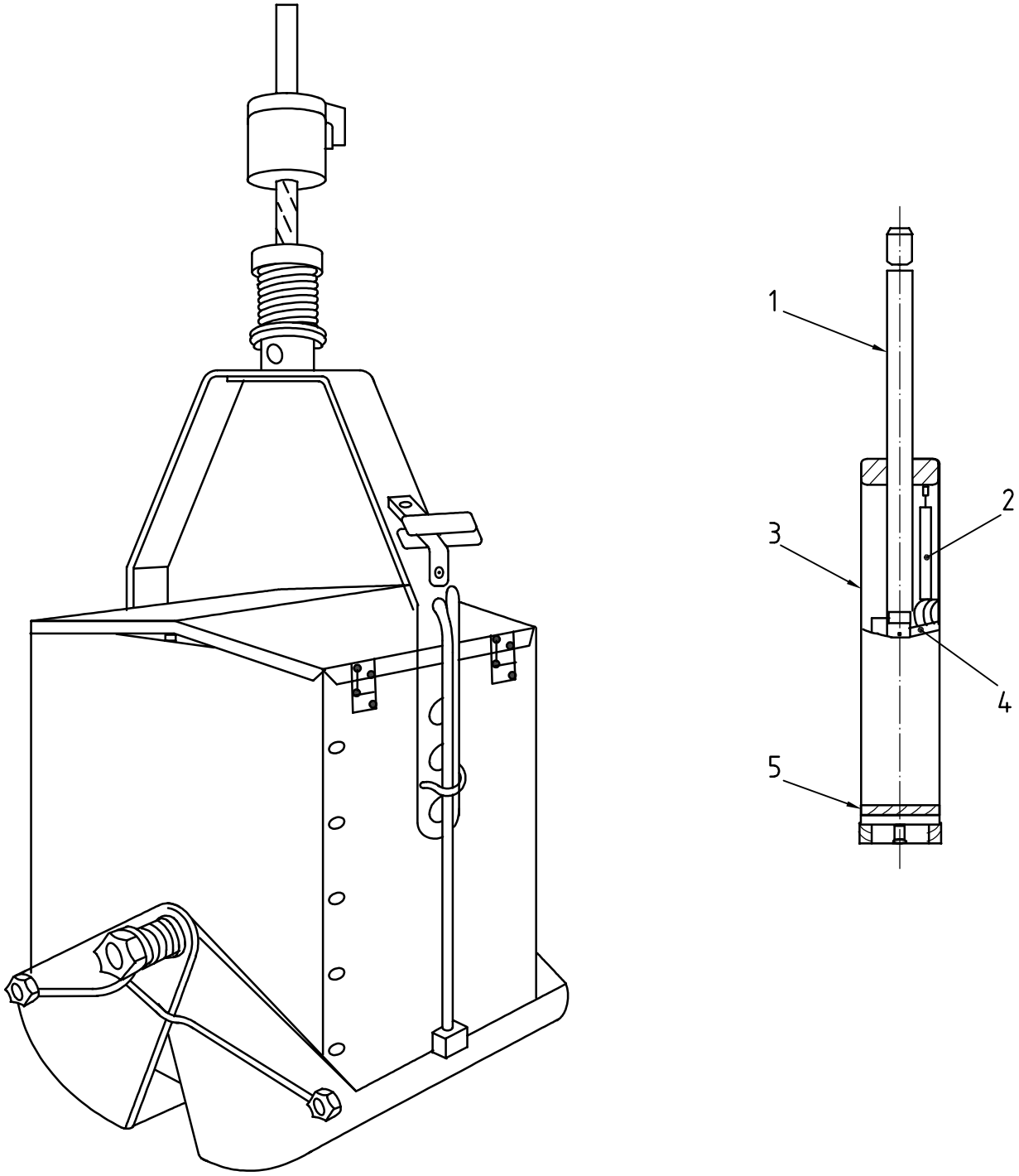
NOTE This part of Figure 7 shows the detail of the spring-loaded bottom inlet/closure mechanism of another design of proprietary bottom sampler.

Figure 7 — Examples of bottom samplers (*continued*)

5.2.4 Residue/deposit samplers

5.2.4.1 Deposit sampler

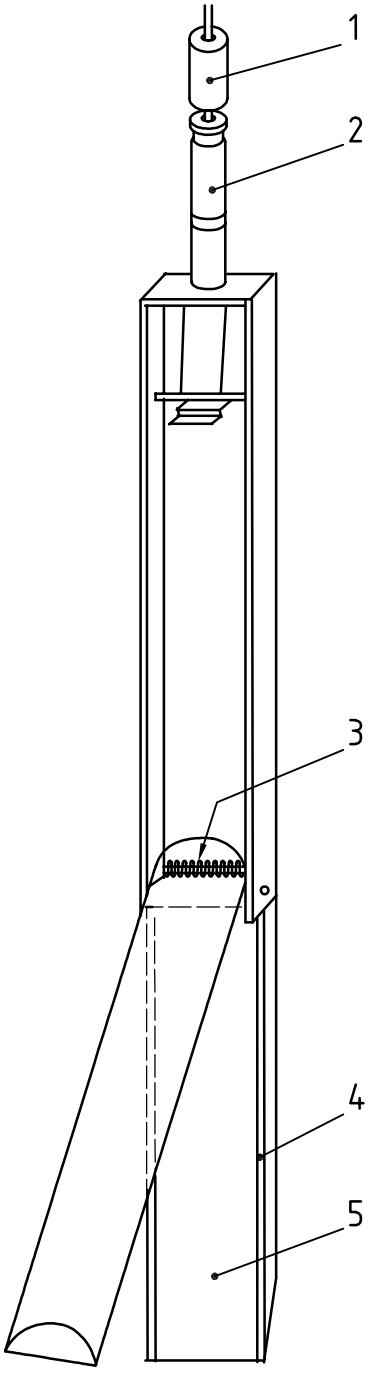
There are two types of deposit samplers in common use. They both rely on spring actuation of either a jaw closure device or suction device. Typical examples are shown in Figure 8.



Key

1 loading rod
2 loading spring
3 main cylinder
4 cylinder seal assembly
5 unloading valve

Figure 8 — Examples of spring actuated and plunger-type residue/deposit samplers



Key

1 weight for triggering closing mechanism
2 closing mechanism
3 closing spring
4 seal
5 heavily weighted sampler body

Figure 8 — Examples of spring actuated and plunger-type residue/deposit samplers (*continued*)

5.2.4.2 Gravitation/ram core sampler

This is a tubular device of uniform diameter either weighted or equipped with a mechanically operated driver to penetrate the deposit layer to be sampled.

5.2.5 Running samplers

A running sampler is a container equipped with a restricted filling device that obtains a sample whilst being lowered and raised through the liquid.

NOTE 1 It has not been established that running samplers fill at a uniform rate, because

a) the tank volume may not be proportional to the depth, or

b) the operator may not be able to raise (or lower) the sampler at the variable rate required for proportionate filling which is approximately proportional to the square root of the depth of immersion.

NOTE 2 A fixed volume running sampler may be based on a sampling-bottlecage or a weighted sampling can. Proprietary fixed volume running sampler devices are also available, which may be provided with a series of selectable orifices to suit different oil depths and viscosities.

NOTE 3 Alternatively, variable volume running samplers, where the capacity of the primary sample receiver increases with the distance travelled through the tank contents, may be available. Such devices are acceptable provided that they can be shown to fill at a consistent rate during travel through the liquid.

5.2.6 All-level sampling devices

An all-level sampler is a container equipped with a restricted filling device that obtains a sample when moved through the liquid in one direction only.

NOTE 1 It has not been established that running samplers fill at a uniform rate, because

a) the tank volume may not be proportional to the depth, or

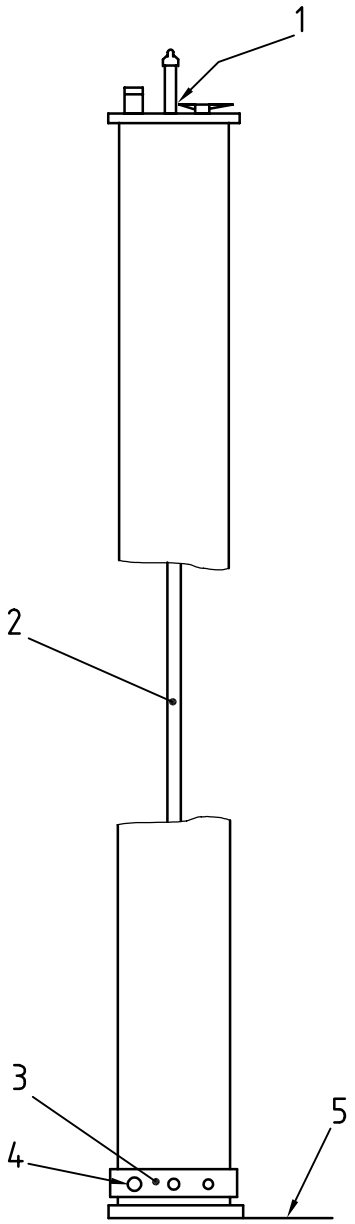
b) the operator may not be able to raise (or lower) the sampler at the variable rate required for proportionate filling which is approximately proportional to the square root of the depth of immersion.

NOTE 2 These devices may be similar to those devices used to take running samples or may be proprietary devices specifically designed to obtain this type of sample.

NOTE 3 A “bottom-up” fixed volume all-level sampler may be based on a sampling bottle/cage (or a weighted sampling can) which is lowered in the closed position to the bottom of the tank, opened, and the sample is collected as it is raised through the liquid.

NOTE 4 Proprietary “top-down” and “bottom-up” fixed volume all-level sampler devices are also available, which are provided with different opening or closing mechanisms. Additional means may be provided to restrict the inlet flow rate to suit different oil depths and viscosities. A typical example of a “top-down” fixed volume all-level sampler is illustrated in Figure 9.

NOTE 5 Alternatively, variable volume all-level samplers may be available. Such devices are acceptable provided that they can be shown to fill at a consistent rate during travel through the liquid.



Key

1 air exhaust opens as sampler fills during lowering
2 suspension rod
3 knurled ring with one orifice, used to select required inlet orifice in main sampler body, to vary speed of filling
4 range of different size inlet orifices in bottom section of main sampler body
5 contact line

NOTE The filling holes are closed by the bottom internal section rising when it makes contact with the tank bottom.

Figure 9 — Example of a "top down" all-level sampler

5.2.7 Restricted and closed system samplers

If safety and/or environmental constraints restrict the use of open tank sampling equipment and procedures, samples may be taken using restricted or closed system samplers operated via a vapour-lock valve. Such systems are particularly suitable for use on pressurized tanks, tanks where the ullage space is inerted, and/or tanks which are part of a vapour balancing/recovery system.

Proprietary samplers are available that are designed to take spot, zone, interface, bottom, running and all-level samples under restricted and/or closed conditions. The sampler is connected to a graduated sampling tape or cable, and inserted in a portable sampling device (PSD) which is fitted with a winding mechanism. The whole assembly is then connected to the vapour-lock valve, which replaces the traditional gauging hatch used in open sampling operations. A typical example of a closed system sampler and vapour-lock valve are illustrated in Figure 10.

Restricted equipment is designed to substantially reduce the release of vapour while the vapour-lock valve is open, but has no special facilities for subsequent sample handling or transfer operations once the vapour-lock valve has been closed. Thus, there may be a small release of vapour to the atmosphere.

Closed equipment is designed to be completely gas-tight at all times, to prevent any release of vapour to the atmosphere. Portable closed sampling devices are therefore normally provided with special facilities to

— ensure gas-tight connections, and

— allow sample transfer to secondary (transportation) receivers without vapour loss.

In addition, other facilities may be provided which permit

— vapours held up within the housing to be displaced back to the tank or to an absorbent canister, or

— the system to be purged with inert gas.

NOTE Closed system conditions do not permit the visual verification that running or all-level samples have been taken representatively in fixed volume sample receivers (i.e. that they are not completely full).

5.3 Drum and can samplers

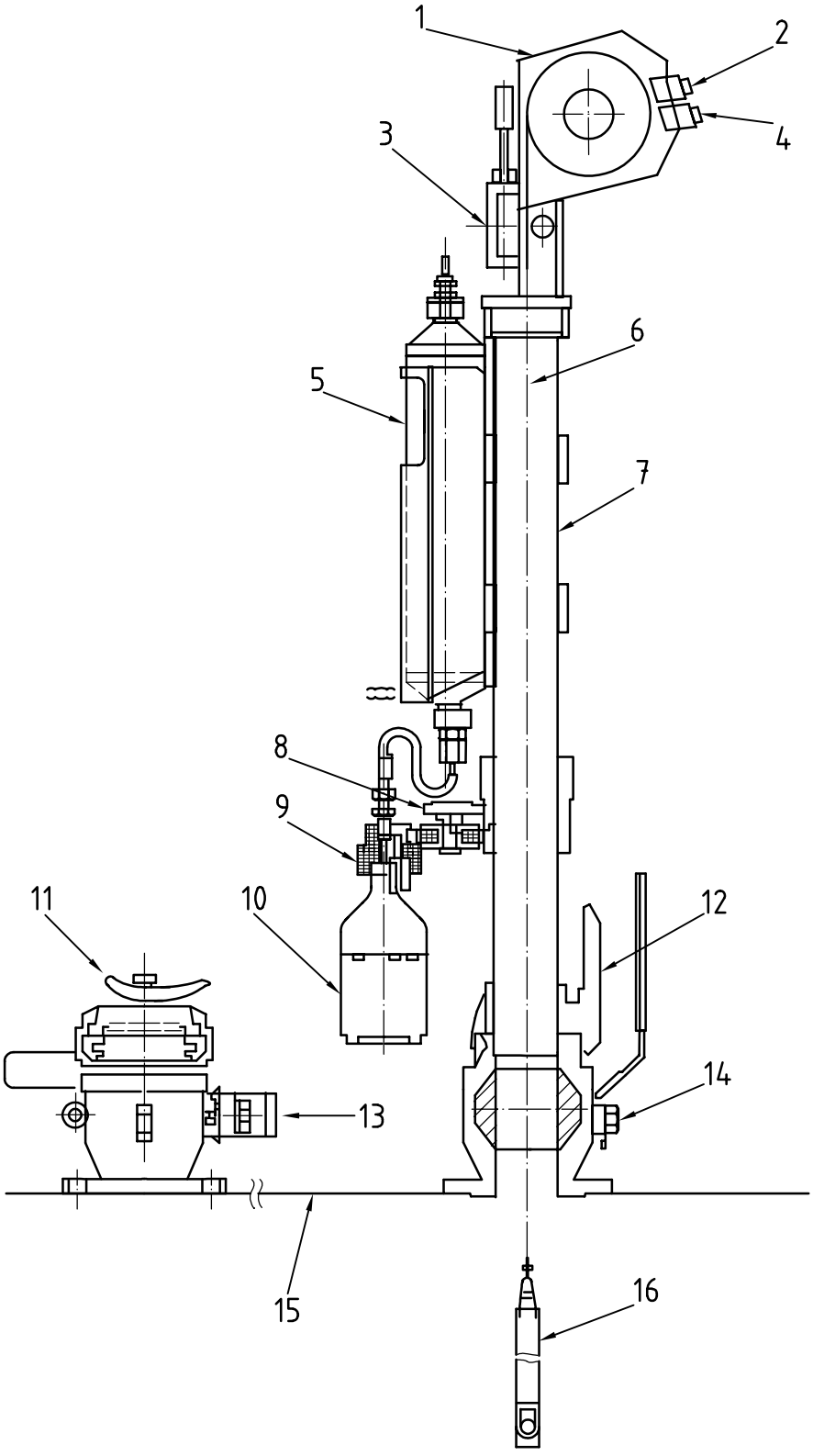
A tube sampler is commonly used for sampling from drums and cans (see Figure 11). This is a tube made of glass, metal or plastics material, with suitable fittings if required to facilitate handling.

A tube sampler may be used to obtain spot samples or bottom samples by temporarily closing the upper opening with the thumb before inserting it into a drum or can to the desired level. When the tube is at the desired level, the thumb is removed from the upper opening, allowing the tube to fill with the sample. Once the tube is filled, the upper opening is once again closed with the thumb, allowing the sample to be withdrawn with the tube sampler. The sample is then transferred to a secondary sample receiver.

Provided that the tube has a uniform cross-sectional area throughout its length, it may also be used to obtain zone or core samples from the whole liquid height within drums or cans. These samples are taken by lowering the tube sampler slowly in the open position so that the rate of sample in-flow equals the rate of descent. Once filled, the upper opening is sealed to allow removal of the tube sampler prior to the transfer of the sample to a secondary sample receiver.

A more sophisticated design of tube sampler, having a closure mechanism at the lower end, may be used similarly for taking a representative zone or core sample.

Drum pump or siphoning devices are not recommended for volatile oils due to the risk of light-end loss. Siphoning should never be attempted by mouth.



Key

1 carter winder
2 pressure valve
3 sight glass
4 relief valve
5 carbon filter
6 graduated tape
7 housing
8 transfer valve
9 transfer block
10 laboratory bottle
11 valve cover
12 quick-connect coupler
13 vapour-lock valve in closed position
14 vapour-lock valve in open position
15 tank top
16 sampling container

Figure 10 — Example of a lock valve and closed system sampler

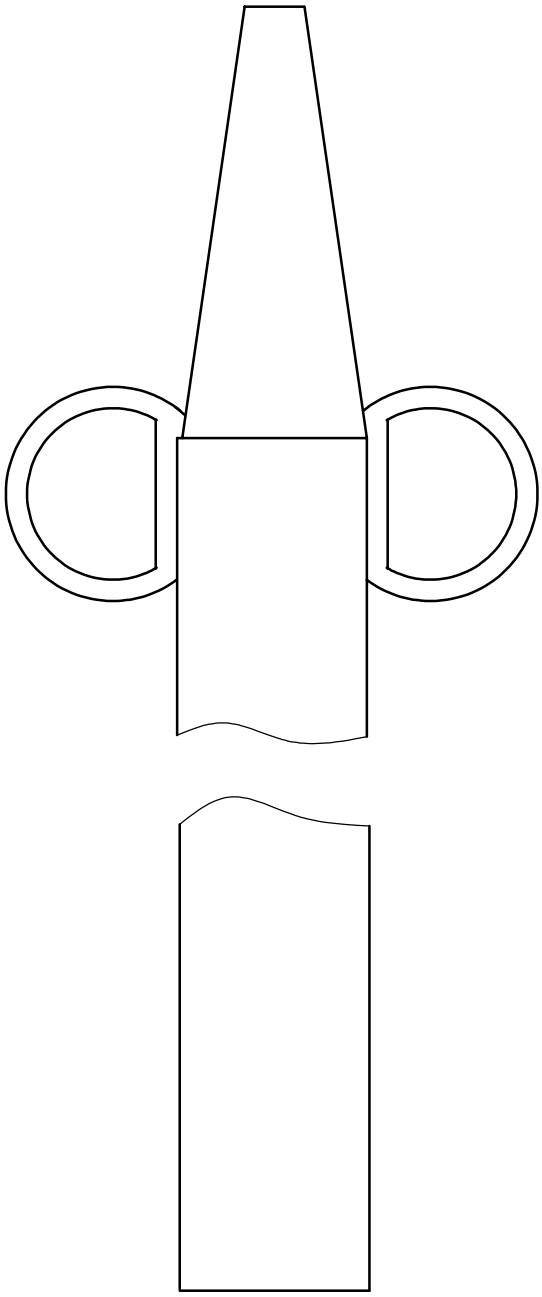


Figure 11 — Example of a tube sampler or thief

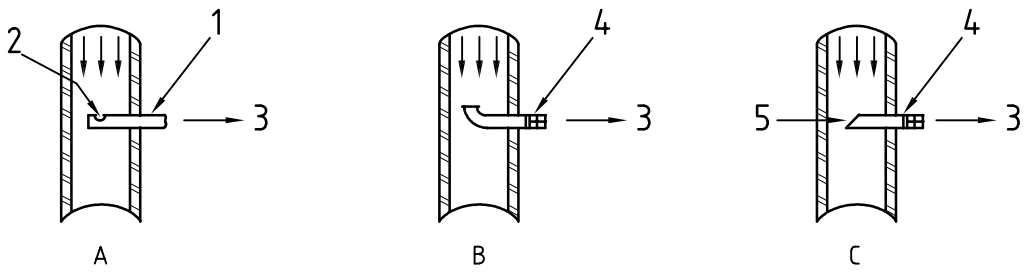
5.4 Pipeline samplers

5.4.1 If an automatic pipeline sampler is required, this shall be in accordance with ISO 3171.

5.4.2 A manual sampler consists of a suitable pipeline probe with an isolating valve. The probe shall extend into the pipeline so that the point of sample entry is not nearer to the pipe wall than a quarter of the internal diameter. The probe entry shall face into the direction of the flow that is being sampled within the pipeline, see Figure 12.

If fixed volume sample containers (e.g. bottles) are to be filled, the probe outlet valve shall have a delivery tube that will be long enough to reach the bottom of the sample container, allowing submerged filling.

If variable volume sample receivers (e.g. floating piston cylinders) are to be filled, the probe outlet valve shall have a delivery tube and connections which permit both the safe flushing of the probe and line contents, and the accumulation of sample in the receiver vessel.



Key

1 manufacturer's standard diameter
2 end of probe closed. Orifice facing upstream. 6,4 mm to 5 cm pipe or tubing
3 to valve
4 6,4 mm to 5 cm pipe or tubing
5 45° bevel

Figure 12 — Examples of probes for manual spot line samplers

5.5 Sample receivers, vessels and containers

Fixed volume sample containers may be glass or suitable plastics bottles, metal covered bottles, or cans, depending on the product to be sampled. Depending upon application, it may be necessary for metal receivers to be lined internally with a suitable coating. Lacquer lined cans and drums may be suitable.

Variable volume sample receivers may be designed for either low pressure or high pressure applications. Low pressure designs include collapsible plastics containers, bladders, and vessels fitted with a flexible internal diaphragm. High pressure variable volume sample receivers are pressure vessels with an internal piston, which moves against a gas buffer as the sample is accumulated against the other side of the piston. A typical example of a high pressure variable volume sample receiver (a floating piston cylinder) is illustrated in Figure 13.

NOTE Some designs of high pressure variable volume receivers have two pistons, which enable the sample to be mixed within the receiver (prior to subsampling) by repeatedly forcing it through a central mixing device (Figure 14).

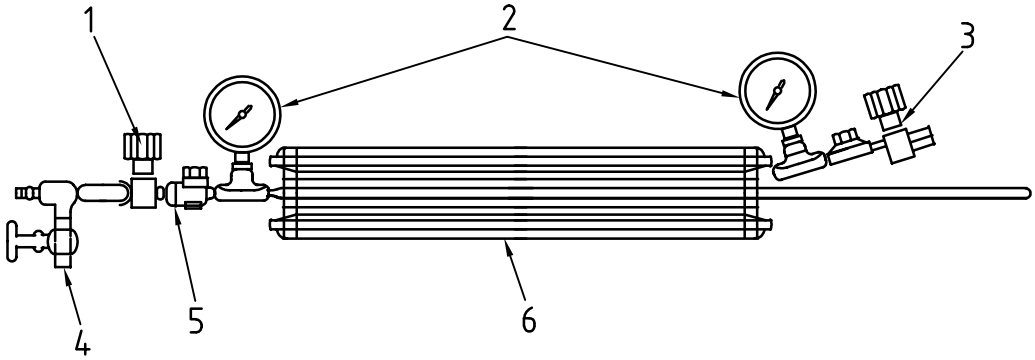
Before use, variable volume sample receivers are normally collapsed, evacuated or reduced to the nominal zero volume (by filling the opposite side of the diaphragm or piston with inert gas). The sample volume expands as the sample is accumulated, either by compressing the inert gas or by cautiously reducing the inert gas pressure.

The sample receiver/container size is dependent on the quantity required for analysis (and/or retention).

Prior to any use, it may be appropriate to rinse the sample receivers, vessels and containers with the fluid that is to be sampled, in order to avoid contamination of the current samples with any residue from previous samples and/or solvents used to clean the receivers.

Wherever possible, the sample should be transported to the laboratory in the vessel it was originally obtained in (the primary sample receiver), and therefore the methods which do not require sample transfer to a secondary vessel should be preferred.

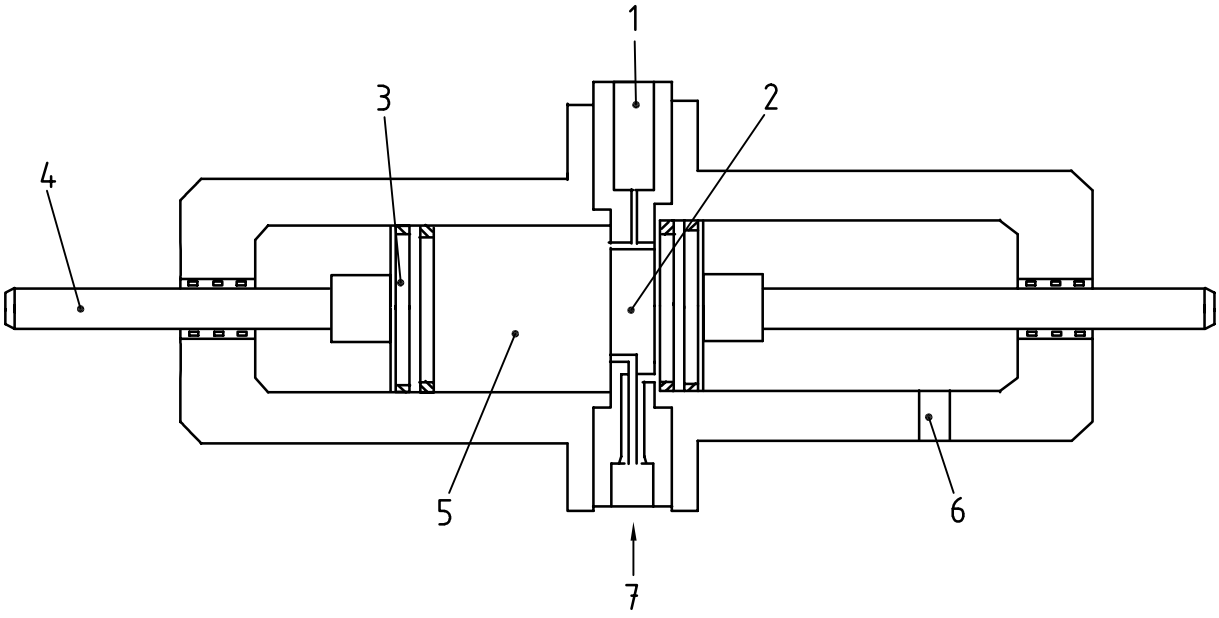
Plastics containers shall not be used for long-term sample storage unless it has been demonstrated that the plastic is suitable (i.e. compatible with the sample) so that the integrity of the sample is not compromised. The use of containers made of non-linear polyethylene may lead to sample contamination and/or sample container failure.



Key

1 fill valve
2 sample gauge
3 preload valve
4 exhaust valve
5 bursting relief
6 constant pressure cylinder

Figure 13 — Example of a single-piston variable volume constant pressure receiver



Key

1 subsampling port
2 mixing baffle
3 piston
4 piston indicator rod
5 sample chamber
6 inert gas port
7 sample inlet

Figure 14 — Example of a double-piston variable volume sample receiver

5.6 Container closures

Corks, or plastics or metal screw-caps may be used for closing fixed volume sample receivers/containers. Natural rubber stoppers shall not be used. Corks shall be of good quality and free from loose pieces or dust. They shall be softened by rolling or squeezing, and pressed well into the neck of the bottle to prevent leakage or evaporation. Where necessary, a protective cover of a suitable material shall be used.

Screw caps with compressible sealing inserts are recommended in preference to corks, when sampling volatile liquids.

Corks shall not be re-used for different types of product, since thorough cleaning is difficult and hydrocarbons may penetrate into the cork and cause contamination of subsequent samples. Re-use of corks is not recommended, but is permissible if the use is restricted to one type of product.

Screw-caps of cans or bottles shall be fitted with discs of cork faced with oil-resistant material, or a compressible plastics sealing insert. Cork discs shall only be used once. They shall be removed before the cleaning of screw-caps and replaced with a new disc prior to the cap being re-used.

Similar closures may be used with low pressure variable volume sample receivers, but high pressure variable volume receivers shall be fitted with suitable valves. Additional blanking cover caps may be provided to seal the valve connection points of high pressure receivers prior to their transportation between the sampling location and the laboratory.

5.7 Sample coolers

A cooling coil made of seamless copper tubing, or other suitable metal tubing, with an appropriate internal diameter, shall be fixed in an open, portable container in such a way that it is immersed in a water or water/ice mixture during use. The cooling coil alternatively shall be fixed within a closed container through which a cooling fluid is circulated.

The inlet end of the tubing shall be furnished with a flange or other appropriate means of connection to the sampling valve. The outlet end shall be open.

Sample coolers shall be efficiently flushed before collecting the cooled sample.

Care is required to ensure that the use of a sample cooler is appropriate (see 7.2.3.2).

6 Safety precautions

Careful consideration shall be given to the nature and known hazards of the products being sampled, which will affect the detailed nature of the precautions to be observed. Guidance on safety precautions is given in Annex A.

7 Procedures for homogeneous petroleum liquids

7.1 Introduction

This clause specifies the general procedures that shall be applied for sampling homogeneous liquids. Additional procedures for sampling crude oil and non-homogeneous liquids are described in Clause 8.

Unless otherwise specified, multiple-spot samples shall be collected using the detailed procedure relevant to the particular application. Typically they are upper, middle and lower; or, upper, middle and suction (outlet) samples.

The minimum number of samples shall be in accordance with Table 1.

NOTE When the liquid level is less than 4,5 m, it may be permissible to take fewer spot samples than shown in Table 1.

Table 1 — Spot samples — Minimum requirements

Liquid level	Required samples		
	Upper	Middle	Lower
$\leqslant$ 3 m		x	
$>$ 3 m and $\leqslant$ 4,5 m	x		x
$>$ 4,5 m	x	x	x

7.2 Precautions

7.2.1 General

7.2.1.1 A sample shall not include any product other than that to be sampled and, if it is necessary to transfer a sample from a primary sampler to a secondary sample container, appropriate precautions shall be taken to preserve the integrity of the sample. Thus, steps shall be taken to avoid sample contamination (e.g. by rainwater or perspiration), and the sampling method shall, whenever practical, avoid sample transfer by permitting the sample to be transported to the laboratory in the container it was originally obtained in (the primary sample receiver).

NOTE The transfer of a sample will often have the following effects:

a) loss of light ends (affecting density and vapour pressure);

b) changes in the relative proportions of oil and contaminants such as water and sediment.

7.2.1.2 Sampling personnel shall be fully instructed in the relevant procedures for the particular sampling application. Specific precautions are necessary when drawing samples for certain tests and the correct sampling procedures shall be closely followed to ensure that the test results are meaningful.

These additional precautions do not form part of this International Standard, but should be set out in the test method or product specification concerned.

7.2.1.3 Samples shall not be drawn from unperforated or unslotted still-wells, guide poles or stand pipes since the contents of an unperforated pipe are not normally representative of the bulk contents of the tank at the same depth or location outside the pipe.

Still-well, guide pole or stand pipe samples shall only be drawn from pipes that have perforations or slots that allow the free flow of product into and out of the pipe.

NOTE A row of perforations, typically with a diameter of 25 mm and spacing of 300 mm, or two rows of overlapping slots of similar width are normally sufficient to allow the free flow of the product into and out of the pipe.

7.2.1.4 For handling samples, use sampling equipment, containers, receivers or samplers that are impervious to and resistant to solvent action by the product handled (see 5.1).

7.2.1.5 Thoroughly inspect all sampling equipment, including closures, to ensure that it is clean and dry.

7.2.1.6 Leave a minimum of 5 % ullage in the container to allow for expansion. If spot samples are drawn from a tank, some of the sample has to be removed from the sample container; this shall be done immediately after the sample container has been withdrawn from the tank.

NOTE 1 Decanting to obtain the ullage is not good practice because it may invalidate the representativity of the sample, particularly if any free water or an emulsion layer is present; however, it cannot always be avoided.

NOTE 2 The ullage requirement only applies to fixed volume sample receivers and containers, not to variable volume sample receivers.

7.2.1.7 Immediately after filling and closing the sampler, sample receiver or container, examine it closely for leaks.

7.2.1.8 If large-volume samples are required which may not, because of volatility or other considerations, be obtained by the bulking of smaller quantities, mix the tank contents thoroughly by the means available (e.g. circulation, tank side mixer). Confirm the homogeneity by tests on samples taken at sufficiently different levels, as described in 4.2 and 8.2.1. The container shall be filled using a sample inlet extending to near the bottom of the container, from tank-side taps, or from a circulation-pump bleed-valve sample point.

7.2.2 Samples for special analysis

If samples are taken for the determination of trace elements, for example lead, specially prepared sample containers may be required. Take such samples directly in the prepared container. Ancillary equipment and sampling cord used shall in no way contaminate the sample. Special care is required to prevent accidental contamination (e.g. by sampling cord being wetted with sea water on a ship's deck prior to being used to sample a product, where the sodium content is a critical analysis in the purchasing specification).

If the tests for which the sample is intended include certain specific requirements, such as copper or silver strip corrosion, take the sample in dark-coloured glassware (or containers of other suitable material), and protect the sample from light prior to testing.

NOTE Any other method of obtaining the sample could affect the results of the corrosion test.

If tests for such properties as water-separation characteristics, oxidation stability, existent gum, etc., are required, then take care to ensure that any sample container has been suitably prepared and is entirely free from contaminants such as flux or other chemicals.

7.2.3 Volatile products

7.2.3.1 When taking samples of volatile crude oils and products, if it is necessary to avoid the loss of light ends, for example for determination of density, vapour pressure or distillation, do not transfer, composite or bulk oil from the original sample container(s). Transport and store the sample in an inverted position to avoid loss of light ends through the closure.

7.2.3.2 Depending on the nature and temperature of the liquid, the ambient temperature and the purpose for which the sample is required, some or all of the following precautions may be necessary:

a) passing the sample through a sample-cooler at the point of sampling;

b) cooling the sample container to a suitable temperature before sampling;

c) keeping the sample container cool until it has been sealed;

d) keeping the sample container cool until it is delivered to the laboratory.

Sample containers may be cooled, if necessary, by immersion in a cooling medium, e.g. crushed ice, but care is required if cooling the sample could result in the partial separation of wax and/or other heavy components.

Cooling of such samples may result in wax or heavy components being deposited on the walls of the sample container so that subsequent subsamples may no longer be representative of the total original sample. Crude oil samples should not be cooled below a temperature of 3 °C above their wax-appearance temperature. In instances where the wax-appearance temperature is greater than ambient, it would be necessary to warm an original sample before subsampling.

7.2.4 Tank side and pipeline sampling

If a tank side or pipeline sampling point is used, adopt the following additional sampling precautions.

a) Before taking tank side or pipeline samples, completely flush the sampling line to ensure removal of all previous contents of the line.

b) When filling fixed volume sample containers, the sample line outlet shall be designed to extend to near the bottom of the sample container during sampling. When filling variable volume sample receivers, facilities shall be available to flush the whole sample line volume to the receiver inlet valve.

c) If the product being sampled is volatile, cool the sample container to a suitable temperature and use a sample cooler (see 5.7), if necessary and appropriate (see 7.2.3.2).

d) If the oil being sampled has a high pour point, it may be necessary to insulate the sample line thermally or to provide means of heating the sampling connections in order to prevent solidification.

7.2.5 Labelling and transport

7.2.5.1 Clearly label sample containers; tie-on labels are preferred. Use indelible marking on the labels.

It is recommended that the following particulars be included in those recorded:

— place at which sample was drawn;

— date;

— initials or other identification mark of operator;

— description of the product;

— quantity represented by sample;

— tank number, package number (and type), name of ship;

— type of sample;

— sampling device or sampler used;

— any additional sampling details.

7.2.5.2 If samples are to be dispatched, all appropriate transportation regulations shall be adhered to. Care shall be taken to ensure that any packaging material does not contaminate the sample when it is subsequently opened.

7.3 Tank sampling

7.3.1 Shore tanks

7.3.1.1 Vertical cylindrical tanks

7.3.1.1.1 Spot samples

Lower the sampling device until its opening is at the required depth, open it in the appropriate manner and maintain it at the required level until it is filled. Retrieve it and either decant a small portion back to the tank to create an ullage space before sealing the sample, or carefully transfer the complete sample to a secondary sample receiver.

In hot climate conditions or whenever there is a significant difference in temperature between the sampler and the product being sampled, the sampler should be conditioned to the temperature of the tank contents by gently raising and lowering it through approximately ± 300 mm for 1 min to 2 min before operating the opening mechanism.

When sampling at different levels, take the samples in sequence from top to bottom, in order to avoid disturbance at a lower level.

In the case of a zone sampler (with essentially full bore top and bottom valves that allow the tank contents to flush through the sampler as it is lowered), lower the sampler in a controlled manner until it is at the required depth. Close the valve(s) as soon as the lowering operation ceases, and retrieve the sample immediately. Carefully transfer the complete sample to a secondary sample receiver.

Where the design of the zone sampler does not permit full flushing during lowering, it is recommended that the sampler be raised and lowered two or three times after reaching the spot sampling location, before closing the valve(s). The raising and lowering shall be through a distance of at least the height of the sampler.

In the case of a top sample, lower the open sampler/container carefully until its neck is just above the surface of the liquid, and then allow the sampler to fall sharply 150 mm below the surface. When the sampler is full, as indicated by the cessation of air bubbles, withdraw it and proceed as for regular spot samples.

7.3.1.1.2 Composite samples

A composite sample may be prepared from representative subsamples of spot samples obtained from within a single tank (e.g. by combining subsamples from the upper, middle and lower spot samples), or may be prepared by combining subsamples representative of individual tanks to provide a composite for a larger oil quantity (e.g. several ship or barge tanks of the same product). Composite samples shall include all the material collected in the primary sampling device without subdivision. The volume collected in the primary sampling device shall be chosen to allow the entire contents of the device to be added to the volume of the other subsamples in the transport container. Compositing of subsamples smaller than the entire contents of a subsample shall only be performed in a laboratory with facilities to assure adequate mixing and measurement of the subsamples.

To prepare either type of composite sample, transfer subsamples of representative individual samples into a composite-sample container, and mix them together gently. The subsamples shall be volume weighted in proportion to the quantity that each represents.

When the subsamples that are to be combined originate from a tank of non-uniform cross-sectional area (or from multiple tanks), the compositing operation shall require careful calculation and measurement of the subsamples to maintain the representative nature of the sample. These operations shall, if practical, be conducted under controlled laboratory conditions.

NOTE Evaporation of light ends, and adhesion of water/sediment to the wall of the original sampler, may influence the representative nature of composite sample(s) (see also 7.2.3).

Do not prepare composite samples for testing unless they are specifically requested, and agreed by the interested parties. As an alternative to physical compositing, individual spot samples shall be tested and a mean value calculated from the individual test results in proportion to the bulk represented by each sample.

7.3.1.1.3 Bottom sampling

Lower the bottom sampler until it rests in an upright position on the bottom of the tank so that the valve opens and the sampler fills. After withdrawing the sampler, examine it closely for leaks. If any are detected, discard the sample, clean the bottom sampler and re-sample. If necessary, transfer the contents into a secondary sample container, taking care that all the sample is properly transferred, including any water or solids that may adhere to the inner walls of the primary bottom sampler.

7.3.1.1.4 Interface sampling

Lower the sampler with the valves open to permit the liquid to flush through the device. At the level desired, close the valve(s) and withdraw the sampler from the liquid.

If a transparent tube is used, any existing interface can be detected visually through the wall of the sampling tube and its position within the tank determined by measurement on the graduated sampling tape. Check that the valves have properly closed; otherwise re-sample.

NOTE The sample may be retained for testing.

7.3.1.1.5 Tap (tank-side) sampling

This is not a preferred method for custody transfer or yield inventory purposes and therefore shall be applied only if no other method of sampling is possible.

The sampling point valves shall be a minimum of 12,5 mm in diameter and shall be fitted to the side of the tank at regular intervals, by connections extending at least 150 mm into the tank, except on floating-roof tanks, in which this is impossible. The lower connection shall be level with the bottom of the suction pipe (see also 7.2.4).

Before a sample is taken, flush the tap or valve connection with the product to be sampled, after which draw off a sample into a container or receiver.

CAUTION — Open the taps with care when sampling under pressure. Make no attempt to clear a blocked connection by rodding through an opened valve.

If the contents of a tank fail to reach the upper or middle sample connections on a tank equipped with three connections, take the sample for the tank as follows.

a) If the level of the contents is nearer the upper sample connection than the middle one, take two-thirds of the sample from the middle connection and one-third from the lower one.

b) If the level is nearer the middle connection than the upper, take one-half of the sample from the middle connection and one-half from the lower one. If the level of the contents is below the middle sample connection, take all the sample from the lower connection.

7.3.1.1.6 All-level sampling

Refer to 5.2.6 for descriptions of the different types of equipment that are available. All-level samplers can be of the “top-down” or “bottom-up” type. As the sample receiver is filled while travelling in a single direction through the tank’s contents, different procedures are required for “top-down” and “bottom-up” techniques.

Follow the manufacturer’s instructions when using proprietary devices.

To obtain a (bottom-up) all-level sample with a bottle fitted to a weighted sampling cage (or with a weighted sampling can), proceed as follows. Stopper the bottle or can, and lower it to the tank bottom (avoiding any bottom-lying free water). Jerk the cord to remove the cork, and raise the sampler back to the surface at a uniform speed without any pause or hesitation. The hauling speed should be chosen so that the bottle or can is about 80 % full, but not more than 90 % full, when withdrawn from the liquid. Cap or stopper the bottle immediately, or carefully transfer the complete sample from the weighted can to a secondary transportation receiver (see also 5.2.6).

If a fixed volume all-level sampler is less than 90 % full when retrieved from the liquid, it may be assumed that oil was flowing into it from all depths during its passage through the tank's contents. If the sampler is more than 90 % full when retrieved from the liquid, the sample may not be representative and should be discarded before re-taking the sample using a faster raising speed.

NOTE 1 The use of fixed volume all-level samplers is not a preferred method for custody transfer or yield inventory applications, as such devices may not fill at a uniform rate. Additionally, the operator may not be able to lower or raise the sampler at the rate required for proportional filling, which is approximately proportional to the square root of the depth of immersion.

Variable volume all-level samplers shall be designed to fill in proportion to the distance travelled through the tank contents.

Refer to 5.2.6 for sample acceptance criteria.

NOTE 2 Care is required with the operation of bottom closing “top-down” types of all-level sampler if a layer of free water can be present at the tank bottom. Some designs of proprietary sampler include an adjustable extension “foot” to trigger closure just above the level of any free water.

7.3.1.1.7 Running samples

Refer to 5.2.5 for descriptions of the different types of equipment that are available.

NOTE The use of fixed volume running samplers is not a preferred method for custody transfer or yield inventory applications, as such devices may not fill at a uniform rate. Additionally, the operator may not be able to lower or raise the sampler at the rate required for proportional filling which is approximately proportional to the square root of the depth of immersion.

To obtain a running sample with a bottle fitted to a weighted sampling cage (or with a weighted sampling can) equipped, if necessary, with a suitable device to restrict the filling rate, proceed as follows. Lower the open can or bottle/cage from the surface of the liquid to the tank bottom (avoiding any bottom-lying free water), and raise it back to the surface at the same uniform speed and without hesitation when changing direction. Select the inlet restriction orifice size and/or the rate of raising and lowering so that the bottle or can is about 80 % full, but not more than 90 % full, when withdrawn from the liquid. Cap or stopper the bottle immediately, or carefully transfer the complete sample from the weighted can to a secondary transportation receiver (see also 5.2.5).

To obtain a running sample with a proprietary running sampler, select the inlet restriction orifice size required for the liquid depth and viscosity, and follow the manufacturer’s instructions. If the sample quantity is more than 95 % of the sampler capacity, discard the sample and select a different size inlet orifice and/or change the speed of lowering and raising to achieve the target sample volume.

If a fixed volume running sampler is not more than 90 % full when retrieved from the liquid, it may be assumed that oil was flowing into it from all depths during its passage through the tank's contents. If the sampler is more than 90 % full when retrieved from the liquid, the sample may not be representative and should be discarded before retaking the sample using a smaller orifice restriction and/or a faster raising and lowering speed. Care is required during the operation of running samplers if a layer of free water is present at the tank bottom.

Free water should not normally be included in such samples, but should be quantified separately by gauging or by bottom sampling with an interface sampler.

7.3.1.2 Horizontal tanks with circular or elliptical cross-sections

Except when noted otherwise, samples shall be taken as spot samples as described in 7.3.1.1.1 from the levels indicated in Table 2. If they are to be combined to give a composite sample as described in 7.3.1.1.2, combine them in the proportions given in Table 2.

NOTE By mutual agreement, a single spot sample at the location corresponding to 50 % of the contained volume may be considered sufficient. Alternatively, it may be acceptable to use one of the other methods described in 7.3.1.1.

Table 2 — Sampling from horizontal cylindrical tanks

Liquid depth (percentage of diameter)	Sampling level (percentage of diameter above bottom)			Composite sample (proportional parts)		
	upper	middle	lower	upper	middle	lower
100	80	50	20	3	4	3
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

7.3.1.3 Tanks with other geometrical shapes

Sample spherical tanks and tanks of irregular shape by taking spot samples as described in 7.3.1.1.1. Determine the actual levels at which the samples are to be taken to allow for the volume distribution over the height of the tank.

7.3.1.4 Tanks fitted with vapour-lock valves

7.3.1.4.1 Use a portable sampling device (PSD) which is compatible with the vapour-lock valve that is installed on the tank, or use a suitable gas-tight adapter. Select the correct sampler for the type of sample that is to be taken (i.e. spot, zone, bottom, interface, running, or all-level) and attach it to the hanging device on the PSD tape/cable. Set any associated sampling valve triggering device and, in the case of running or all-level samplers, select the desired inlet orifice restriction device.

7.3.1.4.2 Confirm that the vapour-lock valve is fully closed before removing the protective cover/cap. Check that the datum surface of the valve and the matching datum surface of the PSD are both clean and free from any foreign material which could prevent the PSD from seating correctly and providing a gas-tight seal.

NOTE Earth continuity between the PSD and the tank structure will also normally be provided via this contact, but a separate earthing connection may also be provided.

7.3.1.4.3 Fit the PSD to the valve, and ensure that it is seated correctly before tightening the coupler device to lock it in position. Where the PSD is provided with a separate earthing connection, connect it to a suitable part of the tank structure to ensure earth (grounding) continuity.

7.3.1.4.4 Open the vapour-lock valve fully and carefully lower the sampler into the tank by unwinding the PSD tape or cable winding handle.

Depending upon the type of sampler used, (i.e. spot, running, all-level, etc.) take the sample(s) in accordance with the procedures specified in 7.3.1.1. When retrieving the sample, ascertain that the sampling device is fully withdrawn above the vapour-lock valve, before closing the valve. After retrieving the sample, ensure that the vapour-lock valve is fully closed before opening the PSD and/or transferring the sample to a secondary receiver.

7.3.1.4.5 When the sample has been obtained with a restricted system PSD, it shall be handled in exactly the same way as the equivalent open samples (see 7.3.1.1) and transferred to a secondary transportation receiver if necessary.

When the sample has been obtained with a closed system PSD, it shall be transferred completely via the gas-fight PSD housing to a fixed or variable volume transportation receiver.

NOTE It is important to verify the cleanliness of the internal components of the PSD housing (as well as the sampler) before re-using either a restricted or closed system PSD.

7.3.1.5 Pressurized tanks with valved sample points

Some pressurized tanks such as LPG Spheres, Bullets, etc. may be fitted with probes to enable sampling from different depths within the tank contents. Alternatively, other valved connections to pressurized tanks may be suitable for taking samples. In either case, use one of the pressurized receiver sampling methods described in 7.4.3.

7.3.2 Tanks on ships or barges

7.3.2.1 General

If open sampling procedures are acceptable, use the procedures detailed in 7.3.1.1 in conjunction with 7.3.2.2 to 7.3.2.4. If restricted or closed system sampling is required, follow the procedures detailed in 7.3.1.4 in conjunction with 7.3.2.2 to 7.3.2.4.

Safety and environmental regulations may limit the release of hydrocarbons to the atmosphere during ship and barge cargo operations. This has resulted in the restriction and, in some cases, the prohibition of traditional methods of obtaining cargo samples via open gauge-hatches or sighting ports. Thus, it is now a common condition specified in ship chartering agreements that ships should have facilities for restricted or closed system measurement and sampling, and that access to the cargo tanks should only be via vapour-lock valves.

The installation of vapour-lock valves should be in accordance with the requirements of the ship's Classification Society and the appropriate port authorities.

NOTE The total load capacity of a vessel is normally subdivided into a number of compartments which may vary in size and geometry. Some tank compartments may not have a uniform volume-to-height ratio, and thus some types of sample may not be representative. In these circumstances, spot samples from each compartment are preferred; however, in practice, time restraints associated with shipping operations normally necessitate the taking of all-level or running samples.

7.3.2.2 Sampling non-inerted, non-pressurized vessels

If open sampling procedures are acceptable, use the procedures described in 7.3.1.1.

If restricted or closed system sampling is required, use one of the procedures described for sampling tanks fitted with vapour-lock valves (see 7.3.1.4).

7.3.2.3 Sampling inerted, but depressurized vessels

For sampling inerted but depressurized vessels, the same procedures and precautions apply as detailed in 7.3.2.2.

7.3.2.4 Sampling inerted and pressurized vessels

For sampling inerted and pressurized vessels, use an appropriate restricted or closed system sampling device (see 5.2.7 and 7.3.1.4).

7.3.3 Railcars

For railcars, if open sampling is acceptable, use the procedures described for sampling horizontal cylindrical tanks (see 7.3.1.2). If restricted or closed system sampling is required, use one of the procedures described for sampling tanks fitted with vapour-lock valves (see 7.3.1.4).

If it is agreed that it may be acceptable to sample from a limited number of railcars in a train that all contain nominally the same material, samples should be drawn from railcars that are selected using a sampling plan in accordance with the general procedures described in 11.1.4.

7.3.4 Road vehicle tanks

For road vehicle tanks, if open sampling is acceptable, use the procedures described for sampling horizontal cylindrical or other geometry tanks as appropriate (see 7.3.1.2 and 7.3.1.3). If restricted or closed system sampling is required, use one of the procedures described for sampling tanks fitted with vapour-lock valves (see 7.3.1.4).

7.4 Pipeline sampling

It is often necessary to obtain manual dynamic pipeline samples, e.g. for instrumentation verification and quality control purposes. It should be noted that such samples are spot samples which may or may not be representative of the bulk transferred quantity.

7.4.1 Non-homogeneous liquids

For sampling non-homogeneous liquids, see Clause 8.

7.4.2 Homogeneous liquids

Sampling of homogeneous liquids shall be performed using a suitable pipeline sampling apparatus (see 5.4.2). Before a sample is drawn, flush the sample line and valve connection with the product to be sampled, after which draw off a sample into a sample container or receiver, taking into account the precautions given in 5.4, 7.2.4 and 7.4.3 as appropriate.

The contents of pipelines may be under considerable pressure and therefore special procedural precautions and equipment may be necessary (see Clause 6). It is recommended that a pressure gauge be provided in the line at each sampling point to enable the pressure to be read before sampling. The line service should be clearly labelled and updated on any change of service.

7.4.3 Spot sampling of high vapour pressure liquids

7.4.3.1 Sampling into single-piston variable volume sample receivers

7.4.3.1.1 Principle

The liquid is sampled at line pressure and maintained at line pressure (or above) during transportation and subsequent subsampling. A typical single-piston receiver is illustrated in Figure 13. The sample is accumulated against one side of the floating piston by carefully reducing the pressure of an inert gas buffer on the opposite side of the piston.

7.4.3.1.2 Receiver selection and leak testing

Select a receiver with the required capacity and a rated working pressure which exceeds the pipeline pressure. Confirm that the piston seal elastomer is compatible with the pipeline liquid, and is capable of operating effectively at the pipeline temperature. Ensure that the receiver is clean and dry.

Pressurize both sides of the receiver with inert gas to at least 100 kPa (1 bar) more than the anticipated pipeline pressure, and test for leaks. Test the receiver piston seals for leakage by pressurizing each side in turn to the same pressure while the other side is open to atmosphere. If a valve, fitting or seal is found to leak, replace it and re-test, or use another receiver.

7.4.3.1.3 Pre-charging the receiver

Open the sample inlet valve. Connect the inert gas side of the receiver to a supply of the appropriate inert gas and slowly pressurize it to at least 100 kPa (1 bar) above the pipeline pressure, so that the piston is fully displaced against the sample inlet end plate. Close all valves and disconnect the inert gas supply. Transport the pre-charged receiver to the sample point.

7.4.3.1.4 Purging the sample point lines and receiver

Connect the pre-charged receiver sample inlet valve to the sampling point.

If the receiver has only a single sample inlet connection, keep the sample inlet valve closed and flush the sample probe and line up to the inlet valve to a closed drain connection (or other safe disposal route such as a flare line).

Some receivers are provided with an additional end plate connection which enables the purging of the receiver sample side dead volume. With this type of receiver, purge the total hold-up volume by connecting the second valve to the disposal line and opening both valves. When the system is fully purged, close the second valve (i.e. the outlet valve) and control the filling by gently reducing the inert gas pressure from the other end of the receiver (7.4.3.1.5).

7.4.3.1.5 Filling the receiver

When filling a single sample inlet receiver, slowly open the inlet valve. A double sample inlet receiver will be ready to fill as soon as the second sample end valve is closed on completion of purging.

The piston shall not move at this stage due to the buffer gas pressure being higher than the pipeline pressure. If the piston does move, reject and use another receiver. Cautiously crack open the inert gas end valve to gradually reduce the inert gas pressure and allow the receiver to fill to no more than 80 % of nominal capacity.

The pressure difference between the sample and inert gas buffer sides of the piston should not exceed 100 kPa (1 bar) at any time during sampling.

Close the sample inlet valve and de-pressurize the sample line to the drain before disconnecting the receiver from the sample point. Finally connect the inert gas inlet valve to an inert gas supply and increase the inert gas pressure to at least 100 kPa (1 bar) above the pipeline pressure. Disconnect the receiver and transport it to the laboratory as soon as possible.

7.4.3.2 Sampling into double-piston variable volume sample receivers

7.4.3.2.1 Principle

The liquid is sampled at line pressure and maintained at line pressure (or above) during transportation and subsequent subsampling. A typical double-piston (internal mixing) receiver is illustrated in Figure 14. Sample is accumulated against only one of the floating pistons by carefully reducing the pressure of the inert gas buffer on the opposite side of that piston. The inert gas pressure on the second piston is kept significantly greater than the pipeline pressure throughout the sample accumulation stage to ensure that the receiver is not overfilled.

Prior to subsampling the primary sample for analysis, it may be homogenized by forcing the total sample quantity repeatedly through fine-bore transverse holes in the central baffle plate. This is achieved by alternately reducing the pressure of one inert gas buffer relative to the other (while maintaining the sample pressure above its bubble point). The efficiency of this multi-phase sample homogenization may be verified by

controlled injection/recovery tests. Consequently, these double-piston receivers are recommended for taking time-synchronized spot samples that are used for the calibration and verification of automatic pipeline water content analysers operating on high pressure pipelines (e.g. unstabilized crude oil or condensate production, fiscal or allocation accounting applications).

7.4.3.2.2 Receiver selection and leak testing

The working (sample) volume of a double-piston variable volume receiver is only 50 % of the total volume. Select a receiver with the required capacity and a rated working pressure which exceeds the pipeline pressure. Confirm that the piston seal elastomer is compatible with the pipeline liquid and capable of operating effectively at the pipeline temperature. Ensure that the receiver is clean and dry. Leak test all components as detailed in 7.4.3.1.2. If a valve, fitting or seal is found to leak, replace it and re-test.

7.4.3.2.3 Pre-charging the receiver

Open the sample inlet valve. Connect both inert gas inlet valves in turn to a supply of the appropriate inert gas and slowly pressurize it to at least 500 kPa (5 bar) above the pipeline pressure, so that both pistons are fully displaced against the central sample inlet/baffle plate. Close all valves and disconnect the inert gas supply. Transport the pre-charged receiver to the sample point.

7.4.3.2.4 Purging the sample point lines and receiver

Connect the pre-charged receiver sample inlet valve to the sampling point. Connect the sample outlet port/septum adapter valve to a closed drain or other safe disposal route.

Open the receiver sample outlet port valve fully and partially open the receiver sample inlet valve (1/4 turn). Carefully open the pipeline sample point valve to thoroughly flush the sample line and receiver dead volume. Control the rate of liquid flow through the receiver by carefully opening the inlet valve and the sample point valve further until the held-up volume has been thoroughly flushed.

7.4.3.2.5 Filling the receiver

Close the receiver sample outlet port valve fully on completion of the flushing operation. Neither piston should move at this stage due to the buffer gas pressure being higher than the pipeline pressure.

Cautiously crack open one (but not both) of the inert gas end valves to gradually reduce the inert gas pressure on one side of the receiver. The reduction in the inert gas pressure will allow sample to accumulate into the working side of the receiver, under the action of the pressure differential between the pipeline and the inert gas buffer. The inert gas valve shall be carefully adjusted to ensure that the corresponding sample inlet flow rate is properly controlled. The pressure difference between the pipeline and the working inert gas buffer should not exceed 100 kPa (1 bar) at any time during the sample accumulation period.

Close the valves when the piston position indicator shows that the receiver has been filled to approximately 90 % of the filled side of the receiver (i.e. about 45 % of the nominal total capacity).

De-pressurize the sample line to the drain before disconnecting the receiver from the sample point. Finally connect the working inert gas inlet valve to an inert gas supply and increase the inert gas pressure to at least 100 kPa (1 bar) above the pipeline pressure. It is not necessary to equalize the pressure between both inert gas buffers, but this may be done provided that the resulting final inert gas pressure is maintained above the pipeline pressure. Disconnect the receiver and transport it to the laboratory as soon as possible.

7.4.3.3 Sampling into fixed volume sample receivers

7.4.3.3.1 Principle

The liquid is sampled at line pressure and maintained at a pressure close to the line pressure during transportation and subsequent subsampling. It is essential to create an ullage volume within the fixed volume

receiver immediately after taking the sample, in order to prevent any unsafe increase in sample pressure due to thermal expansion effects during transportation or storage.

Where the vapour pressure of the liquid being sampled is close to the line pressure, the reduction in sample pressure associated with the creation of the ullage space may result in phase separation that can make subsequent representative subsampling difficult. In these circumstances, a variable volume sample receiver (7.4.3.1 or 7.4.3.2) should usually be used to ensure that the sample is maintained at sufficient pressure to prevent phase separation. However, if the liquid is also refrigerated, the effect of low temperatures on the receiver piston seals should also be considered.

Four main designs of fixed volume receiver are available depending on whether they are provided with

a) one or more inlet/outlet valves, and

b) an internal ullage tube or not.

Two-valve receivers are easier to purge prior to sampling, while an ullage tube simplifies ullaging the receiver by the correct amount (20 % of the total fixed volume) after sampling.

NOTE An internal ullage tube within a fixed volume sample receiver may also be known as an outage tube or dip tube.

7.4.3.3.2 Receiver selection and leak testing

The working (sample) volume of a fixed volume sample receiver is typically 20 % less than the nominal volume to allow for the necessary ullage space. Select a receiver with the required capacity and a rated working pressure that exceeds the pipeline pressure. Verify that the pressure test certification for the sample receiver and the bursting (rupture) disk is current. Ensure that the receiver is clean and dry.

Pressurize the receiver with inert gas to at least 100 kPa (1 bar) more than the anticipated pipeline pressure, and test for leaks. If a valve or fitting is found to leak, replace it and re-test, or use another receiver.

7.4.3.3.3 Purging the sample point line

If the receiver has an internal ullage tube fitted, determine which receiver valve supplies the tube and connect this valve to the sampling point. If the receiver has a second valve, connect that valve to a closed drain or other safe disposal route. Confirm that all valves are initially closed.

Earth continuity should usually be provided to the receiver via the metallic pipework connection to the sample point, but in some cases it may be necessary to provide a separate earth (grounding) connection.

Purge the sample transfer line by displacing at least 150 % of the line's volume (from the sample probe in the pipeline to the sampling point) to the vent immediately prior to the sample point valve. Then close the vent valve.

7.4.3.3.4 Purging the sample receiver

For a one-valve receiver, open the receiver inlet valve to partly fill the receiver. Then close the sample line control valve, and open the line vent valve to purge the receiver. Close the vent valve and repeat the partial filling and venting process at least twice more to purge the receiver as fully as possible.

For a two-valve receiver, open the receiver inlet valve to partly fill the receiver. Then slowly open the receiver outlet valve to its vent. Close the sample line control valve, and allow part of the receiver contents to escape to vent through the receiver outlet valve. Close the receiver vent valve and open the sample line vent valve to allow further venting. Close the sample line vent valve and repeat the partial filling and venting process at least twice more to purge the receiver as fully as possible.

7.4.3.3.5 Filling the receiver

On completion of the purging operation, open the sample line control valve to fill the receiver, then close the receiver inlet valve. Then close the sample line source valve (at the pipeline), and open the sample line vent valve to depressurize the sample line. Finally, close the remaining sample line valves and disconnect the receiver.

7.4.3.3.6 Providing a safe ullage space within the receiver

Immediately after taking the sample, a 20 % ullage shall be provided in the receiver. This may be achieved by either using an integral receiver ullage tube (where fitted), or by weighing the receiver to determine the sample quantity.

If using the ullage tube procedure, position the receiver upright with the sample inlet valve (and ullage tube) at the top.

NOTE 1 Electrostatic safety considerations may mean that it is advisable to fit the receiver with an earth (grounding) connection before proceeding to the next partial venting step.

Cautiously open the receiver inlet valve slightly until liquid is observed to escape. Allow the excess liquid to escape, but, as soon as it is observed that the escaping liquid material changes to vapour quickly, close the valve.

If no liquid escapes initially then the receiver was not filled sufficiently and the sample will not be representative. In these circumstances, the suspect sample should be discarded, and the sampling procedure repeated.

If using the weighing procedure, weigh the filled receiver and deduct the tare weight to determine the total weight of sample that has been taken. Calculate the weight of sample that represents a 20 % ullage, and vent this amount by cautiously opening the receiver inlet valve slightly.

NOTE 2 Electrostatic safety considerations may mean that it is advisable to fit the receiver with an earth (grounding) connection before conducting the venting operation.

Close the receiver inlet valve and re-weigh the receiver to verify that a safe ullage space has been created. If the gross weight still exceeds the tare weight plus 80 % of the original sample weight then the partial venting operation should be repeated.

If the receiver weighing cannot be performed at the sampling location, it is important to ensure that a small quantity of the liquid phase sample is vented immediately to prevent excessive pressure build-up as a result of sample expansion due to any subsequent increase in temperature. The full weighing and ullaging procedure should then take place as soon as possible after transportation to a suitable location where the facilities are available.

7.4.3.3.7 Sample handling

On completion of the preceding steps, immediately check the receiver for leaks with a proprietary leak detecting fluid, with soapy water or by immersing in water. If any leaks are detected, discard the sample and repair or replace the receiver before obtaining another sample.

Label the sample receiver clearly, and prepare it for transportation by packing in a suitable container as required by the appropriate transportation regulations. Transport the receiver to the laboratory/test location without delay. Where intermediate storage is necessary, the sample should be protected from extremes of temperature.

7.5 Dispenser (nozzle) sampling

This procedure is applicable for sampling light fuels from retail-type dispensers. Fit a nozzle extension to allow fuel to be dispensed to the bottom of the sample receiver, without splashing. Where the nozzle is fitted with a

vapour recovery system, a spacer will be needed to hold back the nozzle sleeve. Fill the sample receiver slowly, through the nozzle extension, until it is approximately 85 % full. Remove the nozzle and extension and close or cap the receiver immediately.

If the sample is to be analysed for vapour pressure, chill the receiver prior to filling.

8 Procedures for crude oils and other non-homogeneous petroleum liquids

8.1 General

If available, automatic pipeline sampling as specified in ISO 3171 shall be used for sampling crude oil and non-homogeneous oils in preference to the following manual procedures. The automatic pipeline sampling procedure is applicable to crude oils and other non-homogeneous oils such as so-called “heavy crude oils” and residual fuels because such systems will normally include a mixing device immediately upstream of the sampler, so that the line contents are uniformly dispersed prior to sampling. The sample will also normally be accumulated flow-proportionally, to allow for any changes in flow rate while sampling throughout a batch transfer.

NOTE The manual sampling methods specified in Clause 7 may not provide representative samples for the following reasons.

a) The concentration of dispersed water in the oil is generally higher near the bottom of a tank. A running or all-level sample, or a composite sample of the upper, middle and lower samples may not provide a sample representative of the concentration of all the dispersed water present.

b) The interface between oil and free water may be difficult to locate, especially in the presence of emulsions, layers or water-bearing sediments.

c) The free water level may vary across the tank bottom surface. The bottom may be covered by pools of free water or water/oil emulsion impounded by layers of sediments or wax.

d) Light ends may be lost easily in manual operations, affecting the density and vapour pressure of the sample.

Because circumstances will arise where manual methods of sampling have to be employed, procedures are given in this clause which shall be followed so that a sample may be drawn, which is as representative of the bulk as the techniques allow. The procedures specified are additional to or replace those specified in Clause 7.

8.2 Procedures

8.2.1 Tank sampling

For tank sampling, use one of the following techniques specified in 7.3:

— spot sampling;

— running sampling;

— all-level sampling.

By agreement of all parties, composite spot or zone samples may be prepared, see 7.3.1.2.

Where it is necessary to assess the level of stratification within a tank's contents, draw samples initially from the upper, middle and lower (or suction) levels, transport them to the laboratory or testing location and test them individually for density, water and sediment content.

If the range of the results of these tests lie within $\pm$ 1 kg/m^3 (density) and $\pm$ 0,1 % (V/V) (water content), the tank contents shall be considered as representative of the bulk, and the average results shall be taken.

If the range of the results of these tests does not lie within the specified limits, the tank contents are probably stratified. In these circumstances, additional spot samples shall, if possible, be taken at intervening or equidistant levels, and all the individual test results averaged. For this purpose, the samples for density, water, and sediment analysis shall not be physically composited, but the results of analyses on the separate samples may be mathematically composited.

NOTE In small leased automatic custody transfer (LACT) crude oil tanks of less than 159 m^3 (1 000 barrels), a single spot sample from the middle of the oil is usually sufficient.

8.2.2 Pipeline sampling

8.2.2.1 General

For pipeline sampling of batch transfer quantities, use the procedures described in ISO 3171.

NOTE If it is required to take manual spot samples from pipelines, the procedure will be dependent on whether a fixed volume or variable volume sample receiver is to be used, and on the vapour pressure of the liquid that is to be sampled.

8.2.2.2 Pipeline sampling of low vapour pressure liquids

Fixed volume sample receivers shall be filled directly after thoroughly flushing the sample probe and line. Sample collection shall be by submerged delivery to minimize any risk of the evaporative loss of light end components (5.4.2). A sample cooler may be used if appropriate (5.7).

Low pressure variable volume sample receivers (e.g. collapsible flexible containers, bladders or diaphragm receivers) shall be emptied prior to use. Where appropriate, the receiver may be evacuated. The receiver shall be filled directly after thoroughly flushing the sample probe and line.

8.2.2.3 Pipeline sampling of high vapour pressure liquids

High pressure variable volume sample receivers (e.g. floating piston cylinders) shall be filled in accordance with the procedures detailed in 7.4.3.1 or 7.4.3.2. High pressure fixed volume sample receivers shall be filled in accordance with the procedures detailed in 7.4.3.3.

NOTE It will not normally be appropriate to use low pressure variable volume receivers (e.g. collapsible flexible containers, bladders or diaphragm receivers), or low pressure fixed volume receivers (e.g. bottles or cans) for sampling high vapour pressure pipeline liquids. If such receivers are used, phase separation may occur as the pressure is reduced and light-end components may be lost.

8.2.3 Additional precautions

Give particular attention to the precautions referred to in respect of the following:

a) high pour-point oils (see 7.2.4);

b) volatile oils (see 7.2.3);

c) collection of large-volume samples (see 7.2.1.8);

d) ullage to be left in fixed volume sample containers (see 7.2.1.6);

e) samples for transportation (see 7.2.5 and 8.2.4).

8.2.4 Sample transportation

Transport the samples to the test laboratory in the original sample container, without transfer or compositing (bulking), in order to maintain the integrity of the sample. If it is impossible to transport the sample in the

original container, transfer it to a suitable secondary container following the procedure specified in 9.4 and record the transfer. Samples should be transported to the testing location as soon as possible, or stored in an appropriate cool, dark, dry location.

Fixed volume sample receivers/containers should be transported and stored in an inverted position, if feasible, so that any loss of vapour from the ullage space is prevented, and any leakage is immediately apparent.

9 Sample handling

9.1 General

9.1.1 The method of handling samples between the point at which they are extracted or drawn and the final laboratory analysis of the test portion (or sample storage) shall ensure that the nature and integrity of the samples are maintained.

9.1.2 The method of handling a sample will depend on the purpose for which it has been taken. The laboratory analytical procedure to be used will often require a special handling procedure to be associated with it. For this reason, consult the appropriate method of test so that any necessary instructions as to sample handling can be given to the person drawing the sample. If the analytical procedures to be applied have conflicting requirements, draw separate samples and apply the appropriate procedure to each sample.

9.1.3 Take particular care in respect of the following:

a) liquids containing volatile components, since loss by evaporation can occur;

b) liquids containing water and/or sediment, since separation tends to occur in the sample container;

c) liquids with potential wax deposition, since deposition can occur if a sufficient temperature is not maintained.

9.1.4 When making up composite samples, take great care not to lose light ends from volatile liquids, and not to alter the water and sediment contents. The preparation of composite samples without loss of sample integrity is a very difficult operation and shall be avoided if possible.

9.1.5 Do not transfer samples of volatile liquids to other containers at the sampling location but transport them to the laboratory in the original sample container, cooled and inverted, if necessary. Great care is necessary if a sample contains both volatile components and free water.

9.2 Homogenization of samples

9.2.1 Introduction

Procedures are specified for the homogenization of samples that may contain water and sediment, or are in any other way non-uniform. Homogenization is required before any transfer of a partial quantity of sample, such as subsampling or removal of a test portion. Care is required to ensure that the homogenization process does not in itself cause a loss of representativity, such as through the loss of light components. Procedures for verifying that the sample is satisfactorily mixed before transfer are given in 9.3.

It is not possible to manually agitate samples of liquids containing water and sediment sufficiently to disperse the water and sediment within the sample. Vigorous mechanical or hydraulic mixing is necessary in order to homogenize the sample prior to transfer or subsampling.

Homogenization may be by various methods and will be governed by the sample, the sample receiver and/or the test method(s) used. Whichever method is used, it is recommended that the homogenizing system produces water droplets of sufficiently small size to ensure homogeneity and stability during subsequent handling, subsampling and analysis procedures.

The water content of stable emulsions containing small water droplets cannot be analysed accurately by centrifuge test methods. Alternative methods, such as ISO 10336, ISO 10337 and ISO 9029, should be used.

9.2.2 Homogenization by high-shear mechanical mixer

9.2.2.1 Fixed volume sample receivers

Insert a non-aerating high-shear mechanical mixer into the sample container so that the rotating element reaches to within 30 mm of the bottom.

NOTE 1 A mixer with counter-rotating blades operating at about 3 000 r/min is usually suitable. Other designs may be used if the performance is satisfactory (see 9.3).

In order to minimize loss of light ends from crude oils or other samples containing volatile components, operate the stirrer through a gland in the closure of the sample container. Mix until the sample is completely homogenized. A mixing time of 5 min is sometimes sufficient, but the size of the container and the nature of the sample affect the homogenization time. Verify that the sample has become homogeneous (see 9.3). Optimize the mixing conditions as necessary to achieve homogeneity and enable representative subsampling.

NOTE 2 High-shear mixers frequently produce stable emulsions, and water contents cannot be determined by the centrifuge method (ISO 3734).

Avoid any significant rise in temperature (> 10 °C) during the mixing. If feasible (7.2.3.2), it may be desirable to cool the sample prior to homogenization and/or during the homogenization.

9.2.2.2 Variable volume sample receivers

Operate the integral internal mixing system in accordance with the manufacturer’s recommendations. Verify that the mixing conditions used are capable of homogenizing typical samples by conducting controlled injection/recovery tests (9.3). Optimize the mixing conditions as necessary to achieve homogeneity and enable representative subsampling.

Avoid any significant rise in temperature (> 10 °C) during the mixing. If feasible (7.2.3.2), it may be desirable to cool the sample prior to homogenization and/or during the homogenization.

9.2.3 Circulation with external mixer

External circulation may be applied to both permanently sited sample containers and portable containers; for the latter, use a quick-disconnect coupling. The method may be equally applicable to certain designs of variable volume sample receivers as well as larger fixed volume sample receiver/containers.

Circulate the contents externally using a small pump through a static mixer or spray bar mechanism installed in the piping. Various designs are available; follow the manufacturer's operating instructions.

The hold-up volume of the external mixer, pump and connections should be kept to the absolute minimum. This is to minimize the increase in the ullage volume within a fixed volume receiver during mixing, and thus minimize evaporative losses to the ullage space.

Use a circulating flow rate that is sufficient to theoretically circulate the total sample volume at least once per minute. A typical mixing time is 10 min, but this will vary according to the water content, the type of hydrocarbon components present in the product, the re-circulation flow rate, the total sample volume and the design of the system. Verify that the mixing conditions used are capable of homogenizing typical samples by conducting controlled injection/recovery tests (9.3). Optimize the mixing conditions as necessary to achieve homogeneity and enable representative subsampling.

Avoid any significant rise in temperature (> 10 °C) during the mixing. If feasible (7.2.3.2), it may be desirable to cool the sample prior to homogenization and/or during the homogenization.

When the whole sample is thoroughly mixed, run off the required quantity of subsample from a valve in the circulating line or from valves on the sample receiver, whilst the pump is running. Alternatively, subsamples may be taken via a syringe septum port (located downstream of the static mixer) while the sample is being re-circulated.

If further subsamples are required on a later occasion, the contents of the external mixing loop shall be displaced back to the sample receiver/container on completion of subsampling, otherwise the remaining sample shall be transferred to a suitable secondary receiver/container for retention or disposal as necessary.

After use, the mixing loop shall be thoroughly cleaned with appropriate solvent(s), and then dried with compressed air. Care is required to ensure that there is no risk of the next sample being contaminated by the incorporation of residual sample or cleaning solvents that were held-up within the mixing loop.

NOTE The original empty container may be used to re-circulate solvent through the external mixing loop to clean it.

9.3 Verification of mixing efficiency

9.3.1 General

Whatever means are chosen for obtaining a subsample from a non-homogeneous mixture, verify the suitability of the mixing technique and the time required to obtain a homogeneous sample that can be representatively subsampled.

9.3.2 Homogeneous liquids

If the sample remains homogeneous and stable after mixing (e.g. where completely miscible components such as lubricant additives have been blended), continue the mixing procedure until successive samples drawn from the main bulk of the sample give identical results. This establishes the minimum mixing time.

NOTE As the sample is homogeneous after this time, and will remain so, transfers from the main bulk can be made without further mixing.

9.3.3 Non-homogeneous liquids

If the sample does not remain homogeneous for more than a short period of time after mixing (e.g. if water and sediment are part of the mixture), use the method for the verification of mixing efficiency described in 9.3.4.

NOTE It may be necessary, owing to the components present in the product, to subsample while mixing is still in progress.

9.3.4 Mixing efficiency verification test for non-homogeneous oils (injection/recovery test)

9.3.4.1 Weigh an empty receiver/container using a balance with sufficient resolution to enable the added water content (9.3.4.5) to be determined to 0,01 % (*m/m*) or better.

NOTE The procedure is equally applicable to fixed volume sample receivers and to variable volume sample receivers.

9.3.4.2 Fill the receiver/container with the typical expected sample quantity, using the actual liquid that is to be sampled. The suspended water content of this liquid shall, if possible, be less than 0,1 % (*m/m*), but the liquid used shall have no free water present. Record the liquid temperature.

9.3.4.3 Mix the simulated sample using the anticipated typical conditions (mixer rotational speed, mixing time, recirculation flow rate, number of mixing elements, recirculation time, mixing pressure differential and number of shuttles, etc.). Verify that the mixing has not caused the oil temperature to increase by more than 10 °C.

9.3.4.4 Using a dry syringe, subsample and analyse by Karl Fischer titration (ISO 10336, ISO 10337) to determine the baseline water content. Repeat the subsampling and analysis, and confirm that the results agree within 0,02 % (*m/m*). If the results fall outside this repeatability criterion, increase the severity of the mixing conditions (within the maximum temperature-increase limitation) and re-test until the results agree within 0,02 % (*m/m*). Alternatively, use a more efficient mixing system.

9.3.4.5 Reweigh the receiver/container and contents to determine the quantity of baseline oil that is present. Add a weighed quantity of distilled water to increase the total water content (baseline plus added water content) to a target level of at least 2 % (*m/m)* higher than the maximum concentration that is anticipated in actual samples. If the maximum anticipated concentration is unknown, add sufficient water to bring the total target water content to a minimum of 5 % (*m/m)*.

9.3.4.6 Record the wet oil temperature prior to mixing the simulated sample using the same mixing conditions that were used to obtain the repeatable baseline analysis (9.3.4.4). Verify that the mixing has not caused the oil temperature to increase by more than 10 °C.

9.3.4.7 Subsample the mixed sample immediately using a dry syringe, and analyse the subsample by the same Karl Fischer titration to determine its water content. On completion of this analysis, repeat the subsampling and analysis step to assess the repeatability of the results and the stability of the emulsion created by the chosen mixing conditions.

9.3.4.8 Check the repeatability of the duplicate results, and the agreement between the mean water content found (9.3.4.7) and the total target water content (baseline plus added water content) (9.3.4.5). The maximum permissible range (X) of the duplicate results and the maximum permissible difference between the mean result and the target result (Y) shall not exceed the tolerances given in Table 3.

Table 3 — Maximum tolerances for measured water content

Mean measured water content % *(m/m)*	**Maximum range of the duplicate results** *(X)* % *(m/m)*	**Maximum difference between mean measured and target water contents** *(Y)* % *(m/m)*
$\leqslant$ 4,00	0,10	0,10
4,01 to 6,00	0,15	0,15
6,01 to 10,00	0,20	0,20
$>$ 10	0,25	0,25

9.3.4.9 If the injection/recovery test criteria (Table 3) are met, use the proven mixer conditions for all subsequent subsampling of the same source sample with the same or lesser water content.

9.3.4.10 If the measured water contents do not meet these criteria, repeat the procedure with a fresh sample, but increase the severity of the mixing conditions (e.g. the mixing time and/or speed, flow rate) until the criteria are met or the sample temperature increase restriction is exceeded.

If the injection/recovery test criteria are still not met, re-test using a modified mixing procedure such as re-mixing immediately prior to taking the second subsample, or taking all subsamples while mixing continuously. Alternatively, use a different type of sample mixing system.

If the sample temperature increase restriction is exceeded, consider pre-cooling the sample or cool the sample during mixing where this is practical (7.2.3.2).

9.3.4.11 Do not attempt to subsample crude oil or other non-homogeneous oil samples for water and sediment content analysis unless the representative conformity of the sample mixing and subsampling procedures have been proven by this procedure.

9.3.4.12 Do not determine water content by the centrifuge method (ISO 3734 or ISO 9030) for this verification of the mixing system, as the method cannot be relied upon to give the total water content.

9.4 Transfer of samples

9.4.1 Transfer of sample between receivers/containers shall, if possible, be avoided unless conducted under controlled conditions. If the primary sample cannot be transported directly to the laboratory, it shall be transferred completely to a secondary container that can be transported directly to a laboratory. A complete transfer may be difficult due to evaporative loss of light components and/or the incomplete transfer of heavier components. In such circumstances, a sampling method shall be used that will permit the primary sample to be delivered to the laboratory without any transfer or subsampling. If this is not possible, any sample handling and/or transfer outside of controlled laboratory conditions shall be kept to the absolute minimum.

9.4.2 At every stage involving partial transfer of a sample, it is essential to homogenize the contents of the container from which the sample is being taken using one of the methods specified in 9.2.

9.4.3 Verify the mixing efficiency for each combination of container and mixer by one of the methods specified in 9.3.

9.4.4 Complete any transfer of sample within the period during which the mixture is known to be homogeneous. In some cases (especially during subsampling into more than one container), it may be necessary to continue mixing during the transfer operations.

10 Sampling of tank residues/deposits

Samples of tank residues/deposits are unlikely to be representative and shall only be used for guidance regarding their nature and composition.

Select an appropriate sampling device, depending upon the dimensions of the gauging access point(s) available and the depth of the residue/deposit to be sampled. (See 5.2.4 and Figure 8.) Follow the manufacturer's instructions and transfer the sample from the device into a metal, plastics or glass container, which shall maintain the integrity of the sample.

NOTE Tank residues are organic and/or inorganic sediments forming a layer on the bottom of either marine or land-based tanks. At ambient temperatures, the product is highly viscous and of a soft to stiff consistency.

11 Package sampling

This clause is based on ISO 2859-1, to which reference should be made for details.

11.1 Statistical aspects of sampling packages

11.1.1 Number of samples to be taken

11.1.1.1 General

The variability of the product both within and between packages, the number of packages sampled, and the precision of the test methods may all contribute to errors in the determination of the properties of the product as shown by the test results. The number of samples to be drawn will depend on the number of units of product, the acceptable quality level (AQL) and the inspection level.

11.1.1.2 Sampling to assess uniformity in a package

Draw spot samples from evenly distributed points within the product. Test each sample using a simple test based on an easily assessable characteristic (e.g. density, colour). Any variations in the test results in excess of the repeatability of the test method indicate that the product in the package is not uniform.

11.1.1.3 Sampling to assess the average quality of a batch

A batch consists of a number of packages of a product of a single composition.

a) Single package: If the product has been shown to be uniform (see 11.1.1.2), take a single spot sample, but, if not, take a sufficient number of spot samples and combine them to give a composite sample.

b) Multiple packages: The accuracy with which an estimate of the average quality of the product within a number of packages can be made is dependent on:

   1) the number of packages sampled;

   2) the precision of the test method used;

   3) the variability of the product between packages.

11.1.1.4 Sampling of whole consignment

If all the packages are sampled, this necessarily involves the greatest amount of sampling, and the error in the determination of the average quality will depend on the testing of the samples.

If each of the samples is tested once, the average of the test results will be a measure of the average quality with the lowest uncertainty. If a composite sample is prepared and is tested (in duplicate), the average result will be a measure of the average quality, but with a much higher uncertainty.

11.1.1.5 Sampling of part of the batch

It is not always possible to sample all of a batch or consignment. Plans are given in this International Standard to enable a decision to be made as to the number of discrete samples which need to be taken from a batch in order that a valid conclusion may be arrived at regarding the quality of the total contents of all the packages.

11.1.2 Acceptable quality level (AQL) (see 3.1)

The AQL shall be 2,5 %.

11.1.3 Inspection level

The inspection level determines the relationship between the lot or batch size and the sample size (number of packages per batch and the required number of samples). The normal inspection level (see ISO 2859-1) shall be used.

11.1.4 Sampling plan

11.1.4.1 General

A sampling plan indicates the number of units of product from each batch which are to be inspected (sample size or series of sample sizes) and the criteria for determining the acceptability of the lot or batch (acceptance and rejection numbers) (see Tables 4, 5 and 6).

11.1.4.2 Single sampling plan

The number of sample units inspected shall be equal to the sample size given by the plan. If the number of defectives found in the sample is equal to or less than the acceptance number (see “Ac” in Table 5), the lot or batch shall be considered acceptable. If the number of defectives is equal to or greater than the rejection number (see “Re” in Table 5), the lot or batch shall be rejected.

11.1.4.3 Double sampling plan

The number of sample units inspected shall be equal to the first sample size given by the plan. If the number of defectives found in the first sample is equal to or less than the first acceptance number (see Table 6), the lot or batch shall be considered acceptable.

If the number of defectives found in the first sample is equal to or greater than the first acceptance and rejection number (see Table 6), the lot or batch shall be rejected.

If the number of defectives found in the first sample is between the first acceptance and rejection numbers (see Table 6), a second sample of the size given by the plan shall be inspected. The number of defectives found in the first and second samples shall be accumulated. If the cumulative number of defectives is equal to or less than the second acceptance number, the lot or batch shall be considered acceptable. If the cumulative number of defectives is equal to or greater than the second rejection number, the lot or batch shall be rejected.

Instructions on the use of the plans to determine the acceptability of the batch should be given with each plan (see ISO 2859-1).

11.2 Procedures for sampling packages

11.2.1 Drums and barrels

Place the drum or barrel on its side with the bung up. If the drum does not have a side bung, stand it upright and sample from the top. If detection of water, rust or other insoluble contaminants is desired, let the barrel or drum remain in this position long enough to permit the contaminants to settle.

Remove the bung and place it beside the bung hole with the oily side up. Close the upper end of the clean, dry sampling tube with the thumb, and lower the tube into the oil to a depth of about 300 mm. Remove the thumb, allowing oil to flow into the tube. Again close the upper end with the thumb and withdraw the tube. Rinse the tube with the oil by holding it steady horizontally and turning it so that the oil comes into contact with that part of the inside surface that will be immersed when the sample is taken. Avoid handling any part of the tube that will be immersed in the oil during the sampling operation.

Return the oil used to rinse the tube back to the drum, and allow the tube to drain.

Insert the tube into the oil again, holding the thumb against the upper end. (If an all-level sample is desired, insert the tube with the upper end open.) When the tube reaches the bottom, remove the thumb and allow the tube to fill. Replace the thumb, withdraw the tube quickly and transfer the contents to the sample container. Do not allow the hands to come into contact with any part of the sample. Close the sample container; replace and tighten the bung in the drum or barrel.

11.2.2 Cans

Obtain samples from cans of 20 L capacity or larger in the same manner as from drums and barrels (see 11.2.1) using a tube of proportionately smaller dimensions. For cans of less than 20 L capacity, use the entire contents as the sample, selecting cans as described in ISO 2859-1.

Table 4 — Sampling plans — Sample size code

Batch size	Single sampling code	Double sampling code
2 to 8	A	A
9 to 15	B	A
16 to 25	C	B
26 to 50	D	B
51 to 90	E	C
91 to 150	F	C
151 to 280	G	D
281 to 500	H	D
501 to 1200	J	E
1201 to 3200	K	E
3201 to 10 000	L	F
10 001 to 35 000	M	F
35 001 to 150 000	N	G
150 001 to 500 000	P	G
500 001 and over	Q	H

Table 5 — Sampling plans — Single sampling plan

Sample size code letter	Sample size	AQL = 2,5		
		Ac		Re
A	2			
B	3			
C	5	0	↓	1
D	8		↑	
E	13			
F	20	1	↓	2
G	32	2		3
H	50	3		4
J	80	5		6
K	125	7		8
L	200	10		11
M	315	14		15
N	500	21	↑	22
P	800			
Q	1 250			

AQL = Acceptance quality limit

Ac = Acceptance number

Re = Rejection number

Table 6 — Sampling plans — Double sampling plan

Sample size code letter	Sample	Sample size	Cumulative sample size	AQL = 2,5	
				Ac	Re
A					
B	First	2	2		
	Second	2	4	↓	
C	First	3	3	*	
	Second	3	6		
D	First	5	5	↑	
	Second	5	10		
E	First	8	8		
	Second	8	16	↓	
F	First	13	13	0	2
	Second	13	26	1	2
G	First	20	20	0	3
	Second	20	40	3	4
H	First	32	32	1	4
	Second	32	64	4	5
J	First	50	50	2	5
	Second	50	100	6	7
K	First	80	80	3	7
	Second	80	160	8	9
L	First	125	125	5	9
	Second	125	250	12	13
M	First	200	200	7	11
	Second	200	400	18	19
N	First	315	315	11	16
	Second	315	630	26	27
P	First	500	500	↑	
	Second	500	1000		
Q	First	800	800		
	Second	800	1600		

↓ = Go down in this column till a block with an asterisk (*) or with acceptance/rejection numbers (Ac/Re) is reached. In the latter case, use these numbers and the sample size on the same line to the left of this block. If an asterisk (*) is reached, follow instructions in the footnote below. If the sample size equals or exceeds the lot or batch size, carry out 100 % inspection.

↑ = Go up in this column till a block with an asterisk (*) or with acceptance/rejection numbers (Ac/Re) is reached. In the latter case, use these numbers and the sample size on the same line to the left of this block (not the original sample size). If an asterisk (*) is reached, follow the instructions in the footnote below.

Ac = Acceptance number.

Re = Rejection number.

* = Use the corresponding single sample plan (code letter and AQL for this block) (or, alternatively, use the double sample figures in the nearest block below the asterisk and on the line to the left of this block).

Annex A
(informative)

Guidance on safety precautions

A.1 General

A.1.1 The safety precautions given below apply generally and constitute good practice, but the list is not necessarily comprehensive. The list should be read in conjunction with the appropriate national safety regulations or any recognized code in the petroleum industry. The precautions given below should be taken whenever they do not conflict with local or national regulations.

A.1.2 Personnel should be made aware of the potential hazards and be given instructions in the safety precautions to be observed.

A.1.3 All regulations covering entry into hazardous areas should be observed.

A.1.4 Care should be taken to avoid breathing petroleum vapour during the sampling operations. Protective gloves of hydrocarbon-insoluble materials should be worn. Eye shields or face shields should be worn where there is a danger of splashing. Additional precautions may be necessary when handling sour crude.

A.1.5 When handling leaded fuels, the safety regulations should be observed.

A.2 Safety aspects of equipment

A.2.1 With regard to their mechanical properties, receivers/containers should be designed in accordance with the respective national/international standards.

Pressure tests and other inspection work should be performed according to the local regulations and the results of such tests should be recorded. Cleaning and leak testing operations should be performed at regular intervals.

A.2.2 Cords used for lowering sampling equipment should be electrically conductive, and made from natural anti-static material such as cotton or sisal. They should not be made from man-made fibres.

A.2.3 Portable metal sampling equipment used in flammable atmospheres should be of non-sparking material.

Caution should be exercised when using equipment made of aluminium, magnesium or titanium which may generate incendive sparks when struck against rusted steel. Some countries restrict the use of sampling equipment made from such materials, or from alloys containing more than 15 % (*m/m*) in total of these metals or 6 % *(m/m*) of magnesium.

A.2.4 Sampling personnel should be provided with carriers for their equipment in order that at least one hand may be free.

A.2.5 Lamps and torches (flashlights) should be of an approved type suitable for the electrical classification of the area.

A.2.6 Suitable clothing and equipment to provide protection against all known hazards associated with the product being sampled should be worn.

A.2.7 Sample bottles should be protected with a metal case or mesh safety cover if they are used for sampling products with a Reid vapour pressure (RVP) of between 100 kPa (1,0 bar) and 180 kPa (1,8 bar). Above 180 kPa (1,8 bar) RVP, only suitable metal containers or variable volume sample receivers which are specifically constructed to contain the pressure involved should be used.

A.2.8 Care should be taken to avoid heating of volatile samples in containers with gas-tight closures.

A.3 Safety at sampling points

A.3.1 Sampling points should be provided which enable samples to be taken in a safe manner. Any potential hazards associated with sampling should be clearly marked and it is recommended that a pressure gauge be provided at pipeline sampling points.

A.3.2 The sampling point and equipment should be adequately maintained and regularly inspected, and the results of the inspection recorded.

A.3.3 Safe access to sampling points, with adequate lighting, should be provided. Access ladders, stairways, platforms and handrails should be maintained in a structurally safe condition and regularly inspected.

A.3.4 Adequate and safe drainage for all draining and flushing requirements should be provided.

A.3.5 Any spillages or defects in equipment should be reported immediately.

A.3.6 Care should be taken to avoid breathing petroleum vapours during sampling operations.

A.3.7 Floating-roof tanks should be sampled from the top platform whenever practicable, as toxic and flammable vapors may accumulate above the roof. When it is necessary to descend to the roof for sampling, unless the atmosphere above the roof has been proved to be safe, at least two persons wearing self-contained breathing apparatus should be present.

The second or other person(s) should stand by at the head of the stairway where they can clearly observe the person on the roof. The first person should descend to the roof, take the required samples, and return to the head of the stairway in the minimum time possible.

The following are some of the conditions which may render the atmosphere above the roof hazardous:

a) the product contains hydrogen sulfide and/or volatile mercaptans;

b) the roof is not fully floating;

c) the roof-seal is faulty.

A.4 Static electricity

A.4.1 The following precautions should be taken to avoid danger from static electricity when sampling tanks containing flammable hydrocarbons stored at temperatures above their flash points, or in which a flammable atmosphere of hydrocarbon vapour or mist has been produced.

A.4.2 The contents of storage tanks, road vehicles, railcars, ships or barges should not be sampled during filling, especially when being filled with clean, refined, volatile products capable of giving rise to flammable vapour/air mixtures in the ullage space.

A.4.3 When sampling a tank, the sampling device should be kept firmly earthed at all times, either by direct earth connection to the tank structure, or by firm contact of the suspending cable, cord or tape with the gauge-hatch or vapour-lock valve. When sampling a pipeline, electrical continuity should be maintained between the pipeline and the sample receiver via the connecting pipework.

A.4.4 When tank sampling clean, refined, volatile products, including kerosene and gas oil, which have been loaded at a temperature near or above their flash point, or into tanks which are not gas-free, a relaxation time (commonly 30 min) after completion of transfer or loading should be allowed before introducing any conductive sampling equipment into the tank or container, except in the following circumstances:

a) for a fixed or floating-roof tank, the sampling is carried out from a perforated still-well or dip pipe that extends below the liquid surface and is in direct electrical continuity with the tank shell;

b) for a fixed-roof tank, the tank is fitted with an earthed internal floating cover;

c) for a floating-roof tank, the roof is fully floating, and in direct electrical continuity with the tank shell;

d) the product contains sufficient static-dissipating additive to ensure an overall conductivity greater than 50 pS/m, and no mist or spray is being formed in the ullage space (see the following note).

NOTE Static-dissipating additives can increase the conductivity of hydrocarbon liquids to a level that is sufficient to avoid the accumulation of static electrical charge; an overall conductivity of 50 pS/m is acceptable. The relaxation time for a bound charge in the body of a liquid is so short at this conductivity level that the charge is dissipated almost as it forms. As a result, gauging and sampling may take place without delay or even while filling is in progress, so long as no mist or spray is being formed in the ullage space. Charged droplets can exist in mists or sprays and produce an accumulation of static electricity, regardless of the presence of static dissipation additives in the liquid product.

A.4.5 Footwear and or clothing capable of causing sparks should not be worn in areas where flammable vapours are likely to be present.

A.4.6 Sampling should not be carried out during periods of atmospheric electric disturbance or hail storms.

A.4.7 In order to earth any static charge on their person, the operator should touch some part of the tank structure at least 1 m from any sampling opening immediately before carrying out any sampling operation.

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