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British Standard Methods for **Sampling petroleum products**

Part 2. Automatic pipeline sampling of liquid hydrocarbons

Méthodes d'échantillonnage des produits pétroliers
Partie 2. Hydrocarbures liquides — Échantillonnage automatique en oléoduc

Erdölerzeugnisse; Probenahme
Teil 2. Flüssige Kohlenwasserstoffe; automatische Probenahme an Rohrleitungen

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National foreword

This Part of BS 3195 has been prepared under the direction of the Petroleum Standards Policy Committee and is identical with ISO 3171 : 1988 'Petroleum liquids — Automatic pipeline sampling'. ISO 3171 was prepared by Subcommittee 3 — Static Petroleum Measurement of Technical Committee 28, Petroleum Products and Lubricants, of the International Organization for Standardization (ISO) as the result of discussions in which the United Kingdom participated.

Part 1 of this British Standard is identical with ISO 3170 : 1988. The previous edition of ISO 3171 was not adopted as a British Standard because the methods described were not generally applicable to crude oil; the present revision has been extended to provide for sampling of crude oil and to provide for the mathematical evaluation of sampling system uncertainty and for estimating oil/water dispersion.

Cross-references

International standard	Corresponding British Standard
ISO 3170 — 1988	BS 3195 Methods for sampling petroleum products Part 1 : 1989 Manual sampling of liquid hydrocarbons (Identical)
ISO 3534 — 1977	BS 5532 Statistical terminology Part 1 : 1978 Glossary of terms relating to probability and general terms relating to statistics (Identical)
ISO 3733 — 1976	BS 4385 : 1980 Method for determination of water in petroleum products and bituminous materials (distillation method) (Identical)
ISO 3734 — 1976	BS 2882 : 1980 Method for determination of water and sediment in crude petroleum and fuel oils (centrifuge method) (Identical)
ISO 3735 — 1975	BS 4382 : 1980 Method for determination of sediment in crude petroleum and fuel oils (extraction method) (Identical)
ISO 5168 — 1978	BS 5844 : 1980 Methods of measurement of fluid flow : estimation of uncertainty of a flow-rate measurement (Identical)

There is no British Standard identical to ISO 3165 : 1976, which does not form an essential reference in this standard, but has been included to give additional guidance on safety in sampling. A related British Standard, to which reference may be made, is BS 5309 'Methods for sampling chemical products Part 1 : 1976 Introduction and general principles'.

The Technical Report on 'Statistics for Sampling System Performance' is in course of preparation and will be considered by the responsible BSI Committee when available.

CAUTION. Attention is drawn to clause 13 and the need to observe the safety precautions and the requirements of the Health and Safety at Work etc. Act 1974. With respect to 13.1, 13.8 and 13.10 the user should be familiar with the relevant UK regulations, which should be observed.

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

Petroleum liquids — Automatic pipeline sampling

0 Introduction

The purpose of collecting a sample of the material flowing through a pipeline is to determine the mean composition and quality of the bulk quantity. Samples of the bulk quantity in the line may be analysed to determine composition, water and sediment content, or any other important attributes such as density, viscosity or, with special precautions, vapour pressure.

Manual methods of pipeline sampling are adequate for homogeneous liquids whose composition and quality do not significantly vary with time. If this is not the case, automatic sampling is the recommended procedure since the continuous or repetitive extraction of small samples from a pipeline ensures that any changes in the bulk contents are reflected in the collected sample. In order that the sample shall be as representative as possible it is essential to ensure that the recommendations of this International Standard with respect to the required homogeneity of the liquid at the sampling location and to the required frequency of extraction of the small samples are met.

Consideration should be given to having standby samples provided by manual methods that may be referred to if the automatic sampler fails to perform satisfactorily; however, manual sampling will be subject to uncertainty if pipeline conditions are varying. (See ISO 3170.)

The equipment and techniques described have generally been used for sampling stabilized crude oil, but may also be applied to unstabilized crude oil and refined products provided consideration is given to the relevant safety precautions and the difficulties of sample handling.

Representative sampling of crude oil for density and water and sediment content is a critical process. Extensive studies have shown that, in crude oil transfers, four distinct steps are required for determining representative values:

- a) adequate stream conditioning of the pipeline contents;
- b) reliable and effective sampling, ensuring proportionality between sampling ratio and flow rate in the line;
- c) adequate conservation and transporting of the sample;
- d) adequate conditioning and dividing into parts for accurate laboratory analysis.

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 new equipment not yet developed for commercial use, provided that such equipment enables samples to be obtained that are representative, and is in accordance with the general requirements and procedures of this International Standard.

The annexes to this International Standard contain calculation procedures about pipeline mixing theory and profile testing and also give basic guidelines for sampler location.

It is realized that in many countries some or all of the items covered by this International Standard are subject to mandatory regulations imposed by the laws of those countries; such regulations must be rigorously observed. In cases of conflict between such mandatory regulations and this International Standard, the former should prevail.

1 Scope and field of application

1.1 This International Standard recommends procedures to be used for obtaining, by automatic means, representative samples of crude oil and liquid petroleum products being conveyed by pipeline.

NOTE — Although throughout this International Standard the term crude oil is used, this should be taken to include other petroleum liquids where the technique and equipment are also applicable.

1.2 This International Standard does not apply to the sampling of liquefied petroleum gases and liquefied natural gases.

1.3 The principal purpose of this International Standard is to give guidelines for specifying, testing, operating, maintaining and monitoring crude oil samplers.

1.4 The sampling procedures for crude oil are intended to provide representative samples for the determination of

- a) the oil composition and quality;
- b) the total water content;
- c) other contaminants that are not considered to be part of the crude oil transferred.



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If the sampling procedures for a), b) and c) are in conflict, separate samples may be required.

NOTE — The results of the laboratory analysis may be used for calculating adjustments to the declared quantity of crude oil transferred. The procedure for carrying out the adjustments does not form part of this International Standard.

1.5 Sample handling is included, covering all aspects following collection to the transfer of the sample to laboratory apparatus.

1.6 This International Standard describes the practices and procedures believed at the present time to be the most likely to lead to representative sampling and hence to accurate water determination. However, the accuracy of the water determination on pipeline samples obtained using automatic samplers will depend upon the arrangement and characteristics of all the various elements making up the sampling system, and on the accuracy of the subsequent analytical procedures.

A theoretical technique for evaluating the combined accuracy of the automatic sampling system and the analytical test is given in clause 16. A practical test procedure for field use is described in clause 15.

Normally, the acceptable accuracy limits for a particular automatic sampling system will be specified in agreement between the interested parties.

Table 4 of clause 15 classifies the performance of automatic sampling systems by ratings based on the accuracy of practical test results. These ratings may be used as a guide to possible performance and as a basis for individual agreement.

2 References

ISO 3165, *Sampling of chemical products for industrial use — Safety in sampling*.

ISO 3170, *Petroleum products — Liquid hydrocarbons — Manual sampling*.

NOTE — See also clause 17, Bibliography.

3 Definitions

For the purpose of this International Standard, the following definitions apply.

3.1 acceptable (accuracy) limits: The limits within which the determined concentration of water in a sample is acceptable relative to the true value or other specified value, at the 95 % probability level.

3.2 automatic sampler: A system capable of extracting a representative sample from the liquid flowing in a pipe. The system consists of a sampling probe and/or a separating device, an associated controller and a sample receiver.

3.2.1 intermittent sampler: A system for extracting liquid from a flowing stream, a sample receiver to contain the sample grabs taken from the stream, and a means for controlling the amount of sample taken by varying the sampling frequency or grab volume in relation to flow rate.

3.2.2 continuous sampler: A system for extracting liquid from a flowing stream which has a separating device which continuously withdraws liquid from the main pipeline in relation to flow rate, an intermediate sample receiver, and a means for controlling secondary withdrawal to a final sample receiver.

3.3 calculated sample volume: The theoretical sample volume obtained by multiplying the sample grab volume by the number of actually collected grabs.

3.4 competent person: A person who by reason of his or her training, experience, and theoretical and practical knowledge is able to detect any defects or weaknesses in the plant or equipment and to make an authoritative judgement as to its suitability for further use.

NOTE — This person should have sufficient authority to ensure that the necessary action is taken following his or her recommendation.

3.5 controller: A device which governs the operation of the automatic sampler in order to provide a representative sample.

3.6 fixed-rate sample; time-proportional sample: A sample taken from a pipeline during the whole period of transfer of a batch, composed of equal increments at uniform time intervals.

3.7 flow-proportional sample: A sample taken from a pipeline during the whole period of transfer of a batch, at a rate which is proportional to the rate of flow of the liquid through the pipeline at any instant.

3.8 grab: The portion of liquid extracted from the pipe by a single actuation of the separating device. The sum of all the portions results in a sample.

3.9 homogeneous mixture: A liquid is homogeneous if the composition is the same at all points. For the purposes of this International Standard a liquid is homogeneous if the variation in composition does not exceed the limits provided in 4.4.

3.10 integrity of the sample: The 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.

3.11 isokinetic sampling: Sampling in such a manner that the linear velocity of the liquid through the opening of the sampling probe is equal to the linear velocity of the liquid in the pipeline at the sampling location and is in the same direction as that of the bulk of the liquid in the pipeline approaching the sampling probe.

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

3.12.1 powered mixer: A mixing device which depends on an external source of power for the energy required to mix the liquid.

3.12.2 static mixer: A mixing device having no moving parts and located within a pipe or tube. It depends on the kinetic energy of the moving liquid for the energy required to mix the liquid.

3.12.3 variable-geometry static mixer: A mixing device with parts inside the pipe or tube which can be adjusted to modify its characteristics at different flow rates.

3.13 pipeline: Any section of pipe used for the transfer of liquid. An unobstructed pipe does not have any internal fittings such as a static mixer or orifice plate.

3.14 profile testing: A technique for simultaneous sampling at several points across the diameter of a pipe. Terms used in connection with profile testing are as follows:

3.14.1 overall mean: The average of either the point averages or the profile averages. (Note that the result is the same.)

3.14.2 point: A single sampling orifice in the profile.

3.14.3 point average: The average of the water concentration at the same point in all profiles (neglect points with less than 1 % water).

3.14.4 profile: A set of samples taken simultaneously at several points across a diameter of the pipe.

NOTE — The term is also used to denote the series of sampling points themselves and the set of results obtained by analysis of the samples taken at these points.

3.14.5 profile average: The average of the water concentration at each point in the same profile (neglect the profile if it has less than 1 % water).

3.15 representative sample: A sample having its physical or chemical characteristics identical to the average characteristics of the total volume being sampled.

NOTE — Since errors cannot be quantified exactly, compliance with this ideal can only be expressed as an uncertainty that can be obtained either from practical tests or by theoretical calculation.

3.16 sample: The portion of liquid extracted from the pipeline that is subsequently transferred to the laboratory for analysis.

3.17 sample conditioning: Homogenization necessary to stabilize the sample during sample handling in preparation for analysis.

3.18 sample container: A vessel used for the storage, transportation and preconditioning of the total quantity, or a proportion of the total quantity, of the sample for analytical work or for division into identical small sub-samples to be analysed.

3.19 sample handling: The conditioning, transferring, dividing and transporting of the sample. It includes transferring the sample from the receiver to the container and from the container to the laboratory apparatus in which it is analysed.

3.20 sample loop: A by-pass to the main pipeline being sampled through which a representative portion of the total flow is circulated.

3.21 sample receiver; receptacle: A vessel connected to the automatic sampler in which the sample is collected during the sampling operation. A receiver may be permanently attached to the sampler or it may be portable. In either case, it should be designed to maintain the integrity of the sample.

NOTE — In certain circumstances, it is possible to collect the total sample in more than one sample receiver. In such circumstances, the sample integrity has to be maintained for each individual sample volume.

3.22 sampler performance factor (PF): The ratio between the accumulated sample volume and the calculated sample volume (see 14.6).

3.23 sampling frequency: The number of grabs taken in unit time.

3.24 sampling interval: The time between successive grabs.

3.25 sampling location: The cross-section of the pipe where the sampling probe is, or is proposed to be, located.

3.26 sampling probe: The sampler element that extends into the pipeline.

3.27 sampling ratio: The quantity of pipeline contents represented by one grab.

NOTE — It can be expressed as either the volume, in cubic metres per grab, or the equivalent length of pipeline, in metres per grab.

3.28 separating device: A device that separates a small volume of liquid from the batch of liquid that the small volume represents.

3.29 stream conditioning: The distribution and dispersion of the pipeline contents, upstream of the sampling location.

3.30 time-proportional sample: See 3.6.

3.31 water.

3.31.1 dissolved water: The water contained within the oil forming a solution at the prevailing temperature.

3.31.2 suspended water: The 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.

3.31.3 free water: The water that exists as a separate layer from the oil, and typically lies beneath the oil.

3.31.4 total water: The sum of all the dissolved, suspended and free water in a cargo or parcel of oil.

3.32 worst-case conditions: The operating conditions for the sampler that present the most uneven and unstable concentration profile at the sampling location.

NOTE — This will usually be at minimum flow rate, minimum oil density and minimum oil viscosity but may also be influenced by other factors such as emulsifiers and surfactants.

4 Principles

4.1 Purpose

This clause defines the principles which it is essential to observe during sampling operations in order that the representativity of the sample taken corresponds to the specifications of this International Standard, and meets the acceptability criteria given in 4.4.

4.2 Principles to be observed

In order to determine the oil composition, quality and total water content of a batch of crude oil, samples that are representative of the batch are taken and analysed. The batch may be either a discrete pipeline transfer over a given period of time, or the whole or part of the cargo of a tanker, either loading or unloading.

Representativity depends on four conditions, all of which should be observed, since failure to comply with any one of them could affect the quality of the final result.

4.2.1 The first condition is that the samples that are taken from the pipeline should have the same composition as the average composition of the crude oil over the whole cross-section of the pipeline at the location and time of sampling. It is not easy to comply with this condition, because of the possibility of a variable concentration gradient existing across the section.

This condition requires that at the sampling location:

a) the distribution or concentration of the water in the crude oil should be uniform across the section of the pipeline within the acceptance limits given in 4.4;

b) the diameter of the entry port of the sampling probe should be large in relation to the maximum water droplet size. The port opening should not be smaller than 6 mm (see 7.3).

4.2.2 The second condition is that representativity should persist throughout the period of transfer of the batch, the composition of which may change between the start and finish of sampling. The rate of sampling, whether it is continuous or intermittent, should be in proportion to the flow rate in the pipe. When an intermittent sampler is used the sampling frequency and grab size should both be sufficient to guarantee acceptable representativity.

Furthermore, the representativity of the sample should be maintained in the automatic sampler from the sampling probe up to the final receiver. Samples should be taken with an appliance that complies with the recommendations in clauses 7, 8, 9 and 10.

4.2.3 The third condition is that the sample should be maintained in the same condition as at the point of extraction, without loss from it of liquid, solids or gases and without contamination.

Storage and transfer of samples should comply with the recommendations in clause 11.

4.2.4 The fourth condition concerns division of a sample into a number of sub-samples in such a way as to ensure that each of them has exactly the same composition as the original sample.

The procedure for dividing each sample into sub-samples, and for transferring them to laboratory appliances, is given in clause 12.

NOTE — It should be emphasized that this fourth condition concerns a critical activity and any error introduced is capable of destroying the representativity achieved by the first three.

4.3 Sampling tolerances and validation

In order to ensure that each sample sent to the laboratory for analysis is representative of the whole batch, the composition of the sample should not differ from the composition of the batch by more than the tolerances given in table 4 and as applied in 15.5.

In order to ensure that any departure from the conditions given above (see 4.2) does not result in a sample representativity which exceeds the tolerances given in table 4, each step of the sampling operation should be validated as shown in figure 1.

4.4 General principles for sampling

Hydraulic laws governing the behaviour of heterogeneous liquids which will mix or will not mix in the pipe show that for stream conditioning a sufficiently high energy dissipation rate should be provided to keep drops of water and heavier solid particles suspended in the crude oil. Such an energy dissipation rate may be provided either from the velocity in an unobstructed pipe, or from a mixing device immediately upstream of the sampling location.

In considering the distribution of water over the cross-section, the acceptable limits of the values found in the profile test (clause 6) should be relative to the mean concentration of water in that plane and should be within $\pm 0,05$ g/100 g for samples having a water content up to 1 g/100 g and should be ± 5 % of the mean concentration (relative) for samples having a water content greater than 1 g/100 g [but see also case 2 (4.4.2)].

NOTE — Although the concentrations above are quoted in mass units, they also apply to volume units.

In a horizontal pipe, three cases may be used to describe the ways in which the concentration of the different phases may vary over the cross-section of the pipe depending on hydraulic conditions (flow rate, product density and viscosity, dispersed phase composition, interfacial tension modifiers, etc.).

4.4.1 Case 1 (see figure 2, profile type 1)

In this case, the concentration is the same, within the acceptable limits as defined above, across the entire cross-section of the pipeline for all concentrations of water. The existing conditions are acceptable for sampling, since water is evenly distributed over the pipe cross-section. A representative sample consequently exists at the inlet of the sampling probe, which can be placed at any point on the diameter although it is essential that care should be taken not to place the sampling probe too near the wall in order to minimize wall effects.

4.4.2 Case 2 (see figure 2, profile type 2)

In this case, the concentration varies from one point to another in the cross-section, but with a uniform gradient such that there is at least one point where the concentration is equal to the mean concentration across the entire section. Based on a theoretical analysis, this point is generally found between 0,4 and 0,5 of the vertical diameter from the bottom of a horizontal pipeline.

Sampling at this location will only be acceptable if the water concentration found at the sampling point under worst-case conditions is equal to the mean concentration within the acceptable limits as defined above.

4.4.3 Case 3 (see figure 2, profile types 3a and 3b)

In this case, the concentration across the cross-section of the pipe is non-linear, indicating the presence of segregation (type 3a). Some erratic profiles may also be encountered (type 3b).

As there is a range of concentrations at different points in the cross-section, sampling at a predetermined point is unacceptable, and it will be necessary to install a mixing device (see 5.3).

NOTE — If there is any free water, or an emulsion having a high concentration of water, at the bottom of the pipe, representative sampling is not possible.

4.5 Dispersed phase — variations with time

It is unlikely that the concentration of a dispersed phase component in the bulk liquid will be constant with time. For example in the discharge of crude oil from a marine tanker, in addition to more gradual changes in the base water content, there may also be periods when peaks of relatively high concentrations of water travel down the pipeline. Experimental observations indicate that these "transients" may contain over 50 % water and may be shorter than a minute in duration. Depending on the unloading procedures, the significance of the water discharged in the form of transients may vary relative to the base level carried with the bulk of the discharged cargo.

It is apparent that the representativity of samples taken in such applications will be dependent upon the ability of the automatic sampler system to reflect, both accurately and proportionally, the integrated water content of these peaks in the total collected sample volume.

With intermittent-type samplers, accuracy will depend on the type of equipment and its frequency of operation in relation to the frequency and duration of the transients. With continuous-type samplers, accuracy will depend on the external collection and mixing arrangements and on the rate of secondary sampling if applied. For both types of sampler, the overall duration of the oil transfer, the duration of any water transients and the sampling frequency have statistical significance in the determination of sampling accuracy.

Theoretical analysis of the effect of dispersed phase transients on the performance of the different types of samplers leads to the following general conclusions:

- a) in short-term transfers in which there is a possibility of frequent, short-duration transients, the accuracy of the continuous type of automatic sampler is least affected by the transients;
- b) in short-term transfers in which there may be relatively few long-duration transients, the accuracy of the intermittent-type sampler approaches that of the continuous type of automatic sampler;
- c) for long-term transfers, the mean error introduced by transients of any duration, using either type of sampler, is within the limits of acceptability defined in this International Standard.

4.6 Low water content

Attention is drawn to the fact that if the concentration of water is about 0,1 % (m/m), i.e. near the level at which water is soluble in crude oil, the concentration profiles will show a good uniformity of water distribution under all hydraulic conditions.

5 Selection of sampling point (including stream conditioning)

5.1 General

As has been emphasized in clause 4, it is essential that the sampling probe is presented with a portion of the liquid flowing in the pipeline which is representative of the whole. This depends on the extent of mixing within the pipeline, which in turn depends on a number of factors including flow rate and pipe configuration upstream of the sampling location.

The guidance given in this clause for the selection of the sampling point assumes that the pipeline remains full at all times.

5.2 Initial selection of sampling probe location

5.2.1 The force of gravity tends to promote stratification in horizontal pipelines whereas distribution tends to be more uniform in vertical lines. For this reason, given the choice between vertical and horizontal, and assuming identical operating conditions, the preferred location is in a length of vertical pipe, provided that the pumping rate is significantly higher than the water and sediment settling rate (see annex A, A.2.2).

Reference should be made to annex C for a useful guide to the initial screening of potential sampling locations but annex A is preferred as it provides a more detailed treatment.

5.2.2 The turbulence which exists naturally in a long horizontal pipeline can be, but frequently is not, sufficient to provide adequate mixing. The minimum natural turbulence for adequate mixing depends on flow rate, pipe diameter, viscosity, density and interfacial tension. If the natural turbulence is not sufficient for representative sampling, then the additional mixing offered by specific pipeline components should be introduced (see 5.3 and annex A).

5.2.3 The minimum acceptable flow rate and/or the minimum energy to be provided by a mixing device should be determined. Suitable procedures using formulae or nomographs described in annex A may be used.

5.3 Mixing devices

5.3.1 General

Mixing devices produce homogeneity by the addition of kinetic energy from outside or the conversion of pressure energy from within the pipeline.

In situations where there is an insufficient rate of energy dissipation for pipeline fittings or static mixers to be effective, consideration should be given to the use of powered mixers.

Reference should be made to annex C for a useful guide to the initial screening of potential sampling locations.

5.3.2 Pipeline fittings

Pipeline fittings may be used as in-line mixing devices. The fittings which may be suitable in this respect are valves, orifices, pipe reducers and expanders, manifolds, T-junctions and metering stations. These, however, vary in their effectiveness as mixers and may create concentration zones, leading to zigzag concentration profiles over a downstream distance equivalent to about 20 pipe diameters.

5.3.3 Reduced pipe diameter

If the velocity is too low for sufficient mixing, a length of pipe of reduced diameter can often be installed to advantage but not to the extent that the obstruction caused by the sampling probe will distort the water distribution (see 7.2).

5.3.4 Vertical loops

The improved distribution afforded by a vertical length of pipeline (see 5.2.1) can be obtained by inserting a vertical loop into a horizontal line. If sufficient line pressure is available the flow rate may be increased by reducing the diameter in the vertical loop to below that of the main line. In a vertical loop, the sampling probe should be preferably installed in the downstream leg, at a minimum distance of three, but preferably greater than five, pipe diameters from the upstream bend and a minimum distance of 0,5 pipe diameters from the downstream bend.

It should be noted that water can accumulate at the bottom of vertical piping until these accumulations reach such a size that they are carried forward in the form of slugs. As a result of this phenomenon, water transients occur and this should be taken into account.

5.3.5 Static mixers

Static mixers are commercially available devices specially designed in one or more stages to provide adequate distribution and dispersion of the pipeline constituents prior to sampling. Reference should be made to the manufacturer for advice on the minimum flow rate at which the mixer is still effective.

The maximum flow rate will be limited by the acceptable pressure drop across the mixer, but a wide flow range can be accommodated using a variable geometry mixer.

5.3.6 Powered mixers

Powered mixers can be dynamic versions of the static mixer in which the mixer elements are driven from an external power source to introduce a high shearing action which effects the maximum dispersion of the contacting media. Alternatively, mixing may be achieved when a portion of the flow is re-introduced into the pipeline via a pump and high-velocity injector(s), upstream of the sampling probe.

5.4 Position of the sampling probe

5.4.1 If the contents of the pipeline have been adequately mixed the position of the sampling probe relative to the pipe wall is relatively unimportant. However, to avoid wall effects, which could influence the flow rate as well as the water concentration, the sample should be drawn from the centre of the pipe. In horizontal or vertical pipes the sampling probe should be positioned to withdraw from the shaded areas shown in figure 3.

5.4.2 Under some conditions, centrifugal forces occurring at bends may act to separate the dispersed phase from the bulk liquid. Therefore the exclusive use of single bends to provide mixing upstream of the sampling location is not recommended.

5.4.3 The distance between the mixing device and the sampling probe should be sufficient to avoid the effects of swirl and asymmetry generated by the device but not so great as to lose the mixing effect provided by the device. Recommended distances are between one-half of a pipe diameter and 8 pipe diameters, depending on the type of mixing device being used.

5.5 Checking the location of the sampling probe

Whatever location is chosen for the sampling probe, or whatever additional form of mixing is employed, it is recommended that the concentration profile at the sampling location be checked. The test procedure for this check is described in clause 6. Other methods of checking may be used if they provide equal assurance that the sample taken will be representative.

6 Profile testing

6.1 Introduction

This clause describes methods which may be used to test the uniformity of water distribution across the pipeline at the chosen sampling location.

6.2 Principle

Profile testing can verify that a representative sample is available at the proposed or existing sampling location. The extent of stratification or non-uniformity of water concentration can be determined by taking samples simultaneously at several points across the diameter of the pipe and analysing these for water content. A multipoint probe, as opposed to a traversing probe, is required to eliminate uncertainties due to non-simultaneous sampling.

In order to make a valid judgement regarding uniform dispersion, a minimum of five profile tests should be made, although only three profile tests are required to demonstrate stratification. For acceptable results when using manual methods of test, the mean water concentration should be between 1 and 5 %.

NOTE — If centrifuge tubes are being used for the water determination and the water concentration is above 20 % extreme care should be taken when taking the readings because of the larger volumes between graduations.

If an automatic continuous method of determining water content is employed, then the water content should be within the working range of the instrument. To cope with conditions of complete water separation, the instrument should tolerate 100 % water without failure.

6.3 Methods

The choice is between three methods of test, depending upon the level of water concentration that can be assured throughout the period of testing and the ability to achieve reliable and stable conditions. Generally, a level of at least 1 % water is necessary for a valid test.

- Method 1 employs the injection of additional water into the pipeline. It is the preferred procedure and should be used whenever there is doubt that the natural level of water concentration in the crude oil will be consistently more than 1 % during the test period.

- Method 2 relies upon the natural level of water concentration. To be valid, this method should only be used when there is certainty that the natural water concentration in the crude oil during the period of test will be at least 1 %.

- Method 3 is an alternative method for use with ships. Under some circumstances additional water may be introduced into the ship's compartments or storage tanks to increase the level of water concentration to at least 1 %.

NOTE — A water concentration of 1 % is enough to determine whether the dynamics of the system can provide adequate mixing while minimizing the effects of the inherent measurement error.

6.4 Determining the water concentration profile in a pipeline and validation of the sampling location

6.4.1 Equipment

6.4.1.1 Multipoint sampling probe

The test is performed using the multipoint probe illustrated in figure 4. The probe openings should face upstream into the direction of flow and are internally reamed to minimize entry flow disturbances. The probe should be fitted horizontally into a vertical pipe, or vertically into a horizontal pipe. When the probe is fitted vertically, the recommended direction of flow in the probes is vertically downward where possible. In horizontal pipes, an extra straight-tube without bend is positioned vertically with the entry opening as near as practicable to the bottom wall of the pipeline (see figure 5). This point is used to check that no free water is running along the bottom of the pipe.

The centre of the tube opening of the first and last points of sampling should be located 20 mm from the wall of the pipeline. All tubes leading to the external valves should be approximately the same length to ensure that simultaneous samples are taken.

A probe with a minimum of 5 sample points is recommended for 300 mm (12 in) pipe size or larger. Below 300 mm (12 in) pipe size, three sample points should be used.

NOTE — The internal bore of the tubes should be large enough to avoid blockages. For crude oil, 6 mm is the recommended minimum internal diameter.

As a safety precaution, the probe should be installed and removed during low pressure conditions. However, the probe should be equipped with safety chains and stops to prevent blow out should it be necessary to remove it during operating conditions. (See also clause 13.)

6.4.1.2 Measurement

The measurement of water content at each point across the pipeline may be made by either manual or automatic means.

When measurement is made by a manual laboratory technique, other than the centrifuge method, the sample should be collected into a receiver of adequate capacity for the test, following the procedure described in 6.4.2. When the centrifuge method is used, the sample may be collected directly into the centrifuge tube, but it may be difficult to dispense the exact volume.

Alternatively, when continuous monitors are employed (for example capacitance cells), the samples are passed directly through individual measurement cells, one for each profile point. This procedure has obvious advantages in that profile changes can be seen as they occur by observation of suitable indicating or recording apparatus. This automatic method also avoids the necessity for a very large number of repetitive laboratory tests.

With both methods, provision should be made for the collection and disposal of waste oil passed through the probes during the test period.

6.4.2 Operating procedure

If the application requires a range of crude oils to be sampled, the worst case conditions shall be selected. If this is not possible the formulae in annex A can be used to extrapolate the results to other conditions. The results of extrapolation should be treated with great care.

6.4.2.1 Manual measurement

- a) The sampling frequency should provide at least one profile every 2 min and at least five profiles at the minimum flow rate with a water concentration of at least 1 % (m/m).

- b) If method 1 (see 6.3) is used, water shall be injected upstream from the multipoint probe, at a sufficient distance from the probe and upstream of any mixing elements, so that a representative profile is obtained, but in a manner which will not create an energy dissipation rate significantly higher than the existing one [see annex A, A.4.2.2, equation (26)].

- c) The injection operation should be made in accordance with figure 6, using a pump that will produce the proper flow rate and pressure. If necessary, water should be injected in successive percentages of 1, 2, 3, 4 and 5 % or more of the volume of crude oil flowing in the pipeline. The operation should be repeated at five representative but different flow rates and particularly at the minimum flow rate. These should be constant to within 10 % during the period of each test.

- d) If method 3 (see 6.3) is used, the ship's compartment to be spiked with water should be selected such that it can be pumped without creating trim problems.

- e) Each element of the probe should be rinsed out thoroughly before sampling by running through it an adequate amount of crude oil.

- f) Flow rates in the multipoint probe should be regulated before sampling so that the entry velocity at each point of the probe is the same and is preferably equal to the velocity of the liquid flowing in the pipeline. If not, any differences should be recorded.

- g) Sampling should begin before the calculated water arrival time and continue, without alteration of the flow rates in the probe, until sufficient profiles have been taken to obtain a minimum of five which meet the requirements of 6.2.

- h) Sampling should continue during the time-period that injected water is passing. The time-lag between water injection and arrival at the sampling location should be taken into account (see figure 6).

6.4.2.2 Automatic measurement

- a) The provisions of 6.4.2.1 b), c), d), e), f), g) and h) apply.

- b) A recent development is the use of capacitance cell profile test systems to demonstrate continuously the degree of homogeneity across the profile and with time. The capacitance of water in crude oil mixtures will vary with crude oil composition; it will also vary with temperature and may vary with water droplet size and shape. The individual cells comprising such an automatic profiling system should be zeroed on the particular crude oil being sampled, and steps taken to ensure that temperature and flow rate differences between cells are minimized.

In profile testing, the measurement of interest is the relative concentration of water at each point across the pipe.

Therefore, provided that capacitance cells with similar characteristics are used, there may be no need to adjust the span for different crude oil types.

c) Timing is much less critical than for manual testing, as is the need for steady conditions. A visual display will indicate the profile conditions as they occur, enabling the worst (or best) conditions to be identified.

d) Figure 7 shows a typical bar graph presentation, based on experimental work, from a profile test at three different sampling locations along the pipe.

6.4.3 Typical test data

6.4.3.1 Manual measurement

Annex B shows some typical test data from a profile test at a crude oil terminal. Tests were made at two flow rates and six profiles were obtained for eight points across the pipe for each flow rate.

After excluding all profiles where the water content is less than 1 % (m/m), the point averages, profile averages and overall mean are calculated. The next step is to obtain the percentage deviation of each point average from the overall mean. Profiles should be drawn as shown in annex B.

6.4.3.2 Automatic continuous measurement

With automatic profile test equipment, using continuous water monitors connected to a microprocessor, it is possible to continuously display and record the water profile in any suitable form such that changes caused by flow rate variations or injected water percentage can be observed as they occur. The effect of variable energy mixing devices can also be observed.

Similarly, calculation of mean and average water contents and percentage deviations at points across the pipe can be carried out instantaneously. These results can be displayed or recorded with the profile and other test data.

6.4.4 Presentation and interpretation of results

Each profile may have the general appearance of one of the types P_0 to P_5 shown in figure 8.

a) P_0 represents the most suitable distribution, since the concentration ratio is within the acceptable accuracy limits at any point of the cross-section.

b) P_1 and P_2 represent acceptable distributions, provided that the sampling probe is positioned according to the recommendations in 4.4.2 and 5.4.1.

c) P_3 to P_5 represent unacceptable distributions with concentration ratios outside the acceptable accuracy limits.

Any profile beyond the acceptable accuracy limits indicates that sampling results cannot be expected to be representative. To achieve an acceptable distribution either a higher flow rate or a mixing device should be used.

Annex A gives a simplified theoretical relationship between flow rate and oil characteristics on the one hand, and the profile on the other hand.

7 Sampling probe design

7.1 The sampling probe and/or the separating device should be of sufficient strength to withstand the bending moments imposed by the maximum flow in the main pipeline and to withstand the vibrations caused by vortex shedding. Although the actual determination of strength is difficult, conservative approaches can be made by treating the member as a cantilevered beam.

7.2 The sampling probe should be designed to create the minimum of disturbance to the main pipeline flow. One approach is a pitot-tube type probe entry with a chamfered edge. The entry should face upstream into the direction of the flow.

7.3 It is recommended that the diameter of the sampling probe opening be not less than 6 mm.

8 Sampler design and installation

8.1 Design

8.1.1 Automatic samplers should be designed to procure representative samples of the liquids flowing through pipelines and to store these in one or more receivers. Samplers may be of the continuous or intermittent type.

NOTE — There are two types of intermittent automatic sampling system. One system locates the separating device directly in the main line [see figure 9a)], whereas the other system locates the separating device in a sample loop [see figure 9b)].

8.1.2 The petroleum liquids to be sampled may contain wax, abrasive particles and corrosive components such as sulfur compounds and water. They may, in certain cases, possess poor lubricating properties. These facts should be taken into account in designing an accurate, reliable and durable sampling system.

8.1.3 The installation of piping and valves between the separating device and the receiver should be so designed that there is no separation of components, such as water and crude oil, at any point. It is desirable to keep the volume to a minimum.

8.1.4 A sample loop usually consists of a circulating loop, a suitable pump and a separating device [see figure 9b)]. Any connection between the loop and the separating device should be of minimum volume.

8.1.5 The sample loop entry velocity should be as near as possible to the maximum expected velocity in the main pipeline. If the separating device is not in the immediate vicinity of the sampling probe, care should be taken ensure that the flow rate within the sample loop is high enough to produce fully turbulent flow and thus avoid water settlement.

8.1.6 The inclusion of a device to indicate that there is a flow in the sample loop is recommended. If there is no circulation in the sample loop and sampling continues, erroneous sampling will result.

8.2 Installation

8.2.1 The separating device and the sample receiver should be installed as close as possible to each other in order to minimize dead volume. Connections should be made with short lengths of pipe consistent with the application, and the number of bends should be kept to a minimum. Where possible, the sample receiver should be at the lowest point in the system.

8.2.2 Where for practical reasons it is not possible to install the separating device and the sample receiver close to each other, then a sample loop is recommended. Precautions should be taken to ensure that both the sample in the sample loop and the sample taken from the sample loop are representative.

8.2.3 Automatic samplers should be installed with suitable valves and connections to enable equipment to be flushed automatically or manually either with the liquid being sampled or with some suitable solvent. Proper arrangements should be made for the disposal of the flushings and solvents used. All solvents should be removed from the receiver and the circulating lines to avoid contamination of the next sample.

8.2.4 If necessary, a non-return valve or equivalent device should be installed in the sampling line to prevent any possibility of the sample being returned to the pipeline from which it was drawn. This should not restrict the free passage of sediment into the receiver.

8.2.5 The method of installation of an automatic sampler should provide easy access for maintenance and cleaning; it may be essential that this can be carried out without shutting down the pipeline.

8.2.6 The sampling probe should be installed through a stub-mounted full-bore valve and a suitable insertion and withdrawal mechanism should be provided. If the probe has to be withdrawn without depressurizing the pipeline, means, such as safety chains, should be provided to prevent the probe being ejected by the pipeline pressure. If this system is installed, sufficient space should be allowed for fitting the mechanism and withdrawing the probe.

8.3 Precautions

8.3.1 There may be a tendency for water or heavy particles to drop out in the sampler tubing and components, and appear in

the sample receiver during subsequent sampling. To minimize this, the system should be free from pockets or enlarged sections and should be pitched downwards towards the receiver. Care should also be taken to ensure that there are no pockets in which vapours or water can be trapped.

8.3.2 To prevent solidification of high-pour-point crude oils or products, the separating device, associated tubing and components should be heat-traced and insulated. Care should be taken not to over-heat the sample liquid, although the temperature should be high enough to keep the material in the liquid phase to ensure proper operation of the automatic sampler system. Thermostatic temperature control may be necessary if self-limiting heat tracing is not used.

8.3.3 If sampling lines and systems which are not in use are likely to block through cooling and wax deposition, flushing oil should be used to fill such lines and systems.

8.4 Special characteristics

The general recommendations on automatic samplers for crude oil are applicable to automatic samplers for refined products. However, certain products have specific characteristics which may require special sampling conditions. Some examples are given in table 1. In these cases, the automatic sampler should be adapted so that it fulfils the required sampling conditions.

9 Control equipment

9.1 Function

The functions of the control equipment for an automatic sampler are to control the separating device to take either a flow-proportional sample or a fixed-rate sample, and it may also include a system to

- a) continuously monitor the number of grabs and the volume of sample collected;
- b) verify, at any time during sampling, the proportionality between the sample collection rate and the pipeline flow rate if operating in the flow-proportional mode.

NOTE — Continuous monitoring of density and/or water content may be used as an added monitoring facility.

9.2 Control equipment

9.2.1 The control equipment may be remote from the sampler in a control centre. Alternatively, it may be local to the sampler, in which case consideration should be given to the electrical safety classification of the hazardous area in which it is installed.

9.2.2 The control equipment should enable the operator to

- a) set the equipment for the prospective cargo or batch size or for the necessary sampling rate to meet the required operating criteria (see 14.2);
- b) put the sampler into operation, and switch it off.

9.2.3 Facilities should be provided for monitoring the following during operation:

- a) the flow rate in the main pipeline;
- b) the sample receiver to indicate that the sample volume collected is proportional to the totalized flow in the main pipeline;
- c) the number of grabs.

9.2.4 The equipment should provide an alarm if the flowmeter is indicating flow but no sampling is taking place and vice versa.

9.2.5 Alarms are recommended for

- a) high level in the sample receiver;
- b) low flow in the main pipeline;
- c) low flow in the sample loop;
- d) loss of power;
- e) sampling probe failure (for motor-driven probes).

10 Flow measurement

10.1 Accuracy and rangeability

A flow-proportional sampler requires a flow signal to pace the sampler. The flow transmitter should have enough rangeability (turndown) to produce a reasonably accurate signal over the expected flow range of the pipeline. The accuracy of the flow signal should not degrade the representativity of the sample beyond the tolerance of the analytical technique (see clause 16). An accuracy of better than $\pm 10\%$ of actual flow over the operating flow range should be achieved. When the water concentration figure is higher at low flows it is particularly important that the flowmeter produces a reasonably accurate signal at the minimum flow.

10.2 Use of custody transfer meters for samplers

If the location uses meters for custody transfer, the signals from the custody transfer meters should be used to pace the sampler. If the pipeline flow is measured by several meters, the sampler may be paced by an electronically combined total flow signal or a separate sampler may be provided for each meter.

10.3 Special flowmeters for samplers

If the measurement for custody transfer is by shore tank gauging, a separate flowmeter should be provided for flow-proportional sampling.

Table 1 — Recommended sampler characteristics for crude oil and refined products

Pipeline liquid contents ¹⁾	Special characteristics of the automatic sampler		
	Heating of the cabinet containing the sampling system	Provision of a means of separating the sample from the atmosphere	Receiver kept at + 4 °C
Crude oil	Yes ²⁾	Yes ²⁾	No
Naphtha	No	Yes	No
Gasoline	No	Yes	No
Jet fuel	No	Yes	Yes ⁴⁾
Gas oil/domestic fuel oil	No ³⁾	No	No
Heavy fuel 1/heavy fuel 2	Yes	No	No

1) Pipeline conditioning is not required for homogeneous pipeline contents.

2) Only if required, dependent on type of crude oil and operational requirements.

3) Except in severe climatic conditions.

4) Only if necessary (silver corrosion characteristic).

10.3.1 Insertion turbine meters are often used in large-diameter pipelines. The meter signal is a pulse train which simplifies the electronic design of the controller. Rangeability may be limited and accuracy can be seriously affected by changes in the viscosity of the metered liquid. Insertion turbine meters may be fouled by fibrous matter or other debris in the oil, and therefore consideration should be given to the possibility of installing a second flowmeter.

10.3.2 Ultrasonic flowmeters are often used. The flowmeter signal is a pulse train and current which simplifies the electronic design of the controller. Ultrasonic flowmeters are not affected by viscosity. Minimum flow rate is typically 0,09 m/s.

10.3.3 Orifice plate flowmeters may be used; alternatively, either venturi meters or flow pitot tubes may be used to minimize the permanent pressure loss. The pressure drop across the flow element is measured by a differential pressure transmitter which provides an output that is proportional to the square root of the measurement. A single transmitter installation has a range in the ratio of about 4 to 1. Two transmitters, one with a high and the other with a low differential pressure range, may be used to extend the range. However, the controller design for a two-transmitter installation is complicated and problems can arise.

10.3.4 For small flow rates, a line-size positive displacement meter or turbine meter may be used. The meter installation can be simple since it does not have to provide custody transfer accuracy. Proving and calibration facilities are not required.

10.3.5 Other flowmeters, such as vortex shedding meters, may be used.

10.3.6 It may be possible to use a signal derived from a tank level gauge provided that the precision is within the limits recommended for flowmeters in 10.1.

10.4 Precautions

Any meter should be capable of operating both in the presence of a high concentration of water and in the presence of particulate matter or gas bubbles.

The final step of a cargo off-loading operation is usually the stripping. Consideration should be given to the possibility that during this operation quantities of undefined gas might be fed into the unloading line, simulating a liquid flow at the flowmeter output and also damaging mechanical meters by operating them too quickly.

11 Sample receivers and containers

11.1 Sample receiver

The sample receiver should be equipped with a level indicator or equivalent device, for example a weighing system, for monitoring purposes, and a high-level shut-off or alarm device.

Means should be provided to mix the contents of the receiver to ensure that representative samples are transferred into laboratory apparatus or other containers.

Two types of sample receiver are in current use.

11.1.1 Sample receivers — fixed volume

These are receivers with a fixed volume into which the sample is admitted so that it contacts the enclosed atmosphere.

11.1.2 Sample receivers — variable volume

These are designed to avoid contact between the crude oil and air, thus preventing possible loss of light ends and water vapour. They may be made of a suitable flexible material so that they are fully collapsible or they may consist of a container with a piston or diaphragm precharged with nitrogen or helium. The latter are particularly applicable to liquid petroleum products with high vapour pressure, and to crude oils.

11.2 Sample container

The sample container may be equipped with an internal homogenizer or with the necessary connections for an external mixing system (see 12.2.3).

11.3 Common characteristics of sample receivers and containers

11.3.1 Fixed-volume receivers should be constructed to provide sufficient space for ullage to remain at the end of the sampling period, and a means of relieving any increase in pressure due to expansion under all conditions. The design of the vessel should be such that any expansion due to heating of the contents to the highest temperature to which the vessel might be subjected will not cause the working pressure to be exceeded.

The vessel should be made of petroleum-resistant material. It should be noted that receivers or containers made of a plastics material may not be suitable for long-term storage of samples because permeability of the plastics may result in differential loss of some of the components of the sample.

The vessel should be capable of being vented through a pressure control valve with an appropriate setting to minimize the loss of low-boiling-point components.

11.3.2 Pressure tests and other inspection work should be performed according to the local regulations. Cleaning and leak testing should be performed at regular intervals.

11.3.3 The design and material of construction should be such that the internal surfaces in contact with the liquid are smooth and of a finish which will prevent the retention of water when the contents are transferred or removed for analysis. The design should be such that they can be easily cleaned.

11.3.4 The necessary connections should be fitted to permit sample transfer from the receiver to the container, and from the container to the laboratory apparatus, without contamination or loss.

All connections should be fitted with blanking plugs or end caps, except for the relief valves. Gauge glass and drain valve internal lips should be avoided. Dead legs are not permitted.

If necessary, the sample receiver and its connections should be thermally lagged and/or heat traced to prevent solidification of the sample and facilitate drainage (see 8.4).

11.3.5 The volume of the sample required for analysis and for retention as a reference should meet the requirements of the particular application. Furthermore the local or national fiscal authority may require a minimum quantity of sample to be taken.

This will determine the minimum size of the sample receiver(s) and container(s). The duration of the sampling period, the grab size, the sampling frequency and the requirements for adequate mixing (homogenization) are also relevant considerations in determining the receiver size.

If the total sample is contained in more than one receiver or container, great care should be taken to account for differing volumes in each receiver or container filled successively when either recombining the results of analysis, for example for the average water content of the whole sample volume, or in the preparation of composite samples for other analysis if required.

11.4 Sample labelling

11.4.1 Sample containers should be labelled. Oil-resistant tie-on or wire-on labels are preferred and these should be permanently marked.

11.4.2 It is recommended that the following information be provided on the label:

- a) place at which sample was drawn;
- b) date and time sampling commenced;
- c) date and time sampling was completed;
- d) sample tag number or identification number of the container;
- e) type of liquid being sampled;
- f) quantity of liquid represented by the sample;
- g) tank number and type, name of ship; flowline reference, etc.;
- h) destination of sample;
- i) signature of person responsible for collecting sample.

12 Sample handling

12.1 General

12.1.1 The method of handling samples between the point at which they are extracted or drawn and the laboratory test bench or sample storage are devised to ensure that the nature and integrity of the sample are maintained.

12.1.2 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, then draw separate samples and apply the appropriate procedure to each sample.

12.1.3 Take particular care in respect of the following:

- a) liquids containing volatile material, 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.

12.1.4 When making up composite samples take great care not to lose light ends from volatile liquids and not to alter water and sediment content. This is a very difficult operation and should be avoided if at all possible.

12.1.5 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.

12.2 Homogenization of samples

12.2.1 Introduction

Procedures are specified for the homogenization of samples that may contain water and sediment, or are in any other way non-uniform, before transfer from the sample container to smaller containers or into laboratory test apparatus. Procedures for verifying that the sample is satisfactorily mixed before transfer are given in 12.3.

It is not possible to agitate manually 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 sub-sampling.

Homogenization may be by various methods. Whichever method is used it is recommended that the homogenizing system produces water droplets below 50 μm , but not less than 1 μm . Water droplets of below 1 μm will give a stable emulsion and the water content cannot then be determined by centrifuge methods.

12.2.2 Homogenization by high-shear mechanical mixer

Insert a high-shear mechanical mixer into the sample container so that the rotating element reaches to within 30 mm of the bottom. A mixer with counter-rotating blades operating at about 3 000 min^{-1} is usually suitable. Other designs may be used if the performance is satisfactory.

In order to minimize loss of light ends from crude oils or other samples containing volatile compounds, operate the mixer 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 12.3).

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

Avoid any significant rise in temperature during the mixing.

12.2.3 Circulation with external mixer

This method can be applied to both permanently sited sample containers and portable containers; for the latter, use a quick-disconnect coupling. Circulate the contents externally using a small pump through a static mixer installed in small-bore piping. Various designs are suitable; follow the manufacturer's operating instructions.

Use a circulation flow rate sufficient to circulate the contents at least once per minute. A typical mixing time is 15 min, but this will vary according to the water content, the type of hydrocarbon and the design of the system. When the whole sample is thoroughly mixed, run off the required quantity of sub-sample from a valve in the circulating line whilst the pump is running. Then empty the container and thoroughly clean the entire system by pumping solvent round until all traces of the hydrocarbon and water have been removed.

12.3 Verification of the efficiency of mixing

12.3.1 Whatever means are chosen for obtaining a sub-sample from a non-homogeneous mixture, verify the suitability of the mixing technique and the time required to obtain a suitably mixed sample.

12.3.2 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 consistent 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.

12.3.3 If the sample does not remain homogeneous for more than a short period of time after mixing (for example if water and sediment are part of the mixture), use the special method for the verification of mixing described in 12.3.4.

NOTE — It may be necessary, owing to the characteristics of the hydrocarbon, to sub-sample while mixing is still in progress.

12.3.4 Ensure that the sample as drawn fills the containers to about three-quarters full, and homogenize the sample for a known period of time, which should be recorded. During this period draw off small portions at regular intervals and test each immediately for water content by a standard method (see 12.3.5). When the test results are consistent record the value obtained as the blank water content.

Add an accurately measured quantity of water, between 1 and 2 %, and homogenize for the same period of time as for the blank and take samples as before. If there is good agreement between the water content determined, taking account of the blank water content, and the quantity of water added, then repeat by adding a further accurately measured quantity of water, again between 1 and 2 %. If the results continue to give good agreement then assume that the mixing time is adequate.

If the results do not show good agreement (within the repeatability of the method), then discard them. Revert to the beginning of the procedure, and use a longer period of mixing.

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

12.4 Transfer of samples

12.4.1 If the sample container is not portable, or if it is inconvenient to take samples directly from the container into laboratory test apparatus, transfer a representative sample into a portable container for transport to the laboratory.

12.4.2 At every stage of 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 12.2.

12.4.3 Verify the mixing time for each combination of container and mixer by one of the methods specified in 12.3.

12.4.4 Complete any transfer of sample within the period during which the mixture is known to be homogeneous and stable. This period is short; do not take longer than 20 min to complete any transfer.