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PETROLEUM PRODUCTS - DETERMINATION OF WATER - COULOMETRIC KARL FISCHER TITRATION METHOD

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard
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SLS ISO 12937:2021
(ISO 12937:2000)

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Sri Lanka Standard
PETROLEUM PRODUCTS - DETERMINATION OF WATER - COULOMETRIC KARL FISCHER TITRATION METHOD

NATIONAL FOREWORD

This standard was approved by the Sectoral Committee on Materials, Mechanical Systems and Manufacturing Engineering and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2021- 06 -10.

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This Sri Lanka Standard is identical with **ISO 12937: 2000** published by the International Organization for Standardization (**ISO**).

This Sri Lanka Standard specifies a method for the direct determination of water in petroleum products boiling below 390 ^{0}C.

TERMINOLOGY AND CONVENTIONS

The text of the International Standard has been accepted as suitable for publication as a Sri Lanka Standard. However, certain terminology and conventions are not identical with those used in Sri Lanka Standards.

Attention is therefore drawn to the following:

a) Wherever the “International Standard” appear referring to this standard they should be interpreted as “Sri Lanka Standard”.

b) Wherever page numbers are quoted, they are “**ISO**” page numbers.
c) The coma has been used throughout as a decimal marker. In Sri Lanka Standards it is the current practice to use a full point on the base line as the decimal marker.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test method or observation shall be rounded off in accordance with **SLS 102**. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this standard.

CROSS-REFERENCES

There are no corresponding Sri Lanka Standards available for references given in Clause 2.

INTERNATIONAL STANDARD

ISO 12937

First edition
2000-11-01

Petroleum products — Determination of water — Coulometric Karl Fischer titration method

Produits pétroliers — Dosage de l'eau — Méthode de titrage Karl Fischer par coulométrie

Reference number
ISO 12937:2000(E)

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 12937 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, Subcommittee SC 6, *Bulk cargo transfer, accountability, inspection and reconciliation*.

Annex A forms a normative part of this International Standard. Annex B is for information only.

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Petroleum products — Determination of water — Coulometric Karl Fischer titration method

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the direct determination of water in petroleum products boiling below 390 °C. It covers the mass fraction range 0,003 % (*m/m*) to 0,100 % (*m/m*). It is not applicable to products containing ketones or to residual fuel oils.

This International Standard may be applicable to lubricating base oils. However, the precision has not been established for these materials.

The precision given in clause 12 is based upon data obtained using dual-cell, dual-electrolyte systems.

NOTE 1 A number of substances and classes of compounds associated with condensation or oxidation-reduction reactions interfere in the determination of water by Karl Fischer titration. In petroleum products, the most common interferences are hydrogen sulfide and mercaptan sulfur, however, mass fractions of these below 0,003 % (*m/m*) as sulfur will not cause significant interference over the range 0,003 % (*m/m*) to 0,100 % (*m/m*) water. Other organic sulfur compounds commonly present such as sulfides, disulphides and thiophenes, do not interfere.

NOTE 2 An alternative procedure is provided for information in annex B for the direct determination of water over the range 0,003 % *(V/V)* to 0,100 % *(V/V)* in petroleum products. The limitations under which this alternative volume measurement may be used are listed in annex B.

NOTE 3 For the purposes of this International Standard, the terms "% (*m/m*)" and "% (*V/V*)" are used to represent the mass and volume fraction of a material respectively.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3170:1988, *Petroleum liquids — Manual sampling.*

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

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

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3 Principle

A sample is visually inspected (see 6.2.1). If clear and bright, and free from both water droplets and particulate matter on swirling, a weighed portion is injected into the titration vessel of a coulometric Karl Fischer apparatus in which iodine for the Karl Fischer reaction is generated coulometrically at the anode. When all the water has been titrated, excess iodine is detected by an electrometric end-point detector and the titration is terminated. Based on the stoichiometry of the reaction, one mole of iodine reacts with one mole of water, thus the quantity of water is proportional to the total integrated current according to Faraday's Law.

If the sample is not clear and bright, or water droplets or particulate matter are observed on swirling, a portion of a solution of sodium dioctylsulfosuccinate is added prior to homogenizing with a mixer. A weighed portion is then treated as described above.

4 Chemicals and materials

4.1 Molecular sieve pellets, type 4A.

Activate in an oven at 200 °C to 250 °C for 4 h. Transfer immediately to a dry sealable bottle or desiccator and allow to cool.

4.2 Xylene, reagent grade.

Dry by adding approximately 100 g of activated molecular sieve (4.1) to 2 litres of xylene. Allow to stand overnight.

4.3 Karl Fischer reagents

Use commercially available reagents that meet the performance requirements described in clause 8.

4.3.1 Anode electrolyte solution (anolyte)

Mix 6 parts by volume of commercial Karl Fischer anode solution with 4 parts by volume of dry xylene (4.2).

NOTE Other proportions of Karl Fischer anode solution and xylene may be used, provided they meet the performance criteria of clause 8.

4.3.2 Cathode electrolyte solution (catholyte)

Use commercially available Karl Fischer cathode solution.

4.3.3 Single Karl Fischer solution, for use in place of dual electrolyte solutions (4.3.1 and 4.3.2) in cells with or without a diaphragm.

4.4 Sodium dioctylsulfosuccinate, reagent grade.

NOTE 1 Sodium dioctylsulfosuccinate is also sold under the names dioctyl ester of sodium sulfosuccinic acid and dioctyl sulfosuccinate sodium salt.

NOTE 2 Other anionic surfactants may be used in place of sodium dioctylsulfosuccinate, provided they meet the requirements of normative annex A.

4.4.1 Sodium dioctylsulfosuccinate solution

Dry sodium dioctylsulfosuccinate (4.4) in an oven at 105 °C to 110 °C for 4 h. Transfer immediately to a dry sealable bottle or desiccator and allow to cool. Once cool, dissolve 10 g of dried sodium dioctylsulfosuccinate in dry xylene (4.2) and make up to 100 ml with dry xylene. The mass fraction of water in this solution shall be less than 0,010 % (*m/m*) when checked by the procedure given in clause 9.

4.5 Water, conforming to grade 3 of ISO 3696.

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5 Apparatus

5.1 Automatic coulometric Karl Fischer titrator, capable of meeting the requirements given in clause 8.

5.2 Non-aerating mixer, capable of meeting the homogenization requirements given in A.3.

NOTE Both insertion mixers and circulating mixers, such as those used with automatic sampling containers, are acceptable provided they comply with the principles of annex A.

5.3 Syringes, of glass, with needles of suitable length such that the tip can reach under the surface of the anolyte when inserted through the inlet-port septum. The bores of the needles used shall be kept as small as possible, but large enough to avoid problems arising from back pressure or blocking whilst sampling.

NOTE 1 Needles with bores between 0,5 mm and 0,8 mm have been found suitable.

NOTE 2 Recommended syringe sizes are

a) 10 µl with a fixed needle for periodic checking of the titrator performance,

b) 1 ml or 2 ml for petroleum product samples, and

c) 10 ml for addition of the sodium dioctylsulfosuccinate solution to petroleum product samples which are not clear and bright, or which contain free water or particulate matter.

5.4 Balance, capable of weighing to ± 0,1 mg.

5.5 Volumetric flask, capacity of 100 ml.

5.6 Sealable bottles or desiccators, to hold activated molecular sieve and dried sodium dioctylsulfosuccinate.

5.7 Ovens, capable of maintaining temperatures of 105 °C to 110 °C and 200 °C to 250 °C.

5.8 Cooling bath, if required, capable of meeting the requirements of 6.2.8.

5.9 Thermometer, capable of measuring the sample temperature to the nearest 1 °C.

6 Sampling and sample preparation (see annex A)

6.1 Sampling

Samples shall be drawn in accordance with ISO 3170, ISO 3171, or an equivalent national standard.

If sampling is carried out manually, use a clear borosilicate glass bottle. If an automatic technique is employed, either collect a separate sample for water determination, or treat the whole sample collected in accordance with 6.2.4.

6.2 Sample preparation

6.2.1 If the sample is not in a container suitable for visual inspection, or is opaque, then it should be treated as if it were not clear and bright (see 6.2.4). It should not be transferred to another container.

6.2.2 Immediately prior to analysis, shake the sample vigorously by hand for 30 s and then, when free from bubbles, visually inspect it. Hold the sample up to the light and examine it for haze or lack of clarity and then swirl the sample to produce a vortex and examine both the bottom of the vortex and the bottom of the sample container for water droplets and particulate matter. Record the visual clarity as clear and bright or not clear and bright. Record whether water droplets or particulate matter were, or were not, observed on swirling.

6.2.3 If the sample is both clear and bright, free from water droplets and particulate matter, proceed in accordance with clause 9.

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6.2.4 If the sample is not clear and bright, or if water droplets or particulate matter were observed on swirling, proceed in accordance with 6.2.5 to 6.2.8.

NOTE The precision of this method for samples which are not clear and bright is critically dependent upon the effectiveness of the homogenization stage which is proved periodically, see normative annex A.

6.2.5 Use a clean, dry 10 ml syringe (5.3) to add a volume of sodium dioctylsulfosuccinate solution (4.4.1) as established by the procedure specified in annex A.

NOTE Correction of the sample water content for the water content of the sodium dioctylsulfosuccinate solution is not required because the latter is negligible.

6.2.6 Record the temperature of the sample in degrees Celsius immediately before mixing.

6.2.7 To ensure homogeneity, mix the sample in the original container immediately prior to analysis. The mixing time, mixing power (speed) and mixer position relative to the bottom of the container, shall be that found to be satisfactory for the material and sample size as established by the procedure given in A.3. The sample volume and water content of the sample shall not exceed the maxima validated in A.3.

6.2.8 Record the temperature of the laboratory sample in degrees Celsius immediately after mixing. The rise in temperature between this reading and the reading in 6.2.6 shall not exceed 2 °C, otherwise loss of sample light ends or loss of water may occur. If this criterion cannot be met, the sample shall be placed in a cooling bath (5.8) prior to carrying out the procedure in 6.2.6.

7 Apparatus preparation

7.1 Due to the known reaction of acetone and other ketones with Karl Fischer reagent, the use of such solvents to dry apparatus, sample syringes, homogenizers and sample receivers, is not permitted.

7.2 Follow the manufacturer's directions for preparation and operation of the titration apparatus.

7.3 Seal all joints and connections to the titration cell to prevent atmospheric moisture from entering.

7.4 If a single Karl Fischer solution (4.3.3) is used, add this solution to the cell.

7.5 If separate electrolyte solutions are used, add the anolyte (4.3.1) to the outer compartment of the titration cell to the level recommended by the manufacturer. Add the catholyte (4.3.2) to the inner compartment of the titration cell to a level 2 mm to 3 mm below the level of the anolyte.

7.6 Turn on the titrator and stirrer. Allow the residual moisture in the titration cell to be titrated until the end-point is reached. Do not proceed beyond this stage until the background current (or background titration rate) is constant and less than the maximum recommended by the manufacturer of the instrument (see the note below).

NOTE High background current for a prolonged period may be attributable to moisture on the inside walls of the titration cell. Gentle swirling of the cell will wash the inside with electrolyte. Also check all fittings to ensure atmospheric moisture does not enter the cell. It is recommended that the titrator be permanently switched on to stabilize to a low background current.

7.7 If the vessel becomes contaminated with a petroleum product, thoroughly clean the anode and cathode compartments with xylene (4.2). If the frit becomes blocked, thoroughly clean with xylene (4.2). Do not use acetone or other ketones for cleaning or drying the apparatus.

8 Apparatus check test

The water titrated is a direct function of the coulombs of electricity consumed. However, reagent performance deteriorates with use and shall be regularly monitored by accurately injecting 10 μl of water (4.5). Suggested monitoring intervals are initially when fresh reagents are used and then after every 10 injections. Replace the anolyte (4.3.1) and catholyte (4.3.2) solutions, or the single Karl Fischer solution (4.3.3), whenever one of the following occurs:

a) the result from a 10 µl injection of water is outside 10 000 µg ± 200 µg, or persistently high;

b) unstable background current, or phase separation in the outer compartment, or petroleum products coating the electrodes;

c) the total petroleum product content of the titration cell exceeds one third of the volume of the anolyte;

d) the titrator displays error messages which suggest replacement of the electrolytes (see the manufacturer's instructions).

9 Procedure

Immediately after sample preparation (see 6.2), use a clean, dry syringe (5.3) to withdraw at least three portions of the laboratory sample and discard to waste. Immediately withdraw a 1 ml to 2 ml test portion of the laboratory sample, clean the needle with a paper tissue and weigh the syringe and contents to the nearest 0,1 mg. Insert the needle through the inlet-port septum, start the titration and, with the tip of the needle just below the liquid surface, inject the entire test portion. Withdraw the syringe, wipe the needle with a clean tissue, and reweigh the syringe to the nearest 0,1 mg. After the end-point is reached, record the water titrated from the display on the titrator (5.1).

10 Calculation

Calculate the mass fraction of water, w, expressed as a percentage, using the following equation:

$$w = \frac{100 m_2}{m_1 \times 10^6} = \frac{m_2}{m_1 \times 10^4}$$

where

m_1 is the mass of the test portion, expressed in grams (g);

m_2 is the mass of water displayed by the titrator, expressed in micrograms (µg).

NOTE If the result is additionally required in terms of % (V/V), it can be calculated using the following equation:

$$\varphi = \frac{w \times \rho}{1\ 000}$$

where

φ is the volume fraction of water, expressed as a percentage;

w is the mass fraction of water, expressed as a percentage;

ρ is the density of the sample of petroleum product, expressed in kilograms per cubic metre (kg/m^3) at 15 °C.

11 Expression of results

Report the mass fraction of water in the sample to the nearest 0,001 % (m/m).

12 Precision

12.1 Repeatability, r

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value in only one case in twenty:

$$r = 0{,}018\,74 X^{0{,}5}$$

where X is the average of the test values being compared.

12.2 Reproducibility, R

The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value in only one case in twenty:

$$R = 0{,}068\,77 X^{0{,}5}$$

where X is the average of the test results being compared.

13 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard;

b) the type and complete identification of the product tested;

c) whether the sample was, or was not, clear and bright, and whether water droplets or particulate matter were observed on swirling;

d) if the sample was not clear and bright, or if water droplets or particulate matter were observed on swirling:

- the type, concentration and quantity of emulsifier used (see clause 6);
- the type of mixer, mixing time, mixing speed and the approximate position of the mixer relative to the bottom of the sample container;
- the temperature of the sample before and after mixing;

e) the result of the test (see clause 11);

f) any deviation, by agreement or otherwise, from the procedure specified;

g) the date of the test.

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Annex A
(normative)

Sample handling

A.1 General

A.1.1 The methods 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.

A.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 test methods 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.

A.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.

A.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.

A.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.

A.1.6 Manual samples of light and middle distillates shall be taken in clear borosilicate glass containers.

A.2 Homogenization of samples

A.2.1 Introduction

Samples which are clear and bright and which contain no water droplets or particulate matter on swirling do not require mixing.

Procedures are specified for the homogenization of samples that are not clear and bright, or which contain water droplets or particulate matter on swirling, 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 A.3.

It is not possible to manually agitate samples of petroleum products containing free water and sediment enough to disperse the water and sediment within the sample. Addition of a portion of a suitable emulsifier solution followed by vigorous mechanical or hydraulic mixing is necessary in order to homogenize the sample prior to transfer or sub-sampling.

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A.2.2 Homogenization by high-shear non-aerating insertion mixer

Add a portion of a suitable emulsifier solution as determined in A.3. Insert the mixer (5.2) into the sample container so that the tip of the shaft reaches to within the distance of the bottom verified in A.3. Mix the sample using the mixing time and speed verified in A.3.

In order to minimize the loss of light ends from samples containing volatile compounds, it is preferable to operate the mixer through a gland in the closure of the sample container. Avoid a rise in temperature during mixing that exceeds 2 °C. To meet this requirement, it may be necessary to hold the sample container in a cooling bath during the mixing operation.

Thoroughly wash the shaft of the mixer with solvent and dry it between test runs with different samples.

A.2.3 Homogenization by non-aerating circulating mixing system

Add a portion of a suitable emulsifier solution as determined in A.3 and circulate, by means a small pump, the contents of either permanently sited or portable containers through a static mixer installed externally in small-bore piping. Follow the manufacturer's instructions for the operation of the specific pump design and capacity chosen.

Use the circulating flow rate, mixing time and position of suction and discharge ports as verified in A.3. When the whole sample is thoroughly mixed, run off the required quantity of subsample 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 have been removed. Dry the system by blowing dry compressed air or nitrogen through it to remove all traces of the cleaning solvent.

A.3 Verification of mixing conditions

A.3.1 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 suitably mixed sample.

A.3.2 Fill a preweighed sample container to 80 % capacity with a sample of the petroleum product under test which is clear and bright and free from water droplets and particulate matter on swirling. Determine the blank mass fraction of water in accordance with the procedure given in clause 9. Calculate the mean blank mass fraction of water.

NOTE It will not be necessary to mix this sample prior to determination of the mass fraction of water.

A.3.3 Reweigh the sample container to the nearest 0,1 g to determine the mass of sample within it. Record the temperature of the sample to the nearest 1 °C. Add sufficient water to increase the mass fraction of water to 0,100 % (*m/m*). Record the mass of this water to the nearest 0,1 mg.

A.3.4 Add a portion of a suitable emulsifier solution from a 10 ml syringe. If the product under test is gasoline, kerosine or gas oil, the addition of 1 ml of sodium dioctylsulfosuccinate solution in xylene (4.4.1) per 100 ml of sample, is often sufficient. However, if the product under test is naphtha, the addition of 2 ml of sodium dioctylsulfosuccinate solution in xylene per 100 ml of sample, is often required.

A.3.5 Mix the sample. For a high-shear mixer, record the mixing speed, mixing time and approximate distance between the tip of the shaft and the bottom of the container; 15 000 r/min, 60 s and 20 mm, respectively, are often suitable. For a circulating mixer, record the circulation time and rate and the approximate position of suction and discharge ports. A time of 15 min with a flow rate that circulates the entire contents at least once per minute and the suction port as near the bottom of the container as possible, are often suitable.

A.3.6 After mixing, withdraw two portions of the sample and test in accordance with the procedure specified in clause 9. The time after mixing that the second portion is injected into the titration cell defines the verified maximum period of stability. If there is good agreement, within the repeatability of the method, between the water contents determined and the total quantity of water known to be present (added water plus that found in the blank), record the mixing conditions as adequate.

If the results do not show good agreement, within the repeatability of the method, then discard them. Revert to the beginning of this procedure, and use more severe mixing conditions.

Annex B
(informative)

Alternative test method using volumetric determination of sample size

B.1 Introduction

This alternative test method is used for the determination of water in distillate petroleum products as described in this International Standard, with the exception that a volumetric measure is used for the test portion of the sample injected into the titration vessel.

NOTE The use of this alternative procedure may result in a reduction in precision.

B.2 Apparatus

B.2.1 Syringes, of glass, capacity of 1 ml or 2 ml, accurate to the nearest 0,01 ml (10 µl), with needles of suitable length such that the tip can reach under the surface of the anolyte when inserted through the inlet-port septum. The bores of the needles used shall be kept as small as possible, but large enough to avoid problems arising from back pressure or blocking whilst sampling.

NOTE Needles with bores between 0,5 mm and 0,8 mm have been found suitable.

B.3 Procedure

B.3.1 The procedure described in this International Standard shall be followed except that the test portion is measured by volume rather than by mass and the additional precautionary steps given in B 3.4 are followed.

B.3.2 Due to the known reaction of acetone and other ketones with Karl Fischer reagent, the use of such solvents to dry apparatus, sampling syringes, homogenizers and sample receivers is not permitted.

B.3.3 Prepare the sample in accordance with 6.2.

B.3.4 Using a clean, dry syringe (B.2.1), withdraw at least three portions of the sample and discard to waste. Immediately withdraw a test portion of sample, clean the needle with a paper tissue, check for the presence of gas bubbles and record the volume in the syringe to the nearest 10 µl. If gas bubbles are detected, discharge the syringe and refill it.

B.3.5 Insert the needle through the inlet-port septum, start the titration and, with the tip of the needle just below the liquid surface, inject the sample. After the end-point is reached, record the water titrated from the readout displayed on the instrument.

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B.4 Calculation

Calculate the volume fraction of water, φ, expressed as a percentage, using the following equation:

$$\varphi = \frac{100V_2}{V_1 \times 10^6}$$
$$= \frac{V_2}{V_1 \times 10^4}$$

where

V_1 is the volume of the test portion, expressed in millilitres (ml);

V_2 is the volume of water titrated, expressed in nanolitres (equivalent to the mass of water displayed by the titrator in microgrammes, assuming that 1 nanolitre of water weighs 1 μg).

B.5 Expression of results

Report the volume fraction of water in the sample to the nearest 0,001 % (*V/V*).

B.6 Test report

Add the suffix "Annex B" to the method designation and report in accordance with clause 13.

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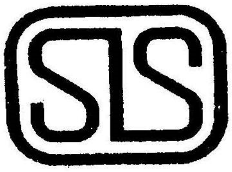

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