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Water quality — Determination of hydrocarbon oil index —

Part 2: Method using solvent extraction and gas chromatography

Qualité de l'eau — Détermination de l'indice hydrocarbure —

Partie 2: Méthode par extraction au solvant et chromatographie en phase gazeuse

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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 part of ISO 9377 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 9377-2 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

ISO 9377 consists of the following parts, under the general title *Water quality — Determination of hydrocarbon oil index*:

— *Part 1: Method using solvent extraction and gravimetry*

— *Part 2: Method using solvent extraction and gas chromatography*

Annexes A, B and C of this part of ISO 9377 are for information only.

Water quality — Determination of hydrocarbon oil index —

Part 2:
Method using solvent extraction and gas chromatography

1 Scope

This part of ISO 9377 specifies a method for the determination of the hydrocarbon oil index in waters by means of gas chromatography. The method is suitable for surface water, waste water and water from sewage treatment plants and allows the determination of a hydrocarbon oil index in concentrations above 0,1 mg/l.

The method is not applicable to the quantitative determination of the content of volatile mineral oil. However, on the basis of the peak pattern of the gas chromatogram, certain qualitative information on the composition of the mineral oil contamination can be derived.

NOTE 1 For the determination of the mineral-oil content of soils and sediment, see ISO/TR 11046.

NOTE 2 The mass concentration of animal and vegetable fat in the test sample should not exceed 150 mg/l, because at higher values the adsorption capacity of the clean-up column packing may not be sufficient.

NOTE 3 In the case of highly polluted waste water, especially if containing a high amount of surfactants, a loss in recovery may occur.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 9377. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 9377 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 5667-3:1994, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*

ISO 8466-1:1990, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function.*

3 Term and definition

For the purposes of this part of ISO 9377, the following term and definition applies.

3.1
hydrocarbon oil index by GC-FID
the sum of concentrations of compounds extractable with a hydrocarbon solvent, boiling point between 36 °C and 69 °C, not adsorbed on Florisil[1)] and which may be chromatographed with retention times between those of *n*-decane ($C_{10}H_{22}$) and *n*-tetracontane ($C_{40}H_{82}$)

NOTE Substances complying with this definition are long-chain or branched aliphatic, alicyclic, aromatic or alkyl-substituted aromatic hydrocarbons.

4 Interferences

Compounds of low polarity (e.g. halogenated hydrocarbons) and high concentrations of polar substances can interfere with the determination.

Surface-active substances interfere with the extraction step.

5 Principle

The water sample is extracted with an extracting agent. Polar substances are removed by clean-up on Florisil. The purified aliquot is analysed by capillary chromatography using a non-polar column and a flame ionization detector (FID). The total peak area between *n*-decane and *n*-tetracontane is measured. The concentration of mineral oil is quantified against an external standard consisting of two specified mineral oils, and the hydrocarbon oil index is calculated.

It is absolutely essential that the test described in this part of ISO 9377 be carried out by suitably qualified staff.

It should be investigated whether and to what extent particular problems will require the specification of additional marginal conditions.

6 Reagents

All reagents shall be reagent grade and suitable for their specific purpose. The suitability of the reagents and solutions shall be checked by carrying out a blank test.

6.1 Water for the preparation of solutions.

Distilled water, or water from a generator of purified water capable of removing organic traces, for example using activated carbon, shall be used.

6.2 Extracting agent.

Single hydrocarbon solvent or technical mixture of hydrocarbons, boiling range 36 °C to 69 °C.

In case of a change of extracting agent, repeatability tests are necessary.

6.3 Sodium sulfate, anhydrous, Na_2SO_4.

6.4 Magnesium sulfate heptahydrate, $MgSO_4 \cdot 7H_2O$.

1) Florisil is a trade name for a prepared diatomaceous substance, mainly consisting of anhydrous magnesium silicate. This information is given for the convenience of users of this part of ISO 9377 and does not constitute an endorsement by ISO of this product.

6.5 Mineral acid, e.g. hydrochloric acid , c(HCl) = 12 mol/l (ρ = 1,19 g/ml).

6.6 Acetone, C_3H_6O.

6.7 Florisil[1)], grain size 150 μm to 250 μm (60 mesh to 100 mesh), heated to 140 °C for 16 h and stored in a desiccator.

6.8 Mixture of mineral oils.

6.8.1 Standard[2)] **mixture.**

Weigh accurately equal amounts of two different types (type A and type B, both containing no additives) of mineral oil and add enough extraction solvent (6.11.2) to give a total hydrocarbon concentration of about 10 mg/ml.

Type A should show discrete peaks in the gas chromatogram. An example is diesel fuel without any additives. See EN 590 for further information. Type B should have a boiling range higher than that of type A and should have unresolved signals in the gas chromatogram. An example of this type is a lubricant without any additives, boiling range 325 °C to 460 °C.

6.8.2 Calibration mixture.

Prepare at least five different calibration solutions by diluting aliquots of standard mixture (6.8.1) with the extraction solvent (6.11.2). The following concentrations may be suitable:

0 (blank), 0,2 mg/ml, 0,4 mg/ml, 0,6 mg/ml, 0,8 mg/ml and 1,0 mg/ml.

Higher concentrations may be advisable for other applications.

Store the calibration mixture tightly sealed in a refrigerator (4 °C to 8 °C). The calibration mixtures are stable for up to six months.

6.8.3 Quality Control (QC) standard.

Prepare a standard solution according to 6.8.1 in acetone (see 6.6) with a mass concentration of e.g. 1 mg/ml. The exact concentration should be about a thousand times the desired application range.

NOTE When using a lubricant for QC, the stock solution is easily prepared in extracting agent (6.2), which is further diluted in acetone about tenfold before spiking the QC water.

Store the solution tightly sealed in a refrigerator (4 °C to 8 °C). It is stable for up to six months.

6.9 Standard mixture of *n*-alkanes for system performance test.

Dissolve *n*-alkanes with even carbon numbers (C_{20}, C_{40} and at least three further *n*-alkanes) in extracting agent (6.2) to give concentrations of approximately 50 μg/ml of the individual components. It may be necessary to use a different solvent, e.g. heptane, for the first solution; in this case dilute this first solution with extracting agent (6.2).

Store the standard mixture tightly sealed in a refrigerator. It is stable for up to six months.

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2) Standards are available from

Fachgruppe I.2
Bundesanstalt für Materialforschung und –prüfung
Richard-Willstätter-Strasse 11
D-12489 Berlin, Germany

This information is given for the convenience of users of this part of ISO 9377 and does not constitute an endorsement by ISO of these products.

NOTE 1 This solution is used to verify the suitability of the gas chromatographic system for the resolution of n-alkanes as well as for the detector response.

NOTE 2 This solution is used to give information on the retention times of the n-alkanes to characterize the hydrocarbons in the sample.

6.10 Reference compounds.

6.10.1 *n*-Decane, $C_{10}H_{22}$.

6.10.2 *n*-Tetracontane, $C_{40}H_{82}$.

6.10.3 *n*-Eicosane, $C_{20}H_{42}$.

6.11 Extraction solvent containing reference compounds.

6.11.1 Extraction solvent stock solution.

Dissolve 20 mg of *n*-tetracontane (6.10.2) in extracting agent (6.2). Then add 20 µl of *n*-decane (6.10.1) and dilute with extracting agent to 1 000 ml.

Store the solution tightly sealed in a refrigerator. It is stable for up to six months.

NOTE *n*-Tetracontane is only moderately soluble in the extracting agent. Slight warming or treatment with ultrasonics accelerates the dissolution process.

6.11.2 Extraction solvent standard solution.

Immediately prior to use, dilute the stock solution (see 6.11.1) with tenfold extracting agent.

6.12 Stearyl stearate test solution, $C_{36}H_{72}O_2$.

Dissolve 200 mg of stearyl stearate in 100 ml of extraction solvent standard solution (6.11.2).

NOTE This solution is used to check the efficiency of the clean-up procedure.

Store the solution tightly sealed in a refrigerator. It is stable for up to six months.

7 Apparatus

7.1 Usual laboratory glassware

Clean all glassware by the usual procedures for this type of analysis and check for purity (measurement of blank). If necessary, rinse the glassware with extracting agent (6.2) and re-check the blank.

7.2 Gas chromatograph, equipped with a non-discriminating injection system and a flame ionization detector.

7.3 Column for gas chromatography, fused silica, with one of the following stationary phases:

non-polar, immobilized 100 % dimethylpolysiloxane, or 95 % dimethyl-/5 % diphenylpolysiloxane, or modified siloxane polymer

and typical dimensions:

length:	5 m to 30 m
internal diameter:	0,25 mm to 0,53 mm
film thickness:	0,25 µm to 1,2 µm

For an example, see annex B.

It is recommended to use a precolumn (e.g. 2 m, 0,53 mm i.d., deactivated fused silica).

7.4 Data system, suitable for integrating the total area of the gas chromatogram and suitable for compensating for 'column bleeding' and for re-integrating after drawing a new baseline.

7.5 Sampling bottles, glass, with ground glass stopper, capacity 250 ml and 1 000 ml, or with screw cap, lined with PTFE (polytetrafluoroethene).

The sampling bottle shall allow direct extraction from the bottle.

7.6 Centrifuge.

7.7 Centrifuge tubes, of capacity 100 ml, with suitable (screw)cap.

7.8 Microseparator, for example see Figure A.2, or other suitable device for phase separation.

7.9 Clean-up columns, made from glass, with frit of sinter porosity 2, for example see Figure A.1.

7.10 Kuderna Danish apparatus, with a 250 ml flask or other suitable concentration apparatus, e.g. a rotary evaporator with controlled vacuum.

7.11 Magnetic stirrer with bar, of length suitable to ensure thorough mixing.

8 Sampling and sample preservation

Sampling and storage shall be in accordance with ISO 5667-3.

Fill the sampling bottle (7.5) to approximately 90 % with the sample, seal tightly and weigh (m_1). Keep the sample at about 4 °C and extract the sample as soon as possible, but in any case within four days.

If necessary, preserve the sample in the field by acidifying with mineral acid (6.5) to pH 2.

Preservation of the sample in the field is recommended for surface and ground water samples. If a high content of humic substances is present in ground waters, indicated by a yellowish-brownish colour of the sample, acidifying of the sample should be avoided to prevent the precipitation of humic acids at the specified pH.

NOTE If formation of emulsions or a concentration of animal and vegetable oils > 150 mg/l is expected, it is advisable to withdraw additionally a smaller sample volume in a 250 ml sampling bottle.

9 Procedure

9.1 Blank test

Carry out blank tests with each series of tests in accordance with 9.3, including all reagents and glassware in the same way as the samples.

9.2 Determination of the recovery

Determine the recovery at regular intervals, preferably in each series of tests, using 900 ml of water (6.1) to which 1,0 ml of the QC standard (6.8.3) has been added. Perform the test starting with 9.3 and calculate the recovery. Ensure that the recovery is between 80 % and 110 %.

9.3 Extraction procedure

Cool the sample to about 10 °C, if necessary, to prevent losses of the extracting agent by volatilization.

Acidify the sample to pH 2 by adding mineral acid (6.5), if this has not been done in the field (see clause 8).

Add about 80 g of magnesium sulfate (6.4) per 900 ml of sample to avoid emulsions.

NOTE 1 The addition of magnesium sulfate is not necessary if samples are known not to form emulsions.

Add 50 ml of extraction solvent standard solution (6.11.2) and a magnetic stirrer bar, close the bottle and stir vigorously for 30 min on the magnetic stirrer (7.11).

Remove the stopper and replace it by the microseparator (7.8).

Add enough water (6.1) to allow withdrawal of the extracting-agent layer from the microseparator, transfer it to the clean-up column (7.9) and proceed according to 9.4.

When transferring the organic phase to the clean-up column, take care to avoid the transfer of water as this will incrust the surface of the sodium sulfate. It is recommended to transfer the organic layer in several steps using a pipette, or, when using the microseparator (7.8) (Figure A.2), to position the meniscus below the cock.

In the case of strong emulsions, centrifuge the extract as follows. Transfer the extracting-agent phase together with the emulsion into a 100 ml centrifuge tube (7.7) and close the tube. Break the emulsion by centrifuging the extract.

NOTE 2 Centrifugation for 10 min to 15 min is usually sufficient.

Extraction by shaking may be adequate, but verify the efficiency.

9.4 Clean-up procedure

Transfer the extracting-agent phase (see 9.3) to a small column (see Figure A.1) filled with 2 g of Florisil (6.7) and covered with a layer of 2 g of sodium sulfate (6.3).

NOTE 1 Pre-rinsing of the column with a few millilitres of extracting agent (6.2) can be useful to prevent the formation of channels.

Let the extracting-agent phase, followed by an additional 10 ml of extracting agent (6.2), run through the column into a suitable concentration apparatus (7.10).

Rinse the column with about 10 ml of extracting agent (6.2).

NOTE 2 Other procedures using the same amount of Florisil for cleaning the extract, e.g. shaking the total extract with 2 g of Florisil during 30 min on a shaking apparatus, may be used as an alternative, provided the results are equivalent to the column testing of Florisil.

9.5 Concentration

Using the evaporation apparatus (7.10), concentrate the extract to a volume of approximately 6 ml.

Concentrate the extract further to slightly less than 1 ml using a gentle flow of nitrogen. Make up to a volume of 1,0 ml with extracting agent (6.2) or calculate the exact volume of the concentrated extract by weighing. Transfer an aliquot of the final extract to a septum vial for gas chromatographic analysis.

NOTE Concentration of the extract to 1,0 ml may be omitted if a high hydrocarbon oil index is expected or if a large quantity, e.g. 100 µl, of the partially or non-concentrated extract is injected by means of the so-called "large-volume injection system".

When using a large-volume injection, it is necessary to bring the extract to a known volume, e.g. 50 ml or 100 ml after treatment with Florisil. In this case the concentration of the calibration solution (6.8) and the calibration mixture of *n*-alkanes (6.9) should be correspondingly lower.

Leave the empty sample bottle to drain for 5 min. Close the bottle with the cap used previously and determine its mass (m_2) to an accuracy of 1 g.

9.6 Suitability testing of Florisil

Check the suitability of Florisil at regular intervals and each time a new batch of dried Florisil is used, as follows:

For this purpose, use a test solution of stearyl stearate (6.12) and a calibration solution of mineral oils (6.8).

Perform the clean-up procedure (9.4) with 10 ml of the stearyl stearate solution, then add extracting agent (6.11.2) to a volume of 25 ml. Transfer an aliquot of the purified solution to a septum vial and analyse by gas chromatography (9.7). Measure the peak areas of stearyl stearate after Florisil treatment. Dilute 0,5 ml of stearyl stearate solution with extracting agent (6.11.2) to 25 ml and analyse by gas chromatography. Calculate the ratio of the peak areas for stearyl stearate in the treated and in the untreated solutions. This ratio should be less than 1. If not, activate the Florisil according to 6.7.

Perform the clean-up procedure (9.4) with 10 ml of 2 mg/ml calibration solution of mineral oils (6.8), then add extraction solvent standard solution (6.11.2) to a volume of 25 ml. Transfer an aliquot of the purified solution into a septum vial and analyse by gas chromatography.

Determine this mineral oil recovery on the basis of the peak area between C_{10} to C_{40} in the treated (with Florisil) and untreated calibration solutions. It should be at least 80 %. If this criterion is not met, wash out the Florisil batch with ample water and activate the Florisil as given in 6.7. If the repeated test shows again that the criterion is not met, try another batch of Florisil with a different batch number.

9.7 Determination by gas chromatography

9.7.1 Adjusting the gas chromatograph

Select a capillary column with one of the stationary phases specified (see 7.3 and annex B) for gas chromatographic analysis. Adjust the gas chromatograph until optimum separation is obtained. The peaks in the gas chromatogram of the standard mixture of *n*-alkanes (6.9) shall be baseline-separated. The relative response (peak area) of *n*-tetracontane ($C_{40}H_{82}$) compared with *n*-eicosane ($C_{20}H_{42}$) should be at least 0,8. If not, the discrimination of the injection system is too high and the injection system shall be optimized or replaced.

9.7.2 Calibration

9.7.2.1 General

For calibration a distinction is made between initial calibration, working calibration and checking of the validity of the calibration curve. Initial calibration determines the working range and the linearity of the calibration function according to ISO 8466-1. Perform this calibration when the apparatus is used for the first time.

In the next step establish the final working range and perform the routine calibration. Repeat this calibration after maintenance (e.g. replacement of the capillary column), after repair of the gas chromatographic systems, and in case the system has not been in use for a longer period of time, or if the validity criteria cannot be met. Check the validity of the initial calibration with each series of samples to be analysed.

Correct all chromatograms for column bleeding. For this purpose run blank chromatograms (chromatograms with solvent only) and use these for baseline correction.

9.7.2.2 Initial calibration

Establish the preliminary working range by analysing at least five dilutions of the calibration standard mixture (6.8). Test for linearity in accordance with ISO 8466-1.

9.7.2.3 Routine calibration

After examining the final working range, analyse a minimum of five dilutions of the standard calibration mixture (6.8). Calculate a calibration function by linear regression analysis of the corrected peak areas. The actual sensitivity of the method may be estimated from the calculated regression function.

9.7.2.4 Check of the validity of the calibration function

Check the validity of the calibration function from the routine calibration with each batch of samples by analysis of one standard solution after every ten samples. The concentration of this standard solution shall lie between 40 % and 80 % of the working range. Make sure that the individual results do not deviate by more than 10 % of the working calibration line. **If this criterion is met, assume the calibration to be valid.** If not, recalibrate in accordance with 9.7.2.3.

For large batches of samples the number of analyses of the standard solution may be reduced, provided that at least three measurements are obtained for calculating the mean result.

9.7.3 Measurement

Measure the sample, the calibration solutions and the blank solution in the gas chromatograph.

At regular intervals record a gas chromatogram (blank chromatogram) by injecting extracting agent (6.2) and analyse under the same conditions as for the sample. Use a chromatogram of extracting agent to correct the areas of the chromatograms of the samples.

NOTE An increase in “column bleeding” may indicate contamination of the injection system or the column.

9.7.4 Integration parameters

Integrate the gas chromatogram between *n*-decane ($C_{10}H_{22}$) and *n*-tetracontane ($C_{40}H_{82}$). Start the integration just after the *n*-decane peak at the signal level in front of the solvent peak (S in Figure B.3). End the integration just before the beginning of the *n*-tetracontane peak on the same signal level (E in Figure B.3). Check all chromatograms visually to ensure correct integration. Draw a straight line from S to E. Mark the beginning and end of the integration on the chromatogram.

NOTE 1 For examples of chromatograms, see annex B.

The presence of peaks between solvent peak and *n*-decane indicates that the sample probably contains low-boiling, volatile hydrocarbons. This should be mentioned in the test report.

Discrete peaks or an increased level of the baseline at the end of the chromatogram (retention time greater than that of *n*-tetracontane) indicate that the sample probably contains hydrocarbons with a high boiling point. This should be mentioned in the test report.

NOTE 2 The range of the carbon numbers of *n*-alkanes present in the sample is determined by comparing the gas chromatogram of the sample extract with that of *n*-alkane standard solution (6.9). The corresponding boiling range can be derived from annex C.

9.8 Calculation

Calculate the hydrocarbon oil index using the equation:

$$\rho = \frac{(A_m - b) \cdot f \cdot V \cdot w}{a \cdot (m_1 - m_2)} \quad (1)$$

where

ρ is the hydrocarbon oil index, in milligrams per litre;

a is the slope of calibration function, in litres per milligram;

A_m is the integrated peak area of the sample extract, in instrument-dependent units;

f is any dilution factor of the sample extract;

m_1 is the mass of the filled sampling bottle, in grams;

m_2 is the mass of the empty sample bottle, in grams;

w is the density of the water sample, in grams per millilitre (for fresh water 1,00 g/ml may be used);

V is the volume of the final extract, in millilitres;

b is the intercept of the y-axis in instrument-dependent units.

9.9 Expression of results

Express the concentration of mineral oil in water as hydrocarbon oil index, in milligrams per litre, to two significant figures.

EXAMPLES

Hydrocarbon oil index	15 mg/l
Hydrocarbon oil index	2,9 mg/l

10 Test report

The test report shall refer to this part of ISO 9377 and contain the following details:

a) identity of the sample;

b) the hydrocarbon oil index, in milligrams per litre;

c) any peculiarities observed during the test;

d) any actions not specified in this part of ISO 9377 which may have influenced the result.

In addition, the following qualitative information can be provided in the test report:

e) the boiling range of the mineral oil detected on the basis of the relative retention time versus the boiling point of the calibration mixture of *n*-alkanes;

f) any presence of volatile hydrocarbons;

g) any presence of hydrocarbons with a high boiling point.

11 Precision

An interlaboratory trial, carried out in summer 1999, resulted in the values given in Table 1.

Table 1 — Precision data

Sample No.	L	n	NAP %	$\overline{\overline{x}}$ mg/l	x_{soll} mg/l	WFR %	s_R mg/l	CV_R %	s_r mg/l	CV_r %
1	35	127	15,3	3,04	2,99	101,6 [a]	0,291	9,6	0,092	3,0
2	35	134	13,6	0,57	0,70	82,0	0,192	33,5	0,037	6,5
3	38	142	10,1	3,61	4,00	90,3 [b]	0,763	21,1	0,210	5,8
4	41	156	0,6	0,74	1,04	71,1 [c]	0,300	40,5	0,105	14,1

L is the number of laboratories after elimination of outliers;

n is the number of results after elimination of outliers;

NAP are the outliers;

$\overline{\overline{x}}$ is the total mean of all results free from outliers;

x_{soll} is the true value;

WFR is the recovery rate;

s_R is the reproducibility standard deviation;

CV_R is the reproducibility coefficient of variation;

s_r is the repeatability standard deviation;

CV_r is the repeatability coefficient of variation.

[a] Two participants in the interlaboratory trial had problems with the quality of petroleum ether.

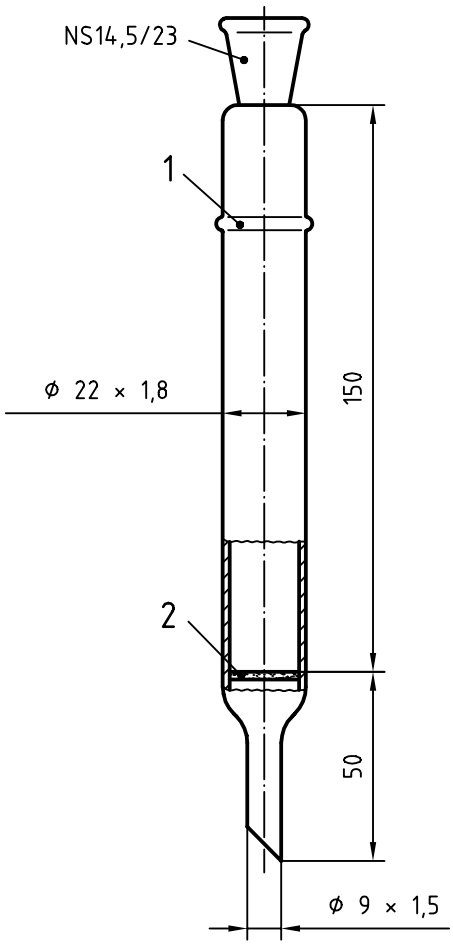
[b] The sample contained surfactants in a concentration exceeding the hydrocarbons by tenfold.

[c] The sample contained a high concentration of C_{12} methyl ester.

Annex A
(informative)

Example of a column and a microseparator

Dimensions in millimetres

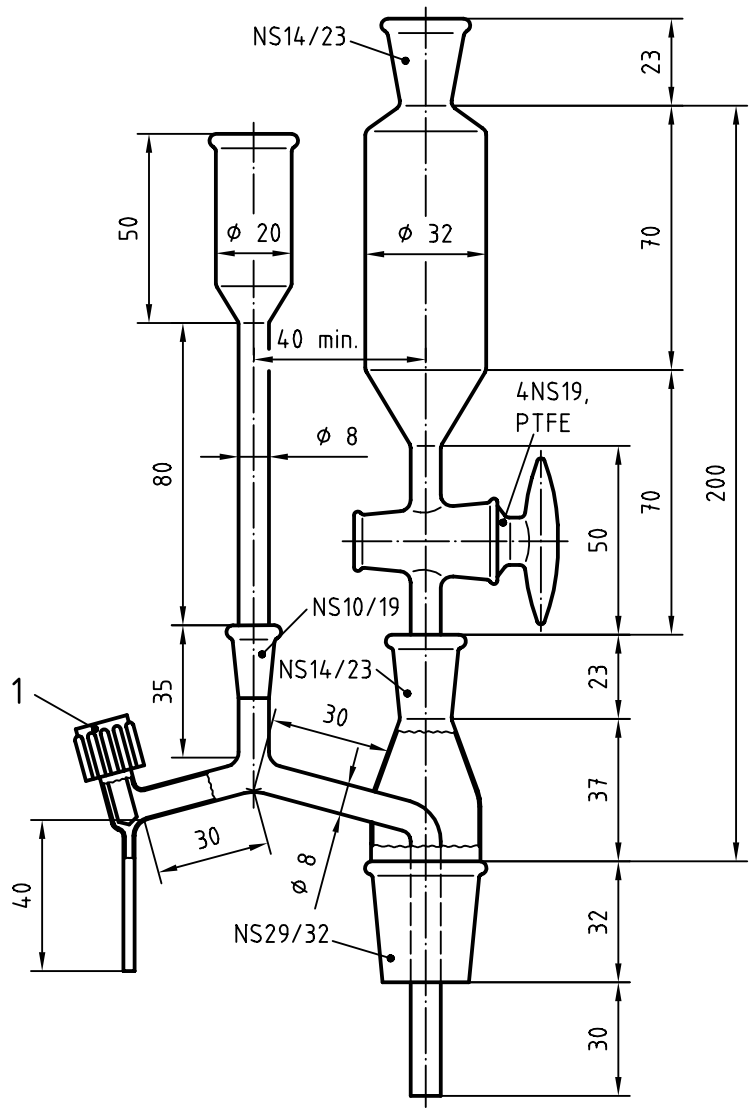


Key

1 Glass bulb
2 Fritted glass filter

Figure A.1 — Example of a column

Dimensions in millimetres



Key

1 Spindle stopcock, PTFE

Figure A.2 — Example of a microseparator

Annex B
(informative)

Examples of gas chromatograms of mineral oil standard and water samples

The gas chromatograms have been recorded under the following conditions:

Injection technique:	programmed temperature vaporization (PTV)
Injection temperature:	50 °C to 300 °C
Injection volume:	1 µl
Column length:	30 m
Column internal diameter:	0,25 mm
Liquid phase:	DB 5 MS
Film thickness:	0,25 µm
Pre-column:	deactivated fused silica capillary
Carrier gas:	hydrogen
Carrier gas pressure:	0,8 bar
Oven temperature programme:	40 °C for 5 min, 10 °C/min to 300 °C, 300 °C for 20 min
Detector:	flame ionization detector
Detector temperature:	300 °C
Make-up gas:	nitrogen
Make-up gas flow:	25 ml

Key

1 *n*-Decane

2 *n*-Tetracontane

Figure B.1 — Gas chromatogram of a standard solution (0,5 mg/l)

Key

1 *n*-Decane

2 *n*-Tetracontane

Figure B.2 — Gas chromatogram of a surface water contaminated with fuel oil

Key

1 *n*-Decane

2 *n*-Tetracontane

Figure B.3 — Gas chromatogram of a waste water

Key

1 *n*-Decane

2 *n*-Tetracontane

Figure B.4 — Gas chromatogram of a waste water contaminated with lubricating oil

Annex C
(informative)

Determination of boiling range of a mineral oil from the gas chromatogram

Table C.1 shows the boiling points of the *n*-alkanes containing between 2 and 44 carbon atoms (up to and including C_{44}). Using these boiling points, it is possible to determine the boiling range of the mineral oil by comparing the gas chromatogram of the calibration mixture of *n*-alkanes with that of the sample extract.

Table C.1 — Boiling points of *n*-alkanes

Number of carbon atoms	Boiling point °C	Number of carbon atoms	Boiling point °C
2	–89	23	380
3	–42	24	391
4	0	25	402
5	36	26	412
6	69	27	422
7	98	28	432
8	126	29	441
9	151	30	450
10	174	31	459
11	196	32	468
12	216	33	476
13	235	34	483
14	253	35	491
15	271	36	498
16	287	37	505
17	302	38	512
18	317	39	518
19	331	40	525
20	344	41	531
21	356	42	537
22	369	43	543
		44	548

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[2] KVERNHEIM A.L. *et al*., Development of a new hydrocarbon index for oil-in-water. *Chemosphere*, **39**, 1999, pp. 2707-2722.

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