BS ISO 11349:2010

BSI Standards Publication

Water quality — Determination of low-volatility lipophilic substances — Gravimetric method

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National foreword

This British Standard is the UK implementation of ISO 11349:2010.

The UK participation in its preparation was entrusted to Technical Committee EH/3/2, Physical chemical and biochemical methods.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Water quality — Determination of low-volatility lipophilic substances — Gravimetric method

Qualité de l'eau — Dosage des substances lipophiles peu volatiles — Méthode gravimétrique

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Foreword

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ISO 11349 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

INTERNATIONAL STANDARD ISO 11349:2010(E)

Water quality — Determination of low-volatility lipophilic substances — Gravimetric method

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this International Standard be carried out by suitably trained staff.

1 Scope

This International Standard defines a method for the determination of lipophilic substances of low volatility in water using gravimetry.

The method is applicable to all kinds of water and allows the determination of low-volatility lipophilic substances which are suspended, emulsified, or dissolved, in concentrations of about 10 mg/l to 500 mg/l. Above this value, the test portion is diluted appropriately.

The method is not applicable to water with a separate oil layer.

2 Normative references

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

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
low-volatility lipophilic substances
sum of substances extractable by non-polar hydrocarbons, determined gravimetrically after drying at 80 °C

NOTE Substances covered by this definition are unpolar or weakly polar, with a boiling point above 250 °C, and include mainly animal oils, vegetable oils, fats, greases, mineral oils, waxes, and non-ionic surfactants.

4 Principle

A test portion of water is extracted with an extracting agent. The extracting agent is evaporated. The mass of low-volatility lipophilic substances is determined by gravimetry.

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5 Interferences

The formation of stable emulsions caused by surface active substances may lead to the inclusion of extracting agents in the emulsion and thus to losses.

6 Reagents

All reagents shall be suitable for the purpose of this method and shall not significantly influence the blank (see 9.1).

6.1 **Water**, as specified in ISO 3696, grade 3, distilled water or deionized water.

6.2 **Extracting agent**. Hydrocarbon or technical mixture of hydrocarbons, boiling range 36 °C to 69 °C (e.g. petroleum ether 40 °C to 60 °C, *n*-hexane).

6.3 **Sodium sulfate**, Na_2SO_4, anhydrous, coarse grained.

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

6.5 **Mineral acid**, e.g. sulfuric acid, $c(H_2SO_4)$ = 2 mol/l (ρ = 1,12 g/ml).

6.6 **Vegetable oil**, as test substance for the determination of the recovery.

NOTE Olive oil has proved to be most suitable. Sunflower oil can be suitable as well.

6.7 **Acetone**, C_3H_6O.

7 Apparatus

Clean all glass apparatus according to normal cleaning procedures and check the purity by blank determination. If necessary, rinse the apparatus with extracting agent (6.2).

Usual laboratory equipment and in particular the following.

7.1 **Sampling vessel**, glass, with glass stopper or polytetrafluoroethene-lined screw cap, e.g. 1 000 ml.

7.2 **Homogenization device**, e.g. Ultraturrax[1)].

7.3 **Extracting vessel**, 1 000 ml.

7.4 **Shaking device** or **magnetic stirrer**.

7.5 **Separating funnel**, e.g. 500 ml.

NOTE For phase separation another suitable device can also be used, e.g. a micro-separator.

7.6 **Erlenmeyer flask** or **round-bottomed flask**, 250 ml.

7.7 **Glass filter funnel with hydrophobic filter**.

7.8 **Measuring cylinders**, glass, capacities 100 ml, 250 ml, and 500 ml, ISO 4788[1].

1) Ultraturrax® is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

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7.9 Suitable concentration device, e.g. Rotavapor[2)].

7.10 Drying oven.

7.11 Desiccator, with a diameter of, for example, 100 mm, 200 mm or 300 mm.

7.12 Balance, capable of being read to at least 0,1 mg.

8 Sampling and sample conservation

Fill the sampling vessel (7.1) to about 80 % by volume with the sample and close tight. If analysis is not carried out the same day, add mineral acid (6.5) to adjust the pH to $\leqslant 2$. Store at about 4 °C and analyse within 7 d.

9 Procedure

9.1 Blank determination

Carry out blank determinations regularly including all reagents and apparatus, in the same way as described for the samples, but replacing the test portion by 500 ml of water (6.1). The blank shall not exceed 3 mg/l.

9.2 Recovery check

Prior to determination of the low-volatility lipophilic substances, check the method by extraction of a test substance as follows.

Transfer 500 ml of water (6.1) to the extracting vessel (7.3) and add about 100 mg of vegetable oil (6.6), precisely weighed and prediluted with about 1 ml of acetone (6.7), and continue the procedure according to this method.

The recovery shall be between 90 % and 105 %.

9.3 Extraction

Acidify the water sample to a pH of $\leqslant 2$ with mineral acid (6.5), if this step has not already been carried out according to Clause 8.

After intensive homogenization (7.2), a test portion of volume, V, between 100 ml and 500 ml is transferred to an extracting vessel (7.3). If necessary, dilute this test portion with water (6.1) up to a final volume of approximately 500 ml.

Add 50 ml of extracting agent (6.2). Shake several times by hand, allow for pressure adjustment by carefully opening the extracting vessel under a hood. Stopper the vessel and shake intensively for 15 min or intensively stir using a magnetic stirrer (7.4). Make sure that the stirring cone reaches the bottom of the vessel.

For phase separation, allow to stand for 20 min in a separating funnel (7.5), then remove the aqueous phase.

Any emulsion formed may be broken by adding in portions up to 20 g of magnesium sulfate heptahydrate (6.4) and/or sodium sulfate (6.3). Shake after each addition of salt and allow for overpressure release. Wait for phase separation and add any remaining emulsion to the water phase.

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2) Rotavapor is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

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Repeat the extraction step with a further 50 ml of extracting agent (6.2) and combine the organic phases.

The volume of the recovered organic phases shall be at least 75 % of the volume of the added extracting agents. If not, repeat the determination with a test portion of smaller volume or with a larger volume of extracting agent.

Dry the organic phase with 10 g of sodium sulfate (6.3).

Filter the extract using a hydrophobic filter (7.7) into a flask (7.6) whose mass, m_1, has previously been recorded. Rinse the vessel, filter, and sodium sulfate two or three times with about 10 ml of extracting agent (6.2) and add the rinsings to the extract.

9.4 Concentration step

Concentrate the extract to about 2 ml using the concentration device (7.9).

Carefully strip the remaining extracting agent either by blowing nitrogen over it in an exhaust hood or by further treatment in the concentration device.

Continue the drying step in an oven (7.10) for 15 min at (80 ± 3) °C.

Allow the flask and its contents to cool in a desiccator (7.11) and weigh. Record the mass as m_2.

Ensure that no crystalline sodium sulfate is visible in the flask.

If the weighed mass is above 250 mg, repeat the analysis procedure from 9.3 onwards with a smaller test portion, diluted as appropriate.

10 Calculation

Calculate the mass concentration of low-volatility lipophilic substances according to Equation (1):

$$\rho = \frac{m_2 - m_1}{V} \tag{1}$$

where

ρ is the mass concentration, in milligrams per litre, of low-volatility lipophilic substances;

m_2 is the mass, in milligrams, of the flask with contents (9.4);

m_1 is the mass, in milligrams, of the empty flask (9.3);

V is the volume, in litres, of the test portion (9.3).

11 Expression of results

Report the mass concentration, in milligrams per litre, of low-volatility lipophilic substances to two significant figures.

EXAMPLE Low-volatility lipophilic substances: 15 mg/l.

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12 Test report

The test report shall contain at least the following information:

a) the test method used, together with a reference to this International Standard (ISO 11349:2010);

b) all the information required for the complete identification of the sample;

c) details of sampling, sample transportation, and sample preparation;

d) details of any sample pretreatment;

e) test result, according to Clause 11;

f) all operation details not specified in this International Standard, or regarded as optional, together with details of any incident that may have influenced the result(s).

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Annex A
(informative)

Precision data

The results from an interlaboratory trial, carried out in Germany in spring 2007 are given in Table A.1.

Table A.1 — Precision data calculated in accordance with ISO 5725-2[2]

Parameter	Sample	
	1	2
	Matrix	
	Industrial waste water I	Industrial waste water II
No. laboratories after elimination of outliers, p	16	13
No. results after elimination of outliers, n	34	28
Percentage of outliers, n_{OP}, %	5,6	12,5
Total mean of all analytical results (after elimination of outliers), $\bar{\bar{\rho}}$, mg/l	14,3	200,4
Reproducibility standard deviation, s_R, mg/l	4,88	19,93
Coefficient of variation of reproducibility, $C_{V,R}$, %	34,2	9,9
Repeatability standard deviation, s_r, mg/l	2,03	10,62
Coefficient of variation of repeatability, $C_{V,r}$, %	14,2	5,3

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Bibliography

[1] ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

[2] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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