



SLOVENSKI STANDARD

SIST ISO 11349:2011

01-junij-2011

Kakovost vode - Določevanje težko hlapnih lipofilnih snovi - Gravimetrijska metoda

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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ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances
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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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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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

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

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

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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.

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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, Na₂SO₄, anhydrous, coarse grained.

6.4 Magnesium sulfate heptahydrate, MgSO₄·7H₂O, coarse grained.

6.5 Mineral acid, e.g. sulfuric acid, $c(\text{H}_2\text{SO}_4) = 2 \text{ mol/l}$ ($\rho = 1,12 \text{ g/ml}$).

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, C₃H₆O.

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. Ultraturrax¹⁾.

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.

7.9 Suitable concentration device, e.g. Rotavapor²⁾.

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 ≤ 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 ≤ 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.

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.