SLOVENSKI STANDARD
SIST ISO 8165-2:2010

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Kakovost vode - Določevanje izbranih enovalentnih fenolov - 2. del: Metoda s plinsko kromatografijo po derivatizaciji

Water quality - Determination of selected monovalent phenols - Part 2: Method by derivatization and gas chromatography

Qualité de l'eau - Dosage des phénols monovalents sélectionnés - Partie 2: Méthode par dérivatisation et chromatographie en phase gazeuse

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INTERNATIONAL STANDARD

ISO 8165-2

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Water quality — Determination of selected monovalent phenols —

Part 2:
Method by derivatization and gas chromatography

Qualité de l'eau — Dosage des phénols monovalents sélectionnés —

Partie 2: Méthode par dérivatisation 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.

International Standard ISO 8165-2 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

ISO 8165 consists of the following parts, under the general title *Water quality — Determination of selected monovalent phenols*:

— *Part 1: Gas-chromatographic method after enrichment by extraction*

— *Part 2: Method by derivatization and gas chromatography*

Annex A of this part of ISO 8165 is for information only.

Water quality — Determination of selected monovalent phenols —

Part 2:
Method by derivatization and gas chromatography

1 Scope

This part of ISO 8165 specifies a method for the determination of phenols by gas chromatography, following pentafluorobenzoyl chloride (PFBC) derivatization. It may in particular be applied to the examination of drinking water, ground water and moderately contaminated surface water. With this method, lower limits of detection may be obtained compared with extraction procedures.

Since other reactive compounds such as amines and in some cases alcohols may also react, this method is not applicable in all cases to the examination of waste water. The applicability to the examination of waste water should be investigated for each individual case.

This method allows the determination of the phenols listed in Table 1 in a concentration range ⩾ 0,1 µg/l. Other monovalent phenols may also be analysed using this method, but the applicability needs to be checked for each individual case.

Table 1 — Phenols to which this method is applicable

phenol	2-cyclopentyl-4-chlorophenol
2-methylphenol	4-chloro-2-benzylphenol
3-methylphenol	6-chloro-5-methyl-2-(1-methylethyl)phenol
4-methylphenol	2,3-dichlorophenol
2,4-dimethylphenol	2,4-dichlorophenol
4-ethylphenol	2,5-dichlorophenol
2,6-bis(1,1-dimethylethyl)-4-methylphenol	2,6-dichlorophenol
2-phenylphenol	2,4,6-trichlorophenol
2-benzylphenol	2,3,5-trichlorophenol
2-benzyl-4-methylphenol	2,4,5-trichlorophenol
2-chlorophenol	2,3,6-trichlorophenol
3-chlorophenol	2,3,4,5-tetrachlorophenol
4-chlorophenol	2,3,4,6-tetrachlorophenol
4-chloro-2-methylphenol	2,3,5,6-tetrachlorophenol
4-chloro-3-methylphenol	pentachlorophenol
6-chloro-3-methylphenol	
2,4-dichloro-3,5-dimethylphenol	
2-chloro-4-*t*-butylphenol	

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 8165. 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 8165 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 5667-1:1980, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes.*

ISO 5667-2:1991, *Water quality — Sampling — Part 2: Guidance on sampling techniques.*

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 Principle

The phenols contained in the unfiltered water sample are extractively derivatized by means of hexane and pentafluorobenzoyl chloride. The completion of the extractive derivatization is verified by the addition of the control solution (5.14). The gas chromatographic measurement uses two capillary columns of different polarity (simultaneous splitting) and detection with electron-capture detectors (ECD).

4 Interferences

Surfactants, emulsifiers or higher concentrations of polar solvents may affect the extractive derivatization step.

Suspended particles in the water may also interfere and reduce the recovery. A second liquid phase in the water sample (e.g. mineral oil compounds, highly volatile halogenated hydrocarbons, emulsified fats and waxes) disturbs sampling, sample preparation and the enrichment. In such cases the examination is restricted to the aqueous phase, and the portion of the non-aqueous phase is reported separately.

If problems are encountered in the use of the gas chromatographic system, reference should be made to the user's manual provided by the instrument manufacturer.

It is absolutely essential that the test described in this part of ISO 8165 be carried out by suitably qualified staff.

5 Reagents

5.1 General requirements

The content of monovalent phenols in water and reagents used shall be negligibly low, compared with the expected concentration levels. The water shall be suitable for trace analysis, its blank being determined in accordance with 8.1 and 8.2. If necessary, the water shall be purified by distillation at pH value > 12.

5.2 Sodium hydroxide solution, c(NaOH) = 1 mol/l.

5.3 Sodium sulfite, Na_2SO_3.

5.4 Sodium hydrogencarbonate solution, $c(NaHCO_3)$ = 1 mol/l.

5.5 Sulfuric acid

Carefully add 1 volume of sulfuric acid, $\rho(H_2SO_4)$ = 1,84 g/ml, to three volume portions of water.

5.6 Hexane, C_6H_{14}, highest purity grade.

5.7 Decane, $C_{10}H_{22}$.

5.8 Pentafluorobenzoyl chloride (PFBC), C_7OClF_5 (verify applicability, since batch-to-batch variations are likely to occur).

The purity of the PFBC is checked by running a blank chromatogram after an overall procedure blank sample preparation. If interfering peaks make a calculation impossible, the PFBC batch shall be rejected.

5.9 Sodium sulfate, anhydrous, Na_2SO_4.

5.10 Methanol, CH_3OH, or **acetone**, C_3H_6O; highest purity grade.

5.11 Phenol stock solutions

As an example, weigh at least 30 mg, to the nearest 0,1 mg, of each of the phenolic compounds in a 100 ml graduated flask and dissolve in methanol or acetone (5.10). For simultaneous determinations, several types of phenol may be dissolved in the appropriate volume of methanol. Keep the solutions cool (about 4 °C) and in dark glass bottles.

The solutions are stable for about four weeks.

5.12 Phenol standard solutions

Pipette 1 ml of the stock solution (5.11) into a 100 ml volumetric flask and make up to volume with methanol. The solution contains 3,0 mg/l of phenol. Prepare further standard solutions in accordance with the working range selected.

5.13 Stock solutions for the internal control solution

As an example, dissolve 0,1 g of 2,4-dibromophenol or 2,5-dibromophenol in 100 ml of methanol (5.10).

5.14 Standard solutions for the control solution

As an example, dilute 1 ml of the stock solution (5.13) with methanol (5.10) to 100 ml.

6 Apparatus

6.1 Flat-bottomed brown glass bottles, with conical shoulder, of capacity 250 ml and 1 000 ml.

Bottles should be rigorously cleaned.

6.2 Separating funnels, of capacity 100 ml and 250 ml, with stopcocks made of polytetrafluoroethene.

6.3 Graduated flasks, of capacity 5 ml, 10 ml and 100 ml.

6.4 Graduated round bottom flask, of capacity 50 ml, with a tapered tip in the base.

6.5 Graduated cylinder, of capacity 250 ml.

6.6 Injection syringes, of capacity 100 µl, 250 µl and 500 µl.

6.7 Gas chromatograph, with a glass insert and electron-capture detector (ECD), gas supply in accordance with the respective manufacturer's instructions.