SLOVENSKI STANDARD
SIST ISO 8165-1:1997

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Kakovost vode - Določanje izbranih enovalentnih fenolov - 1. del: Plinska kromatografska metoda po prekoncentriranju z ekstrakcijo

Water quality -- Determination of selected monovalent phenols -- Part 1: Gas-chromatographic method after enrichment by extraction

Qualité de l'eau -- Dosage des phénols monovalents sélectionnés -- Partie 1: Méthode par chromatographie en phase gazeuse après enrichissement par extraction

Ta slovenski standard je istoveten z: **ISO 8165-1:1992**

ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances

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INTERNATIONAL STANDARD

ISO 8165-1

First edition
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Water quality — Determination of selected monovalent phenols —

Part 1:

Gas-chromatographic method after enrichment by extraction

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

Partie 1: Méthode par chromatographie en phase gazeuse après enrichissement par extraction

Reference number
ISO 8165-1:1992(E)

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.

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 8165-1 was prepared by Technical Committee ISO/TC 147, *Water quality*, Sub-Committee SC 2, *Physical, chemical, 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 after derivatization with pentafluoro-benzoyl bromide*

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Introduction

In the determination of phenols by gas chromatography, several pretreatment methods may be applied depending on the problem to be solved. Basically, the extraction procedure described in this International Standard may be applied to all kinds of water. Compared with derivatization procedures, the limits of determination achievable with this procedure are not quite as low. On the other hand, the derivatization procedures are more likely to be interfered with by compounds such as amines and sometimes alcohols, therefore these procedures cannot be applied to all kinds of waste water.

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Water quality — Determination of selected monovalent phenols —

Part 1:
Gas-chromatographic method after enrichment by extraction

1 Scope

This part of ISO 8165 specifies a method for the determination of the phenols presented in table 1 in a concentration range from 0,1 µg/l to 1 mg/l. The concentration range depends on the nature of the phenols to be determined and on the gas chromatographic method used.

Other monovalent phenols may also be analyzed according to this procedure, the applicability, however, should be investigated for each particular case.

Table 1 — Phenols determinable using this method

Phenol
2-Methylphenol
3-Methylphenol
4-Methylphenol
2,4-Dimethylphenol
4-Ethylphenol
2,6-Di-*tert*-butyl-4-methylphenol
2-Phenylphenol
2-Benzylphenol
2-Benzyl-4-methylphenol
2-Chlorophenol
3-Chlorophenol
4-Chlorophenol
4-Chloro-2-methylphenol
4-Chloro-3-methylphenol
2,4-Dichloro-3,5-dimethylphenol
2-Cyclopentyl-4-chlorophenol
6-Chlorothymol
2,3-Dichlorophenol
2,4-Dichlorophenol
2,5-Dichlorophenol
2,6-Dichlorophenol
2,4,6-Trichlorophenol
2,3,5-Trichlorophenol
2,4,5-Trichlorophenol
2,3,6-Trichlorophenol
2,3,4,5-Tetrachlorophenol
2,3,4,6-Tetrachlorophenol
2,3,5,6-Tetrachlorophenol
Pentachlorophenol
1-Naphthol
2-Naphthol
6-Chloro-3-methylphenol
2-Chloro-4-*tert*-butylphenol
4-Chloro-2-benzylphenol

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 8165. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 8165 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 5667-3:1985, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*

3 Principle

Extraction of the unfiltered sample with diethylether and enrichment of the phenolic compounds in the extract under defined conditions. Gas chromatographic evaluation, using two capillary columns of different polarity (simultaneous splitting) and detection with a flame ionization detector (FID) or an electron capture detector (ECD) in the case of polychlorinated phenols.

4 Interferences

Surfactants, emulsifiers or high concentrations of polar solvents, such as acetone, methanol etc. will affect the extraction. Suspended particles in the water sample may also interfere with the extraction. A second liquid phase in the water sample (e.g. mineral oil compounds, highly volatile chlorinated hydrocarbons, emulsified fats and wax) hamper the pretreatment and the extraction. In this case, only the aqueous phase shall be investigated and the volume of the non-aqueous phase shall be reported with the results.

Interferences of the gas chromatographic system may have various reasons and shall be investigated by the applier with the aid of the operating manual.

5 Reagents

The content of monophenols in water and in the reagents used should be negligibly low. The blank of the water should be determined according to 8.3. If necessary, the water should be purified by distillation of water alkalized with sodium hydroxide (NaOH).

5.1 Sulfuric acid, $\rho = 1{,}84$ g/ml, diluted 1 + 3.

5.2 Sodium hydroxyde solution I, $c = 2$ mol/l.

5.3 Sodium hydroxide solution II, $c = 0{,}2$ mol/l.

5.4 Sodium sulfite (Na_2SO_3).

5.5 Methanol (CH_3OH).

5.6 Dioxane ($C_4H_8O_2$), freshly distilled if necessary.

5.7 Diethylether ($C_4H_{10}O$).

Normally diethylether is stabilized with 2,6-di-tert-butyl-phenol or 2,6-di-tert-butyl-4-methyl-phenol and has to be cleaned prior to use as follows.

Add 10 ml of sodium hydroxide solution I (5.3) to 500 ml of diethylether and distill over a 50 cm long Vigreux column. Discard a residue of 50 ml. The residue may contain peroxides and shall therefore be treated appropiately.

5.8 Silica gel, particle size 0,063 mm × 0,200 mm (equivalent to 70 × 230 mesh).

5.9 Diethylamine ($C_4H_{11}N$), freshly distilled if necessary.

WARNING — Diethylamine is toxic.

5.10 Sodium sulfate (Na_2SO_4), anhydrous.

5.11 Internal standard stock solution.

Dissolve, for example, 1 g of 2,4-dibromophenol or 2,5-dibromophenol in 1 litre of acetone.

1 ml of this solution contains 1 mg of phenol.

5.12 Internal standard solution.

Dilute, for example, 1 ml of internal standard stock solution (5.11) with acetone to 100 ml.

1 ml of this solution contains 10 µg of phenol.

5.13 Phenol stock solution.

Dissolve, for example, 10,0 mg of the respective phenol in methanol in a 100 ml measuring flask and dilute to volume with methanol. The solution contains 0,1 mg/ml of the respective phenol.

Instead of methanol, acetone may also be used.

For the simultaneous determination, several phenols may be dissolved in the respective volume of methanol.

Store the stock solutions in brown glass bottles, tightly stoppered, in a refrigerator.

5.14 Phenol standard solutions.

Pipette 10 ml of the stock solution (5.13) into a 100 ml measuring flask, and dilute to volume with methanol.

The solution contains 0,01 mg/ml of the respective phenol. Prepare the solutions freshly before use.

6 Apparatus

6.1 Storage bottles, brown glass, of capacity 250 ml and 1 000 ml.

6.2 Water bath.

6.3 Distillation apparatus for the distillation of solvents, e.g. a round-bottomed flask, of capacity 1 000 ml, distillation head, condenser, adapter, distillation receiver, e.g. a round-bottomed flask, of capacity 1 000 ml.

6.4 Distillation apparatus for concentration of the extract, consisting of a round-bottomed flask, of capacity 250 ml, with tapered tip, gas inlet tube, distillation head, thermometer, condenser, adapter, and distillation receiver, e.g. a round-bottomed flask, of capacity 50 ml. (See figure 1.)

6.5 Glass column, of length 20 cm and inner diameter 12 mm, tapered at the bottom, which is filled with 5 cm of silica gel (5.8), pre-cleaned with diethylether (see 5.7).

6.6 Shaking apparatus, linear shaker.

6.7 Separating funnels, with polytetrafluoroethylene (PTFE) cocks, of capacity 100 ml, 250 ml and 1 000 ml.

6.8 Measuring flasks, of capacity 5 ml, 10 ml and 1 000 ml.

6.9 Beakers, of capacity 100 ml, 250 ml and 1 000 ml.

6.10 Vigreux column, of length 50 cm.

6.11 Tapered round-bottomed calibrated flask, of capacity 10 ml.

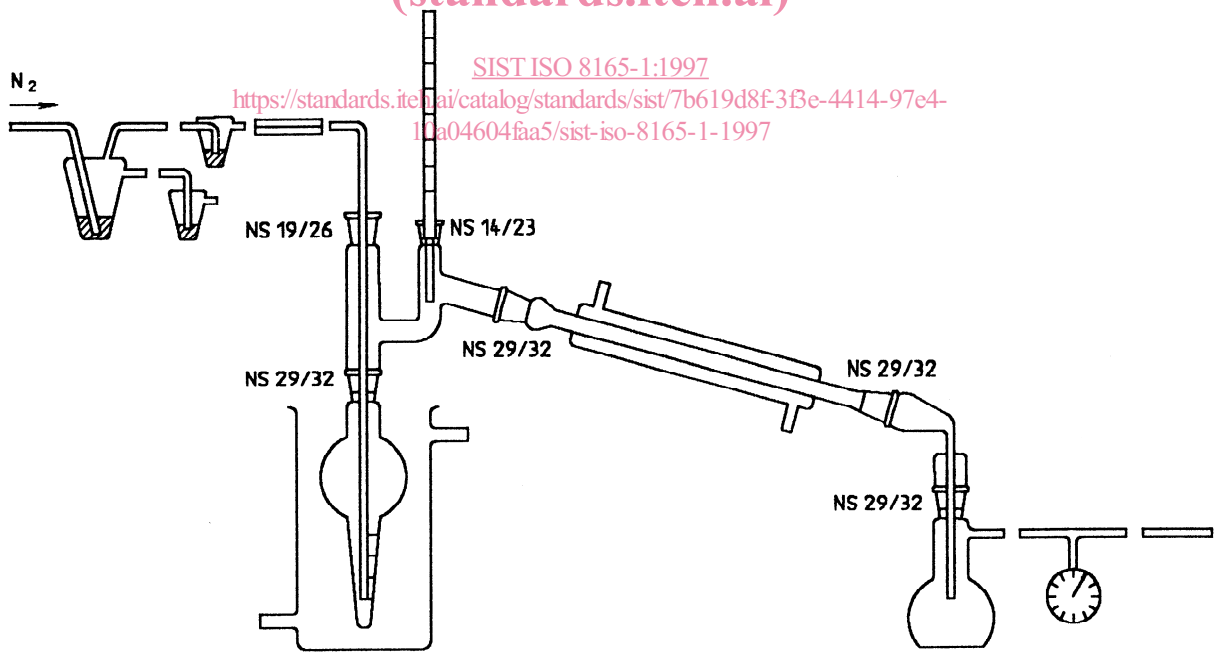

Figure 1 — Apparatus for the concentration of phenols from ether extracts under conditions of isothermic distillation