

Water quality — Determination of selected organic nitrogen and phosphorus compounds — Gas chromatographic methods

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British Standard

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The UK participation in its preparation was entrusted by Technical Committee EH/3, Water quality, to Subcommittee EH/3/2, Physical, chemical & biochemical methods, which has the responsibility to:

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Attention is drawn to the fact that CEN and CENELEC Standards normally include an annex which lists normative references to international publications with their corresponding European publications. The British Standards which implement these international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled “International Standards Correspondence Index”, or by using the “Find” facility of the BSI Standards Electronic Catalogue.

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Summary of pages

This document comprises a front cover, an inside front cover, the EN ISO title page, EN ISO foreword page, ISO title page, pages ii to iv, pages 1 to 23, the annex ZA page, an inside back cover and a back cover.

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This European Standard was approved by CEN on 25 February 2000.

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Foreword

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The text of the International Standard ISO 10695:2000 has been prepared by Technical Committee ISO/TC 147 "Water quality" in collaboration with Technical Committee CEN/TC 230 "Water analysis", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2000, and conflicting national standards shall be withdrawn at the latest by October 2000.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 10695:2000 was approved by CEN as a European Standard without any modification.

NOTE: Normative references to International Standards are listed in annex ZA (normative).

INTERNATIONAL STANDARD

EN ISO 10695:2000

**ISO
10695**

First edition
2000-04-01

Water quality — Determination of selected organic nitrogen and phosphorus compounds — Gas chromatographic methods

*Qualité de l'eau — Dosage de certains composés organiques azotés et
phosphorés sélectionnés — Méthodes par chromatographie en phase
gazeuse*



Reference number
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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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 10695 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Annexes A, B, C and D of this International Standard are for information only.

Water quality — Determination of selected organic nitrogen and phosphorus compounds — Gas chromatographic methods

WARNING — This International Standard makes use of flammable and toxic organic solvents and some toxic organic and phosphorus compounds. Observe the safety regulations in effect.

1 Scope

This International Standard specifies two methods for the determination of certain organic nitrogen and phosphorus compounds in waters by gas chromatography (see Table 1).

The methods may be extended to include additional substances, provided the methods are validated for each individual case.

Clause 3 describes the liquid/liquid extraction method, which is applicable to samples of drinking waters, ground waters, surface waters and waste waters containing up to 0,05 g/l of suspended solids. In the presence of organic matter, suspended matter and colloids, interferences are more numerous and consequently the detection limits of this method can be higher.

NOTE Because of the very low concentrations normally present in the waters, the problem of contamination is extremely important. The lower the level measured, the more precautions have to be observed.

Clause 4 describes the liquid/solid extraction method which is applicable to samples of ground water, surface water and drinking water containing mass concentrations of about $\geq 0,05 \mu\text{g/l}$. Interferences occurring with the examination of some types of surface water can prevent the application of this method.

Detection limits are given for information in annex A.

NOTE When applying this International Standard, the guide on analytical quality control for water analysis (see ISO/TR 13530) should be followed.

Table 1 — Organic nitrogen and phosphorus compounds analysed by these methods

Name	Molecular formula	Molar mass	CAS No. ^a
Atrazine	C ₈ H ₁₄ ClN ₅	215,7	001912-24-9
Cyanazine	C ₉ H ₁₃ ClN ₆	240,7	021725-46-2
Metazachlor	C ₁₄ H ₁₆ ClN ₃ O	277,8	067129-08-2
Parathion (ethyl)	C ₁₀ H ₁₄ NO ₅ PS	291,3	00056-38-2
Parathion (methyl)	C ₈ H ₁₀ NO ₅ PS	263,2	298-00-0
Pendimethalin	C ₁₃ H ₁₉ N ₃ O ₄	281,3	040487-42-1
Propazine	C ₉ H ₁₆ ClN ₅	229,7	000139-40-2
Sebuthylazine	C ₉ H ₁₆ ClN ₅	229,7	007286-69-3
Simazine	C ₇ H ₁₂ ClN ₅	201,7	000122-34-9
Terbuthylazine	C ₉ H ₁₆ ClN ₅	229,7	005915-41-3
Trifluralin	C ₁₃ H ₁₆ F ₃ N ₃ O ₄	335,3	001582-09-8
Vinclozolin	C ₁₂ H ₉ Cl ₂ NO ₃	286,1	050471-44-8

^a Chemical Abstracts Registry Number.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard 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/TR 13530:1997, *Water quality — Guide to analytical quality control for water analysis*.

3 Liquid/liquid extraction

3.1 Principle

The organic nitrogen and phosphorus compounds in the water sample are extracted by liquid-liquid extraction with dichloromethane. After concentration, the sample extracts are analysed by gas chromatography, using a nitrogen-phosphorus detector.

3.2 Reagents

All reagents, including water, shall be of sufficient purity that they do not give rise to significant interfering peaks in the gas chromatograms of the blanks. The purity of reagents used in the procedure shall be verified for each batch of material by running blank determinations (3.5.6).

Reagents shall be stored in glass containers.

NOTE Commercial "pesticide grade" solvents are available. The use of these products is recommended only after verifying their quality. The quality of a solvent is checked by evaporation of about 200 ml down to 1 ml and analysis of the concentrate to determine the compounds subsequently analysed. The solvent should be considered acceptable if it does not give any detectable interfering peaks in the chromatogram for the substance of interest.

3.2.1 Water, purified using, for example, ion exchange and carbon column adsorption.

3.2.2 Dichloromethane (CH_2Cl_2) extraction solvent.

3.2.3 Solvents for dissolution, e.g. acetone or ethyl acetate.

3.2.4 Sodium sulfate, anhydrous

Heat Na_2SO_4 powder at $500\text{ }^\circ\text{C} \pm 20\text{ }^\circ\text{C}$ for $4\text{ h} \pm 30\text{ min}$, cool to about $200\text{ }^\circ\text{C}$ in a muffle furnace and then to ambient temperature in a desiccator containing magnesium perchlorate or equivalent alternative.

3.2.5 Solutions for neutralization

a) **Sodium hydroxide**, aqueous solution, $c(\text{NaOH}) = 1\text{ mol/l}$.

b) **Hydrochloric acid**, aqueous solution, $c(\text{HCl}) = 1\text{ mol/l}$.

3.2.6 Standard stock solutions

Standard stock solutions shall be prepared by dissolving pure or, if available, certified organic nitrogen and phosphorus compounds in acetone (3.2.3).

Unless manufacturer's information or stability trials indicate otherwise, store the solutions at around $+4\text{ }^\circ\text{C}$ in the dark (they can be stored at around $-18\text{ }^\circ\text{C}$, but some septa closures may harden at such a temperature with possible losses of solvent).

For compounds listed in Table 1, the stock solutions are stable for around six months. For other compounds, the laboratory shall check the stability of the solutions.

Prior to use, the solutions shall be brought to ambient temperature.

NOTE 1 A convenient concentration of standard stock solution is obtained by weighing accurately approximately 50,0 mg of each determinand and dissolving it in 100 ml of acetone (3.2.3).

NOTE 2 Certified commercial standards may be used.

3.2.7 Intermediate standard solutions

Prepare intermediate standard solutions by a suitable dilution of the stock solution (3.2.6) with the solvent for dissolution (3.2.3). A typical value is 1 mg/100 ml.

Unless manufacturer's information or stability trials indicate otherwise, store these solutions at around $+4\text{ }^\circ\text{C}$ in the dark.

For compounds listed in Table 1, the intermediate standard solutions are stable up to two months in acetone, except for cyanazine and vinclozolin (one week). For other compounds, the laboratory shall check the stability of the solutions.

3.2.8 Working standard solutions

Prepare at least five different concentrations by suitable dilutions of the intermediate standard solutions (3.2.7) with the solvent for dissolution (3.2.3). A suitable concentration range is 1 μg to 100 μg per 100 ml.