
**Water quality — Determination of
phosphorus — Ammonium molybdate
spectrometric method**

*Qualité de l'eau — Dosage du phosphore — Méthode spectrométrique
au molybdate d'ammonium*

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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 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 6878 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

This second edition cancels and replaces the first edition (ISO 6878:1998), which has been technically revised.

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Introduction

This International Standard specifies the determination of different forms of phosphorus compounds present in ground, surface and waste waters in various concentrations in the dissolved and undissolved state.

The user should be aware that particular problems could require the specification of additional marginal conditions.

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Water quality — Determination of phosphorus — Ammonium molybdate spectrometric 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. It is absolutely essential that tests conducted according to this International Standard be carried out by suitably qualified staff. Molybdate and antimony waste solutions should be disposed of properly.

1 Scope

This International Standard specifies methods for the determination of

- orthophosphate (see Clause 4);
- orthophosphate after solvent extraction (see Clause 5);
- hydrolysable phosphate plus orthophosphate (see Clause 6);
- total phosphorus after decomposition (see Clauses 7 and 8).

The methods are applicable to all kinds of water including seawater and effluents. Phosphorus concentrations within the range of 0,005 mg/l to 0,8 mg/l may be determined in such samples without dilution.

http://www.iso.org/iso/standards A solvent extraction procedure allows smaller phosphorus concentrations to be determined with a detection limit of about 0,000 5 mg/l.

2 Interferences

See Annex A for some known interferences. There may be others and it is recommended to verify whether any such interferences exist and take action to eliminate them.

3 Principle

Reaction of orthophosphate ions with an acid solution containing molybdate and antimony ions to form an antimony phosphomolybdate complex.

Reduction of the complex with ascorbic acid to form a strongly coloured molybdenum blue complex. Measurement of the absorbance of this complex to determine the concentration of orthophosphate present.

Polyphosphate and some organophosphorus compounds are determined if converted to molybdate reactive orthophosphate formed by sulfuric acid hydrolysis.

Many organophosphorus compounds are converted to orthophosphate by mineralization with peroxodisulfate. Nitric acid-sulfuric acid mineralization is used if a more vigorous treatment is required.

4 Determination of orthophosphate

4.1 Reagents

During the analysis, use only reagents of recognized analytical grade and only water having a phosphate content that is negligible compared with the lowest concentration to be determined in the samples.

For low phosphate contents, double-distilled water from an all-glass apparatus is recommended.

4.1.1 Sulfuric acid solution, $c(\text{H}_2\text{SO}_4) \approx 9 \text{ mol/l}$.

Add 500 ml \pm 5 ml of water to a 2 l beaker. Cautiously add, with continuous stirring and cooling, 500 ml \pm 5 ml of sulfuric acid, $\rho = 1,84 \text{ g/ml}$. Mix well and allow the solution to cool to room temperature.

4.1.2 Sulfuric acid solution, $c(\text{H}_2\text{SO}_4) \approx 4,5 \text{ mol/l}$.

Add 500 ml \pm 5 ml of water to a 2 l beaker. Cautiously add, with continuous stirring and cooling, 500 ml \pm 5 ml of sulfuric acid (4.1.1). Mix well and allow to cool to room temperature.

4.1.3 Sulfuric acid solution, $c(\text{H}_2\text{SO}_4) \approx 2 \text{ mol/l}$.

Add 300 ml \pm 3 ml of water to a 1 l beaker. Cautiously add 110 ml \pm 2 ml of sulfuric acid solution (4.1.1), with continuous stirring and cooling. In a measuring flask, dilute to 500 ml \pm 2 ml with water and mix well.

4.1.4 Sodium hydroxide solution, $c(\text{NaOH}) = 2 \text{ mol/l}$.

Dissolve 80 g \pm 1 g of sodium hydroxide pellets in water, cool and dilute to 1 l with water.

4.1.5 Ascorbic acid solution, $\rho = 100 \text{ g/l}$.

Dissolve 10 g \pm 0,5 g of ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$) in 100 ml \pm 5 ml water.

NOTE The solution is stable for 2 weeks if stored in an amber glass bottle in a refrigerator and can be used as long as it remains colourless.

4.1.6 Acid molybdate, Solution I.

Dissolve 13 g \pm 0,5 g of ammonium heptamolybdate tetrahydrate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}]$ in 100 ml \pm 5 ml of water. Dissolve 0,35 g \pm 0,05 g of antimony potassium tartrate hemihydrate $[\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6\cdot \frac{1}{2}\text{H}_2\text{O}]$ in 100 ml \pm 5 ml of water.

Add the molybdate solution to 300 ml \pm 5 ml of sulfuric acid (4.1.1) with continuous stirring. Add the tartrate solution and mix well.

NOTE The reagent is stable for at least 2 months if stored in an amber glass bottle.

4.1.7 Acid molybdate, Solution II.

Cautiously add 230 ml \pm 0,5 ml of sulfuric acid (4.1.1) to 70 ml \pm 5 ml of water, cool. Dissolve 13 g \pm 0,5 g of ammonium heptamolybdate tetrahydrate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}]$ in 100 ml \pm 5 ml of water. Add to the acid solution and mix well. Dissolve 0,35 g \pm 0,05 g of antimony potassium tartrate hemihydrate $[\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6\cdot \frac{1}{2}\text{H}_2\text{O}]$ in 100 ml \pm 5 ml of water. Add to the molybdate-acid solution and mix well.

This reagent is used when the sample is acidified with sulfuric acid (4.1.2) (see also Clauses 6, 7 and 8).

NOTE The reagent is stable for at least 2 months if stored in an amber glass bottle.

4.1.8 Turbidity-colour compensation solution.

On a volume/volume basis, mix two parts of sulfuric acid (4.1.2) and one part of ascorbic acid (4.1.5).

NOTE The reagent is stable for several weeks if stored in an amber glass bottle in a refrigerator.

4.1.9 Sodium thiosulfate pentahydrate solution, $\rho = 12,0$ g/l.

Dissolve $1,20 \text{ g} \pm 0,05 \text{ g}$ of sodium thiosulfate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in $100 \text{ ml} \pm 5 \text{ ml}$ of water. Add $0,05 \text{ g} \pm 0,005 \text{ g}$ of anhydrous sodium carbonate (Na_2CO_3) as preservative.

NOTE The reagent is stable for at least 4 weeks if stored in an amber glass bottle.

4.1.10 Orthophosphate stock standard solution, $\rho_{\text{P}} = 50$ mg/l.

Dry a few grams of potassium dihydrogen phosphate to constant mass at 105°C . Dissolve $0,2197 \text{ g} \pm 0,0002 \text{ g}$ of KH_2PO_4 in about $800 \text{ ml} \pm 10 \text{ ml}$ of water in a 1 000 ml volumetric flask. Add $10 \text{ ml} \pm 0,5 \text{ ml}$ of sulfuric acid (4.1.2) and make up to the mark with water.

Alternatively, use a commercially available stock solution.

The solution is stable for at least 3 months if stored in a well stoppered glass bottle. Refrigeration to about 4°C is recommended.

4.1.11 Orthophosphate standard solution, $\rho_{\text{P}} = 2$ mg/l.

Pipette $20 \text{ ml} \pm 0,01 \text{ ml}$ of orthophosphate stock standard solution (4.1.10) into a 500 ml volumetric flask. Make up to the mark with water and mix well.

Prepare and use this solution each day as required.

NOTE 1 ml of this standard solution contains $2 \mu\text{g P}$.

4.1.12 Hydrochloric acid, $\rho(\text{HCl}) = 1,19$ g/ml.**4.1.13 Hydrochloric acid, $c(\text{HCl}) = 2,5$ mol/l.**

Cautiously add $200 \text{ ml} \pm 10 \text{ ml}$ of hydrochloric acid (4.1.12) to $500 \text{ ml} \pm 10 \text{ ml}$ of water. Mix and cool to room temperature. Make up to 1 000 ml with water.

4.2 Apparatus**4.2.1 Spectrometer, "prism"- or "grating-type" or filter type, capable of accepting optical cells of thickness 10 mm to 50 mm.**

The spectrometer chosen shall be suitable for measuring absorbance in the visible and near infra-red regions of the spectrum. The most sensitive wavelength is 880 nm, but if a loss of sensitivity can be accepted, absorbance may be measured at 700 nm.

NOTE The detection limit of the method is lower if a spectrometer capable of accepting 100 mm optical cells is available.

4.2.2 Filter assembly, to hold a membrane filter of nominal pore size $0,45 \mu\text{m}$.**4.2.3 Glassware.**

Before use, wash all glassware, for example with hydrochloric acid (4.1.13), at approximately 40°C to 50°C and rinse thoroughly with water. Detergents containing phosphate shall not be used.

Preferably the glassware should be used only for the determination of phosphorus. After use, clean it as described above and keep covered until needed again.

Rinse glassware used for the colour development stage occasionally with sodium hydroxide solution (4.1.4), followed by thorough rinsing with water (4.1), to remove deposits of the coloured complex which has a tendency to stick as a thin film on the wall of glassware.

4.3 Sampling and samples

4.3.1 Sampling

Collect the laboratory samples in polyethene, polyvinylchloride or preferably glass bottles. In the case of low phosphate concentrations, use glass bottles.

The use of sampling bottles with cap lines should be avoided as these may contain phosphorus.

4.3.2 Preparation of the test sample

Filter the laboratory sample (4.3.1) within 4 h after sampling. If the sample has been kept cool in the meantime, bring to room temperature before filtration.

Wash a membrane filter of nominal pore size 0,45 µm to ensure it is free of phosphate by passing 200 ml of water, previously heated to approximately 30 °C to 40 °C. Discard these washings. Filter the sample and reject the first 10 ml of sample filtrate. Collect the remainder in a clean, dry glass bottle for the immediate determination of orthophosphate (4.4.4).

If the filtrate is not within the range of pH 3 to pH 10, adjust it with sodium hydroxide (4.1.4) or sulfuric acid solution (4.1.3).

The filtration time should not exceed 10 min. If necessary, a larger diameter filter should be used.

The membrane filter should either be checked for phosphorus content or washed as described. Commercially available membrane filters that are sold free from phosphorus should be washed as described.

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4.4 Procedure

4.4.1 Test portion

Take a volume of test portion not exceeding 40 ml. This maximum volume is suitable for the determination of orthophosphate concentrations of up to $\rho_p = 0,8$ mg/l, when using an optical cell of thickness 10 mm. Smaller test portions shall be used in order to accommodate higher phosphate concentrations as shown in Table 1. Similarly, low phosphate concentrations can be determined by measuring the absorbance in an optical cell of thickness 40 mm or 50 mm.

Table 1 — Sample volumes and concentrations

Orthophosphate concentration mg/l	Volume of test portion ml	Thickness of optical cell mm
0,0 to 0,8	40,0	10
0,0 to 1,6	20,0	10
0,0 to 3,2	10,0	10
0,0 to 6,4	5,0	10
0,0 to 0,2	40,0	40 or 50