Phosphorus, Total, Digestion

DOC316.53.01493

USEPA[1] Acid Persulfate Digestion Method[2] — Method 8190

Scope and application: For water, wastewater and seawater.

[1] USEPA Accepted for wastewater analyses when used with the ascorbic acid (PhosVer 3) method.
[2] Adapted from Standard Methods for the Examination of Water and Wastewater 4500-P B & E.

Test preparation

Before starting

Clean all glassware with 6.0 N (1:1) hydrochloric acid, then fully rinse with deionized water to remove contaminants.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

Items to collect

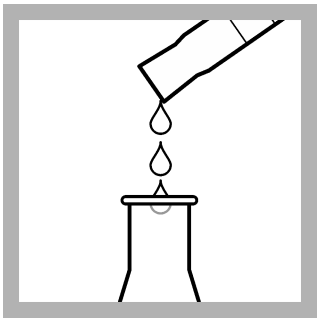
Description	Quantity
Potassium Persulfate Powder Pillow	1
Sodium Hydroxide Solution, 5.0 N	2 mL
Sulfuric Acid Solution, 5.25 N	2 mL
Water, deionized	varies
Cylinder, graduated, 25-mL	1
Flask, Erlenmeyer, 125-mL	1
Hot plate	1

Refer to Consumables and replacement items on page 3 for order information.

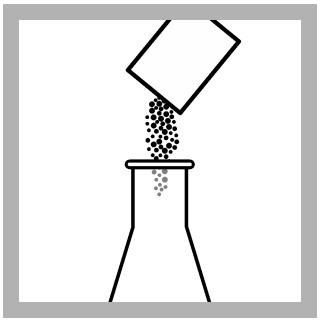
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated sulfuric acid (about 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at or below 6 °C (43 °F) for a maximum of 28 days.
- Let the sample temperature increase to room temperature before analysis.
- Before analysis, adjust the pH to 7 with 5.0 N sodium hydroxide standard solution.
- Correct the test result for the dilution caused by the volume additions.

Acid persulfate digestion procedure

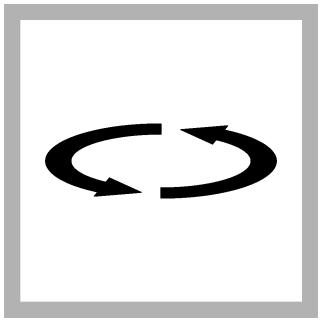


1. Use a graduated cylinder to add 25-mL of sample into the 125-mL Erlenmeyer flask.

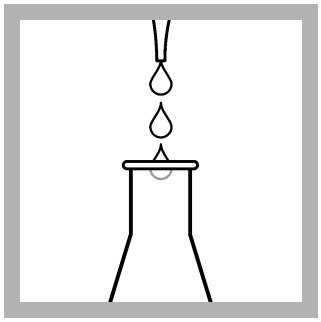


2. Add the contents of one Potassium Persulfate Powder Pillow.

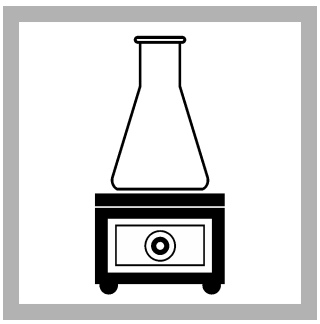
Note: *To identify only inorganic acid hydrolyzable phosphorus (not total including organic), do not add the Potassium Persulfate.*



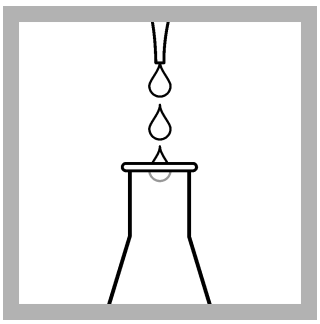
3. Swirl to mix.



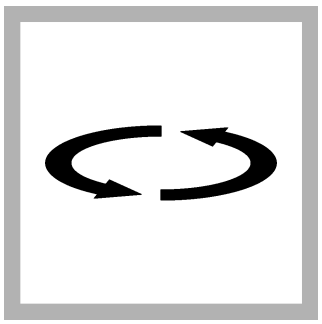
4. Add 2.0 mL of 5.25 N Sulfuric Acid Solution to the flask.



5. Boil the sample gently for 30 minutes. Do not let the flask boil dry.
For the best recovery, concentrate the sample to less than 20 mL. After concentration, maintain the volume of sample near 20 mL by adding small amounts of deionized water. Do not exceed 20 mL.



6. Let the sample cool to room temperature. Add 2.0 mL of 5.0 N Sodium Hydroxide Solution to the flask.



7. Swirl to mix.

8. Pour the sample into a 25-mL graduated cylinder. Adjust the volume to 25 mL by rinsing the flask with deionized water and pouring the rinse water into the cylinder.

9. Proceed with the PhosVer 3 method for reactive phosphorous.

Results of the reactive phosphorus test will include the organic phosphate plus the orthophosphate and the acid-hydrolyzable (condensed) phosphate. The organic phosphate concentration is determined by subtracting the results of an acid hydrolyzable phosphorus test from the test result. Refer to the note in step 2.

Interferences

Interfering substance	Interference level
Alkaline or highly buffered samples	If the pH of the sample after the acid is added is not below pH 1, add additional acid.
Turbidity	Use 50 mL of sample and double the reagent quantities. Use a portion of the digested sample to zero the instrument in the reactive phosphorus procedure. This compensates for any color or turbidity destroyed by this procedure.

Summary of method

Phosphates in organic and condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat provides the conditions for hydrolysis of the condensed inorganic forms. Organic phosphates are converted to orthophosphate by heating with acid and persulfate. Organically bound phosphates are thus determined indirectly by subtracting the result of an acid hydrolyzable phosphorus test (without Potassium Persulfate) from the total phosphorus result. This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods for determining the phosphorus content of the sample. If the ascorbic acid (PhosVer 3) method is used to measure the reactive phosphorus, this method is USEPA accepted for NPDES reporting.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Potassium Persulfate Powder Pillow	1	100/pkg	245199
Sodium Hydroxide Solution, 5.0 N	2 mL	100 mL MDB	245032
Sulfuric Acid Solution, 5.25 N	2 mL	100 mL MDB	244932
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/test	Unit	Item no.
Cylinder, graduated, 25-mL	1	each	50840
Flask, Erlenmeyer, 125-mL	2	each	50543
Hot plate, 7 inch x 7 inch, digital, 120 VAC	1	each	2881500
Hot plate, stirrer, 220–240 VAC	1	each	2881602

Optional reagents and apparatus

Description	Unit	Item no.
Sodium Hydroxide Standard Solution, 5.0 N	1 L	245053
Sulfuric Acid, concentrated, ACS	500 mL	97949
Paper, pH, 0–14 pH range	100/pkg	2601300
Thermometer, non-mercury, –10 to +225 °C	each	2635700
Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	88449
Bottle, sampling, with cap, low density polyethylene, 250-mL	12/pkg	2087076

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