

USEPA¹ Acid Digestion Method²

Method 8180

Scope and application: For water, wastewater, and seawater.

¹ USEPA Accepted for wastewater analyses.

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.

The results of a reactive phosphorus test after acid-hydrolyzable digestion includes the combination of orthophosphate and the acid-hydrolyzable (condensed) phosphate. To find the condensed phosphate concentration, subtract the result of an orthophosphate test without digestion from the result with digestion. Make sure that both results are in the same units, either mg/L PO₄³⁻ or mg/L P. To find the organic phosphorus concentration, subtract the result of an acid hydrolyzable test from the result of a total phosphorus test.

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
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 that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- Do not use a detergent that contains phosphate to clean the sample bottles. The phosphate in the detergent will contaminate the sample.
- Analyze the samples as soon as possible for best results.
- If immediate analysis is not possible, immediately filter and keep the samples at or below 6 °C (43 °F) for a maximum of 48 hours.
- Let the sample temperature increase to room temperature before analysis.

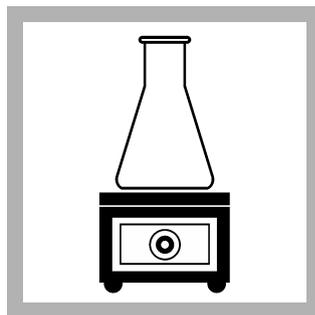
Acid digestion



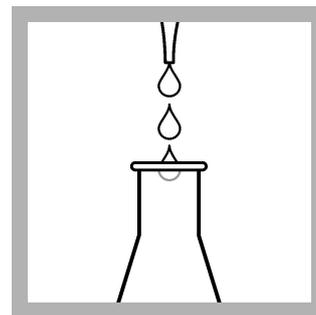
1. Use a graduated cylinder to add 25-mL of sample into the 125-mL Elenmeyer flask.



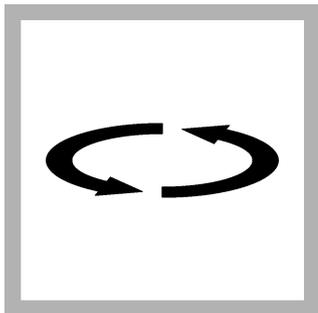
2. Add 2.0 mL of 5.25 N Sulfuric Acid Solution to the flask.



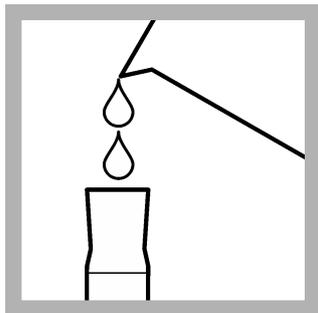
3. Boil the sample gently for 30 minutes. Do not let the flask boil dry. For the best recovery, concentrate the sample to less than 20 mL. After concentration, maintain the volume of sample near 20 mL by adding small amounts of deionized water. Do not exceed 20 mL.



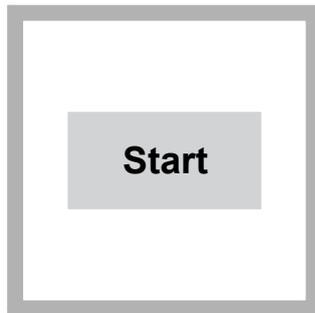
4. Let the sample cool to room temperature. Add 2.0 mL of 5.0 N Sodium Hydroxide Solution to the flask.



5. Swirl to mix.



6. 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.



7. Proceed with a reactive phosphorous test of the expected acid hydrolyzable phosphorous concentration range.

- 480 P React. Mo
- 482 P React. Mo AV
- 485 P React. Amino
- 490 P React. PV
- 492 P React. PV AV
- 535 P React. PV TNT
- 540 P React. HT TNT

Extend the color development time to 10 minutes for the PhosVer 3 method.

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 present in condensed inorganic forms (meta-, pyro- or other polyphosphates) must be converted to reactive orthophosphate before analysis. Pretreatment of the sample with acid and heat hydrolyzes the condensed inorganic forms to orthophosphate. This procedure must be followed by one of the reactive phosphorus (orthophosphate) analysis methods to determine 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.
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

Required apparatus (field applications)

Description	Quantity/test	Unit	Item no.
Heatab cookit, with 1 box Heatabs	—	each	220600
Heatab replacements	—	21/pkg	220700

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
Filter paper, folded, 3–5-micron, 12.5-cm	100/pkg	69257
Funnel, poly, 65-mm	each	108367
Thermometer, non-mercury, –10 to +225 °C	each	2635700

Optional reagents and apparatus (continued)

Description	Unit	Item no.
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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