

Gravimetric Method¹

Method 8277

Scope and application: For wastewater.

¹ Adapted from *Standard Methods for the Examination of Water and Wastewater*.



Test preparation

Before starting

If applicable, use resulting samples from Method 8163 in this procedure. Use the resulting sample and start this method at step 18.

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

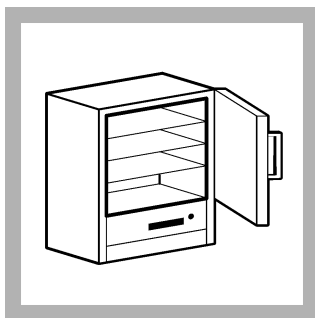
Description	Quantity
Filter flask	1
Filter holder assembly with stopper	1
Filter, 47-mm, glass fiber	1
Graduated cylinder, 100-mL	1
Tongs	1
Evaporating dish	1
Desiccator with desiccant	1
Analytical balance	1
Muffle furnace	1
Vacuum source and tubing	1
Deionized water	1
Steam bath	1
Hot plate	1

Refer to [Consumables and replacement items](#) on page 4 for order information.

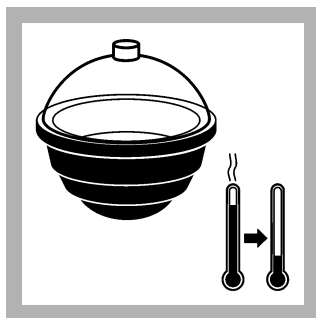
Sample collection preservation and storage

- Collect samples in clean glass or plastic bottles.
- Analyze the samples as soon as possible for best results.
- If prompt analysis is not possible, keep the samples at or below 4 °C (39 °F) for up to 7 days.
- Let the sample temperature increase to room temperature before analysis.

Test procedure



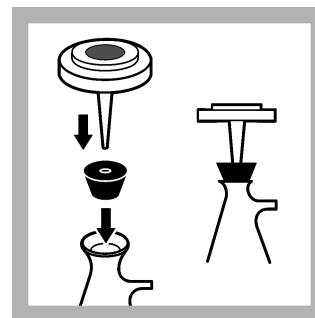
1. Put an evaporating dish in a drying oven at 550 °C (1022 °F) for 1 hour.



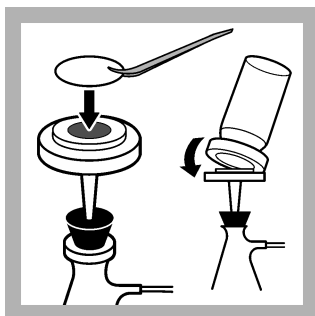
2. Remove the dish from the oven. Let the dish temperature decrease to room temperature in a desiccator.



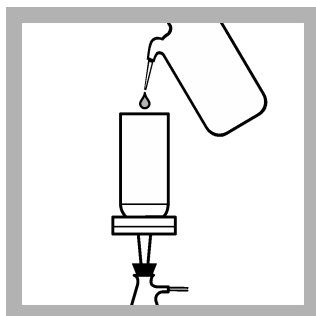
3. Remove all of the residue from the flask with a dilute solution of ammonium hydroxide. Rinse with deionized water.



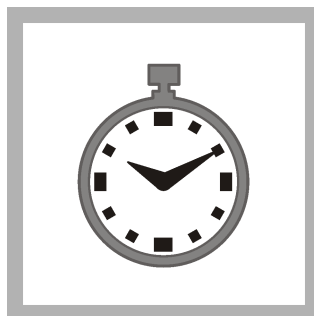
4. Assemble the filter holder/flask assembly using a clean filter flask.



5. Use tweezers to put a 47-mm filter disc in the filter holder. Put the filter holder assembly in the filtering flask.



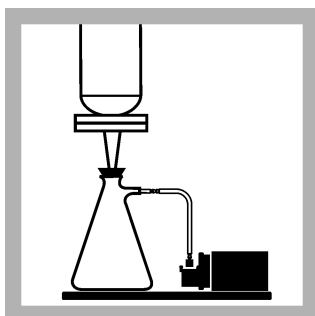
6. Connect vacuum to the filter holder/flask assembly and wash the filter with three separate 20-mL volumes of deionized water.



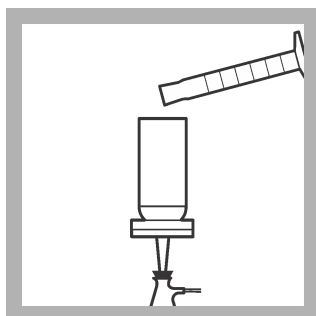
7. Apply vacuum for 2 to 3 minutes after the water has passed through the filter.



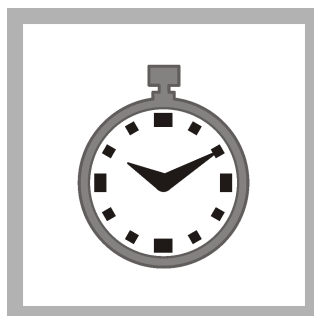
8. Disconnect the vacuum. Discard the washings from the flask.



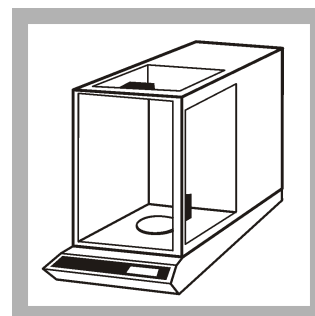
9. Connect vacuum to the filter holder/flask assembly again.



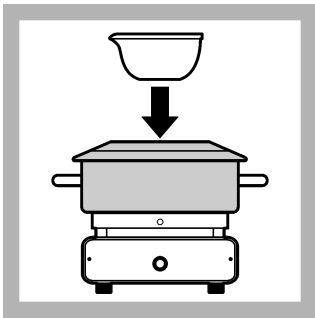
10. Use a clean 100-mL graduated cylinder to pour 100 mL (or more) of a well-mixed, representative water sample into the filter holder. **Note:** For best accuracy, filter as much sample as possible. A sample that contains more than 15 mg of solids can clog the filter. Adjust the volume of the water sample to get the optimum condition. 5–10 completed tests will show if an adjustment is necessary.



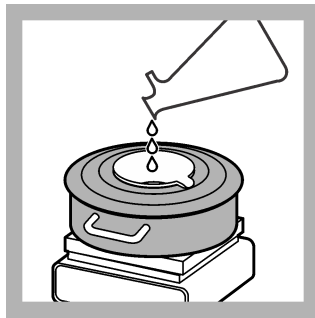
11. Apply vacuum for 2 to 3 minutes after the sample has passed through the filter. Disconnect the vacuum.



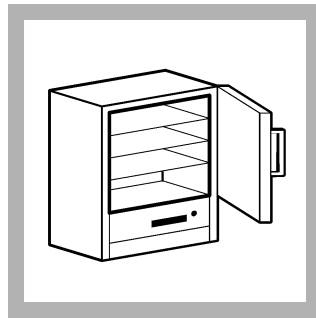
12. Use an analytical balance to weigh the dish to the nearest 0.1 mg (0.0001 g). Record this mg value as A.



13. Put the steam bath on the hot plate. Add water to the steam bath. Put the evaporating dish in the steam bath.



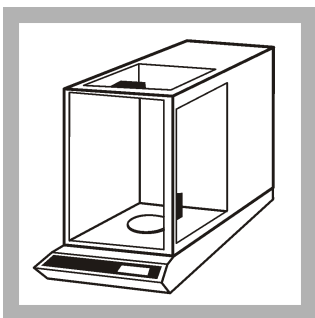
14. Pour the 100-mL filtrate sample from the filter flask into the evaporating dish. Dry for 4 hours. Examine the reservoir of the water bath at regular intervals. Add more water if necessary.



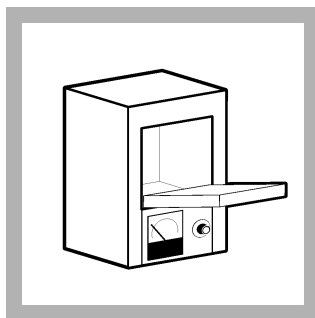
15. Put the evaporating dish in a drying oven at 103–105 °C (217–221 °C) for 1 hour.



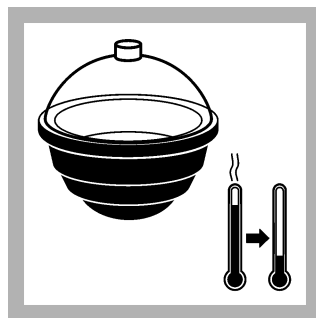
16. Remove the dish from the oven. Let the dish temperature decrease to room temperature in a desiccator.



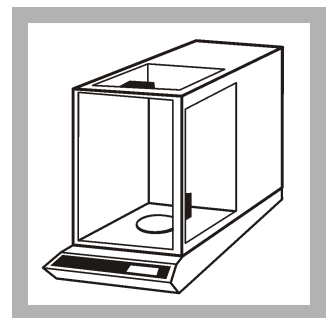
17. Use an analytical balance to weigh the dish to the nearest 0.1 mg (0.0001 g). Record this mg value as B.



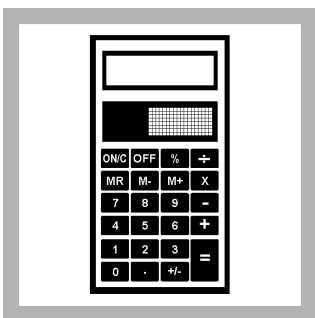
18. Put the evaporating dish into a pre-heated muffle furnace at 550 °C (1022 °F) for 30 minutes.



19. Remove the dish from the oven. Let the dish temperature decrease to room temperature in a desiccator.



20. Use an analytical balance to weigh the dish to the nearest 0.1 mg (0.0001 g). Do steps 16 and 17 until the difference between two successive sample weighings is not more than 4% or 0.5 mg, whichever is greater. Record this mg value as D.



21. The loss of weight is total volatile solids. Weighed residue is total fixed solids. Calculate the test results:

$$(B - D) \times 1000 / C = \text{mg/L Volatile Dissolved Solids}$$

$$(A - D) \times 1000 / C = \text{mg/L Fixed Dissolved Solids}$$

Summary of method

A well-mixed sample is filtered through a glass fiber filter. An aliquot of the filtrate is evaporated in a weighed dish and dried to constant weight in a 103–105 °C (217–221 °F) oven. The dish and sample residue are ignited at 550 °C (1022 °F) for 30 minutes. The loss of sample mass on ignition is the volatile dissolved solids. The remaining residue after ignition is the fixed dissolved solids.

Consumables and replacement items

Required apparatus

Description	Quantity/test	Unit	Item no.
Balance, Analytical, 115 VAC, 60 Hz	1	each	2936701
Cylinder, graduated, 100 mL	1	each	50842
Desiccant, indicating Drierite	1	each	2088701
Desiccator, without stopcock	1	each	1428500
Desiccator plate, ceramic	1	each	1428400
Evaporating dish, porcelain, with lip, 120-mL, 90-mm	1	each	52561
Furnace, muffle, 120 VAC, 50/60 Hz	1	each	1429600
Furnace, muffle, 240 VAC, 50/60 Hz	1	each	1429624
Steam bath, 8 inch diameter	1	each	2347900
Tongs, crucible, 9 inch	1	each	56900
Aspirator, vacuum pump	1	each	213100
Filter disc, 47 mm, glass fiber	1	100/pkg	253000
Filter holder, 47-mm, magnetic base	1	each	1352900
Flask, filtering 1000 mL	1	each	54653
Hot plate/stirrer, 7 x 7 inch, 115 VAC	1	each	2881600
Hot plate, stirrer, 220–240 VAC	1	each	2881602
Oven, drying, 120 VAC	1	each	1428900
Oven, drying, 240 VAC	1	each	1428902
Stopper, rubber, one-hole No. 8	1	6/pkg	211908
Water, deionized	varies	4 L	27256

Optional reagents and apparatus

Description	Quantity/test	Unit	Item no.
Dish, aluminum (63 x 17.5 mm)	1	100/pkg	2164000
Ammonium Hydroxide, 10%	1	500 mL	1473649
Blender, 2-speed, 120 VAC option	1	each	2616100
Blender, 2-speed, 240 VAC option	1	each	2616102
Bottles, with cap, wide mouth, 500 mL poly	1	12/pkg	2087079
Beaker, 250 mL	1	each	50046H
Vacuum Pump, 1.2 CFM 115 V	1	each	2824800
Pump, vacuum, 1.2 cfm, 220 VAC, European plug	1	each	2824802
Pump, vacuum, hand operated	1	each	1428300

Optional reagents and apparatus (continued)

Description	Quantity/test	Unit	Item no.
Stir bar, 22 x 8 mm	1	each	2095350
Magnetic Stirrer	1	each	2881200



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – Call toll-free 800-227-4224
Outside the U.S.A. – Contact the HACH office or distributor serving you.
On the Worldwide Web – www.hach.com; E-mail – techhelp@hach.com

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932