Arsenic

DOC316.53.01005

Silver Diethyldithiocarbamate Method[1] Method 8013

0 to 0.200 mg/L As

Scope and application: For water, wastewater and seawater. Distillation is required. USEPA accepted[2] for reporting for drinking and wastewater analysis.

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater*.
[2] Procedure is equivalent to Standard Method 3500-As for drinking water analysis.

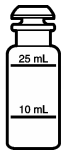
Test preparation

Instrument-specific information

Table 1 shows all of the instruments that can make a user calibration program for this test. The table also shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2612602 
DR 5000 DR 3900	The fill line is toward the user.	

Before starting

A manual calibration is necessary for this procedure, refer to Calibration on page 5. Make a new calibration and update the user program for each new lot of arsenic absorber solution.
Prepare the arsenic absorber solution before starting the test procedure. Refer to Reagent preparation on page 5.
Set up the distillation apparatus in a fume hood to prevent exposure to hazardous gas.
In bright light conditions (e.g., direct sunlight), close the cell compartment, if applicable, with the protective cover during measurements.
Do not use the Pour-Thru Cell or sipper module (for applicable instruments) with this 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
Apparatus (refer to Required apparatus on page 7)	–
Arsenic Standard Solution, 1000–mg/L As	varies
Hydrochloric Acid, ACS	25 mL

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Items to collect (continued)

Description	Quantity
Lead Acetate Solution, 10%	1 mL
Potassium Iodide Solution, 20%	3 mL
Pyridine, ACS	50 mL
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2
Silver Diethyldithiocarbamate	1 g
Stannous Chloride Solution	1 mL
Water, deionized	varies
Zinc, 20-mesh, ACS	6 g

Refer to Consumables and replacement items on page 6 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.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated sulfuric acid (approximately 2 mL per liter). No acid addition is necessary if the sample is tested immediately.
- Keep the preserved samples at room temperature for a maximum of 6 months.
- Before analysis, adjust the pH to approximately 7 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

Test procedure

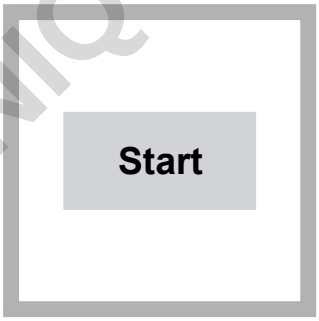
⚠ WARNING

Gas inhalation hazard. Operate the instrument in a fume hood to prevent exposure to hazardous gas.

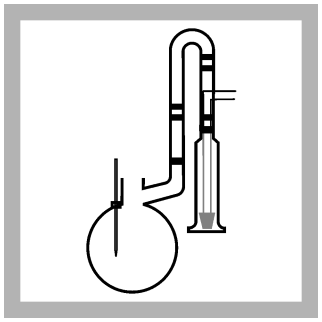


1. Prepare a calibration and user program for this method. Refer to Enter the calibration as a user program on page 5. Record the program number.

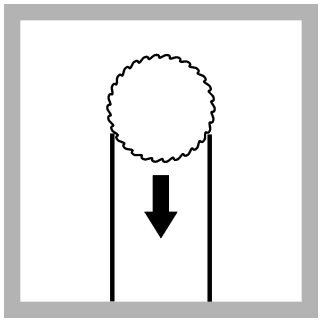
Select User Programs.



2. Start the user-entered program from step 1. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

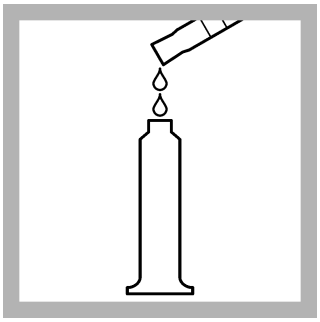


3. Prepare the distillation apparatus, refer to the *Distillation Manual* for assembly instructions. **Do not connect to the aspirator.**

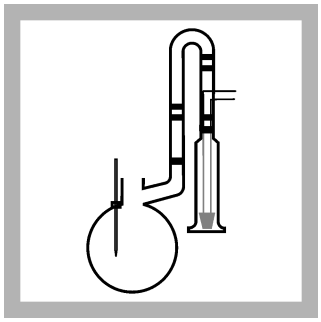


4. Soak a cotton ball with 10% Lead Acetate Solution. Put the cotton ball in the gas scrubber. Make sure that the cotton seals against the glass.

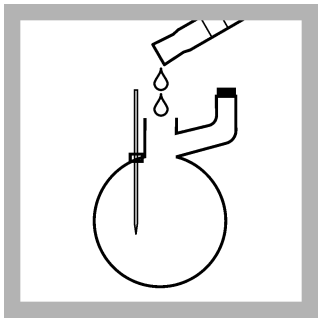
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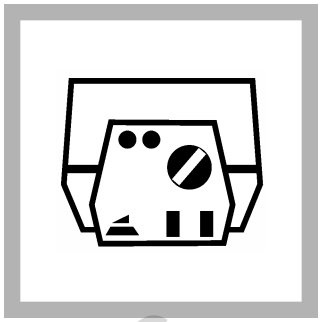
5. Use a graduated cylinder to pour 25-mL of prepared arsenic absorber solution (refer to Reagent preparation on page 5) into the cylinder/gas bubbler assembly.



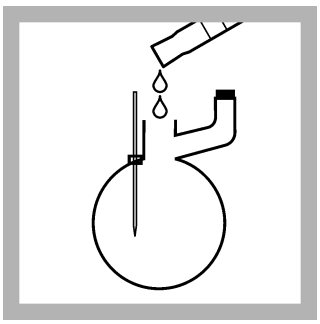
6. Attach the cylinder/gas bubbler assembly to the distillation apparatus.



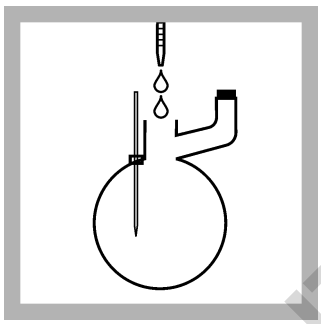
7. Use a graduated cylinder to pour 250 mL of sample into the distillation flask.



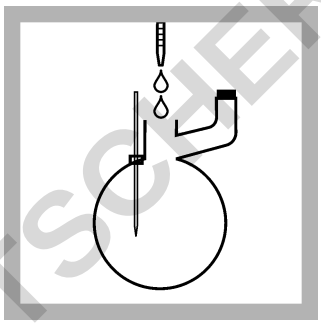
8. Set the heater power to on. Set the stir control to 5. Set the heat control to 0.



9. Use a graduated cylinder to add 25 mL of Hydrochloric Acid ACS to the distillation flask.



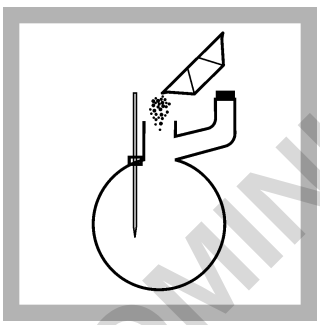
10. Use a serological pipet to add 1 mL of Stannous Chloride Solution to the distillation flask.



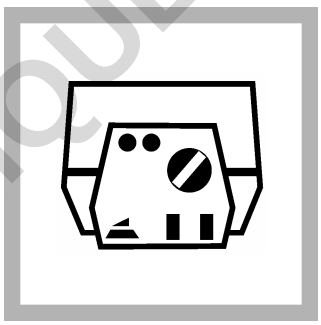
11. Use a serological pipet to add 3 mL of Potassium Iodide Solution to the distillation flask. Put the cap on the flask.



12. Start the instrument timer. A 15-minute reaction time starts.



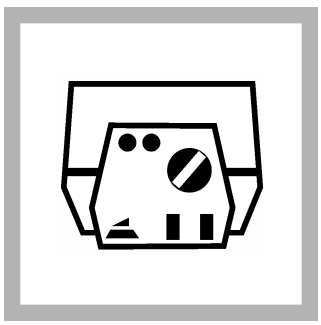
13. When the timer expires, weigh and add 6.0 g of 20-mesh zinc to the distillation flask. **Put the cap on the flask immediately.**



14. Set the heat control to 3.



15. Start the instrument timer. A 15-minute reaction time starts.

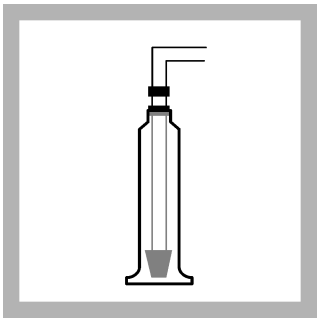


16. When the timer expires, set the heat control to 1.

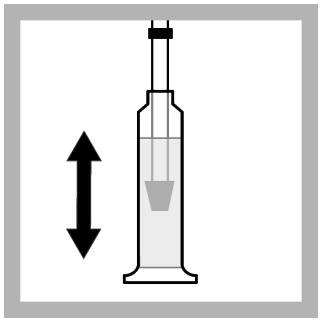
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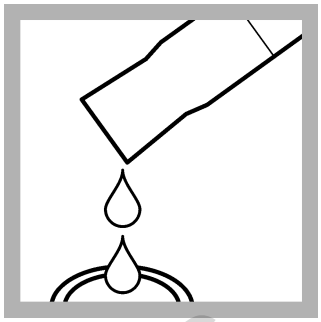
17. Start the instrument timer. A 15-minute reaction time starts.



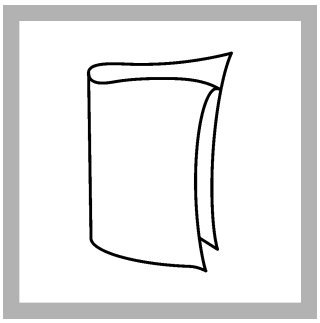
18. When the timer expires, set the heater power to off. Remove the cylinder/gas bubbler assembly as a unit.



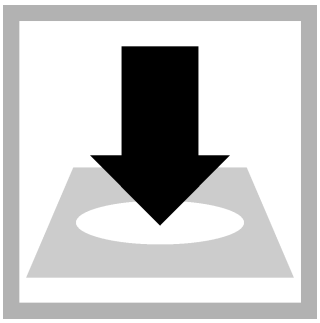
19. Move the gas bubbler up and down in the arsenic absorber solution to rinse the bubbler.



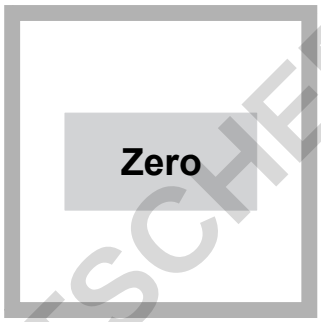
20. Prepare the blank: Fill a dry 10-mL sample cell with unreacted arsenic absorber solution. Put the stopper on the sample cell.



21. Clean the blank sample cell.



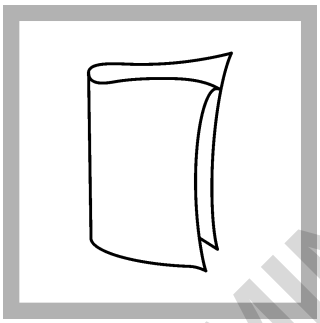
22. Insert the blank into the cell holder.



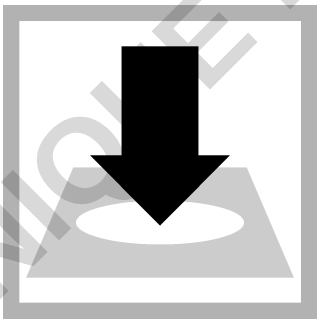
23. Push **ZERO**. The display shows the (non-zero) intercept that is calculated from the user-entered calibration curve.



24. Prepare the sample: Pour the reacted arsenic absorber sample into a sample cell. Put the stopper on the sample cell.



25. Clean the prepared sample cell.



26. Insert the prepared sample into the cell holder.



27. Push **READ**. Results show in mg/L As.

Interferences

Interfering substance	Interference level
Antimony Salts	Can interfere with color development.

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Reagent preparation

⚠CAUTION



Chemical exposure hazard. Obey laboratory safety procedures and wear all of the personal protective equipment appropriate to the chemicals that are handled. Refer to the current safety data sheets (MSDS/SDS) for safety protocols.

Prepare the arsenic absorber solution as follows:

1. Weigh 1.00 g of silver diethyldithiocarbamate on an analytical balance.
2. Add the powder to a 200-mL volumetric flask. Dilute to the mark with pyridine.
3. Mix well to dissolve. Keep the reagent, tightly sealed, for a maximum of 1 month in an amber bottle.

Calibration

This method requires a user-prepared calibration curve. Enter the calibration curve into the instrument as a user program. Make a new calibration curve when a new lot of reagents is used.

Prepare the standard solutions

Prepare the standard solutions for calibration as follows.

Items to collect:

- 1000-mg/L Arsenic Standard Solution
- 1000-mL volumetric flask
- 500-mL volumetric flasks (3), Class A
- 1–10 mL TenSette pipet and tips
- Deionized water

1. Prepare a 10.0-mg/L Arsenic Standard Solution as follows:
   a. Use a pipet to add 10.0 mL of the 1000-mg/L Arsenic Standard Solution into a 1000-mL volumetric flask.
   b. Dilute to the mark with deionized water.
2. Prepare three calibration standard solutions (0.02, 0.04 and 0.20 mg/L arsenic) as follows:
   a. Use a pipet to add 1.0 , 2.0 and 10.0 mL of the 10.0-mg/L Arsenic Standard Solution into three different 500-mL volumetric flasks. Use Class A glassware.
   b. Dilute each flask to the mark with deionized water. Mix well.

Enter the calibration as a user program

After the calibration standards are prepared, make a user program to store the calibration information in the instrument. Select the user program to measure the concentration of samples. The steps that follow are general instructions for all instruments. Refer to the user manual for the instrument that is used for the correct menu options.

1. Use the test procedure to prepare the standard solutions for measurement. Make sure to distill the standard solutions as shown in the test procedure.
2. Go to **User Programs**.
3. For the initial calibration, make a new user program. Set up the basic information for the new program:

Option	Description
Program number	Enter an available number for the user program.

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Option	Description
Program name	Enter a name for the user program, (e.g., the name of the parameter).
Program type	Select single wavelength (for applicable spectrophotometers).

4. Enter the settings for the user program:

Option	Description
Units	mg/L
Wavelength	520 nm
Concentration resolution	0.001
Chemical form	As
Upper limit	0.220
Lower limit	–0.020
Timer 1	15:00
Timer 2	15:00
Timer 3	15:00
Calibration	Read standards

5. Select **CALIBRATION: C = A + BA.** Push **EDIT**.
6. In the Read Standards menu, enter the concentration of the prepared standard solutions.
7. **Prepare the blank solution:** Use a 25-mL sample cell that only contains unreacted arsenic absorber solution.
8. Insert the blank solution into the cell holder and push **ZERO**.
9. Insert the first prepared standard solution into the cell holder. Make sure that the standard solution concentration is selected on the display and push **READ**.
10. Continue to measure the remaining standard solutions.
11. Push **GRAPH**. Make sure that **FORCE ZERO** is off.
12. If the graph is acceptable select **DONE>EXIT**.
13. Save (store) the user program. When the user program is selected in the test procedure, the calibration curve is used to measure the sample concentration.

Summary of Method

A mixture of zinc, stannous chloride, potassium iodide and hydrochloric acid reduces the arsenic into arsine gas in a specially equipped distillation apparatus. The arsine goes through a scrubber that uses a cotton ball soaked with lead acetate for sulfide removal. Then the arsine goes into an absorber tube that contains a solution of silver diethyldithiocarbamate in pyridine. The arsenic reacts to form a red complex that is read colorimetrically. A manual calibration is necessary for this procedure. The measurement wavelength is 520 nm.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Arsenic Standard Solution, 1000-mg/L As	varies	100 mL	1457142
Hydrochloric Acid, ACS	25 mL	500 mL	13449
Lead Acetate Solution, 10%	1 mL	100 mL	1458042
Potassium Iodide Solution, 20%	3 mL	100 mL	1456842
Pyridine, ACS	50 mL	500 mL	1446949

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Consumables and replacement items (continued)

Description	Quantity/test	Unit	Item no.
Silver Diethyldithiocarbamate	1 g	25 g	1447624
Stannous Chloride Solution	1 mL	100 mL	1456942
Water, deionized	varies	4 L	27256
Zinc, 20-mesh, ACS	6 g	454 g	79501

Required apparatus

Description	Quantity/test	Unit	Item no.
Balance, analytical, Zeta series, 80-g capacity	1	each	2936701
Balls, cotton	1	100/pkg	257201
Boat, weighing, 8.9-cm square	2	500/pkg	2179000
Bottle, amber, 237-mL, glass	1	6/pkg	714441
Cap, polypropylene, for amber bottle	1	6/pkg	2166706
Cylinder, graduated, 25-mL	1	each	50840
Cylinder, graduated, 250-mL	1	each	50846
Distillation Apparatus, arsenic accessories	1	set	2265400
Distillation Apparatus, general purpose accessories	1	set	2265300
Flask, volumetric, Class A, 1000-mL, with glass stopper	1	each	1457453
Flask, volumetric, Class A, 200-mL	1	each	1457445
Flask, volumetric, Class A, 500-mL	6	each	1457449
Pipet filler, safety bulb	1	each	1465100
Pipet, serological, 5-mL	1	each	53237
Pipet, volumetric, Class A, 1.00-mL	1	each	1451535
Pipet, volumetric, Class A, 2.00-mL	1	each	1451536
Pipet, volumetric, Class A, 4.00-mL	1	each	1451504
Pipet, volumetric, Class A, 6.00-mL	1	each	1451506
Pipet, volumetric, Class A, 8.00-mL	1	each	1451508
Pipet, volumetric, Class A, 10.00-mL	1	each	1451538
Select one based on available voltage:			
Distillation heater and support for apparatus set, 115 VAC option	1	each	2274400
Distillation heater and support for apparatus set, 230 VAC option	1	each	2274402

Optional reagents and apparatus

Description	Unit	Item no.
Mixing cylinder, graduated, 25-mL	each	189640
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032
Gloves, chemical resistant, size 9–9.5	pair	2410104[1]

[1] Other sizes available

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03/2014, Edition 8