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 (distillation required)

[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

How to use instrument-specific information

The *Instrument-specific information* table displays requirements that may vary between instruments. To use this table, select an instrument then read across to find the corresponding DR 3800, DR 2800, DR 2700information required to perform this test.

Table 43 Instrument-specific information

Instrument	Sample cell	Cell orientation
DR 6000	2612602	Fill line faces right
DR 5000	2612602	Fill line faces user
DR 3900	2612602	Fill line faces user
DR 3800, DR 2800, DR 2700	2612602	Fill line faces right

Before starting the test:

Create a user-entered program for arsenic. See step 1 and *User programming*.
Prepare the arsenic absorber solution as directed in *Reagent preparation*.
Perform a user-entered calibration for each new lot of arsenic absorber solution. See the *Calibration* section. Some variations of the calibration procedure are possible.
In bright light conditions (e.g. direct sunlight) it may be necessary to close the cell compartment with the protective cover during measurements.
Do not use the Pour-Thru Cell with this test.
The arsenic absorber in this test is a silver solution in pyridine. Both silver (D011) and pyridine (D038) are regulated by the Federal RCRA as hazardous waste. In addition, the cotton ball soaked in lead acetate (D008) solution is a hazardous waste. These materials should not be poured down the drain. Refer to a current MSDS sheet for proper disposal.

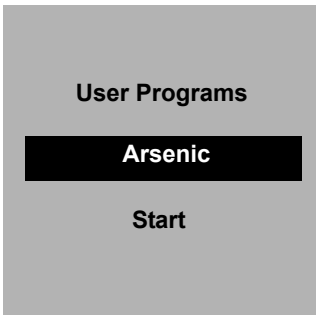
Collect the following items :

Description	Quantity
Apparatus (see *Required apparatus*)	—
Arsenic Standard Solution, 1000-mg/L As	varies
Hydrochloric Acid, ACS	25 mL
Lead Acetate Solution, 10%	1 mL
Potassium Iodide Solution, 20%	3 mL
Pyridine, ACS	50 mL
Sample Cells (see *Instrument-specific information*)	2
Silver Diethyldithiocarbamate	1 g
Stannous Chloride Solution	1 mL
Water, deionized	varies
Zinc, 20-mesh, ACS	6 g

See *Consumables and replacement items* for reorder information.

Silver Diethyldithiocarbamate

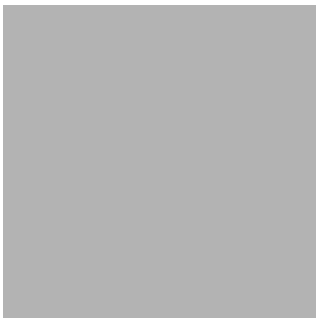
1. Perform the *User programming* procedure. Make note of the program number.



2. To run the test, press **USER PROGRAMS**.

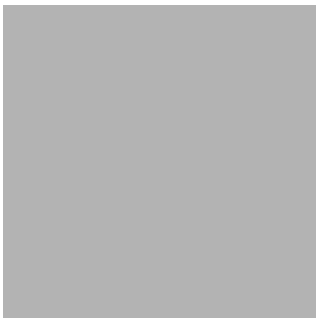
Select the test.

Insert an adapter if required (Table 1). Refer to the user manual for orientation.



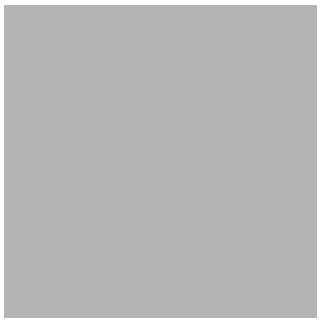
3. Prepare the distillation apparatus for arsenic recovery. See the *Distillation Manual* for assembly instructions. **Do not connect to the aspirator.**

Place the distillation apparatus under a fume hood to vent toxic fumes.



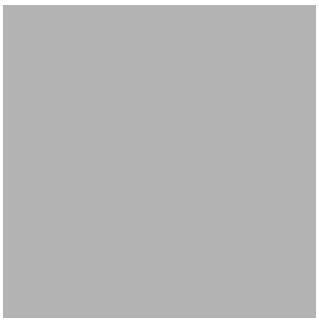
4. Dampen a cotton ball with 10% Lead Acetate Solution. Insert it in the gas scrubber. Be certain that the cotton seals against the glass.

Silver Diethyldithiocarbamate

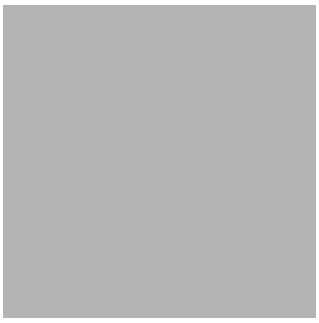


5. Using a graduated cylinder, pour 25-mL of prepared arsenic absorber solution (*Reagent preparation*) into the cylinder/gas bubbler assembly.

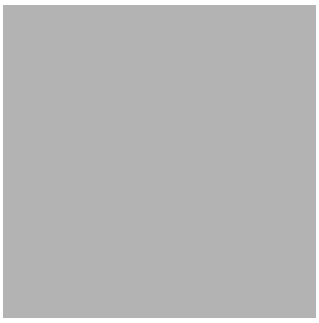
Attach it to the distillation apparatus.



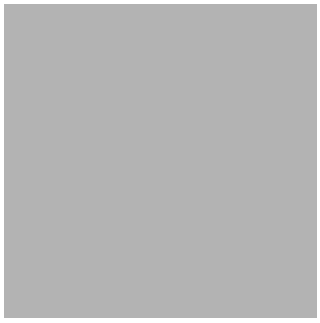
6. Using a graduated cylinder, pour 250 mL of sample into the distillation flask.



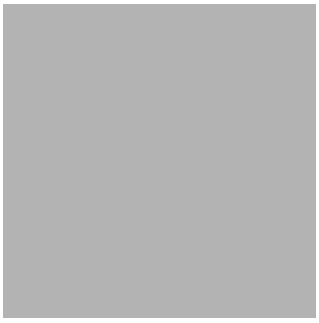
7. Turn on the power switch. Set the stir control to 5. Set the heat control to 0.



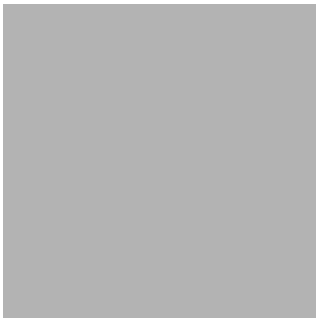
8. Using a graduated cylinder, add 25 mL of Hydrochloric Acid, ACS, to the distillation flask.



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

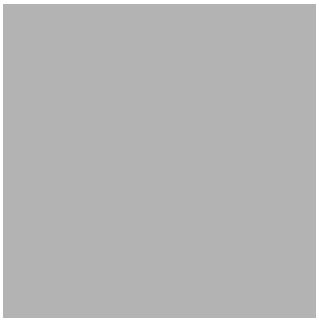


10. Use a serological pipet to add 3 mL of Potassium Iodide Solution to the flask. Cap.

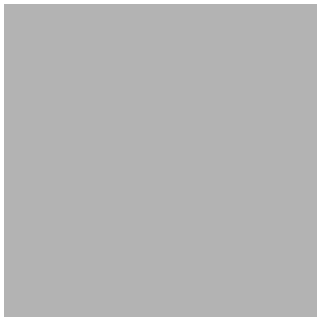


11. Start the instrument timer.

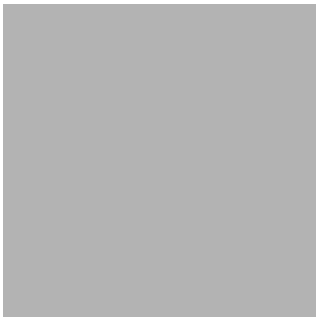
A 15-minute reaction period will begin.



12. When the timer expires, weigh and add 6.0 g of 20-mesh zinc to the flask. **Cap immediately.**

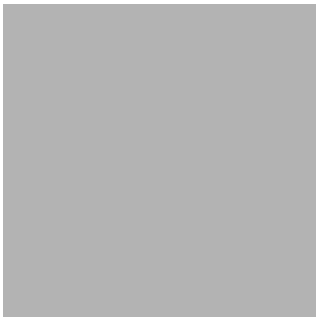


13. Set the heat control to 3.

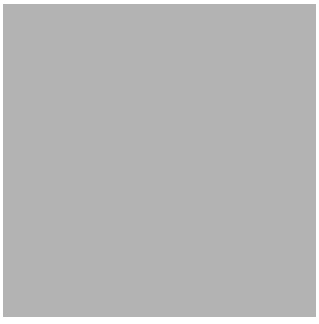


14. Start the instrument timer.

A second 15-minute reaction period will begin.



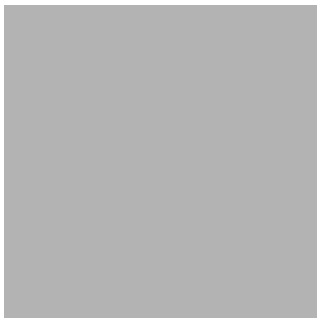
15. When the timer expires, set the heat control to 1.



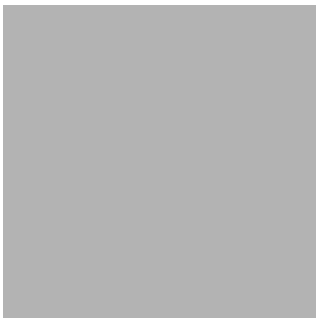
16. Start the instrument timer.

A third 15-minute reaction period will begin.

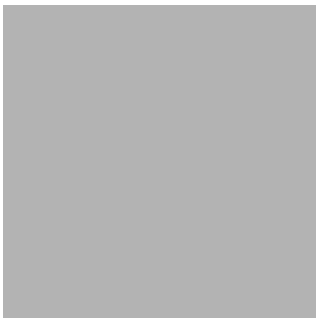
Silver Diethyldithiocarbamate



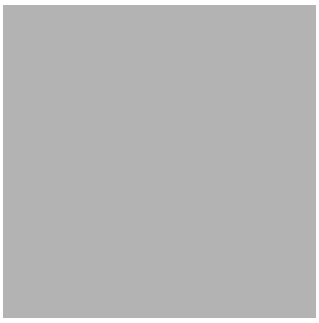
17. When the timer expires, turn off the heater. Remove the cylinder/gas bubbler assembly as a unit.



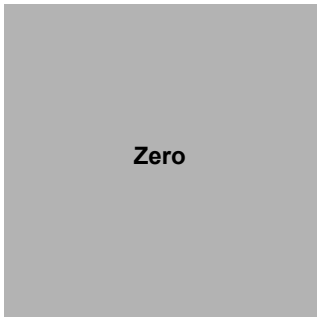
18. Rinse the gas bubbler by moving it up and down in the arsenic absorber solution.



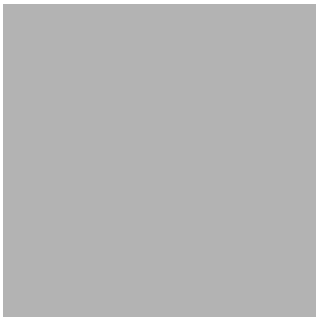
19. Blank Preparation: Fill a dry, 10-mL sample cell with untreated arsenic absorber solution. Stopper.



20. Wipe the blank and insert it into the cell holder.

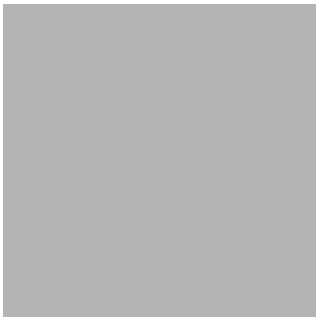


21. ZERO the instrument. The display will show the intercept as calculated from the user-entered calibration curve. This will probably be a non-zero intercept.



22. Prepared Sample: Pour the reacted arsenic absorber sample into a sample cell.

Close the sample cell.



23. Wipe the prepared sample and insert it into the cell holder.

READ the results.

Interferences

Table 44 Interfering substances

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

Sample collection, preservation and storage

Collect samples in acid washed glass or plastic bottles. Adjust the pH to 2 or less with sulfuric acid (about 2 mL per liter)*. Preserved samples may be stored up to six months at room temperature. Correct the test result for volume additions.

* See *Optional reagents and apparatus*.

Reagent preparation

Prepare the arsenic absorber solution as follows:

1. Weigh 1.00 g of silver diethyldithiocarbamate on an analytical balance.
2. Transfer the powder to a 200-mL volumetric flask. Dilute to volume with pyridine. **Use pyridine only in a fume hood and wear chemical resistant gloves.** Read the MSDS before using pyridine.
3. Mix well to dissolve. Store the reagent, tightly sealed, in an amber bottle. The reagent is stable for one month if stored in this manner. Larger volumes of reagent can be prepared if the reagent is used within one month.

Calibration

Standard preparation

Perform a new calibration for each lot of arsenic absorber solution.

1. Prepare a 10.0-mg/L arsenic working standard by pipetting 10.0 mL of Arsenic Standard Solution, 1000 mg/L As into a 1000-mL volumetric flask.
2. Dilute to volume with deionized water.
3. Into three different 500-mL volumetric flasks, pipet 1.0, 2.0, and 10.0 mL of the 10.0 mg/L As stock solution using Class A glassware.
4. Dilute to the mark with deionized water and mix thoroughly. These standards have concentrations of 0.02, 0.04 and 0.20 mg/L As.

   Note: *Distill standards before making the calibration curve.*

User programming

1. Press **USER PROGRAMS** on the main menu.
2. Press **PROGRAM OPTIONS** and **NEW**. Key any available program number (950–999) to use for arsenic testing. Press **OK**.
3. Fill in the appropriate fields using the touch screen when the field is highlighted. Use the alphanumeric keys to name the User Program **ARSENIC**. Press **NEXT** to move to the next screen. Set up the rest of the parameters as follows:

   - Program Type: Single Wavelength
   - Units: mg/L
   - Concentration Resolution: 0.001
   - Chemical Form: As
   - Wavelength: 520 nm
   - Calibration: Read Standards

4. After entering Read Standards, press **NEXT>EXIT**. Fill in the appropriate fields for each of the following. Use the touch screen to activate the parameter and press **EDIT** to enter the data entry screen. Set up the rest of the parameters as follows:

   - Timer1: 15 minutes
   - Timer2: 15 minutes
   - Timer3: 15 minutes
   - Upper Limit: 0.220 mg/L
   - Lower Limit: –0.020 mg/L

5. Press **CALIBRATION: C = A + BA**. Press **EDIT**.

6. The Read Standards will be indicated. Enter the standard concentration values to be used to perform the calibration: 0.00, 0.02, 0.04, and 0.20. To enter the concentration values press **+** and enter the value followed by **OK** for each concentration value.
7. After the values are entered, press the **UP** arrow four times to move the cursor to the 0.00 concentration line.
8. Insert the 25-mL sample cell containing only unreacted arsenic absorber solution into the cell holder. Press **ZERO.**
9. Press the **DOWN** arrow once to move to the next concentration. Insert the prepared sample in the cell holder. Press **READ** to accept the absorbance value. Repeat steps for each standard.

   Note: *Standards must be distilled before absorbance values are measured.*

10. Press **GRAPH**. Make sure **FORCE ZERO** is off.
11. If the graph is acceptable press **DONE>EXIT**.
12. "Store Program?" will appear on the display. Press **YES**.

The program is ready for use.

Some variations of the calibration procedure are possible. See the user manual for details.

Summary of method

Arsenic is reduced to arsine gas by a mixture of zinc, stannous chloride, potassium iodide, and hydrochloric acid in a specially equipped distillation apparatus. The arsine is passed through a scrubber containing cotton saturated with lead acetate for sulfide removal, and then into an absorber tube containing silver diethyldithiocarbamate in pyridine. The arsenic reacts to form a red complex which is read colorimetrically. This procedure requires a manual calibration. Test results are measured at 520 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Catalog number
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
Silver Diethyldithiocarbamate	1 g	25 g	1447624
Stannous Chloride Solution	1 mL	100 mL	1456942
Water, deionized	varies	4 liters	27256
Zinc, 20-mesh, ACS	6 g	454 g	79501

Required apparatus

Description	Quantity	Unit	Catalog number
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

Required apparatus (continued)

Description	Quantity	Unit	Catalog number
Bottle, amber, 237-mL, glass	1	6/pkg	714441
Cap, polypropylene, for amber bottle	1	6/pkg	2166706
Cylinder, graduated, 25-mL	2	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	2	each	53237
Pipet, volumetric, Class A, 1.00-mL	2	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 Apparatus Heater, 115 VAC, 60 Hz	1	each	2274400
Distillation Apparatus Heater, 230 VAC, 50 Hz	1	each	2274402

Optional reagents and apparatus

Description	Unit	Catalog number
Cylinder, mixing, 25-mL	each	189640
Sulfuric Acid, 1.00 N	100 mL	127032
Gloves, chemical resistant, size 9[1]	pair	2410104

[1] Other sizes available.

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