Molybdenum, HR

DOC316.53.01061

Mercaptoacetic Acid Method[1] **Method 8036**

0.2 to 40.0 mg/L Mo (HR) **Powder Pillows or AccuVac® Ampuls**

Scope and application: For water, wastewater, boiler and cooling waters.

[1] Adapted from Analytical Chemistry, 25(9) 1363 (1953).

Test preparation

Instrument-specific information

Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests. Table 2 shows sample cell and adapter requirements for AccuVac Ampul tests. The tables also show all of the instruments that have the program for this test.

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

Table 1 Instrument-specific information for reagent addition

Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906

Table 2 Instrument-specific information for AccuVac Ampuls

Instrument	Adapter	Sample cell
DR 6000 DR 5000 DR 900	—	2427606
DR 3900	LZV846 (A)	
DR 1900	9609900 or 9609800 (C)	
DR 3800 DR 2800 DR 2700	LZV584 (C)	2122800

Before starting

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.
Filter samples that are turbid with filter paper and a funnel.
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

Powder pillows

Description	Quantity
MolyVer® 1 Molybdenum Reagent Powder Pillow, 10-mL	1
MolyVer® 2 Molybdenum Reagent Powder Pillow, 10-mL	1
MolyVer® 3 Molybdenum Reagent Powder Pillow, 10-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to Consumables and replacement items on page 6 for order information.

AccuVac Ampuls

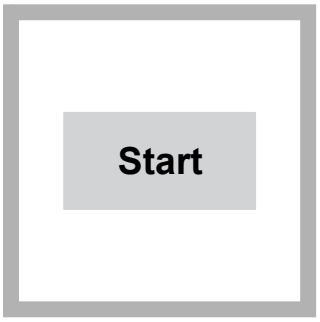
Description	Quantity
CDTA Solution, 0.4 M	4 drops
MolyVer® 6 Reagent AccuVac® Ampul	1
Beaker, 50 mL	1
Stopper for 18-mm tubes and AccuVac Ampuls	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1

Refer to Consumables and replacement items on page 6 for order information.

Sample collection and storage

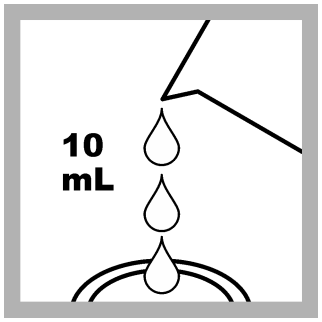
- Collect samples in clean glass or plastic bottles that have been cleaned with 6 N (1:1) hydrochloric acid and rinsed with deionized water.
- To preserve samples for later analysis, adjust the sample pH to less than 2 with concentrated nitric acid (approximately 2 mL per liter). No acid addition is 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 7 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

Powder pillow procedure

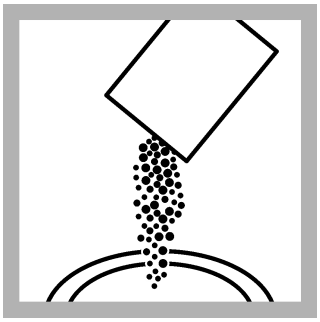


1. Start program **320 Molybdenum HR**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

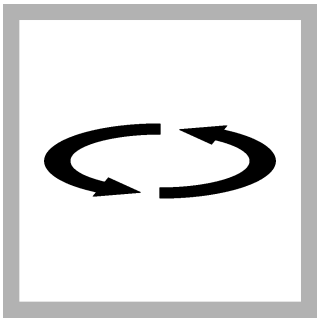
Note: *Although the program name can be different between instruments, the program number does not change.*



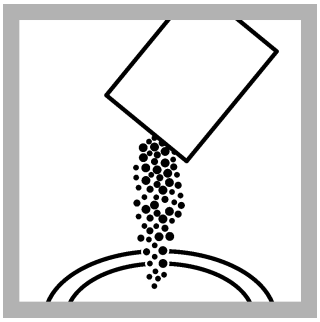
2. Prepare the sample: Fill a sample cell with 10 mL of sample.



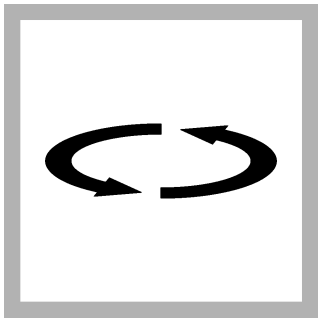
3. Add the contents of one MolyVer 1 reagent powder pillow.



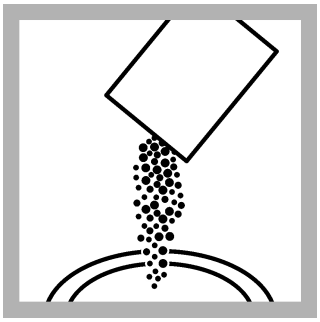
4. Swirl to mix.



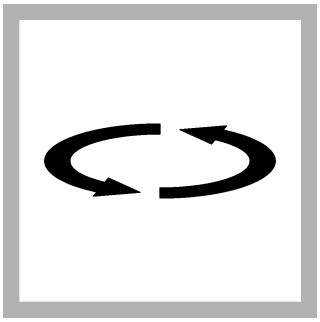
5. Add the contents of one MolyVer 2 reagent powder pillow.



6. Swirl to mix.

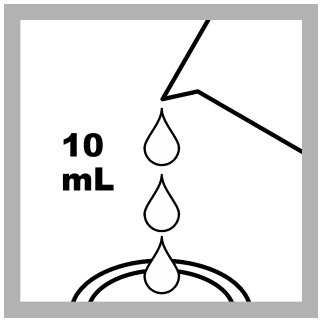


7. Add the contents of one MolyVer 3 reagent powder pillow.

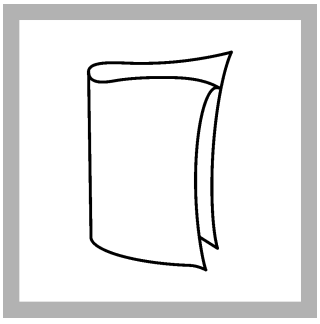


8. Swirl to mix.
A yellow color will show if molybdate molybdenum is present.

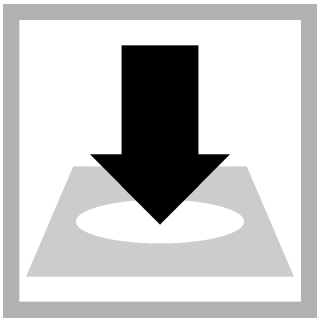
9. Start the instrument timer. A 5-minute reaction time starts.



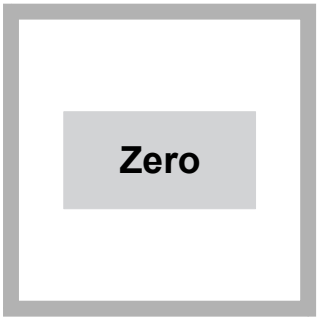
10. Prepare the blank: Fill a second sample cell with 10 mL of sample.



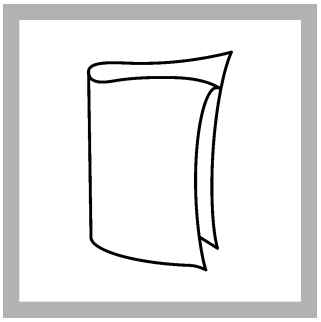
11. When the timer expires, clean the blank sample cell.



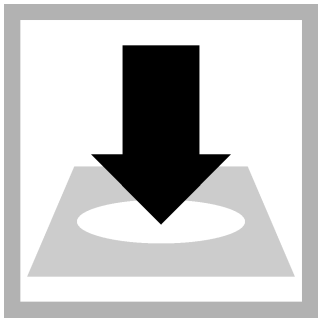
12. Insert the blank into the cell holder.



13. Push **ZERO**. The display shows 0.0 mg/L Mo^{6+}.



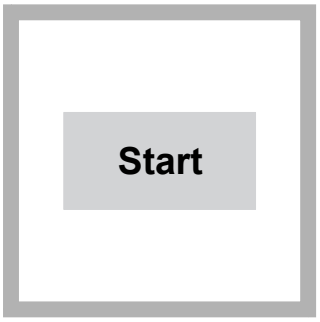
14. Clean the prepared sample cell.



15. Insert the prepared sample into the cell holder.

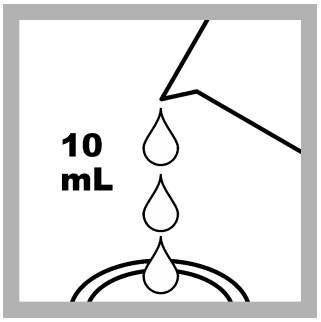
16. Push **READ**. Results show in mg/L Mo^{6+}.

AccuVac Ampul procedure

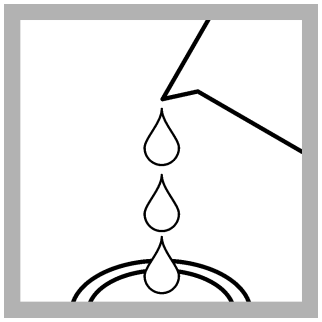


1. Start program **322 Molybdenum HR**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

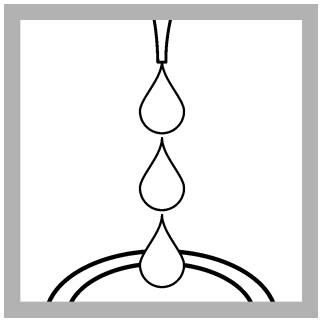
Note: *Although the program name can be different between instruments, the program number does not change.*



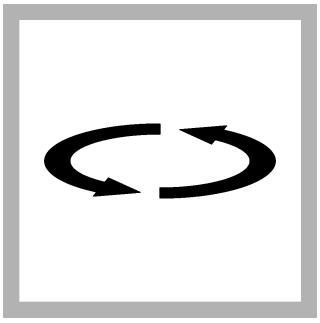
2. Prepare the blank: Fill the sample cell with 10 mL of sample.



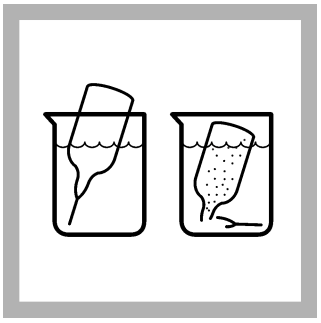
3. Prepare the sample: Collect 40 mL of sample in a 50-mL beaker.



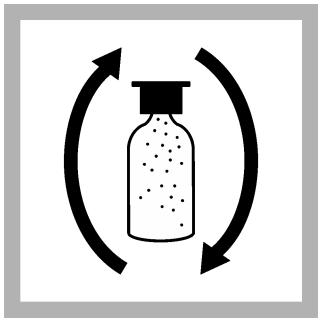
4. Add Add four drops of 0.4 M CDTA Standard Solution to the sample in the beaker .



5. Swirl to mix.

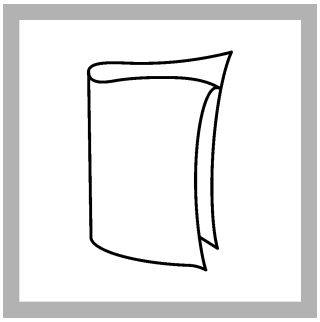


6. Fill an AccuVac Ampul with the treated sample. Keep the tip immersed while the Ampul fills completely.

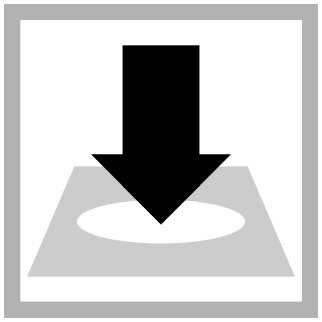


7. Quickly invert the AccuVac Ampul several times to mix.
A yellow color will show if molybdate molybdenum is present.

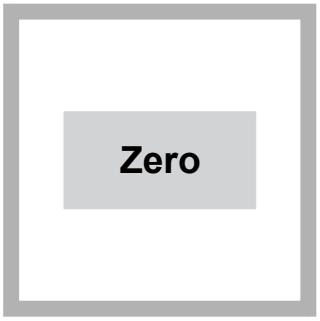
8. Start the instrument timer. A 5-minute reaction time starts.



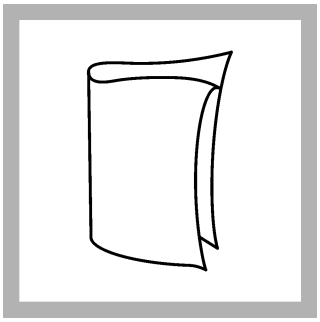
9. When the timer expires, clean the blank sample cell.



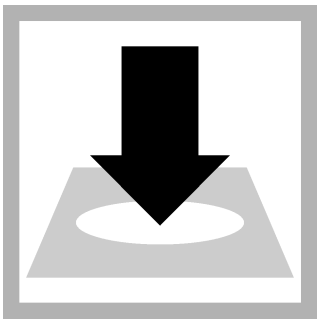
10. Insert the blank into the cell holder.



11. Push **ZERO**. The display shows 0.0 mg/L Mo^{6+}.



12. Clean the AccuVac Ampul.



13. Insert the prepared sample AccuVac Ampul into the cell holder.

14. Push **READ**. Results show in mg/L Mo^{6+}.

Interferences

Interfering substance	Interference level
Aluminum	More than 50 mg/L
Chromium	More than 1000 mg/L
Copper	Samples that contain 10 mg/L or more of copper will show a positive interference that increases over time. Read these samples as soon as possible after the 5-minute reaction period.
Iron	More than 50 mg/L
Nickel	More than 50 mg/L
Nitrite	Add one Sulfamic Acid Powder Pillow to the sample to remove interference from up to 2000 mg/L as NO_2^-.
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment of the sample by the reagents. Sample pre-treatment may be necessary.

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Molybdenum Standard Solution, 1000-mg/L Mo^{6+}
- Mixing cylinders, 50-mL (100-mL for AccuVac Ampuls) (3)
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
2. Go to the Standard Additions option in the instrument menu.
3. Select the values for standard concentration, sample volume and spike volumes.
4. Open the standard solution.
5. Prepare three spiked samples: use the TenSette pipet to add 0.2 mL, 0.4 mL and 0.6 mL of the standard solution, respectively, to three 30-mL portions of fresh sample. Mix well.

   Note: *For AccuVac® Ampuls, add 0.4 mL, 0.8 mL and 1.2 mL of the standard solution to three 60-mL portions of fresh sample.*
6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
7. Select **Graph** to compare the expected results to the actual results.

   Note: *If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.*

Standard solution method

Use the standard solution method to validate the test procedure, the reagents and the instrument.

Items to collect:

- 10 mg/L Molybdenum (as Mo^{6+}) Standard Solution

1. Use the test procedure to measure the concentration of the standard solution.
2. Compare the expected result to the actual result.

   Note: *The factory calibration can be adjusted slightly with the standard adjust option so that the instrument shows the expected value of the standard solution. The adjusted calibration is then used for all test results. This adjustment can increase the test accuracy when there are slight variations in the reagents or instruments.*

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change
320	10 mg/L Mo^{6+}	9.7–10.3 mg/L Mo^{6+}	0.2 mg/L Mo^{6+}
322	10 mg/L Mo^{6+}	9.7–10.3 mg/L Mo^{6+}	0.2 mg/L Mo^{6+}

Summary of method

MolyVer 1 and 2 Reagents are added to buffer and condition the sample. MolyVer 3 provides the mercaptoacetic acid which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration. The measurement wavelength is 420 nm.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Molybdenum Reagent Set, 10-mL, includes:	—	100 tests	2604100
MolyVer® 1 Molybdenum Reagent Powder Pillow, 10-mL	1	100/pkg	2604299
MolyVer® 2 Molybdenum Reagent Powder Pillow, 10-mL	1	100/pkg	2604399

Consumables and replacement items (continued)

Description	Quantity/test	Unit	Item no.
MolyVer® 3 Molybdenum Reagent Powder Pillow, 10-mL	1	100/pkg	2604499
OR			
MolyVer® 6 Reagent AccuVac® Ampul	1	25/pkg	2522025
CDTA Solution, 0.4 M	4 drops	15 mL SCDB	2615436

Required apparatus

Description	Quantity/test	Unit	Item no.
AccuVac Snapper	1	each	2405200
Beaker, 50-mL	1	each	50041H
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards

Description	Unit	Item no.
Molybdenum Standard Solution, 10-mg/L as Mo	100 mL	1418742
Molybdenum Standard Solution, 1000-mg/L as Mo	100 mL	1418642
Water, deionized	4 L	27256

Optional reagents and apparatus

Description	Unit	Item no.
Beaker, 50-mL	each	50041H
Mixing cylinder, graduated, 50-mL	each	189641
Filter paper, 2–3-micron, pleated, 12.5-cm	100/pkg	189457
Funnel, poly, 65-mm	each	108367
Pipet, TenSette®, 0.1–1.0 mL	each	1970001
Pipet tips for TenSette® Pipet, 0.1–1.0 mL	50/pkg	2185696
Sulfamic Acid Powder Pillows	100/pkg	105599

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