Molybdenum, HR

DOC316.53.01061

Mercaptoacetic Acid Method[1]

Method 8036

0.2 to 40.0 mg/L Mo (HR)

Powder Pillows

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 all of the instruments that have the 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
DR6000 DR3800 DR2800 DR2700 DR1900	The fill line is to the right.	2495402 
DR5000 DR3900	The fill line is toward the user.	
DR900	The orientation mark is toward the user.	2401906 

Before starting

Install the instrument cap on the DR900 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

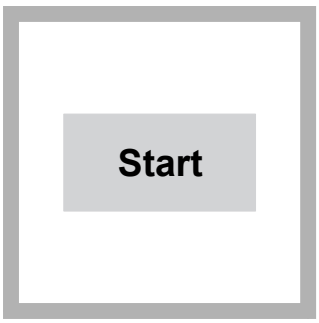
Description	Quantity
MolyVer® 1 Molybdenum Reagent Powder Pillow, 10 mL	1
MolyVer® 2 Molybdenum Reagent Powder Pillow, 10 mL	1
MolyVer® 3 Molybdenum Reagent Powder Pillow, 10 mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on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

Refer to Consumables and replacement items on page 4 for order information.

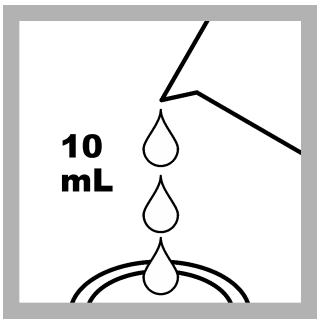
Sample collection and storage

- 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.

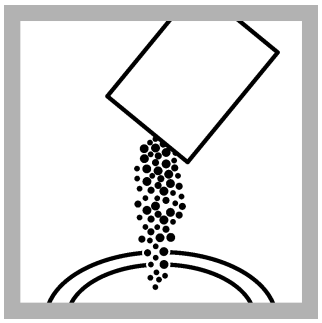
Test procedure



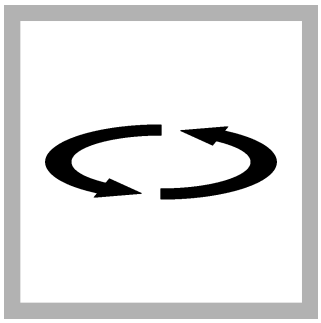
1. Start program **320 Molybdenum HR**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.



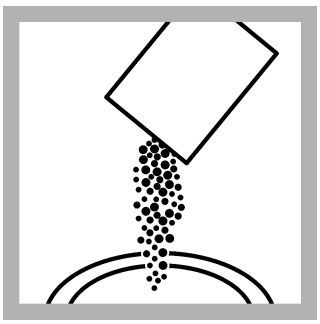
2. Prepare the sample: Fill a sample cell with 10 mL of sample.



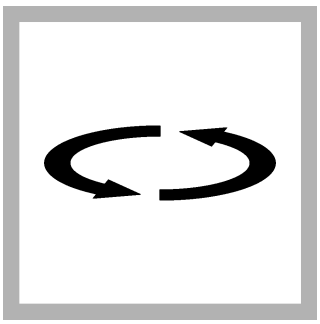
3. Add the contents of one MolyVer 1 reagent powder pillow.



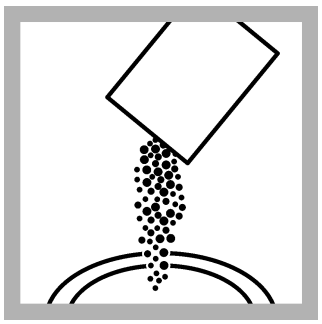
4. Swirl to mix.



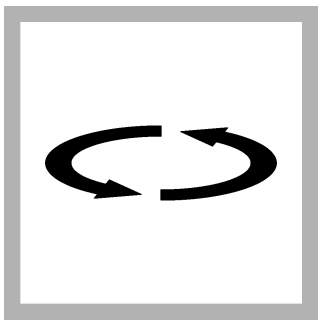
5. Add the contents of one MolyVer 2 reagent powder pillow.



6. Swirl to mix.

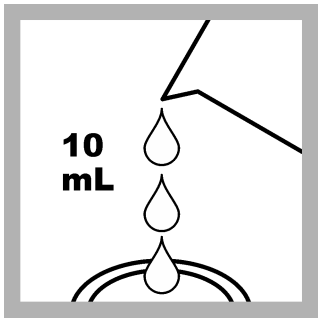


7. Add the contents of one MolyVer 3 reagent powder pillow.

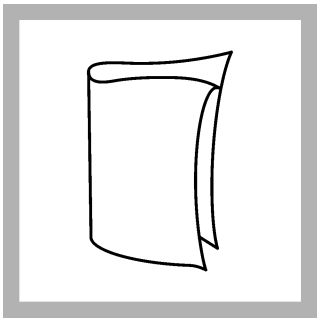


8. Swirl to mix.
A yellow color will show if molybdate molybdenum is present.

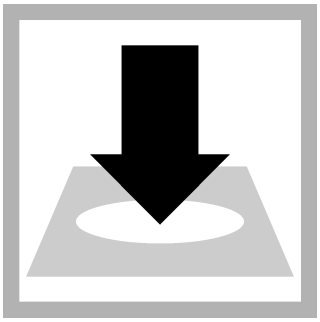
9. Start the instrument timer. A 5-minute reaction time starts.



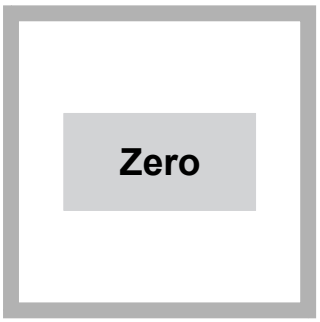
10. Prepare the blank: Fill a second sample cell with 10 mL of sample.



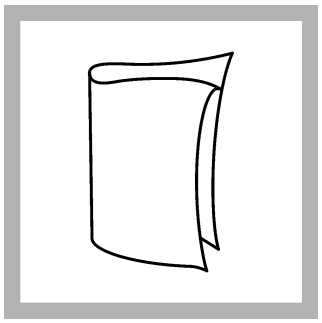
11. When the timer expires, clean the blank sample cell.



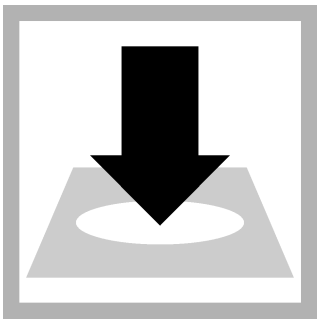
12. Insert the blank into the cell holder.



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+}.

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 pretreatment 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, 30 mL (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.
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 calibration 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 small 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+}

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
MolyVer® 3 Molybdenum Reagent Powder Pillow, 10 mL	1	100/pkg	2604499

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.
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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