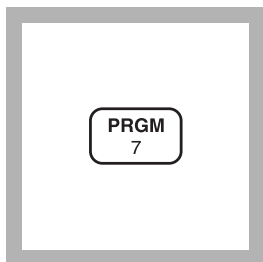


MOLYBDENUM, MOLYBDATE, High Range (0 to 40.0 mg/L)**Mercaptoacetic Acid Method*****For water and wastewater****Using Powder Pillows**

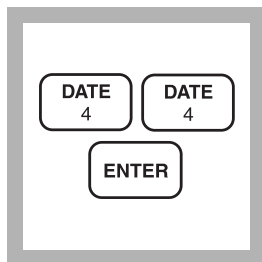
1. Enter the stored program number for high range molybdenum-powder pillows

Press: **PRGM**

The display will show:

PRGM ?

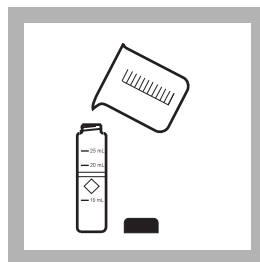
Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).



2. Press: **44 ENTER**

The display will show **mg/L, Mo6** and the **ZERO** icon.

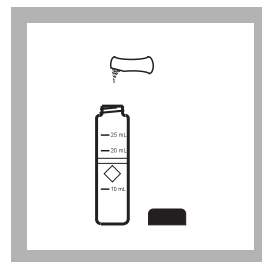
*Note: For alternate form (MoO₄), press the **CONC** key.*



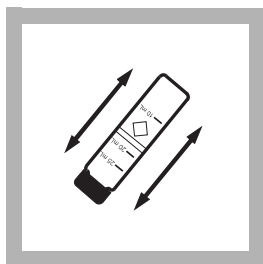
3. Fill a sample cell with 10 mL of sample.

Note: Filter turbid samples.

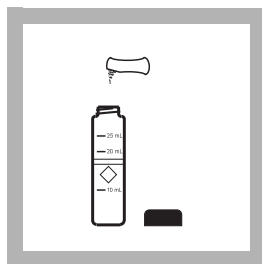
Note: Adjust pH of stored samples before analysis.



4. Add the contents of one MolyVer 1 Reagent Powder Pillow. Cap the cell and invert several times to mix.

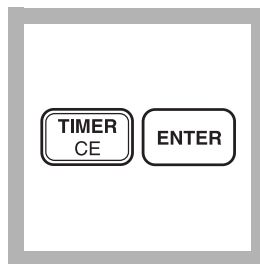


5. Add the contents of one MolyVer 2 Reagent Powder Pillow. Cap the cell and invert several times to mix.



6. Add the contents of one MolyVer 3 Reagent Powder Pillow. Cap the cell and invert several times to mix. This is the prepared sample.

Note: Accuracy is not affected by undissolved powder.

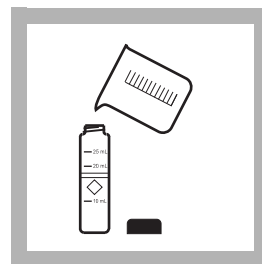


7. Press:

TIMER ENTER

A five-minute reaction period will begin.

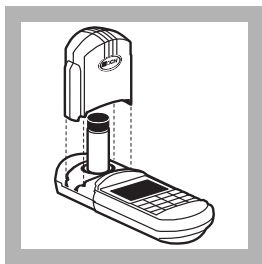
Note: Molybdenum will cause a yellow color to form.



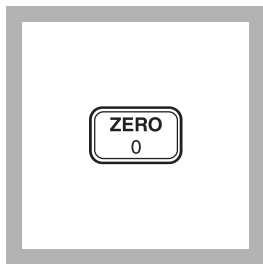
8. After the timer beeps, fill a second sample cell with 10 mL of sample (the blank).

* Adapted from Analytical Chemistry, 25(9) 1363 (1953).

MOLYBDENUM, MOLYBDATE, HR, continued



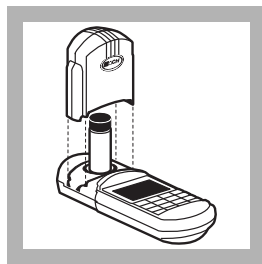
9. Insert the blank into the cell holder. Tightly cover the sample cell with the instrument cap.



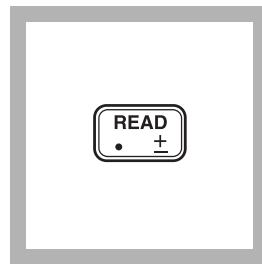
10. Press: **ZERO**
The cursor will move to the right, then the display will show:

0.0 mg/L Mo6

Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.



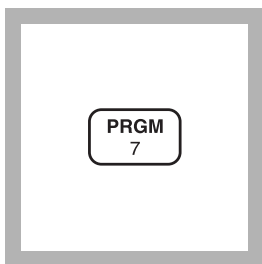
11. Place the prepared sample into the cell holder. Tightly cover the sample cell with the instrument cap.



12. Press: **READ**
The cursor will move to the right, then the result in mg/L molybdenum (or alternate form) will be displayed.

Note: Use of the Standard Adjust feature with each new lot of reagents is highly recommended. See Accuracy Check.

Using AccuVac Ampuls



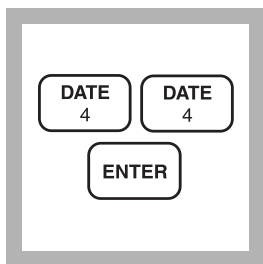
1. Enter the stored program number for high range molybdenum using AccuVac Ampuls.

Press: **PRGM**

The display will show:

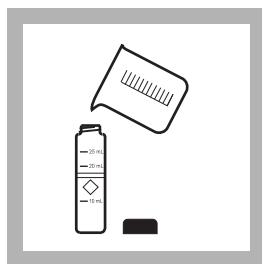
PRGM ?

Note: For most accurate results, perform a Reagent Blank Correction using deionized water (see Section 1).



2. Press: **44 ENTER**
The display will show **mg/L, Mo6** and the **ZERO** icon.

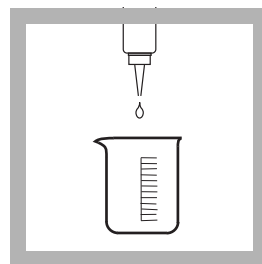
*Note: For alternate form (MoO₄), press the **CONC** key.*



3. Fill a sample cell with at least 10 mL of sample (the blank). Collect at least 40 mL of sample in a 50-mL beaker.

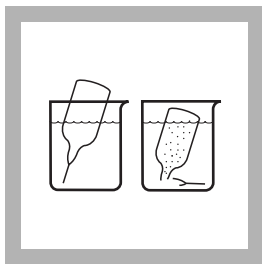
Note: Filter turbid samples.

Note: Adjust the pH of stored samples before analysis.



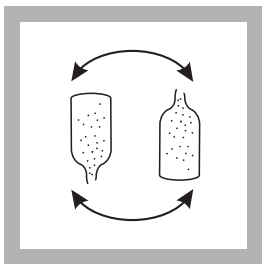
4. Add 4 drops of 0.4 M CDTA Solution to the beaker. Swirl to mix.

MOLYBDENUM, MOLYBDATE, HR, continued



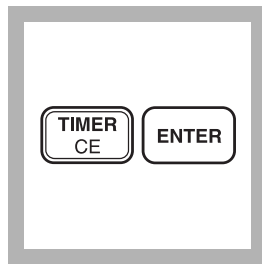
5. Fill a MolyVer 6 Reagent AccuVac Ampul with sample.

Note: Keep the tip immersed while the ampul fills.



6. Invert the ampul repeatedly to mix. Wipe off any liquid or fingerprints.

Note: Undissolved reagent will not affect the result.

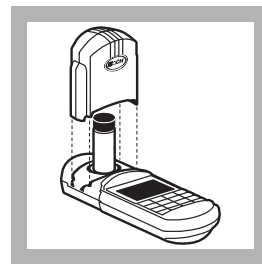


7. Press:

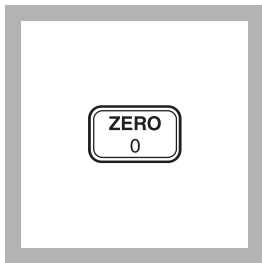
TIMER ENTER

A five-minute reaction period will begin.

Note: If molybdenum is present a yellow color will develop.



8. When the timer beeps, place the blank into the cell holder. Tightly cover the sample cell with the instrument cap.

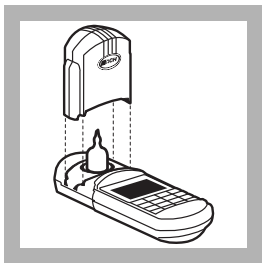


9. Press: **ZERO**

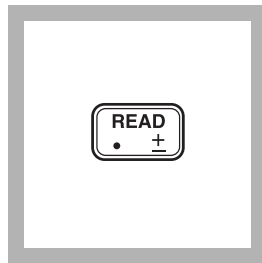
The cursor will move to the right, then the display will show:

0.0 mg/L Mo6

Note: If Reagent Blank Correction is on, the display may flash "limit". See Section 1.



10. Place the AccuVac Ampul in the cell holder. Tightly cover the ampul with the instrument cap.



11. Press: **READ**

The cursor will move to the right, then the result in mg/L molybdenum will be displayed.

Note: Use of the Standard Adjust feature with each new lot of reagent is highly recommended. See Accuracy Check.

Sampling and Storage

Collect samples in clean plastic bottles. Adjust the pH to 2 or less with nitric acid (about 2 mL per liter). Preserved samples can be stored up to 6 months at room temperature. Adjust the pH to 7 with 5.0 N sodium hydroxide before analysis. Correct the test result for volume additions; see *Volume Additions (Section 1)* for more information.

Accuracy Check

Standard Additions Method

- a) Fill three 25-mL graduated mixing cylinders with 25 mL of sample.
- b) Snap the neck off a Molybdenum Voluette Ampule Standard Solution, 500 mg/L Mo⁶⁺.
- c) Use the TenSette Pipet to add 0.1, 0.2 and 0.3 mL of standard, respectively, to the three mixing cylinders. Stopper each and mix thoroughly.
- d) For analysis with AccuVac Ampuls, transfer solutions to dry, clean 50-mL beakers. For analysis with powder pillows, transfer only 10 mL of solution to the sample cells.
- e) Analyze each standard addition sample as described in the procedure. The molybdenum concentration reading should increase 2.0 mg/L for each 0.1 mL of standard added.
- f) If these increases do not occur, see *Standard Additions in Section 1* for troubleshooting information.

Standard Solution Method

To assure the accuracy of the test, use a Molybdenum Standard Solution, 10.0 mg/L Mo⁶⁺. Follow the procedure for powder pillows or AccuVac Ampuls. Results should be between 9.0 and 11.0 mg/L Mo⁶⁺.

Standard Adjust

To adjust the calibration curve using the reading obtained with the 10.0-mg/L standard solution, press the **SETUP** key and scroll (using the arrow keys) to the STD setup option. Press **ENTER** to activate the standard adjust option. Then enter **10.0** to edit the standard concentration to match that of the standard used. Press **ENTER** to complete the adjustment. See *Section 1, Standard Curve Adjustment* for more information.

Method Performance

Precision

In a single laboratory using a standard solution of 20.0 mg/L Mo⁶⁺ and two representative lots of powder pillows with the instrument, a single operator obtained a standard deviation of ±0.3 mg/L Mo⁶⁺.

MOLYBDENUM, MOLYBDATE, HR, continued

In a single laboratory using a standard solution of 20.0 mg/L Mo⁶⁺ and two representative lots of AccuVac Ampuls with the instrument, a single operator obtained a standard deviation of ±0.1 mg/L Mo⁶⁺.

Estimated Detection Limit

The estimated detection limit for program 44 is 0.2 mg/L Mo⁶⁺. For more information on the estimated detection limit, see *Section 1*.

Interferences

Interfering Substance	Interference Levels and Treatments
Aluminum	Greater than 50 mg/L
Chromium	Greater than 1000 mg/L
Copper	Samples containing 10 mg/L copper or more will exhibit an increasing positive interference upon standing. Read these samples as soon as possible after the five-minute reaction period is complete.
Iron	Greater than 50 mg/L
Nickel	Greater than 50 mg/L
Nitrite	Interference from up to 2000 mg/L as NO ₂ ⁻ can be eliminated by adding one Sulfamic Acid Powder Pillow to the sample.
Highly buffered samples or extreme sample pH	May exceed the buffering capacity of the reagents and require sample pretreatment; see <i>Section 1, pH Interferences</i> .

Summary of Method

Powder Pillows

MolyVer 1 and 2 Reagents are added to buffer and condition the sample. MolyVer 1 contains a buffer to control the pH in addition to a chelating agent to mask interferences. MolyVer 3 provides the mercaptoacetic acid, which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration.

AccuVac Ampuls

The CDTA Solution masks metal interferences. The MolyVer 6 reagent provides the mercaptoacetic acid, which reacts with molybdate molybdenum to form a yellow color proportional to the molybdenum concentration.

MOLYBDENUM, MOLYBDATE, HR, continued

REQUIRED REAGENTS (for Powder Pillows)

	Cat. No.
Molybdenum Reagent Set, 10 mL (100 tests)	26041-00
Includes: (1) 26042-99, (1) 26043-99, (1) 26044-99	

Description	Quantity Required		Cat. No.
	Per Test	Unit	
MolyVer 1 Reagent Powder Pillows	1 pillow	100/pkg	26042-99
MolyVer 2 Reagent Powder Pillows	1 pillow	100/pkg	26043-99
MolyVer 3 Reagent Powder Pillows	1 pillow	100/pkg	26044-99

REQUIRED REAGENTS (for AccuVac Ampuls)

MolyVer 6 Molybdenum AccuVac Reagent Set (25 tests)			25220-98
Includes: (1) 25220-25, (1) 26154-36			
CDTA Solution 0.4M	4 drops	15 mL SCDB	26154-36
MolyVer 6 Reagent AccuVac Ampuls	1 ampul	25/pkg	25220-25

REQUIRED APPARATUS (for Powder Pillows)

Sample Cell, 10-20-25 mL, w/cap	2	6/pkg	24019-06
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REQUIRED APPARATUS (for AccuVac Ampuls)

Beaker, 50 mL	2	each	500-41H
Sample Cell, 10-20-25 mL, w/cap	1	6/pkg	24019-06

OPTIONAL REAGENTS

Molybdenum Standard Solution, 10 mg/L Mo ⁶⁺	100 mL	14187-42
Molybdenum Standard Solution, Voluette Ampule, 500 mg/L Mo ⁶⁺ , 10 mL	16/pkg	14265-10
Nitric Acid, ACS	500 mL	152-49
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	2450-32
Sulfamic Acid Powder Pillows	100/pkg	1055-99
Water, deionized	4 L	272-56

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Ampule Breaker Kit	each	21968-00
Cylinder, graduated, mixing, 25 mL	each	20886-40
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, Erlenmeyer, 250 mL	each	505-46
Funnel, poly, 65 mm	each	1083-67
Pipet, serological, 5 mL	each	532-37
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet Tips, for 19700-01 TenSette Pipet	1000/pkg	21856-28

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