Molybdenum

DOC316.53.01062

Ternary Complex Method **Method 8169**

0.02 to 3.00 mg/L Mo (LR) **Powder Pillows**

Scope and application: For boiler and cooling tower waters.

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
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 

Before starting

Samples must be analyzed immediately after collection and cannot be preserved for later analysis.
Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.
After several analyses, the sample cells may have a blue discoloration. Clean the sample cells with 6.0 N (50%) hydrochloric acid, then rinse thoroughly with deionized water.
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
Molybdenum Reagent Set for 20-mL sample	1
Molybdenum 1 Reagent (LR) Molybdate Powder Pillow, 20-mL	1
Molybdenum 2 Reagent Solution	0.5 mL

Items to collect (continued)

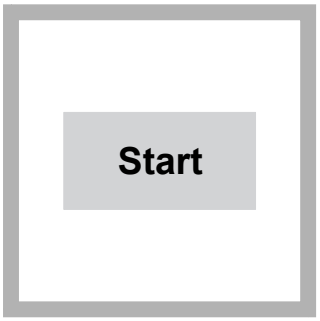
Description	Quantity
Cylinder, graduated mixing, 25-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

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

Sample collection

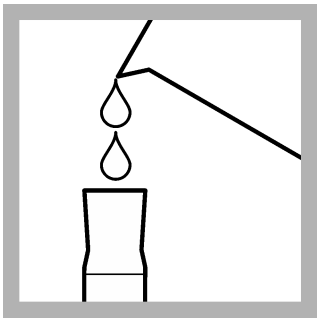
- Samples must be analyzed immediately after collection and cannot be preserved for later analysis.
- Collect samples in clean glass or plastic bottles.
- Filter samples that are turbid with filter paper and a funnel.

Powder pillow procedure

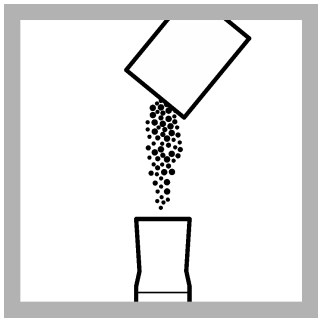


1. Start program **315 Molybdenum LR**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

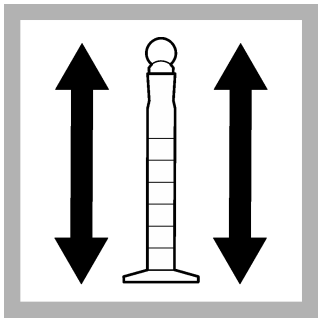
Note: *Although the program name can be different between instruments, the program number does not change.*



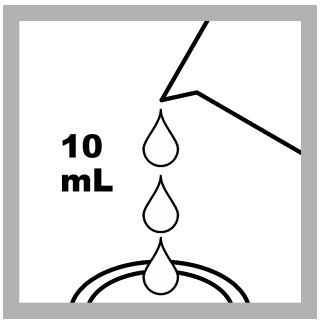
2. Prepare the sample: Fill a 25-mL graduated mixing cylinder with 20 mL of sample.



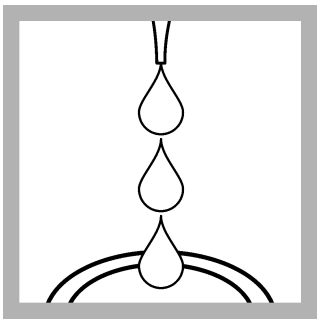
3. Add the contents of one Molybdenum 1 Reagent Powder Pillow to the mixing cylinder.



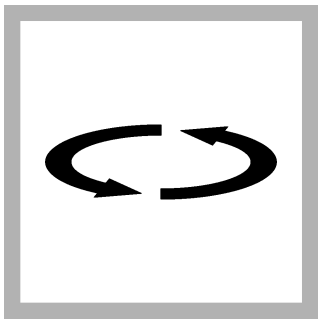
4. Close the cylinder. Shake the cylinder to completely dissolve the reagent.



5. Fill a sample cell with 10 mL of the prepared sample.

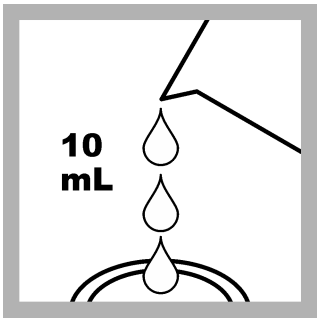


6. Develop the sample: Add 0.5 mL of Molybdenum 2 Reagent Solution to the prepared sample cell.

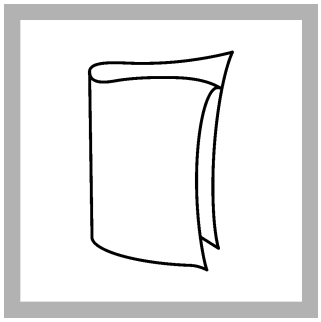


7. Swirl to mix.

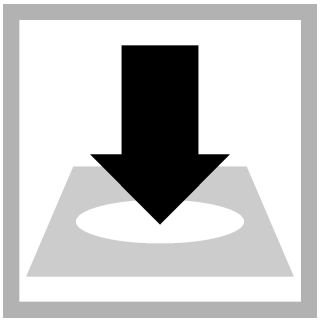
8. Start the instrument timer. A 2-minute reaction time starts.



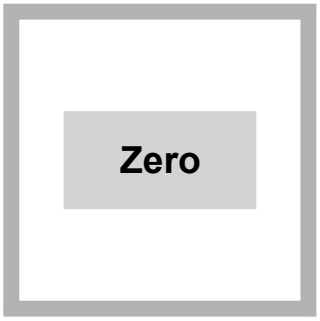
9. Prepare the blank: When the timer expires, fill a second sample cell with 10 mL of the prepared sample.



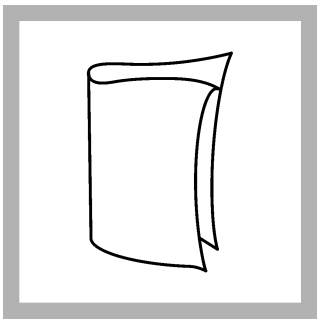
10. Clean the blank sample cell.



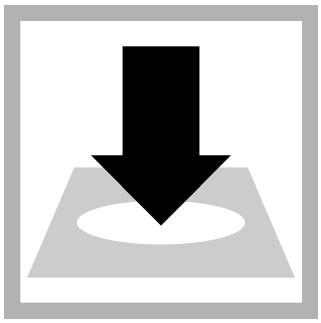
11. Insert the blank into the cell holder.



12. Push **ZERO**. The display shows 0.00 mg/L Mo^{6+}.



13. Clean the developed sample.



14. Insert the developed sample into the cell holder.

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

Interferences

Interference studies were completed with a molybdenum standard solution of 2 mg/L Mo^{6+} that included the potential interfering ion. When the standard solution concentration changed by ±5% with a given ion concentration, the ion was considered to be a substance that interferes. Interference results are summarized in Table 2, Table 3 and Table 4.

Table 2 Substances that cause a negative interference

Interfering substance	Interference level
Alum	More than 7 mg/L
Aluminum	More than 2 mg/L
AMP (Phosphonate)	More than 15 mg/L
Bicarbonate	More than 5650 mg/L
Bisulfate	More than 3300 mg/L
Borate	More than 5250 mg/L
Chloride	More than 1400 mg/L
Chromium	More than 4.5 mg/L. Read the molybdenum concentration immediately after the 2-minute reaction period.
Copper	More than 98 mg/L
Diethanoldithiocarbamate	More than 32 mg/L
EDTA	More than 1500 mg/L
Ethylene Glycol	More than 2% (by volume)

Table 2 Substances that cause a negative interference (continued)

Interfering substance	Interference level
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. Adjust to pH 3–5 with acid (Sulfuric Acid, 1 N) or base (Sodium Hydroxide, 1 N). Correct the test result for the dilution from the volume additions.
Iron	More than 200 mg/L
Lignin Sulfonate	More than 105 mg/L
Nitrite	More than 350 mg/L
Orthophosphate	More than 4500 mg/L
Phosphonohydroxyacetic Acid	More than 32 mg/L
Phosphonate HEDP	Positive interference of about 10% up to 30 mg/L. As the concentration increases above 30 mg/L, a decrease in the molybdenum concentration reading occurs (negative interference).
Sulfite	More than 6500 mg/L

Table 3 Substances that cause a positive interference

Interfering substance	Interference level
Benzotriazole	More than 210 mg/L
Carbonate	More than 1325 mg/L
Morpholine	More than 6 mg/L
Phosphonate HEDP	The presence of the phosphonate HEDP at concentrations up to 30 mg/L will increase the apparent molybdenum concentration reading by approximately 10% (positive interference). Multiply the test result by 0.9 to get the actual Mo^{6+} concentration.
Silica	More than 600 mg/L

Table 4 Non-interfering substances

Interfering substance	Interference level
Bisulfite	9600 mg/L
Calcium	720 mg/L
Chlorine	7.5 mg/L
Magnesium	8000 mg/L
Manganese	1600 mg/L
Nickel	250 mg/L
PBTC (phosphonate)	500 mg/L
Sulfate	12,800 mg/L
Zinc	400 mg/L

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+}
- Graduated cylinder, 250 mL

- Pipet, TenSette®, 0.1–1.0 mL and tips
- Erlenmeyer flasks (3)

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 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 200-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:

- Molybdenum Standard Solution, 10.00-mg/L
- 50-mL volumetric flask, Class A
- 10-mL volumetric pipet, Class A and pipet filler
- Deionized water

1. Prepare a 2.00-mg/L molybdenum standard solution as follows:
   a. Use a pipet to add 10.00 mL of 10.00-mg/L molybdenum standard solution into the volumetric flask.
   b. Dilute to the mark with deionized water. Mix well. Prepare this solution daily.
2. Use the test procedure to measure the concentration of the prepared standard solution.
3. 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
315	2.00 mg/L Mo^{6+}	1.94–2.06 mg/L Mo^{6+}	0.02 mg/L Mo^{6+}

Summary of method

The ternary complex method for molybdenum determination is a method in which molybdate molybdenum reacts with an indicator and a sensitizing agent to give a stable blue complex. The measurement wavelength is 610 nm.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Molybdenum Reagent Set, 20-mL, includes:	—	100 tests	2449400
Molybdenum 1 Reagent (LR) Molybdate Powder Pillow, 20-mL	1	100/pkg	2352449
Molybdenum 2 Reagent Solution	0.5 mL	50 mL MDB	2352512

Required apparatus

Description	Quantity/test	Unit	Item no.
Mixing cylinder, graduated, 25-mL, glass stopper	1	each	189640
Sample cells, 10-mL square, matched pair	2	2/pkg	2495402

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.
Graduated cylinder, 250-mL	each	108146
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
Flask, Erlenmeyer, 250-mL	each	50546
Pipet, volumetric, Class A, 10-mL	each	1451538
Pipet filler, safety bulb	each	1465100
Hydrochloric Acid Solution, 6.0 N (1:1)	500 mL	88449
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	104532
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032

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01/2014, Edition 9