

Alkaline Hypobromite Oxidation Method¹

Method 8024
0.01 to 0.70 mg/L Cr (spectrophotometers)
Powder Pillows
0.01 to 0.60 mg/L Cr (colorimeters)
Scope and application: For water and wastewater.²
¹ Adapted from Standard Methods for the Examination of Water and Wastewater.

² Procedure is equivalent to USEPA and Standard Method 3500-Cr D for wastewater.


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

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.

Prepare a boiling water bath for the test procedure. Use finger cots to hold hot sample cells.

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

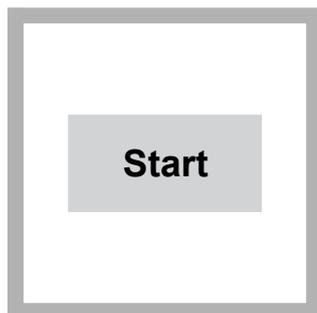
Description	Quantity
Acid Reagent Powder Pillow	1
ChromaVer [®] 3 Chromium Reagent Powder Pillow, 25-mL	1
Chromium 1 Reagent Powder Pillow	1
Chromium 2 Reagent Powder Pillow	1
Hot plate	1
Water bath and rack	1
Finger cots	1 pair
Sample cell for mixing (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Sample cells for measurement (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 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 4 with 5 N sodium hydroxide solution.
- Correct the test result for the dilution caused by the volume additions.

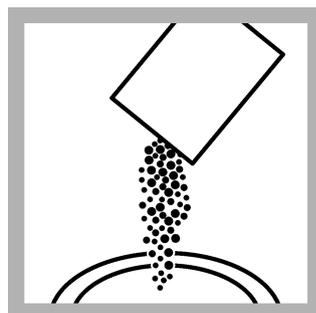
Powder pillow procedure (alkaline hypobromite oxidation method)



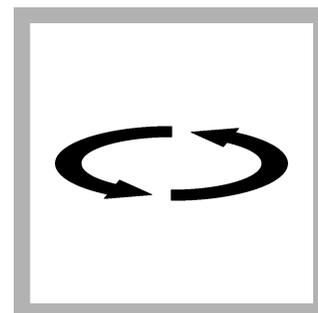
1. Start program 100 Chromium, Total. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.



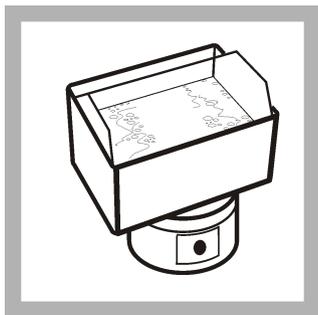
2. Fill a 25-mL sample cell with 25 mL of sample



3. Prepare the sample: Add the contents of one Chromium 1 Reagent Powder Pillow.



4. Swirl to mix.



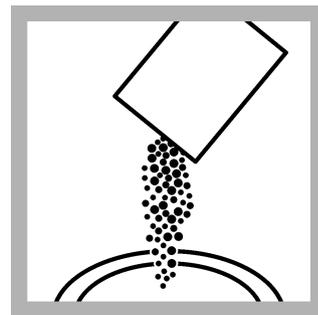
5. Keep the sample cell cap off. Put the prepared sample in a boiling water bath.



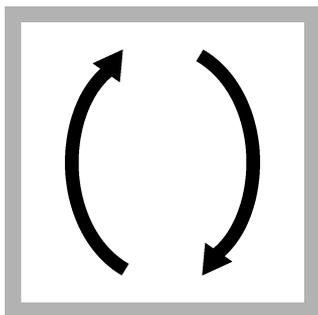
6. Start the instrument timer. A 5-minute reaction time starts.



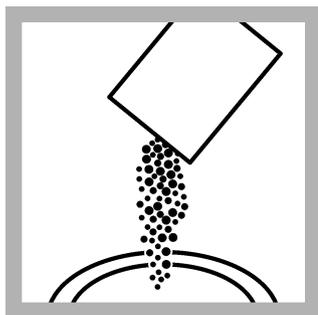
7. When the timer expires, remove the prepared sample from the water bath. Put the cap on. Use running water to cool the sample cell to 25 °C.



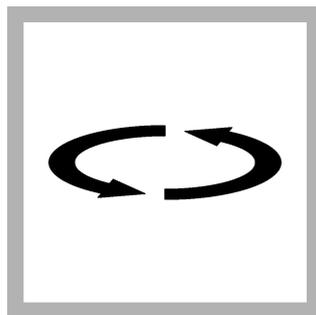
8. Add the contents of one Chromium 2 Reagent Powder Pillow.



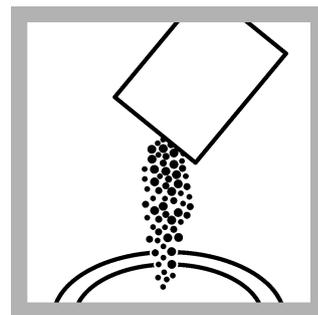
9. Put the stopper on the sample cell. Invert the sample cell to mix.



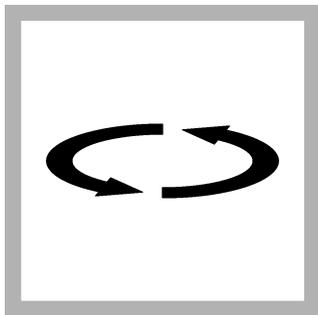
10. Add the contents of one Acid Reagent Powder Pillow.



11. Swirl to mix.



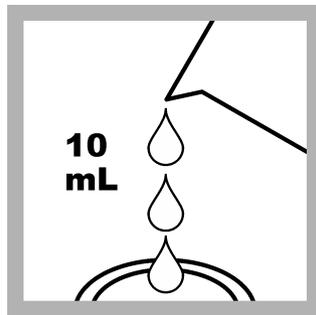
12. Add the contents of one ChromaVer 3 Chromium Reagent Powder Pillow.



13. Swirl to mix.

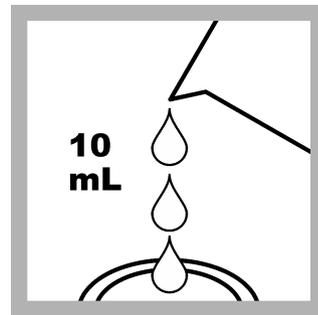


14. Start the instrument timer. A 5-minute reaction time starts.

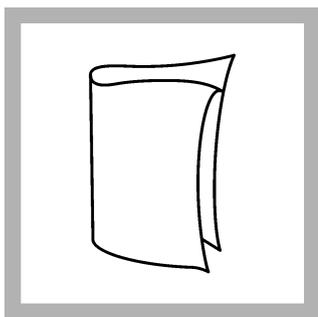


15. During the reaction, pour 10 mL from the mixing bottle into a sample cell for measurement. This is the **prepared sample**.

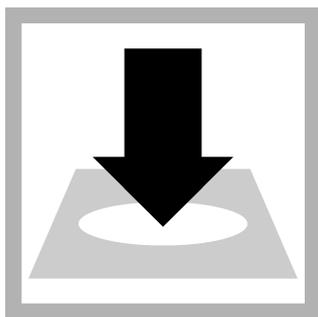
Note: Ignore this step for analysis with the DR 900 because the mixing cell and the measurement cell are the same.



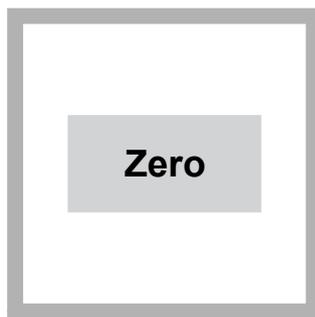
16. **Prepare the blank:** When the timer expires, fill the second measurement cell with 10 mL of the original sample.



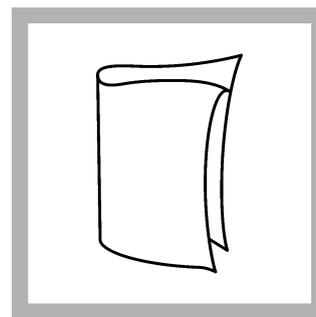
17. Clean the blank sample cell.



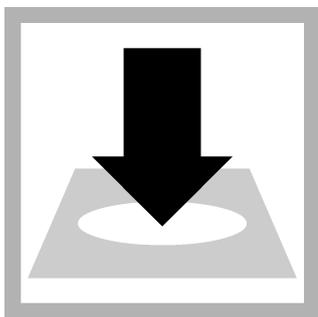
18. Insert the blank into the cell holder.



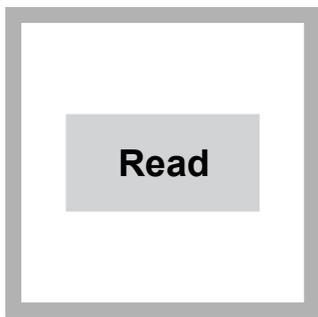
19. Push **ZERO**. The display shows 0.00 mg/L Cr.



20. Clean the prepared sample cell.



21. Insert the prepared sample into the cell holder.



22. Push **READ**. Results show in mg/L Cr.

Interferences

Interfering substance	Interference level
Organic material	May inhibit complete oxidation of trivalent chromium. If high levels of organic material are present, digestion may be required. Complete the analysis as described in this procedure on the digested sample.
Turbidity	For turbid samples, use the test procedure to prepare a 25-mL blank in the same way as the prepared sample, but do not the add the ChromaVer 3 Chromium Reagent Powder Pillow. Use this prepared blank to zero the instrument.
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:

- Trivalent Chromium Standard Solution, 50 mg/L Cr³⁺
- 5-mL volumetric pipet, Class A and pipet filler
- Mixing cylinders (4), 25 mL
- Deionized water
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Prepare a 12.5 mg/L trivalent chromium standard solution as follows:
 - a. Use a pipet to add 5.00 mL of a 50 mg/L Cr³⁺ standard solution into a 25-mL mixing cylinder.
 - b. Dilute to the 20-mL mark with deionized water. Mix well. Prepare this solution daily.

2. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
3. Go to the Standard Additions option in the instrument menu.
4. Select the values for standard concentration, sample volume and spike volumes.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the prepared standard solution, respectively, to three 25-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:

- Trivalent Chromium Standard Solution, Voluette® Ampule, 50 mg/L Cr³⁺
- 500-mL volumetric flask, Class A
- 5-mL volumetric pipet, Class A and pipet filler safety bulb
- Deionized water

1. Prepare a 0.50 mg/L trivalent chromium standard solution as follows:
 - a. Use a pipet to add 5.00 mL of 50 mg/L trivalent chromium 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 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
100	0.500 mg/L Cr	0.47–0.53 mg/L Cr	0.005 mg/L Cr

Summary of method

Trivalent chromium in the sample is oxidized to the hexavalent form by hypobromite ion under alkaline conditions. The sample is acidified. The total chromium content is determined by the 1,5-Diphenylcarbohydrazide method. Determine trivalent chromium by subtracting the results of a separate hexavalent chromium test from the results of the total chromium test. The measurement wavelength is 540 nm for spectrophotometers or 560 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
Chromium, total, Reagent Set	1	100/pkg	2242500
Includes:			
Acid Reagent Powder Pillow	1	100/pkg	212699
ChromaVer [®] 3 Chromium Reagent Powder Pillow, 25-mL	1	100/pkg	1206699
Chromium 1 Reagent Powder Pillow	1	100/pkg	204399
Chromium 2 Reagent Powder Pillow	1	100/pkg	204499

Required apparatus

Description	Quantity/test	Unit	Item no.
Hot plate, 4-inch round	1	each	1206701
OR			
Hot plate, stirrer, 220–240 VAC	1	each	2881602
Water bath and rack	1	each	195555

Recommended standards

Description	Unit	Item no.
Chromium Trivalent Standard Solution, 50-mg/L Cr ³⁺	100 mL	1415142

Optional reagents and apparatus

Description	Unit	Item no.
Finger cots	2/pkg	1464702
Flask, volumetric, Class A, 500 mL, glass	each	1457449
Pipet, volumetric 5.00-mL	each	1451537
Pipet, TenSette [®] , 0.1–1.0 mL	each	1970001
Pipet tips for TenSette [®] Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet, volumetric Class A, 15 mL	each	1451539
Pipet filler, safety bulb	each	1465100
Mixing cylinder, graduated, 25 mL	each	189640



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