Chromium, Hexavalent

USEPA¹ 1,5-Diphenylcarbohydrazide Method²

0.01 to 0.70 mg/L Cr⁶⁺

Scope and application: For water and wastewater; USEPA accepted for reporting for wastewater analysis.³

- ¹ Accepted USEPA and Standard Method 3500 Cr B.
- ² Adapted from Standard Methods for the Examination of Water and Wastewater.
- ³ Procedure is equivalent to USGS method 1-1230-85 for wastewater.

Test preparation

Before starting

At high chromium levels, a precipitate forms after the reagent is added. Sample dilution may be necessary.

Always do tests in sample cells. Do not put the instrument in the sample or pour the sample into the cell holder.

Make sure that the sample cells are clean and there are no scratches where the light passes through them.

Rinse the sample cell and cap with the sample three times before the sample cell is filled.

Make sure that there are no fingerprints or liquid on the external surface of the sample cells. Wipe with a lint-free cloth before measurement.

Cold waters can cause condensation on the sample cell or bubbles in the sample cell during color development. Examine the sample cell for condensation or bubbles. Remove condensation with a lint-free cloth. Invert the sample cell to remove bubbles.

Install the instrument cap over the cell holder before ZERO or READ is pushed.

After the test, immediately empty and rinse the sample cell. Rinse the sample cell and cap three times with deionized water.

The final samples are highly acidic.

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.

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
ChromaVer [®] 3 Chromium Reagent Powder Pillow, 5 or 10 mL	1
Sample cells, 25 mm (10 mL)	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.
- To preserve samples for later analysis, keep the samples at or below 6 °C (43 °F) for up to 24 hours.
- Let the sample temperature increase to room temperature before analysis.

Method 8023 Powder Pillows

Test procedure



1. Set the instrument to $mg/L \ Cr^{6+}$. Refer to the instrument documentation.



2. Prepare the sample: Rinse a sample cell and cap three times with sample. Fill the sample cell to the 10-mL mark with sample.



3. Add one ChromaVer[®] 3 Reagent Powder Pillow to the sample cell.



4. Swirl to mix.



5. Set and start a timer for 5 minutes. A 5-minute reaction time starts.



6. Prepare the blank: Rinse a sample cell and cap three times with sample. Fill the sample cell to the 10-mL mark with sample. Close the sample cell.



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



8. Insert the blank into the cell holder. Point the diamond mark on the sample cell toward the keypad.



9. Install the instrument cap over the cell holder.



10. Push **ZERO**. The display shows "0.00".



11. Remove the sample cell from the cell holder.



12. Clean the prepared sample cell.



13. Insert the prepared sample into the cell holder. Point the diamond mark on the sample cell toward the keypad.



14. Install the instrument

cap over the cell holder.



15. Push **READ**. Results show in mg/L hexavalent chromium (Cr^{6+}) .

Interferences

Interfering substance	Interference level
Iron	May interfere above 1 mg/L
Mercurous and Mercuric lons	Interfere slightly
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment (of the sample) by the reagents. Sample pretreatment may be necessary.
Vanadium	May interfere above 1 mg/L. Allow 10 minutes for the reaction period before reading.
Turbidity	For turbid samples, treat the blank with the contents of one Acid Reagent Powder Pillow. This will make sure that any turbidity dissolved by the acid in the ChromaVer 3 Chromium Reagent will also be dissolved in the blank.

Accuracy check

Standard additions method

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

Items to collect:

- 12.5-mg/L Hexavalent Chromium Standard Solution, 10-mL Voluette[®] Ampules
- Ampule breaker
- Pipet, TenSette[®], 0.1–1.0 mL and tips
- Mixing cylinders, 25-mL (3)
- 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 25-mL portions of fresh sample. Mix well.
- 2. 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.
- **3.** Compare the expected result to the actual result. The expected chromium concentration increase is 0.05 mg/L for each 0.1 mL of standard added.

Standard solution method

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

Items to collect:

- 12.5-mg/L Hexavalent Chromium Standard Solution
- 250-mL volumetric flask, Class A

- 5-mL volumetric pipet, Class A and pipet filler
- Deionized water
- 1. Prepare a 0.25 mg/L hexavalent chromium standard solution as follows:
 - **a.** Use a pipet to add 5.00 mL of 12.5 mg/L hexavalent 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 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 Pocket Colorimeter II during ideal test conditions. Users can get different results under different test conditions.

Precision (95% confidence interval)	
0.25 ± 0.01 mg/L Cr ⁶⁺	

Summary of method

Hexavalent chromium is determined by the 1,5-Diphenylcarbohydrazide method using a single dry powder formulation called ChromaVer 3 Chromium Reagent. This reagent contains an acidic buffer combined with 1,5-Diphenylcarbohydrazide, which reacts to give a purple color when hexavalent chromium is present.

Consumables and replacement items

Required reagents

Description	Quantity/test	Unit	Item no.
ChromaVer [®] 3 Chromium Reagent Powder Pillow, 5 or 10 mL	1	100/pkg	1271099
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/test	Unit	ltem no.
Sample cells, 10-mL round, 25 mm x 60 mm	2	6/pkg	2427606

Recommended standards

Description	Unit	Item no.
Chromium, Hexavalent Standard Solution, 10-mL Voluette [®] Ampules, 12.5-mg/L Cr ⁶⁺	16/pkg	1425610

Optional reagents and apparatus

Description	Unit	ltem no.
Mixing cylinder, graduated, 25-mL	each	2088640
Acid Reagent Powder Pillows	100/pkg	212699
Flask, volumetric, Class A, 100 mL	each	1457442

Optional reagents and apparatus (continued)

Description	Unit	ltem no.
Nitric Acid, concentrated	500 mL	15249
Pipet, serological, 2 mL	each	53236
Pipet, TenSette [®] , 0.1–1.0 mL	each	1970001
Pipet tips for TenSette [®] Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet tips for TenSette [®] Pipet, 0.1–1.0 mL	1000/pkg	2185628
Pipet, volumetric 5.00-mL	each	1451537
Pipet filler, safety bulb	each	1465100
Paper, pH, 1.0–11.0	5 rolls/pkg	39133
Sodium Hydroxide Solution, 5 N	50 mL	245026



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