Chromium, Hexavalent

DOC316.53.01033

USEPA[1] 1,5-Diphenylcarbohydrazide Method[2] **Method 8023**

0.010 to 0.700 mg/L Cr^{6+} (spectrophotometers) **Powder Pillows or AccuVac® Ampuls**

0.01 to 0.60 mg/L Cr^{6+} (colorimeters)

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

Instrument-specific information

Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests. Table 2 shows sample cell and adapter requirements for AccuVac Ampul tests. The tables also show all of the instruments that have the program for this test.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for reagent addition

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

Table 2 Instrument-specific information for AccuVac Ampuls

Instrument	Adapter	Sample cell
DR 6000 DR 5000 DR 900	—	2427606
DR 3900	LZV846 (A)	
DR 1900	9609900 or 9609800 (C)	
DR 3800 DR 2800 DR 2700	LZV584 (C)	2122800

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.
At high chromium levels, a precipitate forms. Sample dilution may be necessary.
Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.
The final samples are highly acidic.
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

Powder pillows

Description	Quantity
ChromaVer® 3 Chromium Reagent Powder Pillows, 5– or 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 6 for order information.

AccuVac Ampuls

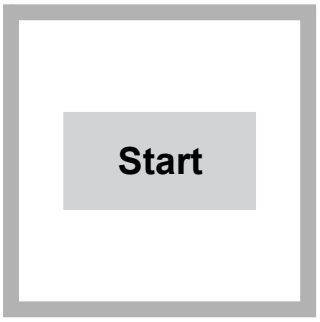
Description	Quantity
ChromaVer®3 AccuVac® Ampuls	1
Beaker, 50-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Stopper for 18-mm tubes and AccuVac Ampuls	1

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

Sample collection and storage

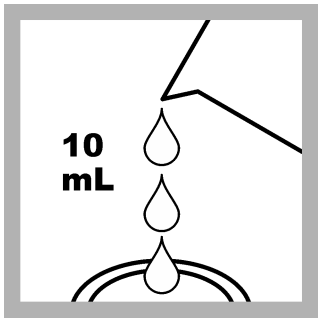
- Collect samples in clean glass or plastic bottles.
- To preserve samples for later analysis, keep the samples at or below 6 °C (43 °F) for up to 24 hours.
- Let the sample temperature increase to room temperature before analysis.

Powder pillow procedure

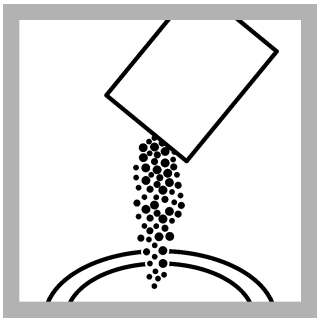


1. Start program **90 Chromium, Hex**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

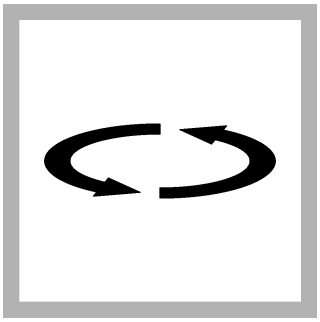
Note: *Although the program name can be different between instruments, the program number does not change.*



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

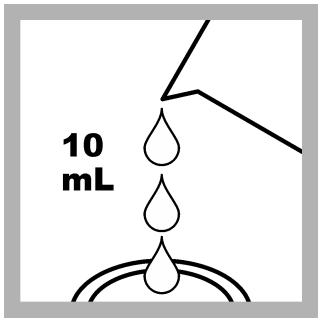


3. Add the contents of one ChromaVer® 3 Reagent Powder Pillow to the sample cell.

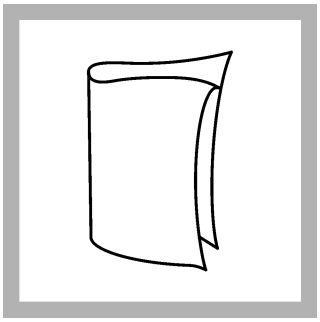


4. Swirl to mix.
A purple color will show if hexavalent chromium is present.

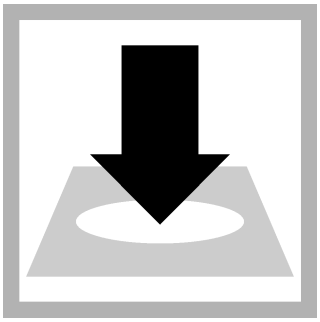
5. Start the instrument timer. A 5-minute reaction time starts.



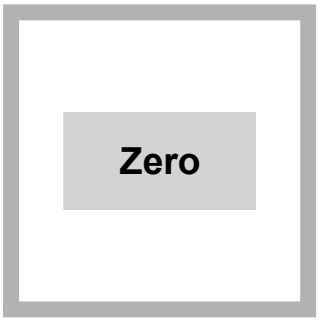
6. Prepare the blank: Fill a second sample cell with 10 mL of sample.



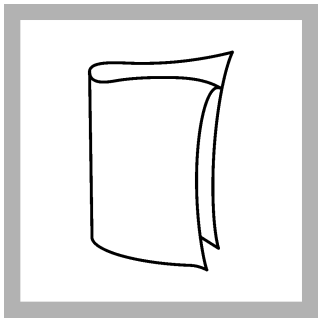
7. When the timer expires, clean the blank sample cell.



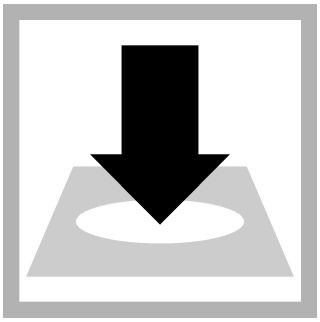
8. Insert the blank into the cell holder.



9. Push **ZERO**. The display shows 0.00 or 0.000 mg/L Cr^{6+}*.



10. Clean the prepared sample cell.

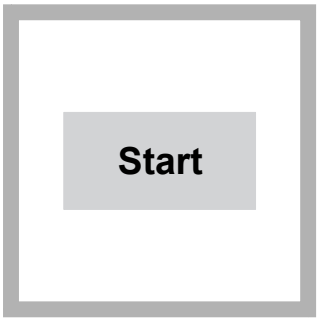


11. Insert the prepared sample into the cell holder.

12. Push **READ**. Results show in mg/L Cr^{6+}.

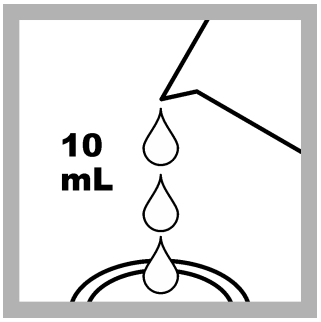
* Colorimeter display shows 0.00 mg/L.

AccuVac Ampul procedure

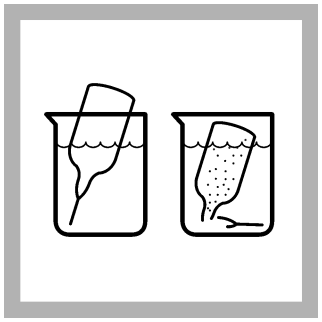


1. Start program **95 Chromium, Hex. AV**. For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.

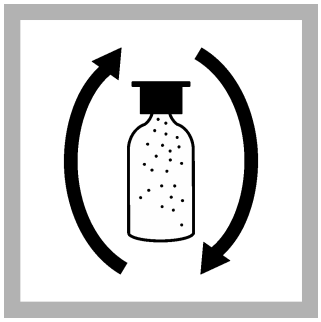
Note: *Although the program name can be different between instruments, the program number does not change.*



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

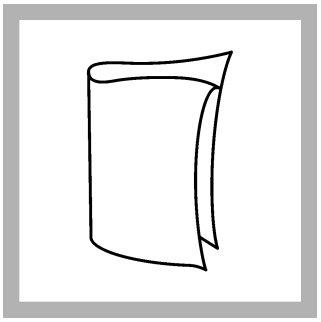


3. Prepare the sample: Collect at least 40 mL of sample in a 50-mL beaker. Fill the AccuVac Ampul with sample. Keep the tip immersed while the AccuVac Ampul fills completely.

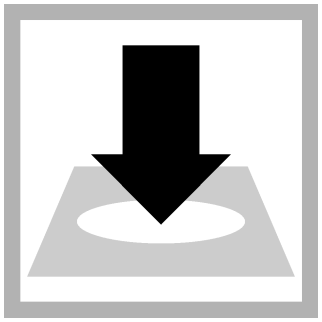


4. Quickly invert the AccuVac Ampul several times to mix.

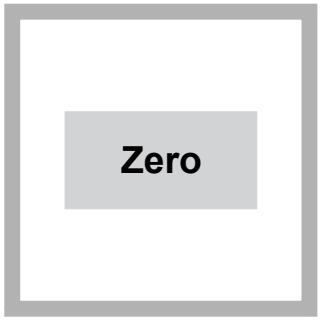
5. Start the instrument timer. A 5-minute reaction time starts.



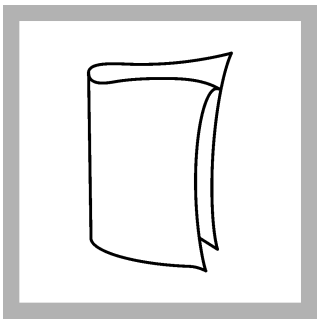
6. When the timer expires, clean the blank sample cell.



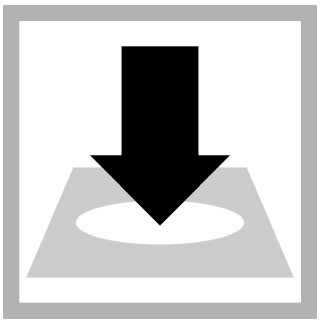
7. Insert the blank into the cell holder.



8. Push **ZERO**. The display shows 0.00 or 0.000 mg/L Cr^{6+}**.



9. Clean the AccuVac Ampul.



10. Insert the prepared sample AccuVac Ampul into the cell holder.

11. Push **READ**. Results show in mg/L Cr^{6+}.

** Colorimeter display shows 0.00 mg/L.

Interferences

Interfering substance	Interference level
Iron	May interfere above 1 mg/L
Mercurous and Mercuric Ions	Interfere slightly
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.
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 (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:

- 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)

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 25-mL portions of fresh sample. Mix well.

   Note: *For AccuVac® Ampuls, add 0.2 mL, 0.4 mL and 0.6 mL of the standard solution to three 50-mL portions of fresh sample.*
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:

- 50.0-mg/L Hexavalent Chromium Standard Solution
- 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 hexavalent chromium standard solution as follows:
   a. Use a pipet to add 5.00 mL of 50.0 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 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
90	0.500 mg/L Cr^{6+}	0.497–0.503 mg/L Cr^{6+}	0.005 mg/L Cr^{6+}
95	0.500 mg/L Cr^{6+}	0.496–0.504 mg/L Cr^{6+}	0.006 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. 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.
ChromaVer® 3 Chromium Reagent Powder Pillow, 5– or 10–mL	1	100/pkg	1271099
OR			
ChromaVer® 3 AccuVac® Ampul	1	25/pkg	2505025
Water, deionized	varies	4 L	27256

Required apparatus

Description	Quantity/test	Unit	Item no.
AccuVac Snapper	1	each	2405200
Beaker, 50-mL	1	each	50041H
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards

Description	Unit	Item no.
Chromium, Hexavalent Standard Solution, 10-mL Voluette® Ampules, 12.5-mg/L Cr^{6+}	16/pkg	1425610
Chromium Hexavalent Standard Solution, 50.0-mg/L Cr^{6+}	100 mL	81042H

Optional reagents and apparatus

Description	Unit	Item no.
Acid Reagent Powder Pillows	100/pkg	212699
Ampule Breaker, 10-mL Voluette® Ampules	each	2196800
Flask, volumetric, Class A, 500-mL glass	each	1457449
Pipet, volumetric 5.00-mL	each	1451537
Pipet filler, safety bulb	each	1465100
Sodium Hydroxide Solution, 5 N	50 mL	245026

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