

Chromium, Total and Hexavalent

DOC316.53.01035

1,5-Diphenylcarbohydrazide Method**Method 10218 (hexavalent) and 10219 (total)****0.03 to 1.00 mg/L Cr****TNTplus™ 854****Scope and application:** For wastewater and process analysis.

Test preparation

Instrument-specific information

[Table 1](#) shows all of the instruments that have the program for this test. The table also shows the adapter and light shield requirements for the applicable instruments that can use TNTplus vials.

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

Table 1 Instrument-specific information for TNTplus vials

Instrument	Adapters	Light shield
DR 6000, DR 5000	—	—
DR 3900	—	LZV849
DR 3800, DR 2800	—	LZV646
DR 1900	9609900 or 9609800 (A)	—

Before starting

DR 3900, DR 3800, DR 2800: Install the light shield in Cell Compartment #2 before this test is started.

Review the safety information and the expiration date on the package.

The recommended sample pH is 3–9.

The sample temperature must be 15–35 °C (59–95 °F) for accurate results.

The recommended temperature for reagent storage is 2–8 °C (35–46 °F).

Use the DRB reactor with 13-mm wells for the digestion. If the reactor has 16-mm wells, insert adapter sleeves into the wells.

The concentration of trivalent chromium is calculated from the difference between chromium (total) and chromium VI (hexavalent).

DR 1900: Go to All Programs>LCK or TNTplus Methods>Options to select the TNTplus number for the test. Other instruments automatically select the method from the barcode on the vial.

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
Chromium, Hexavalent and Total TNTplus Reagent Set	1
DRB200 reactor with 13-mm wells	1
Pipet, adjustable volume, 1.0–5.0 mL	1

Items to collect (continued)

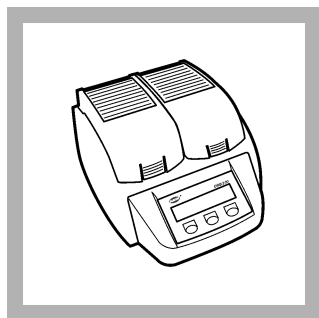
Description	Quantity
Pipet tips, for 1.0–5.0 mL pipet	1
Test tube rack	1

Refer to [Consumables and replacement items](#) on page 5 for order information.

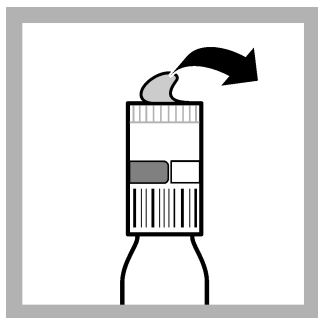
Sample collection and storage

- Collect samples in clean glass or plastic bottles.
- To preserve samples for total chromium analysis, adjust the sample pH to less than 2 with concentrated nitric acid (approximately 2 mL per liter). Keep the preserved samples at or below 6 °C (43 °F) for a maximum of 6 months. Before analysis, adjust the pH to 4 with 5 N sodium hydroxide solution.
- To preserve samples for hexavalent chromium analysis, adjust the sample pH to 8 with 8 N potassium hydroxide. Keep the preserved samples at or below 6 °C (43 °F) for a maximum of 24 hours. No pH adjustment is necessary.
- Let the sample temperature increase to room temperature before analysis.
- Correct the test result for the dilution caused by the volume additions.

Test procedure—total chromium



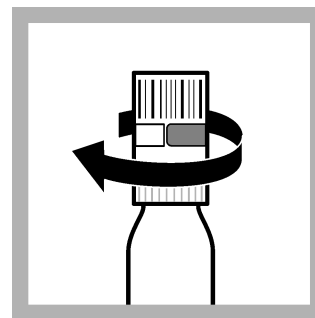
1. Set the DRB200 reactor power to on. Set the temperature to 100 °C.



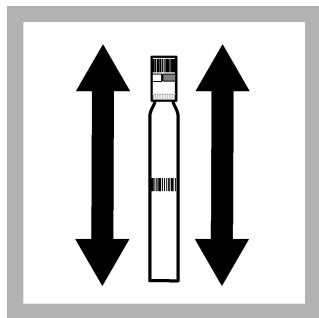
2. Carefully remove the lid from the DosiCap™ Zip cap. Remove the cap from the test vial.



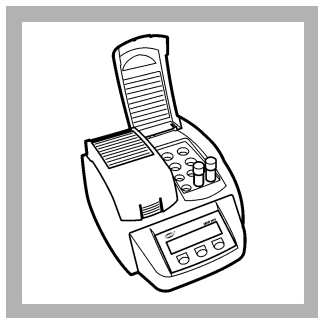
3. Use a pipet to add 2.0 mL of sample to the test vial.



4. Turn the DosiCap Zip over the test vial so that the reagent side goes on the vial. Tighten the cap on the vial.



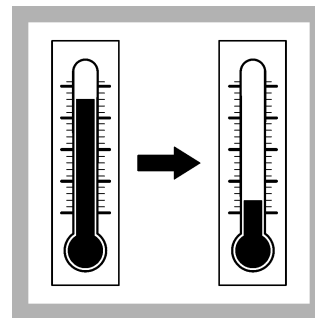
5. Shake the vial 2–3 times to dissolve the reagent in the cap. Look through the open end of the DosiCap to make sure that the reagent has dissolved.



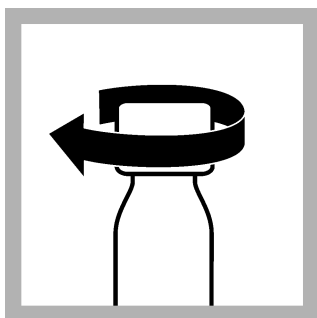
6. Insert the vial in the preheated DRB200 reactor. Close the lid.



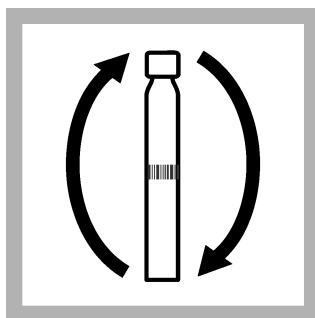
7. Keep the vial in the reactor for 1 hour.



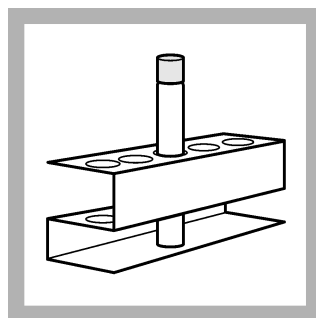
8. When the timer expires, carefully remove the vial from the reactor. Set the vial in a test tube rack. Let the temperature of the vial decrease to room temperature.



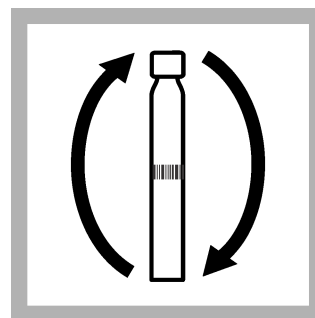
9. Put an orange DosiCap B on the vial.



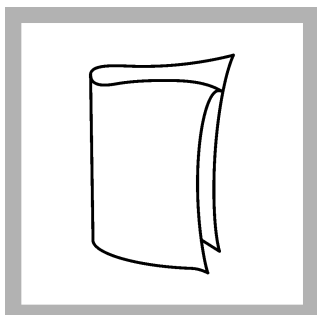
10. Tighten the cap on the vial and invert the vial 2–3 times.



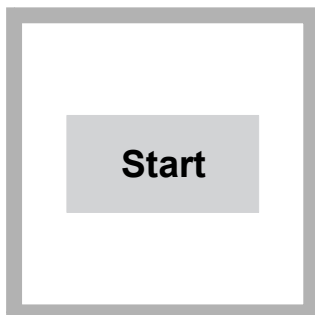
11. Put the vial in the test tube rack for 2–3 minutes.



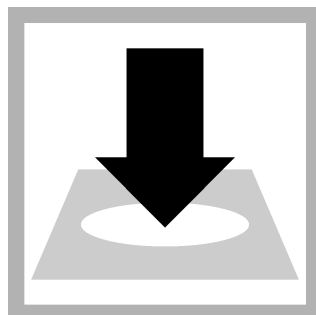
12. When the timer expires, invert the vial 2–3 times.



13. Clean the vial.

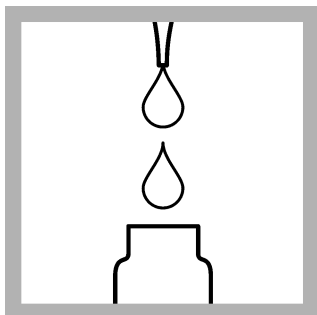


14. DR 1900 only: Select program 854. Refer to [Before starting](#) on page 1.

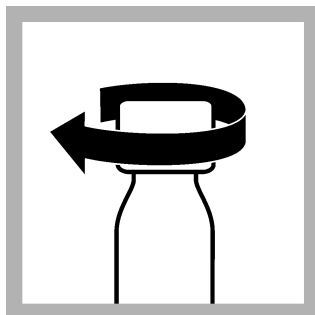


15. Insert the vial into the cell holder. DR 1900 only: Push **READ**. Results show in mg/L Cr.

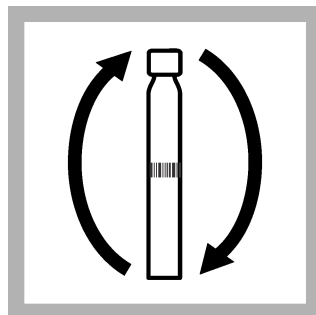
Test procedure—hexavalent chromium



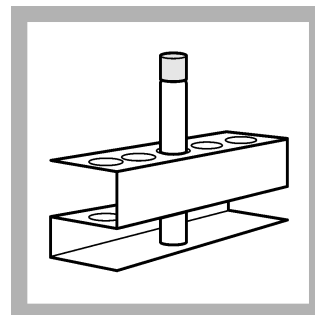
1. Use a pipet to add 2.0 mL of sample to the test vial.



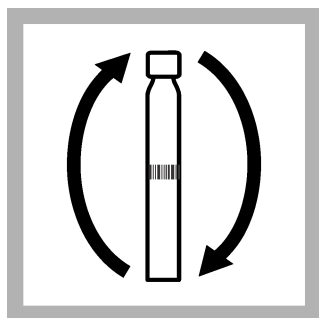
2. Put an orange DosiCap B on the vial.



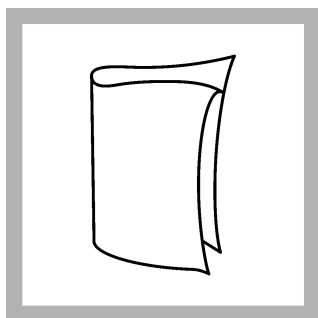
3. Tighten the cap on the vial and invert the vial 2–3 times.



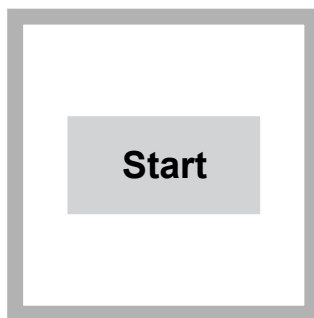
4. Put the vial in the test tube rack for 2–3 minutes.



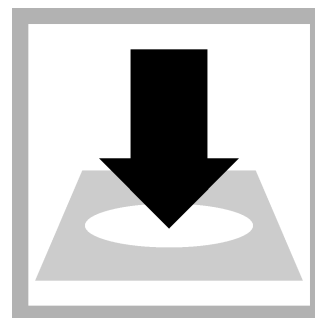
5. When the timer expires, invert the vial 2–3 times.



6. Clean the vial.



7. DR 1900 only: Select program 854. Refer to [Before starting](#) on page 1.



8. Insert the vial into the cell holder. DR 1900 only: Push **READ**. Results show in mg/L Cr.

Reagent blank correction

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. Measure the reagent blank value when a new lot of reagent is used.

1. Use deionized water as the sample in the test procedure to measure the reagent blank value.
2. Set the reagent blank function to on. The measured reagent blank value is shown.
3. Accept the blank value. The reagent blank value is then subtracted from all results until the reagent blank function is set to off or a different method is selected.

Note: As an alternative, record or enter the reagent blank value at a different time. Push the highlighted reagent blank box and use the keypad to enter the value.

Sample blanks

Samples with color or turbidity can cause high results. Samples without color or turbidity do not require sample blanks. The digestion in the total chromium test procedure usually removes all color and turbidity. A sample blank is not necessary. To adjust for color or turbidity, do the hexavalent chromium test procedure. Use the steps that follow to find the sample blank.

1. Do the test procedure, but do not add the reagent.
2. Subtract the value from the final procedure step from the initial sample value to get the corrected sample concentration. The results are given as dissolved hexavalent chromium.

Note: As an alternative, filter samples that have only turbidity through a membrane filter, then analyze.

Interferences

[Table 2](#) shows that the listed ions were individually examined to the given concentrations and do not cause interference. No cumulative effects or influences of other ions were found.

Larger amounts of iron, copper and reducing and oxidizing agents give low-bias results. Lead, mercury and tin give high-bias results.

Undissolved chromium is not found with the determination of chromium(VI). An analyte concentration more than (20 mg/L) in excess of the stated range adversely affects color formation, resulting in a false reading within the method range.

Verify the measurement results with sample dilutions or standard additions.

Table 2 Interfering substances

Interfering substance	Interference level
SO ₄ ²⁻ , Na ⁺ , K ⁺ , NO ₃ ⁻	2000 mg/L
Cl ⁻	1000 mg/L
Ca ²⁺	125 mg/L
Mg ²⁺ , NH ₄ ⁺	100 mg/L
Zn ²⁺ , Ni ²⁺ , Co ²⁺ , Cd ²⁺	50 mg/L
Pb ²⁺	25 mg/L
Cu ²⁺ , Fe ³⁺	10 mg/L
Ag ⁺	5 mg/L
Sn ²⁺	1 mg/L

Accuracy check

Standard solution method

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

Items to collect:

- 50-mg/L Chromium Standard Solution (use trivalent chromium for total chromium procedure)
 - 100-mL volumetric flask, Class A
 - 1.0-mL volumetric pipet, Class A and pipet filler safety bulb
 - Deionized water
1. Prepare a 0.50-mg/L chromium standard solution as follows:
 - a. Use a pipet to add 1.0 mL of a 50-mg/L chromium standard solution into the volumetric flask. Use a trivalent chromium standard solution to verify the total chromium procedure. Use a hexavalent chromium standard solution to verify the hexavalent chromium procedure.
 - 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.

Summary of Method

In the total chromium procedure, all chromium in the sample is oxidized to hexavalent chromium (Cr⁶⁺). The hexavalent chromium then reacts with 1,5-diphenylcarbazide to form 1,5-diphenylcarbazone. The amount of red color formed with hexavalent chromium is directly proportional to the amount of chromium in the sample. To determine trivalent chromium, subtract the results of a separate hexavalent chromium test from the results of the total chromium test. The measurement wavelength is 543 nm.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
Chromium, Hexavalent and Total TNTplus Reagent Set	1	25/pkg	TNT854

Required apparatus

Description	Quantity/test	Unit	Item no.
DRB 200 Reactor, 115 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB20001
DRB 200 Reactor, 230 VAC option, 9 x 13 mm + 2 x 20 mm, 1 block	1	each	DRB20005
Pipet, adjustable volume, 1.0–5.0 mL	1	each	BBP065
Pipet tips, for 1.0–5.0 mL pipet	1	75/pkg	BBP068
Pipet, adjustable volume, 0.2–1.0 mL	1	each	BBP078
Pipet tips, for 0.2–1.0 mL pipet	2	100/pkg	BBP079
Test tube rack	1	each	1864100
Light shield, DR 3800, DR 2800, DR 2700	1	each	LZV646
Light shield, DR 3900	1	each	LZV849

Recommended standards

Description	Unit	Item no.
Chromium Trivalent Standard Solution, 50-mg/L Cr ³⁺	100 mL	1415142
Chromium Hexavalent Standard Solution, 50.0-mg/L Cr ⁶⁺	100 mL	81042H

Optional reagent and apparatus

Description	Unit	Item no.
Reactor adapter sleeves, 16 mm to 13 mm diameter, for TNTplus vials	5/pkg	2895805
Filter membrane, 0.45-micron, 25-mm	100/pkg	2514101
Flask, volumetric, Class A, 100-mL glass	each	1457442
Nitric Acid, concentrated	500 mL	15249
Sampling bottle with cap, low density polyethylene, 500-mL	12/pkg	2087079
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	104532
Sodium Hydroxide Standard Solution, 5.0 N	100 mL MDB	245032



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