**Scope and Application:** For soil. The estimated detection limit for program number 1560 is 0.006 mg/L Cr\(^{6+}\).

1. Press the soft key under **HACH PROGRAM**.
   Select the stored program for hexavalent chromium (Cr\(^{6+}\)) by pressing **1560** with the numeric keys.
   Press: **ENTER**
   **Note:** If samples cannot be analyzed immediately, see Sample Collection, Preservation, and Storage following these steps.

2. The display will show:
   **HACH PROGRAM: 1560 Chromium, Hex.**
   The wavelength (\(\lambda\)), **540 nm**, is automatically selected.

3. Accurately weigh out the required sample size based on anticipated Cr\(^{6+}\) levels (see Table 1). Place soil sample in whirl-pak bag or sample cup.

4. Prepare the extraction solution by adding the contents of one Hexavalent Chromium Extractant Pillow to a 50-mL graduated mixing cylinder. Add 40 mL of deionized water.
   Stopper and invert to dissolve the powder.

<table>
<thead>
<tr>
<th>Expected Cr(^{6+}) Concentration</th>
<th>Sample Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>500–5000 ppb</td>
<td>20 g</td>
</tr>
<tr>
<td>1.0–10 ppm</td>
<td>20 g</td>
</tr>
<tr>
<td>2.50–50 ppm</td>
<td>20 g</td>
</tr>
<tr>
<td>50–1000 ppm</td>
<td>1 g</td>
</tr>
<tr>
<td>500–10,000 ppm</td>
<td>1 g</td>
</tr>
</tbody>
</table>
5. Add the extraction solution to the soil in the whirl-pak bag or sample cup and close tightly. Invert and shake several times to ensure the soil is completely suspended.

6. Press **1500** to enter a 15-minute time period. Press the soft key under **START TIMER**.
   Shake the soil/soil extractant mixture for 15 seconds at 2-minute intervals during the 15-minute time period.
   **Note:** If using a soil shaker, shake for 15 minutes at 200 rpm.
   **Note:** The programmed 8-minute time period is not applicable for soil. It is used for the chromium in water method.

7. Set a funnel in the 50-mL graduated cylinder. Place a piece of pre-folded filter paper into the funnel. Filter the soil/soil extractant mixture into the cylinder and save the filtrate.

8. Determine the required filtrate aliquot size from *Table 2*. Pipet the required aliquot from the 50-mL graduated cylinder into a 25-mL graduated cylinder.

### Table 2 Required Aliquot Size

<table>
<thead>
<tr>
<th>Expected Cr(^{6+}) Concentration</th>
<th>Aliquot Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>500–5000 ppb</td>
<td>10 mL</td>
</tr>
<tr>
<td>1.0–10 ppm</td>
<td>5 mL</td>
</tr>
<tr>
<td>2.50–50 ppm</td>
<td>1 mL</td>
</tr>
<tr>
<td>50–1000 ppm</td>
<td>1 mL</td>
</tr>
<tr>
<td>500–10,000 ppm</td>
<td>0.1 mL</td>
</tr>
</tbody>
</table>
9. Dilute the cylinder to the 25-mL mark with deionized water. Stopper and invert to mix.

10. Fill a sample cell to the 10-mL mark with the diluted filtrate (the blank).

11. Fill another sample cell to the 10-mL mark with the diluted filtrate remaining in the cylinder. Add the contents of a ChromaVer 3 Chromium Reagent Powder Pillow to the cell and swirl to mix. This is the prepared sample.

Note: A purple color will develop if chromium is present.

Note: For best results, determine a reagent blank for each new lot of reagent as follows. Prepare a reagent blank by repeating Steps 8-15, using deionized water as the sample. Zero the instrument on deionized water by pressing the soft key under ZERO. Insert the reagent blank and the blank value will be displayed. Correct for the reagent blank by pressing the soft keys under OPTIONS, (MORE), and then BLANK:OFF. Enter the reagent blank value and press ENTER. Repeat for each new lot of reagent.

12. Press 1000 to enter a 10-minute time period. Press the soft key under START TIMER. A 10-minute reaction period will begin.

Note: If the solution becomes cloudy following reagent addition and does not clear up after 10-minutes, the aliquot size must be reduced until the turbidity is no longer present after the 10-minute reaction period.
13. Place the blank into the cell holder. Close the light shield.

14. Press the soft key under **ZERO**. The display will show:

\[ 0.000 \text{ mg/L Cr}^{6+} \]

**Note:** If you are using a reagent blank correction, the display will show the correction.

**Note:** For alternate concentration units, press the soft key under **OPTIONS**. Then press the soft key under **UNITS** to scroll through the available options. Press **ENTER** to return to the read screen.

**Note:** Results may be expressed as other forms of chromium. Press the soft keys under **OPTIONS** and then **FORM:** to scroll through the available options. Press **ENTER** to return to the read screen.

15. Place the prepared sample into the cell holder. Close the light shield. Results in mg/L apparent hexavalent chromium (or chosen units) will be displayed. Use the following formula to determine the actual hexavalent chromium concentration in the original soil sample:

\[
\text{Cr}^{6+} \text{ (ppb) } = \frac{\text{DR/4000 reading (mg/L)} \times 10^6}{\text{Aliquot (mL)} \times \text{sample size}}
\]

### Interferences

**Table 1 Interfering Substances and Suggested Treatments**

<table>
<thead>
<tr>
<th>Interfering Substance</th>
<th>Interference Levels and Treatments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron</td>
<td>Iron interference levels depend on the volume of the extract aliquot. To avoid iron interference, use the smallest sample and aliquot volume possible. Iron interference prevents color formation. If you get a Cr(^{6+}) concentration of nearly 0, check the sample for iron interference by adding 0.1 mL of a 50-mg/L Cr(^{6+}) standard to the sample and allow an additional 10-minute reaction period. If a pink color develops, iron is not present and the sample does not contain hexavalent chromium. If no color develops, interference is occurring and the aliquot and/or sample size should be decreased until the interferences stop.</td>
</tr>
<tr>
<td>Mercurous &amp; Mercuric Ions</td>
<td>Interferes slightly</td>
</tr>
<tr>
<td>pH</td>
<td>Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require sample pretreatment; see Section 1.3.1 pH Interference.</td>
</tr>
<tr>
<td>Vanadium</td>
<td>1 mg/L. Wait 10 minutes before reading result.</td>
</tr>
</tbody>
</table>
Sample Collection, Preservation, and Storage

Obtain a soil sample representative of the area being tested. If the soil is moist or sampled from depth, it will need to be air dried before sampling if low levels of hexavalent chromium are to be accurately determined. High levels of hexavalent chromium (>100 ppm) do not require as rigorous sampling/sample treatment procedures.

Accuracy Check

Standard Solution

1. Using a TenSette Pipet or a fixed volume pipetter, add 0.2 mL of a 50-mg/L Chromium, Hexavalent, Standard Solution to 20 grams of soil. The soil must not contain any Cr\textsuperscript{6+}.

2. Analyze the sample as described in the above. A reading between 0.3 and 0.4 mg/L should be obtained. The calculation should give a result of 400-600 ppb, with the actual value being 500 ppb.

Method Performance

Precision

Standard: 0.25 mg/L Cr\textsuperscript{6+}

<table>
<thead>
<tr>
<th>Program</th>
<th>95% Confidence Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>1560</td>
<td>0.247–0.253 mg/L Cr\textsuperscript{6+}</td>
</tr>
</tbody>
</table>

For more information on determining precision data and method detection limits, refer to Section 1.5.

Estimated Detection Limit

<table>
<thead>
<tr>
<th>Program</th>
<th>EDL</th>
</tr>
</thead>
<tbody>
<tr>
<td>1560</td>
<td>0.006 mg/L Cr\textsuperscript{6+}</td>
</tr>
</tbody>
</table>

For more information on derivation and use of Hach’s estimated detection limit, see Section 1.5.2. To determine a method detection limit (MDL) as defined by the 40 CFR part 136, Appendix B, see Section 1.5.1.

Sensitivity

Program Number: 1560

<table>
<thead>
<tr>
<th>Portion of Curve:</th>
<th>∆Abs</th>
<th>∆Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Entire Range</td>
<td>0.010</td>
<td>0.0055 mg/L</td>
</tr>
</tbody>
</table>

See Section 1.5.3 Sensitivity Explained for more information.

Summary of Method

Hexavalent chromium in soil is measured via extraction using a concentrated, alkaline extracting reagent. Color development with the 1,5 Diphenylcarbohydrazide method uses a single dry powder formulation called ChromaVer 3 Chromium Reagent. This reagent contains a buffer and 1,5 Diphenylcarbohydrazide, which gives a purple color when hexavalent chromium is present.
Safety

Good safety habits and laboratory techniques should be used throughout the procedure. Consult the Material Safety Data Sheet for information specific to the reagents used. For additional information, refer to Section 1.

Pollution Prevention and Waste Management

The final samples are highly acidic. Neutralize to pH 6–9 and flush to drain for disposal. For information on pollution prevention and waste management, refer to Section 1.

REQUIRED REAGENTS AND STANDARDS

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity Required per test</th>
<th>Unit</th>
<th>Cat. No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>ChromaVer 3 Chromium Reagent Powder Pillows</td>
<td>1</td>
<td>100/pkg</td>
<td>12710-99</td>
</tr>
<tr>
<td>Hexavalent Chromium Soil Extractant Powder Pillows</td>
<td>1</td>
<td>100/pkg</td>
<td>24497-99</td>
</tr>
</tbody>
</table>

REQUIRED EQUIPMENT AND SUPPLIES

- Bags, Whirl-Pak ...........................................2 100/pkg 22331-99
- Sample Cups, disposable plastic, 150-mL ..........2 2500/pkg 22631-74
- Sample Cup Lids ...........................................2 3000/pkg 22632-74
- Balance, laboratory, 300-g ................................1 each 14760-00
- Clippers, large (shears) ..................................1 each 23694-00
- Cylinder, graduated mixing, 25-mL ...................1 each 20886-40
- Cylinder, graduated, 50-mL, poly .....................1 each 2172-41
- DR/4000 1-Inch Cell Adapter ..........................1 each 48190-00
- Filter Paper, folded, 15- cm ............................2 100/pkg 692-58
- Funnel, analytical, poly., 65-mm ......................1 each 1083-67
- Pipetter, 100 µL ..........................................1 each 22753-00
- Pipet Tips, for 22753-00 Pipetter ....................2 10/pkg 22754-10
- Scoop, soil, 1-g ..........................................1 each 26572-01
- Scoop, soil, 10-g .........................................1 each 26572-10

OPTIONAL REAGENTS AND SUPPLIES

- Chromium, Hexavalent, Standard Solution, 50-mg/L Cr⁶⁺ ........................................100 mL 810-42H

OPTIONAL EQUIPMENT AND SUPPLIES

- Demineralizer Assembly, 473-mL ..........................each 21846-00
- DR/4000 Carousel Module Kit ..............................each 48070-02
- DR/4000 Flow Cell Module Kit, 1-inch .....................each 48070-04
- DR/4000 Flow Cell Module Kit, 1-cm ........................each 48070-05
- DR/4000 Sipper Module Kit, 1-inch ........................each 48090-03
- SOILAB Filtration Assembly ................................each 22624-00
- Pipet TenSette, 1.0–10.0 mL ................................each 19700-10
- Pipet Tips, for 19700-10 TenSette Pipet ..................50/pkg 21997-96