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**METHOD 14552Cr6**

**Hexavalent Chromium (Cr<sup>6+</sup>) by Reaction with  
Diphenylcarbazide and Photometry**

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## Acknowledgments

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## Disclaimer

This method has been submitted to the U.S. Environmental Protection Agency for use in EPA's water programs but has not been approved for use by EPA. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

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## Introduction

This method is a convenient, ready to use cell test kit for the analysis of hexavalent chromium ( $\text{Cr}^{6+}$ ). This procedure is based upon “Standard Methods for the Examination of Water and Wastewater,” 18<sup>th</sup> edition, Method 3500-Cr D. The test kit is suitable for both on-site testing and typical laboratory testing. The test kit consists of reagents capable of effecting analytical determinations. This method’s approach, with pre-measured reagents, reduces the analytical errors, the amount of hazardous waste, and increases occupational safety.

The method covers the range 0.05 mg/L to 2.0 mg/L hexavalent chromium ( $\text{Cr}^{6+}$ ).

# Method 14552Cr6

## Hexavalent Chromium (Cr<sup>6+</sup>) by Reaction with Diphenylcarbazide and Photometry

Analyte: Chromium (CAS # Cr 7440-47-3)

### 1.0 Scope and Application

- 1.1 This method is used to measure exclusively hexavalent chromium (Cr<sup>6+</sup>). Diphenylcarbazide, dissolved in a weak phosphoric acid mixture, is oxidized to diphenylcarbazone by Cr<sup>6+</sup>. The Chromium III (Cr<sup>3+</sup>) produced during this reaction, reacts with diphenylcarbazone to give a red-violet color complex that is measured photometrically or near at 543 nm. The reaction, producing the red violet color, only occurs in the presence of Cr<sup>6+</sup>, i.e. chromate or dichromate, as only Cr<sup>6+</sup> gives rise to the reactive, non-hydrolyzed Cr<sup>3+</sup> intermediary during the reaction. Cr<sup>3+</sup> in water samples is either hydrolyzed or complex bound and hence not available for reaction.
- 1.2 This method determines Cr<sup>6+</sup> which occurs naturally in the water (total hexavalent), is dissolved in the water (dissolved hexavalent), or that is converted from other forms (total chromium).
- 1.3 This method is for use in the United States Environmental Protection Agency's (EPA's) data gathering and monitoring programs under the Clean Water Act.
- 1.4 The method detection limit (MDL; 40 CFR 136, Appendix B) has been established at 0.02 mg/L.
- 1.5 The minimum level (ML) for this method has been established as 0.05 mg/L
- 1.6 This method is capable of measuring Cr<sup>6+</sup> in the range of 0.05 to 2.00 mg/L and may be extended to higher levels by serial dilution.
- 1.7 This method is intended for the analysis of Cr<sup>6+</sup> on treated and untreated sanitary and industrial waste waters, and other waste water matrices.
- 1.8 This method is based on a prior Environmental Protection Agency (EPA) association method for the determination of Cr<sup>6+</sup> (Reference 16.1).
- 1.9 Each laboratory that uses this method must demonstrate the ability to generate acceptable results using the procedure in Section 9.2.

### 2.0 Summary of Method

- 2.1 For the determination of total, dissolved, or dissolved total Cr<sup>6+</sup>, pre-treat the samples as described in SM 3500-Cr D (Reference 16.1).
- 2.2 The Spectroquant<sup>®</sup> Cr<sup>6+</sup> Cell Test 14552 contains pre-measured granules of diphenylcarbazide. A six drop aliquot (approximately 0.18 ml) of dimethylsulfoxide-phosphoric acid solution, Spectroquant<sup>®</sup> Reagent Cr-3K, is added to the cell. The cell is closed tightly and mixed vigorously to soak the solids.
- 2.3 After a one minute period, five ml of pre-treated sample is pipetted into the cell test.

- 2.4 After mixing well, allow five minutes for reaction to occur (color formation).
- 2.5 After the five minute reaction period, the intensity of the red-violet color is measured photometrically at or near a wavelength of 543 nm.
- 2.6 The photometric determination can be conducted on either a Merck Spectroquant®-type system photometer or other standard photometric device.
- 2.7 Quality is assured through the use of quality control samples (QCS), calibration of the instrumentation by using calibration test solutions (Cr standard solution) as described in Section 7.4 and Section 17, Table 2, and operation of a formal quality assurance program (Reference 16.2).

### 3.0 Definitions

Definitions for terms used in this method are given in the glossary at the end of the method (Section 18).

### 4.0 Interferences

- 4.1 The reaction of the diphenylcarbazide is nearly specific for Cr<sup>6+</sup>. Hexavalent molybdenum and mercury salts will react to form color with the reagent, but the intensities are much lower than that for Cr<sup>6+</sup> at the specified pH. Concentrations as high as 100 mg Mo<sup>6+</sup> or 10 mg Hg<sup>2+</sup>/L can be tolerated (Reference 16.3).
- 4.2 Vanadium interferes strongly but concentrations up to 10 times that of chromium do not cause appreciable difficulties with recoveries. Concentrations as high as 100 mg V<sup>5+</sup>/L can be tolerated in blanks. Levels at 1 mg V<sup>5+</sup>/L produce false negative results (Reference 16.3).
- 4.3 Iron in concentrations greater than 100 mg/L may interfere (Reference 16.3).

### 5.0 Safety

- 5.1 This method does not address all safety issues associated with its use. The toxicity or carcinogenicity of reagents used in this method has not been fully established. Each chemical and environmental sample should be regarded as a potential health hazard and exposure should be minimized. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses. Additional information on laboratory safety can be found in References 16.4 and 16.5.
- 5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. It is suggested that the laboratory perform personal hygiene monitoring of each analyst using this method and that the results of this monitoring shall be made available to the analyst.

- 5.3** Samples of unknown origin may possess potentially hazardous compounds. Samples should be handled with care (e.g., under a hood), so as to minimize exposure.
- 5.4** This method employs the use of Spectroquant® Cr<sup>6+</sup> Cell Tests, containing pre-measured reagents, which limits the handling of hazardous chemicals.

## 6.0 Equipment and Supplies

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**NOTE:** *Brand names, suppliers, and part numbers are cited for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and materials other than those specified here, but demonstration of equivalent performance that meets the requirements of this method is the responsibility of the laboratory.*

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- 6.1** Sample collection bottles-1-L borosilicate glass or plastic.
- 6.2** Volumetric flasks-various sizes.
- 6.3** Volumetric pipettes-various sizes.
- 6.4** Reaction cells-Spectroquant® Cr<sup>6+</sup> Cell Test, Item 14552.
- 6.5** Laboratory timer.
- 6.6** Rack for cells.
- 6.7** Dry cloths for cleaning cells.
- 6.8** Photometric device.
- 6.8.1** Photometer capable of measuring absorbance at or near 543 nm wavelength, with a cell compartment for tubes 16 x 100-mm-Merck Spectroquant®-type system photometer, or equivalent.

## 7.0 Reagents and Standards

- 7.1** Spectroquant® Cr<sup>6+</sup> Cell Test, item 14552.
- 7.1.1** Diphenylcarbazide-premeasured in cell.
- 7.1.2** Spectroquant® Reagent Cr-3K.
- 7.2** Deionized water.
- 7.4** Chromium stock solution, 1,000 mg/L-catalog no. 1.19780, or equivalent.
- 7.5** Chromium standard solution, 5.0 mg/L-dilute 5.0 ml stock chromium to 1L (1ml = 5 µg).

## 8.0 Sample Collection, Preservation, and Storage

- 8.1 Collect 1L, or at least 100 ml, of sample in a plastic or borosilicate glass bottle, following conventional sampling techniques, as outlined in Reference 16.6 .
- 8.2 Refrigerate samples at 0 to 4°C from the time of collection until analysis, 40 CFR 136, Table II.
- 8.3 If dissolved Cr<sup>6+</sup> is desired, filter the sample immediately, preserve the sample with HNO<sub>3</sub> to pH of <2, and analyze the sample within 24 hours of collection.
- 8.4 If total chromium is to be analyzed, preserve the sample immediately upon sampling with HNO<sub>3</sub> to pH of <2.
- 8.5 Collect an additional aliquot of a sample for each batch (of at least 20 samples) for the field duplicate.

## 9.0 Quality Control

- 9.1 Each laboratory using this method is required to operate a formal quality control (QC) program (Reference 16.2). The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the ongoing analysis of laboratory reagent blanks, precision and recovery standards, and field duplicate samples as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of data thus generated. Laboratory performance, once established, is constantly updated, to determine if the results of analyses meet the performance characteristics of the method, or if any modifications to the procedure are required.
  - 9.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 9.2.
  - 9.1.2 Analysis of field duplicate samples are required to demonstrate method accuracy and precision. The procedure and QC criteria for calculating accuracy and precision on field duplicates (RPD) are described in Section 9.3.
  - 9.1.3 Analyses of laboratory blanks are required to demonstrate freedom from contamination. The procedure and criteria for blank analyses are described in Section 9.4.
  - 9.1.4 The laboratory shall, on an ongoing basis, demonstrate through calibration verification and analysis of the ongoing precision and recovery sample that the analysis system is in control. These procedures are described in Sections 9.5 and 9.6.
  - 9.1.5 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in Sections 9.3.7 and 9.6.3.
  - 9.1.6 Accompanying QC for the determination of Cr<sup>6+</sup> is required per analytical batch. An analytical batch is a set of samples analyzed, to a maximum of 20 samples. Each analytical batch, of up to 20 samples, must be accompanied by a laboratory blank (Section 9.4), and an ongoing precision and recovery sample (OPR, Section 9.6), and (1) field duplicate sample (Section 9.3).

**9.2** Initial demonstration of laboratory capability-The initial demonstration of laboratory capability is used to characterize laboratory performance and method detection limits.

**9.2.1** Method detection limit (MDL)-The method detection limit must be established for the analyte, using the Cr standard solution (Section 7.5). To determine MDL values, take seven replicate aliquots of the diluted Cr standard solution and process each aliquot through each step of the analytical method. Perform all calculations and report the concentration values in the appropriate units. MDLs should be determined every year or whenever a modification to the method or analytical system is made that will affect the method detection limit.

**9.2.2** Initial Precision and Recovery (IPR) - To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

**9.2.2.1** Analyze four samples of the Cr standard solution (Section 7.5) according to the procedure beginning in Section 11.

**9.2.2.2** Using the results of the four analyses, compute the average percent recovery ( $\bar{x}$ ) and the standard deviation ( $s$ , Equation 1) of the percent recovery for  $\text{Cr}^{6+}$ .

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*Equation 1*

$$s = \sqrt{\frac{\sum x^2 - \frac{(\sum x)^2}{n}}{n - 1}}$$

*Where:*

$n$  = number of samples

$x$  = % recovery in each sample

$s$  = standard deviation

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**9.2.2.3** There are no standardized acceptable criteria. This needs to be established by the individual laboratory. At a minimum, the laboratory must meet the default QC requirements (Reference 16.7) where none have been established), which are listed in Section 17, Table 3.



- 9.3** Field Duplicates-Laboratory must run, in duplicate, a minimum of five percent of all samples (one sample in each batch of 20 samples).
- 9.3.1** Compute relative percent difference (RPD) between the two results, using the following equation:

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**Equation 2**

$$RPD = 100 * \frac{(|D_1 - D_2|)}{(D_1 + D_2)/2}$$

where:

*RPD*=Relative percent different

*D*<sub>1</sub>=Concentration of Cr<sup>6+</sup> in the sample

*D*<sub>2</sub>=Concentration of Cr<sup>6+</sup> in the second (duplicate) sample

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- 9.3.2** The relative percent difference criteria for duplicates shall be established by the laboratory, and should at a minimum, meet the default acceptance criteria (Reference 16.7) listed in Section 17, Table 3. If these criteria are not met, the analytical system is judged to be out of control, and the problem must be immediately identified and corrected, and the analytical batch re-analyzed.
- 9.3.3** As a part of the QC program for the laboratory, method precision and accuracy for samples should be assessed and records should be maintained. After the analysis of five duplicate sets of samples, in which the precision passes the test in Section 9.3.2, compute the average relative percent difference (RPD<sub>a</sub>) and the standard deviation of the relative percent difference (s<sub>RPD</sub>).
- 9.4** Laboratory blanks-Laboratory reagent water blanks are analyzed to demonstrate freedom from contamination.
- 9.4.1** Prepare and analyze a laboratory blank initially (i.e., with the tests in Section 9.2) and with each analytical batch. The blank must be subjected to the same procedural steps as a sample.
- 9.4.2** If material is detected in the blank at a concentration greater than the ML (Section 1.5), analysis of samples must be halted until the source of contamination is eliminated and a new blank shows no evidence of contamination. All samples must be associated with an uncontaminated laboratory blank before the results may be reported for regulatory compliance purposes.

- 9.5** Calibration verification-Verify calibration of the photometric device, as per Section 10, for each analytical batch of up to 20 samples. If calibration curve linearity differs more than 10%, run a new calibration curve.
- 9.6** Ongoing Precision and Recovery (OPR)-To demonstrate that the analysis system is in control, and acceptable precision and accuracy is being maintained with each analytical batch, the analyst shall perform the following operations:
- 9.6.1** Analyze a precision and recovery standard (Section 7.5) with each analytical batch according to the procedure beginning in Section 11.
- 9.6.2** Compare the concentration of the OPR with the established limits, or at a minimum the default acceptance limits (Reference 16.7) for ongoing precision and recovery as listed in Section 17, Table 3. If the concentration is in the range specified, the analysis may proceed. If however, the concentration is not in the specified range, the analytical process is not in control. In this event, correct the problem, re-extract the analytical batch, and repeat the ongoing precision and recovery test.
- 9.6.3** The laboratory should add results that pass the specification in Section 9.6.2 to IPR and previous OPR data and update QC charts to form a graphic representation of continued laboratory performance. The laboratory should also develop a statement of laboratory data quality for each analyte by calculating the average percent recovery (R) and the standard deviation of the percent recovery ( $s_r$ ). Express the accuracy as a recovery interval from  $R - 2s_r$  to  $R + 2s_r$ .
- For example, if  $R = 95\%$ , and  $s_r = 5\%$ , the accuracy is 85 % to 105 %.
- 9.7** Quality control sample (QCS)—It is suggested that the laboratory obtain a quality control sample from a source different from the source of the Cr standard solution used routinely in this method (Section 7.5).
- 9.8** The standards used for initial precision and recovery (IPR, Section 9.2.2) field duplicates (RPD, Section 9.3), and ongoing precision and recovery (OPR, Section 9.6) should be identical, so that the most precise results will be obtained.

## 10.0 Calibration and Standardization

- 10.1** The Merck Spectroquant<sup>®</sup>-type system photometers are shipped factory calibrated (Reference 16.8), refer to the manufacturer's documents (Reference 16.9, 16.10, and 16.11). The calibration curve can be verified, and the data from this verification can be stored, modified or re-entered at anytime. However, the factory program settings cannot be changed by the user. When appropriate, the manufacturer supplies a new memo chip (transponder) containing new calibration data.
- 10.2** For other photometric equipment, plot a calibration curve with a minimum of five (5) data points, from standards prepared from the Cr standard solution. The calibration curve should also include a blank.

- 10.2.1** A series of calibration curve standards are prepared as stated in Section 17, Table 2. The calibration curve should span the entire range of the test, and include the highest and lowest concentrations in that range.
- 10.3** Verify the curve, using a calibration standard (mid-point of the curve), with each analytical batch of samples (Section 9.5).
- 10.4** Run a new calibration curve with each new lot of reagents, or when calibration curve linearity differs more than 10%, as stated in 40 CFR part 136, Table IB (Section 9.5).

## **11.0 Procedure**

- 11.1** Adjust the pH of the sample (pre-treated as described in SM 3500-Cr D (Reference 16.1) for the type of Cr<sup>6+</sup> desired) within the range of one to nine.
- 11.2** Add six drops (0.18 ml) of Spectroquant<sup>®</sup> Reagent Cr-3K to a Spectroquant<sup>®</sup> Cr<sup>6+</sup> Cell Test.
- 11.3** Close the cell tightly, and shake vigorously.
- 11.4** Allow this homogenized solution to sit for 1 minute.
- 11.5** Add 5.0 ml of the prepared sample (Section 11.1) into the cell and mix.
- 11.6** Place cell upright in a cell rack. Allow five minutes for reaction.
- 11.7** Wipe the cell clean with a clean dry cloth.
- 11.8** Photometric determination using a Merck Spectroquant<sup>®</sup>-type system photometer.
  - 11.8.1** Switch on the Merck Spectroquant<sup>®</sup>-type system photometer as per manufacturer's suggestions for operation (References 16.9, 16.10, and 16.11).
  - 11.8.2** Place the Spectroquant<sup>®</sup> Cr<sup>6+</sup> Cell Test into the cell compartment with the vertical line aligned with the notch on the instrument, and push down until the cell clicks into place.
  - 11.8.3** Wait as the Merck Spectroquant<sup>®</sup>-type system photometer recognizes the bar code. The Spectroquant<sup>®</sup> Cr<sup>6+</sup> Cell Test product concentration range is displayed, and the instrument is automatically set to the appropriate wavelength and measuring parameters (bar code recognition of item number, test range, cell format, wavelength, and calibration data).
  - 11.8.4** Record the displayed result in mg/L.
- 11.9** Determination using other photometric equipment.
  - 11.9.1** Warm up the instrument as per manufacturer's suggestion for operation.
  - 11.9.2** Set the instrument's wavelength to at or near 543 nm.
  - 11.9.3** Zero the instrument with a reagent water / blank, which has been prepared in the same manner as the standards and samples.
  - 11.9.4** Place the cell into the cell compartment/cell holder.

- 11.9.5** Record the absorbance reading on the instrument.
- 11.9.6** Plot the absorbance reading on the calibration curve, to obtain the concentration of  $\text{Cr}^{6+}$  (mg/L).

## 12.0 Data Analysis and Calculations

- 12.1** If no pre-dilution was performed upon the sample, no calculation is necessary.
- 12.2** If pre-dilution was required, calculate the  $\text{Cr}^{6+}$  (mg/L) as follows:

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### Equation 3

$$\text{Cr}^{6+} = A * \frac{V_2}{V_1}$$

where:

$A$  = Measured concentration of  $\text{Cr}^{6+}$  from photometer (mg/L)

$V_1$  = Volume of sample used for dilution (ml)

$V_2$  = Final total volume of diluted sample (ml)

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- 12.3** Report results to two significant digits for concentrations found above the ML (Section 1.4) in all samples. Report results below the ML as <0.05 mg/L for  $\text{Cr}^{6+}$ .

## 13.0 Method Performance

- 13.1** This method, as equivalent to Standard Method 3500-Cr D (Reference 16.1), should achieve the method performance as cited in Section 6 of that method.
- 13.2** The method detection limit (MDL) study was performed by a single analyst, and was determined as 0.02 mg/L (Section 1.4).
- 13.3** The minimum level (ML) is determined as 0.05 mg/L (Section 1.5).

## 14.0 Pollution Prevention

- 14.1** The reagents used in this method pose little threat to the environment, when managed properly.
- 14.2** Reagents should be ordered consistent with laboratory use, to minimize the amount of expired materials to be disposed.

## 15.0 Waste Management

- 15.1** It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restriction. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations.
- 15.2** For further information on waste management, consult "The Waste Management Manual for Laboratory Personnel" and "Less is Better: Laboratory Chemical Management for Waste Reduction," both available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16<sup>th</sup> Street N.W., Washington, D.C. 20036.

## 16.0 References

- 16.1** "Standard Methods for the Examination of Water and Wastewater," 18<sup>th</sup> Edition, American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, Method 3500-Cr D.
- 16.2** Spectroquant<sup>®</sup> Cr<sup>6+</sup> Cell Test Product Insert, Item 14552, Merck KGaA, Frankfurter Strasse 250, Darmstadt 64271, Germany, Release March 1994.
- 16.3** "OSHA Safety and Health Standards, General Industry," (29CFR 1910), Occupational Safety and Health Administration, OSHA 2206, revised January 1976.
- 16.4** "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3<sup>rd</sup> Edition, 1979.
- 16.5** "Standard Methods for the Examination of Water and Wastewater," 18<sup>th</sup> Edition, American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005, Method 1060.
- 16.6** "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL-Ci, Cincinnati, OH 45268, EPA-600/4-79-019, March 1979.
- 16.7** Protocol for EPA Approval of Alternate Test Procedures for Organic and Inorganic Analytes in Wastewater and Drinking Water-Draft March 1998, Environmental Protection Agency, Office of Water (4303) Washington, DC 20460.
- 16.8** "German Standard Methods for the Examination of Water, Wastewater, and Sludge," Deutsches Institut für Normung e.V., D-10772, Berlin, DIN Method 38402 Part 51, May 1986.
- 16.9** Spectroquant<sup>®</sup> SQ 118 Manual, Merck KGaA, Frankfurter Strasse 250, Darmstadt 64271, Germany, Release July 1998.
- 16.10** Spectroquant<sup>®</sup> NOVA 60 Manual, Merck KGaA, Frankfurter Strasse 250, Darmstadt 64271, Germany, Release July 1998.
- 16.11** Spectroquant<sup>®</sup> VEGA 400 Manual, Merck KGaA, Frankfurter Strasse 250, Darmstadt 64271, Germany, Release July 1998.

## 17.0 Tables

**Table 1. Product Range, Number, and Usage Information**

<u>Range mg/L</u>	<u>Product Number</u>	<u>Sample Volume (ml)</u>	<u>Merck Spectroquant® Photometer Test Code</u>	<u>Wavelength nm</u>
0.05 to 2.0	14552	5.0	039	550

**Table 2. Calibration Standard Preparation**

<u>Product #</u>	<u>Cr Standard Solution</u>	
<u>Range (mg/L)</u>	<u>Volumes (ml)*</u>	<u>Cr Equivalent (mg/L)</u>
14552 (0.05 - 2.0)	0 - 1 - 5 - 10 - 20 - 40	0 - 0.05 - 0.25 - 0.5 - 1.0 - 2.0

\* Dilute all working calibration standards to 100 ml in volumetric flasks.

**Table 3. Acceptance Criteria for Performance Tests**

<b>Acceptance Criterion</b>	<b>Section</b>	<b>Limit (%)</b>
<b>Initial precision and recovery</b>	9.2.2	
Cr <sup>6+</sup> Precision (s)	9.2.2.2	30
Cr <sup>6+</sup> Recovery (X)	9.2.2.2	47 - 153
<b>Field Duplicate</b>	9.3	
Cr <sup>6+</sup> RPD	9.3.1	36
<b>Ongoing precision and recovery</b>	9.6	
Cr <sup>6+</sup> Recovery	9.6	40 - 160

Until the laboratory establishes their own quality control acceptance criteria, the data must, at a minimum, meet these default limits (Reference 16.8).

## 18.0 Definitions

**18.1** The definitions and purposes are specific to this method, but have been conformed to common usage as much as possible.

### 18.1.1 Symbols

°C	degrees Celsius
>	greater than
<	less than
%	percent

### 18.1.2 Alphabetical Characters

g	gram
L	liter
mg	milligram
mg/L	milligram per liter
ml	milliliter
nm	nanometer
µg	microgram

**18.2** Definitions, acronyms, and abbreviations.

**18.2.1** Analyte: Cr<sup>6+</sup>, which is tested by this method.

**18.2.2** Analytical batch: The set of samples analyzed at the same time, to a maximum of 20 samples. Each analytical batch must be accompanied by a laboratory blank (Section 9.4), and ongoing precision and recovery sample (OPR, Section 9.6), and a field duplicate (Section 9.3).

**18.2.3** Cr<sup>6+</sup>: See chromium <sup>6+</sup>.

**18.2.4** Chromium<sup>6+</sup>: The parameter which is tested for by this method.

**18.2.5** Cr standard solution: The standard solution (5 mg/L) which is used to perform all QC tests and calibration of instrument.

**18.2.6** IPR: See initial precision and recovery.

**18.2.7** Initial precision and recovery (IPR): Four aliquots of the diluted Cr standard solution analyzed to establish the ability to generate acceptable precision and accuracy. An IPR is performed the first time this method is used and any time the method or instrument is modified.

**18.2.8** Laboratory blank (method blank): An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, and reagents that are



used with samples. The laboratory blank is used to determine if analyte or interferences are present in the laboratory environment, or the reagents.

- 18.2.9** May: This action, activity, or procedural step is neither required nor prohibited.
- 18.2.10** Merck Spectroquant® - Type System Photometer: Photometers containing information on Spectroquant® products. The instruments automatically set to the appropriate wavelength and measuring parameters through bar code recognition of the test cell. Measuring parameters stored in these instruments are item number, test range, cell format, wavelength, and calibration data.
- 18.2.11** Method detection limit (MDL): The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.
- 18.2.12** Minimum level (ML): The lowest level at which the entire analytical system gives a recognizable signal and acceptable calibration point of the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and preparation procedures have been employed.
- 18.2.13** Must: This action, activity, or procedural step is required.
- 18.2.14** OPR: See ongoing precision and recovery standard.
- 18.2.15** Ongoing precision and recovery standard (OPR): A laboratory blank spike with known quantities of analyte. The OPR is treated exactly like a sample. Its purpose is to establish performance of the method by the analyst.
- 18.2.16** Quality Control Sample (QCS): A sample containing analyte of interest at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory performance using test materials that have been prepared independently from the normal preparation process.
- 18.2.17** Reagent water: Water demonstrated to be low or free from metallic contaminants.
- 18.2.18** Shall: This action, activity, or procedural step is required.
- 18.2.19** Should: This action, activity, or procedural step is suggested, but not required.
- 18.2.20** Spectroquant® Cr<sup>6+</sup> Cell Test: Kit consisting of screw capped round cells (16 x 100 mm) containing pre-measured Cr<sup>6+</sup> reagent and pre-mixed reagent.
- 18.2.21** Transponder: The memo chip, which contains updated information which may include new methods and updated calibration information for downloading into the Merck Spectroquant®-type system photometer.