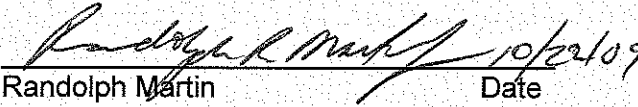

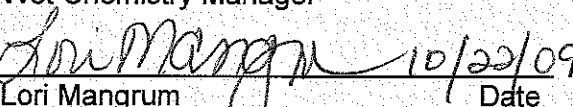
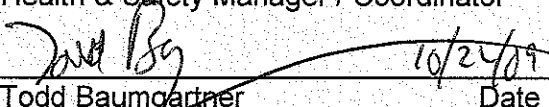
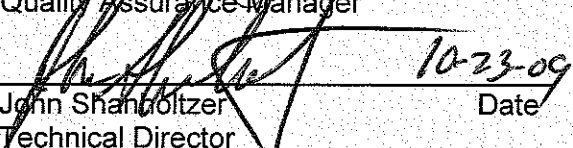


Title: Hexavalent Chromium- Colorimetric Procedure

Method: SM3500-Cr B (21st edition) / SW-846 7196A

Approvals (Signature/Date):

 Randolph Martin Wet Chemistry Manager	 Salvador Ramos Health & Safety Manager / Coordinator
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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP is based upon the methodology described for the determination of hexavalent chromium in water, wastewater, leachates, and soils in accordance with Standard Method 3500-Cr B and SW-846 7196A. The Hach powder pillow is equivalent to the reagents listed in SM3500-Cr B.

The purpose of this SOP is to describe the procedures used to determine the concentration of hexavalent chromium in natural and treated water in the range of 100 to 1000µg in accordance with Standard Method 3500-Cr B. Method SW-846 7196A is used to determine the concentration of dissolved hexavalent chromium in TCLP characteristic extracts and ground waters. This method may also be applicable to certain domestic and industrial wastes, provided that no interfering substances are present. Solids are extracted with a simple DI Leach process and analyzed per Method SW-846 7196A.

The routine target analyte lists, current Reporting Limit (RL), Method Detection Limit (MDL) and precision and accuracy limits associated with this procedure are given in the Method Limit Group (MLGs) in LIMS.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 20 in the Quality Assurance Manual.

If for any reason a part of this SOP cannot be followed, seek the guidance of the Department Manager or the Laboratory Technical Director. All abnormalities must be noted on the data or the benchsheet, in the Non-conformance database in LIMS. See the corrective action procedures and Appendix 4 in the QA Manual

2.0 Summary of Method

This SOP describes the procedures used to determine the concentration of hexavalent chromium in water, wastewater, leachates, and soils.

Aqueous samples are acidified to a pH less than 2 ±0.5 and treated with a color reagent (1,5-diphenylcarbazide) to form a red-violet colored complex. Alternatively, a Hach Chroma Ver 3 powder pillow is added to the samples. The reagent pillow contains the color reagent and a buffer to adjust the pH of the sample to the proper level. The absorption is measured at 540nm and the concentration determined using a standard curve.

This procedure has been modified from the procedures listed in the reference methods to reduce the volumes of sample and reagent needed to perform the test. This procedure has sufficient sensitivity to detect 0.010mg/L hexavalent chromium in an interference-free sample.

3.0 Definitions

- 3.1 Refer to SOP TP-AN-005: Definitions, Terms, and Acronyms and to the current revision of the Tampa's Quality Assurance Manual (TP-QAM) for a complete listing of applicable definitions.

4.0 Interferences

- 4.1 Iron concentrations greater than 1mg/L may produce a yellow color but this is usually not significant when measured at 540nm. Hexavalent molybdenum and mercury salts react to form color with the reagent but the red-violet intensities produced are much lower than those for chromium at the specified pH. Mercury and molybdenum in concentrations up to 200 mg/L can be tolerated. Vanadium interferes strongly but concentrations up to ten times the chromium are tolerable.
- 4.2 Highly buffered samples or extreme sample pH may exceed the buffering capacity of the reagents and require additional acid to bring the pH into the range (pH <2) for color development for method SW-846 7196A.
- 4.3 One sample per batch is spiked in duplicate (MS/MSD) with hexavalent chromium to determine if interferences are present. The sample must be diluted or analyzed by the method of standard additions (MSA) if the recovery is not within 85-115%.
- 4.4 There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing lab ware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.
- 4.5 The entire work area should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.
- 4.6 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. If physical interferences are present, they should be documented.
- 4.7 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Equipment

- Spectrophotometer with a 1cm or wider path length cell, set at wavelength of 540nm. A wider path length increases the sensitivity of the colorimetric test but generally reduces the linear range.

6.2 Supplies

- Disposable plastic beakers or cups (at least 25mL)
- Graduated cylinder (25ml or 50mL)
- Adjustable pipetting device to deliver required volumes
- Volumetric flasks, class A (or equivalent) - appropriate volumes

7.0 Reagents and Standards

- 7.1 All reagents and standards are to be labeled with their unique TALS ID, including name of the material, concentration, date prepared/received, expiration date, and analyst name, according to SOP TP-AN-004, *Standard Material and Reagent Traceability*. Certificates of

analysis or purity must be received with all neat compounds and stock solutions and scanned into TALS.

- 7.2 Lab-Generated De-Ionized Reagent Water: ASTM Type I or Type II. The conductivity must be checked daily in accordance with SOP TP-AN-009: *Conductivity Checks for Laboratory Deionized Water*.
- 7.3 Diphenylcarbazide solution: Dissolve 250mg 1, 5-diphenylcarbazide in 50mL of Acetone. Store in an amber bottle and prepare fresh weekly. Discard if the solution discolors.
- 7.4 Phosphoric Acid - H_3PO_4 , conc.
- 7.5 Sulfuric Acid - H_2SO_4 , conc., 18N, and 6N.
- 7.6 Sodium Hydroxide - NaOH , 1N.
- 7.7 Hach Chroma Ver 3 powder pillows, if color reagent is not used
- 7.8 Hexavalent chromium stock standard: 1000mg/L certified solution. This stock is used to prepare the calibration curve and CCV.
 - 7.8.1 **Intermediate chromium standard (10mg/L)**: Add approx. 50mL of DI water to a 100mL volumetric flask. Transfer 1.0mL of the 1000mg/L stock standard to the flask, dilute to volume with DI water and mix thoroughly. Prepare fresh daily or with each analytical batch.
- 7.9 Second source hexavalent chromium standard: 1000mg/L certified solution. This stock is used to prepare the calibration verification standards: ICV, LCS, MS/MSD.
 - 7.9.1 **Second source intermediate standard (10mg/L)** Add approx. 50mL of DI water to a 100mL volumetric flask. Transfer 1.0mL of the 1000mg/L second source stock to the flask, dilute to volume with DI water and mix thoroughly. Prepare fresh daily or with each analytical batch.
- 7.10 **Continuing Calibration Verification standards (CCV) (0.20mg/L)**: Add about 10mL of reagent water to a 25mL volumetric flask. Transfer 500uL of the 10mg/L second source intermediate standard to the flask, dilute to volume with reagent water, and mix thoroughly.
- 7.11 **Initial Calibration Verification standard (ICV) (0.20mg/L)**: Add about 10mL of reagent water to a 25mL volumetric flask. Transfer 500uL of the 10mg/L second source intermediate standard to the flask, dilute to volume with reagent water, and mix thoroughly.

8.0 **Sample Collection, Preservation, Shipment and Storage**

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time ¹	Reference
Waters	HDPE	100mLs	HNO ₃ , pH < 2; Cool 4 + 2°C	24 Hours	40 CFR Part 136.3
Soils	Glass	50 grams	Cool 4 + 2°C	24 Hours	N/A

¹ Inclusive of digestion and analysis.

9.0 Quality Control

Please refer to Appendix 4 of the laboratory QAM for additional information relating to the QC associated with this method

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

9.1.1 **Method Blank (MB)** -One method blank must be processed with each preparation batch. The method blank consists of DI water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

9.1.2 **Laboratory Control Sample (LCS)** -An LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Corrective action when LCS fails to meet control limits will be re-preparation and re-analysis of the batch.

9.1.3 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks cannot be used for MS/MSD analysis. If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< MDL
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	Statistical Limits ²
Laboratory Control Sample (LCSD)*	1 in 10 or fewer samples	Statistical Limits ²
Matrix Spike (MS) ¹	1 in 20 or fewer samples	Statistical Limits ²
MS Duplicate (MSD) ¹	1 in 10 or fewer samples	Statistical Limits ²

* LCS Duplicate (LCSD) is performed only when there are samples for a TMDL project included in the batch.

¹ The sample selection for MS/MSD are randomly selected, unless specifically requested by a client...predetermined by the extraction lab.

² Statistical control limits are updated annually and are updated into LIMS.

9.2 Instrument QC

9.2.1 Initial Calibration Verification (ICV) 90-110%

9.2.2 Continuing Calibration Verification (CCV) 90-110%

9.2.3 Calibration Acceptance Summary

Step	Standards	Type	Control Limit	Frequency
Initial Cal	0.40ppm, 0.20ppm, 0.10ppm, 0.050ppm, 0.020ppm, 0.010ppm, 0ppm	Linear cal	≥ 0.995	Daily
ICV	0.20ppm	Linear cal	90-110%	After calibration
ICB	0ppm	Linear cal	<MDL	After ICV
CCV	0.20ppm	Linear cal	90-110%	Every 10 samples
CCB	0ppm	Linear cal	<MDL	After CCV
LCS	0.20ppm	Linear cal	85-115%	Every 20 samples
MS/MSD	0.20ppm	Linear cal	85-115%	Every 10 samples

NOTE: ICV, LCS, MS & MSD is required to be from a different source standard than the calibration and CCV standards.

10.0 Procedure

10.1 Sample Preparation

- 10.1.1 Remove the samples from the storage refrigerator and allow the samples to come to room temperature.
- 10.1.2 Turn the spectrophotometer on and set the wavelength to 540nm. Allow 20 min. for warm up.
- 10.1.3 Prepare the calibration standards and spiking solution while the samples are equilibrating.
- 10.1.4 Aqueous samples require no additional preparation steps prior to analysis, however, if dissolved hexavalent chromium is required, filter the samples through a 0.45um syringe filter before analysis.
- 10.1.5 Soils, sediments, and sludges are extracted with a 1:10 DI Water leach prior to the colorimetric analysis (5g into 50ml DI H₂O).

10.2 Calibration

- 10.2.1 Before any instrument is used as a measurement device, the instrument response to known reference materials must be determined. The manner in which various instruments are calibrated depends on the particular type of instrument and its intended use. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.
- 10.2.2 Prepare the calibration standards from the 10mg/L intermediate using the following table for guidance. The calibration curve is valid for 3 months.

Cal Std	Stock Std (mg/L)	mL Stock	Final Vol (mL)	Cal Conc. (mg/L)
BLANK	NA	0	25	0
1	10	0.025	25	0.010
2	10	0.050	25	0.020
3	10	0.125	25	0.050
4	10	0.25	25	0.10
5	10	0.50	25	0.20
6	10	1.00	25	0.40

10.2.3 Make one 25mL aliquot of each calibration standard in labeled cups.

10.2.4 Color Reagent/pH Adjustment

10.2.4.1 If color reagent is used, add 0.5mL of the color reagent to the appropriate aliquots of the calibration standards. Add 0.5mL of concentrated sulfuric acid to each cup.

10.2.4.2 If the Hach pillow is used, add one ChromaVer 3 powder pillow to the appropriate aliquots of the calibration standards.

10.2.5 Wait 5 to 10 minutes for full color development but complete all absorbance readings within 30 minutes of the addition of the pillow, or color reagent and acid.

10.2.6 Transfer the blank aliquot into a cuvette and zero the instrument; i.e., adjust the absorbance to zero.

10.2.7 Transfer the remaining aliquots of the corresponding calibration standards with the added color and measure the absorbance at 540nm. Record the absorbance in the hexavalent chromium logbook.

10.2.8 Each calibration curve point is blank corrected by subtracting an initial reading from a final reading, creating a final result. The absorbance of the calibration blank is subtracted from each point's absorbance reading then plotted as a curve.

10.2.9 Calculate the correlation coefficient of a linear regression curve using an excel calculation spreadsheet. The concentration is plotted along the x-axis and the corresponding corrected absorbance is plotted as the y-coordinate. The correlation coefficient must be equal to or greater than 0.995. If these criteria are not met, the calibration must be repeated. Calculate the concentration of hexavalent chromium using the regression curve.

10.3

Sample Analysis

10.3.1 The analytical batch for aqueous samples consists of up to twenty (20) client samples and the associated quality control items. The quality control items consist of a method (reagent) blank, a lab control standard (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). If insufficient sample is available for the MS/MSD, the LCS is prepared in duplicate.

10.3.2 Analytical Sequence Summary

ANALYTICAL SEQUENCE
Initial Calibration – blank + six cal standards
Initial Calibration Verification (ICV) $\pm 10\%$
Initial Calibration Blank (ICB) <RL
Method Blank
LCS
Sample Readings, MS/MSD
Continuing Calibration Verification (CCV) $\pm 10\%$
Continuing Calibration Blank (CCB) <RL
Sample Readings, MS/MSD
CCV $\pm 10\%$
CCB <RL

10.3.3 MS/MSD (0.20ppm): Spike two 25mL aliquots of the sample chosen for the MS/MSD with 0.50mL of the 10mg/L intermediate standard.

10.3.4 Color Reagent/pH Adjustment

10.3.4.1 If the Hach pillow is used, add one Chroma Ver 3 powder pillow to each aliquot to be analyzed.

10.3.4.2 Check if pH is < 2 after addition of Hach pillow, using pH strips.

10.3.5 Wait 5 to 10 minutes for full color development but complete all absorbance readings within 30 minutes of the addition of the color reagent and acid, or Hach pillow.

10.3.6 If any sample or spiked sample absorbance exceeds the absorbance of the highest calibration standard (0.40mg/L), the sample and/or spiked sample must be diluted so that the absorbance falls within the working range of the instrument.

10.3.7 The samples are color corrected by reading the absorbance of a sample without added color reagent and subtracted from the absorbance of a sample with color reagent added.

10.3.8 Plot the corrected absorbance against the curve to obtain the final result. If the sample requires a dilution, take the absorbance of the diluted sample without color reagent and subtract this from the absorbance of the diluted sample with color reagent. Report the difference and plot it against the curve.

10.3.9 Sample data must be bracketed by acceptable QC.

11.0 Calculations / Data Reduction

11.1 Accuracy

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

11.3 Concentration = mg/kg or L = $\frac{C \times V \times D}{W}$

Where:

C = sample concentration in extract (ppm)

V = Volume of extract (mL)

D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4 Calibration Blank Correction = $\text{ABS}_{\text{calibration standard}} - \text{ABS}_{\text{calibration blank}}$

11.5 Sample Color Correction = $\text{ABS}_{\text{with color}} - \text{ABS}_{\text{without color}}$

11.6 The Reporting Limit (RL) for aqueous samples is calculated

$$RL(\text{mg} / \text{L}) = RL_{\text{gap}} \otimes DF$$

Where:

RL(gap) = RL in Appendices A & B of the TestAmerica Tampa's LQM.

DF = dilution factor.

The TestAmerica Tampa's LQM assumes that DF=1

11.7 Soil Samples

$$C(\text{mg} / \text{kg}, \text{dw}) = C_{\text{curve}} \otimes \frac{FV}{W \otimes \text{solids}} \otimes DF$$

Where:

C_{curve} = concentration of leachate from calibration curve, mg/L.

FV = final volume of the DI Leach
 W = weight of soil leached, kg
 Solids = decimal equivalent of percent solids - (percent solids)/100
 DF = dilution factor

The RL for soil samples is calculated:

$$RL(mg / kg, dw) = RLqap \otimes \frac{0.0025kg}{W \otimes solids} \otimes \frac{FV}{0.10L} \otimes DF$$

Where:

W = weight of sample leached, kg
 FV = final volume of leachate, L
 DF = dilution factor
 Solids = decimal equivalent of percent solids - (percent solids) /100

- 11.8 Determine the volume of standard to be prepared and the volume of the stock standard needed to make the spiking solutions. The following equation can be used:

$$Vi = \frac{Cf \otimes Vf}{Ci}$$

Where:

Vi = volume of stock standard needed to prepare the spiking solution (mL)
 Ci = concentration of stock solution (ug/mL)
 Cf = concentration of spiking solution to prepare (ug/mL)
 Vf = volume of spiking solution to prepare (mL)

The concentration can be expressed in whatever terms the analyst finds most convenient - ug/L, ug/mL, mg/L, etc. The units must be the same for Ci and Cf.

The Tampa's QAM assumes W=0.0025 g; solids=1.0, FV=0.10 L, and DF=1

12.0 Method Performance

12.1 Method Detection Limit Study (MDL) - The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined accordance with 40 CFR, Chapter 1, Part 136, Appendix B and with reference to the laboratory's MDL procedure in Section 20 of the Quality Assurance Manual. An MDL reflects a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Demonstration of Capabilities - Each analyst must perform an Initial Demonstration of Capability (IDOC) in accordance with the procedure outlined in Section 20 of the Quality Assurance Manual.: The evaluation of the IDOC data should be completed prior to the analysis of samples. A Continuing Demonstration of Capability (CDOC) must be performed annually or whenever there is a significant change in the instrument parameters or the associated method.

12.3 Training Requirements – Each analyst's required employee training (such as orientation to the laboratory's policies and procedures and in-house method training) are outlined in Section 18 of the Quality Assurance Manual.

13.0 Data Assessment And Acceptance Criteria For Quality Control Measures

13.1 The following represent data assessment for samples and acceptance criteria for QC measures. Corrective actions for any failure in QC measures are shown in Section 13.

13.2 QC sample acceptance criteria

13.2.1 **Method Blank.** No target analytes may be present in the method blank above the reporting limit. RL > 0.1 mg/L.

13.2.2 **Laboratory Control Sample (LCS).** 85 - 115% The analyte must be within established control limits for accuracy (%Recovery) and precision (RPD). Exceptions are allowed only with QA and project management approval.

13.2.3 **Matrix Spike/Matrix Spike Duplicate (MS/MSD).** 85 - 115% The analyte should be within established control limits for accuracy (%Recovery) and precision (%RPD). Deviations from this may be the results of matrix effects, which are confirmed by passing LCS/LCSD. No specific corrective actions are required in the evaluation of the MS/MSD results provided that the batch LCS is in control. Analysts should use sound judgment in accepting MS/MSD results that are not within control limits, especially if the LCS results are borderline. Check with supervisor, Lab Manager and or Project Manager on reporting out of control limits QC.

13.3 Sample result evaluation

13.3.1 **Dilutions:** If the response for any compound exceeds the working range of the analytical system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit in the upper half of the calibration range. The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

14.0 Corrective Actions For Out Of Control Data

14.1 **Method Blank.** The samples in the batch associated to the defective method blank are evaluated. If the analyte found in the method blank is confirmed to not be present in one or more of the associated samples at any level, the contamination did not affect those samples. This represents a non-impact situation and no specific corrective action is taken. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements. If the analyte found in the method blank is confirmed to be present in one or more of the associated samples, the concentrations are compared. If the concentration in the method blank exceeded 10% of concentration found in one or more samples, the

prescribed corrective action is to re-analyze all affected samples. If the concentration in the method blank was less than 10% of the concentration found in one or more samples, the sample can be reported by qualifying the affected analytes. A nonconformance memo (NCM) is written and discussed with the laboratory supervisor and Project Management for evaluation against project requirements.

14.2 Laboratory control sample. If the analyte is out of control for accuracy (85 - 115%), the associated samples are evaluated. If the recovery is biased high and the associated samples have no positive results for that analyte, a non-impact situation ensues. A nonconformance memo (NCM) is written to notify project management of the situation for evaluation against project requirements. If the recovery is biased high and the associated samples have positive results for that analyte, the prescribed corrective action is reanalysis of the affected sample(s). If the recovery is biased low and the samples have no positive results for that analyte, the prescribed corrective action is reanalysis of the affected sample(s). If the analyte is out of control for precision (RPD), the associated samples are evaluated. All decisions about corrective action(s) are made in consultation with the project manager. If there are no positive results in one or more samples, a non-impact situation ensues for those samples. A nonconformance memo is written to notify project management of the situation for evaluation against project requirements.

14.2.1 If there are positive results for one or more analytes, the likelihood of poor reproducibility increases and corrective action must be evaluated. A nonconformance memo is written and discussed with the laboratory supervisor and Project Management of the situation for a project decision on whether the affected sample(s) should be reanalyzed.

15.0 Contingencies For Handling Out-Of-Control Or Unacceptable Data

15.1 Method blanks. If there is insufficient sample to perform re-analysis; the analyst must notify the project manager for consultation with the client. In this situation, all associated samples are flagged with a "I" qualifier and appropriate comments in the narrative.

15.2 LCS/LCSD. If the batch is not reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Acceptable matrix spike recoveries are not sufficient justification to preclude re-extraction of the batch. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation. An NCM will have to be filled out stating this problem and or a solution.

15.3 Insufficient sample. If there is insufficient sample to repeat the analysis, the situation is discussed with the project manager for consultation with the client and documentation is provided in an NCM.

16.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

17.0 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Tampa's current revision of SOP TP-HAZ-001 *Waste Management*). The following waste streams are produced when this method is carried out.

- Acidic waste generated by the analysis: Samples are neutralized and disposed in sewer system if non-hazardous

18.0 References / Cross-References

- 18.1 *Standard Methods for the Examination of Water and Wastewater*, 21st Edition; American Public Health Association: Washington, DC, 2005
- 18.2 EPA Method 140.1, Revision 2.0 (40 CFR Part 141 December 1994)
- 18.3 TestAmerica's *Environmental Health & Safety Manual* CW-E-M-001, Most current revision.
- 18.4 Tampa's *Quality Assurance Manual* (TP-QAM), most current revision)
- 18.5 *Test Methods for Evaluating Solid Waste*; Third Edition, SW-846; US EPA Office of Solid Waste and Emergency Response: Washington, DC
- 18.6 Reference from Hach and EPA concerning acceptability of the ChromaVer 3 powder pillows. The acceptability of the powder pillows was confirmed by Wayne Turnball of EPA Region IV QA Office (3/3/97)
- 18.7 Hach Method No. 8023 for Chromium, Hexavalent
- 18.8 Acceptance/Approval Letter- February 17, 1983 letter to EPA Region 5 from Robert L. Booth
- 18.9 TestAmerica Tampa SOP's:
 - 18.9.1 TP-HAZ-001 *Waste Management*
 - 18.9.2 TP-AN-005: Definitions, Terms, and Acronym
 - 18.9.3 TP-AN-016 Glassware Cleaning Procedures
 - 18.9.4 TestAmerica Tampa SOP TP-AN-006: *Analytical Batching*
 - 18.9.5 TestAmerica Tampa SOP TP-AN-004: Standard Materials and Reagent Traceability

18.9.6 TestAmerica Tampa SOP TP-AN-009: Conductivity Checks for Laboratory
Deionized Water

19.0 Method Modifications:

None

20.0 Attachments

No additional tables or diagrams are included for this method

21.0 Revision History

- Revision 3 July, 2007
- Revision 4, 15 February 2009
 - Updated references for detection limits from QA Manual to TALS method limit groups
 - Changed expiration of working solutions from 90 days to daily
 - Changed expiration of calibration curve from 6 months to 3 months.
 - Minor spelling and grammatical changes
 - Added Revision history section
 - Updated reference section
- Revision 5, 26 October 2009
 - Updated to new format
 - Added correction to Procedure and Calculation Sections