

OSHA Method ID-215, Hexavalent Chromium

Hexavalent Chromium

CAS number:	18540-29-9
OSHA PEL:	5.0 µg/m ³ 8-Hour Time-Weighted Average (TWA) (2.5 µg/m ³ Action Level)
Procedure:	Chromium plating operations are sampled by drawing workplace air through 37-mm polystyrene cassettes containing NaOH coated binderless quartz fiber filters (NaOHqz). Alternatively, 37-mm or 25-mm diameter polyvinyl chloride (PVC) filters with cellulose backup pads (BUP) in polystyrene cassettes can be used. All other chromium operations are sampled by drawing workplace air through 37-mm or 25-mm polystyrene cassettes containing PVC filters with BUP. Wipe samples are collected by using firm hand pressure to move a 37-mm PVC filter across the surface of interest, or a 37-mm binderless quartz fiber filter (QFF) on rough surfaces. Samples are extracted with 10% Na₂CO₃/2% NaHCO₃ (BE) and phosphate buffer/magnesium sulfate (PBM) solutions. Samples from paint operations require a second extraction with 5% NaOH/7.5% Na₂CO₃ (SPE) and PBM solutions. Samples are analyzed by ion chromatography (IC) with post-column derivatization using 1,5-diphenylcarbazide and UV-vis detection at 540-nm.
Recommended sampling time and sampling rate:	480 min at 2.0 L/min (960 L)
Limit of quantitation:	0.020 µg/m ³ (8-Hour TWA) for 37-mm PVC filter 0.025 µg/m ³ (8-Hour TWA) for 25-mm PVC filter 0.33 µg/m ³ (8-Hour TWA) for NaOHqz filter 0.18 µg/sample for 37-mm QFF filter
Reporting limit:	0.050 µg/m ³ for 37-mm and 25-mm PVC filter 0.33 µg/m ³ for NaOHqz filter and 0.33 µg/sample for 37-mm QFF filter
Special requirements:	All samples must be shipped overnight to the OSHA Technical Center (OTC) within 24 hours of sampling or as soon as possible. Air and wipe samples collected on PVC filters for welding operations must be analyzed within 8 days of sampling. For chromium plating operations, air and wipe samples collected on PVC filters must be analyzed within 6 days of sampling and wipe samples on QFF must be analyzed within 10 days of sampling or stabilized immediately upon arrival as described in Section 3.5.
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1 Introduction

This method harmonizes the sampling, preparation, and analysis of hexavalent chromium [Cr(VI)] collected on four different media for air samples and wipe samples. Validation data for each medium are described in the relevant appendix. The methodologies described in this method replace OSHA Method ID-215 version 2¹ for hexavalent chromium air sampling and OSHA Method W4001² for hexavalent chromium wipe sampling. Changes to IC instrumentation, analytical parameters, sample extraction temperature, extraction apparatus and procedures have been made to allow standardized collection and analysis of hexavalent chromium sampled on different media.

The sampling media used in this method are: One-molar sodium hydroxide coated 37-mm diameter binderless quartz fiber filter media (NaOHqz); 37-mm diameter polyvinyl chloride (PVC) filters; 25-mm diameter polyvinyl chloride (PVC) filters; 37-mm diameter binderless quartz fiber filters (QFF).

2 Sampling Procedure

All safety practices that apply to the work area being sampled should be followed. The sampling equipment should be attached to the worker in such a manner that it will not interfere with work performance or safety. It is very important to record the sampling operation (e.g., painting, chrome plating, welding).

2.1 Apparatus

Air sampling

A personal sampling pump, calibrated to within $\pm 5.0\%$ of the recommended flow rate with a representative sampling device in-line, is used to draw air through a filter cassette. When possible, sample over the duration specified for hexavalent chromium.

Samples from chromium plating operations are collected on NaOHqz filters. NaOHqz filters are 37-mm diameter binderless quartz fiber filter, 0.38 mm thick (SKC Inc., Catalog no. 225-1827) coated with 1.0 M NaOH as described in Appendix C, Section 12. The NaOHqz filters are placed into two-piece cassettes and sampled with a closed face. It is not necessary to use a BUP with NaOHqz filter. It is important to submit a blank sample from the same lot with each set of samples as there is a background amount of Cr(VI). The amount of Cr(VI) found on QFF filters varied from different manufacturers and lots, with amounts of 0 to 50 ng.¹

Samples from chromium plating operations can also be collected on 37-mm diameter, 5- μ m pore size PVC filters (SKC Inc., Catalog no. 225-5-37) with BUPs, or 25-mm diameter, 5- μ m pore size PVC filters (SKC Inc., Catalog no. 225-5-25) with BUPs. The filters are placed into two-piece cassettes with BUPs and sampled with a closed face.

Samples from other operations are collected on 37-mm or 25-mm diameter PVC filters, 5- μ m pore size with BUPs as described above.

Wipe sampling

Wipe samples are collected with 37-mm diameter PVC filters, 5- μ m pore size (SKC Inc., Catalog no. 225-5-37).

On rough surfaces, wipe samples are collected with 37-mm diameter binderless quartz fiber filters (QFF), 0.38 mm thick (SKC Inc., Catalog no. 225-1827).

Labeled vials, 20-mL glass scintillation vials or other appropriately sized glass vials with PTFE lined caps, one for each wipe sample.

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2.2 Reagents

None required

2.3 Technique

Air Sampling

Immediately before sampling, remove the top and end plugs from the cassette. All filters should be from the same lot.

Attach the cassette to the sampling pump so that it is in an approximately vertical position with the inlet facing down during sampling near the worker's breathing zone. Position the sampling pump, cassette, and tubing so that it does not impede work performance or safety of the employee being sampled. Sampled air should not pass through any hose or tubing before entering the sampling cassette.

After sampling the appropriate time, remove the sampler, and replace the top and end plugs. Wrap each sample end-to-end with an OSHA Form-21 seal as soon as possible. The minimum sampling volume recommended is 200 L.

Submit at least one blank sample with each set of samples, making sure the blank filter is from the same lot as the filters used for sampling. Handle the blank sampler in the same manner as the samples except draw no air through it.

Record sample air volume (liters) for each sample, identify the type of operation, and identify any potential interferences. It is important to identify the operation because it affects the sample preparation procedure that is used.

All samples must be shipped overnight to the OSHA Technical Center (OTC) within 24 hours of sampling or as soon as possible.

Wipe Sampling

Labeled 20-mL glass scintillation vials or other appropriately sized glass vials with PTFE lined caps, one for each sample. Prepare enough vials, each labeled with a unique number.

Wear a new pair of clean gloves for each sample. PVC gloves are suggested for sampling Cr(VI) based on a review of a glove manufacturer's chemical resistance and degradation information. Do not wear powdered gloves.

Record the sample vial number and the location where the sample is taken.

Remove the filter from the container with clean PTFE-coated tweezers or plastic tweezers. Do not use metal tweezers to handle the filters as this could contaminate the filters.

Depending on the purpose of the sample, it may be useful to determine the surface loading of the contamination (e.g., in micrograms of analyte per area). For these samples, it is necessary to record the area of the surface wiped (e.g., 100 cm²). This would not be necessary for samples taken to simply show the presence of the contaminant.

Surfaces should not be wetted with water as the water will allow any metal interferences to interact with the Cr(VI), thereby affecting the results.

Firm pressure should be applied when wiping. Start at the outside edge and progress toward the center making concentric squares of decreasing size. Fold the filter in half with the contaminant side inward and repeat.

Without allowing the filter to contact any other surface, fold the filter with the exposed side inward. Place the filter in the sample vial, cap and wrap each sample end-to-end with an OSHA Form-21 seal.

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Record sample location, employee names, surface area (if pertinent), work description, type of operation, personal protective equipment, and any other necessary information, along with any potential interferences.

Submit the samples with at least one blank wipe filter, treated in the same fashion, but without wiping, to the OTC together as soon as possible after sampling. Ship any bulk samples separated from the surface samples.

All samples must be shipped overnight to the OSHA Technical Center (OTC) within 24 hours of sampling or as soon as possible.

3 Analytical Procedure

3.1 Apparatus

- Ion chromatograph with a UV-vis detector and a post-column reagent delivery system (IC system)
- Balances used for weighing reagents (e.g., Mettler Toledo model XP205 series balance and an Ohaus Adventurer Pro AV4101C balance)
- A hot block (e.g., J-Kem MaxQ2000) with a shaker and temperature controller for extracting samples
- A centrifuge (e.g., Thermo Scientific SORVALL ST 16)
- An ultrasonic cleaner (e.g., VWR model 97043-944) for assisting in dissolving reagents and degassing eluents
- A water purification system (e.g., Thermo Scientific Barnstead GenPure UV×CAD plus bench version) to purify the water
- Laboratory solvent filtration apparatus vacuum system (e.g., funnel capacity 300 mL, bottle capacity 2 L) for filtering eluents
- Pipettes (e.g., Eppendorf 10 µL, 100 µL, 1000 µL and 5 mL) for preparing calibration standards as well as to desorb samples
- 20-mL glass scintillation vials (e.g., Wheaton 20-mL glass scintillation vials) with PTFE liner caps for holding and extracting sample filters and as the glassware for preparing calibration standards
- Vial racks for 20-mL glass scintillation vials
- Class A volumetric flasks (100 mL, 500 mL, 1L and 2L) for preparing calibration standard stocks and reagents
- A vacuum filtration apparatus with 0.22-µm nitrocellulose membranes for filtering the eluents
- Polyethylene bottles (1L and 2L) for storing extraction solutions
- Volumetric cylinders (25 mL, 50 mL) for measuring reagent solutions
- Beakers (25 mL, 100 mL, 250 mL, 400 mL, and 1L) for dissolving reagents
- Plastic transfer pipettes (5 mL) for wetting PVC filters to wipe cassette walls and transfer solutions
- Clean PTFE-coated tweezers or plastic tweezers
- 37-mm diameter PVC filters, 5-µm pore size (SKC Inc., Catalog no. 225-5-37) for wiping 37-mm diameter cassette walls
- 25-mm diameter PVC filters, 5-µm pore size (SKC Inc., Catalog no. 225-5-25) for wiping 25-mm diameter cassette walls

Under no circumstances should chromic acid cleaning be used. It is best if glassware used for the analysis of Cr(VI) should be reserved for this purpose only so that maximum analytical sensitivity and absence of outside contamination can be maintained.

3.2 Reagents

- Deionized (DI) water, 18 MΩ-cm
- Sodium carbonate (Na₂CO₃) [CAS no. 497-19-8], reagent grade
- Sodium bicarbonate (NaHCO₃) [CAS no. 144-55-8], reagent grade
- Sodium hydroxide (NaOH) [CAS no. 1310-73-2], reagent grade
- Potassium dichromate (K₂Cr₂O₇) [CAS no. 7778-50-9], reagent grade

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- Magnesium sulfate (MgSO_4) [CAS no. 7487-88-9], anhydrous, reagent grade
- Ammonium sulfate $[(\text{NH}_4)_2\text{SO}_4]$ [CAS no. 7783-20-2], reagent grade
- Ammonium hydroxide (NH_4OH) [CAS no. 1336-21-6], 29% solution
- 1,5-Diphenylcarbazide (DPC) [CAS no. 140-22-7], reagent grade
- Methanol (CH_3OH) [CAS no. 67-56-1], HPLC grade reagent
- Sulfuric acid (H_2SO_4) [CAS no. 7664-93-9], concentrated
- Potassium phosphate dibasic (K_2HPO_4) [CAS no. 7758-11-4], reagent grade
- Potassium phosphate monobasic (KH_2PO_4) [CAS no. 7778-77-0], reagent grade

3.3 Reagent Preparation

Buffer/extraction (BE) solution (2% NaHCO_3 with 10% Na_2CO_3): Add about 700 mL of DI water to a 1-L beaker, add 20 g of NaHCO_3 , stir to dissolve or use an ultrasonic bath, then add 100 g of Na_2CO_3 . When the salts are dissolved, transfer to a 1 L volumetric flask and bring to the mark with DI water. Store the solution in a polyethylene bottle. Prepare this buffer solution every 6 months.

Spray-paint extraction (SPE) solution (5% NaOH + 7.5% Na_2CO_3): Dissolve 25 g of NaOH and 37.5 g of Na_2CO_3 in a 400-mL beaker with about 350 mL DI water, then transfer to a 500 mL volumetric flask and bring up to the mark with DI water. Allow the solution to cool to room temperature and then dilute to the mark with DI water. Transfer and store this solution in a tightly capped polyethylene bottle. Use this solution only for the second extraction of samples from spray-paint operations. Prepare this buffer solution every 6 months.

Magnesium sulfate solution: Place about 150 mL of DI water in a 250-mL beaker, then add 24.8 g of anhydrous magnesium sulfate. Stir or use an ultrasonic bath to dissolve, then transfer to a 250-mL volumetric flask and bring up to the mark with DI water. Store this solution in a tightly capped polyethylene bottle. Prepare this buffer solution every 6 months.

Phosphate buffer (PB) solution (0.5 M KH_2PO_4 with 0.5 M K_2HPO_4): Place about 400 mL of DI water in a 400-mL beaker, slowly add while stirring 34 g KH_2PO_4 (monobasic) and 43.7 g anhydrous K_2HPO_4 (dibasic) (or 57 g $\text{K}_2\text{HPO}_4 \cdot 3\text{H}_2\text{O}$), stir to dissolve or use an ultrasonic bath. Transfer to 500 mL volumetric flask and dilute up to the mark with DI water. Prepare this buffer solution every 6 months.

Phosphate buffer/Magnesium sulfate solution (PBM): 1-part MgSO_4 solution to 2-parts phosphate buffer (PB), i.e., 5 mL MgSO_4 solution is added to 10 mL Phosphate buffer, mix well. Prepare this solution immediately before each analysis.

Buffer Extraction/Phosphate buffer/ Magnesium sulfate solution (DBE): Add 50 mL of BE solution, 15 mL of PBM solution, and 35 mL DI water into a 100-mL beaker, mix well. Magnesium hydroxide will form and slowly precipitate from solution. Allow the precipitation to settle for at least 4 hours. Transfer the "clear" supernatant to 20-mL glass scintillation vials for use in preparation of working standards. Try to avoid transferring any precipitate as it will clog the IC system. This solution is also used for wetting the PVC filters to wipe sample cassette walls. Prepare this buffer solution every 3 months.

Eluent [250 mM $(\text{NH}_4)_2\text{SO}_4$ with 100 mM NH_4OH]: Place about 500 mL of DI water in a 2-L volumetric flask, add 13 mL of 29% ammonium hydroxide, then add 66 g of ammonium sulfate and mix well. Dilute to the mark with DI water. The eluent is filtered through a vacuum filtration system before degassing in an ultrasonic bath for 15 min. Transfer the solution to an IC eluent container. Prepare the eluent every 3 months.

Post-column derivatization reagent [2.0 mM 1,5-diphenylcarbazide (DPC) in 90:10 (1 N H_2SO_4 : methanol)]: Add about 600 mL DI water into a 2-L eluent bottle. Then add 28 mL concentrated sulfuric acid, mix well and allow the solution to cool. Use a 150-mL beaker to dissolve the 0.5 g of DPC in 100 mL of methanol, sonicate if necessary to aid dissolution.

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When the sulfuric acid solution cools to room temperature, add the DPC/methanol solution to an eluent bottle and bring up to the 1 L mark with DI water. Mix well. This solution is stable for 5 days.

Eluent (50 mM NaOH): Add about 500 mL of DI water in a 2-L volumetric flask, add 4 g sodium hydroxide, swirl to dissolve, then dilute up to the mark with DI water. The eluent is filtered through a vacuum filtration system before degassing in an ultrasonic bath for 15 min. Transfer the solution to the eluent container of the IC system. Prepare the eluent every 3 months.

3.4 Standard Preparation

Prepare a stock standard containing about 124 µg/mL of Cr(VI) by dissolving approximately 0.03518 g of $K_2Cr_2O_7$ in 100 mL of DI water. For example: the calculation for a stock standard is: $[0.03518 \text{ g } K_2Cr_2O_7 \times 1000 \text{ mg/g} \times 1000 \text{ µg/mg} \times 0.3535 \text{ (the mass fraction of Cr in } K_2Cr_2O_7\text{)}]/100 \text{ mL} = 124.4 \text{ µg/mL Cr(VI)}$. The stock standards expire after one year.

Make dilutions of the stock standard with DBE solution to matrix-match standards with samples. Prepare five calibration standards. Prepare calibration level 2, 4, and 5 by spiking 10 µL, 30 µL, and 50 µL of the stock solution into 5 mL DBE in 20-mL scintillation vials and mix well. Prepare calibration level 3 by spiking 40 µL of the stock solutions into 10 mL DBE in 20-mL scintillation vial. Prepare calibration level 1 by spiking 20 µL of calibration level 5 solution into 5 mL DBE in a 20-mL scintillation vial. Mix well. Transfer the five calibration standards to 2-mL amber glass autosampler vials. The calibration standard concentrations are 0.0049 µg/mL, 0.2482 µg/mL, 0.4955 µg/mL, 0.7417 µg/mL, and 1.2313 µg/mL. Prepare these diluted analytical calibration standards every 3 months. Recap the punctured autosampler vials that contain standard dilutions at least once a week. If sample concentrations fall outside the range of prepared standards, dilute samples with DBE solution and reanalyze the diluted samples.

3.5 Sample preparation

Air samples or wipe samples from chromium plating operations collected on PVC filters must either be analyzed within 6 days of sampling or be stabilized immediately upon arrival at the analytical laboratory (Appendix A, Sections 4 and 9). Samples are stabilized by removing the filters from the cassettes and placing them into labeled 20-mL glass scintillation vials, then add 5 mL of BE. The interior walls of the sampling cassette should be wiped with the rough side of a PVC filter that has been wet with a drop of DBE, then placed in the same labeled vial as the sample. Acids from chromium plating operations are neutralized by the BE solution, and this allows samples to be stored for up to two weeks before analysis. Just prior to heating the samples for the hot extraction, add 1.5 mL of PBM. The order of BE and PBM solution addition is reversed here because the main interference at room temperature is acids. Cr(III) does not convert to Cr(VI) at room temperature. The addition of PBM prior to heating precipitates out the Cr(III), preventing it from forming Cr(VI) during heating.

Wipe samples from chromium plating operations collected on QFF filters must either be analyzed within 10 days of sampling or be stabilized immediately upon arrival at the analytical laboratory (Appendix D, Section 4.2). Samples are stabilized by placing them in labeled 20-mL glass scintillation vials containing 5 mL of BE which allow samples to be stored for up to two weeks. Samples are analyzed following the same procedure as stabilized PVC filter as above.

Primary extraction for all filters

PVC filters, NaOHqz filters, and QFF filters are digested and analyzed following the same procedure. The primary extraction procedure consists of several steps that must be performed in the given order. These steps result in the formation of precipitates that trap metal interferences and prevent them from reacting with Cr(VI). The PVC filters and QFF used to sample chromium plating operations can be stabilized as described in this Section.

Set the hot block temperature at 93.5 °C.

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Each sample is prepared as follows: remove the filter from the cassette with tweezers and place it in a labeled 20-mL glass scintillation vial with PTFE-lined cap. Wipe the inside walls of the cassette with the rough side of a PVC filter wetted with a drop of DBE and place the filter into the same vial as the sample. The interior walls of the blank-sample cassettes should also be wiped.

Add 1.5 mL of PBM solution to each labeled 20-mL glass scintillation vial and swirl to wet the filter. Next, add 5 mL of BE solution and mix the solution well before proceeding to the next sample. It is important to add the PBM solution first because the freshly precipitated magnesium hydroxide that forms upon the addition of the BE solution suppresses interference from other metal ions. This precipitation occurs immediately upon mixing, so it is important that both sides of the filter are wet.

Heat chromium plating, welding, and other samples at 93.5 °C for 30 minutes while slowly shaking at 120 rpm. Heat paint samples at the same temperature for 90 minutes while slowly shaking at 120 rpm. Carefully watch the process to prevent the samples from boiling or evaporating. If the samples boil or evaporate to dryness, the Cr(VI) will change to Cr(III). Remove the vials from the hot block and allow the samples to cool to room temperature. Then add 3.5 mL of DI water by pipette to dilute the samples into 10 mL. Each vial is shaken vigorously to mix well. Allow the samples sit for at least 4 hours to allow the precipitate to settle or centrifuge at 3000 rpm for 3 minutes. Carefully transfer the supernatant to an autosampler vial. Centrifuge autosampler vials at 3000 rpm for another 3 minutes. Make sure no precipitates are transferred into autosampler vials because they will clog the IC system.

Secondary extraction for spray paint samples

The hardened matrix of paint samples encapsulates Cr(VI) and prevents it from being extracted. Evidence indicates stronger base extractions can recover Cr(VI) in specific paint matrices. Due to the resistant properties of some industrial paints, two separate extractions are necessary to break down the paint matrix and ensure complete recovery of all Cr(VI).

Extract filter samples and cassette wipes from spray painting operations a second time to further break apart the paint matrix, thereby freeing the Cr(VI) for analysis. After the primary extraction is completed and the BE/PBM solution has been removed, extract the PVC filter in 20-mL glass scintillation vials with PTFE liner cap again by adding 1.5 mL of PBM solution and then 5 mL of SPE solution. Swirl the vial slowly until the white precipitate forms.

Heat the solution at 93.5 °C in the hot block for the second time while slowly shaking at 120 rpm for 90 min. Allow additional extraction time for heavily loaded samples. Do not allow the solution to boil or evaporate. Allow the secondary extraction solution to cool to room temperature. Then add 3.5 mL of DI water by pipette to dilute the samples to 10 mL. Each vial is shaken vigorously to mix well. Allow the samples to sit for 4 hours for the precipitation to settle or centrifuge at 3000 rpm for 3 minutes. Carefully transfer the supernatant to an autosampler vial. Then centrifuge autosampler vials at 3000 rpm for another 3 minutes. Make sure no precipitates are transferred because they will clog the IC system.

3.6 Analysis

Analyze samples using an IC instrument and the analytical parameters described below. See Figure 1 for an example chromatogram obtained from a standard containing Cr(VI) mass concentration equivalent to sampling for the recommended time at OSHA 8-hour TWA value. Confirm the presence of hexavalent chromium when an OSHA Permissible Exposure Limit (PEL) value has been exceeded, as described in OSHA Method ID-215, Section 3.8.

IC Conditions

column:	IonPac AS7 analytical column (250-mm × 4-mm i.d.) and IonPac AS7 guard column (50-mm × 4-mm i.d.) at ambient temperature (25 °C)
eluent:	250 mM (NH ₄) ₂ SO ₄ with 100 mM NH ₄ OH

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eluent flow rate: 1.4 mL/min
eluent pump pressure: ~1900 psi
post-column derivatization solution: 2.0 mM DPC in 90:10 1 N H₂SO₄: methanol
post-column derivatization flow rate: 0.5 mL/min
post-column derivatization pump pressure: ~1200 psi
injection size: 50 µL
UV detector: 540 nm
run time: 5.5 min
retention times: 3.7 min

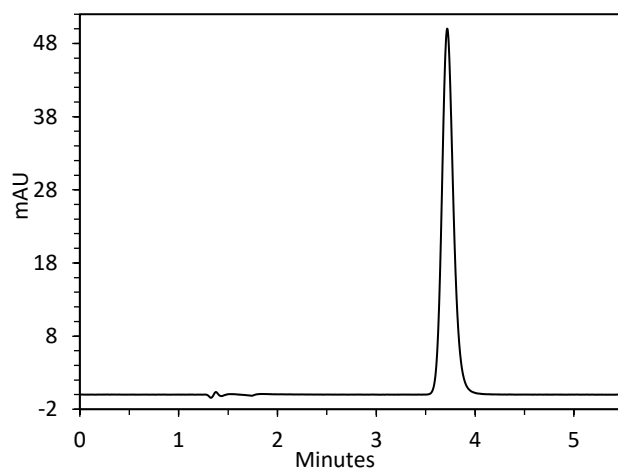


Figure 1. A chromatogram of 0.5 µg/mL hexavalent chromium.

An external standard (ESTD) calibration procedure is used to prepare a calibration curve from the analysis of analytical standards. Bracket samples with analytical standards. Construct a x^{-1} weighted-least-squares linear regression curve by plotting response of standard injections versus micrograms of analyte per sample. See Figure 2 below for an example calibration curve obtained from injection of calibration standards at concentrations defined in OSHA Method ID-215, Section 3.4.

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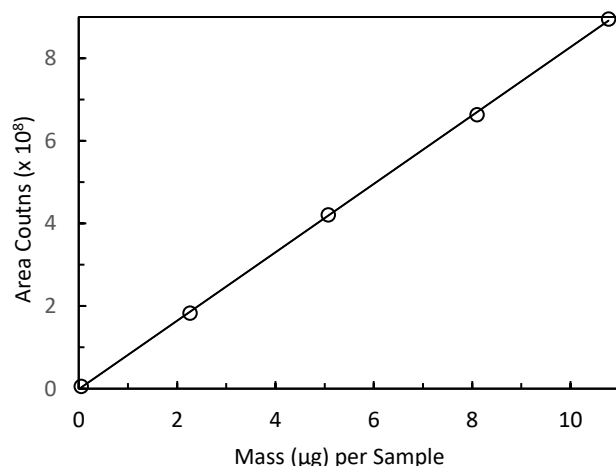


Figure 2. Example calibration curve for hexavalent chromium ($y = 8.23 \times 10^7 x + 1.68 \times 10^6$, $w_i = x^{-1}$).

The possibility of a co-eluting species that does not react with DPC can be tested by injecting the sample with no post-column derivatizing agent added. To confirm the sample without the addition of the post-column derivatizing solution, a solution of 0.1 N H₂SO₄ with methanol (90:10) is substituted for the derivatizing DPC solution, and the sample is rerun under the same conditions as in Section 3.6. If a peak appears at the same retention time as the hexavalent chromium, it is an interference.

3.7 Calculations

Always perform one blank correction for each NaOHqz medium extraction result. One blank correction refers to subtraction of the same lot media blank mass prior to concentration calculation.

For Air Samples

The result for the BE/PBM extraction is:

$$A_{BE/PBM} = Cr(VI)_{BE/PBM} \times Sol Vol_{BE/PBM} - Cr(VI)_{BE/PBMblank} \times Sol Vol_{BE/PBMblank}$$

Where:

$A_{BE/PBM}$	= amount found on filter (µg) from BE/PBM extraction
$Cr(VI)_{BE/PBM}$	= total µg/mL Cr(VI) on filter from BE/PBM extraction
$Sol Vol_{BE/PBM}$	= sample solution volume (usually 10 mL, but if dilution was necessary, make the appropriate calculations here)
$Cr(VI)_{BE/PBMblank}$	= total µg/mL Cr(VI) on blank from BE/PBM extraction
$Sol Vol_{BE/PBMblank}$	= blank solution volume (10 mL)

The result for the SPE secondary extraction (spray paint operations only) is:

$$A_{SPE/PBM} = Cr(VI)_{SPE/PBM} \times Sol Vol_{SPE/PBM} - Cr(VI)_{SPE/PBMblank} \times Sol Vol_{SPE/PBMblank}$$

Where:

$A_{SPE/PBM}$	= amount found on filter (µg) from SPE/PBM extraction
$Cr(VI)_{SPE/PBM}$	= total µg/mL Cr(VI) on filter from SPE/PBM extraction
$Sol Vol_{SPE/PBM}$	= sample solution volume (usually 10 mL, but if dilution was necessary, make the appropriate calculations here)

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$$\begin{aligned}Cr(VI)_{SPE/PBMblank} &= \text{total } \mu\text{g/mL Cr(VI) on blank from SPE/PBM extraction} \\Sol Vol_{SPE/PBMblank} &= \text{blank solution volume (10 mL)}\end{aligned}$$

The total amount of hexavalent chromium for the sample is:

$$A = A_{BE/PBM} + A_{SPE/PBM}$$

Where:

$$\begin{aligned}A &= \text{total } \mu\text{g Cr(VI) in sample after blank correction} \\A_{BE/PBM} &= \text{amount found from filter (}\mu\text{g) BE/PBM extraction} \\A_{SPE/PBM} &= \text{amount found from filter (}\mu\text{g) SPE/PBM extraction}\end{aligned}$$

Cr(VI) air concentration is:

$$Cr(VI), \frac{\mu\text{g}}{\text{m}^3} = \frac{A}{V}$$

Where:

$$\begin{aligned}A &= \text{total } \mu\text{g Cr(VI) in sample after blank correction (}\mu\text{g)} \\V &= \text{air volume (m}^3\text{)}\end{aligned}$$

For painting samples, BE and SPE extraction amounts are added only if results are above the reporting limit. When the sampling volume is less than the recommended minimum sampling volume of 200 L or samples are extracted after the established stability time, a comment shall be included to inform Certified Safety and Health Officials (CSHO) of a potential loss or inaccuracy of Cr(VI) in the sample.

For Wipe Samples

The amount of Cr(VI) per sampler is obtained from the appropriate calibration curve in terms of micrograms per sample. This amount is then adjusted by subtracting the amount (if any) found on the blank using the following formula. For painting samples, the second extraction results from the filter were also calculated with the following formula, then both results are added together if both are above the reporting limit.

$$M_s = M - M_B$$

Where:

$$\begin{aligned}M &= \text{micrograms per sample} \\M_s &= \text{the mass recovered from the sampled surface (}\mu\text{g)} \\M_B &= \text{the mass found on the blank (}\mu\text{g)}\end{aligned}$$

3.8 Qualitative Analysis

When necessary, the identity of an analyte peak can be confirmed by ion chromatography using different analytical columns and analytical parameters described below. Confirm the presence of Cr(VI) by matching the retention time and quality control (QC) sample recoveries within a $\pm 10\%$ difference between the two analyses.

IC Conditions

column:	IonPac AS11 analytical column (250-mm $\times$ 4-mm i.d.) and IonPac AS11 guard column (50-mm $\times$ 4-mm i.d.) at ambient temperature (25 °C)
eluent:	50 mM NaOH
eluent flow rate:	1.0 mL/min

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eluent pump pressure:	~700 psi
post-column derivatization solution:	2.0 mM DPC in 90:10 1 N H ₂ SO ₄ : methanol
post-column derivatization flow rate:	0.22 mL/min
post-column derivatization pump pressure:	~600 psi
UV detector:	540 nm
injection size:	50 µL
retention times:	2.7 min

3.9 Interferences (analytical)

Any compound that produces a detector response at 540-nm and has a similar retention time as Cr(VI) is a potential interference. If potential interferences were reported, they should be considered before samples are extracted. Generally, chromatographic conditions can be altered to separate any interference from the analyte.

When necessary, the identity of an analyte peak may be confirmed with additional analytical data, such as analysis with another analytical column (OSHA Method ID-215, Section 3.8). The presence of a co-eluting species that does not react with DPC can be tested by injecting the sample with no post-column derivatizing reagent being added.

References

1. Ku, J., Eide, M. Hexavalent Chromium (OSHA Method ID-215 version 2), 2006. United States Department of Labor, Occupational Safety and Health Administration website. www.osha.gov (accessed March 2026).
2. Eide, M. Hexavalent Chromium (OSHA Method W4001), 2001. United States Department of Labor, Occupational Safety and Health Administration website. www.osha.gov (accessed March 2026).

Appendix A, 37-mm Diameter Polyvinyl Chloride (PVC) Filter

OSHA PEL: 5.0 $\mu\text{g}/\text{m}^3$ 8-Hour TWA (2.5 $\mu\text{g}/\text{m}^3$ Action Level), General Industry, Shipyards, and Construction. See hexavalent chromium standard for compliance dates and special provisions (71 FR 10100-10385 or 29 CFR 1910.1026; 29 CFR 1915.1026; 29 CFR 1926.1126).

Recommended sampling time
and sampling rate: 480 min at 2.0 L/min (960 L)

Limit of quantitation: 0.02 $\mu\text{g}/\text{m}^3$ (8-Hour TWA)

Reporting limit: 0.05 $\mu\text{g}/\text{m}^3$

Author: James C. Ku, Mary Eide (1998, 2001, 2006), Yanqiu Sun (2026)

1 Introduction

1.1 Previous Methods used by OSHA for Sampling and Analysis of Hexavalent Chromium on 37-mm PVC filter

The methodologies described in this Appendix are based on OSHA Method ID-215 version 2¹ and OSHA Method W4001.² Both methods used Erlenmeyer flask and hot plate without temperature control as extraction apparatus and analyzed by ion chromatography with post-column derivatization using 1,5-diphenylcarbazide and UV-vis detection at 540-nm. Prior to OSHA Method ID-215, OSHA Method ID-103³ was used for sampling and analysis of hexavalent chromium [Cr(VI)]. OSHA ID-103 used 37-mm diameter PVC filters for air sampling, sample extraction with carbonate/bicarbonate buffer solution, and analysis by differential pulse polarography. The carbonate/bicarbonate buffer solution extraction converts both soluble and insoluble chromium-containing chemicals to soluble carbonate compounds and allows both chromium forms to be extracted. OSHA ID-215 utilized a more sensitive and specific ion chromatography analytical method. In addition to carbonate/bicarbonate extraction, OSHA Method ID-215 uses magnesium sulfate/ phosphate buffer⁴ to precipitate potential metal interferences, mainly Fe(II) and Cr(III). OSHA Method W4001 was later developed to provide a wipe sampling method for hexavalent chromium.

1.2 Changes to the Previously Used Method

The specific medium described in this appendix is 37-mm diameter polyvinyl chloride (PVC) filters with 5- μm pore size. This appendix represents an update to the previous OSHA Method ID-215 version and OSHA Method W4001, which were fully validated at the time they were published based on the validation guidelines in effect at that time. Compared to the previous OSHA Method ID-215 version 2 and OSHA Method W4001, this method uses 20-mL glass scintillation vials, hot block, sample extraction temperature of 93.5 °C, no field stabilization requirement, and modified ion chromatography parameters. Thirty-seven mm diameter PVC filters were evaluated as both air and wipe sampling media for hexavalent chromium. Data presented from the previous methods are identified by the statement “Pre-existing data from OSHA Method ID-215 version 2 are presented in this section”, “Pre-existing data from OSHA Method ID-215 version 1⁵ are presented in this section” or “Pre-existing data from OSHA Method W4001 are presented in this section”. The changes were made to standardize collection, extraction and analysis of hexavalent chromium. Pre-existing data from OSHA Method ID-215 version 1 and version 2 are validation data for 37-mm PVC filters as air sampling media. Pre-existing data from OSHA Method W4001 are validation data for 37-mm PVC filters as wipe sampling media.

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1.3 Validation Parameters

The procedures used to develop method validation data are described in *Sampling and Analytical Methods*⁶, and *Evaluation Guidelines for Surface Sampling Methods*.⁷ The target concentration (5.0 µg/m³) for this method evaluation was the OSHA 8-hour time-weighted average (TWA) permissible exposure limit (PEL) for hexavalent chromium. The target mass (T_M) is the equivalent analyte mass per sample collected on a sampler at the target concentration using the recommended sampling time and sampling rate. The action level is half of the target concentration. For the pre-existing data from OSHA Method ID-215 version 2, the target concentration is 1.0 µg/m³.

2 Limit of Detection, Limit of Quantitation, and Reporting Limit

The limit of detection (LOD) is the analyte mass introduced onto the chromatographic column that produces a response greater than 3.3× the standard error of estimate ($S_{y/x}$) divided by the slope of the linear regression equation. The linear regression equation is derived from analyses of three spiked samples at five evenly spaced levels, totaling fifteen spiked media samples. Thirty-seven mm PVC filters were spiked such that the highest mass loading produced a peak approximately 10 times greater than that of a sample blank at or near the chromatographic retention time of the analyte. The limit of quantitation (LOQ) is expressed as a concentration that provides sufficient analyte mass per sample to produce a response greater than $10 \times S_{y/x}$ divided by the slope of the linear regression equation line described above. The spiked media and three sample blanks were analyzed with the recommended analytical parameters in OSHA Method ID-215, Section 3. The resulting data provided the $S_{y/x}$ and the slope values for LOD and LOQ determinations. Results obtained from these analyses are listed in Table A-1 and plotted in Figure A-1.

Table A-1. LOD and LOQ data for hexavalent chromium on 37-mm PVC filters.

mass per sample (µg/sample)	0.0000	0.0162	0.0323	0.0474	0.0646	0.0808
	1.11	2.85	3.96	5.20	6.73	8.31
area counts (×10 ⁶)	1.49	2.77	4.26	5.29	7.20	8.57
	1.56	2.83	4.10	5.46	7.01	8.28

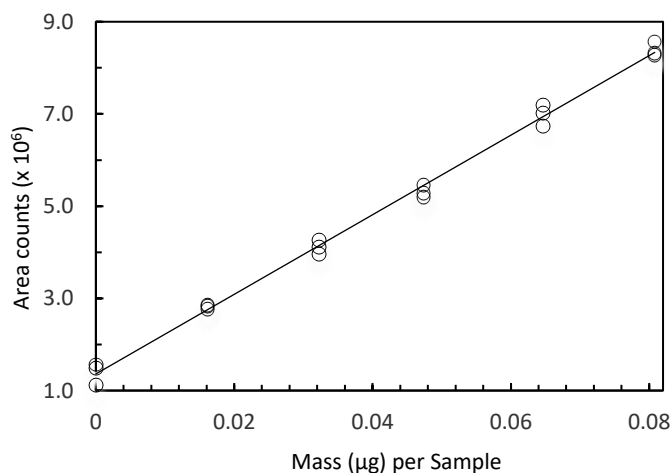


Figure A-1. Plot of data to determine the LOD and LOQ for hexavalent chromium on 37-mm PVC filters ($y = 8.63 \times 10^7 x + 1.36 \times 10^6$, $S_{y/x} = 1.67 \times 10^5$, LOD = 0.00640 µg/sample, LOQ = 0.0194 µg/sample or 0.0202 µg/m³ at the 8-hour TWA).

The reporting limit (RL) is expressed as a concentration at the equivalent mass per sample at the lowest analytical calibration standard providing the recovery is within ±25%. Six replicate 37-mm PVC filters were spiked with 0.0496 µg of hexavalent chromium. After overnight drying in the fume hood at ambient temperature, the samples were analyzed

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using the analytical calibration procedure described in OSHA Method ID-215, Section 3. After analysis, results for hexavalent chromium as a percentage of expected recovery were 117.0%, 119.0%, 117.0%, 119.0%, 109.0%, and 111.0%. The mean percentage of expected recovery was 115.3%. The reporting limit is 0.05 µg/m³ at the 8-hour TWA.

3 Analytical Calibration

Thirty analytical standards over a range of 0.01 to 2.2 times the 8-hour TWA T_M were analyzed with the recommended analytical parameters described in OSHA Method ID-215. Graphical and statistical examination of these data indicate the appropriate regression model and fitting technique were weighted-least-squares linear regression with a x^{-1} weighting factor. Results from these analyses are listed in Table A-2 and plotted with the selected regression model and fitting technique in Figure A-2. Five standards prepared over this range are used for analytical calibration as described in OSHA Method ID-215, Section 3.4.

Table A-2. Analytical calibration data for Cr(VI).

×8-hour TWA T_M (µg/sample)	0.01× (0.0493)	0.23× (1.13)	0.45× (2.26)	0.68× (3.39)	0.90× (4.51)	1.13× (5.64)	1.35× (6.77)	1.62× (8.10)	1.9× (9.44)	2.2× (10.8)
	0.565	10.6	21.2	31.1	41.9	51.3	62.2	74.1	87.1	99.9
area counts ×10 ⁷	0.542	10.3	20.9	30.8	41.2	51.4	61.7	74.5	87.2	98.5
	0.549	10.7	21.2	30.7	40.9	51.4	61.3	75.3	85.9	98.5

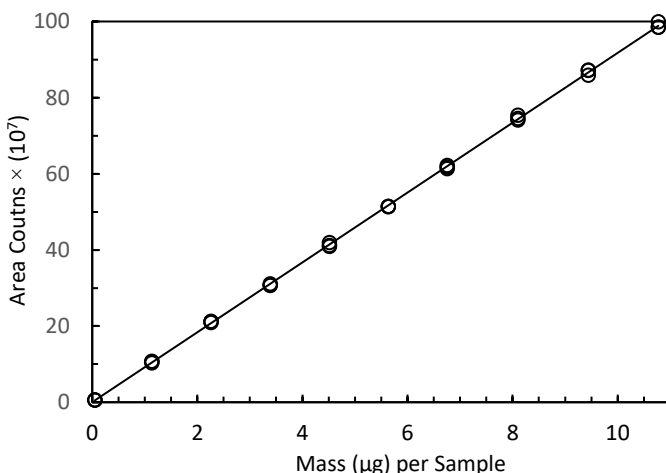


Figure A-2. Plot of data used to determine the analytical calibration procedure for hexavalent chromium ($y = 9.15 \times 10^7 x + 1.05 \times 10^6$, $w_i = x^{-1}$).

4 Post Sampling Storage

Soluble Cr(VI) is defined as Cr(VI) from $K_2Cr_2O_7$ dissolved in DI water; and insoluble Cr(VI) as Cr(VI) from $PbCrO_4$ dissolved in BE solution. Lead chromate ($PbCrO_4$) is not soluble in DI water, but readily soluble in warm BE.

4.1 Soluble Cr(VI) spiked on PVC filters

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

Twenty-one PVC filters were spiked with 960 ng of soluble Cr(VI) and allowed to dry. The spiked filters had 960 L of air at 80% relative humidity (RH) and 23 °C drawn through them at 2 L/min. Three samples were analyzed on the day of preparation. Nine of the filters were stored at a reduced temperature (4 °C) and the other nine were stored in a closed drawer at ambient temperature (about 22 °C). At 5-day intervals, three samples were selected from each of the two

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storage sets and analyzed. Sample results were not corrected for extraction efficiency. The results show a recovery of 96.6% on day 15 for samples stored at ambient temperature, and 96.3% for refrigerated samples. Results from these analyses are listed in Table A-3 and A-4 and Figure A-3 and A-4.

Table A-3. Ambient storage recovery test for soluble Cr(VI) on PVC filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.0	96.7	97.2
5	96.9	95.9	96.0
10	95.8	96.9	96.7
15	97.8	97.6	94.5

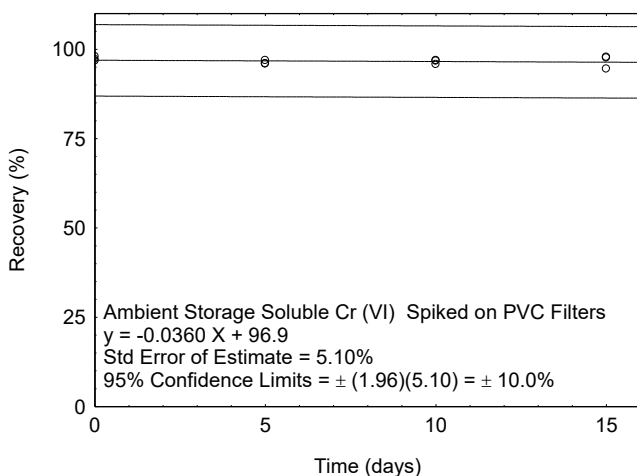
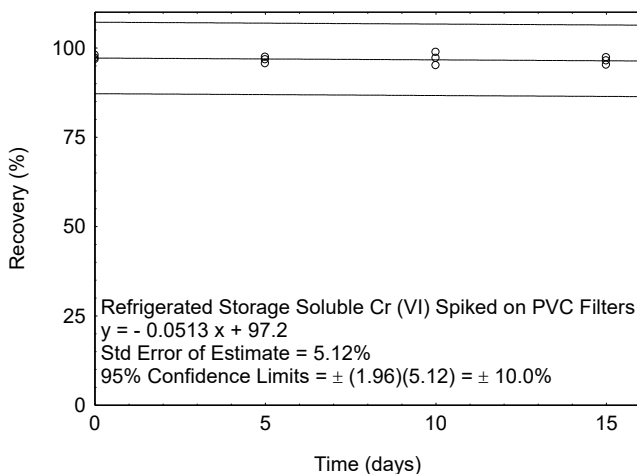


Figure A-3. Ambient storage test for soluble Cr(VI) spiked on PVC filters.

Table A-4. Refrigerated storage recovery test for soluble Cr(VI) on PVC filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.0	96.7	97.2
5	97.1	97.4	95.6
10	97.5	98.8	95.1
15	95.2	96.4	97.3



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Figure A-4. Refrigerated storage test for soluble Cr(VI) spiked on PVC filters.

Note that similar data and results can also be found in OSHA Method W4001 for 37-mm PVC filters validated as wipe sampling media, where the storage samples were prepared by spiking PVC filters without air drawn through.

4.2 Insoluble Cr(VI) spiked on PVC filters

Pre-existing data from OSHA Method ID-215 version 1 are presented in this section.

Twenty-four PVC filters were spiked with 200 ng of Cr(VI) in BE and allowed to dry. The spiked filters had 960 L of air at 80% RH and 23 °C drawn through them at 2 L/min. Six samples were analyzed on the day of preparation. The other filters were stored in a closed drawer at ambient temperature (about 22 °C). At 5-, 15-, and 30-day intervals, six samples were selected and analyzed. Sample results were not corrected for extraction efficiency. The results show a recovery of 93.7% on day 30 for samples stored at ambient temperature. Results from these analyses are listed in Table A-5 and Figure A-5.

Table A-5. Ambient storage recovery test for insoluble Cr(VI) on PVC filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	98.1	97.2	95.6	97.4	97.8	96.0
5	97.4	93.4	91.0	92.3	91.6	95.9
15	99.6	98.8	99.5	97.1	98.5	98.7
30	95.7	92.3	94.2	92.9	93.0	94.1

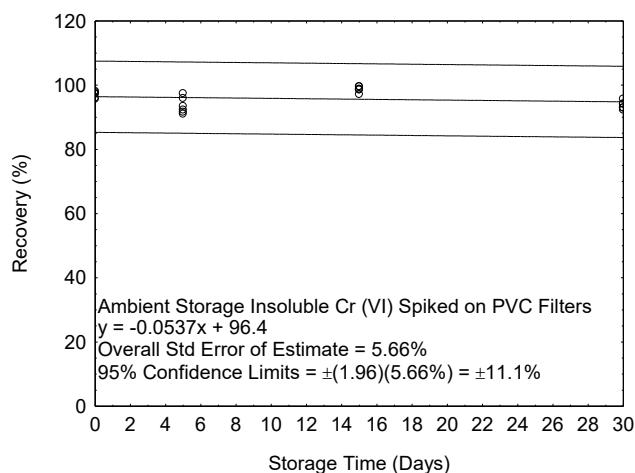


Figure A-5. Ambient storage test for insoluble Cr(VI) spiked on PVC filters.

5 Method Precision and Bias

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The precision at the 95% confidence level was obtained by multiplying the standard error of estimate for the appropriate storage stability test by 1.96 (the z-statistic from the standard normal distribution at the 95% confidence level). In Appendix A, Section 4.1, 95% confidence intervals are drawn about their respective regression lines in the storage graph figures. The precision of the overall procedure of $\pm 10.0\%$ was obtained from the standard error of estimate of 5.1% in Figure A-3 for PVC filters. Each precision includes an additional $\pm 5\%$ for sampling error.

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6 Analytical Recovery and Stability of Prepared Samples

Analytical recovery is affected by the sampling medium, the extraction buffer, extraction temperature and the technique used to extract samples. The data presented demonstrate validity for the extraction buffer, sampling medium, extraction temperature and extraction technique described in this method. If other combinations of these are to be used, testing must be completed to satisfy the requirements found in current OSHA sampling and analysis method validation guidelines. Acceptable testing results must be documented.

6.1 Pre-existing Data Using Hot Plate and Erlenmeyer Flask

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The target concentration refers to 1.0 µg/m³ in OSHA Method ID-215 version 2. The reliable quantitation limit (RQL) is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters obtained for the calculation of the detection limit of the overall procedure, providing 75% to 125% of the analyte is recovered. The RQL for PVC filters extracted with BE/PBM is 3.32 ng/sample (3.5 ng/m³), and 2.67 ng/sample (2.8 ng/m³) for PVC filters extracted with SPE/PBM. Recoveries at this concentration are 96.8% for BE/PBM extraction and 98.3% for SPE/PBM extraction.

Extraction Efficiency for Soluble Cr(VI) with BE/PBM from PVC Filters

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The extraction efficiency of Cr(VI) was determined by liquid-spiking PVC filters with soluble Cr(VI) at masses ranging from 3 to 1920 ng. These samples were stored overnight at ambient temperature and then analyzed. Filters were prepared for analysis following the primary extraction procedure in sample preparation (OSHA Method ID-215 version 2, Section 3.5). The mean extraction efficiency over the range of 3 to 1920 ng was 97.2%. Wet extraction efficiency samples were prepared by loading the filters with water by pulling 960 L of humid air (79% RH at 22 °C) through the filter at 2 L/min before spiking. The wet extraction efficiency was not included in the overall mean.

Table A-6. Extraction efficiency of soluble Cr(VI) BE/PBM from PVC filters.

× target concentration	ng/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	mean (%)
RQL	3	95.6	95.1	97.6	98.9	96.8
0.25	240	96.4	98.0	97.3	95.9	96.9
0.5	480	96.7	98.3	95.9	97.9	97.2
1.0	960	98.1	99.5	97.0	95.8	97.6
1.5	1440	97.8	98.5	96.8	95.3	97.1
2.0	1920	99.1	96.9	96.0	98.5	97.6
1.0 (wet)	960	98.2	97.1	96.3	96.8	97.1

The stability of extracted samples was investigated by reanalyzing the target concentration samples 24 hours after initial analysis. After the original analysis was performed, two vials were recapped with new septa while the remaining two retained their punctured septa. The samples were reanalyzed with fresh standards. The average percent change was -3.4% for samples that were resealed with new septa and -4.5% for those that retained their punctured septa. Each septum was punctured 1 time for each injection.

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Table A-7. Stability of extracted samples for soluble Cr(VI) with BE/PBM from PVC filters with punctured septa replaced.

samples	Initial recovery (%)	after one day recovery (%)	difference (%)
sample 1	98.1	94.0	-4.1
sample 2	98.5	95.8	-2.7
mean	98.3	94.9	-3.4

Table A-8. Stability of extracted samples for soluble Cr(VI) with BE/PBM from PVC filters with punctured septa retained.

samples	Initial recovery (%)	after one day recovery (%)	difference (%)
sample 1	97.0	92.6	-4.4
sample 2	95.8	91.2	-4.6
mean	96.4	91.9	-4.5

Extraction Efficiency for Soluble Cr(VI) with SPE/PBM from PVC Filters

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The extraction efficiency of soluble Cr(VI) was determined by liquid-spiking PVC filters with Cr(VI) at masses ranging from 3 to 1920 ng. These samples were stored overnight at ambient temperature and then analyzed. Filters were prepared for analysis following the secondary extraction procedure in sample preparation of OSHA Method ID-215 version 2, Section 3.5. The mean extraction efficiency over the range of 3 to 1920 ng was 98.4%. Wet extraction efficiency samples were prepared by loading the filters with water by pulling 960 L of humid air (79% RH at 22 °C) through the filter at 2 L/min before spiking. The wet extraction was not included in the overall mean.

Table A-9. Extraction efficiency of soluble Cr(VI) with SPE/PBM from PVC filters.

× target concentration	ng/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	mean (%)
RQL	3	97.3	98.5	99.7	97.6	98.3
0.25	240	98.9	99.1	99.3	98.0	98.8
0.5	480	99.1	97.4	98.0	98.4	98.2
1.0	960	97.9	98.8	97.6	99.2	98.4
1.5	1440	98.4	99.0	97.9	97.0	98.1
2.0	1920	99.9	97.1	98.8	99.1	98.7
1.0 (wet)	960	99.1	98.2	97.2	98.9	98.4

The stability of extracted samples was investigated by reanalyzing the target concentration samples 24 hours after initial analysis. After the original analysis was performed, two vials were recapped with new septa while the remaining two retained their punctured septa. The samples were reanalyzed with fresh standards. The average percentage change was -4.4% for samples that were resealed with new septa and -4.0% for those that retained their punctured septa. Each septum was punctured 1 time for each injection.

Table A-10. Stability of extracted samples for soluble Cr(VI) with SPE/PBM from PVC filters with punctured septa replaced.

samples	Initial recovery (%)	after one day recovery (%)	difference (%)
sample 1	97.9	93.7	-4.2
sample 2	98.8	94.2	-4.6
mean	98.4	94.0	-4.4

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Table A-11. Stability of extracted samples for soluble Cr(VI) with SPE/PBM from PVC filters with punctured septa retained.

samples	Initial recovery (%)	after one day recovery (%)	difference (%)
sample 1	97.6	93.8	-3.8
sample 2	99.2	95.0	-4.2
mean	98.4	94.4	-4.0

Extraction Efficiency for Insoluble Cr(VI) With BE/PBM from PVC Filters

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The extraction efficiency of Cr(VI) was determined by liquid-spiking PVC filters with insoluble Cr(VI) at masses ranging from 3 to 1920 ng. These samples were stored overnight at ambient temperature and then analyzed. Filters were prepared for analysis following the primary extraction procedure in sample preparation of OSHA Method ID-215 version 2, Section 3.5. The mean extraction efficiency over the range of 3 to 1920 ng was 97.8%. Wet extraction efficiency samples were prepared by loading the filters with water by pulling 960 L of humid air (79% RH at 22 °C) through the filter at 2 L/min before spiking. The wet extraction efficiency was not included in the overall mean.

Table A-12. Extraction efficiency for insoluble Cr(VI) with BE/PBM from PVC filters.

× target concentration	ng/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	mean (%)
RQL	3	94.1	96.3	97.1	98.8	96.6
0.25	240	96.6	95.3	98.2	97.6	96.9
0.5	480	97.2	96.9	98.8	97.4	97.6
1.0	960	97.8	98.4	99.1	96.9	98.1
1.5	1440	98.1	99.2	99.5	96.8	98.4
2.0	1920	98.9	99.7	99.8	97.1	98.9
1.0 (wet)	960	97.4	99.2	98.3	97.5	98.1

The stability of extracted samples was investigated by reanalyzing the target concentration samples 24 hours after initial analysis. After the original analysis was performed, two vials were recapped with new septa while the remaining two retained their punctured septa. The samples were reanalyzed with fresh standards. The average percentage change was -3.1% for samples that were resealed with new septa and -3.6% for those that retained their punctured septa. Each septum was punctured 1 time for each injection.

Table A-13. Stability of extracted samples for insoluble Cr(VI) with BE/PBM from PVC filters with punctured septa replaced.

samples	Initial recovery (%)	after one day recovery (%)	difference (%)
sample 1	97.8	95.1	-2.7
sample 2	98.4	94.9	-3.5
mean	98.1	95.0	-3.1

Table A-14. Stability of extracted samples for insoluble Cr(VI) with BE/PBM from PVC filters with punctured septa retained.

samples	Initial recovery (%)	after one day recovery (%)	difference (%)
sample 1	99.1	95.0	-4.1
sample 2	96.9	93.8	-3.1
mean	98.0	94.4	-3.6

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Extraction Efficiency for Insoluble Cr(VI) With SPE/PBM from PVC Filters

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The extraction efficiency of insoluble Cr(VI) was determined by liquid-spiking PVC filters with insoluble Cr(VI) at masses ranging from 3 to 1920 ng. These samples were stored overnight at ambient temperature and then analyzed. Filters were prepared for analysis following the secondary extraction procedure in sample preparation (Section 3.5). The mean extraction efficiency over the range of 3 to 1920 ng was 98.8%. Wet extraction efficiency samples were prepared by loading the filters with water by pulling 960 L of humid air (79% RH at 22 °C) through the filter at 2 L/min before spiking. The wet extraction efficiency was not included in the overall mean.

Table A-15. Extraction efficiency of insoluble Cr(VI) with SPE/PBM from PVC filters.

× target concentration	ng/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	mean (%)
RQL	3	99.3	98.6	97.5	99.3	98.7
0.25	240	98.6	97.9	99.7	99.0	98.8
0.5	480	99.2	98.5	98.0	99.2	98.7
1.0	960	99.8	99.0	98.3	99.5	99.2
1.5	1440	99.3	98.4	99.0	97.8	98.6
2.0	1920	99.8	98.0	97.8	99.7	98.8
1.0 (wet)	960	99.6	97.9	98.8	99.2	98.9

The stability of extracted samples was investigated by reanalyzing the target concentration samples 24 hours after initial analysis. After the original analysis was performed, two vials were recapped with new septa while the remaining two retained their punctured septa. The samples were reanalyzed with fresh standards. The average percentage change was -4.4% for samples that were resealed with new septa and -3.9% for those that retained their punctured septa. Each septum was punctured 1 time for each injection.

Table A-16. Stability of extracted samples for insoluble Cr(VI) with SPE/PBM from PVC filters with punctured septa replaced.

samples	Initial recovery (%)	after one day recovery (%)	difference (%)
sample 1	99.8	95.8	-4.0
sample 2	99.0	94.2	-4.8
mean	99.4	95.0	-4.4

Table A-17. Stability of extracted samples for insoluble Cr(VI) with SPE/PBM from PVC filters with punctured septa retained.

samples	Initial recovery (%)	after one day recovery (%)	difference (%)
sample 1	98.3	94.7	-3.6
sample 2	99.5	95.3	-4.2
mean	98.9	95.0	-3.9

Extraction Efficiency for Soluble Cr(VI) With BE/PBM and SPE/PBM from PVC Filters as Wipe Media

Pre-existing data from OSHA Method W4001 are presented in this section.

The extraction efficiencies of Cr(VI) were determined by liquid-spiking PVC filters with Cr(VI) at concentrations ranging from the reliable quantitation limit (RQL) to 10 times the target concentration. These samples were stored overnight at ambient temperature and then analyzed. The filters were either extracted and digested with the first, main extraction solution, BE, or with the second, spray paint only, SPE solution. The mean extraction efficiency over the working range of the RQL to 10 times the target concentration is 96.6% for samples extracted with the first (main) extraction solvent (BE)

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and 96.8% for samples extracted with the second extraction solvent for paint samples (SPE). (Note, RQL is 3.04 ng/sample. Recovery at this concentration is 95.1%).

Table A-18. Extraction efficiency of Cr(VI) from PVC filters extracted with BE.

× target concentration	ng/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	mean (%)
RQL	3	95.4	96.6	92.7	95.5	95.1
0.1	5	96.4	96.5	97.9	95.9	96.7
1.0	50	97.8	97.2	96.7	97.6	97.3
10.0	500	97.4	97.8	97.0	96.1	97.1

Table A-19. Extraction efficiency of Cr(VI) from PVC filters extracted with SPE.

× target concentration	ng/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	mean (%)
RQL	3	97.8	95.0	97.3	95.5	96.4
0.1	5	97.5	97.1	97.6	96.0	97.1
1.0	50	95.9	97.5	96.3	96.1	96.5
10.0	500	98.0	96.4	97.1	97.9	97.4

6.2 Data Using Hot Block and Scintillation Vial

The analytical recovery (R_A) was determined by liquid-spiking six replicate 37-mm PVC filters across a range of analyte mass values that would be obtained from sampling at 0.1 to 2 times the 8-hour T_C for 480 minutes. Samples were analyzed following overnight drying at ambient temperature (22 °C) in the hood. The R_A was 105.5%. The percent coefficient of variation of the thirty spiked samples (CV_{R_A}) was 3.1%. The data are shown in Table A-20.

Table A-20. Analytical recovery results of 37-mm PVC filters.

× 8-hour TWA T_M	µg/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)	mean (%)
0.1	0.4738	105.9	106.4	107.6	107.3	107.0	107.1	106.9
0.5	2.4926	103.7	106.0	103.0	101.9	105.8	103.0	103.9
1	4.8719	97.6	107.4	104.9	109.1	108.1	102.6	105.0
1.5	7.3645	98.0	106.3	108.9	110.0	107.6	106.8	106.3
2	9.6305	104.8	106.9	98.8	103.8	109.4	108.8	105.4

The stability of sample extracts was determined by liquid-spiking six replicate 37-mm PVC filters with a mass of analyte that would be obtained from sampling at the 8-hour TWA T_C for 480 minutes (calculated to be 5.10 µg). Samples were analyzed following overnight drying at ambient temperature in the hood. Each septum was punctured 1 time for each injection and punctured septa retained in this validation.

All six vials were re-analyzed at 24, 48, and 72 hours after the initial analyses, with all vials remaining in the autosampler tray kept at room temperature (22 °C). Freshly prepared standards (in DBE buffer without media) were used for each analysis event. The linear regression equation determined from these results was $y = -0.000283x + 1.02$. The change in recovery (Δ_{ps_TWA}) of hexavalent chromium calculated from the regression line generated for the 3-day storage test was 0.085%. Results from the analysis are listed in Table A-21.

Table A-21. Stability of prepared sample data of Cr(VI) on 37-mm PVC filters for 3 days (8-hour TWA).

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	101.8	101.8	101.8	102.6	102.7	103.4
1	102.7	101.8	101.9	102.2	102.3	102.8
2	103.2	101.8	102.2	102.3	102.4	102.8
3	102.2	102.2	102.2	101.9	102.2	102.5

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All six vials were re-analyzed at 1, 3, and 5 weeks after the initial analyses together with freshly prepared standards for each analysis event, with all vials standing at room temperature. Freshly prepared standards were used for each analysis event. The results of these analyses are provided in Table A-22. The linear regression equation determined from these results was $y = 0.00198x + 1.02$. The change in recovery (Δ_{ps_TWA}) of hexavalent chromium calculated from the regression line generated for the 5-week storage test was 0.99%.

Table A-22. Stability of prepared sample data of Cr(VI) on 37-mm PVC filters for 5 weeks (8-hour TWA).

time (week)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	101.8	101.8	101.8	102.6	102.7	103.4
1	101.8	102.6	101.3	102.5	102.6	103.0
3	102.0	102.4	103.4	102.8	102.3	102.7
5	103.3	104.6	102.9	102.7	103.2	103.2

The action level ($\frac{1}{2}$ 8-hour TWA) stability of sample extracts was examined by retaining the solvent extract of six spiked 37-mm PVC media at mass loading levels of half T_M (2.49 μg). Each septum was punctured 1 time for each injection and punctured septa retained in this validation.

All six vials were re-analyzed at 24, 48, and 72 hours after the initial analyses, with all vials remaining in the autosampler tray and kept at room temperature. Freshly prepared standards (in DBE buffer without media) were used for each analysis event. The linear regression equation determined from these results was $y = -0.00357x + 1.02$. The change in recovery (Δ_{ps_action}) of hexavalent chromium calculated from the regression line generated for the 3-day storage test was 1.1%. Results from the analysis are listed in Table A-23.

Table A-23. Stability of prepared sample data of Cr(VI) on 37-mm PVC filters for 3 days (Action Level).

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	101.7	103.3	104.0	103.4	104.0	100.4
1	100.3	102.1	101.7	101.5	102.1	98.9
2	100.3	102.1	102.7	102.5	101.9	98.5
3	100.4	102.4	102.6	102.0	102.3	99.5

All six vials were re-analyzed at 2, 3, and 5 weeks after the initial analyses together with freshly prepared standards for each analysis event, with all vials standing at room temperature. Freshly prepared standards were used for each analysis event. The results of these analyses are provided in Table A-24. The linear regression equation determined from these results was $y = 0.00128x + 1.02$. The change in recovery (Δ_{ps_action}) of hexavalent chromium calculated from the regression line generated for the 5-week storage test was 0.64%.

Table A-24. Stability of prepared sample data of Cr(VI) on 37-mm PVC filter for 5 weeks (Action Level).

time (week)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	101.7	103.3	104.0	103.4	104.0	100.4
2	100.4	102.1	102.5	102.1	102.0	98.9
3	101.0	103.0	102.8	103.3	102.9	99.1
5	101.7	102.3	102.5	102.9	102.9	99.7

7 Sampler capacity

7.1 Removal Efficiency as Air Sampling Media

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

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It was not possible to safely generate a test atmosphere of Cr(VI); therefore, retention efficiency tests were performed to support the recommended air volume.

A retention efficiency test for soluble Cr(VI) on 37-mm PVC filters was performed by spiking 1920 ng of Cr(VI) onto the filters, allowing them to dry, and then placing them into 37-mm polystyrene cassettes with a BUP. A sampling train was constructed by placing a cassette containing the spiked 37-mm PVC filter with a BUP and a second cassette with a blank 37-mm PVC filter and a BUP. The air flowed through the cassette containing the spiked 37-mm PVC filter and then through the second (back) cassette. These sampling trains each had 960 liters of air with 80% relative humidity at 23 °C drawn through them at 2 L/min. There was no Cr(VI) found on the 37-mm PVC filters on the back cassettes or on the BUPs.

Table A-25. Retention efficiency test for soluble Cr(VI) spiked on 37-mm PVC filters.

cassette location	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)	mean (%)
front PVC	98.5	97.4	96.9	96.0	98.6	97.5	97.5
front BUP	0.0	0.0	0.0	0.0	0.0	0.0	0.0
back PVC	0.0	0.0	0.0	0.0	0.0	0.0	0.0
back BUP	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total	98.5	97.4	96.9	96.0	98.6	97.5	97.5

7.2 Removal Efficiency as Sampler Cassette Wipes

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The practice of wiping interior walls of filter sampling cassettes was initiated because of the following study. The interior walls of cassettes used for compliance samples received at OTC over a three-month period beginning in August 2000 were wiped with a PVC filter wet with DBE/PBM.¹ The compliance samples were from three industries, chromium plating (plating), spray painting (painting), and welding (welding). Samples were analyzed within three days of the date of receipt. There was Cr(VI) found on most of the cassette walls. Percentages (amount on cassette walls/amount found on filter) ranged from 0-20% for plating, 3.8-13% for painting, and 0-123% for welding.

The removal efficiency of Cr(VI) from the walls of cassettes was determined by spiking the interior wall of the top piece of a 37-mm cassette with either 100 ng of K₂Cr₂O₇ or 100 ng of PbCrO₄, allowing it to dry overnight, and then wiping the cassette. Three different means of wiping the cassette were tested: wiping with a dry PVC filter, wiping with a PVC filter wetted with a drop of DI water, and wiping the cassette with a PVC filter wetted with a drop of DBE/PBM solution. There is a smooth and rough side on a PVC filter. All wiping was performed with the rough side towards the spiked surface using a gloved hand. Filters were prepared following the primary extraction procedure in sample preparation (OSHA Method ID-215, Section 3.5). The recoveries were similar for PVC filter wetted with a drop of DI water or DBE/PBM solution. The use of DBE/PBM solution will help eliminate interference problems, so that solution is used for cassette wiping.

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Table A-26. Removal efficiency (%) of cassette wipes using 37-mm PVC filter.

sample #	dry filter K ₂ Cr ₂ O ₇	DI water filter K ₂ Cr ₂ O ₇	DBE/PBM filter K ₂ Cr ₂ O ₇	dry filter PbCrO ₄	DI water filter PbCrO ₄	DBE/PBM filter PbCrO ₄
1	87.5	97.7	98.3	79.3	86.9	88.5
2	90.2	95.6	99.9	88.6	89.4	95.8
3	88.6	94.6	95.6	86.8	88.7	93.5
4	89.5	97.1	94.9	86.5	92.8	94.9
5	86.5	96.9	97.8	92.8	91.4	92.6
6	91.2	93.9	98.3	84.6	85.9	91.4
mean	88.9	96.0	97.5	86.4	89.2	92.8

7.3 Surface Removal Efficiency as Wipe sampling Media

Pre-existing data from OSHA Method W4001 are presented in this section.

Removal efficiency refers to the ability of the PVC filters to absorb or otherwise capture surface contaminants when the filter is moved across a surface under firm pressure. The surface used to evaluate the removal efficiency was a PTFE sheet. This type of surface approaches the smooth and non-porous characteristics of an ideal surface. The variety of surfaces found in workplaces will likely be less than ideal, so the media will have a lower removal efficiency. The amount of analyte found on the filter after sampling will indicate that at least that amount was present on the surface that was sampled. Six surfaces were spiked at the target concentration of Cr(VI), 0.050 µg/100 cm². Samples were collected from each surface using the technique described in OSHA Method ID-215, Section 2.3 and analyzed. The results are shown in Table A-27. The mean removal efficiency of the six samples was 96.9% for PVC filters. The removal efficiency was calculated as amount of analyte recovered divided by amount of analyte spiked on the surface.

Table A-27. Sampler removal efficiency data for Cr(VI) from PTFE using PVC filters.

theoretical (µg/sample)	recovered (µg/sample)	recovery (%)
0.05	0.0490	98.0
0.05	0.0491	98.2
0.05	0.0488	97.6
0.05	0.0476	95.2
0.05	0.0479	95.8
0.05	0.0482	96.4

8 Effect of Humidity and Low Sampling Concentration

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The ability of PVC filters to retain Cr(VI) in a relatively dry atmosphere was tested by spiking 1920 ng of Cr(VI) onto each of three filters and placing them into polystyrene cassettes. The cassettes then had 960 liters of air with 20% RH at 23 °C drawn through them at 2 L/min. All the samples were immediately analyzed. The analytical results were 97.8%, 99.4% and 98.1% of theoretical, with a mean of 98.4%.

The ability of PVC filters to retain Cr(VI) at low concentration was tested by spiking 96 ng of Cr(VI) onto each of three filters and placing them into polystyrene cassettes. The cassettes then had 960 liters of air with 79% RH at 23 °C drawn through them at 2 L/min. All the samples were immediately analyzed. The analytical results were 96.8%, 97.7% and 94.9% of theoretical, with a mean of 96.5%.

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9 Sampling and Analytical Interferences

Acid is a negative interference in chrome plating operations. Samples should be taken on NaOHqz filters in chrome plating operations. Chromium plating samples collected on PVC filters must either be analyzed within 6 days of sampling or be stabilized in a solution of 10% Na_2CO_3 with 2% NaHCO_3 immediately upon arrival at the analytical laboratory (OSHA Method ID-215, Section 3.5). Welding samples must be analyzed within 8 days after sampling. Spray-paint samples are extracted with hot BE/PBM followed by a second extraction with hot SPE/PBM (Appendix A, Section 4). It is routine procedure to wipe the interior walls of sampling cassettes for all air samples analyzed at OTC (Appendix A, Section 7). The sample filter and cassette wiping PVC filter are put in the same vial and analyzed together instead of analyzed separately. Wipe cassettes of blank samples and treat blank samples the same as the other samples.

Suspected interferences information should be reported to the laboratory with submitted samples. The interference studies were performed in OSHA Method ID-215 version 2 and should be consulted for specific information about the metal species of interest. The major positive interference is Cr(III), which can be oxidized to Cr(VI). The major negative interference is Fe(II), which can reduce Cr(VI) to Cr(III). Other reducing metal species can also change Cr(VI) to Cr(III).

Oxidation Metal Interference

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The ability of PVC filters to maintain Cr(VI) in the presence of interferences was tested several ways. Reducing metal species have the potential to reduce Cr(VI) to Cr(III). Also, Cr(III) may oxidize to Cr(VI) when heated in an alkaline solution. These interferences are greatly reduced by the addition of PBM to the BE, causing the other metal species to precipitate. Reducing metal species react with Cr(VI), changing it to Cr(III). Various ratios of Cr(VI) to Fe(II) were studied and the recoveries were 29.2, 72.5, and 91.4% for ratios of 1:10, 1:5 and 1:1, when samples were extracted with BE alone and analyzed the same day they were extracted. The Cr(VI) loading was 1000 ng. Fe(II) continued to react with Cr(VI) in BE solution, decreasing the amount of Cr(VI) recovered over time. Additionally, other reducing metals with the potential to affect recovery were tested in the following ratios and the subsequent recoveries were obtained: 1:10 Cr(VI): Mo(VI), 98.5% recovery; 1:10 Cr(VI): Mn(II), 94.4% recovery; 1:10 Cr(VI): Fe(III) 103% recovery; 1:10 Cr(VI): V(V) 103% recovery; 1:10 Cr(VI): Cu(I), 101% recovery; and 1:10 Cr(VI): Cr(III), 103% recovery. Cr(VI) loading was 1000 ng in these tests.

Magnesium (II) (from magnesium chloride) was added to the BE to precipitate reducing metal species such as Fe(II), and thereby preventing them from reacting with Cr(VI). The ratio in the above paragraph gave the lowest results (1:10 Cr(VI): Fe(II) was tested with Mg(II) and the recovery results improved to 92.7%. Phosphate buffer (0.5 M KH_2PO_4 0.5 M $\text{K}_2\text{HPO}_4 \cdot 3\text{H}_2\text{O}$) and MgCl_2 were added to 1:10 Cr(VI): Fe(II) and the recovery results improved even more to 96.6%. The precipitate using MgCl_2 was very fine; consequently, MgSO_4 was tested as the source of Mg(II) and the use of MgSO_4 resulted in a larger mesh precipitate. Magnesium sulfate and phosphate buffer gave a recovery of 95.8%.

There is a positive interference from Cr(III) because it can change to Cr(VI) in an alkaline solution, in increasing amounts as alkalinity and temperature increases. SPE solution was tested for this effect because it is more alkaline than BE solution. The recovery without PBM was 104.7% for SPE extraction, and 100.6% with PBM for PVC filters spiked with 250 ng of Cr(VI) and 5 mg of Cr(III). The addition of the mixture of MgSO_4 and phosphate buffer (PBM) eliminated this relatively small positive interference.

The major interference in welding operations is Fe(II), which reacts with Cr(VI) to form Cr(III). Twenty-seven storage stability samples were prepared in the following manner to test this effect. A storage test was performed by spiking PVC filters with 960 ng of soluble Cr(VI) and 0.5 mg Fe(II) separately on differing spots on the same filter and allowing the filters to dry. The dried spikes on the same filter were rubbed together to mix them. Cr(VI) and Fe(II) react slowly in the dry state, but they react more quickly in a water solution to form Cr(III). For this reason, Cr(VI) and Fe(II) could not be placed in the same solution, or the solutions spiked on top of each other, but instead had to be mixed in a dry state. The spiked PVC filters then had 960 L of air with 80% RH at 23 °C drawn through them at 2 L/min. Three samples were

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analyzed on the day of preparation. Twelve of the filters were stored at reduced temperature (4 °C) and the other twelve were stored in a closed drawer at ambient temperature (about 22 °C). At 3 to 4-day intervals, three samples were selected from each of the two storage sets and analyzed. Sample results were not corrected for extraction efficiency. The Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis states “A change in recovery of more than 10% in 15 days is a significant uncorrectable bias and must be avoided.”¹ The loss exceeded 10% after 7 days, showing that the samples must be analyzed within 8 days of sampling.

Table A-28. Ambient storage recovery test for soluble Cr(VI) and Fe(II) spiked on PVC filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.9	96.7	97.1
3	94.6	92.3	93.6
7	90.4	88.9	89.9
10	84.2	83.6	82.9
14	81.9	82.3	80.5

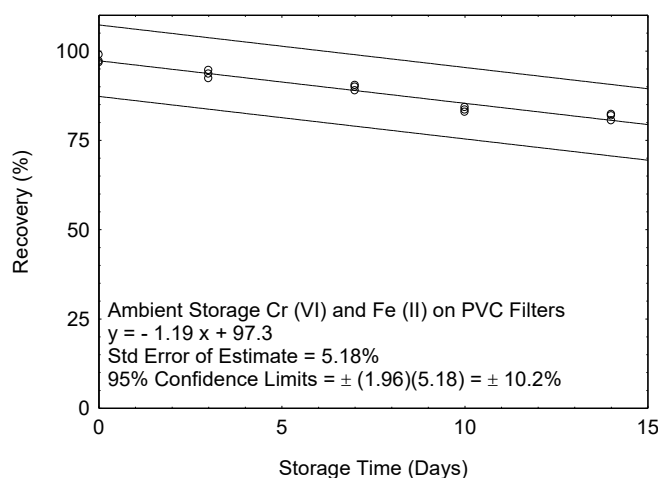


Figure A-6. Ambient storage test for soluble Cr(VI) and Fe(II) using PVC filters.

Table A-29. Refrigerated storage recovery test for soluble Cr(VI) and Fe(II) spiked on PVC filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.9	96.7	97.1
3	94.3	92.9	95.2
7	90.1	89.5	88.9
10	85.3	83.8	84.9
14	80.2	81.4	80.9

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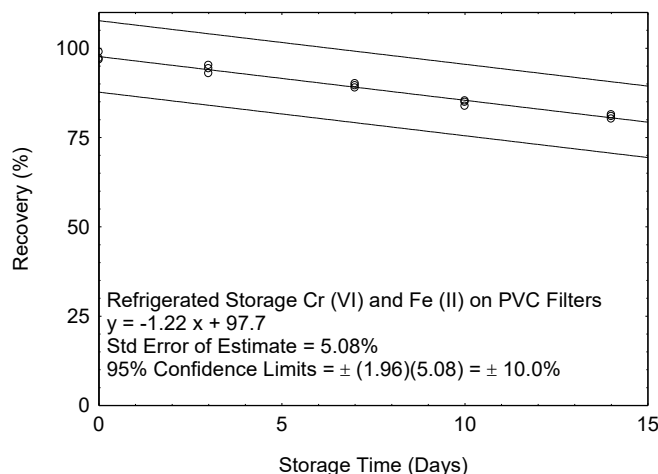


Figure A-7. Refrigerated storage test for soluble Cr(VI) and Fe(II) using PVC filters.

Acid Interference

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The presence of acid in chrome plating operations causes a negative interference due to reaction between Cr(VI) and acid to form Cr(III). Most chrome plating baths contain H₂SO₄. Accordingly, a mixture of H₂SO₄ and Cr(VI) was prepared in water and used to spike PVC filters. Twenty-seven filters were spiked with 960 ng of soluble Cr(VI) and 50 ng of H₂SO₄ and then allowed to dry. The spiked PVC filters each had 960 L air with 80% RH at 23 °C drawn through them at 2 L/min. Three samples were analyzed on the day of preparation. Twelve of the filters were stored at reduced temperature (4 °C) and the other twelve were stored in a closed drawer at ambient temperature (about 22 °C). At 3 to 4-day intervals, three samples were selected from each of the two storage sets and analyzed. Sample results were not corrected for extraction efficiency. The "Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis states "A change in recovery of more than 10% in 15 days is a significant uncorrectable bias and must be avoided."¹ The loss exceeded 10% after 6 days, showing that the samples must be analyzed within 6 days of collection.

Table A-30. Ambient storage recovery test for Cr(VI) and H₂SO₄ on PVC filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.0	96.8	97.2
3	92.6	91.3	93.8
7	86.1	84.1	82.9
10	79.9	81.8	78.5
14	73.6	77.8	75.9

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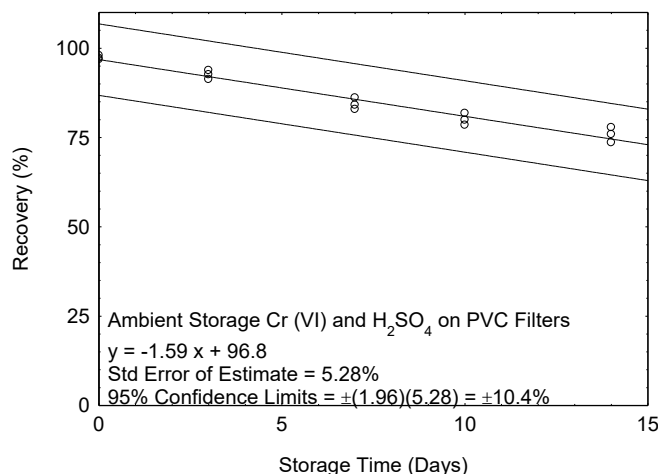


Figure A-8. Ambient storage test for Cr(VI) and H₂SO₄ spiked on PVC filters.

Table A-31. Refrigerated storage recovery test for Cr(VI) and H₂SO₄ on PVC filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.0	96.8	97.2
3	91.8	93.5	92.3
7	85.5	86.8	84.4
10	80.2	78.1	79.9
14	73.2	74.9	75.9

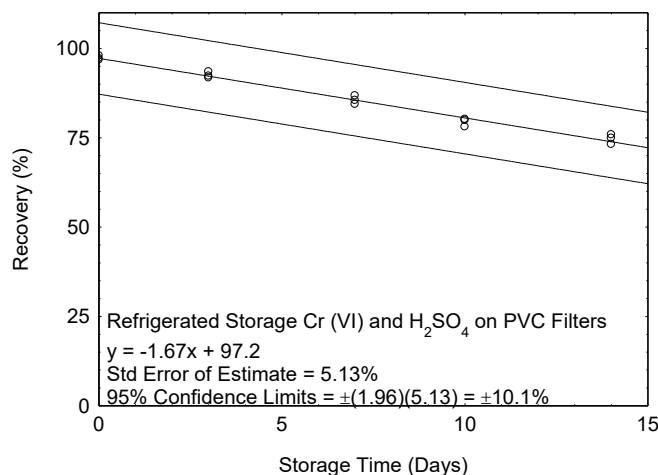


Figure A-9. Refrigerated storage test for Cr(VI) and H₂SO₄ spiked on PVC filters.

Chromium plating samples can be stabilized by neutralizing the acid. A storage stability test was performed by placing spiked filters into BE after preparation to demonstrate this stabilization effect. The PVC filters were prepared by spiking them with 960 ng of Cr(VI) and 50 ng of H₂SO₄ and allowing them to dry before drawing 960 L of air at 80% RH and 23 °C through them at 2 L/min. The filters were placed into a vial containing 5 mL of BE immediately after drawing humid air through them. Three samples were analyzed on the day of preparation. Twelve of the filters were stored at reduced temperature (4 °C) and the other twelve were stored in a closed drawer at ambient temperature (about 22 °C). At 3 to 4-day intervals, three samples were selected from each of the two storage sets and analyzed. Sample results were not corrected for extraction efficiency.

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Table A-32. Ambient storage recovery test for Cr(VI) and H₂SO₄ on PVC filters and placed immediately in BE.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	99.1	97.9	98.1
3	96.6	98.0	95.9
7	95.8	96.1	94.2
10	97.1	95.3	93.8
14	94.3	96.8	92.4

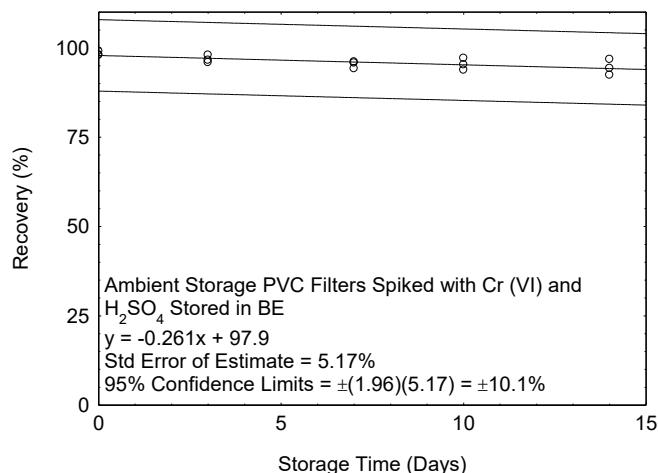


Figure A-10. Ambient storage test for Cr(VI) and H₂SO₄ spiked on PVC filters and stored in 5 mL of BE buffer.

Table A-33. Refrigerated storage recovery test for Cr(VI) and H₂SO₄ on PVC filters and placed immediately in BE.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	99.1	97.9	98.1
3	97.9	98.9	96.9
7	99.2	97.3	97.5
10	98.2	96.9	95.8
14	94.3	95.9	96.7

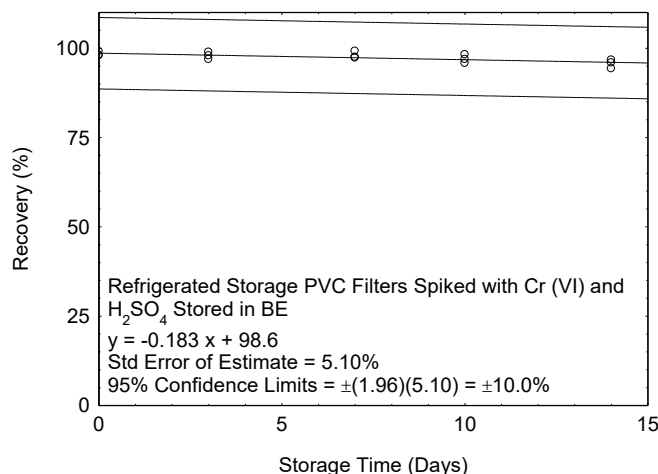


Figure A-11. Refrigerated storage test for Cr(VI) and H₂SO₄ spiked on PVC filters and stored in 5 mL of BE buffer.

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Painting Matrix Interference

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The ability of the two-step extraction process to extract Cr(VI) from a paint matrix was tested by spiking PVC filters with two drops of Sunfire 421 paint (acrylic-urethane enamel obtained from Sherwin-Williams Co., Cleveland, OH) and allowing the spikes to dry. This paint contains lead chromate. Five samples were extracted with acid and analyzed for total chromium by ICP following OSHA Method ID-125G⁸ to determine the amount of chromium in the paint so that theoretical loading on the spiked filters could be calculated. Five samples were extracted with BE/PBM, five samples were extracted with SPE/PBM, and five samples were extracted with BE/PBM followed by a second extraction with SPE/PBM. The recoveries were 46.2% for BE/PBM, 69.8% for SPE/PBM, and 101% for BE/PBM followed by SPE/PBM, illustrating that the two-step extraction is effective. Based on Appendix A, Section 4.2 ambient storage sampling data for lead chromate, the painting samples are stable up to 30 days after sampling.

Acid Interference Tests as Wipe Media

Pre-existing data from OSHA Method W4001 are presented in this section.

Cr(VI) and H₂SO₄ spiked on PVC filters

In chrome plating environments, the presence of acid causes a negative interference due to the reaction between the Cr(VI) and acid to form Cr(III). Most chrome plating baths contain H₂SO₄, so a mixture of H₂SO₄ and Cr(VI) was prepared in water to spike the filters with. Storage samples were prepared by spiking PVC filters with Cr(VI) and H₂SO₄. The PVC filter was spiked with 50 ng of Cr(VI) and 0.5 ng H₂SO₄ and allowed to dry. Twenty-one storage samples were prepared. Three samples were analyzed on the day of preparation. Nine of the filters were stored at reduced temperature (4°C) and the other nine were stored in a closed drawer at ambient temperature (about 22 °C). At 5-day intervals, three samples were selected from each of the two storage sets and analyzed. There was an immediate reaction between the Cr(VI) and the sulfuric acid, causing some of the Cr(VI) to change to Cr(III). Results were corrected for this bias. The results show a recovery of 78.0% on day 15 for samples stored at ambient temperature, and 81.0% for refrigerated samples.

Table A-34. Ambient storage recovery test for Cr(VI) and H₂SO₄ on PVC filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.5	98.8	100.7
5	91.1	91.5	93.6
10	85.8	83.9	84.7
15	74.5	79.4	79.5

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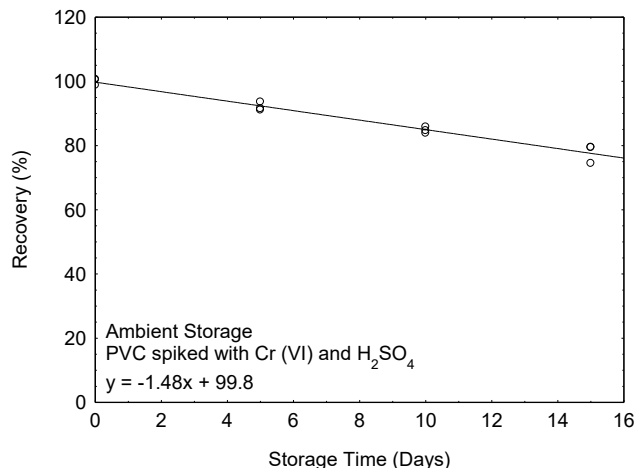


Figure A-12. Ambient storage test for Cr(VI) and H₂SO₄ spiked on PVC filters.

Table A-35. Refrigerated storage recovery test for Cr(VI) and H₂SO₄ on PVC filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.5	98.8	100.7
5	90.0	90.9	91.7
10	84.1	86.8	85.1
15	79.3	83.5	82.9

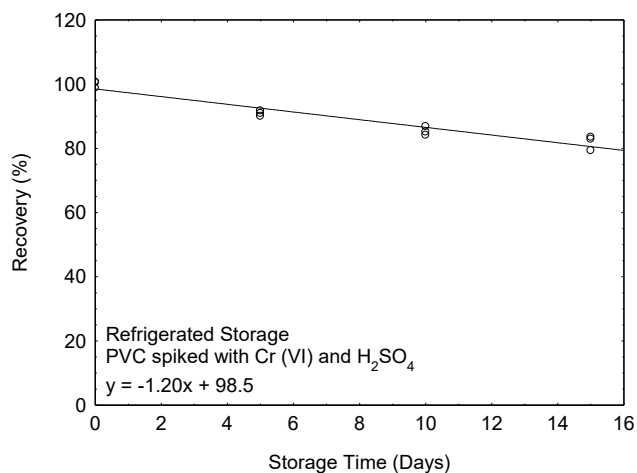


Figure A-13. Refrigerated storage test for Cr(VI) and H₂SO₄ spiked on PVC filters.

Cr(VI) and H₂SO₄ spiked on PVC filters placed immediately in 5 mL of an aqueous solution containing 10% Na₂CO₃ with 2% NaHCO₃.

The "Evaluation Guidelines for Surface Sampling Methods" states that a drop in recovery of greater than 10% upon storage for 15 days is a significant uncorrectable bias and should be avoided¹. The recoveries for the PVC filters spiked with Cr(VI) and H₂SO₄ on day 15 were 78.0% for ambient and 81.0% for refrigerated samples. To eliminate this negative bias from the H₂SO₄, the PVC filters were spiked with Cr(VI) and H₂SO₄ and then placed into a vial containing 5 mL of an aqueous solution containing 10% Na₂CO₃ with 2% NaHCO₃ (BE buffer) immediately. The PVC filter was spiked with 50 ng of Cr(VI) and 0.5 ng H₂SO₄. Twenty-one storage samples were prepared. Three samples were analyzed on the day of preparation. Nine of the filters were stored at reduced temperature (4°C) and the other nine were stored in a

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closed drawer at ambient temperature (about 22 °C). At 5-day intervals, three samples were selected from each of the two storage sets and analyzed. There was an immediate reaction between the Cr(VI) and the sulfuric acid, causing some of the Cr(VI) to change to Cr(III). Results were corrected for this bias. The recoveries on day 15 were 97.4% for ambient storage, and 98.0% for refrigerated storage.

Table A-36. Ambient storage recovery test for Cr(VI) and H₂SO₄ on PVC filters and placed in BE buffer.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.7	97.9	101.5
5	99.6	96.9	98.9
10	98.4	97.6	97.9
15	98.9	95.6	98.3

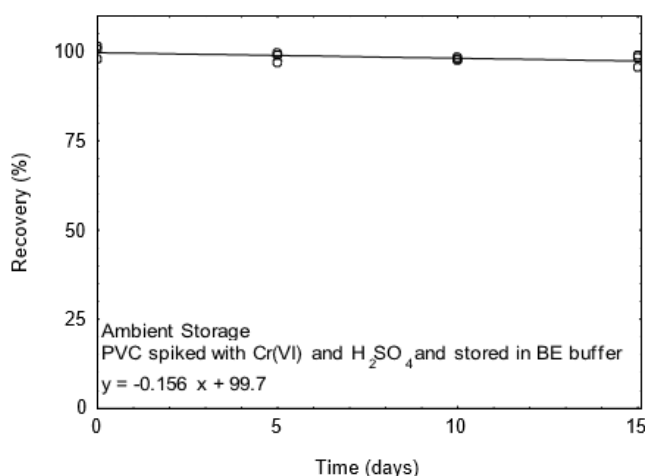


Figure A-14. Ambient storage test for Cr(VI) and H₂SO₄ spiked on PVC filters and stored in 5 mL BE buffer.

Table A-37. Refrigerated storage recovery test for Cr(VI) and H₂SO₄ on PVC filters and placed in BE buffer.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.7	97.9	101.5
5	99.2	98.9	99.1
10	97.9	98.4	99.0
15	98.5	98.9	96.9

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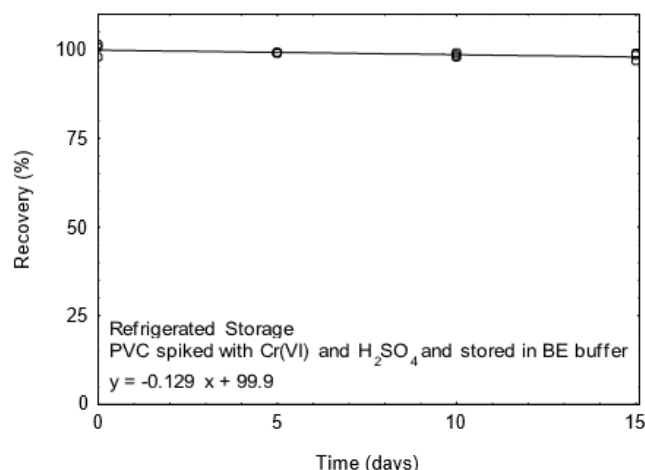


Figure A-15. Refrigerated storage test for Cr(VI) and H₂SO₄ spiked on PVC filters and stored in 5 mL BE buffer.

10 Analytical Reproducibility

As Air Sampling Media

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

Reproducibility samples were prepared by spiking six 37-mm PVC filters with 1.0 µg of soluble Cr(VI) and allowed to dry, and then drawing 960 liters of air at 80% RH and 23 °C through them at 2 L/min. The resulting samples were submitted to the OSHA Technical Center for analysis using OSHA Method ID-215 version 2 after being stored for 10 days at 23 °C. The reproducibility data for PVC filters is presented in Table A-38. No sample result for Cr(VI) had a deviation greater than the precision of the overall procedure presented in Appendix A, Section 5.

Table A-38. Reproducibility data for Cr(VI) spiked on PVC filters.

theoretical (µg/sample)	recovered (µg/sample)	recovery (%)	deviation (%)
1.0	0.968	96.8	-3.2
1.0	0.954	95.4	-4.6
1.0	0.938	93.8	-6.2
1.0	0.945	94.5	-5.5
1.0	0.975	97.5	-2.5
1.0	0.966	96.6	-3.4

As Wipe Sampling Media

Pre-existing data from OSHA Method W4001 are presented in this section.

Six PTFE surfaces were spiked at the target level of 50 ng Cr(VI). A chemist, other than the one developing the method, conducted sampling on the PTFE surfaces as described in OSHA Method ID-215, Section 2.3 using 37-mm PVC filters. The test was repeated with a second chemist performing the sampling. The samples were analyzed. The first chemist was able to achieve a removal efficiency of 96.0% (Table A-39) and the second chemist 95.1% (Table A-40).

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Table A-39. Sampling reproducibility 1st chemist samples for Cr(VI) from PTFE using PVC filters.

theoretical (ng/surface)	recovered (ng/sample)	recovery (%)
50	48.2	96.4
50	48.7	97.4
50	48.4	96.8
50	47.2	94.4
50	48.3	96.6
50	47.2	94.4

Table A-40. Sampling reproducibility 2nd chemist samples for Cr(VI) from PTFE using PVC filters.

theoretical (ng/surface)	recovered (ng/sample)	recovery (%)
50	46.5	93.0
50	47.9	95.8
50	48.6	97.2
50	48.7	97.4
50	46.9	93.8
50	46.7	93.4

Six samples were prepared by spiking 37-mm PVC filters in the same manner that was used in the preparation for the storage study. The samples were submitted to the OSHA Technical Center for analysis. The samples were analyzed after being stored for 13 days at 23 °C. Sample results were corrected for extraction efficiency (Appendix A, Section 6.1). The average recovery was 97.6% (Tables A-41).

Table A-41. Analytical reproducibility data for Cr(VI) using PVC filter.

theoretical (ng/surface)	recovered (ng/sample)	recovery (%)
50	48.1	96.2
50	49.2	98.4
50	47.9	95.8
50	48.6	97.2
50	49.1	98.2
50	49.8	99.6

11 Estimation of Uncertainty

While systematic biases such as analyte storage loss are examined, and limits are placed on these, an estimation of uncertainty that encompasses both potential random and systematic error was not completed. Instead, the overall standard error of estimate was calculated from the random error inherent to the points about the regression line produced by the ambient storage test described in Appendix A, Section 5, as prescribed by the OSHA validation guidelines in use at the time OSHA Method ID 215 version 2 was originally validated. See Section 5 of this appendix for details.

References

1. Ku, J., Eide, M. Hexavalent Chromium (OSHA Method ID-215 version 2), 2006. United States Department of Labor, Occupational Safety and Health Administration website. www.osha.gov (accessed March 2026).
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7. Lawrence, R. Evaluation Guidelines for Surface Sampling Methods, 2001. United States Department of Labor, Occupational Safety & Health Administration website. www.osha.gov (accessed March 2026).
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Appendix B, 25-mm Diameter Polyvinyl Chloride (PVC) Filter

OSHA PEL:	5.0 $\mu\text{g}/\text{m}^3$ 8-Hour TWA (2.5 $\mu\text{g}/\text{m}^3$ Action Level), General Industry, Shipyards, and Construction. See hexavalent chromium standard for compliance dates and special provisions (71 FR 10100-10385 or 29 CFR 1910.1026; 29 CFR 1915.1026; 29 CFR 1926.1126).
Recommended sampling time and sampling rate:	480 min at 2.0 L/min (960 L)
Limit of quantitation:	0.03 $\mu\text{g}/\text{m}^3$ (8-Hour TWA)
Reporting limit:	0.05 $\mu\text{g}/\text{m}^3$
Author:	James C. Ku, Mary Eide (1998, 2006), Yanqiu Sun (2026)

1 Introduction

1.1 Previous Methods used by OSHA for Sampling and Analysis of Hexavalent Chromium on 25-mm PVC filter

The methodologies described in this Appendix are based on OSHA Method ID-215 version 2.¹ That method used Erlenmeyer flask and hot plate without temperature control as extraction apparatus and analyzed by ion chromatography with post-column derivatization using 1,5-diphenylcarbazine and UV-vis detection at 540-nm. Prior to OSHA Method ID-215, OSHA Method ID-103² was used for sampling and analysis of hexavalent chromium [Cr(VI)]. OSHA ID-103 used 37-mm diameter PVC filters for air sampling, sample extraction with carbonate/bicarbonate buffer solution, and analysis by differential pulse polarography. The carbonate/bicarbonate buffer extraction converts both soluble and insoluble chromium-containing chemicals to soluble carbonate compounds and allows both chromium forms to be extracted. OSHA ID-215 utilized a more sensitive and specific ion chromatography analytical method. In addition to carbonate/bicarbonate extraction, OSHA Method ID-215 uses magnesium sulfate/phosphate buffer³ to precipitate potential metal interferences, mainly Fe(II) and Cr(III).

1.2 Changes to the Previously Used Method

The specific medium described in this appendix is 25-mm diameter polyvinyl chloride (PVC) filters with 5- μm pore size. This appendix represents an update to the previous OSHA Method ID-215 version, which was fully validated at the time it was published based on the validation guidelines in effect at that time. Compared to the previous OSHA Method ID-215 version 2, this method uses 20-mL glass scintillation vials, hot block, sample extraction temperature of 93.5 °C, no field stabilization requirement, and modified ion chromatography parameters. Twenty-five-mm diameter PVC filters were evaluated as air sampling media for hexavalent chromium. Data presented from the previous method are identified by the statement "Pre-existing data from OSHA Method ID-215 version 2 are presented in this section". The changes were made to standardize collection, extraction and analysis of hexavalent chromium. Pre-existing data from OSHA Method ID-215 version 2 are validation data for 25-mm PVC filters as air sampling media.

1.3 Validation Parameters

The procedures used to develop the method validation data are described in *Sampling and Analytical Methods*.⁴ The target concentration (5.0 $\mu\text{g}/\text{m}^3$) for this method evaluation was the OSHA 8-hour time-weighted average (TWA) permissible exposure limit (PEL) for hexavalent chromium. The target mass (T_M) is the equivalent analyte mass per sample collected on a sampler at the target concentration using the recommended sampling time and sampling rate.

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The action level is half of the target concentration. For the pre-existing data from OSHA Method ID-215 version 2, the target concentration is 1.0 µg/m³.

2 Limit of Detection, Limit of Quantitation, and Reporting Limit

The limit of detection (LOD) is the analyte mass introduced onto the chromatographic column that produces a response greater than $3.3 \times$ the standard error of estimate ($S_{y/x}$) divided by the slope of the linear regression equation. The linear regression equation is derived from analyses of three spiked samples at five evenly spaced levels, totaling fifteen spiked media samples. Twenty-five-mm diameter polyvinyl chloride (PVC) filters were spiked such that the highest mass loading produced a peak approximately 10 times greater than that of a sample blank at or near the chromatographic retention time of the analyte. The limit of quantitation (LOQ) is expressed as a concentration that provides sufficient analyte mass per sample to produce a response greater than $10 \times S_{y/x}$ divided by the slope of the linear regression equation line described above. The spiked media and three sample blanks were analyzed with the recommended analytical parameters in OSHA Method ID-215, Section 3. The resulting data provided the $S_{y/x}$ and the slope values for LOD and LOQ determinations. Results obtained from these analyses are listed in Table B-1 and plotted in Figure B-1.

Table B-1. LOD and LOQ data for hexavalent chromium on 25-mm PVC filters.

mass per sample (µg/sample)	0.0000	0.0162	0.0323	0.0474	0.0646	0.0808
	0.940	2.36	3.76	4.84	6.24	7.34
area counts ($\times 10^6$)	1.14	2.53	3.45	5.20	6.46	7.98
	1.14	2.39	3.63	5.18	6.05	7.53

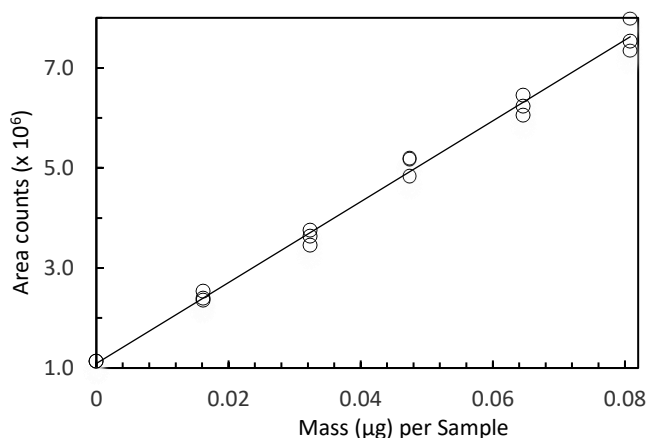


Figure B-1. Plot of data to determine the LOD and LOQ for hexavalent chromium on 25-mm PVC filters ($y = 8.08 \times 10^7 x + 1.09 \times 10^6$, $S_{y/x} = 1.91 \times 10^5$, LOD = 0.00779 µg/sample, LOQ = 0.0236 µg/sample or 0.0246 µg/m³ at the 8-hour TWA).

The reporting limit (RL) is expressed as air concentration at the equivalent mass per sample at the lowest analytical calibration standard providing the recovery is within $\pm 25\%$. Six replicate 25-mm PVC filters were spiked with 0.0496 µg of hexavalent chromium. After drying overnight in the fume hood at ambient temperature, the samples were analyzed using the analytical calibration procedure described in OSHA Method ID-215, Section 3. After analysis, results for hexavalent chromium as a percentage of expected recovery were 111.0%, 115.0%, 119.0%, 123.1%, 117.0%, 121.1%. The mean percentage of expected recovery was 117.7%. The reporting limit is 0.05 µg/m³ at the 8-hour TWA.

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3 Analytical Calibration

The analytical data can be found in OSHA method ID-215 Appendix A, Section 3.

4 Post Sampling Storage

The post sampling storage data can be found in OSHA method ID-215 Appendix A, Section 4.

5 Method Precision and Bias

The method precision and bias data can be found in OSHA method ID-215 Appendix A, Section 5.

6 Analytical Recovery and Stability of Prepared Samples

Analytical recovery is affected by the extraction buffer, the sampling medium, extraction temperature and the technique used to extract samples. The data presented demonstrate validity for the extraction buffer, sampling medium, extraction temperature and extraction technique described in this method. If other combinations of these are to be used, testing must be completed to satisfy the requirements found in current OSHA sampling and analysis method validation guidelines. Acceptable testing results must be documented.

6.1 Pre-existing Data Using Hot Plate and Erlenmeyer Flask

The pre-existing data using a hot plate and Erlenmeyer flask data can be found in OSHA method ID-215 Appendix A, Section 6.1.

6.2 Data using Hot Block and Scintillation Vial

The analytical recovery (R_A) was determined by liquid-spiking six replicate 25-mm PVC filters across a range of analyte mass values that would be obtained from sampling at 0.1 to 2 times the T_C for 480 minutes. Samples were analyzed following overnight drying at ambient temperature (22 °C) in the hood. The R_A was 105.1%. The percent coefficient of variation of the thirty spiked samples (CV_{R_A}) was 2.5%. The data are shown in Table B-2.

Table B-2. Analytical recovery results of 25-mm PVC filters.

xtarget concentration	µg/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)	mean (%)
0.1	0.496	105.6	105.3	108.1	105.3	105.1	106.0	105.9
0.5	2.480	103.3	100.1	104.0	100.0	104.9	103.2	102.6
1.0	4.960	104.0	108.3	107.4	105.9	107.7	103.9	106.2
1.5	7.440	105.2	106.7	106.9	106.3	105.4	98.4	104.8
2.0	9.920	103.1	105.9	111.3	107.5	104.4	104.8	106.2

The stability of sample extracts was determined by liquid-spiking six replicate 25-mm PVC filters with a mass of analyte that would be obtained from sampling at the 8-hour TWA T_C for 480 minutes (calculated to be 5.10 µg). Samples were analyzed following overnight drying at ambient temperature in the hood. Each septum was punctured 1 time for each injection and punctured septa retained in this validation.

All six vials were re-analyzed at 24, 48, and 72 hours after the initial analyses, with all vials remaining in the autosampler tray kept at room temperature (22 °C). Freshly prepared standards (in DBE buffer without media) were used for each analysis event. The linear regression equation determined from these results was $y = -0.00317x + 1.02$. The change in recovery (Δ_{ps_TWA}) of hexavalent chromium calculated from the regression line generated for the 3-day storage test was 0.95%. Results from the analysis are listed in Table B-3.

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Table B-3. Stability of prepared sample data of Cr(VI) on 25-mm PVC filters for 3 days (8-hour TWA).

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	101.3	101.6	101.9	103.0	103.3	101.6
1	101.3	101.4	101.6	101.0	102.2	101.1
2	100.8	100.9	101.7	101.1	102.1	100.4
3	101.0	101.0	101.3	101.0	101.7	100.9

All six vials were re-analyzed at 1, 3, and 5 weeks after the initial analyses together with freshly prepared standards for each analysis event, with vials remaining at room temperature. Freshly prepared standards were used for each analysis event. The results of these analyses are provided in Table B-4. The linear regression equation determined from these results was $y = 0.000661x + 1.03$. The change in recovery (Δ_{ps_TWA}) of hexavalent chromium calculated from the regression line generated for the 5-week storage test was 0.33%.

Table B-4. Stability of prepared sample data of Cr(VI) on 25-mm PVC filters for 5 weeks (8-hour TWA).

time (week)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	101.3	101.6	101.9	103.0	103.3	101.6
1	103.1	103.3	103.3	102.9	103.0	103.1
3	103.2	102.1	103.2	102.5	102.6	102.6
5	103.0	102.1	102.8	102.6	102.8	103.3

The action level ($\frac{1}{2}$ 8-hour TWA) stability of sample extracts was examined by retaining the solvent extract of six spiked 25-mm PVC media at mass loading levels of half T_M (2.49 μg). Each septum was punctured 1 time for each injection and punctured septa retained in this validation.

All six vials were re-analyzed at 24, 48, and 72 hours after the initial analyses, with all vials remaining in the autosampler tray and kept at room temperature. Freshly prepared standards (in DBE buffer without media) were used for each analysis event. The linear regression equation determined from these results was $y = -0.00258x + 1.02$. The change in recovery (Δ_{ps_action}) of hexavalent chromium calculated from the regression line generated for the 3-day storage test was 0.78%. Results from the analysis are listed in Table B-5.

Table B-5. Stability of prepared sample data of Cr(VI) on 25-mm PVC filters for 3 days (Action Level).

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	102.3	102.8	100.8	105.2	99.8	103.9
1	100.4	102.8	101.3	104.9	100.0	103.1
2	101.1	102.5	100.5	103.4	99.4	103.0
3	100.7	102.3	101.1	104.2	99.5	102.7

All six vials were re-analyzed at 2, 3, and 5 weeks after the initial analyses together with freshly prepared standards for each analysis event, with all vials standing at room temperature. Freshly prepared standards were used for each analysis event. The results of these analyses are provided in Table B-6. The linear regression equation determined from these results was $y = 0.00117x + 1.02$. The change in recovery (Δ_{ps_action}) of hexavalent chromium calculated from the regression line generated for the 5-week storage test was 0.58%. Results from the analysis are listed in Table B-6.

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Table B-6. Stability of prepared sample data of Cr(VI) on 25-mm PVC filters for 5 weeks (Action Level).

time (week)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	102.3	102.8	100.8	105.2	99.8	103.9
2	101.8	102.9	101.5	105.0	100.2	103.3
3	102.1	102.9	101.7	105.1	99.3	103.8
5	102.3	103.6	101.7	106.0	100.0	104.8

7 Sampler capacity

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section

It was not possible to safely generate a test atmosphere of Cr(VI); therefore, retention efficiency tests were performed to support the recommended air volume.

A retention efficiency test for soluble Cr(VI) on 25-mm PVC filters was performed by spiking 1920 ng of Cr(VI) onto the filters, allowing them to dry, and then placing them into 25-mm polystyrene cassettes with a BUP. A sampling train was constructed by placing a cassette containing the spiked 25-mm PVC filter with a BUP and a second cassette with a blank 25-mm PVC filter and a BUP. The air flowed through the cassette containing the spiked 25-mm PVC filter and then through the second (back) cassette. These sampling trains each had 960 liters of air with 80% relative humidity at 23°C drawn through them at 2 L/min. There was no Cr(VI) found on the 25-mm PVC filters on the back cassettes or on the BUPs.

Table B-7. Retention efficiency test for soluble Cr(VI) spiked on 25-mm PVC filters.

cassette location	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)	mean (%)
front PVC	99.9	98.8	95.3	97.7	96.9	98.2	97.8
front BUP	0.0	0.0	0.0	0.0	0.0	0.0	0.0
back PVC	0.0	0.0	0.0	0.0	0.0	0.0	0.0
back BUP	0.0	0.0	0.0	0.0	0.0	0.0	0.0
total	99.9	98.8	95.3	97.7	96.9	98.2	97.8

8 Effect of Humidity and Low Sampling Concentration

The effect of humidity and a low sampling concentration data can be found in OSHA method ID-215 Appendix A, Section 8.

9 Sampling and Analytical Interferences

The sampling and analytical interferences data can be found in OSHA method ID-215 Appendix A, Section 9.

Twenty-five-mm PVC filter medium was evaluated as air sampling media. Chromium plating samples collected on PVC filters must either be analyzed within 6 days of sampling or be stabilized immediately upon arrival at the analytical laboratory. Welding samples must be analyzed within 8 days after sampling (Appendix A, Sections 4 and 9). Spray-paint samples are extracted with hot BE/PBM followed by a second extraction with hot SPE/PBM (OSHA Method ID-215, Section 3.5). It is routine procedure to wipe the interior walls of sampling cassettes for all air samples analyzed at OTC (Appendix A, Section 7). The sample filter and cassette wiping PVC filter are put in the same vial and analyzed together instead of analyzed separately. Wipe blank samples cassettes and treat blank samples the same as the other samples. Confirm the presence of hexavalent chromium as described in OSHA Method ID-215, Section 3.8.

Suspected interferences information should be reported to the laboratory with submitted samples. The interference studies were performed in OSHA Method ID-215 version 2 and should be consulted for specific information about the

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metal species of interest. The major positive interference is Cr(III), which can be oxidized to Cr(VI). The major negative interference is Fe(II), which can reduce the Cr(VI) to Cr(III). Other reducing metal species can also change Cr(VI) to Cr(III).

10 Analytical Reproducibility

The analytical reproducibility data can be found in OSHA Method ID-215 Appendix A, Section 10.

11 Estimation of Uncertainty

While systematic biases such as analyte storage loss are examined, and limits are placed on these, an estimation of uncertainty that encompasses both potential random and systematic error was not completed. Instead, the overall standard error of estimate was calculated from the random error inherent to the points about the regression line produced by the ambient storage test described in Appendix A, Section 5, as prescribed by the OSHA validation guidelines in use at the time OSHA Method ID 215 version 2 was originally validated. See Section 5 of Appendix A for details.

References

1. Ku, J., Eide, M. Hexavalent Chromium (OSHA Method ID-215 version 2), 2006. United States Department of Labor, Occupational Safety and Health Administration website. www.osha.gov (accessed March 2026).
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4. Sampling and Analytical Methods, 2024. United States Department of Labor, Occupational Safety & Health Administration Website. www.osha.gov (accessed March 2026).

Appendix C, NaOH Coated Binderless Quartz Fiber Filter (NaOHqz)

OSHA PEL:	5.0 µg/m ³ 8-Hour TWA (2.5 µg/m ³ Action Level), General Industry, Shipyards, and Construction. See hexavalent chromium standard for compliance dates and special provisions (71 FR 10100-10385 or 29 CFR 1910.1026; 29 CFR 1915.1026; 29 CFR 1926.1126).
Recommended sampling time and sampling rate:	480 min at 2.0 L/min (960 L)
Limit of quantitation:	0.33 µg/m ³ with one blank correction
Reporting limit:	0.33 µg/m ³ with one blank correction
Author:	James C. Ku, Mary Eide (1998, 2001, 2006), Yanqiu Sun (2026)

1 Introduction

1.1 Previous Methods used by OSHA for Sampling and Analysis of Hexavalent Chromium on NaOHqz Filter

The methodologies described in this Appendix are based on OSHA Method ID-215 version 2.¹ The method used Erlenmeyer flask and hot plate without temperature control as extraction apparatus and analyzed by ion chromatography with post-column derivatization using 1,5-diphenylcarbazide and UV-vis detection at 540-nm. Prior to OSHA Method ID-215, OSHA Method ID-103² was used for sampling and analysis of hexavalent chromium [Cr(VI)]. OSHA ID-103 used 37-mm diameter PVC filters for air sampling, sample extraction with carbonate/bicarbonate buffer solution, and analysis by differential pulse polarography. The carbonate/bicarbonate buffer solution extraction converts both soluble and insoluble chromium-containing chemicals to soluble carbonate compounds and allows both chromium forms to be extracted. OSHA ID-215 utilized a more sensitive and specific ion chromatography analytical method. In addition to carbonate/bicarbonate extraction, OSHA Method ID-215 uses magnesium sulfate/phosphate buffer³ to precipitate potential metal interferences, mainly Fe(II) and Cr(III). OSHA Method ID-215 adopted NaOHqz filter as the sampling medium in plating operations.⁴

1.2 Changes to the Previously Used Method

The specific medium described in this appendix is 1.0 M NaOH coated 37-mm binderless quartz fiber filter, 0.38 mm thick (NaOHqz). This appendix represents an update to the previous OSHA Method ID-215 version, which was fully validated at the time they were published based on the validation guidelines in effect at that time. Compared to the previous OSHA Method ID-215 version 2, this method uses 20-mL glass scintillation vials, hot block, sample extraction temperature of 93.5 °C, no field stabilization requirement, and modified ion chromatography parameters. NaOHqz filters were evaluated as air sampling media for hexavalent chromium. Data presented from the previous methods are identified by the statement "Pre-existing data from OSHA Method ID-215 version 2 are presented in this Section". The changes were made to standardize collection, extraction and analysis of hexavalent chromium. Pre-existing data from OSHA Method ID-215 version 2 are validation data for NaOHqz filters as air sampling media.

1.3 Validation Parameters

The procedures used to develop the method validation data are described in *Sampling and Analytical Methods*⁵, and *Evaluation Guidelines for Surface Sampling Methods*.⁶ The target concentration (5.0 µg/m³) for this method evaluation was the OSHA 8-hour time-weighted average (TWA) permissible exposure limit (PEL) for hexavalent chromium. The

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target mass (T_M) is the equivalent analyte mass per sample collected on a sampler at the target concentration using the recommended sampling time and sampling rate. The action level is half of the target concentration. For the pre-existing data from OSHA Method ID-215 version 2, the target concentration is $1.0 \mu\text{g}/\text{m}^3$.

2 Limit of Detection, Limit of Quantitation, and Reporting Limit

One blank correction was required for the samples collected on the NaOHqz filters. The limit of detection (LOD) is the analyte mass introduced onto the chromatographic column that produces a response greater than $3.3 \times$ the standard error of estimate ($S_{y/x}$) divided by the slope of the linear regression equation and multiplied by the square root of two. The linear regression equation is derived from analyses of three spiked samples at five evenly spaced levels, totaling fifteen spiked media samples. NaOHqz filters were spiked such that the highest mass loading produced a peak approximately 10 times greater than that of a sample blank at or near the chromatographic retention time of the analyte. The limit of quantitation (LOQ) for NaOHqz filters is expressed as a concentration that provides sufficient analyte mass per sample to produce a response greater than $10 \times S_{y/x}$ divided by the slope of the linear regression equation described above and multiplied by the square root of two. The spiked media and three sample blanks were analyzed with the recommended analytical parameters in OSHA Method ID-215, Section 3. The resulting data provided the $S_{y/x}$ and the slope values for LOD and LOQ determinations. Results obtained from these analyses are listed in Table C-1 and plotted in Figure C-1.

Table C-1. LOD and LOQ data for hexavalent chromium on NaOHqz filters.

mass per sample ($\mu\text{g}/\text{sample}$)	0.000	0.103	0.309	0.515	0.721	0.927
area counts ($\times 10^7$)	0.679	1.37	2.92	4.47	6.13	7.36
	0.680	1.18	2.87	4.35	6.31	7.87
	0.685	1.51	3.06	4.78	6.41	7.92

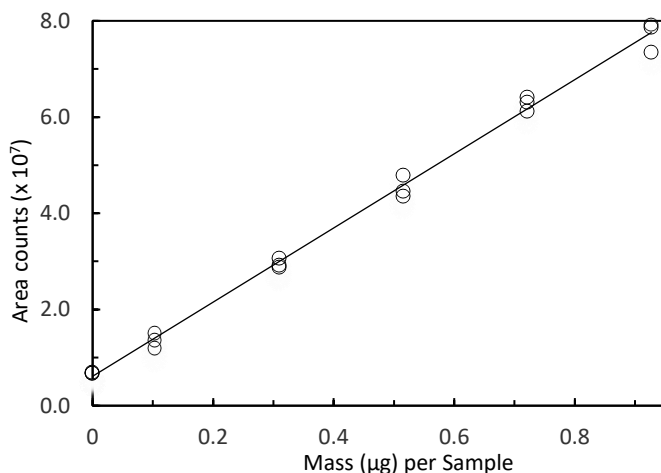


Figure C-1. Plot of data to determine the LOD and LOQ for hexavalent chromium on NaOHqz filters ($y = 7.71 \times 10^7 x + 6.11 \times 10^6$, $S_{y/x} = 1.74 \times 10^6$, LOD = $0.106 \mu\text{g}/\text{sample}$, LOQ = $0.320 \mu\text{g}/\text{sample}$ or $0.333 \mu\text{g}/\text{m}^3$ at the 8-hour TWA) with one blank correction.

The reporting limit (RL) is expressed as a concentration at the equivalent mass per sample at the lowest analytical calibration standard providing the recovery is within $\pm 25\%$. Six replicate NaOHqz filters were spiked with $0.330 \mu\text{g}$ of hexavalent chromium. After drying overnight in the fume hood at ambient temperature, the samples were analyzed using the analytical calibration procedure described in OSHA Method ID-215, Section 3. After analysis, results for hexavalent chromium as a percentage of expected recovery were 104.7%, 104.4%, 108.3%, 108.9%, 113.8% and 106.2%. The mean percentage of expected recovery was 107.7%. The reporting limit is $0.33 \mu\text{g}/\text{m}^3$ at the 8-hour TWA.

OSHA Method ID-215, Hexavalent Chromium

3 Analytical Calibration

The data can be found in OSHA method ID-215 Appendix A, Section 3.

4 Post Sampling Storage

Soluble Cr(VI) is defined as Cr(VI) from $K_2Cr_2O_7$ dissolved in DI water; and insoluble Cr(VI) as Cr(VI) from $PbCrO_4$ dissolved in BE solution. Lead chromate ($PbCrO_4$) is not soluble in DI water, but readily soluble in warm BE.

4.1 Soluble Cr(VI) Spiked on NaOHqz Filters

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

Storage samples were prepared by spiking NaOHqz with 960 ng of soluble Cr(VI). The spiked NaOHqz had 960 L of air at 80% relative humidity (RH) and 23 °C drawn through them at 2 L/min. Twenty-seven storage samples were prepared. Three samples were analyzed on the day of preparation. Twelve of the filters were stored at reduced temperature (4 °C) and the other twelve were stored in a closed drawer at ambient temperature (about 22 °C). At 3- to 4-day intervals, three samples were selected from each of the two storage sets and analyzed. Sample results were not corrected for extraction efficiency. On day 14 the recovery was 96.3% for samples stored at ambient temperature and 96.0% for samples stored at refrigerated temperature. The recovery was obtained from the equation of the storage graphs.

Table C-2. Ambient storage recovery test for soluble Cr(VI) spiked on NaOHqz filter.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	99.0	98.6	97.1
3	97.4	98.8	98.3
7	96.9	98.1	98.3
10	98.0	96.9	97.2
14	94.9	95.8	96.6

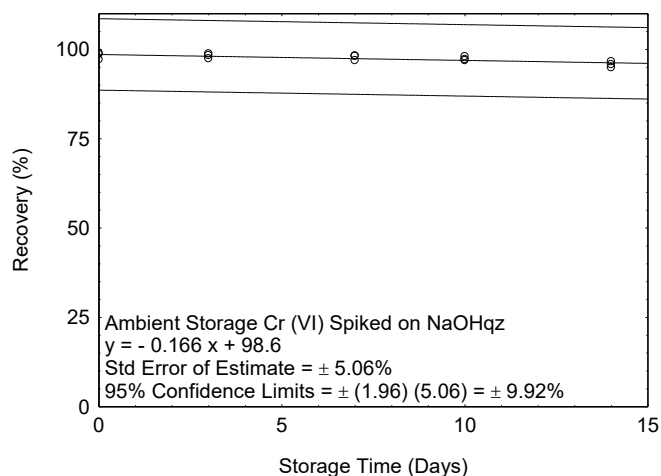


Figure C-2. Ambient storage test for soluble Cr(VI) spiked on NaOHqz filter.

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Table C-3. Refrigerated storage recovery test for soluble Cr(VI) spiked on NaOHqz filter.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	99.0	98.6	97.1
3	99.0	97.4	96.6
7	98.2	96.1	96.9
10	98.1	94.9	95.9
14	97.1	95.2	96.1

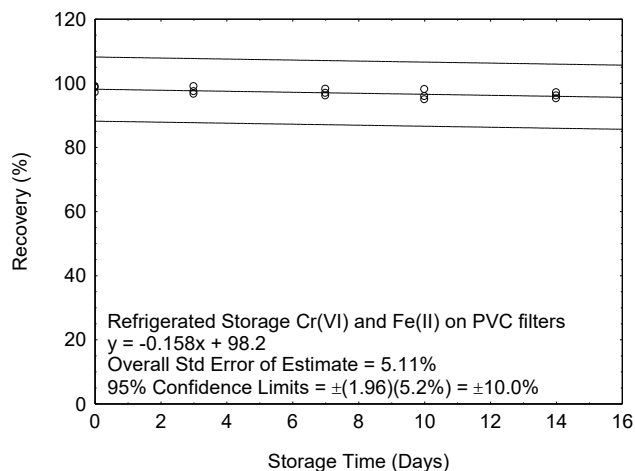


Figure C-3. Refrigerated storage test for soluble Cr(VI) spiked on NaOHqz filter.

4.2 Insoluble Cr(VI) Spiked on NaOHqz Filters

Because NaOHqz filters are only used in chromium plating operations sampling, the only form present is soluble Cr(VI).

4.3 Cr(VI) and H₂SO₄ Spiked on NaOHqz Filters

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The main interferences in chromium plating operations are the acids in the baths (mainly sulfuric acid but also possibly phosphoric acid or other mineral acids) and Cr(III). The ability of NaOHqz to retain Cr(VI) in the presence of these interferences was tested by spiking 960 ng Cr(VI) onto each of three filters, along with 100 ng of Cr(III) and 50 ng of H₂SO₄ and then placing them into polystyrene cassettes. The cassettes then had 960 liters of air with 79% RH at 23 °C drawn through them at 2 L/min. All the samples were immediately analyzed. The analytical results were 98.9%, 99.7% and 99.5% of theoretical, with a mean of 99.4%.

The storage stability of Cr(VI) in the presence of H₂SO₄ was tested by spiking 27 NaOHqz filters with 960 ng of soluble Cr(VI) and 50 ng of H₂SO₄ and allowing the filters to dry. The spiked NaOHqz had 960 L of air with 80% RH at 23 °C drawn through them at 2 L/min. Three samples were analyzed on the day of preparation. Twelve of the filters were stored at reduced temperature (4 °C) and the other twelve were stored in a closed drawer at ambient temperature (about 22 °C). At 3 to 4-day intervals, three samples were selected from each of the two storage sets and analyzed. The test results show excellent storage stability.

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Table C-4. Ambient storage stability/recovery test of soluble Cr(VI) and H₂SO₄ on NaOHqz filter.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.2	99.1	96.7
3	97.8	96.9	98.4
7	96.3	97.7	98.1
10	95.8	97.8	96.2
14	94.5	95.9	96.9

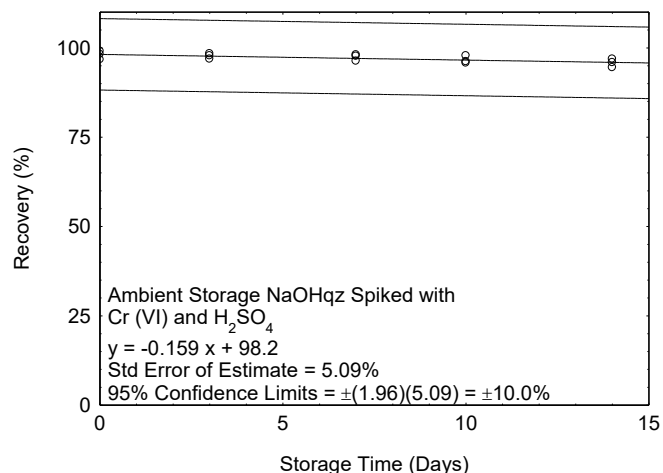


Figure C-4. Ambient storage test for soluble Cr(VI) and H₂SO₄ spiked on NaOHqz filter.

Table C-5. Refrigerated storage stability/recovery test of soluble Cr(VI) and H₂SO₄ on NaOHqz filter.

time (days)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.2	99.1	96.7
3	97.7	98.3	96.4
7	97.6	95.9	96.6
10	97.9	95.5	94.8
14	96.9	94.4	95.9

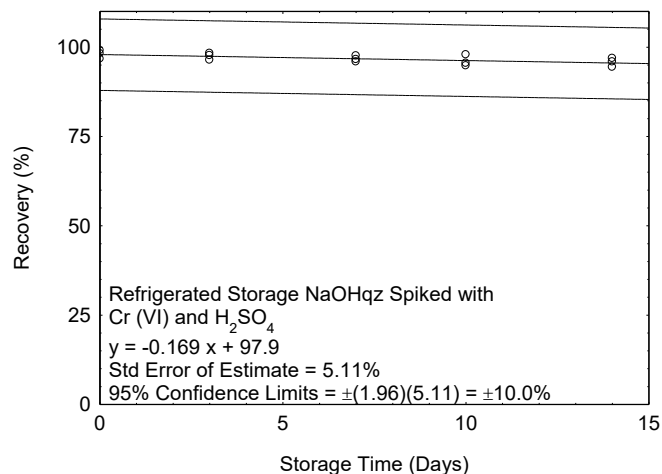


Figure C-5. Refrigerated storage test for soluble Cr(VI) and H₂SO₄ spiked on NaOHqz filter.

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5 Method Precision and Bias

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The precision at the 95% confidence level was obtained by multiplying the standard error of estimate for the appropriate storage stability test by 1.96 (the z-statistic from the standard normal distribution at the 95% confidence level). In Appendix C, Section 4, 95% confidence intervals are drawn about their respective regression lines in the storage graph figures. The precision of the overall procedure of $\pm 9.92\%$ was obtained from the standard error of estimate of 5.06% in Figure C-2 for NaOHqz filters. Each precision includes an additional $\pm 5\%$ for sampling error.

6 Analytical Recovery and Stability of Prepared Samples

Analytical recovery is affected by the extraction buffer, the sampling medium, extraction temperature and the technique used to extract samples. The data presented demonstrate validity for the extraction buffer, sampling medium, extraction temperature and extraction technique described in this method. If other combinations of these are to be used, testing must be completed to satisfy the requirements found in current OSHA sampling and analysis method validation guidelines. Acceptable testing results must be documented.

6.1 Pre-existing Data Using Hot Plate and Erlenmeyer Flask

The target concentration refers to $1.0 \mu\text{g}/\text{m}^3$ in OSHA Method ID-215 version 2. The reliable quantitation limit (RQL) is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters obtained for the calculation of the detection limit of the overall procedure, providing 75% to 125% of the analyte is recovered. The RQL for NaOHqz is $3.12 \text{ ng}/\text{sample}$ ($3.2 \text{ ng}/\text{m}^3$). Recovery at this concentration is 97.8%.

Extraction Efficiency for Soluble Cr(VI) with BE/PBM from NaOHqz Filter

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The extraction efficiency of Cr(VI) was determined by liquid-spiking NaOHqz with soluble Cr(VI) at masses ranging from 3 to 1920 ng. These samples were stored overnight at ambient temperature and then analyzed. Filters were prepared for analysis following the primary extraction procedure in sample preparation (OSHA Method ID-215, Section 3.5). The mean extraction efficiency over the range of 3 to 1920 ng was 97.7%. Wet extraction efficiency samples were prepared by loading the filters with water by pulling 960 L of humid air (79% RH at 22 °C) through the filters at 2 L/min before spiking. The wet extraction efficiency was not included in the overall mean.

Table C-6. Extraction efficiency for soluble Cr(VI) from NaOHqz filter with BE/PBM.

*target concentration	ng/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	mean (%)
RQL	3	96.9	99.2	97.9	97.2	97.8
0.25	240	98.9	97.4	96.4	96.4	97.3
0.5	480	98.8	97.7	99.1	96.8	98.1
1.0	960	99.2	98.2	97.1	95.7	97.6
1.5	1440	98.9	97.1	99.0	96.0	97.8
2.0	1920	98.3	96.9	97.5	98.2	97.7
1.0 (wet)	960	99.2	96.7	97.5	96.2	97.4

The stability of extracted samples was investigated by reanalyzing the target concentration samples 24 hours after initial analysis. After the original analysis was performed, two vials were recapped with new septa while the remaining two retained their punctured septa. The samples were reanalyzed with fresh standards. The average percentage change was -4.2% for samples that were resealed with new septa and -3.9% for those that retained their punctured septa. Each septum was punctured 1 time for each injection.

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Table C-7. Stability of extracted samples for soluble Cr(VI) with BE/PBM from NaOHqz filter with punctured septa replaced.

samples	Initial recovery (%)	after one day recovery (%)	difference (%)
sample 1	99.2	95.3	-3.9
sample 2	98.2	93.9	-4.3
mean	98.7	94.6	-4.1

Table C-8. Stability of extracted samples for soluble Cr(VI) with BE/PBM from NaOHqz filter with punctured septa retained.

samples	Initial recovery (%)	after one day recovery (%)	difference (%)
sample 1	97.1	93.3	-3.8
sample 2	95.7	91.7	-4.0
mean	96.4	92.5	-3.9

6.2 Data using Hot Block and Scintillation Vial

The analytical recovery (R_A) was determined by liquid-spiking six replicate NaOHqz filters across a range of analyte mass values that would be obtained from sampling at 0.1 to 2 times the 8-Hour T_C for 480 minutes. Samples were analyzed following overnight drying at ambient temperature (22 °C) in the hood. The R_A was 105.2%. The percent coefficient of variation of the thirty spiked samples (CV_{R_A}) was 3.4%. The data are shown in Table C-9.

Table C-9. Analytical recovery results of NaOHqz.

× target concentration	µg/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)	mean (%)
0.1	0.496	106.8	110.0	111.4	116.3	107.8	100.4	108.8
0.5	2.480	102.4	102.6	102.3	102.3	109.0	108.0	104.4
1	4.960	107.3	103.2	108.7	103.9	103.3	103.2	104.9
1.5	7.440	104.0	105.0	103.3	103.0	102.9	101.7	103.3
2	9.920	101.5	102.7	102.6	105.0	108.7	105.2	104.3

The stability of sample extracts was determined by liquid-spiking six replicate NaOHqz filters with a mass of analyte that would be obtained from sampling at the 8-Hour TWA T_C for 480 minutes (calculated to be 5.10 µg). Samples were analyzed following overnight drying at ambient temperature in the hood. Each septum was punctured 1 time for each injection and punctured septa retained in this validation.

All six vials were re-analyzed at 24, 48, and 72 hours after the initial analyses, with all vials remaining in the autosampler tray kept at room temperature (22 °C). Freshly prepared standards (in DBE buffer without media) were used for each analysis event. The linear regression equation determined from these results was $y = -0.00153x + 1.05$. The change in recovery (Δ_{ps_TWA}) of hexavalent chromium calculated from the regression line generated for the 3-day storage test was 0.46%. Results from the analysis are listed in Table C-10.

Table C-10. Stability of prepared sample data of Cr(VI) on NaOHqz filters for 3 days (8-hour TWA).

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	105.3	99.0	106.9	109.1	108.7	103.7
1	103.7	98.3	106.3	107.5	107.4	102.7
2	103.6	97.7	106.1	104.6	107.9	103.7
3	104.2	98.3	107.0	108.4	108.8	103.7

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All six vials were re-analyzed at 1, 3, and 5 weeks after the initial analyses together with freshly prepared standards for each analysis event, with vials standing at room temperature. Freshly prepared standards were used for each analysis event. The results of these analyses are provided in Table C-11. The linear regression equation determined from these results was $y = 0.00262x + 1.06$. The change in recovery (Δ_{ps_TWA}) of hexavalent chromium calculated from the regression line generated for the 5-week storage test was 1.31%.

Table C-11. Stability of prepared sample data of Cr(VI) on NaOHqz filters for 5 weeks (8-hour TWA).

time (week)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	105.3	99.0	106.9	109.1	108.7	103.7
1	105.6	102.6	109.7	109.6	109.7	104.8
3	105.4	102.8	109.5	109.5	109.9	104.3
5	104.9	103.2	109.7	110.3	110.3	104.6

The action level ($\frac{1}{2}$ 8-hour TWA) stability of sample extracts was examined by retaining the solvent extract of six spiked NaOHqz media at mass loading level of $\frac{1}{2} T_M$ (2.49 μg). Each septum was punctured 1 time for each injection and punctured septa retained in this validation.

All six vials were re-analyzed at 24, 48, and 72 hours after the initial analyses, with all vials remaining in the autosampler tray kept at room temperature. Freshly prepared standards (in DBE buffer without media) were used for each analysis event. The results of these analyses are provided in Table C-12. The regression line equation determined from these results was $y = -0.00252x + 1.10$. The change in recovery (Δ_{ps_action}) of hexavalent chromium calculated from the regression line generated for the 3-day storage test was 0.76%.

Table C-12. Stability of prepared sample data of Cr(VI) on NaOHqz filters for 3 days (Action Level).

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	107.3	110.0	104.8	116.7	114.7	110.1
1	106.7	108.6	104.1	114.6	113.4	109.0
2	107.0	109.1	104.4	115.5	113.3	108.8
3	106.3	109.3	103.9	115.4	114.0	109.0

All six vials were re-analyzed at 2, 3, and 5 weeks after the initial analyses together with freshly prepared standards for each analysis event, with all vials standing at room temperature. Freshly prepared standards were used for each analysis event. The results of these analyses are provided in Table C-13. The linear regression equation determined from these results was $y = 0.00184x + 1.11$. The change in recovery (Δ_{ps_action}) of hexavalent chromium calculated from the regression line generated for the 5-week storage test was 0.92%.

Table C-13. Stability of prepared sample data of Cr(VI) on NaOHqz filters for 5 weeks (Action Level).

time (week)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	107.3	110.0	104.8	116.7	114.7	110.1
2	107.5	109.4	109.8	115.9	114.1	109.7
3	108.2	110.5	110.3	116.5	114.4	110.2
5	107.5	109.9	110.3	116.5	114.3	110.1

7 Sampler capacity

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

It was not possible to safely generate a test atmosphere of Cr(VI); therefore, retention efficiency tests were performed to support the recommended air volume.

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A retention efficiency test for soluble Cr(VI) on NaOHqz was performed by spiking 1920 ng of Cr(VI) onto the filters, allowing them to dry, and then placing them into 37-mm polystyrene cassettes. A sampling train was constructed by placing the cassette containing the spiked NaOHqz in series with a second cassette containing a clean NaOHqz. The air flowed through the cassette containing the spiked NaOHqz and then through the second (back) cassette. These sampling trains each had 960 liters of air at 80% relative humidity at 23 °C drawn through them at 2 L/min. There was no Cr(VI) found on the NaOHqz in the back cassettes.

Table C-14. Retention efficiency test for soluble Cr(VI) spiked on NaOHqz.

cassette location	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)	mean (%)
front NaOHqz	99.2	98.8	97.1	97.9	96.8	99.0	98.1
back NaOHqz	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total	99.2	98.8	97.1	97.9	96.8	99.0	98.1

8 Effect of Humidity and Low Sampling Concentration

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

The ability of NaOHqz to retain Cr(VI) in a relatively dry atmosphere was tested by spiking 1920 ng of Cr(VI) onto each of three filters and placing them into polystyrene cassettes. The cassettes then had 960 liters of air with 19% RH at 24 °C drawn through them at 2 L/min. All the samples were immediately analyzed. The analytical results were 97.1%, 96.3% and 98.4% of theoretical, with a mean of 97.3%.

The ability of NaOHqz to retain Cr(VI) at low concentration was tested by spiking 96 ng of Cr(VI) onto each of three filters and placing them into polystyrene cassettes. The cassettes then had 960 liters of air with 79% RH at 23 °C drawn through them at 2 L/min. All the samples were immediately analyzed. The analytical results were 98.6%, 97.1% and 95.9% of theoretical, with a mean of 97.2%.

9 Sampling and Analytical Interferences

The main interferences in chromium plating operations are the acids in the baths which will be neutralized by sodium hydroxide on the NaOHqz filters. Suspected interferences should be reported to the laboratory with submitted samples. The interference studies were performed in OSHA Method ID-215 version 2 and should be consulted for specific information about the metal species of interest. The major positive interference is Cr(III), which can be oxidized to Cr(VI). The major negative interference is Fe(II), which can reduce the Cr(VI) to Cr(III). Other reducing metal species can also change Cr(VI) to Cr(III). It is routine procedure to wipe the interior walls of sampling cassettes for all air samples analyzed at OTC (Appendix A, Section 7). The sample filter and cassette wiping PVC filter are put in the same vial and analyzed together instead of analyzed separately. Wipe blank samples cassettes and treat blank samples the same as the other samples. Confirm the presence of hexavalent chromium as described in OSHA Method ID-215, Section 3.8.

10 Analytical Reproducibility

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

Reproducibility samples were prepared by spiking six NaOHqz filters with 1.0 µg of soluble Cr(VI) and allowed to dry, and then drawing 960 liters of air at 80% RH and 23 °C through them at 2 L/min. The resulting samples were submitted to the OSHA Technical Center for analysis using OSHA Method ID-215 version 2 after being stored for 7 days at 23 °C. The reproducibility data for NaOHqz filters are presented in Table C-15. No sample result for Cr(VI) had a deviation greater than the precision of the overall procedure presented in Appendix C, Section 5.

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Table C-15. Reproducibility data for Cr(VI) spiked on NaOHqz filters.

theoretical (µg/sample)	recovered (µg/sample)	recovery (%)	deviation (%)
1.0	0.983	98.3	-1.7
1.0	0.949	94.9	-5.1
1.0	0.968	96.8	-3.2
1.0	0.986	98.6	-1.4
1.0	0.959	95.9	-4.1
1.0	0.971	97.1	-2.9

11 Estimation of Uncertainty

While systematic biases such as analyte storage loss are examined, and limits are placed on these, an estimation of uncertainty that encompasses both potential random and systematic error was not completed. Instead, the overall standard error of estimate was calculated from the random error inherent to the points about the regression line produced by the ambient storage test described in Appendix C, Section 5, as prescribed by the OSHA validation guidelines in use at the time OSHA Method ID 215 was originally validated. See Section 5 of this appendix for details.

12 Preparation and Storage of the 1.0 M NaOH Coated Binderless Quartz Fiber Filters (NaOHqz Filters)

Pre-existing data from OSHA Method ID-215 version 2 are presented in this section.

NaOHqz filters are prepared by placing binderless quartz fiber filters onto the rim of clean glass beakers (20 or 50-mL size), pipetting 0.5 mL of a 1.0 M NaOH (4 g NaOH in 100 mL of DI water) solution onto each filter, and allowing the filter to dry under nitrogen at either ambient temperature for four hours or in a vacuum oven at 100 °C for 30 min. After filters are dry, analyze three NaOH coated binderless quartz fiber filters with one blank QFF filter to determine the amount of background Cr(VI). If the background Cr(VI) is above 0.1 µg, fresh filters should be prepared after the source of the contamination is identified. The source of contamination may be the NaOH, binderless quartz fiber filters, or both. Binderless quartz fiber filters can be heated in BE and then rinsed in DI water to remove the contamination. The filters can be stored in room air for 1 month or under nitrogen for 4 months. The filters used in this evaluation were prepared at OTC.

References

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3. Vitale, R. J., Mussoline, G. R., Petura, J. C., James, B. R. Hexavalent Chromium Extraction from Soils. *J. Environ. Qual.* **1994**, Vol. 23, 1249-1256.
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Appendix D, Binderless Quartz Fiber Filter (QFF)

OSHA PEL:	OSHA has a housekeeping requirement that all surfaces are maintained as free as practicable from accumulations of hexavalent chromium (29 CFR 1910.1026).
Limit of quantitation:	0.18 µg/sample
Reporting limit:	0.33 µg/sample
Author:	Mary E. Eide (2001), Yanqiu Sun (2026)

1 Introduction

1.1 Previous Methods used by OSHA for Sampling and Analysis of Hexavalent Chromium on QFF Filter

The methodologies described in this Appendix are based on OSHA Method W4001.¹ OSHA Method W4001 was developed to provide a means of taking wipe samples for hexavalent chromium. The method used Erlenmeyer flask and hot plate without temperature control as extraction apparatus and analyzed by ion chromatography with post-column derivatization using 1,5-diphenylcarbazide and UV-vis detection at 540-nm.

1.2 Changes to the Previously Used Method

This appendix represents an update to the previous OSHA Method W4001, which was fully validated at the time it was published based on the validation guidelines in effect at that time. Compared to the previous OSHA Method W4001, this method uses 20-mL glass scintillation vials, hot block, sample extraction temperature of 93.5 °C, no field stabilization requirement, and modified ion chromatography parameters. The specific medium described in this appendix is 37-mm binderless quartz fiber filter, 0.38 mm thick (QFF). Data presented from the previous methods are identified by the statement "Pre-existing data from OSHA Method W4001 are presented in this section". The changes were made to standardize collection, extraction and analysis of hexavalent chromium. Pre-existing data from OSHA Method W4001 are validation data for QFF filters as wipe sampling media.

1.3 Validation Parameters

The procedures used to develop the method validation data are described in *Sampling and Analytical Methods*², and *Evaluation Guidelines for Surface Sampling Methods*.³ The target concentration is 5 µg/sample in this validation. The target mass (T_M) is 5.0 µg. The action level is half of the target concentration. Note, the target concentration is 0.050 µg/100 cm² in the pre-existing data from OSHA Method W4001.

2 Limit of Detection, Limit of Quantitation, and Reporting Limit

The limit of detection (LOD) is the analyte mass introduced onto the chromatographic column that produces a response greater than $3.3 \times$ the standard error of estimate ($S_{y/x}$) divided by the slope of the linear regression equation. The linear regression equation is derived from analyses of three spiked samples at five evenly spaced levels, totaling fifteen spiked media samples. Binderless quartz fiber filters (QFF) were spiked such that the highest mass loading produced a peak approximately 10 times greater than that of a sample blank at or near the chromatographic retention time of the analyte. The limit of quantitation (LOQ) is expressed as a concentration that provides sufficient analyte mass per sample to produce a response greater than $10 \times S_{y/x}$ divided by the slope of the linear regression equation line described above. The spiked media and three sample blanks were analyzed with the recommended analytical parameters in OSHA

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Method ID-215, Section 3. The resulting data provided the $S_{y/x}$ and the slope values for LOD and LOQ determinations. Results obtained from these analyses are listed in Table D-1 and plotted in Figure D-1.

Table D-1. LOD and LOQ data for hexavalent chromium on binderless quartz fiber filters.

mass per sample (µg/sample)	0.000	0.103	0.309	0.515	0.721	0.927
	0.415	1.167	2.673	4.266	5.807	7.688
area counts ($\times 10^7$)	0.449	1.266	2.611	4.235	5.995	7.662
	0.545	1.071	2.712	4.238	6.151	7.900

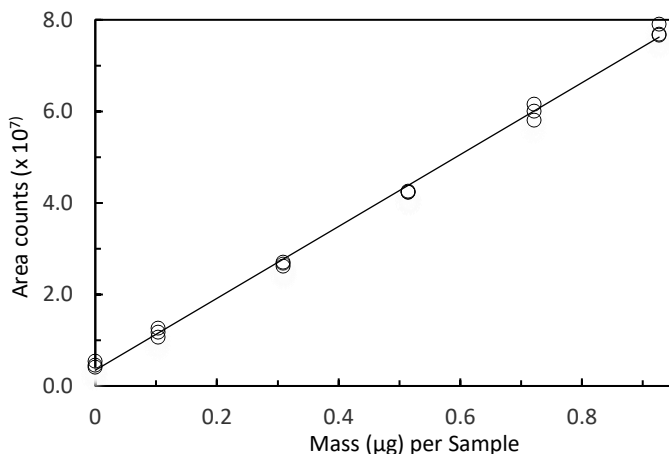


Figure D-1. Plot of data to determine the LOD and LOQ for hexavalent chromium on binderless quartz fiber filters ($y = 7.85 \times 10^7 x + 3.45 \times 10^6$, $S_{y/x} = 1.41 \times 10^6$, LOD = 0.0593 µg/sample, LOQ = 0.180 µg/sample).

The reporting limit (RL) is expressed as a concentration at the equivalent mass per sample at the lowest analytical calibration standard providing the recovery is within $\pm 25\%$. Six replicate QFF filters were spiked with 0.330 µg of hexavalent chromium. After drying overnight in the fume hood at ambient temperature, the samples were analyzed using the analytical calibration procedure described in OSHA Method ID-215, Section 3. After analysis, results for hexavalent chromium as a percentage of expected recovery were 111.0%, 102.5%, 104.7%, 102.5%, 113.2%, 114.1%. The mean percentage of expected recovery was 108.0%. The reporting limit is 0.33 µg/sample.

3 Analytical Calibration

The data can be found in OSHA Method ID-215 Appendix A, Section 3.

4 Post Sampling Storage

4.1 Cr(VI) Spiked on QFF Filters

Pre-existing data from OSHA Method W4001 are presented in this section.

Storage samples were prepared by spiking binderless quartz fiber filters with Cr(VI). The binderless quartz fiber filter was spiked with the target concentration of Cr(VI) and allowed to dry. Twenty-one storage samples were prepared. Three samples were analyzed on the day of preparation. Nine of the filters were stored at reduced temperature (4°C) and the other nine were stored in a closed drawer at ambient temperature (about 22 °C). At 5-day intervals, three samples were selected from each of the two storage sets and analyzed. Sample results were not corrected for extraction efficiency. The recoveries on Day 15 were 96.4% at ambient temperature and 96.2% at refrigerated temperature. Results from these analyses are listed in Table D-2 and D-3 and Figure D-2 and D-3.

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Table D-2. Ambient storage recovery test for Cr(VI) on binderless quartz fiber filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.2	98.1	97.1
5	97.9	96.1	96.7
10	96.1	96.6	97.2
15	97.3	97.2	95.1

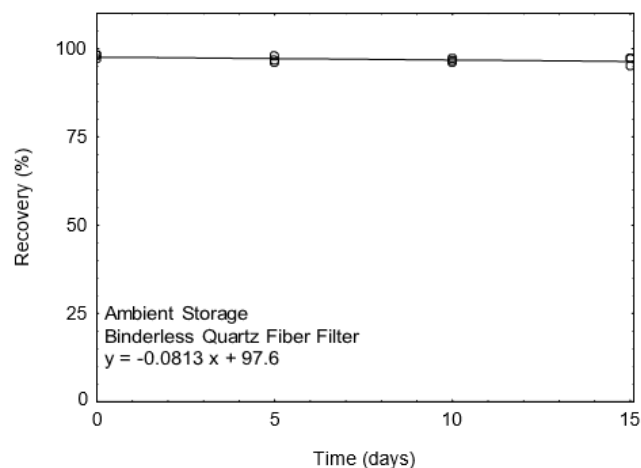


Figure D-2. Ambient storage test for Cr(VI) spiked on binderless quartz fiber filters.

Table D-3. Refrigerated storage recovery test for Cr(VI) on binderless quartz fiber filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	98.2	97.1	98.3
5	97.1	97.9	96.7
10	97.3	98.2	96.1
15	96.1	96.3	95.3

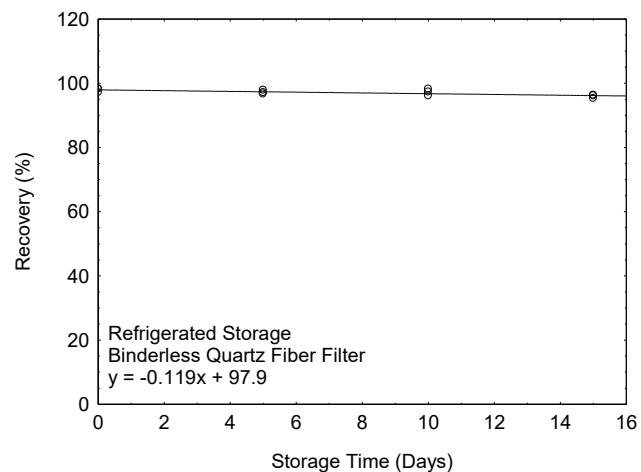


Figure D-3. Refrigerated storage recovery test for Cr(VI) spiked binderless quartz fiber filters.

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4.2 Cr(VI) and H₂SO₄ Spiked on QFF Filters

Pre-existing data from OSHA Method W4001 are presented in this section.

Binderless quartz fiber filters were spiked with a mixture of H₂SO₄ and Cr(VI) in water at concentrations of 50 ng Cr(VI) and 0.5 ng H₂SO₄ and allowed to dry. Twenty-one storage samples were prepared. Three samples were analyzed on the day of preparation. Nine of the filters were stored at reduced temperature (4°C) and the other nine were stored in a closed drawer at ambient temperature (about 22 °C). At 5-day intervals, three samples were selected from each of the two storage sets and analyzed. There was an immediate reaction between the Cr(VI) and the sulfuric acid, causing some of the Cr(VI) to change to Cr(III). Results were corrected for this bias. The results show a recovery of 86.1% on day 15 for samples stored at ambient temperature, and 88.1% for refrigerated samples. Results from these analyses are listed in Table D-4 and 5 and Figure D-4 and 5.

Table D-4. Ambient storage recovery test for Cr(VI) and H₂SO₄ on binderless quartz fiber filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.9	99.3	99.8
5	94.8	93.0	95.1
10	90.1	91.2	89.9
15	85.4	86.8	86.1

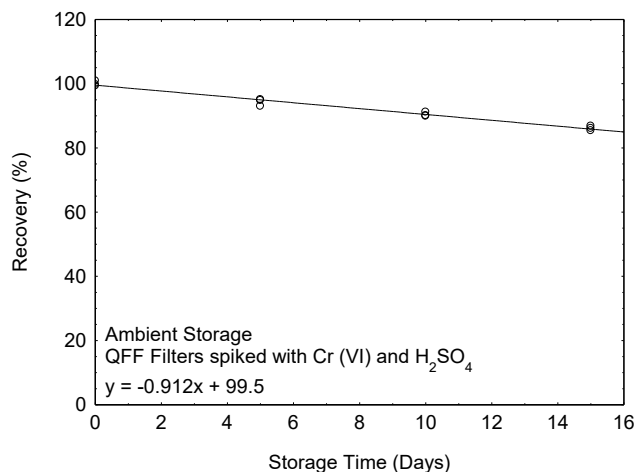


Figure D-4. Ambient storage test for Cr(VI) and H₂SO₄ spiked on binderless quartz fiber filters.

Table D-5. Refrigerated storage recovery test for Cr(VI) and H₂SO₄ on binderless quartz fiber filters.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.9	99.3	99.8
5	96.4	93.2	95.4
10	93.7	92.2	93.4
15	87.9	89.2	87.3

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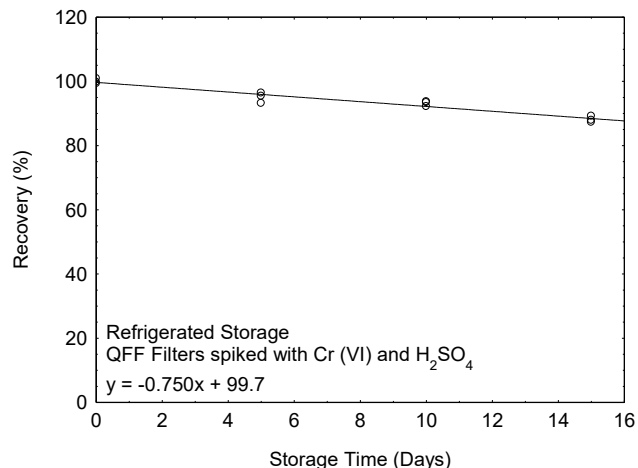


Figure D-5. Refrigerated storage test for Cr(VI) and H₂SO₄ spiked on binderless quartz fiber filters.

4.3 Cr(VI) and H₂SO₄ Spiked on QFF Filter Placed Immediately in 5 mL of Solution Containing 10% Na₂CO₃ with 2% NaHCO₃

Pre-existing data from OSHA Method W4001 are presented in this section.

Because the recovery for the binderless quartz fiber filters spiked with the mixture of Cr(VI) and H₂SO₄ was 86.1% on day 15 for samples stored at ambient temperature, and 88.7% for refrigerated samples, placing the filters into 5 mL of an aqueous solution containing 10% Na₂CO₃ with 2% NaHCO₃ (BE buffer) immediately after spiking was performed. The binderless quartz fiber filter was spiked with 50 ng of Cr(VI) and 0.5 ng H₂SO₄. Twenty-one storage samples were prepared. Three samples were analyzed on the day of preparation. Nine of the filters were stored at reduced temperature (4°C) and the other nine were stored in a closed drawer at ambient temperature (about 22 °C). At 5-day intervals, three samples were selected from each of the two storage sets and analyzed. There was an immediate reaction between the Cr(VI) and the sulfuric acid, causing some of the Cr(VI) to change to Cr(III). Results were corrected for this bias. The recoveries on day 15 were 97.9% for ambient samples and 98.4% for refrigerated samples. Results from these analyses are listed in Table D-6 and 7 and Figure D-6 and 7.

Table D-6. Ambient storage recovery test for Cr(VI) and H₂SO₄ on QFF filters and placed in BE buffer.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.8	99.9	99.3
5	96.9	97.9	98.9
10	98.4	98.5	97.9
15	98.3	97.5	97.6

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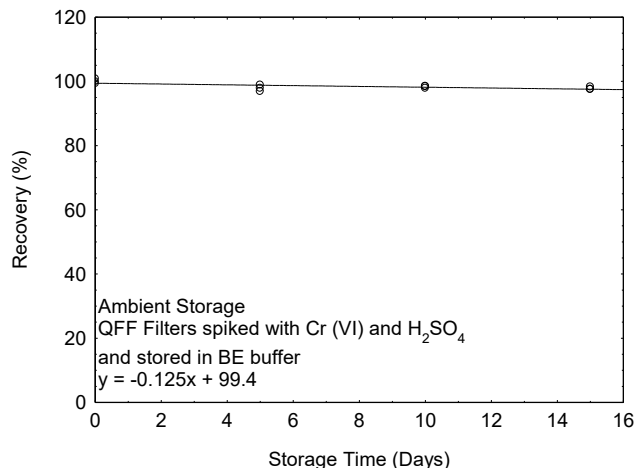


Figure D-6. Ambient storage test for Cr(VI) and H₂SO₄ spiked on binderless quartz fiber filters in BE buffer.

Table D-7. Refrigerated storage recovery test for Cr(VI) and H₂SO₄ on QFF filters and placed in BE buffer.

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)
0	100.8	99.9	99.3
5	98.8	97.9	99.3
10	97.9	98.9	98.5
15	99.1	98.1	97.9

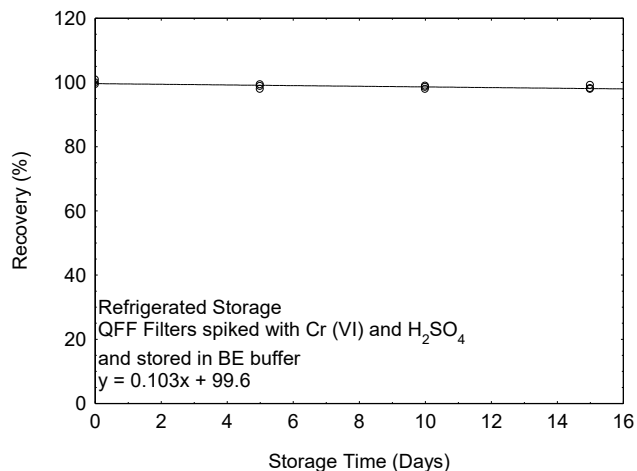


Figure D-7. Refrigerated storage test for Cr(VI) and H₂SO₄ spiked on QFF filters stored in BE buffer.

5 Method Precision and Bias

No pre-existing data from OSHA Method W4001 in this section.

6 Analytical Recovery and Stability of Prepared Samples

Analytical recovery is affected by the extraction buffer, the sampling medium, extraction temperature and the technique used to extract samples. The data presented demonstrate validity for the extraction buffer, sampling medium, extraction temperature and extraction technique described in this method. If other combinations of these are to be used, testing

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must be completed to satisfy the requirements found in current OSHA sampling and analysis method validation guidelines. Acceptable testing results must be documented.

6.1 Pre-existing Data Using Hot Plate/Water Bath and Erlenmeyer flask

Pre-existing data from OSHA Method W4001 are presented in this section.

The reliable quantitation limit (RQL) is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters obtained for the calculation of the detection limit of the overall procedure, providing 75% to 125% of the analyte is recovered. The RQL for QFF filter is 2.23 ng/sample. Recovery at this concentration is 94.6%.

The extraction efficiencies of Cr(VI) were determined by liquid-spiking binderless quartz fiber filters with Cr(VI) at concentrations ranging from the RQL (0.06) to 10 times the target concentration. These samples were stored overnight at ambient temperature and then analyzed. The filters were either extracted and digested with the first, main extraction solution, BE, or with the second, spray paint only, SPE solution. The mean extraction efficiency over the working range of the RQL to 10 times the target concentration is 97.3% for samples extracted with the first (main) extraction solvent (BE) and 96.2% for samples extracted with the second extraction solvent for paint samples (SPE). Results from these analyses are listed in Table D-8 and D-9.

Table D-8. Extraction efficiency of Cr(VI) from binderless quartz fiber filters extracted with BE.

×target concentration	ng/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	mean (%)
RQL	3	96.3	95.4	95.9	96.3	96.0
0.1	5	97.2	96.9	96.8	97.5	97.1
1.0	50	98.4	97.2	98.2	97.9	97.9
10.0	500	97.9	98.3	98.1	98.1	98.1

Table D-9. Extraction efficiency of Cr(VI) from binderless quartz fiber filters extracted with SPE.

×target concentration	ng/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	mean (%)
RQL	3	96.1	95.3	95.7	96.2	95.8
0.1	5	97.2	96.7	96.8	95.8	96.6
1.0	50	97.4	96.3	95.9	95.0	96.2
10.0	500	96.3	95.2	97.2	96.5	96.3

6.2 Data using Hot Block and Scintillation Vial

The analytical recovery (R_A) was determined by liquid-spiking six replicate QFF filters across a range of analyte mass values that would be obtained from sampling at 0.1 to 2 times of T_M . Samples were analyzed following overnight drying at ambient temperature (22 °C) in the hood. The R_A was 102.7%. The percent coefficient of variation of the thirty spiked samples (CV_{R_A}) was 4.9%. The data are shown in Table D-10.

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Table D-10. Analytical recovery results of binderless quartz fiber filters.

× target concentration	µg/sample	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)	mean (%)
0.1	0.496	97.9	99.5	109.9	108.2	100.6	105.4	103.6
0.5	2.480	97.9	99.7	99.9	100.8	102.2	98.9	99.9
1	4.960	88.4	98.2	101.3	103.6	101.8	101.9	99.2
1.5	7.440	99.2	106.4	111.0	97.4	108.3	107.6	105.0
2.0	9.920	101.3	104.9	107.3	109.1	110.6	103.1	106.1

The stability of sample extracts was determined by liquid-spiking six replicate QFF filters with a mass of analyte that would be obtained from sampling at T_M (calculated to be 5.10 µg). Samples were analyzed following overnight drying at ambient temperature in the hood. Each septum was punctured 1 time for each injection and punctured septa retained in this validation.

All six vials were re-analyzed at 24, 48, and 72 hours after the initial analyses, with all vials remaining in the autosampler tray kept at room temperature (22 °C). Freshly prepared standards (in DBE buffer without media) were used for each analysis event. The results of these analyses are provided in Table D-11. The linear regression equation determined from these results was $y = 0.000133x + 0.990$. The change in recovery (Δ_{ps}) of hexavalent chromium calculated from the regression line generated for the 3-day storage test was 0.04%.

Table D-11. Stability of prepared sample data of Cr(VI) on binderless quartz fiber filters for 3 days (T_M).

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	102.1	104.7	93.7	93.3	101.6	101.0
1	101.5	103.7	92.7	92.2	100.0	100.1
2	102.3	104.3	93.3	92.9	101.4	100.6
3	101.9	104.1	93.8	92.9	101.3	100.6

All six vials were re-analyzed at 1, 3, and 5 weeks after the initial analyses together with freshly prepared standards for each analysis event, with all vials standing at room temperature. Freshly prepared standards were used for each analysis event. The results of these analyses are provided in Table D-12. The linear regression equation determined from these results was $y = 0.00817x + 1.01$. The change in recovery (Δ_{ps}) of hexavalent chromium calculated from the regression line generated for the 5-week storage test was 4.1%.

Table D-12. Stability of prepared sample data of Cr(VI) on binderless quartz fiber filters for 5 weeks (T_M).

time (week)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	102.1	104.7	93.7	93.3	101.6	101.0
1	102.8	105.6	102.9	104.3	105.4	104.2
3	103.5	104.6	102.4	104.2	105.8	103.7
5	104.4	105.4	103.3	104.6	105.7	104.8

The half target mass level ($\frac{1}{2} T_M$) stability of sample extracts was examined by retaining the solvent extract of six spiked QFF media at mass loading level of $\frac{1}{2} T_M$ (2.49 µg). Each septum was punctured 1 time for each injection and punctured septa retained in this validation.

All six vials were re-analyzed at 24, 48, and 72 hours after the initial analyses, with all vials remaining in the autosampler tray and kept at room temperature. Freshly prepared standards (in DBE buffer without media) were used for each analysis event. The linear regression equation determined from these results was $y = -0.00192x + 1.09$. The change in recovery (Δ_{ps}) of hexavalent chromium calculated from the regression line generated for the 3-day storage test was 0.58%. Results from the analysis are listed in Table D-13.

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Table D-13. Stability of prepared sample data of Cr(VI) on binderless quartz fiber filters for 3 days ($\frac{1}{2} T_M$).

time (day)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	105.1	107.5	105.8	114.4	111.1	109.1
1	104.9	105.9	104.0	114.0	109.4	108.2
2	105.1	107.2	104.6	114.0	109.7	109.0
3	105.6	106.2	103.9	113.8	109.6	109.0

All six vials were re-analyzed at 2, 3, and 5 weeks after the initial analyses together with freshly prepared standards for each analysis event, with all vials standing at room temperature. Freshly prepared standards used for each analysis event. The results of these analyses are provided in Table D-14. The linear regression equation determined from these results was $y = 0.000256x + 1.09$. The change in recovery (Δ_{ps}) of hexavalent chromium calculated from the regression line generated for the 5-week storage test was 0.13%.

Table D-14. Stability of prepared sample data of Cr(VI) on binderless quartz fiber filter for 5 weeks ($\frac{1}{2} T_M$).

time (week)	sample 1 (%)	sample 2 (%)	sample 3 (%)	sample 4 (%)	sample 5 (%)	sample 6 (%)
0	105.1	107.5	105.8	114.4	111.1	109.1
2	106.1	105.5	109.3	112.4	110.7	106.4
3	105.0	106.0	109.0	112.7	110.5	106.7
5	105.7	106.9	109.5	113.4	110.3	106.5

7 Sampler Removal Efficiency

Pre-existing data from OSHA Method W4001 are presented in this section.

Removal efficiency of binderless quartz fiber filters was determined by placing the Cr(VI) on a PTFE sheet. This type of surface approaches the smooth and non-porous characteristics of an ideal surface. The variety of surfaces found in workplaces, including skin, will likely be less than ideal. The media will have a lower removal efficiency on less ideal surfaces. The amount of analyte found on the filter after sampling will indicate at least the amount present on the surface that was sampled. Six surfaces were spiked at the target concentration of Cr(VI), $0.050 \mu\text{g}/100 \text{ cm}^2$. Samples were collected from each surface using the technique described in OSHA Method ID-215, Section 2.3 and analyzed. The results are shown in Table D-15. The mean removal efficiency of the six samples was 97.7% for QFF filters. The removal efficiency was calculated as amount of analyte recovered divided by amount of analyte spiked on the surface.

Table D-15. Sampler removal efficiency data for Cr(VI) from PTFE using binderless quartz fiber filters.

theoretical ($\mu\text{g}/\text{surface}$)	recovered ($\mu\text{g}/\text{sample}$)	recovery (%)
0.05	0.0489	97.8
0.05	0.0493	98.6
0.05	0.0492	98.4
0.05	0.0483	96.6
0.05	0.0484	96.8
0.05	0.0489	97.8

8 Effect of Humidity and Low Sampling Concentration

No pre-existing data from OSHA Method W4001 in this section.

9 Sampling and Analytical Interferences

Acid is a negative interference in chrome plating operations. Samples should be taken on NaOHqz filters in chrome plating operations. The storage results indicate that the samples taken in chrome plating operations with the quartz

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fiber filter should be stabilized in a 20 mL scintillation vial containing 5-mL of an aqueous solution containing 10% sodium carbonate (Na_2CO_3)/ 2% sodium bicarbonate (NaHCO_3) immediately after receiving or be analyzed within 10 days of sampling (Appendix D, Section 4.2). Confirm the presence of hexavalent chromium as described in OSHA Method ID-215, Section 3.8.

Suspected interferences information should be reported to the laboratory with submitted samples. The interference studies were performed in OSHA Method ID-215 version 2 and should be consulted for specific information about the metal species of interest. The major positive interference is Cr(III), which can be oxidized to Cr(VI). The major negative interference is Fe(II), which can reduce the Cr(VI) to Cr(III). Other reducing metal species can also change Cr(VI) to Cr(III).

10 Analytical Reproducibility

Pre-existing data from OSHA Method W4001 are presented in this section.

Six PTFE surfaces were spiked at the target level of 50 ng Cr(VI). A chemist, other than the one developing the method, conducted sampling on the PTFE surfaces as described in OSHA Method ID-215, Section 2 using binderless quartz fiber filters. The test was repeated with a second chemist performing the sampling. The samples were analyzed. The first chemist was able to achieve a removal efficiency of 95.9% (Table D-16) and the second chemist 96.0% (Table D-17).

Table D-16. Sampling reproducibility 1st chemist samples for Cr(VI) from PTFE using binderless quartz fiber filter.

theoretical (ng/surface)	recovered (ng/sample)	recovery (%)
50	47.2	94.4
50	48.9	97.8
50	47.1	94.2
50	47.5	95.0
50	48.8	97.6
50	48.4	96.8

Table D-17. Sampling reproducibility 2nd chemist samples for Cr(VI) from PTFE using binderless quartz fiber filters.

theoretical (ng/surface)	recovered (ng/sample)	recovery (%)
50	47.5	95.0
50	48.6	97.2
50	48.3	96.6
50	48.8	97.6
50	47.1	94.2
50	47.7	95.4

Six samples were prepared by spiking binderless quartz fiber filters in the same manner that was used in the preparation for the storage study. The samples were submitted to the OTC for analysis. The samples were analyzed after being stored for 5 days at 23 °C. Sample results were corrected for extraction efficiency. The average recovery was 96.5% (Table D-18).

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Table D-18. Analytical reproducibility data for Cr(VI) using binderless quartz fiber filters.

theoretical (ng/surface)	recovered (ng/sample)	recovery (%)
50	47.3	94.6
50	48.4	96.8
50	47.9	95.8
50	48.2	96.4
50	48.8	97.6
50	48.9	97.8

11 Estimation of Uncertainty

While systematic biases such as analyte storage loss are examined, and limits are placed on these, an estimation of uncertainty that encompasses both potential random and systematic error was not completed.

References

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3. Lawrence, R. Evaluation Guidelines for Surface Sampling Methods; OSHA Salt Lake Technical Center, U.S. Department of Labor: Salt Lake City, UT, 2001, unpublished.