Standard Operating Procedure

Analysis of Ambient Air Particulate Matter for Hexavalent Chromium by Ion Chromatography

DEQ17-LAB-0006-SOP
Version 2.0

January 2025

State of Oregon
DEQ Department of Environmental Quality

This document was prepared by
Oregon Department of Environmental Quality
Laboratory and Environmental Assessment Division
7202 NE Evergreen Parkway, Suite 150
Hillsboro, OR 97124
Contact: Sara Krepps
Phone: 503-693-5700
www.oregon.gov/deq

DEQ17-LAB-0006-SOP
Version 2.0
Last updated: January 17, 2025

Printed copies of this document are considered uncontrolled.

State of Oregon
DEQ Department of Environmental Quality

Translation or other formats

Español | 한국어 | **繁體中文** | **Русский** | **Tiếng Việt** | العربية

800-452-4011 | TTY: 711 | deqinfo@deq.oregon.gov

Non-discrimination statement

DEQ does not discriminate on the basis of race, color, national origin, disability, age or sex in administration of its programs or activities. Visit DEQ's Civil Rights and Environmental Justice page.

Approvals

Prepared by:

_____ Date: _____
Joe Hyland, Chemist

Reviewed by:

_____ Date: _____
Zach Mandera, Inorganic Section Manager

Approved by:

_____ Date: _____
Sara Krepps, Laboratory Quality Assurance Officer

Signed Copy on File

Table of Contents

1. Scope and Application 6
1.1. Applicable Matrices 6
1.2. Detection Limits 6
2. Summary 6
3. Personnel-Qualifications/Responsibilities 6
4. Interferences 7
5. Safety 7
6. Equipment and Supplies 7
7. Reagents 8
8. Standards 8
9. Sample Collection, Preservation, Shipment, and Storage 9
10. Calibration and Standardization 9
11. Quality Control 10
11.1. Data Assessment and QC Acceptance Criteria 10
11.2. Corrective Actions and Contingencies for Out-of-Control Data 11
12. Procedure 13
12.1. Preparation of Filters 13
12.2. Filter Extraction 13
12.3. Sample Analysis 14
13. Calculations 15
14. Records Management 15
14.1. Log books 16
14.2. Record Retention 16
15. Method Performance 17
16. Maintenance 17
17. Pollution Prevention 18
18. Waste Management 18
19. Definitions 18
20. Deviations from Referenced Methods 19
21. References 19
22. Revision History 19

List of Tables

Table 1: List of target analytes and Limits of Quantitation (LOQ's)....6
Table 2: Standards....9
Table 3. Quality Control Elements and Acceptance Criteria....10
Table 4: List of Corrective Actions for Out-of-Control QC Data....12

List of Appendices

Appendix A: Attachment A Job Safety Assessment (JSA)....21
Appendix B: Method Performance....23
Appendix C: Analytical Batch Review Form....24

1. Scope and Application

This procedure provides step-by-step instructions for analyzing Hexavalent Chromium in ambient air particulate matter collected on Sodium Bicarbonate impregnated ashless cellulose filters.

1.1. Applicable Matrices

This method applies to the analysis of ambient air particulate matter collected on sodium bicarbonate impregnated ashless cellulose filters.

1.2. Detection Limits

Determine the method detection limit (MDL) every year according to the procedure in DEQ18-LAB-0053-SOP Limits of Detection (LOD) and Quantitation (LOQ). The LOQ is also commonly known as the Method Reporting Limit (MRL). Spike a standard onto at least seven prepared filters at a concentration around three times the estimated detection limit. Extract these filters according to the method outlined in section 12. The Limit of Quantitation (LOQ) is generally 3 to 5 times the LOD. The lowest point in the calibration curve (0.1 ng/L) is equal to the LOQ.

Table 1: List of target analytes and Limits of Quantitation (LOQ's)

Analyte	Instrument Range, ng/mL Digest-aqueous	LOD, ng/mL Digest-aqueous	LOD, ng/ m^3 Reporting units	LOQ, ng/mL Digest-aqueous	LOQ, ng/ m^3 Reporting units
Hexavalent Chromium	0.1-2.0	0.04	0.019	0.1	0.046

2. Summary

This SOP covers the determination of Hexavalent Chromium in ambient air particulate matter collected on bicarbonate-impregnated ashless cellulose filters. Extract the filters in 20 mM Sodium Bicarbonate in deionized (DI) water via sonication for 1 hour. Analyze the extract by ion chromatography using a system comprised of a guard column, an analytical column, a post-column derivatization module, and a UV/VIS detector. In the analysis procedure, Hexavalent Chromium exists as chromate due to the near neutral pH of the eluent. After separation through the column, the Hexavalent Chromium forms a complex with the 1,5-diphenylcarbohydrazide (DPC) which can be detected at 530 nm.

3. Personnel-Qualifications/Responsibilities

The analyst should be trained by a chemist who has previously demonstrated their proficiency at performing the method. The analyst should either be a Chemist 2 or work directly with a Chemist 2 or higher.

As required by the DEQ Laboratory Quality Manual (LQM), the analyst will conduct an Initial Demonstration of Capability (IDOC) prior to reporting data. The analyst should conduct an annual Demonstration of Capability

(DOC). DOCs and IDOCs are conducted according to DEQ20-LAB-0011-SOP Method Validation, Verification & Personnel DOC.

4. Interferences

Sodium Bicarbonate used as the stabilizing medium on the Hexavalent Chromium on filters may cause interferences with the analysis. Higher concentrations of the Sodium Bicarbonate impregnating solution may cause flow restrictions during the ambient air sampling. The use of an impregnated filter of smaller pore size may cause flow restrictions during sampling.

5. Safety

Analysts working in the LEAD facility must review the laboratory's Chemical Hygiene Plan (DEQ04-LAB-0006-SFTY) and the Emergency Action Plan (DEQ04-LAB-0050-SFTY).

Refer to the Job Safety Assessment (JSA) for this procedure and conduct analysis in accordance with the safety precautions specified in the Appendix.

Health and Safety has an online database for SDS sheets. Please refer to the Workplace Safety Home page on QNet for accessing this online database.

6. Equipment and Supplies

- A freezer capable of maintaining a temperature of less than -15°C
- Analytical balance
- Ultrasonicator
- Ion chromatograph (Currently using Dionex Integrion)
- Autosampler (Currently using Dionex AS-DV)
- Post-Column Derivatization Module
- UV/VIS absorbance detector
- Dionex AG7 guard column or equivalent
- Dionex AS7 separator column or equivalent
- Data system, including computer, Windows 7, and Chromeleon software
- Eluent and Post Column Derivatization Solution Reservoirs
- 47mm ashless cellulose filters
- Teflon-coated or plastic forceps
- Disposable nitrile gloves
- Clean container for sodium bicarbonate impregnation of filters
- Laminar flow hood

- Volumetric Flasks and Pipettes in a variety of sizes

7. Reagents

0.12 M Sodium Bicarbonate Impregnating Solution
In a 1L volumetric flask, dissolve 10.0 g of Sodium Bicarbonate in ~500 mL DI water. Dilute to 1L with DI water. This reagent should be made fresh before use.

20 mM Sodium Bicarbonate Solution
In a 1 L volumetric flask, dissolve 1.68 g of Sodium Bicarbonate in ~500 mL DI water. Dilute to 1 L with DI water. This reagent should be made fresh before use.

Eluent
Eluent solution is 250 mM Ammonium sulfate and 100 mM Ammonium hydroxide in DI water. This Eluent is good for three months. Prepare as follows:

> In a 2 L volumetric flask, dissolve 66.0 g of Ammonium Sulfate in ~1 L DI water and add 13mL of Ammonium Hydroxide. Dilute to 2 L with DI water. Degas eluent before use. Because ammonia and some related chemicals are target analytes for other analyses performed in the lab there is a potential for contamination. Steps to minimize this contamination should be taken and can include: always keeping eluent capped when it is not in a hood, using a snorkel hood above the eluent tank and above the waste container, rinsing or washing glassware used in the preparation of eluent as far away as practical from where prep or analysis of ammonia samples is done, and wiping reagent bottles after they are used to make eluent.

Post-Column Derivatizing Reagent (PCDR)
In a 1 L volumetric flask, dissolve 0.5 g of 1,5-Diphenylcarbazide in 100 mL of HPLC-grade Methanol. The 1,5-Diphenylcarbazide may take 30 minutes or more to fully dissolve. In a separate container, add 25 mL of 98% sulfuric acid to about 500 mL of DI water and allow time for cooling. This can happen at the same time as the 1,5-Diphenylcarbazide is dissolving. Once the acid solution has cooled and the 1,5-Diphenylcarbazide has dissolved, pour the acid solution into the flask and then fill the flask to the 1 L mark with DI water. Invert and swirl several times to mix. This reagent is stable for a week. To minimize waste, prepare in 1 L quantities as needed. When not in use, store PCDR in refrigerator in opaque container. Degas PCDR before use.

8. Standards

The working analytical range for this method is 0.1 to 2.0 ng/mL. Dilute samples with concentrations above the highest calibration point into the calibration range with 20mM Sodium Bicarbonate solution.

Note: The stocks and dilution schemes listed below are examples of the method used to make working standards. These may be changed as appropriate to achieve similar working standard concentrations.

Prepare all standards using class A volumetric flasks.

Primary Stock 1000 ug/mL Hexavalent Chromium Purchase Certified Reference Material from, for example, Ultra Scientific or Accustandard. Dilute appropriately with 20mM Sodium Bicarbonate solution to create working standards.

Secondary Stock 1000 ug/mL Hexavalent Chromium Purchase Certified Reference Material from a supplier other than the supplier of the primary stock. Dilute appropriately with 20mM Sodium Bicarbonate solution to create the ICV.

Calibration (Working) standards (0.1 to 2.0 ng/mL range): These are made fresh daily.

Table 2: Standards

Standard ID	Volume Source Std (mL)	Source Std	Final volume (ml)	Final conc. (ng/mL)
Dilution Stock	1	Primary Stock	100	10,000
Working Stock	1	Dilution Stock	100	100
STD 1	0.1	Working Stock	100	0.1
STD 2	0.2	Working Stock	100	0.2
STD 3	0.5	Working Stock	100	0.5
STD 4	1.0	Working Stock	100	1.0
STD 5	2.0	Working Stock	100	2.0

9. Sample Collection, Preservation, Shipment, and Storage

Use clean Teflon-coated or plastic tweezers and disposable Nitrile gloves when handling filters.

Store unused, Sodium Bicarbonate impregnated filters in the freezer until needed for sampling or for use in the laboratory as spikes or blanks. The filters are frozen to prevent the Sodium Bicarbonate from reacting with possible interfering substances present in the air.

Hexavalent chromium filters that are loaded in filter holders and ready to be transported to the sampling site are stored at ≤0º C. After sampling, loaded filters are stored at ≤0º C until analysis. See DEQ01-LAB-004-QAPP for full details on filter handling in the field.

The maximum holding time from sampling to extraction is 21 days. Analyze as soon as practical following extraction. Analysis must occur within 24 hours of extraction. If a sample must be extracted and/or analyzed beyond these limits, qualify the result appropriately.

10. Calibration and Standardization

Run a five-point calibration each day prior to analysis.

Use all standards within one day of preparation. Perform the calibration under the same instrument conditions used for analysis of the samples. Designate the standard results in the sequence as “standard” type. The software then utilizes the results to calculate standard curve and correlation coefficient. The correlation

coefficient must be ≥0.995. The fit of all standards (the calculated value compared to the true value) must be within ±10%.

11. Quality Control

Test Method Validation: Refer to the Laboratory Quality Manual DEQ91-LAB-0006-LQM for Test Method Validation requirements. Conduct a Test Method Validation after significant method modifications or instrument repairs.

11.1. Data Assessment and QC Acceptance Criteria

Table 3. Quality Control Elements and Acceptance Criteria

QC Element	Frequency	Acceptance Criteria	Comments
Calibration Curve "r" value	Initial calibration	≥0.995	A linear curve is used and is not forced through origin.
Initial Demonstration of Capability (IDOC) and Continuing Demonstration of Capability.	At end of method development and prior to reporting data, and thereafter yearly.		DOCs and IDOCs are conducted according to DEQ20-LAB-0011-SOP Method Validation, Verification & Personnel DOC.
Initial Calibration Verification (ICV)	Immediately following Initial Calibration	Recovery 90%-110%	0.75ng/mL standard made from secondary stock
Initial Calibration Blank (ICB)	Immediately following Initial Calibration	< LOD	
Low Calibration Value (LCV) Check	Immediately following Initial Calibration	Recovery 50%-150%	0.1 ng/mL standard
Method Blank (BLK)	At least once per analytical batch	< LOD	Prepared filter, extracted with samples
Laboratory Control Sample (LCS)	At least once per analytical batch	Recovery 90%-110%	1.0 ng/mL second source - spiked onto prepared filter, extracted with samples
Continuous Calibration Verification standard (CCV)	1 after every 10 samples and at the end of each batch	Recovery 90%-110%	0.5ng/mL standard
Continuous Calibration Blank (CCB)	1 after every 10 samples and at the end of each batch	< LOD	

QC Element	Frequency	Acceptance Criteria	Comments
Replicate Samples	At least once per analytical batch	Within 10% RPD for all values > 5x LOD otherwise within 1x LOQ	Use field primary if available
Field Primary and Field Duplicate (FP/FD)	Per Air Monitoring staff procedures	Within 20% RPD for all values > 5x LOD otherwise within 2x LOQ	
Equipment Blank (EB)Surrogate Recovery	Per Air Monitoring staff procedures	< LOD	
MRL verification sample (MRLv)	Minimally one per quarter in any quarter in which samples are analyzed.	Recovery 50%-150%	Follow procedure in DEQ18-LAB-0053-SOP, Method Detection and Reporting Limits (MDL & MRL)
MDL verification sample (MDLv)	Minimally two per quarter, in separate batches prepped on separate days and analyzed on separate days in any quarter in which samples are analyzed.	Must meet qualitative criteria	Follow procedure in DEQ18-LAB-0053-SOP, Method Detection and Reporting Limits (MDL & MRL)

11.2. Corrective Actions and Contingencies for Out-of-Control Data

If the samples cannot be reanalyzed or the analysis otherwise brought under-control:

a) Identify if there is any guidance in the relevant QAPP or SAP and follow that guidance.

b) Contact the lab project manager and verify the importance of the data and to see if the analyte can be reported as estimated.

- If so, report the affected analyte(s) with a data qualifier that assigns a DQL of B.
- If not, void the analysis and add data qualifier(s) to assign a DQL of C or D to the result.

c) Determine with the lab project manager if an alternate sample can be used for their evaluation purposes.

d) If it seems appropriate in your best professional judgment, speak with the lab sample tracker or sample collector about obtaining an alternate sample that may provide the required information.

e) If the lab project manager cannot be reached, consult with the section manager or QA officer to determine if the data should be reported as an estimate (DQL B), rejected (DQL C) or voided (DQL C or D).

f) In every case, the problem(s) and attempted corrections should be noted with the analytical batch data, in the LIMS system for final reporting, and in the laboratory notebook if instrumental problems have occurred.

Table 4: List of Corrective Actions for Out-of-Control QC Data

Quality Control Element	Corrective Action
Initial 5-point calibration standards	Repeat analysis of calibration standards. If calibration is still out-of-control, prepare new calibration standards and reanalyze.
Initial Calibration Verification (ICV)	Reanalyze ICV. If ICV is still out-of-control, recalibrate.
Initial Calibration Blank (or ICB)	Reanalyze ICB. If ICB is still out-of-control, recalibrate.
Low Calibration Value (LCV) Check	Reanalyze LCV. If LCV is still out-of-control, recalibrate.
Method Blank (BLK)	Reanalyze BLK. If BLK is still out-of-control, flag data of all associated samples. Qualify the BLK with QC::B.
Laboratory Control Standard (LCS)	Reanalyze LCS. If LCS is still out-of-control, flag data of all associated samples. Qualify the LCS with QC::B.
Continuing Calibration Verification (CCV)	Reanalyze CCV. If CCV is still out of control, flag data bracketed by unacceptable CCV. Qualify the unacceptable CCV with QC::B.
Continuing Calibration Blank (CCB)	Reanalyze CCB. If CCB is still out of control, flag data bracketed by unacceptable CCB. Qualify the unacceptable CCB with QC::B.
Field Primary and Field Duplicate (FP/FD) and Equipment Blank (EB)	Inspect sample records and inform field staff of findings (whether or not any discrepancies are discovered). Flag sample(s) and create either an FP/FD or FB report as appropriate. These are printed from Element in the data review screen using either. Rev_AirReplicateVDup.rpt or Rev_FieldBLK.rpt.

12. Procedure

12.1. Preparation of Filters

Before a new lot of filters can be used, test at least 2 filters per box of unprepared filters for contamination by extracting and analyzing them. If contamination is detected, discard the whole lot.

1. Soak the filters in the 0.12 M Sodium Bicarbonate impregnating solution overnight (not more than 18 hours).
2. Dry the filters completely on a screen rack in the laminar flow hood.
3. Before a new set of prepared filters can be used, test three or more (about 5-10%) of the newly prepared filters by extracting and analyzing them. If contamination is detected, discard all the filters prepared in that set.
4. Load dry filters directly in to filter holders, or store in dated Petri dishes in a freezer until needed.
5. When a filter is loaded into a filter holder, assign it a unique filter ID. Label both the filter holder and a 1 liter plastic jar with this unique filter ID and store the filter holder in the plastic jar in a freezer. It is now ready to be deployed in the field. There needs to be a traceable record of each filter placed in the freezer ready to be deployed in the field that includes the unique filter ID and the date it was placed in the freezer. The current practice is to record this information on a log on a clipboard attached to the front of the Cr6+ freezer in sample control, and when this clipboard is full, some of the oldest sheets are transferred to a binder (the maintenance of the logsheets and binder are the responsibility of AQM staff). Additional filter prep information is also maintained in a spreadsheet which can be viewed by following this link: \\deqlead-lims\Inorganic_InstData\107692-Integrion_IC\Filter Tracking.xlsx

12.2. Filter Extraction

Due to the oxidation/reduction and conversion problems of trivalent chromium and hexavalent chromium, the extraction should be performed immediately prior to analysis. It is important that the IC be equilibrated, calibrated and ready for analysis. One unused filter each will be prepared and used for a method blank and an LCS with every batch.

1. Create a batch and sequence in LIMS and print batch sheet and sequence sheet.
    a. Add up to 20 samples to be digested to the batch. This will cause Element to automatically add in a blank and a blank spike.
    b. Right click on the blank and blank spike and under special info change CR6+ VOLUME (M3) to 21.6
    c. Add the appropriate spike ID and volume to the blank spike
    d. Add one duplicate per preparation batch. This is actually a replicate and not a normal laboratory duplicate. This is indicated by right clicking on the DUP and its source sample and, under

special info, changing the REPLICATE field to “Replicate”. A comment that says “Replicate” should also be added to the DUP.

2. After verifying that the unique filter ID on the filter holder, 1L bottle containing filter holder, and bench sheet all match, remove the exposed filter from the filter holder using tweezers and disposable Nitrile gloves. Fold the filter, place it in a labeled 14 mL polystyrene test tube and cap tube. For the blank and blank spike, an unexposed filter is used. Add the appropriate amount of spike solution to the tube designated as the blank spike.
3. Add 10 mL of the 20 mM Sodium Bicarbonate in DI water solution to each test tube, cap the tube, then shake it vigorously. Place the tubes in a test tube rack and sonicate for 1 hour.
4. Record ID numbers for all support equipment and reagents used on the bench sheet. Also record the start and finish times for sonication. Make sure the sonication start time matches the prepared time in Element and that all reagents match those listed in Element.
5. After sonication, shake the tubes vigorously again and pour about 5 mL of the sample extract into a labeled, 5 mL disposable autosampler vial. For the replicate analysis, prepare a second autosampler vial from the same test tube.

12.3. Sample Analysis

Prior to the start of the run, record the pressure for both the main pump and the auxiliary pump in the maintenance log book. The analysis time for each injection is approximately 12 minutes.

1. Place the eluent and PCDR lines in DI water and first turn the main pump and then the auxiliary pump. This is done under the AXP_PUMP and PUMP_ECD tabs of the Chromeleon software. **Do not run the auxiliary pump when the main pump is off.**
2. While pumping DI water through the system, turn on the lamps. This is done under the UV tab of the Chromeleon software. It is important that the pumps are on when the lamps are turned on. **Running the lamps for an extended period of time while the pumps are not on can cause overheating and damage the lamps.**
3. Open the most recent analytical run and re-save it with the name of the new sequence.
4. From the instrument computer, open the sequence in Element and export it to the desktop.
5. Copy and paste the calibration and sample IDs into the instrument sequence. It can be useful to run at least one conditioning blank before the calibration to verify that the instrument is running well and that there is no contamination.
6. Once the lamps (detection wavelength is 530 nm) have been warming up for at least two hours, turn the pumps off and switch the eluent and PCDR lines back from DI water to eluent and PCDR and immediately turn the pumps back on. **Pumps should not be off more than about 10 minutes to avoid damage to the lamps.**
7. Make sure that the eluent flow rate is 1.5 mL/minute and the post-column Reagent flow rate is 0.5 mL/min, and then allow the instrument to reach a steady base line. This usually takes about an hour.

8. Click the start button. The instrument may ask you to verify the reagents. This is done from the "Inventory" tab, and you will have to click start again afterwards.
9. After the run completes, place the eluent and PCDR lines in DI water and first turn on the main pump and then the auxiliary pump. **Do not run the auxiliary pump when the main pump is off.** Allow the system to flush for at least an hour and then turn off the pumps starting with the auxiliary pump. Because an analytical run is often set up and allowed to run over night, this flush often happens the next day.

13. Calculations

The Chromeleon software plots the calibration and quantifies the analyte in initial units of ng/mL using the calibration curve. Element converts the initial result into a final result in units of ng/m3 (see equation below). Element also calculates precision (RPD) and accuracy (% recovery) values; RPD and % recovery equations can be found in section 18.3 of the LEAD Quality Manual (DEQ91-LAB-0006-LQM).

Custom equation for calculating final results:

{IRESULT} * {DILN} * ({FINI} / {INI}) / {SXINFO8}

Where:

- IRESULT is initial result in ng/mL
- DILN is dilution factor at instrument (default = 1)
- FINI is preparation final amount in mL (default = 10)
- INI is preparation initial amount in filters (default = 1)
- SXINFO8 is air volume sampled onto filter in m3 (default = 21.6)

14. Records Management

For detailed instructions on how to generate and review data electronically, please refer to DEQ24-LAB-0007-SOP, Generating Instrument Data Packages Electronically.

Instrument data are generated in an individual subfolder labeled by Sequence ID in the **!In_Process** folder under the instrument folder on the instrument network drive: **\\deqlead-lims\Inorganic_InstData\107692-Integrion_IC.**

The sequence folder should include the following documentation:

1. Completed Analytical (Peer Review) Checklist and any communications needed to document anomalies (such as emails)

   01_SequenceID_Checklist.pdf

01_SequenceID_Email.pdf

2. Element® EB and FP/FD Crystal reports for any FAILING field QC *(not needed in data pack if no failing field QC)*

   02_SequenceID_EB.pdf

   02_SequenceID_FPFD.pdf

3. Element® benchsheet(s) *(may be omitted if not used to record original observations) (will be included for NH3N distillation and NITS treated batches)*

   05_SequenceID_BatchID.pdf

4. Raw Data (including all calibration information)

   08_SequenceID_Data.pdf

The analyst generates the preceding documentation and passes it on to a second chemist for peer review by cutting the batch folder from the **!In_Process** folder and pasting it to the **!Needs_Review** folder (there should be no copy of the folder remaining in the **!In_Process** folder after this step). If the sequence contains only QC samples (IDOC, LOD study, etc.), the analyst should differentiate the sequence folder by adding a brief descriptor after the sequence ID (e.g. SequenceID_LOD) and should email the peer reviewer to notify them that the QC data is ready for review since sequences with only QC data will not show up in Element® queries. For sequences containing samples, the analyst and reviewer will determine what mode of communication works best for them (Element® status update only, email, or a combination).

The peer reviewer combines the individual files into a single pdf file in the order listed above using **SequenceID.pdf** (e.g., **S24E001.pdf**) as the filename. This file is saved to the **!Peer_Reviewed** folder for the instrument. Once the combined file is generated, checked for completeness, and saved to the **!Peer_Reviewed** folder, the peer reviewer shall delete the sequence folder from the **!Needs_Review** folder on the instrument server. The combined file is the official analytical raw data record. The reviewer attaches this file to the sequence in Element using the Sequence PDF button on the sequence screen. The file located in the **!Peer_Reviewed** folder is kept for a minimum of 24 hours to allow server backup to occur. On a periodic basis, the peer reviewer shall delete old files.

After peer review, the reviewing chemist submits a single copy of the Analytical (Peer Review) Checklist to the Inorganic Section QA Chemist to notify them that the data has been reviewed. This copy is a convenience copy only and shall not be considered the official record.

14.1. Log books

The analyst is to keep an instrument maintenance logbook that includes daily conditions such as pressure and all instrument repairs/maintenance. Also, a reagent logbook is maintained for reagents prepared frequently from the same parent reagents (e.g. eluent and PCDR).

14.2. Record Retention

Records are retained according the DEQ Records Retention Policy. See document retention schedule for details and guidance on archiving. A project QAPP may require more stringent record retention.

Integrion Backup Procedure

DEQ maintains at least the most recent 3 months of data on the instrument's controller (i.e. PC computer). On a monthly basis a copy of all Chromeleon files, including raw data, is backed up to the DEQ LEAP server (DEQLEAD_LIMS) by following these steps:

1. Close all Chromeleon applications (Console/Studio, Instrument Configuration Manager, Administration Console, Services Manager)
2. Open the SERVICES tab from the task manager.
3. Stop all Chromeleon services and the SQL Server services in the following order by right-clicking on the desired service and selecting "Stop Service":
    a. Chromeleon 7 Instrument Controller Service: this should also stop the Chromeleon 7 Real Time Kernel Service automatically.
    b. Chromeleon 7 License Service: this should also stop the Chromeleon 7 Discovery Service, Chromeleon 7 Data Vault Service, Chromeleon 7 Data Processing Service, and Chromeleon 7 Scheduler Service automatically.
    c. Chromeleon 7 User Management Service and Data Processing Service.
    d. Chromeleon 7 Cache.
    e. SQL Server (SQLEXPRESS).
4. Confirm that all necessary services are stopped by checking the "status" column on the services list. If the service is stopped, the status column will be blank.
5. Open Windows Explorer and navigate to local computer C:\ProgramData\Dionex\Chromeleon
6. Copy the entire "Chromeleon" folder to deqlead-lims\Inorganic_InstData\107692-Integrion_IC\Chromeleon Full Backup\Year\Month (Year and Month folders are named for the year and month of the back up).
7. Once back-up is complete, re-start each of the services listed in 14.3.3 in the opposite order from which they were stopped.

15. Method Performance

Refer to Appendix B: Method Performance.

16. Maintenance

The system should be flushed with DI water for at least an hour weekly. This is usually done after analysis (see section 12.3.9) but if it happens that no samples are analyzed in a week, the DI water flush should still be done.

Maintenance of the IC and post-column system is integral to achieving valid results. DEQ maintains a service contract on the instrument and should not hesitate to contact either the telephone customer support or the field service engineer whenever a problem develops.

The operator shall record all maintenance, repair, or troubleshooting activities in the instrument maintenance log book including that performed by any third party.

17. Pollution Prevention

When possible, minimize the amount of chemicals used in the preparation and analysis to reduce waste.

18. Waste Management

The eluent waste should be placed in an appropriately labeled waste container in the laboratory. Eluent waste can be disposed of in the neutralizer. In the laboratory, there should be a satellite hazardous waste container for the Hexavalent Chromium standards. Hexavalent Chromium standard waste is disposed of in the high metals waste container. Hazardous wastes stored in satellite locations are bulked quarterly by the laboratory's hazardous waste coordinator. See Chemical Waste Management SOP DEQ04-LAB-0057-SOP for more information.

19. Definitions

Standard Definitions applicable to laboratory quality systems can be found in Appendix A of the LEAD Quality Manual (DEQ91-LAB-0006-LQM).

Specific definitions are explained below.

IC: Ion chromatograph

DI: Deionized

UV/VIS: Ultraviolet-Visible

PCDR: Post-column Derivatizing Reagent

ICV: Initial Calibration Verification

ICB: Initial Calibration Blank

LPM: liter(s) per minute

M: molar

mM: millimolar

20. Deviations from Referenced Methods

Extraction is done with 10mL of 20 mM sodium bicarbonate instead of 15mL. The CARB method increased extraction volume with revision 4.0. Our MDL is sufficient maintaining the 10mL extraction volume.

CARB method calls for filters to be impregnated ten at a time in a glass petri dish and to be dried on a drying rack for 24 hours covered by a kim wipe to avoid contamination. We saturate all the filters to be impregnated at one time in a covered plastic tub labeled for the purpose, and we dry them uncovered in a laminar flow hood (instead of covering them). This speeds up drying time and has less potential for contamination.

21. References

Extraction and Analysis of Hexavalent Chromium by Ion Chromatography, CARB MLD-039, version 4.0

Standard Operating Procedure for the *Determination of Hexavalent Chromium in Ambient Air Analyzed by Ion Chromatography (IC)*, ERG No.: 0143.04.003

Technical Assistance Document for the National Air Toxics Trends Stations Program, Revision 2

Technical Assistance Document for the National Air Toxics Trends Stations Program, Revision 3

Thermo manuals for all instrument components and supplies

22. Revision History

The editor shall increment the revision number with each approved revision. A new document is assigned a revision number of 1.0. The revision number of a document that receives routine or minor editing is updated by incrementing the minor number by one (i.e., 1.0 becomes 1.1) The revision number of a document that has undergone major revisions is updated by incrementing the major number by one and setting the minor number to zero (i.e., 1.1 becomes 2.0). Revisions to documents should be clearly identified in a "Revision History" section of the document. The Revision History documents the specific changes made to the controlled document, who made the changes, and the date (month and year) the changes were made.

Revision History

Revision	Date	Changes	Editor
1.0	02/22/2017	New document	PJH
1.1	01/08/2018 to 03/28/2018	Changed acronym PDR to PCDR to avoid confusion Added back up procedure to 14.3. Added detail to Element batching process in 12.2 Based on NATTS TAD 3, updated replicate and FP/FD QC requirements to be calculated from LOD rather than LOQ. Also updated frequency and mechanism for documenting replicates.	PJH & ZM

		Added field QC to table 3 Minor edits for clarity and grammar throughout and to update hyperlink file paths. Updated performance data in appendix B	
1.2	05/24/2021 07/01/2021 09/28/2021	Updated SOP to new template Added references to new DOC SOP Added revision number to reference method In multiple places, clarified extraction to analysis hold time from “same day” to “within 24 hours” since analysis often goes overnight. This also now matches the language in the reference method. Cleaned up back up procedure. Added detail to 12.3 Sample analysis A few minor grammar/wording changes Table 1 footnote removed by PJH and added to paragraph above based on LKM comment. Section 4: PJH corrected Sodium carbonate to Bicarbonate after LKM inquired about the chemical. Section 7: PJH added that reagents are made fresh daily after LKM inquired about expiration dates. Added that Eluent is good for three months. Section 10: PJH changed “curves” to “curve” (singular) after LKM inquired if there was more than one curve. Section 11.2, Table 4: Added what qualifiers to use. Section 14.2: Added that repairs and maintenance are added to Instrument Maintenance Logbook. Section 18: PJH added additional information and added the Chemical Waste Management SOP for reference as requested by LKM Section 19: PJH removed an acronym after LKM questioned what the acronym was.	PJH KPB LKM
2.0	08/06/2024 01/17/2025	Conversion to new template Removed hyperlinks Updated reagent recipes (1.0M H2SO4 to 0.9M in PCDR, and corrected ammonium sulfate from 33.0g to 66.0g in 2L so that molarity matches recipe for eluent) Section 9 updated to include storage requirements Completely rewrote section 14 to reflect GEL process Updated control charts and review checklist in appendices	RSL PJH

Appendix A: Attachment A Job Safety Assessment (JSA)

Hazard Analysis		
	Activity:	Cr^{+6}
	Program/Location	DEQ Laboratory
	SOP:	DEQ17-LAB-0006-SOP
	Analysis by:	LSO
	Date:	09/28/2021

Required PPE:

Gloves - Nitrile	Gloves – Cut Resistant	Safety Glasses	Safety Goggles	Lab Coat
				

Required/Recommended Trainings:

1. Review of Chemical Hygiene Plan
2. SIM-plicity Training
3. Review of relevant lab SOPs
4. Compressed Gas Safety Training
5. PPE Policy Review

TASK	HAZARDS	SEVERITY	CONTROLS
1. Computer Use/Data Entry	Repetitive motion injuries	Low	Follow ergonomic recommendations
2. Sample removal from refrigerators	(E) – Wet and cold temperatures	Medium	Gloves
	(CW) – Unknown samples and sample preservatives	Medium	Inspect samples for hazards prior to removing contents
3. Sample Handling and Preparation of Reagents and Standards	(CW) – Glass shards from broken sample vials, glass pipettes or needles on microsyringes.	Medium	Inspect glassware and prior to handling. Store sharps in a safe manner to prevent accidental punctures. Use puncture resistant gloves when handling fragile or damaged glassware.

	(E) – Solvent, acid or heavy metal exposure, unknown sample contaminant exposure, waste stream exposure	High	Always work in approved hood wearing appropriate PPE: lab coat, safety glasses/goggles and gloves suitable for chemicals in use. Dispose of all waste products in approved manner.
4. Sample Analysis/Instrument Operation and Maintenance	(CW) – Heated zones of instruments	Low	Allow instrument components and vacuum pumps to cool prior to performing maintenance. Wear thermal gloves where practical.
	(CW) – Shock hazard from electrical components	Low	Ensure instruments are turned off and unplugged when performing any work which may result in a shock hazard, such as board replacement or repair.
	(CI) – Pinch point hazard when operating automated instrumentation	Low	Ensure areas are clear of body parts and/or obstructions in swing radius of moving parts.
	(OE) – Disposing of acid waste solution in carboy	Medium	Limit volume of accrued waste to an amount that can be comfortably lifted (i.e. less than half-full). Follow ergonomic recommendations for lifting.
	(E) – Unknown sample contaminants	High	Wear all appropriate PPE when handling sample digestates or performing instrument maintenance. Dispose of all waste products in approved manner.

* Codes for Potential Hazards

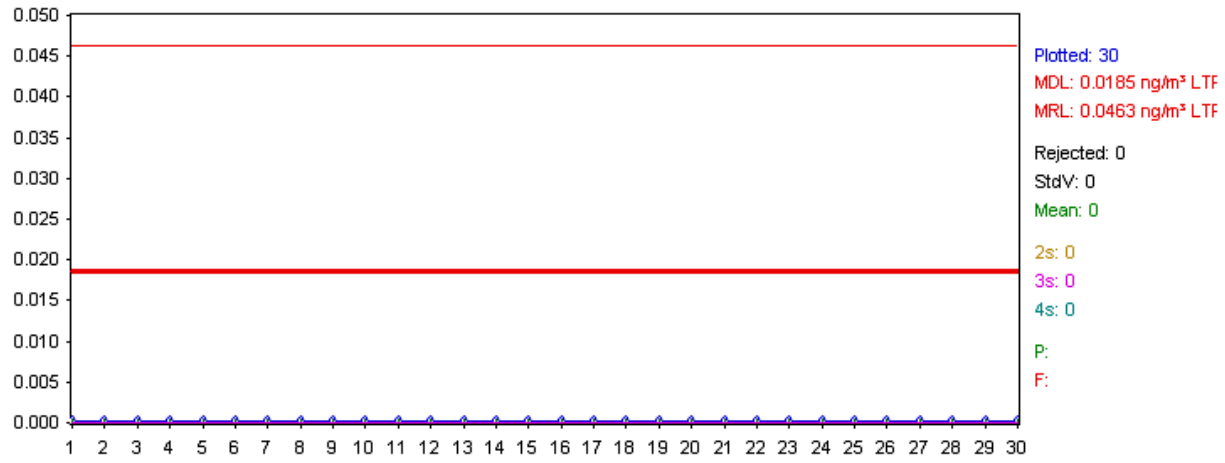
(BIO) Biological		(CO) Caught On		(FS) Fall – Same Level
(CB) Contacted By		(CW) Contact With		(OE) Overexertion
(CBT) Caught Between		(E) Exposure		(SA) Struck Against
(CI) Caught In		(FB) Fall To Below		(SB) Struck By

Risk Severity Level Key Low | Medium | High | Very High

Appendix B: Method Performance

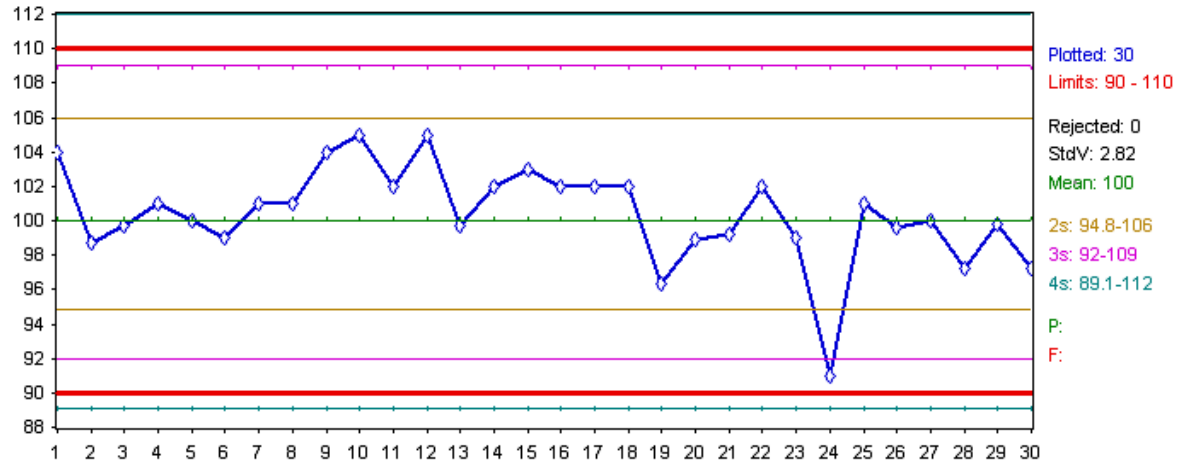
Printed:	21-Jan-25 09:18	Matrices:	Air::LAB
Client:	All Clients	Instruments:	107692-IC-Integrion
Project:	All Projects	Prepared By:	All Extractionists
From:	01/21/2024	Analyzed By:	PJH
To:	01/21/2025	Extractions:	All Extractions

Hexavalent Chromium [Cr(VI)] ***BLK***



Printed:	21-Jan-25 09:21	Matrices:	Air::LAB
Client:	All Clients	Instruments:	107692-IC-Integrion
Project:	All Projects	Prepared By:	All Extractionists
From:	01/21/2024	Analyzed By:	PJH
To:	01/21/2025	Extractions:	All Extractions

Hexavalent Chromium [Cr(VI)] ***BS %R***



Appendix C: Analytical Batch Review Form

OREGON DEPT OF ENVIRONMENTAL QUALITY
Laboratory and Environmental Assessment Division
Analytical Batch Summary and Review Checklist
Ion Chromatography (IC): Air Particulate Hexavalent Chromium

Analytical Batch Information

SOP: DEQ17-LAB-0006-SOP
Version: 1.3 **Date:** 1/21/2025

Sequence ID: _____
Method: CARB MLD-039, rev 4.0
Instrument: 107692 Integrion
Analyst: _____ **Date:** _____

Work Order #'s: _____

Preparation Batch Information

Prep. Batch ID(s): _____

Standards & Reagents

Standard and reagent records in Element. Supplemental reagent information in logbook: L20170216B

LOQ 0.046 **LOD** 0.019 **ng/m³**

		Analyst		Analyst Comments (see below as well)	Reviewer		review notes
Calibrations		**Pass**	**Fail***		**Agree**	**C.A.R.****	
Linear Curve	$r^2 \geq 0.995$	☐	☐		☐	☐	
Curve fit	90-110% for all cal points	☐	☐		☐	☐	
ICV (2nd source)	Recovery: 90-110%	☐	☐		☐	☐	
ICB/CCB	Conc < LOD	☐	☐		☐	☐	
LCV	Recovery: 50-150%	☐	☐		☐	☐	
CCV	Recovery: 90-110%	☐	☐		☐	☐	
Analytical Quality Control		**Pass**	**Fail***		**Agree**	**C.A.R.****	
Batch Size	≤ 20 samples	☐	☐		☐	☐	
MB	Conc < LOD	☐	☐		☐	☐	
LCS	Recovery: 90-110%	☐	☐		☐	☐	
Sample QC		**Pass**	**Fail***	**N/A**	**Agree**	**C.A.R.****	
Replicate Analysis	RPD: ≤ 10% (or ≤ LOQ if avg ≤5X LOD)	☐	☐		☐	☐	
Manual Integrations	Verified and appropriate.	☐	☐	☐	☐	☐	
Field QC		**Pass**	**Fail***	**N/A**	**Agree**	**C.A.R.****	
Field Blank(s)	Conc < LOD	☐	☐	☐	☐	☐	
Field Duplicates	RPD: ≤ 20% (or ≤ 2xLOQ if avg ≤5X LOD)	☐	☐	☐	☐	☐	
Data Management		**Yes**	**N/A**		**Yes/Agree**	**C.A.R.****	
Data Calculations/Entry/Upload Complete & Accurate		☐			☐	☐	
LIMS: Chemist Initials, Instrument ID, Update Status		☐			☐	☐	
Is optional batch narrative attached?		☐	☐				

Follow up after initial review	**Yes**	**N/A**	
Corrective actions that were required (C.A.R.) by the reviewer have been completed:	☐	☐	

Signatures & Dates

Analyst	Date	Reviewer	Date:

Additional Analyst Comments	**Reviewer comments**

See back for additional comments

Printed copies of this document are considered uncontrolled.

OREGON DEPT OF ENVIRONMENTAL QUALITY

Laboratory and Environmental Assessment Division

Analytical Batch Summary and Review Checklist

Ion Chromatography (IC): Air Particulate Hexavalent Chromium

Additional Analyst Comments	Reviewer comments

The batch was selected for an internal audit: ☐

* If a QC element fails and data is reported with a qualifier, include in the comments the QC result value, the qualifier code, and the grade. If data is not reported, comment "Affected data not reported".
** C.A.R = Corrective Actions Required--Comment should state if a "green sheet" or "issue" was created.