Standard Operating Procedure

EDB, DBCP, 1,2,3-TCP in Water GC-ECD 504.1

DEQ93-LAB-0021-SOP
Version 7

April 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
Hillsboro, OR 97124
Contact: Sara Krepps
Phone: 503-693-5700
www.oregon.gov/deq

DEQ93-LAB-0021-SOP
Version 7
Last updated: April 7, 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:** _____
Brian Jordan, Chemist

Reviewed by:

_____ **Date:** _____
Jeremy Unrau, Organic Section Manager

Approved by:

_____ **Date:** _____
Sara Krepps, Laboratory Quality Assurance Officer

Table of Contents

List of Tables

List of Appendices

1. Scope and Application

This method is suitable for analysis of 1,2-Dibromoethane (EDB), Dibromochloropropane (DBCP), and 1,2,3-Trichloropropane (1,2,3-TCP) in drinking water and ground water samples. This method meets the analytical requirements specified in EPA Method 504.1 and may be used for regulatory purposes in drinking water matrices.

1.1 Applicable Matrices

This method is applicable to following matrices:

- Drinking water
- Ground water samples

1.2 Detection Limits

Limits of Detection (LOD's) for the analytes in Table 1 are determined in reagent water following the requirements of 40CFR part 136, Appendix B, Revision 2 and the Limits of Detection (LOD) and Quantitation (LOQ) SOP (DEQ18-LAB-0053-SOP). LOD's are updated annually or following significant changes in method or instrumentation. Current detection limits studies may be found on the lab QA network drive.

Table 1: List of target analytes and Limits of Quantitation

Compound	CAS Number	LOQ[1], µg/L	ULOQ[2], µg/L
1,2-Dibromoethane (EDB)	106-93-4	0.05	2.857
1,2-Dibromo-3-Chloropropane (DBCP)	96-12-8	0.05	2.857
1,2,3-Trichloropropane (1,2,3-TCP)	96-18-4	0.05	2.857

[1] *The LOQ or Limit of Quantitation is also commonly known as the method reporting limit (MRL).*

[2] *The Upper Limit of Quantitation (ULOQ) may be extended by sample dilution.*

2. Summary

Thirty-five mL of the aqueous sample is extracted with 2 mL of Hexane. The extract is then analyzed for 1,2-Dibromoethane (EDB), Dibromochloropropane (DBCP), and 1,2,3-Trichloropropane (1,2,3-TCP), by Gas Chromatography with an Electron Capture Detector (ECD). Aqueous calibration standards are extracted and analyzed in an identical manner as the samples in order to compensate for possible extraction biases.

3. Personnel/Qualifications

The analyst should meet the minimum qualifications for a Chemist II position. A chemist who has previously demonstrated their proficiency at performing the method should train the analyst. An Initial Demonstration of Capability (IDOC) is to be conducted and passed prior to reporting data as required by the Drinking Water Certification Program and the DEQ Laboratory Quality Manual (LQM). The Drinking Water Certification Program also requires the analyst to conduct and pass a Demonstration of Capability (DOC) each year.

4. Interferences

Impurities contained in the extraction solvent usually account for the majority of the analytical problems. Laboratory blanks should be analyzed on each new bottle of solvent before use. Whenever interference is noted in the reagent water blank, the extracting solvent should be reanalyzed. Low level interferences generally can be removed by distillation of the solvent.

Protect interference-free solvents by storing in an area known to be free of organochlorine solvents.

Current column technology suffers from the fact that EDB at low concentrations may be masked by very high levels of dibromochloromethane (DBCM), a common disinfection by-product of chlorinated drinking waters. Analysis of DBCM along with standards is necessary in order to confirm that no co-elution with EDB is occurring. Currently, the DBCM standard utilized is purchased from Ultra Scientific; catalog # HC-100-1.

5. Safety

The analytes and solvents mentioned in this method-be they detected in samples, used in extraction, or used to make standards-are hazardous and every effort should be made to avoid contact with them. A laboratory coat, organic-impervious gloves and a fume hood are all good preventatives in this effort. In general, disposable Nitrile gloves provide effective protection from the solvents and analyte standards used in this method as well as possible biological hazards from the field samples themselves. If any solvent or standard contacts the gloves, the analyst should immediately remove the gloves and replace them with new.

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.

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

Please refer to the attached Job Safety Analysis for further safety information when performing this analytical method.

6. Equipment and Supplies

- Agilent 6890 Series Gas Chromatograph, split/splitless/PTV injector
- Agilent 7683 Autosampler
- Agilent ChemStation
- Restek RTX-CLPesticides2 (30 m X 0.25 mm ID X 0.2 µm film thickness) or equivalent
- Restek Rxi-XLB column (30 m X 0.25 mm ID X 0.5 µm film thickness) or equivalent
- 40 mL purge vials
- 1.5 mL extract vial
- Volumetric pipets, 2 mL
- Gas-tight syringes, 10, 25, 50, 100, 250, 500, and 1000 µL
- Volumetric flasks, 10 and 25 mL
- Pasteur Pipettes, 5 to 6 inch in length
- Standard Storage vials, approximately 10 mL

7. Reagents

- Hexane, pesticide grade or better.
- Methanol, ACS grade or better.
- Reagent water
- Sodium chloride, crystal, ACS reagent grade
- Sodium thiosulfate ACS reagent grade; for sample preservation– (0.04g/mL) mix 1g in 25 mL vol flask.

8. Standards

8.1 Standard Materials

- 1,2-Dibromoethane – 99 %
- 1,2- Dibromo-3-chloropropane – 99%
- 1,2,3-Trichloropropane – 99%

8.2 Stock Standard Solutions

Place approximately 9 mL of methanol in a 10 mL volumetric flask, wait until the wetted surfaces have dried, about 10 minutes, then add one or two drops of EDB directly to the methanol, ensuring the EDB does not touch the sides of the flask. Reweigh the flask, bring the solution to volume, stopper and shake the flask. Transfer the stock standard to a standards vial and store in the standards freezer. Repeat for DBCP and 1,2,3-TCP.

$$\text{Stock Conc, } \mu\text{g/mL} = \frac{\text{Std Wt, mg}}{10\text{ mL}} \times \frac{1000\ \mu\text{g}}{1\text{ mg}} \qquad (1)$$

Purchased certified standards are currently utilized as a substitute for in-house stock standards.

8.3 Primary Dilution (Working) Standard Solutions

Use stock standard solutions to prepare a 10 µg/mL working standard that contains all analytes in methanol. Using a stock such as Restek 504.1 Cal Mix (Cat. No. 30239) at 200 µg/mL, the following calculation is used:

$$\text{Volume needed, } \mu\text{L} = 500\ \mu\text{L} = \frac{(10\ \mu\text{g/mL}) \times (10\text{ mL})}{200\ \mu\text{g/mL}} \times \frac{1000\ \mu\text{L}}{1\text{ mL}} \qquad (2)$$

This 10 ug/mL Working Stock is used to prepare two Standard Stocks, one at 1000 ug/L and the second at 35 ug/L. These two stocks are used for the preparation of the calibration curve.

8.4 Calibration Standards

Prepare standards by adding the appropriate amount of working standard to 35 mL of reagent water in a 40 mL purge vial via syringe. Equation 3 shows an example calculation appropriate for a 100 uL volume of 1000 ug/L Standard Stock into 35 mL of DI water required to prepare the Cal Level 7 standard at 2.857 µg/L.

$$100\ \mu\text{L} \times \frac{1000\ \mu\text{g/L}}{35000\ \mu\text{L}} = 2.857\ \mu\text{g/L} \qquad (3)$$

Prepare calibration standards at a minimum of 7 concentrations from the working standard. The levels should bracket the expected concentration in the sample. Standards are prepared following the table below:

Table 2: Calibration Standard Preparation Volumes

Stock Conc, µg/L	Stock Volume, uL	Final Vol, ml	Sample Conc, µg/L	Conc in Extract, µg/L	Cal Level
1000	100	35	2.857	50.00	7
	50	35	1.429	25.00	6
	25	35	0.714	12.50	5
	10	35	0.286	5.00	4
35	100	35	0.100	1.75	3
	50	35	0.050	0.875	2
	20	35	0.020	0.350	1 (LOD CK)

9. Sample Collection, Preservation, Shipment, and Storage

9.1 Sample Collection

Samples are collected in 40 mL purge vials. 75 µL of 0.04 g/mL freshly prepared Sodium thiosulfate must be added to all samples to suppress the formation of Dibromochloromethane.

When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 10 min). Adjust the flow to about 500 mL/min and collect samples from flowing stream.

When sampling from well, fill a wide-mouth bottle with sample, and carefully fill 40mL sample bottle.

9.2 Sample Preservation

The samples must be chilled to 4 ºC on the day of collection and that temperature maintained with ice during shipping. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure that they will be ≤ 4 ºC on arrival at the laboratory.

9.3 Sample Storage

Store samples and field reagent blanks at 4 ºC until analysis time. Refrigerator temperature must be monitored; and sample storage area must be free of organic solvent vapors.

Samples must be extracted within 14 days of collection. Extracts may be held up to 24 hours.

10. Calibration and Standardization

10.1 Initial Calibration

The calibration standard should contain EDB, DBCP, and 1,2,3-TCP at a concentration at or below the Limit of Quantitation for each compound; and calibration may not be forced through the origin. Currently, seven calibration standard levels are prepared from 0.02 µg/L to 2.857 µg/L. Each calibration standard is analyzed accordingly and an initial calibration curve independent of past calibrations is prepared.

Table 3: Summary of Initial Calibration Requirements

No. of calibration standards	7 (3 minimum, 5 recommended)
Low calibration standard	0.020 µg/L
High calibration standard	2.857 µg/L
Signal Response	Peak Area
Calibration Method	1st choice: Average Response Factor 2nd choice: Linear 3rd choice: Quadratic
Acceptance criteria	Linear: $r^2 \geq 0.990$ ARF: RSD ≤ 20%
Calibration frequency	With Each Analysis
Initial Calibration Verification	Second Source Standards Conc: 0.714 µg/L Acceptance Criteria: 70-130%
Thresholds	If used, Chemstation reporting thresholds will be set no higher than ½ the low calibration point.

10.2 Calibration Verification

The working calibration curve must be verified each day by analyzing standards at a minimum of 2 levels. If the concentration of any of the analyte varies from the known value by more than 20%, the test must be repeated using a fresh calibration standard. If the results still do not agree, a new calibration curve must be generated.

Table 4: Summary of Continuing Calibration Requirements

Frequency	Beginning of each analytical sequence End of each analytical sequence Every 12 hours of operation
Concentration(s)	Vary the concentrations of the calibration standards used
Acceptance criteria	Recovery: 80 – 120%
Corrective Actions for Failures	1) Reanalyze the CCV 2) Recalibrate, if the reanalysis of the CCV fails 3) Reanalyze the preceding samples (to the last check that passed) after acceptable calibration is restored.

Performance of the entire analytical system should be checked using data gathered from analyses of reagent water blanks, standards, and quality control check standard. If one of the analytes in the calibration check is outside the 20% criteria, the preceding samples (to the last check that passed) must be rerun after acceptable calibration is restored.

11. Quality Control

No analytical results are valid, and no further analyses should be performed if the QC parameters are not met. For any QC problems, the source must be identified and corrected before any further analysis is done and all analyses performed since the last time all QC parameters were met must either be appropriately flagged or repeated (if possible) as needed.

Quality control (QC) requirements include determination of the Limit of Detection (LOD), Demonstration of Capability (DOC) followed by regular determination of instrument performance and analysis of reagent blanks, field blanks, and spikes.

All analysts and laboratories using this method are required to operate a formal Quality Control (QC) program. The laboratory is required to maintain performance records that define the quality of the data thus generated.

11.1 Data assessment and Quality Control Acceptance Criteria

Data assessment and QC acceptance criteria are of primary importance in assessing the quality of data resulting from analytical batch. Be especially attentive to analytical bias; negative bias is of equal concern to positive bias. Any samples returning “hits” for any analyte(s) above the upper end of the calibration range must be diluted and reanalyzed so that all detected analytes quantitate within the calibration range. The following data assessment and QC data are required for all analyses:

Table 5: Listing of required quality control elements, including frequency and acceptance limits

QC Element	Frequency	Acceptance Criteria
Initial Demonstration of Capability (IDOC)[1]:	Initial method development	Mean concentration 70-130% of true value, RSD ≤ 20%
Continuing Demonstration of Capability (CDOC)	Annually	Successful PT or CDOC
Limit of Detection Study[2]	• Initial method development • Each new analyst Annually, thereafter	Signal to Noise Ratio: 3 to 5
Initial Calibration Verification (ICV)	Immediately following every ICAL	Recovery: 70 – 130%
Continuing Calibration Verification (CCV)	• Start and End of every analytical sequence Every 12 hours of operation	Recovery: 80 – 120%
Method Blank	Daily	must be ≤MDL
Field Reagent Blank	Daily (when available)	must be ≤ MDL
LCS – must be at 0.25 µg/L	10% of samples analyzed	Recovery: 70 – 130%

QC Element	Frequency	Acceptance Criteria
LOD check standard – spiked at current LOD concentration.	weekly	Recovery: 60 – 140%
DBCM RT/Interference Check	Daily	RT NOT in EDB window
Matrix Spike (MS)	5% of samples analyzed	Recovery: 65 – 135%, RSD ≤ 30% (MSD if performed)
Quality Control Sample (QCS)	Quarterly or with every use, whichever is longer (if another second source QC standard is not analyzed)	Recovery: 70 – 130% or as determined by study

[1]IDOCs generally consist of 4 LCS @ mid-point concentration. See laboratory SOP on Demonstrations of Capability for more information. [2]See laboratory SOP on Determining the LOD/LOQ.

Before processing samples, the analyst must demonstrate that all glassware and reagent interferences are under control. Each time a set of samples are extracted, or new reagent bottles are used, a LRB must be analyzed.

The analyst is permitted to modify GC columns and GC conditions. Each time such modifications are made, the analyst is required to determine a new IDOC. The IDOC is performed by extracting a BLK and 4 to 7 LCS's using reagent water preserved with sodium thiosulfate. Spike target analytes at a concentration of about 10 to 20 times the LOD or at the mid-point of the calibration curve for each compound of interest. Extract and analyze the IDOC according to the procedure section. The IDOC must meet the requirements in Table 4.

11.2 Corrective Actions for Out-of-Control Data

Corrective actions must be taken for all data found to be out-of-control, as summarized in the Table: List of corrective actions to be used in addressing out-of-control QC data. Out-of-control QC data can often follow samples that are especially "dirty" or have high concentrations of analytes. If this is suspected, it is often best to stop the analysis, clean the sample introduction system, and reanalyze the samples after they have been diluted.

In some cases, out-of-control data may be indicative of a serious instrumental problem that may require a service visit. When the corrective actions given below fail to correct the identified problem, assess the situation with the senior chemist.

Table 6: List of corrective actions to be used in addressing out-of-control QC data.

Quality Control Element	Corrective Action
Method Blank	If any peak, which will prevent the determination of the analyte is identified, the source of contamination must be determined and eliminated before processing samples.
ICV	Recalibrate.
LCS	Reanalyze all samples associated with the failed LCS.
CCV	Reanalyze CCV. Upon subsequent failure, recalibrate.
LOD check standard	Reanalyze. Determine the source of problem and repeat the test.

Quality Control Element	Corrective Action
Matrix Spike (MS)	Confirm. If LCS is not in control, flag data as estimated and may have matrix effects.
QCS	Identify and correct the source of the problem.

11.3 Contingencies for Handling Out-of-Control Data

On rare occasion the analysis cannot be brought under-control. In these cases contingencies plans must be employed. The following list provides a guideline for handling out-of-control data:

- Contact the project manager and verify the importance of the data. If the analyte is of minimal importance, determine if it can be reported as an estimate or not reported at all.
- Determine with the project manager if an alternate sample can be used for their evaluation purposes.
- If it seems appropriate in your best professional judgment, speak with the project manager about obtaining an alternate sample that may provide the required information.
- If QC data suggests the analysis is showing a low bias, report **only** that data that exceeds a regulatory limit. Be sure and clearly comment this information in the LIMS system and in the analytical documentation.
- If QC data suggests the analysis is showing a high bias, report **only** that data that is below the LOQ. Be sure and clearly comment this information in the LIMS system and in the analytical documentation.
- In *every* case, the problem(s) and attempted corrections should be noted in the instrument logbook (when instrumental problems have occurred), with the analytical batch data, and in the LIMS system for final reporting.

12. Procedure

12.1 Sample Preparation

- Remove samples and standards from storage and allow them to reach room temperature.
- For samples and field reagent blanks, remove the vial cap (shake sample first to homogenize) and pipette off or decant about 5.0 mL.
- Replace the cap and weigh the container to the nearest 0.1 g (for subsequent sample volume determination).
- For calibration and check standards, LOD check standard, LCS, and laboratory reagent blanks, measure a 35 mL volume of reagent water using a 50 mL graduated cylinder and transfer it to a 40 mL vial.

Note: Remember that standards used in this analysis are procedural and must have 75 µL of 0.04 g/mL freshly prepared sodium thiosulfate added to all samples and standards to suppress the formation of Dibromochloromethane.

- Add an appropriate volume of the calibration working standard to 35 mL reagent water of those vials that will be used to extract the calibration and check standards, as well use laboratory fortified blanks (see section 11.4).
- Use a micro syringe and rapidly inject the standard into the mid part of the vial.

12.2 Micro Extraction

- Add 6 g NaCl +/- 0.4 g to the vial and shake until dissolved (about 20 sec).
- Add 2.0 mL of Hexane, shake vigorously for one minute, and then allow phases to separate. (If stored at this stage, keep the container upside down.)
- Carefully transfer approximately 0.3 mL of the hexane extract via pipette to a 1.5 mL extract vial with an insert for analysis. Transfer the remaining hexane phase into a second vial and reserve it at 4 ºC for a reanalysis if necessary. (Note: Transferring the extract to a 4mL vial can aid in separating the solvent phase from the aqueous phase).

12.3 Determination of Sample Volume

- Determine the volume of samples and field blanks by discarding the remaining water/hexane mixture.
- Reweigh the empty sample vial and calculate the net weight of sample to the nearest 0.1g, by subtracting the empty container weight from the full container weight.
- This mass (in g) equals the volume of water extracted (in mL).

12.4 Cleanup Procedure

No cleanup is performed.

12.5 Sample Analysis

The daily calibration standards are injected in order of increasing concentration. A seven point calibration table is generated. Extracts are loaded onto the autosampler (blanks run before samples) and the sampling sequence initiated. If the analyte concentration in an extract is higher than the highest calibration standard, an appropriate dilution from the second extract vial for that sample is prepared and analyzed.

12.6 Current GC Parameters

Samples are analyzed on method ‘504_1.M’ and data is processed on method ‘P504_1.M’ using the HP Chemstation. Below are the GC operation conditions:

Injector:
5 µL, 1 time

Inlet program:
Mode — Solvent vent
Gas — He
Heater — 10 °C
Pressure — 17.74 psi
Total flow — 106.6 mL/min
Vent flow — 20.0 mL/min
Vent pressure — 0.0 psi, until 0.50 min
Purge flow to split vent — 100 mL/min @ 2.00 min.
Gas saver ‘ON” — 20 mL/min @ 10.00 min

Column:
Pressure — 17.76 psi
Column flow — 1.8 mL/min
Average velocity — 38 cm/sec

Oven program:
Initial temp — 35 °C
Oven max — 320 °C
Equilibration —0.5min

Detectors:
Heater — 370 °C
Makeup gas — N_2
Column + Makeup set to constant flow — 40 mL/min

Column comp1 – front; column comp 2 - rear

Table 7: Inlet program operating parameters

Ramp	(°C/min)	Next °C	Hold (min)
Initial	-	-10	0.70
Ramp 1	720	275	15.00

Table 8: Oven program operating parameters

Oven Ramp	°C/min	Next °C	Hold min	Run Time (min)
Initial	-	35	2.00	2.00
Ramp 1	10	200	0	13
Ramp 2	40	300	2	18
Post run	-	-	-	-

12.7 Dual Column Analysis and Confirmation

Each analyte is reported only if properly identified and quantified on each column. In general, each analyte can be reported from either column, as long as the same column is reported throughout the sequence and the column is in calibration. The analyst must use their discretion as to which column to report from by taking into consideration CCV recoveries, baseline noise, coelutions, etc.

Sample results are confirmed by using two dissimilar columns. The agreement between the results should be evaluated after the identification has been confirmed. Large differences in the numerical results from the two analyses may be indicative of positive interferences with the higher of the results, which could result from poor separation of target analytes, or the presence of a non-target compound. However, they may also result from other causes. Thus, in order to ensure that the results reported are appropriate for the intended application, the analyst should make a formal comparison, as described below.

Calculate the relative percent difference (RPD) between the results from each column. See Section 17.2 for the calculation. If the RPD is significantly higher, e.g., >40% RPD, check the chromatograms to see if an obviously interfering or overlapping peak is causing an erroneously high difference. If no such peaks are observed, examine the baseline of the peak. A poorly integrated baseline may cause a difference between the peaks.

If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, then it may be appropriate to report the lower result.

13. Calculations

- Sample volume (V_s): is calculated as equal to the net sample weight.

$$V_s = \text{gross wt.} - \text{bottle tare}$$

- Sample concentration:

$$\text{Concentration, } \mu g/L = C_i \times \frac{2}{V_s}$$

where C_i is the instrument output in µg/ L, and V_s is the measured sample volume.

Results should be reported to three significant figures.

- Relative Percent Difference:

$$RPD = \frac{|(X1 - X2)|}{|(X1 + X2)|/2} \times 100$$

Where X1 and X2 are the results of samples 1 and 2, respectively.

- Relative Standard Deviation:

$$\%RSD = \frac{stdev}{|mean|} \times 100$$

- Limit of Detection:

$$LOD = st(n-1,1-alpha=0.99)$$

where:
s = standard deviation of replicate analysis
n = number of replicates
$t_{(n-1,\ 1\text{-}alpha\ =\ 0.99)}$ = Student's t value for the 99% confidence level with n-1 degrees of freedom

- Recovery:

$$\%\,\text{Recovery} = \frac{Cs - Cu}{Cn} \times 100$$

where: Cs = Measured concentration of the spiked sample aliquot

Cu = Measured concentration of the unspiked sample aliquot (use 0 for the LCS)
Cn = Nominal concentration increase that results from spiking the sample, or the nominal concentration of the spiked aliquot (for LCS).

14. Records Management

In 2019, the Department of Environmental Quality Laboratory began an effort to convert to a primarily paperless system and is in a period of transition. All records are managed in accordance with the state record retention schedule, regardless of format. Physical records are stored either onsite or in secure offsite document storage. Electronic documents are stored on internal servers prior to being transferred to the State Archives. The Oregon Records Management System is a statewide program for managing electronic public records. ORMS is a cloud-based system for governing records and information management.

Data files on the GC must be backed up on a regular basis and should be done monthly.

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\Organic_InstData\51968 Dual ECD

Chromatograms are to be collated based on run order combined with the following documentation as PDFs:

1. Analytical Checklist PDF (YYMMDD_Checklist_etc.pdf), contains the checklist, Element sequence, and Element Evaluation Reports for field blanks and field duplicates.
2. Curve data PDF (YYMMDD_Curve_data.pdf) includes:
    a. Calibration Point Difference report,
    b. Calculated Concentration report,
    c. Raw data Imported from calibration,
    d. Calibration Curve Fit report,
    e. Calibration Plot report,
    f. Calibration Status report,
    g. Data Analysis Parameters report
    h. All Quantitation reports which include chromatograms associated with the calibration,
    i. Acquisition times,
3. Sample data PDF (YYMMDD_Sample_data.pdf) includes samples chromatograms in run order, including manual integrations (before & after) and peak deletions, generated using the Detailed Macro. Where possible, use the “Continuing Calibration Report” to calculate percent recoveries for Minimum Reporting Level Checks, Calibration Standard, ICV, LCS, and CCV percent recoveries.

The analyst generates the preceding documentation and passes it on to a second chemist for peer review by cutting the sequence 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.

In other instances, the analyst may combine the multiple pdfs into one master report pdf, named as SequenceID.pdf, e.g. S25A123.pdf. The individual files listed above will be ordered in the master report as

designated; the contents of report 01 will be the first pages of the master report (which should be the checklist), followed by the contents of report 02, then 03, etc. In this instance, the pdf, not a folder containing multiple pdfs, is removed from the **!In_Process** folder and transferred to the **!Needs_Review** folder for peer review.

The preceding documentation is put into the order listed above and it is passed on to a second chemist for peer review. Access to the data is limited to DEQ employees since the facility is secured. Data packets should be maintained in accordance with the State records retention schedule.

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: e.g.

15. Method Performance

Current Method Validation data may be found on the QA drive of the Shared directory.

16. Maintenance

Daily inspection of the GC system is a good practice prior to operating the instrument. Any work done on the instrument regarding maintenance should be recorded on the GC-ECD maintenance log book.

17. Pollution Prevention

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

No solvents are utilized in this method except the very small volumes required to make calibration standards. The only other chemicals used in this method are the materials used in preparing standards and sample preservatives. All are used in very small amounts and pose little or no danger to the environment.

For information about pollution prevention that may be applicable to laboratories and research institutions, consult *Less is Better: Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, 202.872.4477.

18. Waste Management

The Oregon Department of Environmental Quality requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions.

Recycling is the first course of action for waste management in the laboratory. Since quantitative limitations combined with laboratory efficiency specify that all samples requiring volatile analysis should be collected in clean, un-used 40 mL glass vials, the laboratory has made a concerted effort to recycle these vials through the recycling program. All packing materials (i.e., cardboard boxes) and waste paper are to be recycled.

Waste Disposal: All samples, standards and sample extracts that are no longer of use and that meet the criteria to be defined as hazardous waste must be treated and/or disposed of as such following all appropriate procedures/protocols in the Lab.

The aqueous liquid that remains in the sample vials following analysis should be removed from the vials and the vials can then be recycled. The liquid should be sparged with air in a fume hood until the contaminants are driven off and it can then be disposed of in the sink.

For further information on waste management consult *The Waste Management Manual for Laboratory Personnel*, available from the American Chemical Society.

19. Definitions

Standard Definitions applicable to laboratory quality systems can be found on in the Laboratory Quality Manual, DEQ91-LAB-0006-LQM.

20. Deviations from Referenced Methods

None

21. References

Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures Quality Assurance, Fifth Edition, EPA 815-R-05-004, January 2005

Method 504.1. 1,2-Dibromoethane (EDB), 1,2-Dibromo-3-Chloro-Propane (DBCP), and 1,2,3-Trichloropropane (1,2,3TCP) in Water By Microextraction and Gas Chromatography. Revision 1.1; Munch, J.W.; National Exposure Research Laboratory Office of Research and Development U.S. Environmental Protection Agency: Cincinnati, OH; 1995.

22. Revision History

Revision	Date	Changes	Editor
3.1	Feb 07	Updated SOP to current DEQ Laboratory requirements.	RAD
4.0	May 08	Updated to current DEQ SOP format, included recent performance data, clarified procedure.	KDK
4.1	08/21/2012	Revised following DW Audit. Updated formatting. Added dual column confirmation criteria.	SLK

4.2	05/23/2014	Annual review. Updated ICAL requirements table 2 with Threshold information.	SLK
	05/29/2014	Added suggestions to SOP from last EPA audit. Update Standard information in sections 8.3 and 8.4, Added clarification note to sample preparation section 12.1.	KDK
4.3	6/30/2015	Update 6.0 column info, 8.3-8.4 standard prep, 10.1 cal info, 12.6 instrument details, changed focus from “batch” to “sequence” in 12.7	KDK
4.4	3/18/2016	Update 9.1 and Table 2 for correct calibration concentrations and units Updated version and date of SOP	RP NMM
4.5	6/1/2017	Updated 4.0 for clarity about checks for DBCM co-elution, 5.0 for column used, and 11.6 GC parameters.	BJ
4.6	6/26/2018	Updated 11.6 GC parameters.	BJ
4.7	8/28/2019	Updated cover pg. for QAO. Updated Section 1.2 for LOD, Section 7.4 & Table 4 to remove LOQ check requirement. Updated 13.0 Record management for accuracy.	NMM
4.8	10/12/2020	Updated Section 11.6 for GC parameters. Corrected revision history for duplicate Rev 4.5 entry.	BJ
4.9	11/3/2021	Added section 6.0 equipment and supplies, changed instrument manufacturer. Clarified 9.3 language on sample hold time.	JU
5.0	01/09/2023	Updated Table 4. MB and FB criteria ≤MDL. Updated section 12.0 for analysis method. Updated section 5.0 Safety for online SDS. Section 14 Records Management for State Retention Schedule and removing “Print” to prepare for GEL. Section 15 Method Performance for storage on the QA drive.	BJ NM
6.0	01/26/2024	Routine review & minor editorial edits made. Changed < symbol to ≤ for IDOC RSD limit per the reference method. New template	BJ/NM RL
7.0	4/2/2025 4/7/2025	Updated Records Management for electronic data packages. Minor editorial edits	BJ NM

Appendix A: Job Safety Assessment

Hazard Analysis		
	Activity:	Chromatographic Analysis
	Program/Location	DEQ Laboratory
	Position # (s):	0160, 0167, 0193, 2603, 2645, 3010, 3012, 3276, 3277
	Analysis by:	Health & Safety
	Date:	2/21/2019

Required PPE:

Gloves - Nitrile	Safety Glasses	Lab Coat	Gloves - Thermal	Gas Cylinder Hand Truck	Blast Shield	Safety Goggles
						

Required/Recommended Trainings:

1. 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 Preparation/Dilutions	(CW) – Glass shards from broken sample vials, glass pipettes or needles on microsyringes	Medium	Inspect glassware and extract vials prior to handling. Store syringes in a safe manner to prevent accidental punctures
	(E) – Solvent, acid or heavy metal exposure, unknown sample contaminant exposure	High	Always work in approved hood wearing appropriate PPE: lab coat, safety glasses/goggles, gloves suitable for chemicals in use. Some procedures may require using a blast shield
3. Extract removal from refrigerators	(E) – Wet and cold temperatures	Medium	Gloves
	(CW) – Glass shards from broken sample vials; unknown	Medium	Inspect extracts for hazards prior to removing contents

	samples and sample preservatives		
4. Instrumental Analysis	(E) – Compressed gases and cryogenics;	High	Proper storage/use of gas cylinder & use proper technique when transporting cylinders. Use only compatible regulators with proper fittings. Test all connections for leaks. Wear thermal gloves when handling liquid nitrogen.
	(CW) - burns from heated instrument zones.	Medium	Cool heated zones prior to performing instrument maintenance.
	(E) – Solvents, hazardous waste, used pump oil	High	Always work in approved appropriate PPE: lab coat, safety glasses, gloves suitable for chemicals in use.
	(CBT) – Pinch points on autosampler when moving	Low	Ensure areas are clear of body parts and/or obstructions in swing radius of autosampler arm.
	(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.

* 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