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Trihalomethanes - EPA Method 551.1

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1. Scope and Application

1.1. Method 551.1 is used to determine the concentrations of trihalomethane compounds in drinking water. Water samples are extracted with Pentane. The extract is injected in a temperature programmable gas chromatograph with an electron capture detector. Identifications are obtained by analyzing a standard curve under identical conditions used for samples and comparing resultant retention times. Analytes are quantitated using procedural standard calibration.

1.2. This method is restricted to analysts who have completed the requirements of the initial demonstration SOP. See SOP reference 13.2.

2. Definitions

2.1. Refer to Section 3 and Section 4 of the Georgia EPD Laboratory Quality Assurance Manual for Quality Control definitions.

2.2. Primary Source (PS) – A standard that is used to make up the calibration points of a curve.

2.3. Second Source (SS) – A standard made from a manufacturer other than that of the primary source.

2.4. Initial Calibration Verification (ICV) – An ICV is a second source standard that is used to verify the correctness of the primary sources calibration curve. The ICV is run at a level equal to that of a Laboratory Control Sample (LCS) or the midpoint on the calibration curve.

2.5. Field Duplicate (Original sample and Field Duplicate) – Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of original sample and field duplicate gives a measure of the precision associated with sample collection, preservation, storage, and as well as with laboratory procedures.

3. Interferences

3.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in chromatograms.

3.1.1. Glassware must be scrupulously cleaned with hot water and detergent followed by de-ionized water then rinsed with methanol followed by acetone.

3.1.2. The use of high purity reagents and solvents helps minimize interference problems.

3.2. Plastics should never be used at any point in this analysis.

3.3. Interfering contamination may occur when a sample containing low concentrations of analytes is analyzed immediately following a sample containing relatively high concentrations of analytes.

3.3.1. When contamination from one chromatography run to the next is suspected samples are reanalyzed employing the use of any necessary dilutions and solvent blanks to eliminate the problem.

3.4. Matrix interferences may be caused by contaminants that are co-extracted from the sample.

3.4.1. All samples are run on two chromatography columns which greatly minimize interference problems.

4. Safety

4.1. Refer to Laboratory Chemical Hygiene Plan.

5. Apparatus and Equipment

5.1. Sample Container: New Pre-certified 60 ml glass vials with a screw cap lined with Teflon.

5.2. Disposable Pasteur Pipets: glass, used for extract transfer.

5.3. Volumetric Flasks: 5 ml, 10 ml, 25 ml, and 100 ml.

5.4. pH paper: narrow range.

5.5. Micro Syringes: various sizes.

5.6. Autosampler vials: 2 ml with Teflon lined septum and screw top.

5.7. Graduated cylinders: 10 ml, and 50 ml

5.8. Balance: Analytical, capable of accurately weighing to 0.01g.

5.9. Balance: Analytical, capable of accurately weighing to the nearest 0.0001g.

5.10. Chlorine Test Strip: capable of testing residual chlorine up to 10 µg/ml.

5.11. Gas Chromatograph capable of temperature and flow programming, with a linearized electron capture detector (ECD), and a capillary column split/splitless injector.

5.11.1. TotalChrom software or equivalent; capable of temperature and flow programming of a split/splitless injection gas chromatograph.

5.11.2. Rtx 624 Polysiloxane capillary column (0.53 mm ID or 0.32 mm ID) or equivalent phase.

5.11.3. Rtx 5 Polysiloxane capillary column (0.53 mm ID or 0.32 mm ID), or equivalent phase, for confirmations.

5.12. Electron capture detector.

5.13. Detergent: Steris Labklenz or equivalent.

5.14. Autopipette Dispenser: Capable of dispensing 5.0 ml ± 0.04 ml of an organic solvent.

6. Reagents and Standards

6.1. Pentane: pesticide grade or equivalent.

6.2. Acetone: pesticide grade or equivalent.

6.3. Methanol: pesticide grade or equivalent.

6.4. Reagent Water: Purified water which does not contain any measureable quantities of target analytes or interfering compounds for each compound of interest (deionized, HPLC, Milli-Q water or equivalent). Milli-Q water has a resistivity of 18 MΩ·cm or greater @ 25° C and a TOC of 50µg/L or less.

6.5. Sodium Sulfate: granular, anhydrous, reagent grade suitable for pesticide analysis or equivalent.

6.5.1. Sodium Sulfate is baked for 4 hours at 450° C then stored in a glass container.

6.6. Potassium Phosphate: reagent grade, monobasic.

6.7. Sodium Phosphate: reagent grade, dibasic.

6.8. Ammonium Chloride: reagent grade.

6.9. Phosphate Buffer/Ammonium Chloride Preservative:

6.9.1. Prepare a dry homogenous mixture of 1% sodium phosphate / 99% potassium phosphate, by weight. Example: Combine 6 g sodium phosphate and 594 g potassium phosphate for total weight of 600 g into a large stainless steel bowl. Add 3.6 g of ammonium chloride to the 600 g mixture.

6.9.2. Completely cover mixture with methanol and stir. Pour off methanol and dispose of properly in the appropriate waste stream.

6.9.3. Next, completely cover mixture with acetone and stir. Pour off acetone and dispose of properly in the appropriate waste stream.

6.9.4. Finally, completely cover mixture with Pentane and stir. Pour off Pentane and dispose of properly in the appropriate waste stream. Repeat this step.

6.9.5. Spread mixture onto clean aluminum foil under a hood to dry overnight. Break up any clumps using a metal spoon and transfer to glass jars with Teflon lined caps for storage until use in sample collection vials.

6.10. Calibration Standard Primary Stock Solutions

6.10.1. *Primary Chloroform Stock Solution*: 200 µg/ml Chloroform stock made from 5000 µg/ml vendor stock.

Table 6.10.1. 1 – 551.1 Primary Chloroform Stock Solution in Acetone

Compound	Initial Concentration (µg/ml)	Aliquot (ml)	Final Concentration (µg/ml)
Chloroform	5000	1.0	200

Total Volume of Standard Aliquots	1.0 ml
Addition of Acetone to Standard Aliquots	24 ml
Final Volume of Spiking Stock Standard in Acetone	25 ml

6.10.2. *Primary Bromoform Stock Solution:* 200 µg/ml Bromoform stock made from 5000 µg/ml vendor stock.

Table 6.10.2. 1 – 551.1 Primary Bromoform Stock Solution in Acetone

Compound	Initial Concentration (µg/ml)	Aliquot (ml)	Final Concentration (µg/ml)
Bromoform	5000	1.0	200

Total Volume of Standard Aliquots	1.0 ml
Addition of Acetone to Standard Aliquots	24 ml
Final Volume of Spiking Stock Standard in Acetone	25 ml

6.10.3. *Primary Dichlorobromomethane and Dibromochloromethane Stock Solutions:* individual vendor stocks of these compounds at 200 µg/ml concentrations.

6.11. Spiking Solutions

6.11.1. *Analyte Spiking Solution*: appropriate aliquots of the 200 µg/ml primary stock standards are made up in Acetone to the final concentrations given in Table 6.11.1. 1.

Table 6.11.1. 1 – 551.1 Analyte Spiking Solution in Acetone

Compound	Initial Concentration (µg/ml)	Aliquot (ml)	Final Concentration (µg/ml)
Chloroform	200	2.5	5.0
Dichlorobromomethane	200	1.0	2.0
Dibromochloromethane	200	1.0	2.0
Bromoform	200	2.5	5.0

Total Volume of Standard Aliquots	7.0 ml
Addition of Acetone to Standard Aliquots	93 ml
Final Volume of Spiking Stock Standard in Acetone	100 ml

6.11.2. *Surrogate Spiking Solution*: 10 µg/ml surrogate spiking solution in Acetone made from a 2000 µg/ml Decafluorobiphenyl (DFBP) vendor stock.

Table 6.11.2. 1 – 551.1 Surrogate Spiking Solution in Acetone

Compound	Initial Concentration (µg/ml)	Aliquot (µl)	Final Concentration (µg/ml)
Decafluorobiphenyl (DFBP)	1000	500	10

Total Volume of Standard Aliquots	500 µl
Addition of Acetone to Standard Aliquots	49.50 ml
Final Volume of Surrogate Spiking Solution in Acetone	50 ml

6.11.3. *Internal Standard Spiking Solution*: 5 µg/ml internal standard spiking solution in Acetone made from a 100 µg/ml Dibromochloropropane (DBCP) vendor stock.

Table 6.11.3. 1 – 551.1 Internal Standard Spiking Solution in Acetone

Compound	Initial Concentration (µg/ml)	Aliquot (µl)	Final Concentration (µg/ml)
Dibromochloropropane (DBCP)	100	500	5.0

Total Volume of Standard Aliquots	500 µl
Addition of Acetone to Standard Aliquots	9.5 ml
Final Volume of Internal Standard Spiking Solution in Acetone	10 ml

6.11.4. *LPC Stock Solution #1*: appropriate aliquot of Lindane analyte vendor stocks at 100 µg/ml are made up in Pentane to the final concentration of 0.02 µg/ml as given in Table 6.11.4. 1.

Table 6.11.4. 1 – 551.1 LPC Stock Solution #1 (TCMX) in Pentane (1st Dilution)

Compound	Initial Concentration (µg/ml)	Aliquot (ml)	Final Concentration (µg/ml)
TCMX	100	0.02	0.02

Total Volume of Standard Aliquots	0.02 ml
Addition of Pentane to Standard Aliquots	99.98 ml
Final Volume of LPC Standard in Pentane	100 ml

6.11.5. *LPC Stock Solution #2*: appropriate aliquots of LPC analyte vendor stocks at 100 µg/ml and LPC Stock Solution #1 at 0.02 µg/ml (see 6.11.4.) are made up in Pentane to the final concentrations given in Table 6.11.5. 1.

Table 6.11.5. 1 – 551.1 LPC Stock Solution #2 in Pentane (2nd Dilution)

Compound	Initial Concentration (µg/ml)	Aliquot (ml)	Final Concentration (µg/ml)
TCMX	0.02	1.0	0.0002
HCCP	100	0.02	0.02
Dichlorobromomethane	100	0.03	0.03
Trichloroethylene	100	0.03	0.03

Total Volume of Standard Aliquots	1.08 ml
Addition of Pentane to Standard Aliquots	98.92 ml
Final Volume of LPC Standard in Pentane	100 ml

6.12. Initial Calibration Verification (ICV) Standard Stock Solutions

6.12.1. *ICV Second Source Stock Solutions*: individual vendor stocks of the four THM compounds at 100 µg/ml in Methanol.

6.12.2. *ICV Spiking Solution*: appropriate aliquots of the 100 µg/ml ICV stock standards (see 6.12.1. are made up in Acetone to the final concentrations given in Table 6.12.2. 1.

Table 6.12.2. 1 – 551.1 ICV Spiking Solution in Acetone

Compound	Initial Concentration (µg/ml)	Aliquot (ml)	Final Concentration (µg/ml)
Chloroform	100	0.50	5.0
Dichlorobromomethane	100	0.20	2.0
Dibromochloromethane	100	0.20	2.0
Bromoform	100	0.50	5.0

Total Volume of Standard Aliquots	1.4 ml
Addition of Acetone to Standard Aliquots	8.6 ml
Final Volume of Spiking Stock Standard in Acetone	10 ml

6.12.3. *ICV Surrogate Spiking Solution*: 10 µg/ml ICV surrogate vendor stock in Acetone made from 1000 µg/ml Decaflurobiphenyl second source vendor stock.

Table 6.12.3. 1 – 551.1 ICV Surrogate Spiking Solution in Acetone

Compound	Initial Concentration (µg/ml)	Aliquot (µl)	Final Concentration (µg/ml)
Decafluorobiphenyl (DFBP)	1000	100	10

Total Volume of Standard Aliquots	100 µl
Addition of Acetone to Standard Aliquots	9.9 ml
Final Volume of Surrogate Spiking Solution in Acetone	10 ml

6.13. Should alternate concentrations of vendor standards be required due to availability, adjust aliquots or final concentrations as needed to meet the final concentrations in Tables 6.10.1. 1 through 6.12.3. 1.

6.14. Calibration Curve Standards

6.14.1. Method 551.1 utilizes a procedural curve. All initial calibration, calibration verification, and continuing calibration standards are extracted in the same manner as samples, including the addition of preservation chemicals.

6.14.2. Prepare eight different concentrations equivalent to the concentration levels in Table 6.14.3. 1.

6.14.3. The calibration curve consists of the calibration standards at the following concentrations in µg/L:

Table 6.14.3. 1 551.1 Calibration Levels Concentrations

Compound	Level 1 (µg/L)	Level 2 (µg/L)	Level 3 (µg/L)	Level 4 (µg/L)	Level 5 (µg/L)	Level 6 (µg/L)	Level 7 (µg/L)	Level 8 (µg/L)
Chloroform	0.5	1	5	10	30	40	60	80
Dichlorobromomethane	0.2	0.4	2	4	12	16	24	32
Dibromochloromethane	0.2	0.4	2	4	12	16	24	32
Bromoform	0.5	1	5	10	30	40	60	80
Decafluorobiphenyl-ss	10	10	10	10	10	10	10	10
IS: DBCP	50	50	50	50	50	50	50	50

6.14.4. The calibration levels are made by dilution of appropriate aliquots of the Analyte Spiking Solution (Table 6.11.1. 1) into 50 ml of preserved reagent water followed by extraction. Surrogate and internal standards are added as directed in section 10.

Table 6.14.4. 1 551.1 Calibration Level Spike Volumes into 50 ml Water (Procedural Curve)

Level / Amount	Level 1 (µl)	Level 2 (µl)	Level 3 (µl)	Level 4 (µl)	Level 5 (µl)	Level 6 (µl)	Level 7 (µl)	Level 8 (µl)
Analyte Spiking Solution (Table see 6.11.1. 1)	5.00	10.0	50.0	100	300	400	600	800

6.14.5. The ICV is made at the same concentration as Level 6 of the calibration curve (400 µl aliquot of the ICV Spiking Solution (Table 6.12.2. 1) into 50 ml of preserved reagent water) and extracted as directed in section 10. , except that the ICV Surrogate Spiking Solution (Table 6.12.3. 1) is used in place of the calibration surrogate spiking solution.

7. Sample Collection

7.1. Drinking water samples for EPA Method 551.1 are collected in new pre-certified 60 ml glass vials with Teflon lined screw caps and preserved with 1.0 g of phosphate buffer/ammonium chloride prior to shipping. Two vials are required for every entry point plus two additional vials are sent for an MS and MSD for every 10th entry point or more frequently is needed.

7.1.1. A residual chlorine check is done in the field by the collector. The collector writes down the numerical value for residual chlorine in mg/L (ppm) on the sampling form.

7.1.2. The shipping and receiving staff log in the samples and enter the information for residual chlorine in the DNR_LAB Labworks field. The analyst prints or checks the backlog to determine samples to be analyzed.

7.1.3. The backlog report contains the residual chlorine concentration determined by the collector. If the residual chlorine measured by the collector is less than 8 mg/L, the 1.0 g phosphate buffer/ammonium chloride in the vial was sufficient to convert free chlorine to monochloramine.

7.1.4. If the collector reports 8 mg/L or more residual chlorine, the sample must be recollected.

7.1.5. If the residual chlorine is not listed on the sampling form then the analyst must test it by using chlorine test strips.

7.2. Samples are cooled to 0-6° C (not frozen) and extracted within 14 days after collection. Extracts must be analyzed within 14 days of extraction and stored at –10° C or less.

8. Calibrations

8.1. Initial Calibration

8.1.1. An eight-point calibration is performed for all single peak components (see Table 6.14.3. 1 for standard concentrations). The calibration system uses traceable certified standards. The calibration is an internal standard, procedural calibration.

8.1.1.1. The calibration may be an internal standard calibration based on mean relative response factors (see calculations 11.1. - 11.2.) with a relative standard deviation (%RSD) of < 10% (see calculation 11.3.) **or** a linear fit internal standard curve or a quadratic fit internal standard curve with a correlation coefficient (r^2) of ≥ 0.980 (or a coefficient of variation (r) ≥ 0.990).

8.1.1.2. First Order Linear Calibration using Least Squares Regression:

8.1.1.2.1. If linear regression is used, linearity through the origin cannot be assumed in a linear least squares fit. The instrument responses versus the concentrations of the standards for 8 points is determined using the instrument data analysis software and the regression will produce the slope and intercept terms for a linear equation. The regression calculation will generate a correlation coefficient (r) that is a measure of "goodness of fit" of the regression line to the data. A value of 1.0 is a perfect fit, 551.1 requires a correlation coefficient, $r \geq 0.990$ or $r^2 \geq 0.980$. See calculation 11.4.

8.1.1.2.2 Alternatively, second order quadratic fit may be used with an acceptable correlation of coefficient of $r \geq 0.990$ (or $r^2 \geq 0.980$). Note: quadratic fit will be calculated by chromatographic software. See calculation 11.5.

8.2. Retention Time Windows

8.2.1. Once per year or after major maintenance, retention time windows must be established. The width of the retention time windows for each analyte, surrogate, and internal standard is defined as ± 3 times the standard deviation of the mean absolute retention time established over the course of a day (24-hours) for all CCCs analyzed during that period. See calculation 11.6.

8.3. Calibration Verification

8.3.1. A second source calibration verification standard (ICV) must be analyzed after each initial calibration and at least once per quarter, even if the system has not been recalibrated. All analytes must be within ± 20% Drift of the expected values (see calculation 11.9.)

8.4. Record Keeping:

8.4.1. Documentation of an instrument calibration is reviewed for adherence to quality criteria and archived with the project records.

8.5. Daily Calibration Verification and Continuing Calibration:

8.5.1. A continuing calibration check standard (CCC) ensures the instrument target compound retention times and quantitation parameters meet method performance criteria. Preceding each analysis set, after every tenth sample analysis and after the final sample analysis, a calibration standard, alternating at two different concentration levels, should be analyzed as a continuing

calibration check to verify that instrument calibration accuracy does not exceed +/- 20% Drift from the expected value. See calculation 11.9.

8.5.1.1. If the CCC fails and can be immediately reanalyzed and meets the method criteria at this time then the samples that were analyzed immediately before this CCC are considered to be valid.

8.5.1.2. If the CCC fails and is not immediately reanalyzed, then the samples bracketed by the failed CCC must be reanalyzed with CCCs that pass method criteria.

8.5.1.3. If a valid CCC cannot be achieved then the instrument is considered to be out of calibration for the compounds in this method and the instrument must be recalibrated.

8.5.1.4. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated, and accepted or analyzed on an instrument that is meeting acceptable criteria.

8.5.2. An LPC (Laboratory Performance Check) standard is run at the beginning of each sample sequence prior to the analysis of samples to determine signal to baseline noise ratio and chromatographic performance.

Table 8.5.2. 1, LPC Requirements and Acceptance Criteria

Parameter	Analyte	Conc., µg/ml in Pentane	Acceptance Criteria
Instrument Sensitivity	TCMX	0.000200	Detection of Analyte: Signal to Noise > 3[1,2]
Chromatographic Performance	Hexachlorocyclopentadiene	0.0200	0.80 <PGF > 1.15[3,4]
Column Performance	Dichlorobromomethane Trichloroethylene	0.0300 0.0300	Resolution > 0.50[5,6]
	2 closest eluting peaks[7]	Varies[8]	Resolution > 0.50[6]

[1] *See 8.5.2.1.*
[2] *See calculation 11.12.1.*
[3] *See 8.5.2.2.*
[4] *See calculation 11.12.2.*
[5] *See 8.5.2.3.*
[6] *See calculation 11.12.3.*
[7] *See 8.5.2.4.*
[8] *8.5.2.4.1.*

8.5.2.1. Lindane signal to noise ratio must be greater than 3:1 (see calculation 11.12.1.).

8.5.2.2. Chromatographic Performance: the Peak Gaussian Factor (PGF) for Hexachloropentadiene must be between 0.80 and 1.15 (see calculation 11.12.2.).

8.5.2.3. Column Performance: Resolution between Dichlorobromomethane and Trichloroethylene must be greater than 0.5 (see calculation 11.12.3.).

8.5.2.4. Resolution between the two closest eluting target analytes, internal standard, or surrogate (may be different on different columns) must be greater than 0.5 (see calculation 11.12.3.). Selection of peaks is dependent upon column and chromatographic temperature and pressure programming for each instrument.

8.5.2.4.1. Select concentrations which provide responses similar to 0.03 µg/ml Dichlorobromomethane.

8.5.3. Daily Retention Time Update:

8.5.3.1. Retention Times (RT) are updated once per 24 hour period when GC analyses are performed. The first CCC is processed using Totalchrom or equivalent software. The new RTs are saved in a copy of the processing method used for analyzing this batch of samples. To the existing processing method an extension is added by using – Month-Day-Year. Then hard copies of the calibration parameters are added to the data package for that batch of samples. (**NOTE**: If an analytical sequence is stopped for any reason longer than a typical work shift a new retention time update is necessary for the next sequence.)

8.6. Sample Concentration

8.6.1. Sample results are expressed in µg/L.

8.6.2. If an analyte response is calibrated by Mean Relative Response Factor, ($\overline{RRF}$) (see calculation 11.2.), the chromatographic software calculates the sample concentrations per calculation 11.10.

8.6.3. If an analyte response is calibrated by linear regression, the chromatographic software calculates the sample concentrations per calculation 11.11.

8.6.4. If an analyte response is calibrated by quadratic fit, the chromatographic software calculates the sample concentrations per calculation 11.11.

8.6.5. If the initial sample volume is less than 50 ml then volume from the duplicate sample container may be combined to achieve 50 ml. If a duplicate is not available then the sample is recollected.

9. Quality Control

9.1. Refer to Table 14.1. 1 for Reporting Limits (RLs), Appendix A, Table A.1 for Quality Assurance criteria and Table 14.1. 2 for Quality Control procedures associated with this method.

9.2. A Method Detection Limit Study for all analytes must be performed once per year. See SOP reference 13.6.

9.3. See SOP reference 13.2. for training and certification procedures.

9.4. See SOP reference 13.3. for control charting procedures.

9.5. Control Limits:

9.5.1. The default control limits from EPA Method 551.1 are 80 - 120% recovery for LCS recoveries. These are the same as the %Drift for CCCs as LCSs and CCCs are essentially the same per Method 551.1. However, the EPD Laboratory extracts LCS and LCSD QC samples which are separate from the CCCs. The EPD Laboratory applies LCS recovery limits to LCSDs. Note, unless specified

by method, the EPD Laboratory does not validate batch quality based on LCSD recoveries.

9.5.2. By default, the EPD Laboratory sets static LCS/LCSD precision control limits to be 0 - 20% RPD.

9.5.3. Ten percent of all routine samples must be spiked. Also, 10% of all samples must be run in duplicate. Analysis of MS/MSD pairs as directed in Section 9.6.2. below results in 10% spike duplicates, meeting the method criteria. EPA Method 551.1 requires recovery control limits of 80 - 120% for matrix spike analytes. The EPD Laboratory applies MS recovery limits to MSDs.

9.5.4. The EPD Laboratory sets static MS/MSD precision to be 0 - 20% RPD.

9.5.5. The default control limits for surrogates from EPA Method 551.1 are 80 - 120% recovery. These limits are static, i.e. not adjusted through the use of control charts.

Note: Analysts must use the control limits presented in Appendix A, Table A.1. Those limits cannot exceed the default limits in Table 9.5.5. 1.

Table 9.5.5. 1 - Default QC Limits*

	Analyte	Default LCL % Recovery	Default UCL % Recovery	Default Precision %RPD
LCS/LCSD	Chloroform	80	120	20
	Dichlorobromomethane	80	120	20
	Dibromochloromethane	80	120	20
	Bromoform	80	120	20
Surrogate	DFBP	80 (8.0 µg/L)	120 (12 µg/L)	NA
MS/MSD	Cloroform	80	120	20
	Dichlorobromomethane	80	120	20
	Dibromochloromethane	80	120	20
	Bromoform	80	120	20

9.5.6. See the EPD Laboratory Procedures for Control Charting and Control and Control Limits SOP (SOP reference 13.3.) for further details.

9.6. Batching:

9.6.1. Batch samples in groups of 1 to 20 samples.

9.6.2. For batches of 1 to 10 routine samples, one MS/MSD pair must be spiked. For batches of 11 to 20 routine samples, a second MS/MSD pair must be spiked using different samples for each pair.

9.6.3. For batches of 1to 10 routine samples, one Field Duplicate must be extracted and analyzed. For batches 11 to 20 routine samples, a second Field Duplicate must be extracted and analyzed using different samples for each Duplicate.

9.7. Assessing the Internal Standard Response:

9.7.1. The response of the internal standard must be monitored for all samples and standards analyzed by this method during each analysis day. The internal standard response should not deviate from the mean internal standard response of the calibration curve standards by more than 20% on the quantitative column/detector. If > 20% deviation occurs with an individual extract, a second aliquot of that extract spiked with the appropriate amount of internal standard is analyzed. If this aliquot produces an acceptable internal standard response, report results for this aliquot. If the deviation is greater than 20% for this aliquot the sample is either re-extracted if within method hold times, or recollected. A reanalyzed sample aliquot should be followed by a CCC to validate the calibration of the instrument. If this CCC fails after two attempts (see 8.5.1.) the IS acceptance criterion for quantitative column/detector, the instrument is considered to be out of calibration. A new calibration must be performed and validated before samples may be analyzed using this instrument.

9.8. Method Detection Limit Study (MDL):

9.8.1. MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.

9.8.2. The actual MDL varies depending on instrument and matrix.

9.8.3. The MDL must be determined annually for each instrument prior to results being reported for that instrument. The MDL determined for each compound must be less than the reporting limit for that compound.

9.8.4. An MDL study may be done two different ways. The two different ways are considered and initial MDL study and a continuous MDL study. Both ways will be explained below.

9.9. Initial MDL study:

9.9.1. An initial MDL study may occur when a new instrument is brought online, changes to the method (which affect the compound of interest's peak area), and lastly major instrument repairs have been made.

9.9.2. An initial MDL study will consist of the following operating parameters, 7 MDL samples and 7 MDL blanks. The 7 MDL samples study is performed by preparing 7 spiked vials, MDLSpike, spiked at the lowest calibration point of the curve, and preparing 7 clean blank vials filled with DI water, MDLBlank. These 7 sets of spiked and blank vial "pairs" are analyzed over 3 separate days, there may or may not be a non-analysis day between each of the 3 days. A total of 14 vials are prepared, 7 spiked and 7 blanks.

9.10. Continuous MDL study:

9.10.1. A Continuous MDL study is preferred over the initial except in a few cases. For a continuous MDL study to be used on an instrument it must have a minimum of 7 MDL samples and 7 MDL blanks extracted over the course of multiple batches over a year. It is required that at a minimum 2 MDL samples and 2 MDL blanks must be ran per quarter per instrument. If this requirement is not met, then the initial MDL study must be performed for that instrument. (See section 9.9.2 for requirements.)

9.10.2. A continuous format MDL study is performed where one vial is spiked as an MDLSpike, at the lowest point of the calibration curve and analyzed with every batch of samples along with the method blank vial as an MDLBlank.

9.10.3. The results of the MDLBlank will be entered into Labworks using the Method Blank test code, $B_551T. The MDLSpike result will be entered using the $ML551T. The MDL Spiked Amount will be entered into the test code $MA551T. The instrument used for the MDL and Blank analysis will be selected using the test code INSTR-551T.

9.10.4. MDL studies must be pulled on a yearly basis or an initial MDL study must be performed before the current MDLs for the instrument expire.

10. Procedure

10.1. Remove sample bottles, standards and reagents, surrogate solution, QC spike solution and internal standard solution from cold storage and allow them to equilibrate to room temperature prior to sample preparation and/or analysis.

10.2. With each batch of samples extracted, prepare a volume comparison (reference) vial. Measure 50 ml of reagent water in a graduated cylinder, pour into a new, pre-preserved 60 ml vial, cap, and mark the volume on the vial with a permanent marker. *Note: This vial is to also be used as the batch method Blank in section 10.3.*

10.2.1. The analyst that creates the reference vial will adjust sample volumes to 50 ml by pouring out a portion of each sample and comparing it to the mark on the reference vial. During adjustment, a small portion of the sample is poured into the sample's vial cap. If necessary, this portion can be used to adjust the final sample volume. Following volume adjustment, test the pH of the remaining portion in the cap with pH paper to verify the sample pH is within a range of 4.5 to 5.5. Discard the contents of the cap after pH check. *Do not measure the pH by inserting the pH paper directly into the sample in the 60 ml vial. Do not use the portion in the cap to adjust the volume in the vial after that portion has been tested for pH.*

10.3. Sample Extraction:

10.3.1. A batch consists of a Blank, LCS, LCSD, one or two MS and MSD pairs, an MDL, and one or more Field Duplicates, and up to 20 samples.

10.3.1.1. Field samples, MS and MSD volumes are adjusted and pH values checked per section 10.2.1. prior to proceeding to the next step.

10.3.2. LCS, LCSD, Blank, and MDL's are made up in pre-preserved 60 ml glass vials (same as sample collection vials).

10.3.2.1. Spike 2 aliquots of reagent water for the LCS and LSCD with 300 µl of the Analyte Spiking Solution (see 6.11.1.).

10.3.2.2. Spike two aliquots of the same sample for the MS and MSD with 300 µl of the Analyte Spiking Solution (see 6.11.1.) for the calculation of precision and accuracy.

10.3.2.3. At least every 2 weeks, spike two aliquots of reagent water with the Analyte Spiking Solution (see 6.11.1.) at two different levels for CCCs (see Table 6.11.1. 1).

10.3.3. Add 0.05 ml (or 50 µl) of Surrogate Spiking Solution (see 6.11.2.) to all samples, blanks, and QC samples. Cap and mix by slowly and carefully inverting the sample vial 2 times with minimal shaking. Equivalent to 10 µg/L DFBP in a 50 ml sample.

10.3.4. Add exactly 5.0 ml of Pentane.

10.3.4.1. The 5.0 ml of Pentane aliquot may be measured with:

10.3.4.1.1. A bottle mounted autopipette. The autopipette must be calibrated a minimum of once each day of use.

10.3.4.1.2. If a bottle mounted autopipette is not available, an appropriate class A volumetric glassware may be used (pipette, cylinder, flask, etc.).

10.3.5. Add 20 g of Sodium sulfate, Na_2SO_4, to the sample vial.

10.3.6. Recap, agitate, and place vial in a secure horizontal position to prevent the Na_2SO_4 from solidifying at the bottom of the sample while other samples are being prepared for extraction.

10.3.7. Extract the sample mixture by vigorously and consistently shaking the vial by hand for 4 minutes.

10.3.8. Allow the Pentane and water phase to separate (approximately 2 minutes).

10.3.9. Transfer exactly 1 ml of Pentane phase (avoid water) into an autosampler vial. Transfer a second 2 ml aliquot of the Pentane phase to an autosampler vial for use if needed as a spare extract of the same sample.

10.3.10. Add 0.01 ml (10 µl) of the Internal Standard Spiking Solution (see 6.11.3.) to each autosampler vial (the vials containing 1 ml of extract), cap and gently invert to mix. Note: Amount of internal standard, DBCP, in the 1 ml extract is equal to 50 µg/L. It is not necessary to spike the second spare vial with the internal standard at this point (spike with IS only if vial is needed).

10.3.11. When not in use, store extracts in a freezer at -10° C or cooler, protected from light. Dispose in the appropriate waste stream (see SOP reference 12.1.) when the extract holding time has expired.

10.4. Sample Analysis:

10.4.1. Analyze all sample extracts and QC using a Gas Chromatograph equipped with an Electron Capture Detector.

10.4.2. A detect is considered to be positive if the quantitation amount is greater than the reporting limit for that compound. When a positive detect is found then the

sample is run on a second dissimilar confirmation column. The blanks, LCS, and MS values are based on the primary column. If the results from this column are out of acceptable range due to matrix interferences or other problems, the results may be reported from the confirmatory column provided calibration criteria are met.

10.4.3. A single peak analyte is identified as positive if detected within its appropriate retention time window on both columns.

10.5. Dilutions:

10.5.1. Upon analysis of the extract, if a target compound response is greater than that of the highest standard of the calibration curve, the sample must be diluted with the final extraction solvent (Pentane) so that, upon analyzing the dilution (in a valid analysis sequence), the target response is between the lowest concentration standard (or the reporting limit, whichever is higher) and the highest concentration standard.

11. Calculations

11.1. Relative Response Factor (*RRF*):

11.1.1. Calculate the relative response factors (*RRF*) for each target compound relative to the appropriate internal standard (i.e., standard with the nearest retention time) using the following equation:

$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

11.1.2. Where:

RRF = Relative response factor
A_x = Area of the peak for the compound to be measured
A_{is} = Area of the peak for the internal standard
C_{is} = Concentration of internal standard spiking mixture
C_x = Concentration of the compound in the calibration standard

11.1.3. NOTE: The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC analyses is the same from run to run, and that the volume of sample extract and QC sample extract introduced into the GC is the same for each analysis. C_{is} and C_x must be in the same units.

11.2. Mean Relative Response Factor ($\overline{RRF}$):

11.2.1. Calculate the mean RRF ($\overline{RRF}$) for each compound by averaging the values obtained at the five concentrations using the following equation:

$$\overline{RRF} = \sum_{i=1}^{n} \frac{x_i}{n}$$

11.2.2. Where:

$\overline{RRF}$ = Mean relative response factor
x_i = RRF of the compound
n = Number of values

11.3. Percent Relative Standard Deviation (*%RSD*):

11.3.1. Using the RRFs from the initial calibration, calculate the *%RSD* for all target compounds using the following equations:

$$\%RSD = \frac{\delta_{n-1}}{\overline{RRF}} \; x \; 100$$

and

$$\delta_{n-1} = \sqrt{\sum_{i=1}^{n} \frac{(RRF_i - \overline{RRF})^2}{n-1}}$$

11.3.2. Where:

δ_{n-1} = Sample standard deviation of initial response factors (per compound)
RRF_i = Relative response factor at a concentration level
$\overline{RRF}$ = Mean of initial relative response factors (per compound)
n = Number of values

11.4. Linear Regression Response Equation

$$Y = ax + b$$

This rearranges to:

$$x = Y - b/a$$

11.4.1. Where:

Y = Instrument response
a = Slope of the line
b = Intercept
x = Concentration in the extract or standard

11.4.2. For an internal standard calibration:

$$\frac{A_x}{A_{is}} = a\left(\frac{C_x}{C_{is}}\right) + b$$

11.4.3. Where:

A_x = Area of the peak for the compound to be measured

A_{is} = Area of the peak for the internal standard
C_{is} = Concentration of internal standard spiking mixture
C_x = Concentration of the compound in the calibration standard
a = Slope of the line
b = Intercept

11.4.4. This rearranges to:

$$C_x = C_{is}\left(\left(\frac{A_x}{A_{is}}\right) - \left(\frac{b}{a}\right)\right)$$

11.5. Second Order Quadratic Fit Equation

11.5.1. $Y = ax^2 + bx + c$

11.5.2. Where:
Y = Instrument response
a = Slope of the line
b = Intercept
c = constant
x = Concentration in the extract or standard

11.5.3. Subtract Y from c to get modified equation $0 = ax^2 + bx + c$

11.5.4. Solve for x using the quadratic formula:

$$x = \frac{-b \pm \sqrt{b^2 - 4ac}}{2a}$$

11.5.5. A positive and negative value will be generated. Use positive value.

11.5.6. For an internal standard calibration:

$$\frac{A_x}{A_{is}} = a\left(\frac{C_x}{C_{is}}\right) + b$$

11.5.7. Where:
A_x = Area of the peak for the compound to be measured
A_{is} = Area of the peak for the internal standard
C_{is} = Concentration of internal standard spiking mixture
C_x = Concentration of the compound in the calibration standard
a = Slope of the line
b = Intercept

11.5.8. This rearranges to:

$$C_x = C_{is}\left(\left(\frac{A_x}{A_{is}}\right) - \left(\frac{b}{a}\right)\right)$$

11.6. Relative Retention Times (*RRT*):

11.6.1. Calculate the *RRT*s for each target compound over the initial calibration range using the following equation:

$$RRT = \frac{RT_c}{RT_{IS}}$$

11.6.2. Where:

RT_c = Retention time of the target compound
RT_{IS} = Retention time of the internal standard

11.7. Mean Relative Retention Time:

$$\overline{RRT} = \sum \frac{RRT}{n}$$

11.7.1. Where:

$\overline{RRT}$ = Mean retention time for target compound
RRT = Retention time for the target compound
n = number of values

11.8. Dilution Factor

11.8.1. When dilutions are required, sample extracts are diluted and a dilution factor (DF) calculated. If the sample volume is not exactly 50 ml, that must also be factored into the DF as follows:

$$DF = \frac{V_f}{V_{aliquot}} * \frac{V_s}{V_{ideal}}$$

11.8.2. Where:

DF = Dilution factor
V_f = Final volume after dilution
$V_{aliquot}$ = Amount of extract diluted
V_s = Actual sample volume
V_{ideal} = Ideal sample volume of 50 ml

11.8.3. If there is no dilution performed and a sample of exactly 50 ml is used, DF = 1.

11.9. Percent Drift, %Drift

$$\%\text{Drift} = \frac{(\text{Concentration}_{\text{Calculated}} - \text{Concentration}_{\text{Expected}})}{\text{Concentration}_{\text{Expected}}} * 100$$

11.9.1. Where:
Concentration $_{Calculated}$ = Concentration calculated from result
Concentration $_{Expected}$ = Theoretical concentration of the standard

11.10. Sample Concentration Calculation for Mean Relative Response Factor Calibrations ($\overline{RRF}$):

$$C_x = \frac{A_x C_{is} DF}{A_{is} \overline{RRF}}$$

11.10.1. Where:
C_x = Compound concentration
A_x = Area of the compound to be measured
A_{is} = Area of the internal standard
C_{is} = Concentration of the internal standard spiking mixture,
$\overline{RRF}$ = Mean relative response factor from the initial calibration
DF = Dilution factor. If 50 ml sample and no dilution is performed, DF = 1

11.10.2. NOTE: The equation above is valid assuming a consistent volume and concentration of the internal standard spiking solution throughout calibration and sample analysis.

11.11. Alternate Sample Concentration Calculation using linear regression:

11.11.1. Utilizing linear regression calculation 11.4.4. sample concentrations for linear regression internal standard calibrations are calculated as :

$$C_x = C_{is}\left(\frac{\left(\frac{A_x}{A_{is}}\right) - b}{a}\right) * DF$$

11.11.2. Where:
A_x = Area of the peak for the compound to be measured
A_{is} = Area of the peak for the internal standard
C_{is} = Concentration of internal standard spiking mixture
C_x = Concentration of the compound in the calibration standard
a = Slope of the line
b = Intercept
DF = Dilution factor. If 50 ml sample and no dilution is performed, DF = 1

11.11.3. This rearranges to:

$$C_x = C_{is}\left(\left(\frac{A_x}{A_{is}}\right) - \left(\frac{b}{a}\right)\right)*\text{DF}$$

11.12. LPC Calculations:

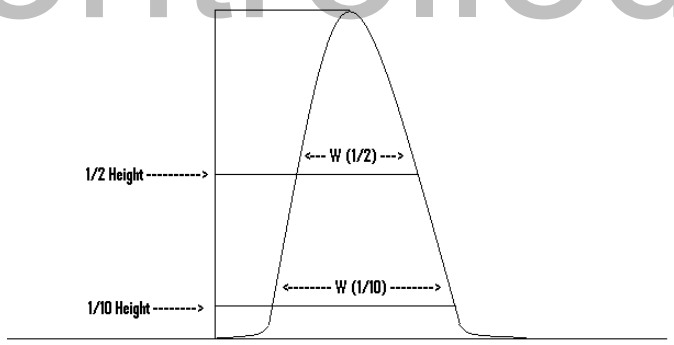
11.12.1. Sensitivity:

11.12.1.1. Instrument sensitivity is determined by comparing the LPC peak height of the weakest responding analyte, TCMX, in the LPC to signal noise. The height of this peak must be 3 times greater than the largest noise peak.

$$\frac{S}{N} = \frac{\mu V_{Analyte}}{\mu V_{Noise}}$$

11.12.2. Chromatographic Performance (Peak Gausian Factor – PGF):

11.12.2.1. The PGF is calculated on Hexachlorocyclopentadiene in the LPC chromatogram. The PGF is equal to 1.83 times the ratio of that peak's half-height width and the tenth height peak width:



Peak Gaussian Factor - Measuring width at half peak height - W (1/2) and one tenth height - W (1/10)

11.12.2.2. $PGF = 1.83\left(\frac{W_{1/2}}{W_{10}}\right)$

11.12.2.3. Where:

PGF = Peak Gaussian Factor

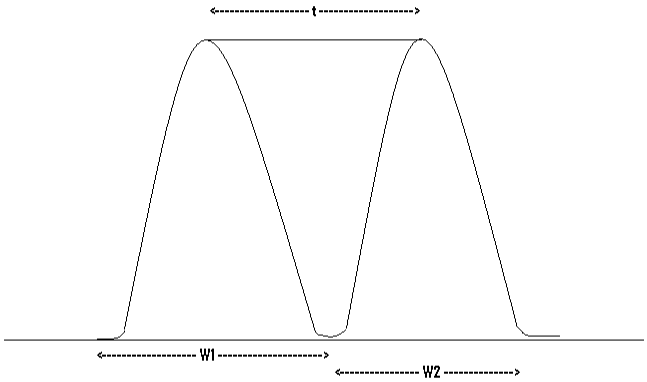
$W_{½}$ = the peak width at ½ height from base in seconds

$W_{1/10}$ = the peak width at $^1/_{10}$ height from base in seconds

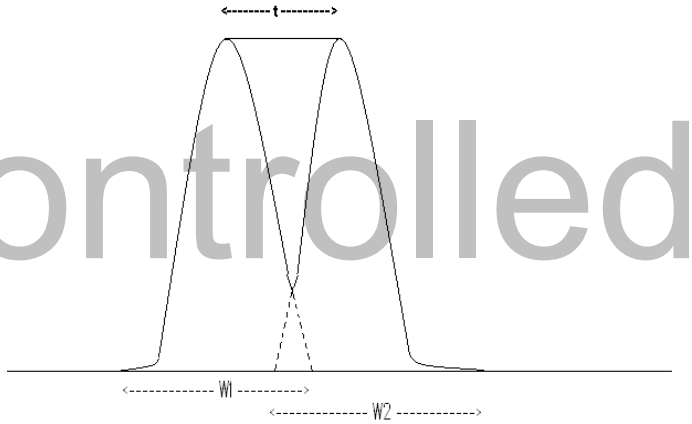
11.12.2.4.

11.12.3. Column Performance (Resolution):

11.12.3.1. Column performance is determined by calculating the resolution factor between Dichlorobromomethane and Trichloroethylene, in the LPC.



Column Performance - Peak Resolution for Resolved Peaks



Column Performance - Peak Resolution for Overlapping Peaks

11.12.3.2. Resolution between two peaks is determined by dividing the difference in the peak apex retention times by the average of the widths of the two peaks at the baseline. If the peaks overlap as in the second example above, estimate the peak widths as shown:

$$R = \frac{t}{\left(\frac{W1 + W2}{2}\right)}$$

11.12.3.3. Where:

R = Resolution between two peaks
t = the difference in elution times between the two peaks
W1, W2 = the peak widths at the baseline of the two peaks

11.12.4 LPCs must be measured with a ruler or other straight edge to mark time values on the chromatograms and calculated manually using equations from section 11.11.

Alternatively, Total Chrom or equivalent chromatography software or the values on the Total Chrom LPC System Suitability Report may be used to calculate LPC results.

11.12.5 RPD Equation for Original Sample and Field Duplicate

$$RPD = \frac{(FD1-FD2)}{1/2(FD1+FD2)} X100$$

11.12.5.1 Where:

FD1 = Original Sample

FD2 = Field Duplicate

12. Waste Management

12.1. See GA EPD Laboratory SOP-EPD Laboratory Waste Management Standard Operating procedures.

13. References

13.1. EPA/600/4-88-039 - EPA Method 551.1, Revision 1.0, 1995

13.2. GA EPD Laboratory SOP's- Initial Demonstration of Capability SOP 6-001, online revision and/or Continuing Demonstration of Capability SOP 6-002, online revision.

13.3. GA EPD Laboratory SOP- EPD Laboratory Procedures for Control Charting and Control and Control Limits SOP, SOP 6-025, online revision.

13.4. GA EPD Laboratory SOP- EPD Laboratory Waste Management SOP, SOP 6-015, online revision.

13.5. Manual for the Certification of Laboratories Analyzing Drinking Water, EPA/815-R-05-004, January 2005

13.6. GA EPD Laboratory SOP- Determination of Method Detection Limit, Method Detection Limit SOP 6-007, online revision.

13.7. GA EPD Laboratory Quality Assurance Plan, online revision.

13.8. GA EPD Laboratory Safety/Chemical Hygiene Plan & Fire Safety Plan, online revision.

14. Practical Reporting Limits (RLs), Precision and Accuracy Criteria, and Quality Control Approach

14.1. Refer to Appendix A, Table A.1 for precision and accuracy criteria.

Table 14.1. 1 - RLs for EPA Method 551.1

Parameter/Method	Analyte	Matrix (WATER)	
		RL	Unit
551.1	Chloroform	1.0	µg/l
	Dichlorobromomethane	1.0	µg/l
	Dibromochloromethane	1.0	µg/l
	Bromoform	1.0	µg/l

Table 14.1. 2 - Summary of Calibration and QC Procedures for EPA Method 551.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA Method 551.1	Trihalomethanes	Eight point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD for all analytes ≤10% or use linear least squares regression r ≥0.990 ($r^2 \geq 0.980$)	Correct problem then repeat initial calibration	
		Second-source Calibration verification (ICV)	Once per initial calibration	All analytes within ±20% of expected value	Correct problem then repeat initial calibration	
		Acetone Blank	Once per initial calibration	No analytes detected >RL	Correct problem then repeat initial calibration	
		Retention time window calculated for each analyte	Once per year or after major maintenance that would affect RTs.	±3 times standard deviation for each analyte retention time from study conducted over the course of a day		
		Retention time window update	Must be done every analytical sequence	First CCC of each sequence and the first CCC of each 24 hour period.	Correct problem then reanalyze all samples since the last retention time check	
		Calibration verification (CCC)	Beginning each analysis seq.,prior to the analysis of samples, after every 10 samples, and at the end of the analysis sequence	All analytes within ±20% of expected value.	Rerun CCC once. If passes use sample results. If does not pass correct problem repeat initial calibration verification, if necessary and reanalyze all samples since last successful calibration verification	
		Laboratory Performance Check (LPC)	Prior to the analysis of each sample sequence	See 8.5.2. for LPC criteria	Correct problem and reanalyze	

Table 14.1. 2 - Summary of Calibration and QC Procedures for EPA Method 551.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA Method 551.1	Trihalomethanes	IDC- Demonstrate ability to generate acceptable accuracy and precision using four replicate analyzes of a QC check sample + a blind & a blank. Analyst must also produce a passing MDL study with 7 MDL spikes and 7 MDL blanks	Once per analyst	QC acceptance criteria, see Appendix A for recoveries but RSD of 15% or less. **See section 9.8 for MDL requirements**	Locate and fix problem then rerun or reextract demonstration for those analytes that did not meet criteria	
		CDC – Continuing Demonstration of Capability	Required every Six Months after IDC for each analyst	See **Appendix A, Table A.l**	Locate and fix problem then rerun or reextract demonstration for those analytes that did not meet criteria	
		Method Blank Solvent Blank	One per analytical batch	No analytes detected > RL.	Analyze second extract aliquot. If this does not pass correct problem then re-analyze or re-extract the blank, LCS/LCSD and all samples in the affected batch.	
		LCS/LCSD for all analytes	One LCS/LCSD per analytical batch	QC acceptance criteria, see Appendix A	If an LCS/LCSD fail, it may be reran at least 24 hours from the original run or up to 12 hours from the end of the sequence. Then if the rerun of the LCS/LCSD result with a failure then all samples associated with the batch must be re-extracted.	

Table 14.1. 2 - Summary of Calibration and QC Procedures for EPA Method 551.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA Method 551.1	Trihalomethanes	Surrogate spike	Every sample, spiked sample, standard and method blank	QC acceptance criteria, see Appendix A	Analyze second extract aliquot. If this does not pass, correct problem then re-extract and re-analyze sample	
		Internal Standard	Every sample, spiked sample, standard, and method blank	± 20% deviation from expected value	Reanalyze extract. If it fails the second analysis, re-extract sample and reanalyze	
		Matrix Spike (MS/MSD)	Minimum frequency of 10% samples. One MS/MSD per batch of 1-10 samples. Two MS/MSD per batch of 11-20 samples.	QC acceptance criteria, see Appendix A	Flag QC sample report.	
		Second-column confirmation	100% for all positive results	If used for quantitation same as for initial or primary column analysis	Same as for initial or primary column analysis	
		MDL study	Once per year or after major maintenance of the instrument	All Spiked MDLs must have a value greater than 0. Minimum Detection Limits established shall be < the RLs in Table 14.1	Re-do MDL Study	None
		MDL analysis	Once per batch or as needed to acquire data points per SOP 6-007, online revision	All Spiked MDLs must have a value greater than 0. All other QC in the MDL blank and MDL sample (i.e. Surrogate Spike or Internal Standard, etc. if included) must meet established criteria	Correct problem and re-run the MDL sample or MDL blank once and initiate a corrective action. If the re-run fails a second time, do not use MDL data. Update corrective action, and use associated sample data	None
		Results reported between MDL & RL	none	none	None	
		Quarterly ICV	Once per Quarter	All analytes within ± 20% of expected value	Correct problem then repeat calibration	

Table 14.1. 2 - Summary of Calibration and QC Procedures for EPA Method 551.1

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA Method 551.1	Trihalomethanes	Residual Chlorine Check	Whenever needed. If collector does not check residual chlorine.	Must be checked for every sample.	Check residual chlorine levels and add information to extraction sheet.	
		Field Duplicate (FD)	Minimum frequency of 10% of samples. One duplicate per batch of 1 to 10 samples. Two duplicates per batch of 11 to 20 samples.	RPD for all analytes < 20%	Do not reanalyze. Generate Corrective Action and comment sample.	

15. Associated Labworks Test Codes

15.1. Parent Test Code

15.1.1. $551T – Analysis results

15.2. Extraction Test Code

15.2.1. 551TE – 60 mL clear vial – Liquid/ Liquid extraction

15.3. QC Test Codes

15.3.1. $B_551T – Extraction Blank Results

15.3.2. $LA551T – LCS/LCSD Spike Amount

15.3.3. $LS551T – LCS Results

15.3.4. $LS551T – LCSD Results

15.3.5. $LR551T – LCS Percent Recovery

15.3.6. $L2551T – LCSD Percent Recovery

15.3.7. $LP551T – LCS/LCSD Precision

15.3.8. $A_551T – MS/MSD Spike Amount

15.3.9. $S_551T – MS Results

15.3.10. $D_551T – MSD Results

15.3.11. $R_551T – MS Percent Recovery

15.3.12. $RD551T – MS Percent Recovery

15.3.13. $P_551T – MS/MSD Precision

15.3.14. $MA551T – MDL Spike Amount

15.3.15. $ML551T – MDL Results

15.3.16. INSTR-551T – Instrument associated with batch

Appendix A, Table A.1 – Quality Assurance Criteria for EPA Method 551.1

QC Type	Analyte	Accuracy (%R) LCL - UCL	Precision (%RPD)
LCS/LCSD*	Chloroform	82 - 119	20
	Dichlorobromomethane	83 - 118	20
	Dibromochloromethane	84 - 118	20
	Bromoform	82 - 120	20
Surrogate**	Decafluorobiphenyl	80 - 120	NA
	Decafluorobiphenyl (as µg/l)	8.0 - 12	NA
MS/MSD***	All Analytes	80 - 120	20

*LCS/LCSD recovery based on control charts of data collected from 12/31/2018 to 01/01/2021. The EPD sets a static LCS/LCSD precision of 0-20% RPD.

**EPA Method 551.1 sets a static recovery range of 80-120% for the surrogate.

***EPA Method 551.1 sets a static limit of 80-120% recovery for Matrix Spike analytes. EPA Method 551.1 sets a static range of 0-20% RPD for MS/MSD pairs.

Updates:

Appendix A added. Updated for online revision.