15 March 2023

Mike McCrum
Project Manager
Bureau of Land Management, Alaska State Office
222 West 7th Ave #13
Anchorage, Alaska 99513
Email: mmcrum@blm.gov

SUBJECT: 2023 Groundwater and Surface Water Baseline Monitoring Work Plan Addendum at Red Devil Mine, Alaska

Dear Mr. Euler and Mr. McCrum,

This letter presents the *2023 Groundwater and Surface Water Baseline Monitoring Work Plan Addendum* (*2023 Work Plan Addendum*) to the *2019 Groundwater and Surface Water Baseline Monitoring Work Plan* (*2019 Baseline Monitoring Work Plan*; Bureau of Land Management [BLM], 2019). The *2019 Baseline Monitoring Work Plan* included a Field Sampling Plan (FSP) and Quality Assurance Project Plan (QAPP) and established the field and analytical methods and procedures, data quality objectives, and the quality assurance/quality control measures to be employed during the spring and fall baseline monitoring of groundwater and surface water at Red Devil Mine, Alaska.

The *2023 Work Plan Addendum* describes the main elements of the 2023 spring and fall baseline monitoring and any relevant updates to the *2019 Baseline Monitoring Work Plan* (BLM, 2019). In addition, updated laboratory standard operating procedures (SOPs) and a Health and Safety Plan (HASP) have been provided as appendices.

Sundance Consulting, Inc. (Sundance), appreciates the opportunity to continue this project. If you have any questions or concerns regarding the *2023 Work Plan Addendum*, please contact me at (505) 321-4515 or crust@sundance-inc.net .

Sincerely,

Colleen Rust, PMP, PG-WA, AZ, AK, CPG, Project Manager
Sundance Consulting Inc.

Enclosures

2023 Groundwater and Surface Water Baseline Monitoring Work Plan Addendum
Appendix A. Laboratory Standard Operating Procedures
Appendix B. Health and Safety Plan

1.0 PROJECT SCOPE AND OBJECTIVES

Sundance Consulting, Inc. (Sundance), will complete bi-annual spring and fall groundwater and surface water baseline monitoring at Red Devil Mine in two different sampling events: one in late May to early June and the second in early September. The spring sampling event will be conducted shortly after spring breakup when surface runoff and water table elevation is at or near the annual maximum. The fall sampling event will be conducted during early fall when stream flow is typically approaching a minimum prior to freeze up and water table elevations begin a seasonal decline.

1.1 Groundwater Monitoring

Sundance will complete the following scope of work:

1. Mobilize to the project site all personnel, equipment, and material needed to perform monitoring activities.
2. Measure depth to static water level at all monitoring wells listed in Table 1, including those monitoring wells that are not scheduled for sampling collection. Depth to water measurements will be completed for all monitoring wells within a 24-hour period during both the spring and fall sampling events. Data will also be downloaded from transducers installed in the monitoring wells designated in Table 1 during the same 24-hour period.
3. Collect groundwater and surface water samples following methods and protocols defined in the *2019 Groundwater and Surface Water Baseline Monitoring Work Plan* (*2019 Baseline Monitoring Work Plan*) (Bureau of Land Management [BLM], 2019) and this 2*023 Groundwater and Surface Water Baseline Monitoring Work Plan Addendum* (*2023 Work Plan Addendum*). Note that some monitoring wells in Table 1 will not be sampled. Surface water sampling stations along Red Devil Creek are listed in Table 2.
4. Transport all samples to the laboratory under chain of custody such that laboratory analysis is performed within holding times prescribed for all analytical methods.
5. Coordinate with the BLM at least once a week while data collection is ongoing.
6. Demobilize personnel, equipment, and material from the site at completion of field data collection period.

Table 1. Baseline Groundwater Monitoring Well Summary

Monitoring Well ID	Groundwater Sample	Bladder Pump	Transducer	Northing	Easting
MW01		X		6848472.892	588897.3617
MW03				6848581.065	588979.8349
MW04				6848564.133	588907.3826
MW06	X	X		6848656.180	588995.1024
MW07				6848393.712	588891.9008
MW08				6848354.155	588997.7221
MW09	X	X		6848491.702	588970.2143
MW10	X	X		6848470.815	588950.2887

Monitoring Well ID	Groundwater Sample	Bladder Pump	Transducer	Northing	Easting
MW11				6848454.172	588935.9930
MW12				6848354.269	588742.4843
MW13				6848408.425	588879.9700
MW16	X	X		6848553.570	588947.8710
MW17	X	X		6848551.484	588948.1433
MW18				6848531.638	588988.1867
MW19				6848571.165	589025.1547
MW20				6848592.800	588967.1091
MW21				6848622.174	589000.2127
MW22				6848638.537	589022.4639
MW23				6848688.270	589038.5637
MW24				6848641.681	588975.4127
MW25				6848629.149	588940.7024
MW26	X	X		6848622.444	588918.9978
MW27	X	X		6848587.889	588919.2896
MW28	X	X		6848585.430	588917.8712
MW29	X	X		6848700.653	588897.0048
MW30				6848630.777	588855.5862
MW31				6848717.532	588561.9681
MW32				6848725.007	589122.1639
MW33	X	X		6848780.504	589096.4901
MW34				6848558.600	589074.0454
MW35				6848567.313	589075.9173
MW36				6848564.717	589084.2718
MW39				6848849.533	588651.3045
MW40	X	X		6848767.128	588726.9626
MW42	X	X		6848618.255	588792.7568
MW43	X	X		6848605.028	588834.4352
MW44	X	X		6848863.449	588738.0061
MW45	X	X		6848923.057	588700.9894
MW46	X	X		6848994.838	588631.5805
MW47	X	X		6849057.390	588604.4875
MW48				6848841.960	588787.3862
MW49	X	X		6848819.986	588882.4892
MW50	X	X	X	6848912.827	588629.5502
MW51	X	X	X	6848980.258	588580.2529
MW52	X	X		6849032.846	588522.5802
MW53	X	X	X	6848849.577	588568.7948
MW54	X	X	X	6848903.625	588517.9083
MW55	X	X		6848988.233	588449.4053

Monitoring Well ID	Groundwater Sample	Bladder Pump	Transducer	Northing	Easting
MW56	X	X	X	6848851.666	588696.7599
MW57	X	X	X	6848793.412	588612.8689
MW58	X	X	X	6848819.169	588496.7913
MW59	X	X	X	6848851.296	588656.2038

Acronyms and Abbreviations
ID – Identification
MW – Monitoring Well

Notes:
During static water level measurements, Sundance will measure the elevation difference between the top of casing to adjacent ground surface. The measured difference shall be reported with water table elevation results as a check on impacts of seasonal frost heave on monitoring well casing elevation.
Northing and Easting are in Alaska State Plane coordinates.

1.2 Surface Water Monitoring in Red Devil Creek

Sundance will conduct surface water monitoring to evaluate discharge rate and surface water quality at five locations on lower Red Devil Creek. The descriptions, location identifiers and coordinates of the five sampling stations are summarized in Table 2.

Table 2. Baseline Surface Water Monitoring Summary

Surface Water ID	Description	Northing	Easting
RD10SW	Red Devil Creek, downstream of the reservoir, upstream of the NTCRA.	6848181.15	588654.77
RD15SW	Red Devil Creek, new station immediately downstream of the newly aligned section (post-NTCRA) of Red Devil Creek, near the former baseline monitoring station RD13SW	6848571.56	588940.59
RD05SW	Seep on left bank of Red Devil Creek	6848581.63	588942.34
RD06SW	Red Devil Creek, near Settling Pond #3	6848656.07	589028.13
RD08SW (RD08BSW)	Red Devil Creek, near the confluence of Red Devil Creek and Kuskokwim River, downstream of sediment trap constructed during the NTCRA	6848790.31	589133.43

Acronyms and Abbreviations
NTCRA – non-time critical removal action
RD – Red Devil Mine
SW – Surface water

Notes:
The surface water monitoring location RD08SW was significantly impacted and eroded by 2022 spring breakup flooding. A new 2022 location, RD08BSW, was identified upstream of the flood debris depositional zone and has been updated on associated figures.
Northing and Easting are in Alaska State Plane coordinates.

1.3 Laboratory Analysis

The parameters with the most significant overall risk at Red Devil Mine are the primary focus of spring and fall baseline monitoring. The number of samples for laboratory analyses and the analytical methods applied to those samples are summarized in Table 3 and Table 4.

Table 3. Groundwater Analytical Summary

Analysis	Method	Primary Samples	Field Duplicates	MS/MSD	Trip Blank	Equipment Blank*	Total Samples
Total TAL Metals	EPA 6020B	28	3	2	3	2	38
Total Low-Level Mercury	EPA 1631E	28	3	2	3	2	38
Dissolved Low-Level Mercury	EPA 7470A	28	3	2	3	2	38

Acronyms and Abbreviations
EPA – U.S. Environmental Protection Agency

Notes:
Eurofins Frontier Global Sciences, LLC (Eurofins TestAmerica) will be the primary lab for all analyses.
All low-level mercury containers for surface water and groundwater will be contained together in separate coolers and the trip blank will accompany each cooler.
* Equipment blanks collected only for reusable water level meters due to dedicated pumps in each monitoring well.

Table 4. Surface Water Analytical Summary

Analysis	Method	Primary Samples	Field Duplicates	MS/MSD	Trip Blank	Equipment Blank*	Total Samples
Total TAL Metals	EPA 6020B	5	1	1	1	0	8
Total Low-Level Mercury	EPA 1631E	5	1	1	1	0	8
Dissolved Low-Level Mercury	EPA 7470A	5	1	1	1	0	8
Solids, Total Dissolved (TDS)	EPA 160.1	5	1	1	0	0	7
Solids, Total Suspended (TSS)	EPA 160.2	5	1	1	0	0	7
Inorganic Ions (Cl, F, SO4)	EPA 300.0	5	1	1	0	0	7
Nitrate-Nitrite as Nitrogen	EPA 353.2	5	1	1	0	0	7
Alkalinity as Carbonate and Bicarbonate (CO3/HCO3)	EPA 310.1	5	1	1	0	0	7

Acronyms and Abbreviations
EPA – U.S. Environmental Protection Agency

Notes:
Eurofins Frontier Global Sciences, LLC (Eurofins TestAmerica) will be the primary lab for all analyses.
All low-level mercury containers for surface water and groundwater will be contained together in separate coolers and the trip blank will accompany each cooler.
* No equipment blanks are required, only dedicated one-time use equipment is used to collect surface water samples.

1.4 Schedule

Sundance bi-annual spring and fall groundwater and surface water baseline monitoring are expected to take place in late May to early June 2023 and early September 2023 and will take approximately one week to complete.

1.5 Field Quality Control Samples and Frequency

Field quality control (QC) samples may include trip blanks, field blanks, rinsate blanks, and field duplicates to be submitted blind to the analytical laboratory. Field QC samples and frequency will be collected in accordance with the *2019 Baseline Monitoring Work Plan* (BLM, 2019) with the exceptions noted in Table 3 and Table 4.

1.6 Quality Assurance

General updates to the Quality Assurance Project Plan (QAPP) are required due to changes in contractors, business mergers, and updates to standard operating procedures (SOPs) over time. The personnel organization and contact information for the BLM, BLM contractors, and analytical laboratory personnel has been updated in Table 5.

Table 5. Contact Information

Organization	Contact	Title	Telephone	Address
BLM	Mike McCrum	PM	(907) 271-4426	222 West 7th Ave #13 Anchorage, Alaska 99513
Sundance Consulting Inc. (BLM Contractor)	Colleen Rust	PM	(505) 321-4515	10400 Academy Rd NE Suite 200 Albuquerque, NM 87111
	Steve Offner	QA Manager	(678) 447-5048	
	George Garner	HSO	(251) 605-1931	
Eurofins Environment Testing Northwest, LLC (Subcontracted Laboratory)	Pauline Matlock	PM	(253) 248-4965	5755 8th St E, Tacoma, WA 98424

Acronyms and Abbreviations
BLM – Bureau of Land Management
HSO – Health and Safety Officer
PM – Project Manager
QA – quality assurance

1.7 Structure Evaluation

Structures remaining from mining activities or from the 2014 Interim Action (BLM, 2014) will be evaluated as part of the bi-annual spring and fall baseline monitoring events. The following areas will be examined and photographed to document changes in condition.

- Dolly Sluice and Dolly Sluice Delta
- Barge Landing near the Red Devil Creek Delta
- Reworked areas along Red Devil Creek (e.g., stockpiles, realignment and gabon structures)

Condition of structures, vegetation condition, presence of erosional features, and other general site conditions will be recorded and evaluated as part of each bi-annual spring and fall baseline monitoring event.

1.8 Laboratory Standard Operating Procedures

The subcontracted laboratory, Eurofins Environment Testing Northwest, LLC, located in Tacoma, Washington, SOPs have been updated to reflect changes to internal operating procedures. The updated laboratory SOPs are included in Appendix A.

1.9 Health and Safety Plan

A Health and Safety Plan (HASP) has been updated to reflect changing conditions and operations at Red Devil Mine. The HASP includes updated BLM Risk Management Worksheets, activity hazard analyses and COVID-19 language. The updated HASP is included in Appendix B.

2.0 REFERENCES

U.S. Department of the Interior Bureau of Land Management (BLM), 2014. *Non-Time Critical Removal Action Memorandum, Red Devil Mine Site, Red Devil, Alaska*. June 2014.

BLM, 2019. *Final Work Plan 2019 Groundwater and Surface Water Baseline Monitoring, Red Devil Mine, Alaska*. Prepared by Ecology and Environment Inc. (E.&E.), May 2019.

BLM, 2022. *Addendum to the Final Work Plan 2019 Groundwater and Surface Water Baseline Monitoring, Red Devil Mine, Alaska*. Prepared by Sundance Consulting Inc., April 2022.

Appendix A. Laboratory SOPs

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eurofins	Always check on-line for validity. **Mercury in Water by Oxidation, Purge & Trap and CV-AFS (EPA Method 1631, Rev E)**	Level:
Document number: **EFGS-T-AFS-SOP2992**		**Standard Operating Procedure**
Old Reference: **FGS-137**		
Version: **6.1**		Organisation level: **4-Business Unit**
Approved by: **GW2R, KOJ6** Effective Date **28-SEP-2021**	Document users: **EFGS_AFS, EFGS_S-and-R, EFGS_TraceMetal**	Responsible: **EFGS_SMM**

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1) Revision Log:

Revision: 06	**Effective Date:** This version	
Section	**Justification**	**Changes**
2.15	Required	Updated reference
3	Required	Added link to CHP/QM/standard SOP
6	Required	Delete references to the bubbler system
6	Required	Added description of the 2600 system
10.1/10.2	Required	Updated and added links to SDS/CHP
11.4	Required	Added link to QM
14.3	Required	Corrected the recipe for the preparation of 0.2N BrCl
14.8	Required	Updated NIST 1641 Standard (d to e).
14.8/9	Updated	Removed info regarding the receipt, prep, and storage of standards and added link to new standard SOP. Renumbered sections.
14.9	Required	Added HNO3 and RINSE
17	Required	Replaced algorithms with reference to SOP2822
19.3	Required	Reworded to clarify MD's aren't required
19.14	Required	Added carryover section. Blank now required after samples over 200 ng/L only.

2) Reference:

2.1 EPA Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry, 2002.

2.2 Method 1669, "Method for Sampling Ambient Water for Determination of Metals at EPA Ambient Criteria Levels," U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology, Engineering and Analysis Division (4303), 401 M Street SW, Washington, DC 20460, April 1995 with January 1996 revisions.

2.3 Bloom, N.S.; and Tsalkitzis, E. Standard Operating Procedure FGS-012 Determination of Total Mercury in Aqueous Media (Modified EPA Method1631). Frontier GeoSciences Inc., Quality Assurance Manual 1995.

2.4 Bloom, N.S.; Ultra-Clean Sample Handling, Environmental Lab 1995, March/April, 20.

2.5 Bloom, N.S.; Horvat M., and Watras C.J. Results of the International Mercury Speciation Intercomparison Exercise. Wat. Air Soil Pollut. 1995, 80, 1257.

2.6 Bloom, N.S.; Crecelius, E.A. Determination of Mercury in Seawater at Sub-nanogram per Liter Levels.Mar.Chem.1983, 14, 49.

2.7 Bloom, N.S.; Crecelius, E.A. Distribution of Silver, Lead, Mercury, Copper, and Cadmium in Central Puget Sound Sediments Mar. Chem 1987, 21, 377-390.

2.8 Bloom, N.S.; Fitzgerald, W.F. Determination of Volatile Mercury Species at the Picogram Level by Low-Temperature Gas Chromatography with Cold-Vapor Atomic Fluorescence Detection. Anal. Chem. Acta. 1988, 208, 151.

2.9 Cossa, D.; Couran, P. An International Intercomparison Exercise for Total Mercury in Seawater. App.Organomet. Chem.1990, 4, 49.

2.10 Fitzgerald, W.F.; Gill, G.A. Sub-Nanogram Determination of Mercury by Two-Stage Gold Amalgamation and Gas Phase Detection Applied to Atmospheric Analysis. Anal. Chem. 1979, 15, 1714.

2.11 Gill, G.A.; Fitzgerald, W.F. Mercury Sampling of Open Ocean Waters at the Picogram Level Deep Sea Res.1985, 32, 287.

2.12 EPA Method 30.B, Determination of total vapor phase mercury emissions from coal-fired combustion sources using carbon sorbent traps.

2.13 Chemical Hygiene Plan, Eurofins Frontier Global Sciences, current version.

2.14 TNI Environmental Laboratory Sector, Vol 1, Management and Technical Requirements, ELV1-2016.

2.15 Department of Defense / Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, prepared by DoD Environmental Data Quality Workgroup (EDQW) and DOE Consolidate Audit Program (DOECAP) Data Quality Workgroup (DOE-DQW), Version 5.3, May 2019.

3) Cross Reference:

Document	Document Title
EFGS-Q-QM-QM5805	Quality Manual
EFGS-S-HS-12066	Chemical Hygiene Plan
EFGS-Q-QD-SOP41510	Volumetric Verification and Pipette and Dispenser Operation & Maintenace
EFGS-S-SB-SOP5139	Cleaning of Sampling Equipment and Bottles for Trace Metals Analysis
EFGS-S-CS-SOP2794	Ultra-Clean Aqueous Sample Collection
EFGS-S-HS-SOP2991	Waste Disposal Procedures for Client Sample Waste
EFGS-T-AFS-SOP2822	Determination of Total Mercury by Flow Injection AFS (Mod 1631E)
EFGS-T-TM-SOP2839	Stock and Prepared Standards

4) Purpose:

4.1 This SOP is designed to ensure that all reproducible traceable procedures in EPA 1631 are followed in the standardization of the total mercury analyzers and in the analysis of samples for total mercury, as well as to establish the limits wherein data will be considered acceptable.

5) Scope:

5.1 This Standard Operating Procedure (SOP) describes a method for the determination of total mercury (Hg) in filtered and unfiltered water by oxidation, purge and trap, desorption, and cold vapor atomic fluorescence spectrometry (CVAFS).

5.2 This method is designed for the determination of mercury in the range of 0.5-40 ng/L (ppt). Application may be extended to higher levels by selection of a smaller sample size, as long as the instrument value (intensity) remains within the calibration curve.

5.3 The control limits used in this procedure are from EPA 1631E.

6) Basic Principles:

6.1 Total mercury analyses are split into two categories: waters and solids. For analysis of aqueous samples, a dilution (up to 1x) of oxidized sample is added to a 50mL vial. If less than 30mL of sample is used, the sample is diluted to a final volume of at least 30mL with 1% BrCl. The final volume is neutralized with 25uL of 25% hydroxylamine-hydrochloride (NH2OH-HCl). Approximately 25mL of sample is drawn into the system by an auto sampler. (Please refer to Figure 1. for a detailed system flow diagram of the Tekran 2600 analysis system.) Sample is then mixed with 3% stannous chloride (SnCl2) reducing Hg2+ to Hg0 before entering the phase separator.

6.2 As sample travels down the phase separator Hg0 is liberated by a counter flow of ultra pure argon (or nitrogen). Mercury then travels through a soda-lime acid vapor trap, a switching valve (V2), and amalgamates onto the fixed “sample trap”. Following primary amalgamation V2 is triggered, switching on the flow of pure argon through the sample trap. At the same time the sample trap is heated, and mercury is released into the pure argon gas stream, passing through a second switching valve (V1) and amalgamating onto the “analytical trap”. The analytical trap is then heated releasing mercury into the AFS detector. All event timing and peak integration is carried out via a PC running TekMDS-2 software running the EPA 1631 event timing file (ETF). NOTE: This method may also be run using 15ml vials and selecting the appropriate ETF with in the TekMDS-2 software. Sample must fill the vial up to the 15ml mark. All other procedures, reagent concentrations, and dilutions should be kept the same.

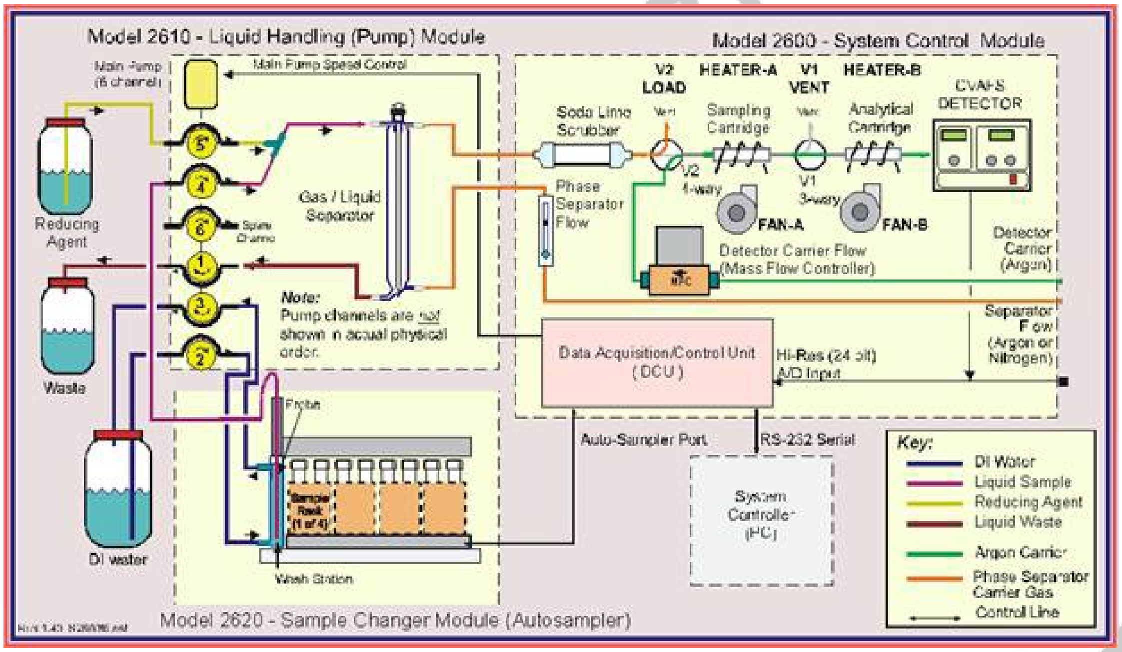

Figure 1 - Tekran 2600 Flow Diagram

7) Reference Modifications:

7.1 There were no significant modifications to this method.

8) Definitions:

8.1 Analytical Duplicate (AD): A representative sample (that yielded a result within the calibration curve) is analyzed a second time during the analytical run. The second analysis should be at the same aliquot as the original.

8.2 Analytical Run – The continuous analysis of one or more batches during the same 12 hour-shift. Each analytical day requires a minimum five-point calibration curve, ICV, at least 3 IBLs, and CCV/CCB every ten runs. An analytical day must conclude with a CCV/CCB.

8.3 Analytical Spike and Analytical Spike Duplicate (AS/ASD): A representative sample is selected and spiked, with a dilution of the primary source, during the analytical run, at a target concentration of 1-5X the ambient concentration of the sample. These QC samples are used to indicate sample matrix effects on the analyte of interest. Non-detectable samples are spiked at 1 – 5 x of the MRL/PQL.

8.4 Batch: 20 client samples or less grouped for preparation. See Quality Assurance Section for batch requirements.

8.5 Calibration Standards (CAL) – a series of standards that will be used to calibrate the instrument, made from a primary source stock standard. Three calibration blanks plus at least five different concentrations are required, beginning with one at PQL concentration.

8.6 Certified Reference Material (CRM) – a standard of known composition that is certified by a recognized authority and representing a sample matrix. It is used to verify the accuracy of a method.

8.7 Continuing Calibration Blank (CCB): An instrument blank that is used to monitor the ambient blank concentration after the Continuing Calibration Verification (CCV).

8.8 Continuing Calibration Verification (CCV): An aliquot of standard from a secondary source as the calibration standard, at a value of 5ng/L. This standard is analyzed after every 10 analytical runs, and determines whether the instrument is maintaining calibration.

8.9 Continuing Demonstration of Capability (CDOC)

8.10 Control Limit (CL) – the limit of the range of acceptability for the quality control samples

8.11 Equipment Blank (EB): Reagent water processed through the sampling devices and placed in a sample container prior to using the equipment to collect samples and used to demonstrate that the sampling equipment is free from contamination.

8.12 Field Blanks (FB): A sample of reagent water placed in a sample container in the field and used to demonstrate that samples have not been contaminated by sample collection or transport activities. EPA-1631E recommends the analysis of at least one field blank per 10 samples collected at the same site at the same time. Analyze the blank immediately before analyzing the samples in the batch.

8.13 Initial Calibration Verification (ICV): A standard that is prepared from a secondary source stock standard with a value of 5ng/L. This standard is run immediately following the calibration curve and verifies instrument calibration.

8.14 Initial Blank Level (IBL): An instrument blank that is used to demonstrate the ambient blank concentration of the instrument.

8.15 Initial Demonstration of Capability (IDOC).

8.16 Laboratory Control Sample (LCS and LCSD) or Quality Control Sample (QCS): A sample (and duplicate) containing a known concentration of mercury that is used to monitor complete method performance. The preferred LCS is a matrix matched Certified Reference Material (CRM), but a blank spike meets the requirement also. In LIMS, the LCS is always referred to as a Blank Spike (BS), whether it is matrix matched or not.

8.17 Limit of Detection (LOD) – The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. The LOD shall be at least 2 times but no greater than 4 times the DL for multiple analyte methods or 2 times but no greater than 3 times the DL for single analyte methods.

8.18 Limit of Quantitation (LOQ) – equal to PQL and verified on a quarterly/annual basis, depending on the preparation, by spiking within 2 times the LOQ and showing a recovery between 70 – 130%.

8.19 LIMS: Laboratory Information Management System. Computer software used for managing samples, standards, and other laboratory functions.

8.20 May: This action, activity, or procedural step is optional.

8.21 May Not: This action, activity, or procedural step is prohibited.

8.22 Matrix Spike (MS) and Matrix Spike Duplicate (MSD): A representative sample is selected and spiked with a dilution of the primary source at a known concentration. The MS and MSD are run through the entire analytical process just as the samples are. These QC samples will indicate sample matrix effects on the analyte of interest.

8.23 Method Blank (MBLK) or Preparation Blank (PB): For waters, reagent water that is prepared and analyzed in a manner identical to that of samples. For digested solids, preparations blanks consist of the same reagents used to digest the samples, in the same volume or proportion and are carried through the complete sample preparation and analytical procedure. Boiling chips are used as a blank matrix for solids. Preparation blanks are referred to as BLK in LIMS.

8.24 Method Detection Limit (MDL): A limit derived from 40 CFR, Part 136, Appendix B. This method produces a defined value that is the minimum concentration that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero from a given matrix.

8.25 Method Duplicates/Method Triplicates (MD/MT): A second or third separate sample dilution, taken from the same source sample, prepared and analyzed in the laboratory separately. An MSD may be used as a duplicate.

8.26 Reagent water: 18 MO minimum, reagent water starting from a pre-purified (distilled, Reverse Osmosis, etc.) source.

8.27 Must: This action, activity, or procedural step is required.

8.28 Ongoing Precision and Recovery (OPR): A dilution of a secondary source resulting in an instrumental concentration of 5.0 ng/L mercury.

8.29 PM: Project Manager.

8.30 Practical Quantitation Limit (PQL), Method Reporting Limit (MRL): The minimum concentration that can be reported quantitatively. The PQL is often described as 1-10 times higher than MDL. Eurofins Frontier defines the PQL as the lowest concentration that can achieve 70-130% recovery for 10 replicate sample preparations. In LIMS, the PQL is referred to as the MRL.

8.31 Primary Source: The stock standard used to make the calibration standard. Procedural Method: A method where standards and samples are run through the analytical procedure exactly the same. By NELAC definition, this SOP is a procedural method.

8.32 Secondary Source: The stock standard used to make the OPR/ICV/CCV standard.

8.33 Shall: This action, activity, or procedure is required.

8.34 Should: This action, activity, or procedure is suggested, but not required.

8.35 Stock Standard Solution (SSS) – a standard of analyte that is purchased from a certified source for the preparation of working standards.

8.36 Total mercury: As defined by this method, all bromine monochloride-oxidizable mercury forms and species found in aqueous solutions. This includes, but is not limited to, Hg(II), Hg(0), strongly organo-complexed Hg(II) compounds, adsorbed particulate Hg(P), and several tested covalently bound organomercurials (i.e. CH_3HgCl, $(CH_3)_2Hg$, and $C_6H_5HgOOCCH_3$). The recovery of mercury bound within microbial cells may require additional preparation steps (i.e. UV oxidation, or oven digestion).

8.37 Travel or Trip Blank (TB): A sample of reagent water placed in a sample container in the laboratory and used to demonstrate that samples have not been contaminated by transport activities.

9) Interferences:

9.1 Gold and iodide are known interferences. At a mercury concentration of 2.5 ng/L and at increasing iodide concentrations from 30 to 100 mg/L, test data have shown that mercury recovery will be reduced from 100 to 0 percent. At iodide concentrations greater than 3 mg/L, the sample should be pre-reduced with $SnCl_2$ (to remove brown color immediately prior to analysis). If samples containing iodide concentrations greater than 30 mg/L are analyzed, it may be necessary to clean the analytical system with 4N HCl after the analysis.

9.2 Water vapor has the potential to create recovery interferences. To prevent interference from water, ensure that soda-lime pre-traps and gold traps remain dry.

9.3 The presence of high concentrations of silver and/or gold can cause $SnCl_2$ to precipitate out of solution. High concentrations of these metals can sometimes be found in the matrix spike samples from the digestion sets that are shared with the trace metals group. When analyzing digestates where the matrix spike samples have been spiked with silver or gold, the matrix-spiked samples must not be used for mercury analysis. Instead, an alternate matrix spike and matrix spike duplicate (MS/MSD) should be prepared and analyzed. If this is not possible, an Analytical Spike/Analytical Spike Duplicate (AS/ASD) must be analyzed on the ambient sample.

10) Safety Precautions, Pollution Prevention and Waste Handling:

10.1 Personnel will don appropriate laboratory attire according to Chemical Hygiene Plan (CHP) *EFGS-S-HS-12066*. This may include, but is not limited to: laboratory coat, apron, eye protection, face shield and protective gloves.

10.2 The toxicity or carcinogenicity of each laboratory reagent has not been fully established. Therefore, every chemical must be treated as a potential health hazard. Exposures shall be reduced as much as possible. Analysts and technicians are expected to familiarize themselves with the characteristics and risks of each chemical they work with by referring to the SDS located at https://msdsmanagement.msdsonline.com/company/5c1df5b3-747d-4789-8104-42457dc3a3e5/

10.2.1 Note: Use particular caution when preparing and using BrCl, as it releases extremely irritating, corrosive fumes similar in effect to free chlorine. Always handle this reagent in an approved fume hood

10.3 All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known disease-causative agents. Eurofins Frontier will reimburse the expense of Hepatitis A and B immunizations for any laboratory staff member who desires this protection.

10.4 Hydrochloric acid: Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion. Slightly hazardous in case of inhalation (lung sensitizer). Non-corrosive for lungs. Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering. For more information see SDS.

10.5 See Eurofins Frontier Global Sciences Chemical Hygiene Plan (CHP) for general information regarding employee safety, waste management, and pollution prevention.

10.6 Pollution prevention information can be found in the current Eurofins Frontier Global Sciences Chemical Hygiene Plan (CHP), which details and tracks various waste streams and disposal procedures.

10.7 All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations. Any waste generated by this procedure should be disposed of according to SOP *EFGS-S-HS-SOP2991* “Waste Disposal Procedure for Client Sample Waste,” which provides instruction on dealing with laboratory and client waste.

11) Personnel Training and Qualifications:

11.1 An analyst must perform an initial demonstration of capability (IDOC) that includes four replicates of a secondary source before being qualified to analyze samples without supervision. Continuing DOC will be maintained and monitored via performance on CRMs and other QC samples.

11.2 The analyst/laboratory technician must have read this SOP and other relevant SOPs and have the training documented on the applicable form(s). The analysis may be questioned on SOP by supervisor(s) and/or trainers.

11.3 Training is documented by the employee and supervisor, and is kept on file in the QA Office. The employee must read, understand, and by signing the training document, agree to perform the procedures as stated in all Standard Operating Procedures (SOPs) related to this method.

11.4 All employees must read the *Quality Manual (QM)* and complete the annual Ethics training.

11.5 All training documents including IDOCs, CDOCs, Initial QA orientation, and Ethics training are stored by the Quality Assurance Manager in the employees training file for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

11.6 Chemical Safety Training, Compressed Gas Training, Chemical Hygiene Plan documentation, and Shipping of Hazardous goods, are stored by the Health and Safety Officer for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

12) Sample Collection, Preservation, and Handling:

12.1 Aqueous samples are collected in rigorously cleaned fluoropolymer (e.g. Teflon) or PETG bottles and caps (as described in *EFGS-S-SB-SOP5139* "Cleaning of Sampling Equipment and Bottles for Mercury Analysis"). Certified clean glass bottles with fluoropolymer lids may be used if mercury is the only analyte of interest.

12.1.1 Aqueous samples are preserved upon receipt with 0.2N BrCl that has tested low in mercury. Samples are typically preserved to 1% BrCl v/v, but may require further oxidation due to high levels of organic matter or mercury.

12.2 All samples should be collected utilizing clean techniques, so as not to cross-contaminate samples with mercury. See *EFGS-S-CS-SOP2794* "Ultra Clean Aqueous Sample Collection" and EPA Method 1669 for aqueous sample techniques.

13) Apparatus and Equipment:

13.1 LIMS – Element, version 6.11 or higher; Computer – Windows 7 or 8

13.2 Tekran Operating System – see Instrument Software Inventory *EFGS-S-IT-WI7061*

13.3 Model 2600 System Control Module – manufactured by Tekran, this module contains dual stage pre-concentration/desorption units, a high resolution analogue to digital converter, and a high sensitivity AFS Detector (IDL<1pg).

13.4 Model 2610 Pump Unit – manufactured by Tekran, this module contains a precision 6 channel peristaltic pump with optical speed feedback. Speed may be varied manually or under computer control. The pump head has three four-roller channels to providing high pumping rates for wash station circulation and waste removal, and three eight-roller channels to providing slower/low pulse delivery of $SnCl_2$ and sample (one extra channel).

13.5 Model 2620 Auto-Sampler – a specially modified Gilson Model 223 auto-sampler, includes reticulating wash station and allows for automatic sample handling with the 2600 analysis system.

13.6 50mL Polypropylene Vials with screw caps, manufactured by Environmental Express (Item No: SC475), or equivalent pre-cleaned and/or tested vials, graduated to 50mL.

13.7 Three-stop tubing, various inner diameters of Marprene for $SnCl_2$ (1.02mm), for wash station fill (2.38mm), wash station drain (2.79mm), and phase separator drain (2.79mm) Three stop silicone tubing (2.05mm) is used for sample feed.

13.8 Teflon Fittings and FEP tubing – of various sizes and lengths. Tubing from sample probe, $SnCl_2$, rinse water, phase separator, to and from wash station, and between gold traps is 1/16 inch (inner diameter) FEP.

13.9 Soda-Lime trap – a 14cm x 1.1cm diameter glass tube containing 12-14g of reagent grade, non-indicating 8-14 mesh soda-lime ($Ca(OH)_2$+NaOH) aggregates, packed between portions of silanized glass wool. This trap is purged of mercury by placing it on the output of the phase separator and purging it to air with a 0.5% BrCl rinse solution and 3% $SnCl_2$ for approximately 20 minutes with Ar (or N_2)at 400 mL/min.

13.10 Phase separator – manufactured by Tekran, the phase separator utilizes laminar fluid flow of sample over a frosted rod counter current to a stream of argon gas at 400mL/min.

13.11 Gold Coated Quartz Sand Trap ("sample trap") – manufactured by Tekran or made in house, heated from 0% to 100%, 8 Amp maximum.

13.12 Pure Gold Bead Trap ("analytical trap") – manufactured by Tekran, heated from 0% to 100%, 8 Amp maximum.

13.13 Tekran 2500 Atomic Fluorescence Spectrophotometer (AFS) or equivalent: A high sensitivity AFS Detector (IDL<1pg) with a required wavelength of 253.7 nm and associated software.

13.14 Flow meter/needle valve: A unit capable of controlling and measuring gas flow to the cold vapor generator at 200-500 mL/min.

13.15 Teflon Fittings: Connections between components and columns are made using Teflon FEP tubing and Teflon friction fit tubing connectors.

13.16 Soda-Lime pre-trap: A 10cm x 0.9cm diameter Teflon tube containing 2-3 g of reagent grade, non-indicating 8-14 mesh soda-lime ($Ca(OH)_2$+NaOH) aggregates, packed between portions of silanized glass wool. This trap is purged of mercury by placing it on the output of a clean cold vapor generator and purging it with ~3-5% HCl and ~600 µL of $SnCl_2$ for approximately 20 minutes with N_2 at 40 mL/min.

13.17 Gold Traps: Made from 12 cm lengths of 6 mm OD quartz tubing, with a 4-way crimp 3.0 cm from one end. The tube is filled with approximately 2.5 cm of 20/40 mesh gold-coated quartz sand, the end of which is then plugged with quartz wool. Gold-coated sand traps are heated to 450-500°C (the coil should have a barely visible red glow when the room is darkened) with a coil consisting of 75 cm of 24-gauge nichrome wire at a potential of 10 VAC. Potential is applied and finely adjusted with an auto-transformer.

13.18 Agilent Integrator Recorder or equivalent: Any multi-range chart recorder or integrator with 0.1-5.0 mV input and variable speeds is acceptable. Data capture software may also be used.

13.19 Pipettes: Calibrated variable pipettes with a range of 20 µL – 10 mL. Used to make solutions and sample dilutions. Pipettes are to be calibrated weekly according to SOP *EFGS-Q-QD-SOP41510*.

13.20 Analytical Balance – capable of accurately weighing to the nearest 0.1mg, and able to tare at least one gram. The analytical balances are verified for accuracy on a daily basis according to *EFGS-Q-QD-SOP2710*, "Balance Verification, Calibration and Maintenance."

14) Reagents and Standards:

14.1 Reagent Water: 18-MO ultra pure deionized water starting from a pre-purified (distilled, R.O., etc.) source is used. To remove any remaining trace metals and organics, an activated carbon cartridge is placed between the final ion exchange bed and the 0.2-µm filter. Reagent water used in the mercury lab is checked monthly for total mercury concentrations, and must test below 0.25ng/L.

14.2 Hydrochloric Acid (HCl): Concentrated (36-38% weight basis). Must be trace-metal purified and reagent grade. HCl is typically monitored through performance of the BrCl. Sometimes it will be necessary to test the HCl directly. Do not prep blank correct. This reagent should test below 5.0 ng/L. This solution is considered stable until the expiration date on the bottle, set by the manufacturer.

14.3 0.2N Bromine Monochloride (BrCl):

14.3.1 27 g of KBr is added to a 2.5 L bottle of concentrated HCl (pre-analyzed and below 5 ng/L Hg). The bottle is inverted in a fume hood to mix the acid and KBr. The solution sits overnight, allowing the KBr to dissolve.

14.3.2 38 g of KBrO3 (certified to be low in Hg) is slowly added to the acid. As the KBrO3 is added, the solution should go from yellow to red to orange.

CAUTION: This process generates copious quantities of free halogens (Cl2, Br2, BrCl) which are released from the bottle. Add the KBrO3 SLOWLY in a well operating fume hood.

14.3.3 Loosely cap the bottle and allow to sit for at least 1 hour in a fume hood before tightening. Once tightly capped, invert bottle to make sure all of the solids go into solution.

14.3.4 The expiration time for this reagent is set by default to six months in LIMS. There is no suggested expiration date in EPA method 1631E, therefore it can be extended, as long as the primary reagent has not expired. The mercury concentration of the BrCl is monitored through the preparation of water preparation blanks.

14.3.5 This reagent must test below 0.20ng/L. Do not prep blank correct. Analyze one replicate per bottle.

14.4 Hydroxylamine hydrochloride: Dissolve 300g of NH2OH-HCl in reagent water and bring the volume up to 1L. This solution may be purified by the addition of 1mL SnCl2 solution and purging overnight at 500mL/min with mercury-free N2. The working reagent is a 25% solution that is made by adding one part reagent water to one part 50% hydroxylamine hydrochloride. This reagent must test below 0.25ng/L.

14.4.1 The expiration time for this reagent is set by default to six months in LIMS. There is no suggested holding time in EPA method 1631E; therefore the holding time can be extended, as long as the primary reagent has not expired (the stannous chloride that's added for purification is not considered a primary reagent).

14.5 Stannous Chloride (SnCl2): Weigh out 500 g SnCl2 using a calibrated balance that also has been verified for the day. Dissolve with three 100 mL aliquots of concentrated HCl and transfer to a 1L I-CHEM glass bottle, which contains approximately 300 mL of reagent water. Bring this solution up to approximately 1 L of volume and purge overnight with mercury-free N2 at 500 mL/min to remove all traces of mercury. Store tightly capped. The working reagent is a 25 % solution that is made by adding one part reagent water to one part 50 % stannous chloride.

14.5.1 This reagent must test below 0.20 ng/L. Do not prep blank correct. Analyze one replicate per bottle.

14.5.2 The expiration time for this reagent by default is set to six months in LIMS. There is no suggested holding time in EPA method 1631E; therefore the holding time can be extended, as long as the primary reagent has not expired.

14.6 Argon Grade 4.7 or better (ultra high-purity grade): Argon that has been further purified by the removal of mercury using a gold trap that is located in line between the gas output and the analyzer gas input.

14.7 Nitrogen Grade 4.5 (standard laboratory grade): Nitrogen that can be further purified of mercury using a gold trap that is located in-line.

14.8 Total Mercury Stock and Prepared Standards:

14.8.1 See SOP *EFGS-T-TM-SOP2839* for information regarding the receipt, documentation, preparation, and storage of purchased and prepared standards used by EFGS.

14.8.2 Total Mercury Stock Standard Solution (Stock): Certified mercury standard purchased from High Purity Standards (1000 µg/mL (1 000 000 ng/mL) primary source) or Absolute Standards (100 µg/mL (100 000 ng/mL) secondary source), or any equivalent standard.

14.8.3 100,000 ng/mL Spiking Standard: Made from the Primary Stock Standard (High Purity, or equivalent vendor). Dilute 10 mL of the stock standard to 100 mL of reagent water containing 2 % BrCl. (Can also be made by preserving Secondary Stock Standard to 2% BrCl).

14.8.4 10,000 ng/mL Spiking Standard: If made from the Primary Stock Standard (High Purity, or equivalent vendor). Dilute 1.0 mL of the stock standard to 100 mL of reagent water containing 2 % BrCl. If made from Secondary Stock Standard, dilute 10mL of stock standard to 100mL with reagent water containing 2% BrCl.

14.8.5 1,000 ng/mL Spiking Standard: If made from the Primary Stock Standard (High Purity, or equivalent vendor). Dilute 0.250 mL of the stock standard to 250 mL RO water containing 2% BrCl. If made from Secondary Stock Standard dilute 2.5mL of stock standard to 250mL with RO water containing 2% BrCl.

14.8.6 100 ng/mL Spiking Standard: Made from a stock standard or dilution of a stock standard with a concentration of 100,000 ng/mL. Dilute 0.100 mL of the 100,000 ng/mL dilution to 100 mL of reagent water containing 2 % BrCl. Expiration date is currently set at 3 months or when the stock standard expires, whichever is shorter.

14.8.7 Calibration Standard (10 ng/mL): Must be made from a dilution of the Primary Stock Standard (High Purity, or equivalent vendor). Typically made by diluting 0.5mL of a 10,000 ng/mL Primary Spiking Standard to 500 mL of reagent water containing 2 % BrCl. Transfer to glass or Teflon bottle. The calibration standard is considered stable for three months or until the stock standard expires.

14.8.8 Calibration Standard (1 ng/mL): Must be made from a dilution of a Primary Stock Standard. Typically made by diluting 1.0mL of a 100 ng/mL Primary Spiking Standard to 100mL with Reagent water containing 2% BrCl.

14.8.9 Initial Calibration Verification (ICV): A 10 ng/mL ICV solution is prepared using the Secondary Stock Standard (Absolute Standards, or equivalent vendor). Use 0.100 mL (100 µL) of the Secondary Stock Standard to 1000 mL Milli-Q containing 2 % BrCl. Transfer to one 1000 mL glass or Teflon bottle. The ICV standard is considered stable for three months or until the stock standard expires. It is recommended to alternate expiration date with the CAL standard.

14.8.10 Continuing Calibration Verification (CCV): For CCV analysis, use 200 µL of the 10 ng/mL CAL standard (documented in LIMS as SEQ-CAL3). The True Value is 20 ng/L.

14.8.11 Certified Reference Material (CRM) for Total Mercury in Water: A 1.5679 mg/L solution (1.557 mg/kg at a density of 1.007 g/mL) is prepared by adding a 5.0 mL of CRM NIST 1641e (from ampoule) into a 1000 mL flask containing of reagent water. This solution is diluted to 1000 mL, and an additional 10 mL of 0.2N BrCl is added, resulting in a final volume of 1010 mL. Preparing the solution in this manner makes a 1:200 dilution of the stock CRM. This solution is considered stable for one year, or until the stock standard expires. Results are corrected for the additional 1 % BrCl in the analysis Excel spreadsheet and in LIMS.

14.8.12 Ongoing Precision and Recovery (OPR) for “Strict” 1631E: A 5.0 ng/L solution is prepared by adding 100 µL of the 100 ng/mL secondary spiking standard into 2000 mL reagent water. An additional 1 % BrCl (20 mL) of BrCl is added, so that the final volume is 2020 mL. This standard is analyzed at 100 mL at the instrument, and preparation blank corrected exactly in the same manner as samples

14.9 Nitric Acid (HNO3): concentrated (69%-70% weight basis). Trace metal purified reagent-grade HNO3 is pre-analyzed and lot sequestered. Several brands (Baker, Fisher, Omnitrace) have been found to have lots with acceptably low levels of trace metals. This reagent should be from a lot number that has been previously tested to be low for the analytes of interest. This reagent shall be entered into LIMS and the expiration date is set to the same as the manufacturer’s expiration date.

14.9.1 Nitric Acid, 5%

14.10.1.1 Rinse bottle labeled:

Metals Rinse, 5% HNO3

To be used for CV-AFS (THg only) and ICPMS preps/analysis.

15) Calibration:

15.1 Follow the calibration in *EFGS-T-AFS-SOP2822* while still adhering to the QA/QC criteria of this method.

16) Procedure:

16.1 Follow the procedure in *EFGS-T-AFS-SOP2822* while still adhering to the QA/QC criteria of this method.

17) Calculations:

17.1 The Tekran 2500 is no longer operated by the laboratory. For the calculations that are based on data generated from a Tekran 2600, see *EFGS-T-AFS-SOP2822*.

18) Statistical Information/Method Performance:

18.1 Method Detections Limits (MDL) are determined during method development and then annually thereafter according to 40 CFR Part 136, Section B and and CA-Q-S-006.

18.2 The Practical Quantitation Limit (PQL) is the reporting limit for this method and is included as the lowest calibration point (2003 NELAC regulation 5.5.5.2.2.1.h.3). The PQL is determined by running ten samples with a concentration that will produce a recovery of 70-130 %. The PQL is referred to as the Method Reporting Limit (MRL) in LIMS.

18.3 Using clean handling techniques and reagents tested low for Hg content, the LOD value for Total Hg in water is typically less than 0.2 ng/L, while the PQL is 0.50 ng/L.

18.4 Current LODs, LOQs, MDLs, and PQLs are stored at: Cuprum\General and Admin\Quality Assurance\MDLs & PQLs.

19) Quality Assurance/Quality Control:

19.1 A minimum of three preparation blanks and one LCS/LCSD (preferably NIST 1641e or equilivalent), must be analyzed per preparation batch. The upper control limit for each preparation blank is equal to the PQL.

19.2 Matrix Spikes: One Matrix Spike/Matrix Spike Duplicate (MS/MSD) must be performed for every 10 samples. The recovery of the MS/MSD must be between 71%-125% recovery, and the Relative Percent Difference (RPD) below 24%. If an MS/MSD is out of control, the analyst should investigate to identify the source of the failure. The MS and MSD may be used as duplicates. Some failures may be qualified using QA Qualification Flow Charts (Appendix A).

19.2.1 For aqueous samples, the MS/MSD is spiked at 1 to 5 times the ambient concentration, with 0.25 ng being the minimum spiking level. Sample dilutions for the MS/MSD should be the same as the ambient sample dilution, if sufficient sample volume exists. NEVER ADD SPIKE DIRECTLY TO THE ORIGINAL SAMPLE CONTAINER UNLESS OTHERWISE INSTRUCTED.

19.3 Matrix Duplicates – Not required. A Matrix Duplicate (MD) or Matrix Triplicate (MT) may be analyzed for every batch of 20 samples upon client request. The MSD may serve as the MD if necessary. The Relative Percent Difference (RPD) and the Relative Standard Deviation (RSD) of duplicate samples must be less than 24%. Some failures may be qualified using QA Qualification Flow Charts.

19.3.1 For aqueous samples, analyze the parent, duplicate and triplicate at the same dilution.

19.4 Laboratory Control Standard (LCS) or Quality Control Sample (QCS): For every batch of samples, at least one LCS is processed and analyzed. The recovery of the LCS must be within 80-120% for the aqueous NIST 1641e or equilivalent. An LCS Duplicate (LCSD) should accompany the LCS.

19.4.1 A Certified Reference Material (CRM) is the preferred LCS, but a Blank Spike may serve as an LCS if an appropriate CRM does not exist. The spiking level is based on client request, historical data, or a default of mid-curve. A duplicate blank spike must also be prepared as an LCSD.

19.5 Ongoing Precision and Recovery (OPR): An OPR must be analyzed at the beginning and end of each analytical batch, or at the end of each 12-hour shift. The recovery of the OPR must be within 77-123% to be considered in control.

19.6 All calibration standards must be traceable to the original standard source. The calibration curve must be established at the beginning of the analytical run. It must include at least five different concentrations, with the lowest concentration equal to the PQL. The average response factor of each calibration standard is used to calculate the sample values. The RSD of the response factors must be less than 15% of the mean or the calibration fails.

19.7 ICV and CCV control limit is 77-123%. The CCV is analyzed every 10 analyses, and at the end of an analytical run. CCBs are always analyzed after the CCVs.

19.8 Field Blanks: To be compliant with EPA 1631, clients must submit a field blank for each set of samples (samples collected from the same site at the same time, to a maximum of 10 samples).

19.8.1 If no field blanks are submitted by the client, their data will be flagged with "FB-1631." "Required equipment/field/filter blank not submitted by the client. The sample has been analyzed according to 1631E, but does not meet 1631E criteria."

19.9 Method or Preparation Blanks (BLK): Method blanks are used to demonstrate that the analytical system is free from contamination that could otherwise compromise sample results. Method blanks are prepared and analyzed using sample containers, labware, reagents, and analytical procedures identical to those used to prepare and analyze the samples.

19.9.1 A minimum of three 1 % BrCl method blanks per analytical batch are required. Any sample requiring an increased amount of reagent must be accompanied by at least one method blank that includes an identical amount of reagent.

19.9.2 If the result for any 1 % BrCl method blank is found to contain =0.50 ng/L Hg (0.25 ng/L for DOD, 0.1835 for WI-DNR), the system is out of control. Mercury in the analytical system must be reduced until a method blank is free of contamination at the 0.50 ng/L level.

19.9.3 For method blanks containing more than 1% BrCl, the control limit is equal to 0.50 ng/L multiplied by the final preservation percentage of BrCl. For example, for a method blank preserved to 2 % BrCl, the control limit for the blank is 0.50 ng/L * (102/101), or 0.50 ng/L. For 3% BrCl the control limit is (103/101)*0.50ng/L, or 0.51ng/L.

19.10 Instrument Blanks (IBL): A minimum of three instrument blanks must be analyzed with each analytical batch.

19.10.1 If the instrument blank is found to contain more than 0.50ng/L, (0.25 ng/L for DOD, 0.15 for WI-DNR), the system is out of control. The problem must be investigated and remedied and the samples must be reanalyzed.

19.10.1.1 The mean result for all instrument blanks must be <0.25ng/L with a standard deviation of 0.10 ng/L.

19.11 The analytical day must close with a CCV(OPR)/CCB.

19.12 Since the method is done in real-time, it is EFGS' position that a single non-compliant QC sample result does not automatically invalidate a data set. All data points that can be explained and rerun with a passing result can be qualified. If the source of error cannot be corrected for a QC standard that day, none of the data can be validated. In the event that the system becomes out of control during the analysis day, all results bracketed between valid QC data points shall still be considered valid (CCV, OPR, CCB, etc.).

19.13 The Control Limits are established from EPA 1631E.

19.14 Carryover: In 2020 a carryover study was completed monitoring Hg levels up to five times the top calibration point (200 ng/L). No carryover was detected during this study. Any sample ran with a concentration of Hg over 200 ng/L requires a blank be ran directly after to ensure there is no carryover. Additional samples may only be ran after the blank shows a concentration below 0.50 ng/L.

20) Corrective Action

20.1 The data is reviewed as in the QC section (or matrix specific QC section) for all parameters that pass specific requirements. If the data does not meet QC requirements, it is qualified or submitted for reruns. Data may be qualified (based on scientific peer review) by the Group Supervisor, Project Manager, Lab Manager, or QA Officer.

20.1.1 Continuing Calibration Verification (CCV): If a recovery falls outside acceptance criteria, recalibrate the instrument and reanalyze all affected samples since the last acceptable CCV or immediately analyze two additional CCVs. If both CCVs are within acceptance criteria, the samples may be reported without reanalysis and the analysis of the next bracket may continue beginning with a CCB.

20.1.1.1 If either of the two CCVs fails, the analysis must be terminated, the problem resolved, the instrument recalibrated and then all of the affected samples since the last acceptable CCV reanalyzed.

20.2 Control Chart data is generated through LIMS to monitor the performance of the CCV, LCS, MS, and MSD. This is done by the QA department.

20.3 Due to the real-time nature of the CVAFS method, failures must be investigated as they happen. If the source of the problem can be identified, and corrected, the samples may be rerun. If source of problem cannot be isolated, see the Senior Analyst, Group Supervisor, or Laboratory Manager for instructions.

20.4 The Senior Analyst, Group Supervisor, Laboratory Manager, or QA Officer must be informed if QC fails. It is also advisable to always alert the Project Managers.

21) List of Attachments

Table 1: QC Requirements for Total Mercury

QC Parameter	Acceptance Criteria
Initial Calibration Verification (ICV)	79-121% Recovery
Continuing Calibration Verification (CCV)	77-123% Recovery
Ongoing Precision and Recovery (OPR)	77-123% Recovery
Initial Calibration Blank (ICB)/ Continuing Calibration Blank (CCB)	Individually, IBL and CCB ≤0.50ng/L, (≤0.25 ng/L for DOD/DOE, ≤0.15 for WI-DNR), but the mean of all the IBLs shall be ≤0.25ng/L with a standard deviation of ≤0.10ng/L.
Laboratory Control Standard (LCS) or Quality Control Standard (QCS)	80-120% Recovery for NIST1641e; 75-125% for all other CRMs. RPD≤24%
Calibration Curve RSD (Referred to as "Corr. RSD CF" in Excel spreadsheet).	RSD of Calibration Response Factor ≤15%
Lowest Calibration Point	70-130%
1% BrCl Method Blank (BLK)	≤0.50ng/L, (≤0.25 ng/L for DOD, ≤0.15 for WI-DNR)
Matrix Duplicate (MD) and Analytical Duplicate (AD)	≤ 24% RPD
Matrix Spike and Matrix Spike Duplicate (MS/MSD); Analytical Spike (AS) and Analytical Spike Duplicate (ASD)	71-125% Recovery ≤ 24% RPD

EFGS-Q-QD-SOP2710 Class 1 Weight Calibration, Balance Verification, Calibration & Maintenance
EFGS-Q-QD-SOP41510 Volumetric Verification and Pipette and Dispenser Operation & Maintenance
EFGS-Q-QM-QM5805 Quality Assurance Manual
EFGS-S-CS-SOP2794 Ultra-Clean Aqueous Sample Collection
EFGS-S-HS-SOP2991 Waste Disposal Procedures for Client Sample Waste
EFGS-S-IT-WI7061 Eurofins Frontier Global Sciences - Instrument and Instrument Software Inventory
EFGS-S-SB-SOP5139 Cleaning of Sampling Equipment and Bottles for Trace Metals Analysis
EFGS-T-AFS-SOP2822 Determination of Total Mercury in Various Matrices by FI-AFS
EFGS-T-TM-SOP2839 Stock and Prepared Standards
EFHS-S-HS-12066.0004 Chemical Hygiene Plan
EFQA-Q-QD-SOP10098 Procedures for Determining IDLs, MDLs, LODs, PQLs, LOQs, OPRs, IQs, OQs and PQs

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End of document

Document number: **EFGS-T-WC-SOP40897**	Always check on-line for validity. **Determination of Nitrate-Nitrite in Water (EPA 353.2 & SM 4500-NO3- I)**	Level: **Standard Operating Procedure**
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Version: **1**		Organisation level: **4-Business Unit**
Approved by: **KOJ6, R0UO** Effective Date **23-SEP-2021**	Document users: **EFGS_Wet Inst**	Responsible: **EFGS_WET_M**

EUROFINS FRONTIER GLOBAL SCIENCES

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1) Revision Log:

Revision: 1		**Effective Date: This version**
Section	Justification	Changes
throughout	Required	Replaced TestAmerica Seattle with EFGS
Section 12.1 and 12.2	Required	Updated instrument and instrument software
Section 12.7	Required	Updated sample cup type/description
Section 13.1	Required	Updated prep and pH of buffer
Section 13.2	Required	Updated prep of color reagent
Section 13.6.1	Required	Updated to describe Smartchem method for ICAL
Section 14	Required	Updated procedure, shut down and maintenance for new instrument

2) Reference:

2.1 SM 4500-NO3- I. Cadmium Reduction Flow Injection Method. Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998

2.2 EPA 353.2, Determination of Nitrate-Nitrite by Automated Colorimetry. Methods for the Determination of Inorganic Substances in Environmental Samples. EPA 600-R-93/100. August 1993.

3) Cross Reference:

Document	Document Title
EFGS-Q-QM-QM5805	QA Manual
EFGS-S-HS-12066	Chemical Hygiene Plan
CW-E-M-001	Corporate Environmental Health and Safety Manual
EFGS-Q-QD-SOP41490	Use and Maintenance of Glassware
EFGS-S-HS-SOP2991	Waste Disposal Procedures for Client Sample Waste
EFGS-Q-QD-SOP41922	Receipt, Preparation, Storage and Verification of Standards and Reagents and Traceability Protocols
EFGS-Q-QD-SOP2994	Lot Testing for Cleanliness - Verification of Quality Materials
CA-Q-P-003	Calibration Curves and the Selection of Calibration Points
EFGS-Q-QD-SOP2814	Personnel Training and Demonstration of Capability Procedures
CA-Q-S-006	Detection and Quantitation Limits
EFGS-Q-QD-SOP2812	Procedures for Nonconformance, Corrective Actions and the Control of Non-Conforming Work
EFGS-P-DR-SOP2801	Data Review and Validation and Monthly Logbook Reviews
EFGS-Q-QD-SOP41510	Volumetric Verification and Pipette and Dispenser Operation & Maintenance
EFGS-Q-QD-SOP2819	Control Charting and Measurement Uncertainties

4) Scope:

4.1 This procedure is for the determination of nitrite-nitrogen (NO2-N) and nitrate-nitrogen (NO3-N) in both fresh and saline water and wastewater samples. The applicable range of this method is 0.050 to 2.0 mg/L as Nitrogen (N). The range may be extended with sample dilution.

4.2 On occasion, clients may request slight modifications to this SOP. These modifications are addressed on a case by case basis with the supporting demonstration of sensitivity and accuracy (e.g. MDLs, linearity check or PT sample) verified prior to implementation. Any modifications would be written into project special instructions (e.g. Quality Assurance Project Plans), authorized by the laboratory, and mentioned in the report narrative.

5) Basic Principles:

5.1 The method is based upon colormetric principles. The actual analysis is specific for nitrite-nitrogen (NO2-N) by reaction with sulfanilamide and N-(1-napththyl)-ethylenediamine dihydrochloride (NED) to form a highly colored azo dye. Intensity of the dye is proportional to the concentration of NO2 in the test sample. Intensity of the dye is measured colorimetrically and compared to known standards that have been run through the same chemical reaction.

5.2 Nitrate (NO3) analysis is accomplished by first passing the sample through a copper treated cadmium reduction column where NO3 is reduced to NO2. The resulting solution is then reacted as above. If only NO2 is to be determined the cadmium reduction column is by-passed.

6) Reference Modifications:

None.

7) Definitions:

7.1 Batch - A group of 20 or less samples prepared and/or processed together within the same shift using the same reagents. Each batch must contain a minimum QC of one method blank, one laboratory control sample, one matrix duplicate, and one or two matrix spikes.

7.2 Bench Sheet – Work list created from the laboratory's LIMS which includes all of the pertinent information for the batch including sample IDs, quantity of sample, prep date/time, spiking standard(s) ID, QC samples, QC source samples, etc.

7.3 Reduction Efficiency - Proper operation of the cadmium reduction column must be monitored for each analytical run. This is accomplished by analysis of standard samples containing equivalent concentrations of both NO2 and NO3. These standards will be run immediately after the calibration curve verification standards and blanks. Reduction efficiency is calculated as the ratio of measured peak height or area for nitrate relative to that for nitrite.

8) Interferences:

8.1 Glassware. In order to eliminate contaminates from glassware, all glassware must be cleaned in accordance with SOP *EFGS-Q-QD-SOP41490* prior to being utilized for sample preparation.

8.2 Suspended solids may block the flow of sample through the cadmium reduction column. This may be eliminated through filtration (0.45 µm membrane) of the sample.

8.3 The presence of iron, copper or other metals in high concentration may suppress the determination of Nitrite. The addition of EDTA to the ammonium chloride buffer will minimize this problem.

8.4 The presence of oil and grease will coat the surface of the cadmium reduction column. Oil and grease may be removed from the sample by extraction using an organic solvent (Hexane) prior to analysis for Nitrite or Nitrate.

8.5 The laboratory analyst will perform the method in accordance with this SOP. The analyst will resolve non-conformances in methods and data, either individually or with the assistance of the department supervisor or operations manager. Deviations from this SOP must be documented. Bench sheets and raw data must capture information related to a deviation. The laboratory analyst or supervisor will report deviations or non-conforming events to the operations, project and/or QA manager via a non-conformance report.

8.6 The Department Supervisor and/or QA Manager will assist the laboratory analyst in resolving non-conformances.

8.7 The Department Supervisor will review and approve data, data qualifiers, non-conformance reports, methodology, and final reports for all analyses performed in his/her department.

8.8 The QA Manager shall verify adherence to this SOP through annual audits, non-conformance reports, and performance evaluation studies.

9) Safety Precautions, Pollution Prevention and Waste Handling:

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

9.1 Specific Safety Concerns or Requirements

None

9.2 Primary Materials Used

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

Material(1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Hydrochloric Acid	Corrosive Poison	5 ppm-ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Phosphoric Acid	Corrosive	1 mg/m3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sulfuric Acid (Used for pH adjustment)	Corrosive Oxidizer Dehydrator	1 mg/m3 TWA	Inhalation produces damaging effects on the mucous membranes and

	Poison Carcinogen		upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Potassium Nitrate	Oxidizer	None	Causes irritation to the respiratory tract, skin and eyes. Symptoms may include coughing, shortness of breath. Symptoms include redness, itching, and pain.
N-1-naphthylethylenediamine dihydrochloride (NED) (3-0-2)		None listed	Inhalation can cause respiratory tract irritation and delayed pulmonary edema. Skin contact can cause itching.

1 - Always add acid to water to prevent violent reactions.
2 - Exposure limit refers to the OSHA regulatory exposure limit.

9.3 Pollution Control

It is EFGS's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., processing one set of MDLs on all applicable instruments, examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention." Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

9.4 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to *EFGS-S-HS-SOP2991*.

9.4.1 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out:

9.4.1.1 Acidic waste generated by the auto-analyzer is collected in a large carboy labeled "hazardous waste." When the carboy is full it is taken to the waste disposal room and bulked into the acid waste with high metals drum, which is sent out for waste water treatment.

9.4.1.2 Any remaining sample left in the test tubes after analyses are emptied into the carboy labeled "hazardous waste." When the carboy is full it is taken to the waste disposal room and bulked into the acid waste with high metals drum, which is sent out for waste water treatment. The empty test tubes are collected in designated bin for disposable glassware and test tubes. When the bin is full it is disposed into the dumpster.

9.4.1.3 Expired standards/reagents are disposed of according to their hazard classification.

Acids and Bases may be neutralized and disposed through the public sewer system. Ammonium chloride (NH_4Cl), Disodium ethylenediamine tetra-acetic acid dehydrate ($Na_2EDTA\ 2H_2O$) and sulfanilamide and 1 g N-(1-naphthyl)ethylenediamine dihydrochloride (NED) may be disposed of into the toxic solids waste stream, class 9, which is sent out for incineration. Spent Cd Columns are placed with Lab Pack waste.

10) Personnel Training and Qualifications:

10.1 An analyst must perform an initial demonstration of capability (IDOC) that includes four replicates of a secondary source before being qualified to analyze samples without supervision. Continuing DOC will be maintained and monitored via performance on LCSs and other QC samples, as well as obtaining acceptable results on proficiency testing exercises.

10.2 Training is documented by the employee and supervisor, and is kept on file in the QA Office. The employee must read, understand, and by signing the training document, agree to perform the procedures as stated in all Standard Operating Procedures (SOPs) related to this method.

10.3 All employees must also, on a yearly basis, read the Quality Manual (QM), and complete the yearly Ethics training.

10.4 All training documents including IDOCs, CDOCs, Initial QA orientation, and Ethics training are stored by the Quality Assurance Manager in the employees training file for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

10.5 Chemical Safety Training, Compressed Gas Training, Chemical Hygiene Plan documentation, and Shipping of Hazardous goods, are stored by the Health and Safety Officer for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

11) Sample Collection, Preservation and Handling:

11.1 Nitrate or Nitrite

No preservation, hold time of 48 hours, store at 0-6°C in glass or plastic containers.

11.2 Nitrate+Nitrite

Adjust to a pH of <2 with H_2SO_4, hold time of 28 days, store at 0-6°C in glass or plastic containers.

12) Apparatus and Equipment:

12.1 KPM Analytics Smartchem 200. This instrument includes the cadmium reduction column as specified in the method.

12.2 Smartchem operation software, version 3.1.54

12.3 25 mm syringe filter, 0.45 µm (Millipore Millex-HV)

12.4 Auto pipettes, 40 – 200 µL, 200 – 1000 µL and 1 – 5 mL, with appropriate tips, accurate to ±3%

12.5 Analytical balance capable of weighing to 0.0001 g

12.6 Class A Volumetric glassware (50 mL, 100 mL and 1000 mL)

12.7 4mm sample cups, Fisher catalog 02-544-4 or equivalent

13) Reagents and Standards:

All reagents and standards used in this procedure must conform to the requirements specified in SOP *EFGS-Q-QD-SOP2994* Verification of Quality of Materials. Preparation of reagents and standards will be documented in the reagent module in TALS in accordance with SOP *EFGS-Q-QD-SOP41922* Standards. All initial instrument calibrations must be verified with a standard obtained from a second manufacturer. If a standard from a second manufacturer is not available, verification may be achieved by using a different lot from the primary manufacturer. However, the supervisor must obtain written warranties that the two references were not prepared from the same reference material. Certificates of Analysis or Traceability must be labeled with the TALS ID number and attached to the standard information in TALS.

13.1 Ammonium chloride buffer (pH 9.1)

13.1.1. In a 1 L volumetric flask, dissolve 85.0 g ammonium chloride (NH4Cl) and 1.0 g disodium ethylenediamine tetra-acetic acid dehydrate (Na2EDTA 2H2O) in about 800 mL DI water. Dilute to the mark and invert to mix. Adjust the pH to 9.1 with 10 N sodium hydroxide solution. Note: **Reagent grade NH4Cl may be contaminated with NO_x.** Generally this buffer is prepared 4L at one time, and stored in the amber glass bottle expressly for that purpose.

13.1.2. Dilute 200ml of this buffer solution to 1L with DI water, and add to the nitrate buffer bottle on the instrument.

13.2 Sulfanilamide color reagent

13.2.1 Add 150ml of DI water to a 250ml volumetric flask. Add 25ml of concentrated phosphoric acid (H_3PO_4). Mix thoroughly then add 10g of sulfanilamide (4-$NH_2C6H_4SO_2NH_2$), and 0.5g of N-(1-napthyl)ethylenediamine (aka NEDA) ($C_{10}H_7NHCH_2NH_2 \cdot 2HCl$). Stir to dissolve. Finally, add 2ml of Smartchem Probe Rinse Solution, and bring to 250ml volume. Store the solution in amber glass bottle.

13.3 Purchased/Verified NO3 and NO2 standards at 100 mg/L NOx as N

13.4 Initial Calibration Verification Standards (ICV). This will be a mid-range standard (e.g., 1 mg/L) of nitrite nitrogen derived from a source other than that used for generation of the standard curve. Verification standards are generally purchased in a more concentrated form and will have to be diluted to the desired concentration. The verification standards are considered equivalent to Laboratory Control Samples (LCS) or Blank Spikes (BS). ICV should be prepared fresh for each use.

Verification standards are run immediately after the initial calibration (Initial Calibration Verification or ICV) and again after every ten samples processed (Continuing Calibration Verification or CCV). Each verification standard must be followed by a calibration verification blank (ICB and CCB) in order to verify absence of blank contamination and/or carry-over.

13.5 Reduction Efficiency Standards. Prepare a nitrate standard at a concentration equivalent to that of the calibration verification standard, 1 mg/L. The result for this standard will be compared to the ICV nitrite standard in order to assess reduction efficiency of the cadmium column.

13.6 Intermediate working standards (2 mg/L as nitrogen) for generation of the standard curve for nitrite. Add 1.00 mL of 100 mg/L stock to 45 mL DI water contained in a 50 mL volumetric flask. Mix and dilute to volume.

13.6.1 The Smartchem method will automatically create its own calibration curve using the 2mg/L stock solution. If necessary, the calibration points can be created manually using the folloiwing information:

13.6.2 Prepare a series of 5 standards (including a dilution water blank) in 50 mL volumetric flasks by dilution of the 2 mg/L intermediate standard as shown in the table below.

Vol intermediate (mL)	Final volume (mL)	Final conc (mg/L as nitrogen)
0.00	50	0.00
1.25	50	0.05
2.50	50	0.10
12.5	50	0.50

25.0	50	1.00

13.7 Analysts are expected to check those areas where standards are stored on a monthly basis and dispose of expired standards according to sec. 9.4.1.3

13.8 Calibration Standards and Second Source:

13.8.1 Source #1 (NO2 as N 100 µg/mL)

13.8.2 Source #2 (NO2 as N 100 µg/mL)

13.8.3 Source #3 (NO3 as N 100 µg/mL)

13.8.4 Source #4 – Intermediate Working Standard (2 mg/L): Transfer 2.0 mL of source #1 to a 100 mL class A volumetric flask, dilute to the mark with DI water and invert to mix.

13.8.5 Make calibration standards and ICV fresh each day

Standard	**Spike**
Calibration Zero (0.00 mg/L)	No spike
Standard A (0.05 mg/L)	1.25 mL spike of source #4
Standard B (0.10 mg/L)	2.50 mL spike of source #4
Standard C (0.50 mg/L)	12.5 mL spike of source #4
Standard D (1.00 mg/L) (LCS & CCV)	25.0 mL spike of source #4
Standard E (2.00 mg/L)	50.0 mL spike of source #4
ICV (1.00 mg/L)	0.50 mL spike of source #2

13.8.6 Transfer the following volumes with a 200 µL or 2000 µL micropipette into a 50 mL class A volumetric flask, dilute to mark with DI water and invert to mix. Pour standard into a disposable beaker.

14) Procedure:

14.1 Prepare reagents and standards as described in 13.6.1

14.2 Instrument operating parameters are defined in the Smartchem software and in the Smartchem Method number N03-001-A.

14.3 Access the Diagnostic menu in the smartchem software and click on the NO3 tab. Click the large **Prime** button to prime the nitrate reduction unit.

14.4 If needed, filter samples, blank, blank spike and matrix spike(s) through 0.45 µm syringe filters and pour into the disposable sample tubes. Place filtered samples into the auto-sampler rack and enter sample identification as required by the data system. The software automatically adds interim QC checks between every 10 samples and at the end of the run.

14.6 Matrix spikes. Matrix spikes should be conducted for a minimum of 1 in every 10 samples analyzed (2 spikes per batch of 20 samples). Add 0.25ml of the 100ppm nitrite to 50ml of sample. Samples should be spiked at a level high enough to be detected above the original sample, or at least 4 times the MDL. Initiate the sample run.

14.7 During the sequence run, the instrument will first create calibration points and analyze them to determine the calibration curve before any samples are analyzed. The software will display the calibration on the curve when the calculation is complete.

14.7.1 If the calibration curve does not pass ($r^2 < 0.995$) The software will display a warning that the calibration is unacceptable. ***However, the instrument will continue to keep running samples. Stop the analysis and restart if this occurs.***

14.7.2 The calibration data are plotted in a calibration curve area vs. concentration (see corporate SOP CA-Q-P-003, Calibration Curves). Alternatively, calibration curve can be height vs. concentration.

14.8 Shut Down

14.8.1 If the run has successfully completed, print the data and the calibration curve. This data is entered into TALS and the documents scanned as raw data from the batch.

14.8.2 Release pump tubing on the nitrate assembly if the instrument will not be run again within the next 5-7 days.

14.9 Data Review

14.9.1 Upon completion of the analytical run, the primary analyst must review all data for compliance with criteria documented in Section 16.0 and evaluate control charts according to SOP *EFGS-Q-QD-SOP2819*. The primary analyst will enter the data into TALS upon completion of their initial review and update the status.

14.9.2 The Supervisor (or designate) must perform a secondary peer review of the data as entered into TALS. Upon satisfactory completion of this review, the Supervisor (or designate) will update the status of the data set to second level reviewed, indicating the data is ready for reporting to the client.

14.10 Instrument Maintenance

All maintenance and repairs need to be documented in the instrument's maintenance logbook. The logbook must include the instrument name, serial number for each major component (e.g., GC, autosampler) and the date of start-up. When an instrument is not capable of analyzing samples, it needs to be tagged "Out of Service". Logbook entries must include a description of the problem and what actions were taken to address the problem. After an instrument has undergone maintenance or repairs, the system is evaluated using a CCV or ICAL. If the evaluation is successful, the analyst documents in the logbook that the "System returned to control as indicated by a passing CCV" (or ICAL, MB, etc as may be the case).

For specific information on system maintenance and troubleshooting refer to the Smartchem software manual.

If instrument has difficulties with repeated calibration failures, or Low QC recoveries, re-priming the cadmium column may be necessary. Refer to the Diagnostic menu in the Smartchem software for instructions.

15) Calculations:

15.1 Calibration Curves

See corporate SOP CA-Q-P-003, Calibration Curves and the Selection of Calibration Points

15.2 Spike Recovery

$$\% \text{ Recovery} = C_s / C_a \ \text{X} \ 100$$

where:
C_s = Observed spike concentration less sample concentration
C_a = spike level

Background sample concentrations less than the reporting limit will be ignored in this calculation.

15.3 Relative Percent Difference (RPD)

$$\text{RPD} = \frac{|X_1 - X_2|}{(X_1 + X_2)/2} * 100$$

where:

X1 = Concentration of sample analyte
X2 = Concentration of duplicate analyte

16) Quality Assurance/Quality Control and Corrective Action:

At a minimum, an analytical batch must contain a method blank and laboratory control sample for every 20 samples or less. The batch must also contain a matrix spike per 10 samples. A matrix spike duplicate cannot be used in lieu of the second matrix spike. The matrix spikes must be from different sample sources, one matrix spike for each batch of 10.

16.1 Method Blank (MB): An interference-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analysis. The method blank is used to document contamination resulting from the analytical process. In this method, the method blank is equivalent to the ICB/CCB. The method blank should not contain any analyte of interest above one-half the reporting limit. Failure of the method blank, unless able to qualify, requires the batch be reanalyzed.

16.2 Laboratory Control Sample (LCS): A known matrix spiked with the target analytes that is used to document laboratory performance. A LCS is analyzed for each analyte using the same sample preparation and analytical methods used for samples at a frequency of one set per 20 samples or batch. The control limits are 90% - 110% for the LCS sample recovery. If the % recovery of the LCS fail, determine if there is a problem (i.e., the instrument malfunctioned, the solutions are suspect, there was a carryover problem), make the necessary adjustments and reanalyze the LCS. If it fails again, initiate a non-conformance memo and re-prepare the entire batch. If the instrument appears not to have malfunctioned, initiate a non-conformance memo and re-prepare the entire batch.

16.3 Matrix Spike (MS): An aliquot of sample spiked with a known concentration of target analyte. The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix. The control limits for the MS are 90% - 110%. If the % recovery of a MS fails, check for sample related reasons, i.e., large dilutions or high levels of target analytes and qualify the data accordingly.

If an MS is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed.

16.4 Matrix Spike Duplicate (MSD): An interlaboratory split sample which is used to document the precision of a method in a given sample matrix. A matrix duplicate may be either a sample replicate or matrix spike duplicate. The control limits for the RPD are specified in TALS. If the RPD fails, check for sample related reasons, e.g., large dilutions or high levels of target analytes, and qualify the data accordingly.

If a Duplicate is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed.

16.5 Initial Calibration: The correlation coefficient (r) must be > 0.995. If not recalibrate. If re-calibration does not result in meeting the acceptance criteria, consult your supervisor.

The ICV must return a value within 10% of the known concentration.

The ICB must return a value less than the established method reporting limit (MRL).

If the calibration does not meet the acceptance criteria, the instrument must be re-calibrated. If re-calibration does not result in meeting the acceptance criteria, see your supervisor for corrective action.

16.6 Continuing Calibration Verification: The initial calibration should be verified with both a continuing calibration verification (CCV) and a verification blank (CCB), analyzed in that order, every ten analytical samples and at the end of the run. All samples must be bracketed by both acceptable CCV and CCB. Samples that are not bracketed by CCV and CCB within acceptance criteria must be re-analyzed. Acceptance criteria are as defined for the initial calibration.

16.7 All sample results must fit within the range of the standard curve. Samples beyond the calibration range are diluted to the mid point of the curve, if possible, and re-analyzed. The Lachat analyzer provides for either manual or automated dilution of the sample.

16.8 Reduction efficiency checks: Reduction efficiency is calculated as the ratio of area counts for NO3 relative to the area counts for NO2 times 100. This number must be within range of 85% to 115%. If not, recondition and/or replace the cadmium reduction column. Follow manufacture's specifications for any column reconditioning.

16.9 All standard and reagent identifications are recorded on the raw data.

16.10 Anomalous situations occurring during sample preparation and analysis must be documented on the bench sheet, and non-conformance reports must be issued if necessary. Possible anomalous situations resulting in non-conformance memos include loss of a sample or batch QC through spillage or breakage. Please see the SOP for Laboratory Corrective Action Procedures, *EFGS-Q-QD-SOP2812*.

17) Statistical Information and Method Performance:

17.1 A Method Detection Limit (MDL) study must be performed annually according to the current SOP for MDL completion (CA-Q-S-006).

17.2 An IDOC must be completed according to the procedures specified in SOP *EFGS-Q-QD-SOP2814*.

EFGS-P-DR-SOP2801 Data Review and Validation and Monthly Logbook Reviews
EFGS-Q-QD-SOP2812 Procedures for Nonconformance, Corrective Actions and the Control of Non-Conforming Work
EFGS-Q-QD-SOP2814 Personnel Training and Demonstration of Capability Procedures
EFGS-Q-QD-SOP2819 Control Charting and Measurement Uncertainties
EFGS-Q-QD-SOP2994 Lot Testing for Cleanliness - Verification of Quality of Materials
EFGS-Q-QD-SOP41490 Use and Maintenance of Glassware
EFGS-Q-QD-SOP41510 Volumetric Verification and Pipette and Dispenser Operation & Maintenance
EFGS-Q-QD-SOP41922 Receipt, Preparation, Storage and Verification of Standards and Reagents and Traceability Protocols
EFGS-Q-QM-QM5805 Quality Assurance Manual
EFGS-S-HS-SOP2991 Waste Disposal Procedures for Client Sample Waste
EFHS-S-HS-12066.0004 Chemical Hygiene Plan

End of document

Version history

Version	Approval	Revision information	
1	23.SEP.2021		

eurofins	Always check on-line for validity. **Anions by Ion Chromatography [Methods 300.0, 9056A]**	Level: **Standard Operating Procedure**
Document number: **EFGS-T-WC-SOP40994**		
Old Reference: **TA-WC-0161**		
Version: **1.1**		Organisation level: **4-Business Unit**
Approved by: **R0UO** Effective Date: **03-NOV-2022**	Document users: **EFGS_Wet Inst**	Responsible: **EFGS_WET_M**

EUROFINS FRONTIER GLOBAL SCIENCES

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DOCUMENT IS NOT CONTROLLED WHEN PRINTED

1) Revision Log:

Revision: 1		**Effective Date:** This version
Section	**Justification**	**Changes**
throughout	Required	Replaced TestAmerica Seattle with EFGS

2) Reference:

2.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA Publication SW846, 3rd Edition, Final Update IIIB (December 1996), Method 9056A, “Determination of Inorganic Anions by Ion Chromatography”, Revision 0, September 1994.

2.2 Method 300.0, “Determination of Inorganic Anions by Ion Chromatography”, Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio, Revision 2.1, August 1993.

3) Cross Reference:

Document	Document Title
EFGS-Q-QM-QM5805	QA Manual
EFGS-S-HS-12066	Chemical Hygiene Plan
CW-E-M-001	Corporate Environmental Health and Safety Manual
EFGS-S-HS-SOP2991	Waste Disposal Procedures for Client Sample Waste
EFGS-Q-QD-SOP41922	Receipt, Preparation, Storage and Verification of Standards and Reagents and Traceability Protocols
EFGS-Q-QD-SOP2710	Class 1 Weight Calibration, Balance Verification, Calibration & Maintenance
EFGS-T-WC-SOP41035	Percent Moisture/Percent Solids
EFGS-Q-QD-SOP41510	Volumetric Verification and Pipette and Dispenser Operation & Maintenance

EFGS-Q-QD-SOP2812	Procedures for Nonconformance, Corrective Actions and the Control of Non-Conforming Work
EFGS-Q-QD-SOP2814	Personnel Training and Demonstration of Capability Procedures
EFGS-Q-QD-SOP41889	Quality Control Program
CA-Q-P-003	Calibration Curves and the Selection of Calibration Points
CA-Q-S-006	Detection and Quantitation Limits

4) Scope:

4.1 Analytes, Matrix(s), and Reporting Limits

This procedure describes the determination of the anions fluoride, chloride, nitrite, bromide, nitrate, and sulfate in water samples by ion chromatography, based on EPA Method 300.0 and SW-846 Method 9056A.

This procedure can also be applied to leachates from soil samples. The soil leaching procedure is described in Section 15.5.3.

The anions included in this procedure and their routine working ranges for interference-free samples are as follows:

Analyte	CAS Number	Working Range* (mg/L)	Working Range* (mg/kg)
Fluoride	16984-48-8	0.2 – 10	1.0 – 100
Chloride	16887-00-6	0.5 – 100	5.0 – 1000
Nitrite as N	14797-65-0	0.4 – 10	2.5 – 100
Bromide	24959-67-9	0.5 – 10	4.0 – 100
Nitrate as N	14797-55-8	0.2 – 10	1.5 – 100
Sulfate	14808-79-8	1.2 – 100	10 – 1000

*The working range can be extended by dilution of the sample.

The reporting limits for the following analytes are based on a 25 uL injection volume. Current reporting and detection limits are maintained in LIMS (TALS).

NOTE 1: Report nitrite (NO2-) as N, nitrate (NO3-) as N.

NOTE 2: Depending client or project requirements, reporting limits may be higher than those listed in TALS.

NOTE 3: Reporting limits for soils are based on the DI Leach procedure using a soil to water ratio of 1:10. Client-specific soil to water ratios may differ.

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

5) Basic Principles:

5.1 Aqueous samples are all filtered with a 0.45 µm filter (Pall Acrodisc Nylon Membrane #4438T or equivalent). High concentration samples must be diluted for analysis. Soil samples are leached using deionized water in accordance with Section 15.5.3, and the water leach is analyzed.

5.2 A small volume of sample is introduced into the ion chromatograph with the AS-AP autosampler to flush and fill a constant volume loop.

5.3 The sample is pumped through three different ion exchange columns and into a conductivity detector. The first two columns, a guard column and a separator column, are packed with low-capacity, strongly basic anion exchange resin. Ions are separated based on their affinity for the exchange sites of the resin. The last column is a suppressor column that reduces the background conductivity of the eluent to a low or negligible level and converts the anions in the sample to their corresponding acids.

5.4 The separated anions in their acid forms are measured using an electrical conductivity cell. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area and comparing it to a calibration curve generated from known standards.

6) Reference Modifications:

Item	Method	Modification
1	300.0	Method 300.0 specifies that target analytes must be less than the MDL in the Laboratory Reagent Blank. Eurofins Frontier Global Sciences QA SOP (*EFGS-Q-QD-SOP41889*) defines the acceptance limit for the method blank as the laboratory reporting limit (RL) and not the MDL. If specified in client or project requirements, the method blank acceptance limit may be set at the MDL.
2	9056A	Method 9056A specifies bomb combustion

		for solid waste samples. Method 300.0 specifies water leaching for solid samples. This SOP specifies a deionized water leach procedure. In this respect, this SOP complies with Method 300.0, but deviates from Method 9056A.

7) Definitions:

This procedure includes the drinking water QC terminology from Method 300.0 and the solid waste terminology from SW-846 Method 9056A. Where there are two terms for the same concept, the cross reference is explained below. The frequency and evaluation of these QC samples are discussed in Sections 15 and 18.

7.1 Calibration Blank (CB) - A volume of reagent water fortified with the same matrix as the calibration standards, but without the addition of any of the analytes of interest.

7.2 Laboratory Reagent Blank (LRB, also referred to as a Method Blank) - For water samples, which do not require any preparation steps, the calibration blank and the method blank are the same thing. When soils are being analyzed, the method blank consists of the same reagents and preparation steps as applied to samples.

7.3 Laboratory Fortified Blank (LFB, also referred to as a Laboratory Control Sample, LCS) - An aliquot of reagent water or other blank matrix to which known quantities of method analytes are added in the laboratory. The LCS is analyzed exactly like a sample and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of measurements the meet data quality objectives for accuracy and precision.

7.4 Laboratory Fortified Sample Matrix (LFM, also referred to as a Matrix Spike, MS) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix affects the accuracy of the analytical result. The background concentrations of the analyte in the sample matrix must be determined if method analytes or other interference is present in the laboratory environment, the reagent, or the apparatus.

7.5 Instrument Performance Check Solution (IPC, also referred to as Initial and Continuing Calibration Verification Standards, ICV and CCV) - The ICV and CCV serve to monitor instrument drift from the beginning to the end of a given analytical sequence.

7.6 Linear Calibration Range (LCR) - The concentration range over which the instrument response is linear.

7.7 Quality Control Sample (QCS) - The QCS provides an independent verification of the accuracy of calibration standards and instrument performance. For the purposes of this SOP, the second-source ICV provides this verification (see Section 18.12).

8) Interferences:

8.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion or cause the retention times of the other anions to shift. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

8.2 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or an elevated baseline in the ion chromatograms.

8.3 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other low molecular weight organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

8.4 The acetate anion elutes early during the chromatographic run. The retention times of the anions may also differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

8.5 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

9) Safety Precautions, Pollution Prevention and Waste Handling:

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

9.1 Specific Safety Concerns or Requirements

9.1.1 Exercise caution when using syringes with attached filter assemblies. Application of excessive force has, upon occasion, caused a filter disc to burst during the process.

9.2 Primary Materials Used

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

Material (1)	**Hazards**	**Exposure Limit (2)**	**Signs and symptoms of exposure**
Potassium Nitrate	Oxidizer	None	Causes irritation to the respiratory tract, skin and eyes. Symptoms may include coughing, shortness of breath. Symptoms include redness, itching, and pain.
Sodium Fluoride	Poison	2.5 Mg/M3-TWA as F	Highly Toxic. Causes severe irritation to the respiratory tract, symptoms may include coughing, sore throat, and labored breathing. Causes irritation, with redness and pain. Solutions are corrosive. Eye irritant! May cause irritation and serious eye damage. Effects may not appear immediately.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.

9.3 Pollution Control

It is EFGS's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

9.4 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP *EFGS-S-HS-SOP2991*. The following waste streams are produced when this method is carried out.

9.4.1 Waste Streams Produced by the Method

9.4.1.1 IC process waste – aqueous carbonate/bicarbonate eluent waste: Non-hazardous may be disposed of through the public sewer system.

10) Personnel Training and Qualifications:

10.1 An analyst must perform an initial demonstration of capability (IDOC) that includes four replicates of a secondary source before being qualified to analyze samples without supervision. Continuing DOC will be maintained and monitored via performance on LCSs and other QC samples, as well as obtaining acceptable results on proficiency testing exercises.

10.2 Training is documented by the employee and supervisor, and is kept on file in the QA Office. The employee must read, understand, and by signing the training document, agree to perform the procedures as stated in all Standard Operating Procedures (SOPs) related to this method.

10.3 All employees must also, on a yearly basis, read the Quality Manual (QM), and complete the yearly Ethics training.

10.4 All training documents including IDOCs, CDOCs, Initial QA orientation, and Ethics training are stored by the Quality Assurance Manager in the employees training file for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

10.5 Chemical Safety Training, Compressed Gas Training, Chemical Hygiene Plan documentation, and Shipping of Hazardous goods, are stored by the Health and Safety Officer for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

11) Sample Collection, Preservation and Handling:

11.1 Samples are stored at 0-6°C in the container received. Samples should be collected in scrupulously clean glass or polyethylene bottles.

11.2 The holding time for samples submitted for bromide, fluoride, chloride, and/or sulfate analysis is 28 days.

11.3 Holding time for samples submitted for nitrate-N, and/or nitrite-N is 48 hours.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters NO2, NO3	HDPE	50 mLs	Cool 0-6°C	48 Hours	40 CFR Part 136.3
Waters Fl, Cl, Br, SO_4, NO_2/NO_3	HDPE	50 mLs	Cool 0-6°C	28 Days	40 CFR Part 136.3
Soils	Glass	3 grams	Cool 0-6°C	180 Days	N/A

12) Apparatus and Equipment:

12.1 Instrumentation and Software

- Dionex Ion chromatographic system, including: automated sampler; gradient pump; degas module; inline filter; guard column (4 x 50 mm; Ion pac AG14A, AG18 or equivalent); analytical column (4 x 250 mm; Ion pac AS14A, AS18 or equivalent); anion self-regenerating suppressor column; conductivity detector; computer interface; and, computer data handling system.

Note: Other columns may be used. These were the columns in place at the time the SOP was prepared.

- Analytical balance, capable of accurately weighing to the nearest 0.0001 g

12.2 Computer hardware and software

- Computer with a minimum 1GB memory, Pentium 4 processor, 80 G hard drive or equivalent or as recommended by instrument manufacturer.
- Data acquisition/processing system: Chromeleon Version 7.2 or higher
- LIMS system: TALS version 1.0 or higher

12.3 Supplies

- Dedicated Volumetric Flasks (Class A): 2-L (eluent prep) and 100 mL (standards prep)
- Dedicated glass syringes, 10 uL to 1000 uL
- Dedicated glass pipettes, 5 mL to 10 mL
- Adjustable pipettes that are verified quarterly.
- 5 mL Sample vials with filter caps, 2 um filter.
- 0.45 µm filter (Pall Acrodisc Nylon Membrane #4438T or equivalent)

13) Reagents and Standards:

13.1 Document reagent/standards and reagent/standard preparation in TALS using the reagent module as described in SOP *EFGS-Q-QD-SOP41922*.

13.2 Reagent water: Type II, free of anions of interest.

13.3 Eluent solution:

13.3.1 AS14 Eluent concentrate purchased from Dionex, P/N 053560. A 100x dilution is required before use.(20ml concentrate to 2000ml DI water). After dilution the solution is 3.5mM Sodium Carbonte and 1.0 mM Sodium Bicarbonte. Or eluent generator EGCII KOH purchased from Dionex, P/N 058900. This is a cartridge that is installed on the IC and the working eluent is generated as need by the instrument.

13.3.2 Dionex Integrion: AS22 Eluent concentrate, P/N 063965.A 100x dilution is required before use. After 100X dilution the solution is 4.5 mM Sodium Carbonate and 1.4 mM Sodium Bicarbonate.

13.4 Calibration and Continuing Calibration Check Standard solutions, purchased from Environmental Express, Accustandard (Multi-Compound Anion Standard) or another vendor that offers certified standards. The following concentrations are suggestions and differ as required by the instrument*: These standards have a shelf life of 12 to 18 months. They will not be used past the expiration date.

Anion	Concentration, ug/mL
Fluoride	100
Chloride	1000
Bromide	100
Nitrate	100
Sulfate	1000

Anion	Concentration, ug/mL
Nitrite	100

*alternate equivalent source at similar concentrations may be purchased.

13.5 Working ICAL standards. The Environmental Express or Accustandard stock or other certified standard is diluted as described below to give the final concentrations listed below: These working standards are good for 2 months.

Standard Level	Aliquot (mls)	Final Volume (mls)	Concentration (mg/l) Fluoride, Nitrite, Bromide, Nitrate	Concentration (mg/l) Chloride, Sulfate
Level #1	0.0	100	0	0
Level #2**	0.1	100	0.1	1.0
Level #3	0.2	100	0.2	2.0
Level #4	0.5	100	0.5	5.0
Level #5	1.0	100	1.0	10.0
Level #6	2.0	100	2.0	20.0
Level #7*	5.0	100	5.0	50.0
Level #8	10.0	100	10.0	100.0

*This level is used as the CCV standard.
**This level is used as the CCVL standard (if required).

13.6 Initial Calibration Verification (ICV). Purchased from a different source than the ICAL Standards. (Currently Environmental Express standards)

Standard	Aliquot (mls)	Final Volume (mls)	Concentration (mg/l) Fluoride, Nitrite, Bromide, Nitrate	Concentration (mg/l) Chloride, Sulfate
ICV	5.0	100	5	50

13.7 Laboratory Control Spike (LCS). This solution can be made from the standard stock solution or the outside check solution. (Currently Environmental express standards.)

Standard Level	Aliquot (mls)	Final Volume (mls)	Concentration (mg/l) Fluoride, Nitrite, Bromide, Nitrate	Concentration (mg/l) Chloride, Sulfate
LCS	5.0	100	5	50

13.8 Matrix Spike (MS). The matrix spikes are prepared and spiked at the following levels.

Matrix Spike	Aliquot (mls)	Final Volume (mls)	Concentration (mg/l) Fluoride, Nitrite, Bromide, Nitrate	Concentration (mg/l) Chloride, Sulfate
MS	0.25	5	5	50

13.9 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards and dispose of them according to SOP *EFGS-S-HS-SOP2991*.

14) Calibration:

14.1 Ion chromatographic operating parameters and start up procedure:

14.1.1 Flow rate/psi: 1 mLs/minute, approximately 2000 psi.

14.1.2 Conductivity with eluent: ≤ 25 umhos.

14.1.3 Typical retention times: see attached.

14.1.4 Program schedule:

- pull up previous schedule;
- save new schedule under current date;
- delete sample ID and information;
- copy ICAL, ICV, and ICB from original sequence;
- click “yes”;

- click “yes”;
- put CCV (RTC, retention time check), CCB, MB, LCS, LCSD, Samples and Sample Dup into autosampler;
- place vials in AS-DV/AS-AP autosampler;
- click “resume”;
- click on current schedule;
- click on "ok";
- click on "run";
- click on "start"; and
- click on "ok".

14.2 Calibration

14.2.1 Initial Calibration Procedures. Analyze all six working standards and a blank. Typical retention times are listed in Table 2. Prepare separate calibration curves for each anion of interest by plotting peak size in area, or peak height units of standards against concentration values (see corporate SOP CA-Q-P-003, Calibration Curves and the Selection of Calibration Points). Quadratic models are not permitted. The ICV and ICB must be analyzed immediately following the initial calibration. The ICV must agree within 10% of the true value. There are no exceptions (DOD QSM).

14.2.2 Continuing Calibration Procedures. The CCV serves as the daily calibration and retention time check. At the beginning of each analysis batch and after every ten sample analyses, a continuing calibration verification (CCV) standard is analyzed. The CCV must agree within 10% of the true value, or all samples analyzed since the last successful CCV must be reanalyzed. There are no exceptions (DOD QSM).

15) Procedure:

15.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of management to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP *EFGS-Q-QD-SOP2812*. The NCM shall be filed in the project file and addressed in the case narrative.

15.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

15.3 Attachment 2 summarizes the recommended operating conditions for the ion chromatograph. Included in this table are estimated retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the data quality objectives can be met. If operating conditions are different than those listed in the SOP, they should also be documented in the maintenance logbook.

15.4 Check system calibration daily by analyzing a CCV and CCB (see Sections 18.14 and 18.16) and, if required, recalibrate as described in Section 14. A passing CCV must be run daily before any samples are analyzed.

15.5 Sample Preparation

15.5.1 Water samples are prepared for analysis by filtering an aliquot through a 0.45 µm membrane type filter. Record the filter ID in the batch information of the analytical batch.

15.5.2 Check the Balance Logbook to determine if the daily calibration check was completed. If the balance requires a check, verify the calibration as detailed in *EFGS-Q-QD-SOP2710*.

15.5.3 The following extraction should be used for solid materials. Prepare QC using Teflon beads and spike LCS (LCSD) and MS/MSD with appropriate reagent. Record the lot number of the Teflon beads in the batch information in the TALS DI_Leach prep batch. Add an amount of reagent water equal to 10 times the weight of dry solid material taken as a sample. This slurry is mixed together for 30 minutes using a shaker table. Filter the resulting slurry before injecting using a 0.45 u membrane type filter. This can be the type that attaches directly to the end of the syringe. Record the filter ID in the batch information of the analytical batch.

15.6 Sample Analysis

15.6.1 Load samples into 1.5 mL sample vials. The majority of the volume is used to flush the loop; 25 uL of the sample is injected into the instrument.

15.6.2 If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

15.6.3 Compute sample concentration by comparing sample peak response with the standard curve.

Report results in mg/L: NO2- as N

NO3 as N

NOTE: Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases this peak migration may produce poor resolution or identification.

15.8 Following is a typical analytical sequence:

- ICAL and ICV and ICB or CCB and CCV (CCVL if required)
- Method Blank
- LCS and LCSD (if included)
- 7 injections (or 8 if LCSD is not included)
- CCV (CCVL if required) and CCB
- 10 injections
- CCV and CCB
- 10 injections
- CCV and CCB

15.9 Retention Times, Retention Time Windows and Anion Identification

15.9.1 Each sample or QC standard analyte is identified by comparing the peak retention time to the established retention time and retention time window.

15.9.2 The retention time for each compound will be set using the midpoint standard of the calibration curve or on days when a calibration is not performed the initial CCV will be used.

15.9.2.1 Retention time window widths are calculated for each analyte after a major maintenance event (i.e., column change). Using data obtained from the analysis of at least five continuing calibration verification standards over a 24-hour period, calculate the mean retention time and standard deviation for each analyte. Assign a retention-time window of + 3 standard deviations around the mean for each analyte. If the standard deviation is 0.00, use a default retention-time window of 0.3 minutes. Each analyte's peak window is set in Chromeleon software in the component table by the analyst.

15.9.2.2 Calculate the mean and standard deviation for the RTs for each analyte as follows:

$$\text{Mean RT} = \overline{RT} = \frac{\sum_{i=1}^{n} RT_i}{n} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RT_i - \overline{RT}\right)^2}{n-1}}$$

Where:

RTi = Retention time for the ith injection.
n = Number of injections (typically 3).
SD = Standard deviation.

15.9.2.3 The Chromeleon software can chart and calculate retention time mean and standard deviation using the charting program described in the software manual.

15.9.2.4 Set the width of the RT window for each analyte at ± 3 standard deviations of the mean RT for that analyte. If the RT window as calculated above is less than ± 0.3 minute, use ± 0.3 minute as the RT window. This allows for slight variations in retention times caused by sample matrix. The retention time windows are entered into the component table of the Chromeleon software. See attachment 8 for example.

15.9.2.5 The retention time study must be recorded in the instrument maintenance logbook. Note if the retention time was updated based on the standard deviation calculation or was set to the default 0.3 minutes.

15.9.2.6 The width of each window remains the same until new windows are generated following the installation of a new column, or in response to an RT failure.

15.9.3 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, prepare a new sample spiked with a known amount of the anion under question and reanalyze in order to confirm the presence or absence of analyte.

NOTE: Concentration can affect retention time and cause peak migration. Late eluting species, e.g., nitrate and sulfate, exhibit the greatest amount of change, although all anions are affected to some degree. In some cases, this peak migration may produce poor resolution or misidentification. If a peak has shifted outside of its retention time window (as confirmed by a CCV or Matrix Spike), change the window in the software and reprocess the chromatogram. Document the reason for reprocessing the chromatogram along with the date and initials.

15.9.4 Should more complete resolution be needed between peaks, the eluent can be diluted. This will spread out the run but will also cause the later eluting anions to be retained longer. The analyst must determine to what extent the eluent is diluted. This dilution should not be considered a deviation from the method.

15.10 If the response for the peak exceeds the working range of the system, dilute the sample with reagent water and reanalyze.

15.11 Preventative and Routine Instrument Maintenance

All maintenance and repairs need to be documented in the instrument's maintenance logbook. If operating conditions are different than those listed in the SOP, they should also be documented in the maintenance logbook. The logbook must include the instrument name, serial number for each major component (e.g., IC, autosampler, column) and the date of start-up. When an instrument is not capable of analyzing samples, it needs to be tagged "Out of Service". Logbook entries must include a description of the problem and what actions were taken to address the problem. After an instrument has undergone maintenance or repairs, the system is evaluated using a CCV or ICAL. If the evaluation is successful, the analyst documents in the logbook that the "System returned to control as indicated by a passing CCV" (or ICAL, MB, etc as may be the case).

15.11.1 Refer to the ICS-2000 Instrument Manual maintenance procedures in attachment 5.

15.11.2 Refer to the HPIC Instrument Manual maintenance procedures in attachment 6.

15.12 Trouble Shooting

See Attachment 7 of this SOP

16) Calculations:

16.1 Calibration Curves

See corporate SOP CA-Q-P-003, Calibration Curves and the Selection of Calibration Points

16.2 Accuracy

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

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

16.3 Precision (RPD)

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

16.4 Concentration

16.4.1 Liquid samples, report as mg/L, direct instrument reading

16.4.2 Solid samples, report as mg/kg:

$$\text{mg/kg} = \frac{(\text{instrument reading})\ (\text{final volume})}{(\text{sample weight})\ (\text{dry weight correction})}$$

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

17) Statistical Information and Method Performance:

17.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure (see SOP CA-Q-S-006). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

17.2 Demonstration of Capabilities

Analyst initial and continuing Demonstrations of Capability (DOC) are performed before any client samples are analyzed and are updated annually. See SOP *EFGS-Q-QD-SOP2814* for details.

17.3 Training Requirements

See SOP *EFGS-Q-QD-SOP2814* for detailed training requirements.

18) Quality Assurance/Quality Control and Corrective Action:

18.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. The process of establishing control limits, and the use of control charts are described more completely in *EFGS-Q-QD-SOP41889*, Quality Control Program. Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP *EFGS-Q-QD-SOP2812*. This is in addition to the corrective actions described in the following sections.

18.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents.

18.3 Attachment 1 reconciles the various QC requirements specified in the reference methods with the QC requirements specified in this SOP.

18.4 Before analyzing samples, the laboratory must establish a method detection limit (MDL) and the linear calibration range (LCR) as described in Section 17.1. In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument they will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 17.2 for more details.

18.5 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See SOP *EFGS-Q-QD-SOP41889* for further details.

18.6 Method Blank (same as Laboratory Reagent Blank, LRB)

A method blank (MB) is required with every batch of 20 or less samples. The MB is deionized water taken through the procedure as if it were a sample.

Acceptance Criteria: The MB must not contain anions of interest above one-half the reporting limit or above one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL).

NOTE: Some programs (e.g., DoD) require control of method blanks to have a concentration less than or equal to one-half of the RL. Some programs (LaMP) and Method 300.0 and Method 9056A require no detections in the method blank more than 10% of the low limit calibration check solution. This can not be obtained in most cases. EFGS will only evaluate the method blank to 1/2 the RL or Project DQOs and when specific DQOs are not provided by the client the RL will be defined as the DQO.

Corrective Action: If the method blank exceeds allowable levels, laboratory contamination is suspected and corrective action must be taken before continuing. All samples associated with the failed blank must be reanalyzed.

18.7 Laboratory Control Sample (same as Laboratory Fortified Blank, LFB)

One Laboratory Control Sample (LCS) is required with each analytical batch. Depending on client or project requirements, an LCS duplicate may also be analyzed. The LCS and LCSD are prepared as described in section 15.5 and spiked as described in section 13.7. An LCS that is determined to be within acceptance criteria effectively demonstrates that the analytical system is in control and validates system performance for the samples in the associated batch.

If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed.

Acceptance Criteria: For Method 300.0, the LCS recovery for each analyte of interest must be within 90-110%. For Method 9056A, the LCS recovery for each analyte of interest must be within laboratory statistical control limits, not to exceed 80-120%. The absolute value of the relative percent difference (RPD) between the LCS and LCSD must be </= 15%. Control limits are maintained in the LIMS. The process of establishing control limits is described in more detail in SOP *EFGS-Q-QD-SOP41889*.

NOTE: Some programs (e.g., DoD) require program defined control limits.
Corrective Action: If the LCS recovery falls outside of the established control limits, and/or when the RPD for the LCS/LCSD exceeds the RPD limit, check instrument conditions and the standards being used for problems. Correct any problems before continuing. Reanalyze all samples associated with the failed LCS.

18.8 Matrix Spike / Matrix Spike Duplicate (MS/MSD, same as Laboratory Fortified Matrix)

For Method 9056A, one MS/MSD pair is required with each analytical batch of 20 or fewer samples. For Method 300.0, one MS is required for every 10 routine samples. The MS and MSD are prepared as described in section 15.5 and spiked as described in Section 13.8.

If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed.

Acceptance Criteria: For Method 300.0, the MS and MSD recovery for each analyte of interest must be within 90-110%. For Method 9056A, the MS and MSD recovery for each analyte of interest must be within laboratory statistical control limits. The absolute value of the relative percent difference (RPD) between the MS and MSD must be </= 15%. Control limits are maintained in the LIMS. The process of establishing control limits is described in more detail in SOP *EFGS-Q-QD-SOP41889*.

Corrective Action: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures:

- Check calculation and instrument performance;

- Verify, if possible, that the MS and MSD were spiked correctly;

- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering compounds seen on chromatograms, or interference demonstrated by prior analyses); and

- Document the failure in an NCM and note it on the final report.

For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample non-homogeneity; and document the failure in an NCM.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

18.9 Matrix Duplicate

For clients requiring a matrix duplicate, one will be processed in each analytical batch of 20 or fewer samples.

If a duplicate is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed.

Acceptance Criteria: The relative percent difference (RPD) between the sample and Duplicate must be less than 10%, or less than the established control limit, depending on project requirements. The process of establishing control limits is described in more detail in SOP *EFGS-Q-QD-SOP41889*. Control limits are maintained in the LIMS.

Corrective Action: See corrective actions under section 18.8.

18.10 Instrument QC

18.11 Initial Calibration (ICAL)

18.11.1 An initial calibration is performed every three months, or as needed, based on instrument performance and maintenance.

18.11.2 Calibrate the instrument at seven levels. See Section 13.5 for preparation of calibration standards. Note that it is generally NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or linear range is adjusted accordingly. The only exception is that a level may be removed from the calibration if the reason is clearly documented, for example a cracked tube, and a minimum of five levels remain (six for quadratic curves). Curves may not be forced through the origin.

18.11.3 Construct a calibration curve using a least squares linear regression or a quadratic curve (see corporate SOP CA-Q-P-003, Calibration Curves and Attachment 4 for the verbiage from the method update rule).

Acceptance Criteria: Six calibration points must be used when using a quadric curve. The r2 of a 2nd order quadratic curve must be 0.990 or greater. The absolute value of the correlation coefficient must be 0.995 or greater.

Corrective Action: If the correlation coefficient is less than the acceptance limit, recheck instrument conditions and calibration standards. Samples cannot be analyzed until the initial calibration is successful.

18.12 Initial Calibration Verification (ICV)

The second-source ICV and is described in Section 7.6; it is analyzed immediately following the ICAL.

Acceptance Criteria: The ICV recovery for each anion must be 90-110%. The retention time for each analyte in the ICV must be within =/- 10% of the established retention time for that analyte.

Corrective Action: If the recovery and/or retention time is outside of the acceptance limits, repeat the test. If the test fails on the second attempt, then the problem must be investigated and the instrument recalibrated for the failed analyte(s). There are no exceptions (DOD QSM).

18.13 Initial Calibration Blank (ICB)

An ICB is analyzed following the ICV.

Acceptance Criteria: The result must be less than one-half the reporting limit.

Corrective Action: If the blank is above the acceptance limit, check for carryover or the need for instrument maintenance. The instrument must be recalibrated, and all samples analyzed since the last successful CCV must be reanalyzed.

18.14 Continuing Calibration Verification (CCV, same as IPC in Method 300.0)

A CCV is required at the beginning of every run, after every 10 or fewer samples and after the last sample.

Acceptance Criteria: The CCV recovery must be 90-110%. The retention time for each analyte in the CCV must be within of the established retention time window for that analyte.

Corrective Action: If the recovery and/or retention time is outside of the acceptance limits, all samples analyzed since the last successful CCV must be reanalyzed. There are no exceptions (DOD QSM).

18.15 Low-Level Continuing Calibration Verification (CCVL)

For any samples analyzed under the BP LaMP program a CCVL is required at the beginning of every run, after every 10 or fewer samples and after the last sample.

Acceptance Criteria: The CCVL recovery must be 90-110%. The retention time for each analyte in the CCVL must be within the established retention time window for that analyte.

Corrective Action: If the recovery and/or retention time is outside of the acceptance limits, all samples analyzed since the last successful CCVL must be reanalyzed.

18.16 Continuing Calibration Blank (CCB)

A CCB is analyzed after each CCV.

Acceptance Criteria: The result must be less than one-half the reporting limit.

Corrective Action: If the blank is above the acceptance limit, check for carryover or the need for instrument maintenance. All samples analyzed since the last successful CCB must be reanalyzed.

Note: CCVs need to be followed by a CCB. CCV cannot be preceded by a CCB, unless a blank is analyzed before each sample in the bracket.

18.17 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

19) List of Attachments:

Attachment 1: Quality Control Summary

Attachment 2: Suggested Standard Instrument Operating Parameters

Attachment 3: Example Ion Chromatogram

Attachment 4: Verbiage on quadratic calibrations from the method update rule (MUR)

Attachment 5: Dionex Integrion HPIC Maintenance

Attachment 6: Dionex ICS-2000 Maintenance

Attachment 7: Troubleshooting Scheme for Anion Chromatograph Systems

Attachment 8: Example Screen Shot Showing Retention Times and RT Windows

Attachment 1: Quality Control Summary

QC Samples	Frequency	Acceptance Criteria	Corrective Action	Reference Method Equivalent
Retention time (RT) window width determination	At method set-up and after major maintenance (e.g. column change)	RT width is ± 3 times std dev for each analyte RT over a 24-hour period.	NA	NA
Minimum 3-point Initial Calibration	Initial calibration prior to sample analysis	One of the options below: Linear least squares regression: r ≥ 0.990 **Linear least squares regression for DoD: r ≥ 0.995** For quadratic regression: r^2 ≥ 0.990.	Terminate analysis; correct the problem; recalibrate. Problem must be corrected. No samples may be run until ICAL has passed.	NA
Initial Calibration Verification (ICV)	Immediately following the initial calibration.	90 - 110% of true value RT must be ± 10% of established RT.	Repeat once, and recalibrate and reanalyze if it fails a second time.	QC Reference Sample (9056A) IPC and QCS (300.0)
Initial Calibration Blank (ICB)	After the ICV and prior to sample analysis.	< RL. **For DoD, LaMP**: ≤ ½ RL.	Re-prepare and reanalyze	Calibration Blank (300.0)
Method Blank (MB)	1 per QC batch	σ; RL **For DoD, LaMP**: ≤ ½ RL.	See Section 9.6 or SOP *EFGS-Q-QD-SOP41889*.	LRB (300.0)
Laboratory Control Sample (LCS)	1 per QC batch	Within laboratory historical limits but not to exceed 90-110% recovery **For DoD**: control limits may not exceed the limits listed in the QSM.	Recalibrate and reanalyze all samples associated with unacceptable LCS	LFB (300.0)
Matrix Spike Sample/Matrix Spike Duplicate (MS/MSD)	1 MS/MSD pair per QC batch for 9056A. 1 MS/MSD pair per 10 samples for 300.0.	%R within laboratory historical limits but not to exceed 80-120% recovery and RPD σ; laboratory historical limits not to exceed 20%. **For DoD**: limits listed in the QSM.	If LCS and CCVs are in control, then document in an NCM, unless project requires reanalysis.	Matrix Spike (9056A) LFM (300.0)
Continuing Calibration Verification (CCV)	Between each group of 10 injections and at the end of the analytical sequence.	90 - 110% of true value	Repeat. If repeat fails, reanalyze all samples since the last acceptable CCV.	Mid-range Calibration Standard (9056A) IPC (300.0)
Continuing Calibration Blank (CCB)	Between each group of 10 injections and at the end of the analytical sequence	< RL **For DoD**: ≤ ½ RL.	Repeat. If repeat fails, reanalyze all samples since the last acceptable CCB.	Calibration Blank (300.0)
Sample Duplicate	**For DoD,** 1 per 10 samples. BP LaMP 1 per 20 - may use either MSD or SDUP	%D σ; 10% between sample and duplicate	Correct problem and reanalyze sample and duplicate.	NA

Attachment 2: Suggested Standard Instrument Operating Parameters

Typical Retention Times

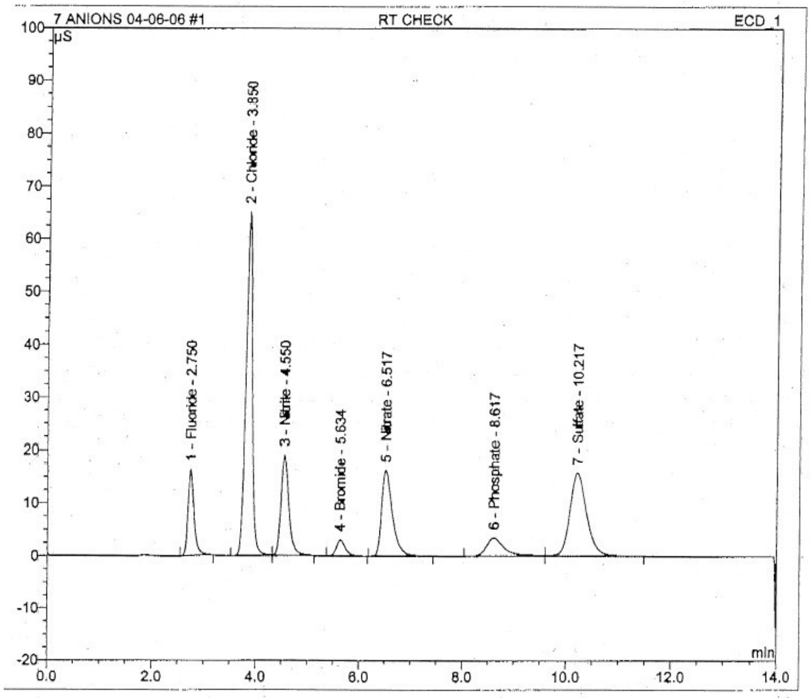
Analyte	Peak #	Retention Time (Minutes)
Fluoride	1	3.3
Chloride	2	4.7
Nitrite as N	3	5.7
Bromide	4	7.1
Nitrate as N	5	8.1
Sulfate	7	11.9

Instrument Conditions

Anion Guard Column	AG14A, AS18 or equivalent (4X50mm)
Anion Separator Column	AS14A, AS18 or equivalent (4X250mm)
Supressor Device	SRS 300 Self-Regenerating Suppressor (4mm)
Pump Rate	1.0 mL/min
Sample Loop	25 µL
Eluent	1.0 mM sodium bicarbonate, 32.0 mM sodium carbonate or Potassium hydroxide cartridge
Detector Output	Baseline conductivity should be between 24 - 28 µS prior to sample analysis.

Anion Guard Column	AG22 fast (4mm)
Anion Separator Column	AS22 fast (4x150mm)
Supressor Device	AERS Carbonate (4mm)
Pump Rate	1.0 mL/min
Sample Loop	25 µL
Eluent	4.5 mM sodium bicarbonate, 1.4 mM sodium carbonate
Detector Output	Baseline conductivity should be between 18 - 22 µS prior to sample analysis.

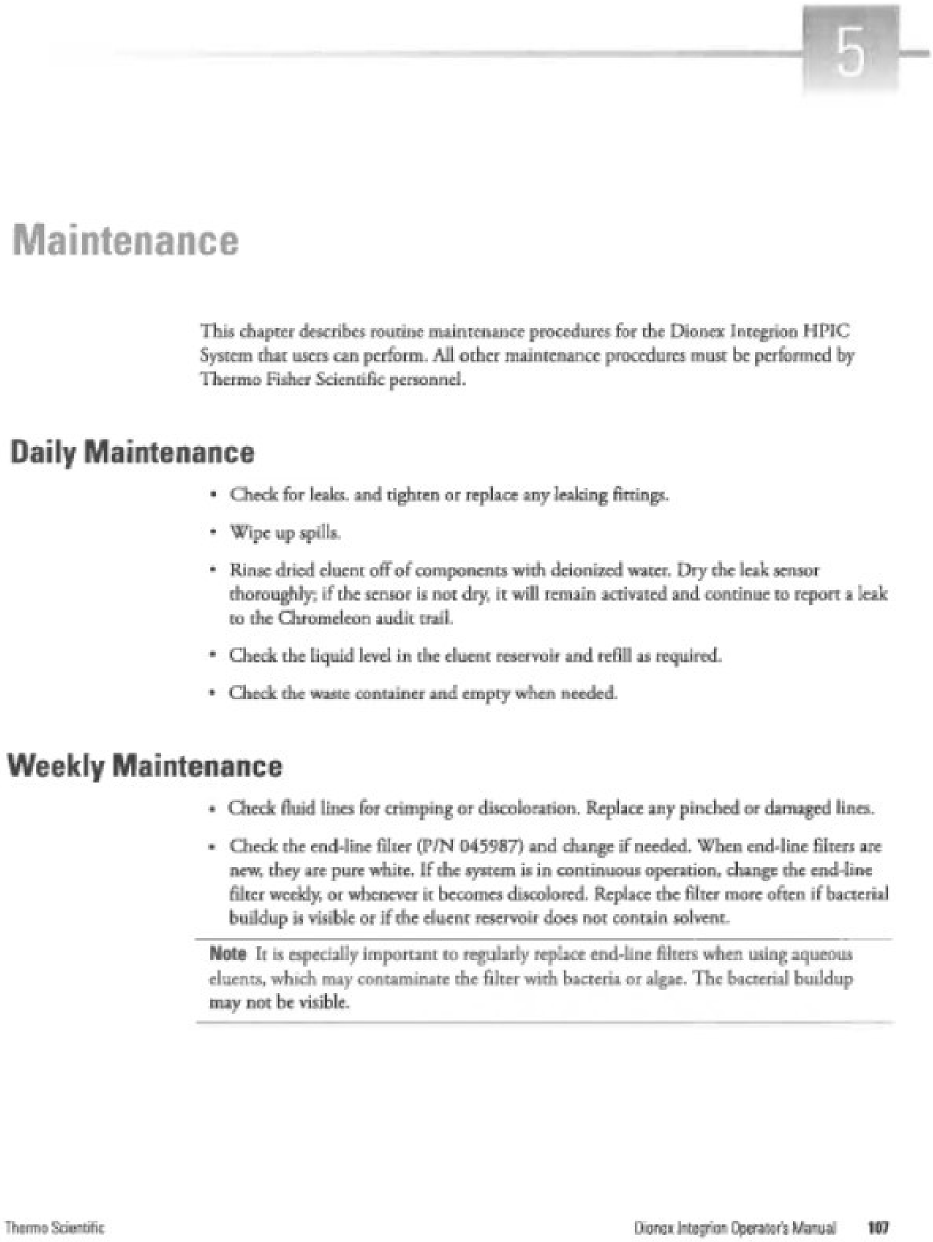
Attachment 3: Example Ion Chromatogram



Attachment 4: Verbiage on quadratic calibrations from the method update rule (MUR)

Changes in calibration model. (A) Linear calibration models do not adequately fit calibration data with one or two inflection points. For example, vendor-supplied data acquisition and processing software on some instruments may provide quadratic fitting functions to handle such situations. If the calibration data for a particular analytical method routinely display quadratic character, using quadratic fitting functions may be acceptable. In such cases, the minimum number of calibrators for second order fits should be six, and in no case should concentrations be extrapolated for instrument responses that exceed that of the most concentrated calibrator. Examples of methods with nonlinear calibration functions include chloride by SM4500–Cl–E–1997, hardness by EPA Method 130.1, cyanide by ASTM D6888 or OIA1677, Kjeldahl nitrogen by PAI–DK03, and anions by EPA Method 300.0.

Attachment 5: Maintenance for Dionex HPIC



5

Maintenance

This chapter describes routine maintenance procedures for the Dionex Integrion HPIC System that users can perform. All other maintenance procedures must be performed by Thermo Fisher Scientific personnel.

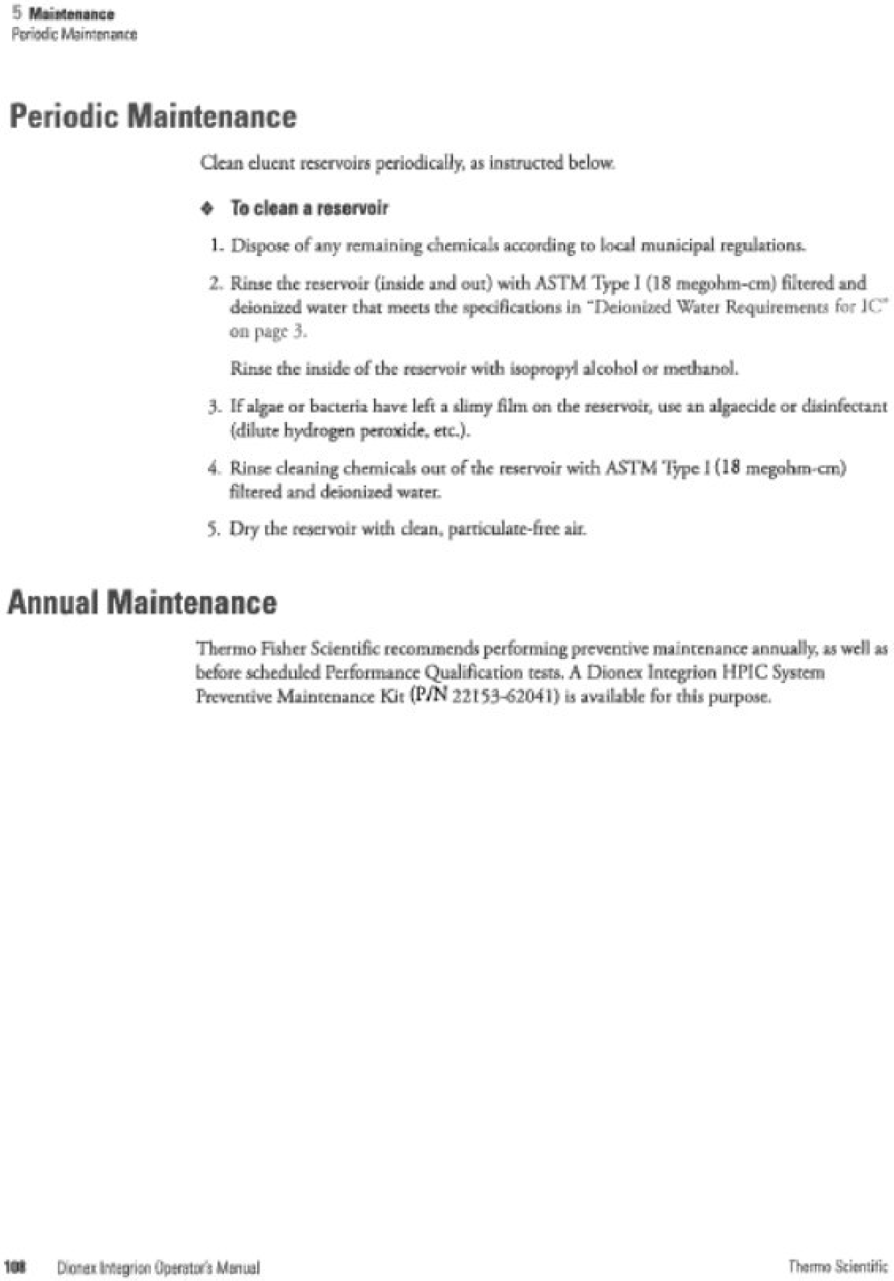
Daily Maintenance

- Check for leaks, and tighten or replace any leaking fittings.
- Wipe up spills.
- Rinse dried eluent off of components with deionized water. Dry the leak sensor thoroughly; if the sensor is not dry, it will remain activated and continue to report a leak to the Chromeleon audit trail.
- Check the liquid level in the eluent reservoir and refill as required.
- Check the waste container and empty when needed.

Weekly Maintenance

- Check fluid lines for crimping or discoloration. Replace any pinched or damaged lines.
- Check the end-line filter (P/N 045987) and change if needed. When end-line filters are new, they are pure white. If the system is in continuous operation, change the end-line filter weekly, or whenever it becomes discolored. Replace the filter more often if bacterial buildup is visible or if the eluent reservoir does not contain solvent.

Note It is especially important to regularly replace end-line filters when using aqueous eluents, which may contaminate the filter with bacteria or algae. The bacterial buildup may not be visible.



Periodic Maintenance

Clean eluent reservoirs periodically, as instructed below.

◆ **To clean a reservoir**

1. Dispose of any remaining chemicals according to local municipal regulations.
2. Rinse the reservoir (inside and out) with ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications in "Deionized Water Requirements for IC" on page 3.

   Rinse the inside of the reservoir with isopropyl alcohol or methanol.
3. If algae or bacteria have left a slimy film on the reservoir, use an algaecide or disinfectant (dilute hydrogen peroxide, etc.).
4. Rinse cleaning chemicals out of the reservoir with ASTM Type I (18 megohm-cm) filtered and deionized water.
5. Dry the reservoir with clean, particulate-free air.

Annual Maintenance

Thermo Fisher Scientific recommends performing preventive maintenance annually, as well as before scheduled Performance Qualification tests. A Dionex Integrion HPIC System Preventive Maintenance Kit (P/N 22153-62041) is available for this purpose.

Attachment 6: Maintenance for Dionex ICS-2000

4. Click the **Add** button on the **Batch List** tab and add the sequence you created in Step 2. See Figure 3-14 for an example.

Name	Operator
ICS-2000\Test	amarquardt

Figure 3-14. Batch Dialog Box with an Example Sequence Added to the Batch List

5. To start the batch, click **Start**.

3.12 Maintenance

This section describes routine maintenance procedures that users may perform. All other maintenance procedures must be performed by Dionex personnel.

As Needed

- Regularly check the eluent reservoir and refill when needed.

Daily

- Check the ICS-2000 component mounting panel (see Figure 2-8) for leaks or spills. Wipe up spills. Isolate and repair leaks (see Section 4.2). Rinse off any dried eluent with deionized water.
- Check the waste container daily and empty when needed.

Weekly

- Once a week, check fluid lines for crimping or discoloration. Relocate any pinched lines. Replace damaged lines.
- Check the junctions between the pump heads and the pump casting for evidence of liquid leaks. If the piston seal wash tubing is not connected, check the drain tubes at the rear of the pump heads for evidence of moisture. Normal friction and wear may gradually result in small liquid leaks around the piston seal. If unchecked, these leaks can gradually contaminate the piston housing, causing the pump to operate poorly. If leaks occur, replace the piston seals (see Section 5.6).
- Check the end-line filter (P/N 045987) and change if needed. When new, end-line filters are pure white. If the system is in continuous operation, change the end-line filter weekly, or whenever it becomes discolored. Replace the filter more often if bacterial buildup is visible or if the eluent does not contain solvent.

NOTE It is especially important to regularly replace end-line filters when using aqueous eluents, which may contaminate the filter with bacteria or algae. The bacterial buildup may not be visible.

Every Six Months

- Calibrate the cell (see Section 5.1.3).
- Calibrate the vacuum degas assembly (see Section 5.1.5).
- Replace the pump piston rinse seals and piston seals (see Section 5.6).

Yearly

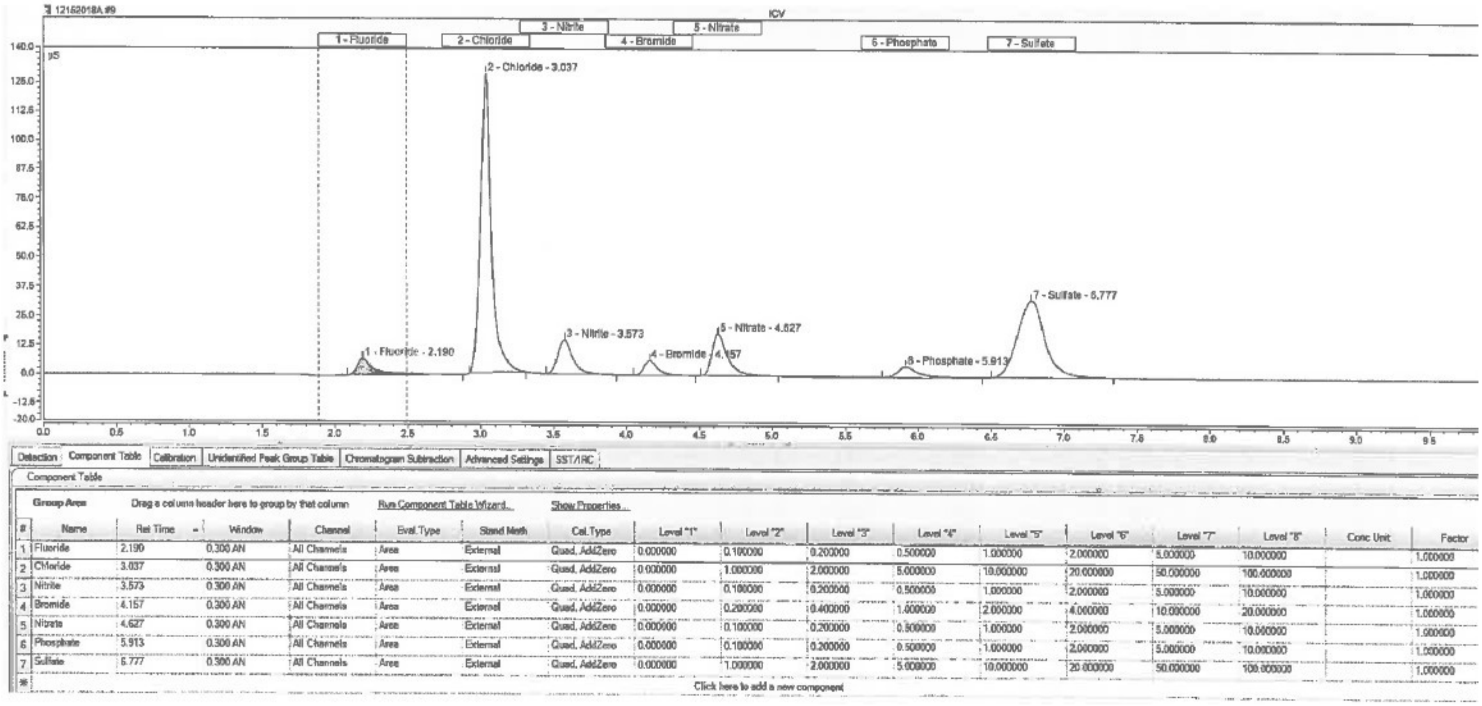
- Rebuild the injection valve (see Section 5.4).
- Replace the AS40 Automated Sampler tip and tubing. The AS40/ASM Maintenance Kit (P/N 055647) contains all of the components required to replace the sampling tip and the tubing between the tip and the injection valve. Instructions are included with the kit.
- Perform the AS or AS50 (USB) preventive maintenance procedure. The AS or AS50 (USB) Maintenance Kit (P/N 060581) contains all of the required components and instructions for performing the procedure.

Attachment 7: Troubleshooting Scheme for Anion Chromatograph Systems

Troubleshooting Scheme for Anion Chromatography Systems

Symptom	Possible source of error	Action
• High background conductivity, but a relatively low noise.	• The eluent has been contaminated with an anion of a strong acid.	• Check the electrolytic conductivity of the deionised water used to prepare the eluent and standards, and for dilution of the samples. • Prepare a new eluent (→ page 5). Change also the stock solution.
• High background conductivity in combination with high noise.	• The suppressor reaction does not work. • High backpressure in the detector. • The membrane of the suppressor is worn out or is inhibited by metal ions or hydrophobic cations.	• Check that the regenerant solution flows in a closed loop, as it should. • Check the detector by manually flushing water through the cell. Clean if necessary. • Wash the membrane with an alkaline magnesium EDTA solution (→ page 15). If this do not restore the full function of the suppressor, it is worn out and needs to be replaced with a new unit.
• High (often regular) noise with normal background level.	• Trapped air or malfunctioning valves in the eluent pump.	• Remove air from the pump and carefully degas the eluent (→ page 5). • Rinse with isopropanol (→ page 7). Change the pump valves if these are worn out.
• The sensitivity for anions of weak acids has decreased.	• Incomplete suppression. • The pH after the suppressor is too low.	• Check the regenerant flow – thereafter check if the cartridge lifetime is exceeded. • Check the pH of the eluate after the suppressor (→ page 14) and compare to the SOP (→ page 20).
• The sulfate or fluoride peak has decreased height and broadened, while other peaks are as usual.	• The suppressor or the separator column is contaminated by metal ions.	• Wash the suppessor membrane with an alkaline magnesium EDTA solution (→ page 15). If the problem remains the suppressor needs to be changed. • Run a clean-up procedure on the separator column. Follow the manufacturers instructions. Disconnect the column from the system before starting the clean-up.
• The baseline is drifting.	• The system has not stabilised yet. • Leakage in the flow system, temperature variations, debris on the column filters.	• Wait until the baseline has stabilised (Note: This can take a relatively long time). • Choose the simplest action first. Try also shutting off all pumps one by one. The injector can be a tricky source off error (→ page 8).
• Negative peaks.	• High background. • The cables between the detector and the recorder/integrator are connected with opposite polarity (signal to ground & vv).	• Check the regenerant flow, and if the regenerant cartridge has been consumed. • Make sure that – and + out from the detector connects to the corresponding terminals on the recorder/integrator. Possibly switch the cords between – and +.
• High backpressure.	• The column inlet filter is clogged. • The injector needs service or is placed in a position between *Load* and *Inject*.	• Disconnect the column. Compare with normal backpressure in SOP. • If the pressure remains when the column is disconnected the injector is the source. Check the position of the handle and perform service if necessary (→ page 8).

Attachment 8: Example Screen Shot Showing Retention Time and RT Windows



#	Name	Ret Time	Window	Channel	Eval.Type	Stand Meth	Cal.Type	Level "1"	Level "2"	Level "3"	Level "4"	Level "5"	Level "6"	Level "7"	Level "8"	Conc Unit	Factor
1	Fluoride	2.190	0.300 AN	All Channels	Area	External	Quad, AddZero	0.000000	0.100000	0.200000	0.500000	1.000000	2.000000	5.000000	10.000000		1.000000
2	Chloride	3.037	0.300 AN	All Channels	Area	External	Quad, AddZero	0.000000	1.000000	2.000000	5.000000	10.000000	20.000000	50.000000	100.000000		1.000000
3	Nitrite	3.573	0.300 AN	All Channels	Area	External	Quad, AddZero	0.000000	0.100000	0.200000	0.500000	1.000000	2.000000	5.000000	10.000000		1.000000
4	Bromide	4.157	0.300 AN	All Channels	Area	External	Quad, AddZero	0.000000	0.200000	0.400000	1.000000	2.000000	4.000000	10.000000	20.000000		1.000000
5	Nitrate	4.627	0.300 AN	All Channels	Area	External	Quad, AddZero	0.000000	0.100000	0.200000	0.500000	1.000000	2.000000	5.000000	10.000000		1.000000
6	Phosphate	5.913	0.300 AN	All Channels	Area	External	Quad, AddZero	0.000000	0.100000	0.200000	0.500000	1.000000	2.000000	5.000000	10.000000		1.000000
7	Sulfate	6.777	0.300 AN	All Channels	Area	External	Quad, AddZero	0.000000	1.000000	2.000000	5.000000	10.000000	20.000000	50.000000	100.000000		1.000000

12066 Chemical Hygiene Plan
2710 Class 1 Weight Calibration, Balance Verification, Calibration & Maintenance
2812 Procedures for Nonconformance, Corrective Actions and the Control of Non-Conforming Work
2814 Personnel Training and Demonstration of Capability Procedures
2991 Waste Disposal Procedures for Client Sample Waste
41035 Percent Moisture/Percent Solids [Method SM 2540 G]
41510 Volumetric Verification and Pipette and Dispenser Operation & Maintenance
41889 Quality Control Program
41922 Receipt, Preparation, Storage and Verification of Standards and Reagents and Traceability Protocols
5805 Quality Assurance Manual

End of document

Version history

Version	Approval	Revision information	
1	10.SEP.2021		
1.1	01.NOV.2022		

eurofins	Always check on-line for validity. **Determination of Solids in Waters and Wastes [Methods SM 2540B, SM 2540C, SM 2540D, EPA 160.1, 160.2 and 160.3]**	Level: **Standard Operating Procedure**
Document number: **EFGS-T-WC-SOP41106**		
Old Reference: **TA-WC-0125**		
Version: **1**		Organisation level: **4-Business Unit**
Approved by: **KOJ6, R0UO** Effective Date **04-OCT-2021**	Document users: **EFGS_WET grav**	Responsible: **EFGS_WET_M**

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DOCUMENT IS NOT CONTROLLED WHEN PRINTED

1) Revision Log:

Revision: 1	**Effective Date:** This version	
Section	**Justification**	**Changes**
throughout	Required	Replaced TestAmerica Seattle with EFGS

2) Reference:

2.1 Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.

2.2 Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005.

2.3 Method 160.1, "Residue, Filterable (Gravimetric, Dried at 180°C)", Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio.

2.4 Method 160.2, "Residue, Non-Filterable (Gravimetric, Dried at 103-105°C)", Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio.

2.5 Method 160.3, “Residue, Total (Gravimetric, Dried at 103-105°C)”, Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio.

3) Cross Reference:

Document	Document Title
EFGS-Q-QM-QM5805	QA Manual
EFGS-S-HS-12066	Chemical Hygiene Plan
CW-E-M-001	Corporate Environmental Health and Safety Manual
EFGS-S-HS-SOP2991	Waste Disposal Procedures for Client Sample Waste
EFGS-Q-QD-SOP41922	Receipt, Preparation, Storage and Verification of Standards and Reagents and Traceability Protocols
EFGS-Q-QD-SOP2710	Class 1 Weight Calibration, Balance Verification, Calibration & Maintenance
EFGS-T-WC-SOP41035	Percent Moisture/Percent Solids
EFGS-T-WC-SOP40959	Settleable Matter or Settleable Solids (SM 2450F)
EFGS-Q-QD-SOP2812	Procedures for Nonconformance, Corrective Actions and the Control of Non-Conforming Work
EFGS-Q-QD-SOP2814	Personnel Training and Demonstration of Capability Procedures
EFGS-Q-QD-SOP41889	Quality Control Program

4) Scope:

4.1 This SOP is applicable to the determination of total solids, total suspended solids and total dissolved solids using gravimetric techniques. This SOP is applicable to drinking, surface, and saline waters and domestic and industrial wastes.

4.2 The methods cover a practical range of 10 mg/L to 20,000 mg/L (TSS: 2 mg/L - 20,000 mg/L). As a practical matter, the final residue weight should be limited to about 200 mg.

4.3 The procedure for settleable solids is in SOP # *EFGS-T-WC-SOP40959*.

4.4 The procedure for percent moisture in solid samples is in SOP # *EFGS-T-WC-SOP41035*.

5) Basic Principles:

5.1 Total Solids (TS): A well-mixed aliquot of the sample is quantitatively transferred to a preweighed evaporating dish and evaporated to dryness at 103-105°C. The increase in weight over that of the empty dish represents the total solids.

5.2 Total Dissolved Solids (TDS): A well-mixed sample is filtered through a glass fiber filter. The filtrate is quantitatively transferred into a preweighed evaporating dish and is evaporated to dryness and then dried to constant weight at 180 °C. The increase in weight over that of the empty dish represents the total dissolved solids. The filter from this procedure may also be used for TSS determination.

5.3 Total Suspended Solids (TSS): A well-mixed sample is filtered through a pre-weighed glass fiber filter. The residue on the filter is dried to constant weight at 103-105 °C. The increase in weight over that of the pre-weighed filter represents the TSS content. The filtrate from this procedure may be used for TDS determination.

6) Reference Modifications:

Samples are shaken vigorously and then a sample aliquot is quickly poured out. This is a deviation from the Standard Method of stirring the sample and measured volume is pipette on to the filters.

7) Definitions:

7.1 Total Solids (TS): The term applied to the residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at 103-105°C. Total solids includes "total suspended solids," the portion of solids retained by a filter, and "total dissolved solids," the portion that passes through the filter.

7.2 Total Dissolved Solids (TDS): Those solids capable of passing through a glass fiber filter and dried to constant weight at 180 °C. TDS is also referred to as filterable residue.

7.3 Total Suspended Solids (TSS): Those solids which are retained by a glass fiber filter and dried to constant weight at 103-105 °C. TSS is also referred to as non-filterable residue.

7.4 Aliquot: A representative portion of a sample.

7.5 Reagent Water: Deionized water which is free of the analyte(s) of interest.

8) Interferences:

8.1 Method interferences may be caused by contaminants, reagents, glassware, and other sample processing hardware. All these materials must be routinely demonstrated to be free from interferences under the conditions of analysis by running method blanks.

8.2 Non-homogeneous samples may give erroneous results. Samples should be mixed as thoroughly as possible before removing an aliquot for analysis.

8.3 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the bench sheet.

8.4 Samples containing large amounts of solids may filter slowly. Prolonged filtration times resulting from filter clogging may produce high TSS results due to increased colloidal materials captured on the clogged filter.

8.5 Oil and grease in the samples will cause unreliable results due to difficulty in drying to constant weight. Floating oil and grease, if present, should be included in the sample and dispersed by a blender device before aliquoting.

8.6 Filtration apparatus, filter material, pre-washing, post-washing, and drying temperatures are specified because these variables have been shown to affect the results.

8.7 The temperature at which the residue is dried has an important bearing on the results because weight losses due to volatilization of organic matter, mechanically occluded water, water of crystallization, and gases from heat-induced chemical decomposition, as well as weight gains due to oxidation, depend on temperature and time of heating.

8.8 Each sample requires close attention to desiccation after drying. Minimize opening the desiccator because moist air enters. Some samples may be stronger desiccants than those used in the desiccator and may take on water.

8.9 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

8.10 Samples containing high concentrations of bicarbonate may require careful and possibly prolonged drying to ensure that all the bicarbonate is converted to carbonate.

8.11 Too much residue in the drying vessel will crust over, trapping water that will not be driven off during drying. Total residue should be limited to about 200 mg.

8.12 Some samples may have fine suspended solids which will pass through the glass fiber filter causing high TDS results.

8.13 Aluminum pans should not be used for TS or TDS analyses. Components in some samples may react to form aluminum compounds, causing unreliable results.

8.14 For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material prior to TSS determination.

9) Safety Precautions, Pollution Prevention and Waste Handling:

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

9.1 Primary Materials Used

There are no materials used in this method that have a serious or significant hazard rating. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

9.2 Pollution Control

It is EFGS's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

9.3 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP *EFGS-S-HS-SOP2991*. The following waste streams are produced when this method is carried out".

9.3.1 Waste Streams Produced by the Method

9.3.1.1 There are no special waste streams associated with this method.

10) Personnel Training and Qualifications:

10.1 An analyst must perform an initial demonstration of capability (IDOC) that includes four replicates of a secondary source before being qualified to analyze samples without supervision. Continuing DOC will be maintained and monitored via performance on LCSs and other QC samples, as well as obtaining acceptable results on proficiency testing exercises.

10.2 Training is documented by the employee and supervisor, and is kept on file in the QA Office. The employee must read, understand, and by signing the training document, agree to perform the procedures as stated in all Standard Operating Procedures (SOPs) related to this method.

10.3 All employees must also, on a yearly basis, read the Quality Manual (QM), and complete the yearly Ethics training.

10.4 All training documents including IDOCs, CDOCs, Initial QA orientation, and Ethics training are stored by the Quality Assurance Manager in the employees training file for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

10.5 Chemical Safety Training, Compressed Gas Training, Chemical Hygiene Plan documentation, and Shipping of Hazardous goods, are stored by the Health and Safety Officer for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

11) Sample Collection, Preservation and Handling:

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

Method	Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
TS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
TDS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
TSS	Waters	HDPE	100 mLs	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3

12) Apparatus and Equipment:

- Analytical balance capable of weighing to 0.0001 g. The balance calibration is checked each day of use with three Class 1 weights that bracket the range of use. Details for this procedure are covered in SOP *EFGS-Q-QD-SOP2710*.
- Vacuum filtration apparatus.
- Vacuum pump equipped with moisture trap.
- Glass fiber filter disks, 47 mm, without organic binder (Gelman Type A/E) or equivalent.
- Evaporating dishes, glass, 150 mL capacity.
- Desiccators providing sufficient space for storage of samples in process separate from filters and evaporating dishes.
- Desiccant containing a color indicator of moisture concentration or an instrumental indicator.
- Drying ovens set at 103-105 °C and 180 ± 2 °C. Separate ovens should be maintained at appropriate temperatures if possible.
- Thermometers, NIST traceable.
- Graduated, Class A, cylinders, assorted sizes.
- Volumetric flasks, Class A, assorted sizes.
- Aluminum weighing dishes large enough to hold a 47 mm filter.
- Forceps for handling beakers.
- Crucible tongs.
- Tweezers for handling filters.

- Zetex gloves or other gloves capable of providing protection at 180 °C.

12.1 Preparation of Glass Fiber Filter Disc for TSS

12.1.1 Place the glass fiber filter discs, one at a time, on the membrane filter apparatus with wrinkled surface up.

12.1.2 While vacuum is applied, wash the disc with three successive (approximately) 20 mL volumes of distilled water.

12.1.3 Remove all traces of water by continuing to apply vacuum after water has passed through and discard washings.

12.1.4 Remove filter from membrane filter apparatus and place in a labeled, aluminum weighing dish and dry in an oven at 103-105 °C for one hour.

12.1.5 Remove the weighing dish from the oven and place in a desiccator and cool to room temperature.

12.1.6 Weigh the cooled filter to the nearest 0.1 mg using an analytical balance. Record the weight and the dish identification number on the bench sheet.

12.1.7 Alternatively and preferred is the use of certified dried and pre-weighed filters from Environmental Express P/N F93447MM.

12.2 In most cases, TDS analysis will be done using disposable pre-weighed bags,that are inserted into Teflon beakers for oven drying. (Environmental Express part TDS-100). In the event the disposable vessels are unavailable, prepare glass beakers as follows: Glass beakers for TDS, minimum 150 mL volume, must be thoroughly cleaned, rinsed with de-ionized water and baked at 180 ± 2 °C for TDS and 104 ± 2 °C for TS at least one hour before use. Transfer to a desiccator and allow to cool completely before use.

13) Reagents and Standards:

13.1 Document reagent/standards and reagent/standard preparation in TALS using the reagent module as described in SOP *EFGS-Q-QD-SOP41922*.

13.2 De-ionized water.

13.3 LCS solution:

For TS and TDS, place 1.0 g of sodium chloride to a 1000 mL volumetric flask and dilute to volume with deionized water. Mix well. Prepare fresh every three months. Alternatively, a commercially available LCS solution may be used. True values are TS = 1100 mg/L and TDS = 1000 mg/L.

For TSS place 0.5g of Celite™ diatomaceous earth and 1g of sodium chloride into a 1000ml volumetric flask and mix well. Alternately a certified commercial standard can be used instead, e.g. NSI solutions (P/N QCI-057) or equivalent. TSS = 500 mg/L.

13.4 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards and dispose of them according to SOP *EFGS-S-HS-SOP2991*.

14) Calibration:

14.1 Since this method is based on gravimetric techniques, there is no calibration in the usual sense. Proper balance operation will be verified daily or prior to sample analysis by following the lab-specific balance calibration SOP *EFGS-Q-QD-SOP2710*. Analytical balance calibration must be performed daily (every 24 hours).

14.2 Oven temperature must be checked daily and uncorrected and corrected temperatures recorded either on the bench sheet or in an oven temperature logbook.

15) Procedure:

15.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo (see SOP # *EFGS-Q-QD-SOP2812* for details) and must be approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

15.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance with a cause and corrective action described.

15.3 Proper sample identification is extremely important in any analytical procedure. Labeling of evaporating dishes and filters holders must be done in a manner to ensure connection with the proper sample.

15.4 If possible, analyze all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab and reporting group.

15.5 Non-representative particulates such as leaves, sticks, fish, and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result. The presence/removal of these artifacts should be noted on the bench sheet.

15.6 If samples are visibly oily, this should be noted on the bench sheet.

15.7 If there is limited sample volume or high solids content, smaller amounts of sample may need to be processed than detailed in the following sections. This occurrence must be noted on the bench sheet and reporting limits must be adjusted appropriately.

15.8 Sample Preparation

15.8.1 Before any vessel, dish or sample preparation is started, check the balance log for the balances that will be used to ensure that the balance checks have been done that day. If the checks have not been done, do not proceed until the checks are completed. See SOP *EFGS-Q-QD-SOP2710*.

15.8.2 Total Solids

15.8.2.1 Total solids use the vessels or glass beaker in the same manner as specified for total dissolved solids. If unable to use the disposable vessels as described in section in section 12.2, then prepare beakers as follows:

15.8.2.2 Remove the beaker from the oven furnace using tongs and heat resistant gloves.

15.8.2.3 Cool and store beaker in desiccator until dish reaches room temperature or until needed.

Note: Analyst must transfer the beaker with gloves or tongs to prevent added weight due to oil from fingerprints.

15.8.2.4 Weigh immediately before use to the nearest 0.1 mg. Record the weight on the bench sheet.

15.8.3 Total Dissolved Solids

15.8.3.1 If unable to use the disposable TDS vessels as described in section in section 12.2, then prepare beakers as follows: Preparation of heat clean beakers to 180 ± 2 °C for one hour.

- Heat resistant gloves and tongs must be used when removing items from the oven.

- Store and cool beaker in desiccator until dish reaches room temperature or until needed.

Note: Analyst must transfer the beaker with gloves or tongs to prevent added weight due to oil from fingerprints.

- Weigh immediately before use to the nearest 0.1 mg. Record the weight of the dish on the bench sheet.

15.8.4 Total Suspended Solids

15.8.4.1 Preparation of the glass fiber filters is described in Section 12.1.

15.9 Sample Analysis

15.9.1 Check the Balance Logbook to determine if the daily calibration check was completed. If the balance requires a check, verify the calibration as detailed in *EFGS-Q-QD-SOP2710*.

15.9.2 Total Solids

15.9.2.1 Shake the sample vigorously and quickly aliquot the sample. Transfer a measured aliquot of well-mixed sample to a pre-weighed disposable vessel or glass beaker. Record the volume of sample (to the nearest mL) on the bench sheet.

15.9.2.2 Choose an aliquot of sample sufficient to contain a residue of at least 10 mg but less than 200 mg, up to a total volume of 100 mls. To obtain a weighable residue, successive aliquots of sample may be added to the sample vessel.

15.9.2.3 For the LCS, measure 50 mL of the TS/TDS LCS Solution (Section 13.3) and pour into the vessel or beaker.

15.9.2.4 For the MB, measure 100 mL of reagent water and pour into the vessel/beaker.

15.9.2.5 Place the tray of samples into a drying oven at 103-105 ^{0}C and evaporate to dryness.

15.9.2.6 Dry the evaporated sample for at least one hour at 103-105 °C.

15.9.2.7 Remove the tray of weighing dishes from the oven using heat-resistant gloves. Place in a desiccator and cool to room temperature.

15.9.2.8 Weigh the dish to the nearest 0.1 mg. Record the weight on the bench sheet.

15.9.2.9 Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or until loss of weight is less than 4% of the previous weight or 0.5 mg, whichever is less. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

Note: When weighing dried sample, be alert to change in weight due to air exposure and/or sample degradation.

15.9.2.10 Calculate results according to the equation provided in Section 16.3.1. Use the final weight achieved for calculating TS.

15.9.3 Total Dissolved Solids

15.9.3.1 TDS estimation and Estimated sample volume calculation:

TDSe=conductivity*0.65 mLe=(25/TDSe)*1000

Alternately use, use the chart in Attachment 1

Use these to determine the estimated TDS and the volume of sample to be filtered. If the conductivity is very low a large amount of sample will be calculated to filter. Do not filter more than 100 mls of sample due to beaker capacity. Seek guidance from Manager for questions.

15.9.3.2 Thoroughly rinse the entire filtration apparatus with reagent water before filtering each sample.

15.9.3.3 Assemble the filtering apparatus, place a glass fiber filter in the apparatus, pre-wet the filter using reagent water, and begin suction.

Note: If the sample also requires TSS, pre-weigh the prepared filter and refer to Section 15.9.4 for additional guidance.

15.9.3.4 Shake the sample vigorously and quickly aliquot the sample. Filter an aliquot of sample sufficient to yield between 10 and 200 mg dried residue to the funnel by means of a graduated cylinder, up to 100 mls. If more than 10 minutes are required to complete filtration, decrease sample volume or increase filter size. If total filterable residue is low, a larger volume may be filtered.

Note: Multiple filters may be used if performing only TDS analysis.

15.9.3.5 A smaller amount should be filtered if the sample is high in TSS or is otherwise slow to filter.

15.9.3.6 Record the volume of sample used (to the nearest mL) on the benchsheet.

15.9.3.7 For the method blank, process 100 mL of reagent water as the sample. Blank result should be less than method detection limit.

15.9.3.8 For the LCS, process 50 mL of the LCS Solution. Refer to Section 13.3 for instructions on how to prepare the LCS.

15.9.3.9 Filter the sample through the glass fiber filter.

15.9.3.10 Rinse the graduated cylinder, funnel walls, and filter with three successive 10 mL portions of reagent water and allow for complete drainage between washings. Continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

15.9.3.11 Transfer the filtrate (including the washings) to a pre-weighed disposable vessel, placed in a Teflon beaker on a tray. Rinse the receiving flask with 10-25 mL of reagent water and transfer washings into the dish to ensure complete transfer of the sample.

15.9.3.12 Evaporate the samples to dryness in a drying oven at 170 ± 5 °C.

15.9.3.13 Dry the evaporated sample in an oven for at least one hour at 180 ± 2 °C.

15.9.3.14 Use forceps or heat resistant gloves to remove the samples from the oven. Place in a desiccator and cool to room temperature.

15.9.3.15 Weigh the dish to the nearest 0.1 mg. Record the combined weight of the dried residue and the dish on the bench sheet.

15.9.3.16 Return the samples to the oven at 180°C for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or until loss of weight is less than 0.5 mg. Continue weighing until a constant weight is obtained or prepare a Nonconformance Memo, if a constant weight is not achieved in three drying cycles.

15.9.3.17 Calculate results according to the equation in Section 16.3.2. Use the final weight achieved for calculating TDS.

15.9.4 Total Suspended Solids

15.9.4.1 Assemble the filtering apparatus, place the pre-weighed glass fiber filter in the apparatus, pre-wet the filter using reagent water and begin suction.

Note: Handle the filters only with tweezers.

15.9.4.2 Selection of Sample Volume

For the MB, filter 500ml of DI water.
For the LCS, shake the bottle **vigorously** and filter 50 ml.
For a 4.7 cm diameter filter, filter an aliquot of sample sufficient to yield between 2.5 mg and 200 mg of dried residue, up to 1000 mls. If the weight of the captured residue is less than 2.5 mg, the sample volume must be increased up to 1000ml to provide at least 2.5 mg of residue. If the weight of the captured residue is >200 mg the sample volume must be reduced.
If insufficient sample is provided by the client to meet the dried residue requirement an NCM must be generated in TALS to indicate this deviation from the method requirements.
If during filtration of this initial volume, the filtration rate drops rapidly or if filtration time exceeds 5-10 minutes, a smaller volume of sample should be processed.

Note: If the sample appears high in TSS, start with a smaller sample volume.

15.9.4.3 Shake the sample vigorously and quickly aliquot the sample. It is important to pour out the sample immediately after shaking so that the solids do not have time to settle. A smaller amount should be filtered if the sample is high in TSS or is otherwise slow to filter. Record the volume of sample filtered (to the nearest mL) on the benchsheet.

Note: If Total Dissolved Solids (TDS) is also required, the filtrate may be used. Refer to Section 15.9.3 for additional guidance.

15.9.4.4 Remove all traces of water by continuing to apply vacuum after the sample has passed through.

15.9.4.5 With suction on, rinse the graduated cylinder, filter, suspended solids residue, and filter funnel wall with three 10 mL portions of reagent water allowing complete drainage between washings.

15.9.4.6 Remove all traces of water by continuing to apply vacuum for about three minutes after the sample has passed through.

15.9.4.7 Carefully remove the filter from the filter support and transfer to an aluminum weighing dish. If the filter is torn or damaged during this process, the sample must be reanalyzed. Take care to keep the filter face-up during the transfer so that the residue does not fall off.

15.9.4.8 Dry the filter at least one hour at 103-105 °C.

15.9.4.9 Use heat resistant gloves to remove the tray of dishes from the oven. Place in a desiccator and cool to room temperature.

15.9.4.10 Cool the samples in a desiccator, weigh (to the nearest 0.1 mg), and record the weight on the bench sheet.

15.9.4.11 Return the samples to the oven for another hour, cool in a desiccator, and reweigh. Repeat the drying, cooling, desiccating, and weighing cycle until a constant weight is obtained or weight difference is less than 0.5 mg. If a constant weight is not achieved in three drying cycles, prepare a Nonconformance Memo.

15.9.4.12 Calculate the results using the formula given in Section 16.3.3. Use the final weight achieved for calculating TSS.

16) Calculations:

- If smaller or larger sample volumes are processed than are specified in the method, the reporting limit must be adjusted accordingly.
- All data are subject to two levels of review, which are documented on a checklist.
- If multiple weighing cycles are required, the lowest final sample weight is used for calculating solids content.

16.1 Accuracy

$$\text{LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

16.2 Precision (RPD)

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

16.3 Concentration

16.3.1 Total Solids:

$$\text{Total Solids, mg/L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

16.3.2 Total Dissolved Solids:

$$\text{Total Dissolved Solids, mg/L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of dried residue + dish (mg)
B = weight of dish (mg)
C = volume of sample (mL)

16.3.3 Total Suspended Solids:

$$\text{Total Suspended Solids, mg/L} = \frac{(A - B) \times 1000}{C}$$

Where: A = weight of filter + residue (mg)
B = weight of filter (mg)
C = volume of sample filtered (mL)

17) Statistical Information and Method Performance:

17.1 Method Detection Limit Study (MDL)

Method detection limits (MDL) are not determined for this procedure.

17.2 Demonstration of Capabilities

Analyst initial and continuing Demonstrations of Capability (DOC) are performed before any client samples are analyzed and are updated annually. See SOP *EFGS-Q-QD-SOP2814* for details.

17.3 Training Requirements

See SOP *EFGS-Q-QD-SOP2814* for detailed training requirements.

18) Quality Assurance/Quality Control and Corrective Action:

18.1 Before analyzing samples, the laboratory must establish a method detection limit (MDL) as described in Section 17.1.

18.2 In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument they will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 17.2 for more details.

18.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. See QC SOP (*EFGS-Q-QD-SOP41889*) for further details.

18.4 Method Blank

A method blank is required with every batch of 20 or fewer samples. The blank is deionized water taken through the procedure like a sample. The general requirement is that the method blank result must be less than the reporting limit or one-tenth of the concentration found in the associated samples (for samples with concentrations above the RL).

Corrective Action: If the method blank exceeds allowable levels, all associated samples must be reanalyzed.

18.5 Laboratory Control Sample (LCS/LCSD)

One LCS is required with each analytical batch. Historical control limits are based on three standard deviations of past results, and must be 80-120% or tighter. The process of establishing control limits is described in more detail in the QC SOP *EFGS-Q-QD-SOP41889*.

Corrective Action: If the LCS exceeds allowable levels, all associated samples must be reanalyzed.

NOTE: For DoD projects, an LCSD must be prepared and evaluated when adequate sample volume for a duplicate is not provided.

18.6 Duplicate Sample Analysis

A duplicate pair is required with every 10 samples. (Twice for each analytical batch of 20). Historical control limits are based on three standard deviations of past results, and must be 20% RPD. Note that the control limits only apply to samples with results greater than 5 times the RL. The process of establishing control limits is described in more detail in the QC SOP *EFGS-Q-QD-SOP41889*.

Corrective Action: If the RPD is greater than 20%, the result should be qualified and a NCM should be initiated that describes the problem.

18.7 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

19) List of Attachments:

Attachment 1: Quick reference for TDS volumes to use based on conductivity

Attachment 1: Quick reference for TDS volumes to use based on conductivity

Conductivity (µS/cm)	TDS Volume (ml)
1-2000	100
2000-6000	50
6000-10000	10
10000-15000	5
15000-25000	2
25000-70000	1
70000+	0.5

EFGS-Q-QD-SOP2710 Class 1 Weight Calibration, Balance Verification, Calibration & Maintenance
EFGS-Q-QD-SOP2812 Procedures for Nonconformance, Corrective Actions and the Control of Non-Conforming Work
EFGS-Q-QD-SOP2814 Personnel Training and Demonstration of Capability Procedures
EFGS-Q-QD-SOP41889 Quality Control Program
EFGS-Q-QD-SOP41922 Receipt, Preparation, Storage and Verification of Standards and Reagents and Traceability Protocols
EFGS-Q-QM-QM5805 Quality Assurance Manual
EFGS-S-HS-SOP2991 Waste Disposal Procedures for Client Sample Waste
EFGS-T-WC-SOP40959 Settleable Matter or Settleable Solids [Methods EPA 160.5 and SM 2540F]
EFGS-T-WC-SOP41035 Percent Moisture/Percent Solids [Method SM 2540 G]
EFHS-S-HS-12066.0004 Chemical Hygiene Plan

End of document

Version history

Version	Approval	Revision information	
1	02.OCT.2021		

eurofins	Always check on-line for validity.	Level:
Document number: **EFGS-T-WC-SOP41381**	**Alkalinity by Titration [Methods 310.1, SM 2320B]**	**Standard Operating Procedure**
Old Reference: **TA-WC-0101**		
Version: **3**		Organisation level: **4-Business Unit**
Approved by: **H3WW, KOJ6** Effective Date **12-JAN-2022**	Document users: **EFGS_WET titr/spec**	Responsible: **EFGS_WET_M**

EUROFINS FRONTIER GLOBAL SCIENCES

1) Revision Log:

Revision: 3	**Effective Date:** This version	
Section	**Justification**	**Changes**
13.5	Required	Added calculation detail for titrant standardization requirement

Revision: 2	**Effective Date:** 1/9/2022	
Section	**Justification**	**Changes**
13.3	Required	Added titrant standardization requirement

2) Reference:

2.1 Methods for the Analysis of Water and Wastes, EPA-600/4-79-020, March 1983, Method 310.1.

2.2 Standard Methods for Analysis of Water and Wastewater, 19th edition, 1995, Method 2320B.

2.3 Annual book of ASTM Standards, Part 31, “Water”, p 115, D-1067, Method D, 1976.

2.4 Mettler DL 70 Operation Instruction Manual.

3) Cross Reference:

Document	Document Title
EFGS-Q-QM-QM5805	QA Manual
EFGS-S-HS-12066	Chemical Hygiene Plan
CW-E-M-001	Corporate Environmental Health and Safety Manual
EFGS-S-HS-SOP2991	Waste Disposal Procedures for Client Sample Waste
EFGS-Q-QD-SOP41922	Receipt, Preparation, Storage and Verification of Standards and Reagents and Traceability Protocols
EFGS-Q-QD-SOP2710	Class 1 Weight Calibration, Balance Verification, Calibration & Maintenance
EFGS-Q-QD-SOP41510	Volumetric Verification and Pipette and Dispenser Operation & Maintenance
EFGS-Q-QD-SOP2812	Procedures for Nonconformance, Corrective Actions and the Control of Non-Conforming Work
EFGS-Q-QD-SOP2814	Personnel Training and Demonstration of Capability Procedures
EFGS-Q-QD-SOP41889	Quality Control Program

4) Scope:

4.1 Analytes, Matrix(s), and Reporting Limits

4.1.1 This method is applicable for the measurement of alkalinity in drinking, surface, and saline waters, and domestic and industrial wastes.

4.1.2 This method is applicable to all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 50 mL.

4.1.3 The reporting limit for both methods is 5 mg/L. For current reporting limits please see TALs.

4.1.4 Automated titrimetric analysis is equivalent.

4.1.5 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 14.2.1 in the Quality Assurance Manual.

5) Basic Principles:

Samples are titrated with 0.02N sulfuric acid for <1000 mg/L CaCO3 or 0.1N sulfuric acid for > 1000 mg/L CaCO3 to an endpoint of pH 4.5. The end point is determined with a pH meter. Results are reported as mg/L calcium carbonate.

6) Reference Modifications:

None

7) Definitions:

7.1 pH **-** At a given temperature, the intensity of the acidic or basic character of a solution is indicated by pH or hydrogen ion activity. Because of the ionic interactions in all but very dilute solutions, it is necessary to use the “activity” of the hydrogen ion and not its molar concentration. The approximate equivalent to molarity can be presumed only in very dilute solutions. A logarithmic scale is used for pH in order to express a wide range of hydrogen ion activities. Neutral pH is 7.0 at 25°C, while acidic pH’s are <7 and basic pH’s are >7.

7.2 Alkalinity **–** A measure of the acid-neutralizing capacity of water.

7.3 Conductivity **-** A measure of the ability of water to carry an electrical current.

8) Interferences:

8.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or high salinity.

8.2 Coatings of oily material or particulate matter can impair electrode response. Gentle wiping or detergent washing, followed by distilled water rinsing can usually remove coatings.

8.3 Samples with high concentrations of salts of weak organic or inorganic acids may have interferences. Alkalinity for samples with high concentrations of mineral acids should be determined using an electrometric endpoint of 3.9, using the ASTM procedure.

9) Safety Precautions, Pollution Prevention and Waste Handling:

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

9.1 Specific Safety Concerns or Requirements

9.1.1 None

9.2 Primary Materials Used

There are no materials with a health rating of 3 or 4 used in this method. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

9.3 Pollution Control

It is EFGS’s policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for “Waste Management and Pollution Prevention.”

9.4 Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP *EFGS-S-HS-SOP2991*.

9.4.1 The following waste streams are produced when this method is carried out:

9.4.1.1 Acidified samples are disposed of into the neutralization tank.

10) Personnel Training and Qualifications:

10.1 An analyst must perform an initial demonstration of capability (IDOC) that includes four replicates of a secondary source before being qualified to analyze samples without supervision. Continuing DOC will be maintained and monitored via performance on LCSs and other QC samples, as well as obtaining acceptable results on proficiency testing exercises.

10.2 Training is documented by the employee and supervisor, and is kept on file in the QA Office. The employee must read, understand, and by signing the training document, agree to perform the procedures as stated in all Standard Operating Procedures (SOPs) related to this method.

10.3 All employees must also, on a yearly basis, read the Quality Manual (QM), and complete the yearly Ethics training.

10.4 All training documents including IDOCs, CDOCs, Initial QA orientation, and Ethics training are stored by the Quality Assurance Manager in the employees training file for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

10.5 Chemical Safety Training, Compressed Gas Training, Chemical Hygiene Plan documentation, and Shipping of Hazardous goods, are stored by the Health and Safety Officer for ten years after the employee is no longer working for Eurofins Frontier Global Sciences.

11) Sample Collection, Preservation and Handling:

11.1 The sample container must be filled completely, sealed and stored between 0-6°C.

11.2 Care must be taken to minimize exposure of the sample to the atmosphere; open the sample immediately before analysis.

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

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	HDPE	50 mLs	Cool 0-6°C	14 Days	40 CFR Part 136.3

12) Apparatus and Equipment:

12.1 Method 310.1

- pH meter, Orion SA 720 or equivalent.
- Combination electrode with temperature compensating probe.
- Laboratory balance, minimum 0.001g accuracy. Check the Balance logbook to determine if the daily calibration check has been completed. If it has not, the analyst must perform this check according to SOP *EFGS-Q-QD-SOP2710*.
- Spatula or scoopula.
- Volumetric pipet, 20.0-mL, 3.0-mL

- Volumetric flask, 1 liter
- Mettler DL70 Autotitrator
- 100mL plastic cups for use in auto sampler

12.2 Computer hardware and software

- Computer with a minimum 1GB memory, Pentium 4 processor, 80 G hard drive or equivalent or as recommended by instrument manufacturer.
- Data acquisition/processing system: LIMS system: TALS version 1.0 or higher

13) Reagents and Standards:

13.1 Document reagent/standards and reagent/standard preparation in TALS using the reagent module as described in SOP *EFGS-Q-QD-SOP41922*.

13.2 Deionized (DI) water.

13.3 0.02N Sulfuric acid solution purchased standardized, Fisher part number, SA226-4.

13.4 0.1 N Sulfuric acid solution purchased standardized, Fisher part number SA220-4. See certificate of analysis for exact Normality that is used for constant with the auto titrator.

13.5 All acid titrants, whether prepared in the lab or purchased, must be standardized prior to initial use, and at least monthly following that. Standardize the titrant solution by potentiometric titration of 15.00 mL 0.05N Na2CO3. This needs to be performed one month after opening of titrant solution and performed monthly.

On the instrument titrate 15mL of 0.05N NaCO3 solution (Section 13.6) until it reaches a pH of about 4.5.

Calculate the normality of the acid:

$$N = \frac{A \times B}{53 \times C}$$

Where:

A = g Na2CO3

B= mL Na2CO3 solution used for titration

C = mL acid used to reach end-point pH of 4.5

After standardizing the titrant, the new Normaity **MUST BE** entered into TALS for each batch in the batch information under "Normality of First Titrant" and updated on the auto titrator.

13.6 1000 mg/L Alkalinity Standard, purchased standard, Absolute Standards #54142. 100 gm/L ICV may be diluted from the certified stock standard by a factor of 10. Prepare as needed.

13.7 100 mg/L Spike Standard for LCS and CCV.

Stock standard; in a 1000 mL volumetric flask, dissolve 0.1060 g Anhydrous Sodium Carbonate (oven dried at 250 °C for four hours) in 700 mL previously boiled DI water and then bring to 1000 mL with DI water for a 100 mg/L CaCO3 standard. Standard may be stored for up to a week.

13.8 Managers/supervisors or a designee are expected to check their areas on a monthly basis for expired standards/reagents and dispose of them according to SOP *EFGS-S-HS-SOP2991*.

14) Calibration:

14.1 Calibrate the **Mettler DL70** autotitrator as follows:

14.1.1 The date must be entered into the auto-titrator every day before beginning analysis. Turn on the instrument and when prompted enter the month, press 'START', enter the day, press 'START', enter the year and press 'START'.

14.1.2 The pH probe must be calibrated every day before beginning analysis. Press '3' on the keypad followed by the 'ELEC CALIB' button. Place enough pH 4.0 buffer solution in a sample cup to cover the probe (approx. 30-mL) and when the 'Buffer A' light blinks press 'Start'. When the 'Buffer B' light blinks, replace the pH 4.0 buffer solution with a pH 10.0 buffer solution. When calibration is complete the instrument will print out a calculated slope. This slope must be between –52 and –62 mV/pH to be valid. If the slope is outside of this range, the probe must be recalibrated.

14.2 Calibrate the **Mettler DL70** as follows:

14.2.1 The date must be entered into the auto-titrator every day before beginning analysis.

14.2.2 The pH probe must be calibrated every day before beginning analysis. With about 30 mL of pH buffer solution 4.0 and 10.0 in the first two positions on the autosampler, run User Method 1. When calibration is complete the instrument will print out a calculated slope. This slope must be between –52 and –62 mV/pH to be valid. If the slope is outside of this range, the probe must be recalibrated. Ideally, the midpoint should be 7.0 ± 0.1. Record the slope in the logbook.

15) Procedure:

One-time procedural variations are allowed only if deemed necessary in the professional judgment of management to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP # *EFGS-Q-QD-SOP2812*. The NCM shall be filed in the project file and addressed in the case narrative.

15.1 Sample Preparation

The sample must not be filtered, diluted, concentrated, or altered in any way except at the request of the client (this request must be indicated in an NCM).

15.2 Autotitrator Volume Check

15.2.1 Review the calibration label on the titrator to insure that the last volume check conducted on the titrator applies to the current quarter. If not, check the dispensed volume according to the procedures detailed in SOP *EFGS-Q-QD-SOP41510*.

15.3 Sample Analysis for the Mettler DL70

15.3.1 Measure 30 mL of sample into sample cup and place cups into autosampler. Note: Autosampler spins clockwise, so samples need to be loaded counter clockwise from the 1st position.

15.3.2 Run User Method 3. Enter sample IDs into the run log.

15.3.3 Open sequence with an ICV, LCS, Sample, DUP. Include a CCV after every 10th samples and at the end of the batch.

15.3.4 A high-level CCV should be used for samples that exceed titration limit using the 0.02 N sulfuric acid and the samples plus the high-level CCV must be re-titrated using 0.1 N sulfuric acid.

15.4 Instrument Maintenance

15.4.1 Volumetric verification of the Mettler DL70 autotitrator must be performed quarterly.

15.4.2 No regular maintenance is required for this instrumentation.

15.4.3 All instrument maintenance must be documented in the instrument maintenance logbook.

15.5 Troubleshooting

15.5.1 Refer to Appendix A, Mettler Troubleshooting Guide.

16) Calculations:

16.1 Accuracy

$$\text{LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

16.2 Precision (RPD)

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

16.3 Concentration

$$\text{Alkalinity, mg CaCO3/L} = \frac{\text{mL of acid x N of acid x 50,000}}{\text{mL of sample}}$$

16.4 Calculate the individual forms of alkalinity as follows:

Result of Titration	Hydroxide Alkalinity	Carbonate Alkalinity	Bicarbonate Alkalinity
P = ND	ND	ND	T
P < T/2	ND	2P	T - 2P
P = T/2	ND	2P	ND
P > T/2	2P - T	2(T - P)	ND
P = T	T	ND	ND

Where:

T = Total Alkalinity = Alkalinity at pH 4.5

P = Phenolphthalein Alkalinity = Alkalinity at pH 8.3

17) Statistical Information and Method Performance:

17.1 Method Detection Limit Study (MDL)

A method detection limit (MDL) study is not performed for this analysis.

17.2 Demonstration of Capabilities

Analyst initial and continuing Demonstrations of Capability (DOC) are performed before any client samples are analyzed and are updated annually. See SOP *EFGS-Q-QD-SOP2814* for details.

17.3 Training Requirements

See SOP *EFGS-Q-QD-SOP2814* for detailed training requirements.

18) Quality Assurance/Quality Control and Corrective Action:

18.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section.

18.2 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in SOP *EFGS-Q-QD-SOP41889*, Quality Control Program.

18.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

18.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP *EFGS-Q-QD-SOP2812*. This is in addition to the corrective actions described in the following sections.

18.5 Batch Definition

A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a laboratory control sample (LCS), and duplicate (DUP). As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

18.6 Method Blank (MB)

Method blanks (MB) are not applicable to this technique.

18.7 Laboratory Control Sample (LCS)

At least one LCS (see Section 13.6) must be processed with each batch. If there isn't sufficient sample for a duplicate sample analysis (18.8), then a LCSD must also be processed. However, LCSD is required for BP/DOD samples. The LCS is used to monitor the accuracy of the analytical process.

Acceptance Criteria: The LCS result must be within 15% of the true value.

Corrective Action: If LCS recoveries are outside established control limits, the analytical system is out of control and corrective action must be taken.

If recoveries are above control limits and carbonate is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

In other circumstances, the entire batch must be re-prepared and reanalyzed.

18.8 Duplicate Sample Analysis

A duplicate pair is required with each analytical batch and must be within 20% RPD. If there isn't sufficient sample for a duplicate sample analysis, then a LCSD (9.8) must also be processed. Note that the control limits only apply to samples with results greater than 5 times the RL. The process of establishing control limits is described in more detail in the QC SOP *EFGS-Q-QD-SOP41889*.

Corrective Action: If the RPD is greater than 20% the sample should be reanalyzed if within holding time and sufficient sample is remaining.

18.9 Continuing Calibration Verification (CCV)

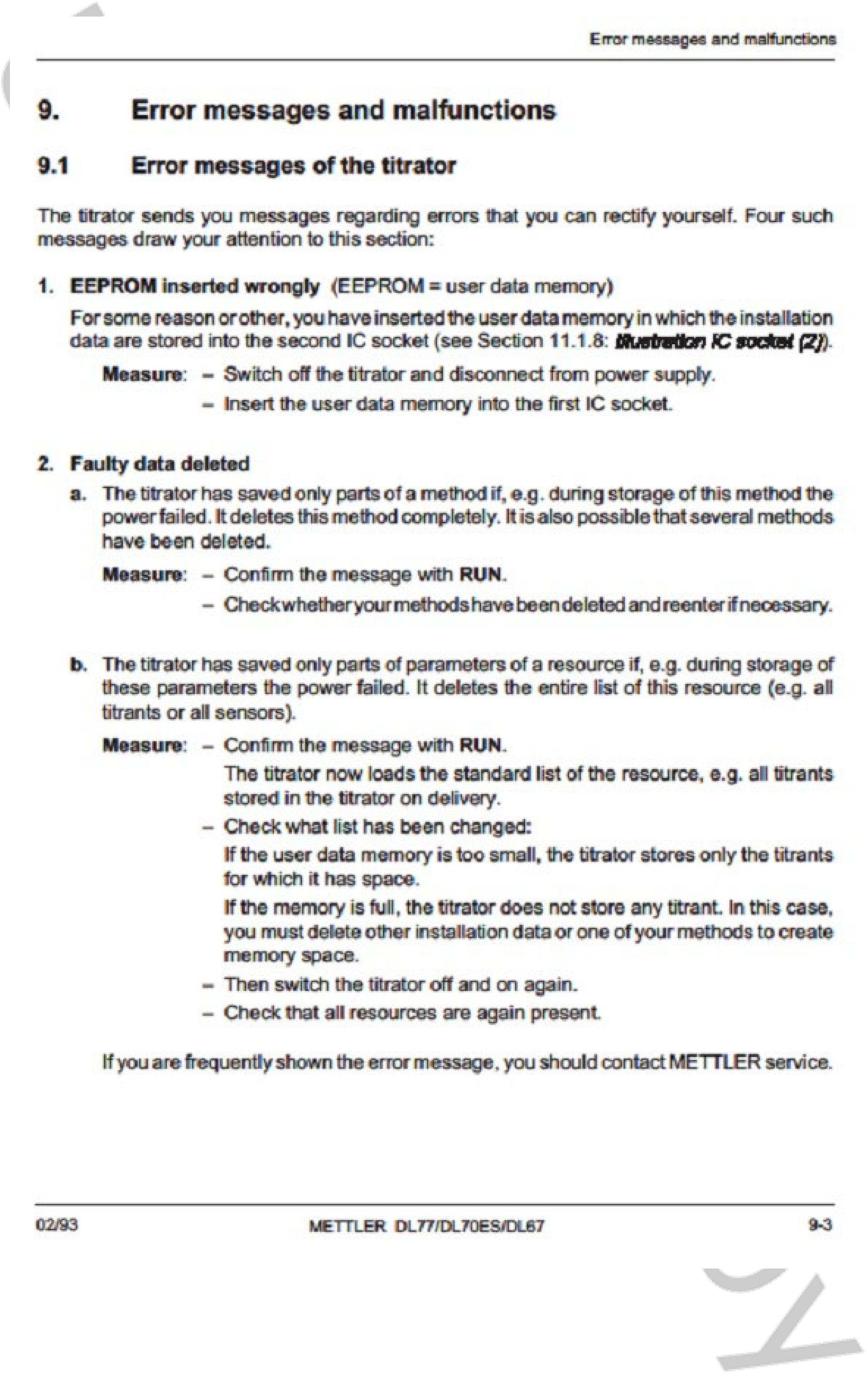
A CCV is required after every 10 or fewer samples and after the last sample. Acceptance Criteria: The CCV recovery must be 85-115%. Corrective Action: If the recovery is outside of the acceptance Limits, all samples analyzed since the last successful CCV must be reanalyzed.

18.10 Any extra QC that is analyzed in a batch or sequence must be evaluated using the same criteria as the corresponding QC above.

19) List of Attachments:

Appendix A: Mettler Troubleshooting Guide

Appendix A: Mettler Troubleshooting Guide



9. Error messages and malfunctions

9.1 Error messages of the titrator

The titrator sends you messages regarding errors that you can rectify yourself. Four such messages draw your attention to this section:

1. **EEPROM inserted wrongly** (EEPROM = user data memory)

   For some reason or other, you have inserted the user data memory in which the installation data are stored into the second IC socket (see Section 11.1.8: ***Illustration IC socket (2)***).

   Measure: – Switch off the titrator and disconnect from power supply.
   – Insert the user data memory into the first IC socket.

2. **Faulty data deleted**

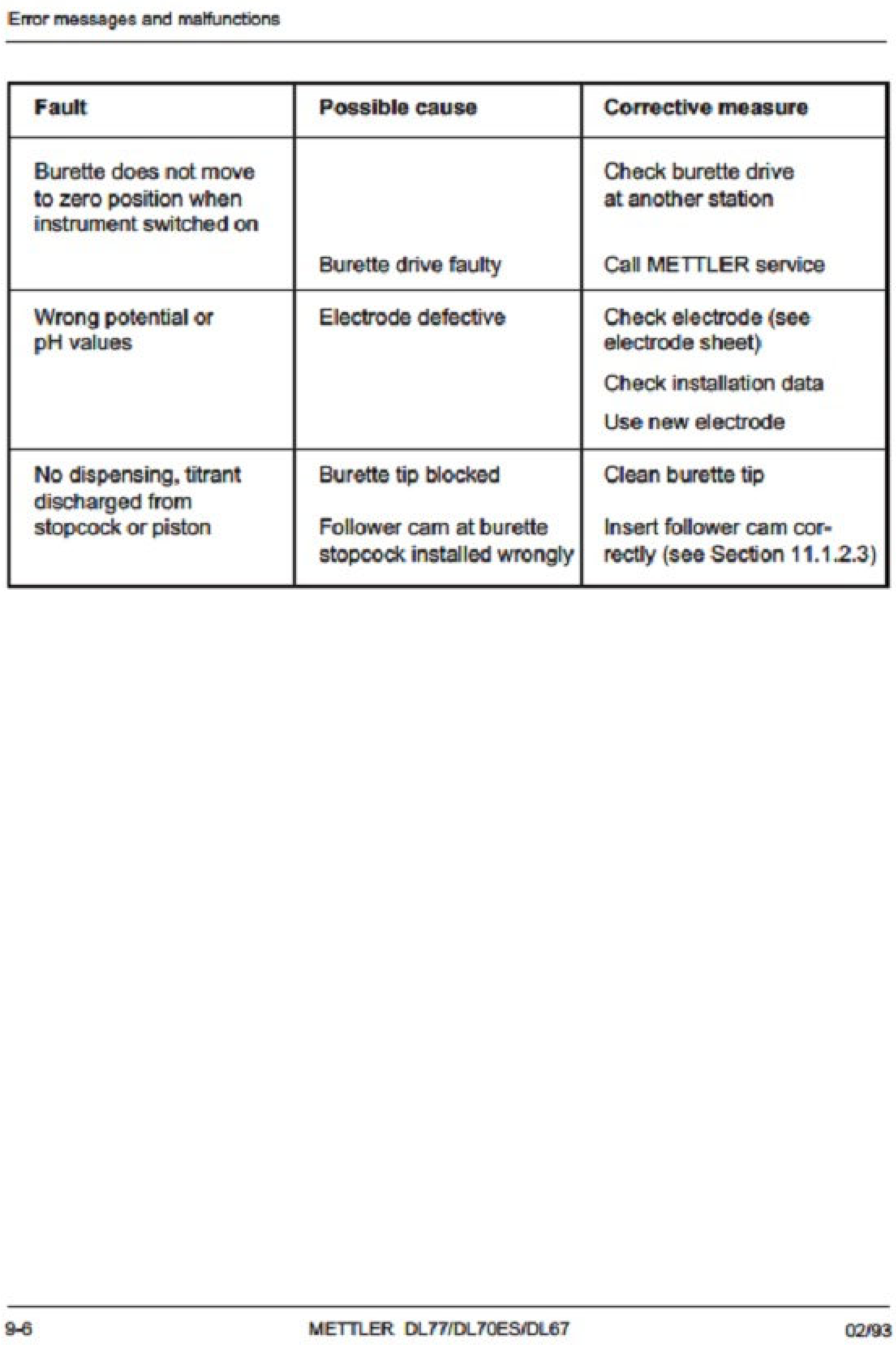
   a. The titrator has saved only parts of a method if, e.g. during storage of this method the power failed. It deletes this method completely. It is also possible that several methods have been deleted.

   Measure: – Confirm the message with **RUN**.
   – Check whether your methods have been deleted and reenter if necessary.

   b. The titrator has saved only parts of parameters of a resource if, e.g. during storage of these parameters the power failed. It deletes the entire list of this resource (e.g. all titrants or all sensors).

   Measure: – Confirm the message with **RUN**.
   The titrator now loads the standard list of the resource, e.g. all titrants stored in the titrator on delivery.
   – Check what list has been changed:
   If the user data memory is too small, the titrator stores only the titrants for which it has space.
   If the memory is full, the titrator does not store any titrant. In this case, you must delete other installation data or one of your methods to create memory space.
   – Then switch the titrator off and on again.
   – Check that all resources are again present.

   If you are frequently shown the error message, you should contact METTLER service.



Error messages and malfunctions

Fault	Possible cause	Corrective measure
Burette does not move to zero position when instrument switched on		Check burette drive at another station
	Burette drive faulty	Call METTLER service
Wrong potential or pH values	Electrode defective	Check electrode (see electrode sheet) Check installation data Use new electrode
No dispensing, titrant discharged from stopcock or piston	Burette tip blocked	Clean burette tip
	Follower cam at burette stopcock installed wrongly	Insert follower cam correctly (see Section 11.1.2.3)

9-6 METTLER DL77/DL70ES/DL67 02/93

EFGS-Q-QD-SOP2710 Class 1 Weight Calibration, Balance Verification, Calibration & Maintenance
EFGS-Q-QD-SOP2812 Procedures for Nonconformance, Corrective Actions and the Control of Non-Conforming Work
EFGS-Q-QD-SOP2814 Personnel Training and Demonstration of Capability Procedures

EFGS-Q-QD-SOP41510 Volumetric Verification and Pipette and Dispenser Operation & Maintenance
EFGS-Q-QD-SOP41889 Quality Control Program
EFGS-Q-QD-SOP41922 Receipt, Preparation, Storage and Verification of Standards and Reagents and Traceability Protocols
EFGS-Q-QM-QM5805 Quality Assurance Manual
EFGS-S-HS-SOP2991 Waste Disposal Procedures for Client Sample Waste
EFHS-S-HS-12066.0004 Chemical Hygiene Plan

End of document

Version history

Version	Approval	Revision information	
1	23.JUL.2021		
2	09.JAN.2022		
3	12.JAN.2022		

THE STATE of ALASKA
GOVERNOR MICHAEL J. DUNLEAVY

Department of Environmental Conservation

DIVISION OF SPILL PREVENTION AND RESPONSE
Contaminated Sites Program
Laboratory Approval Program

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Anchorage, Alaska 99501
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February 7, 2021

Shawn Kusma
Eurofins – Seattle
5755 8th Street East
Tacoma, WA 98424

RE: Contaminated Sites Laboratory Approval **20-004**

Thank you for submitting a completed application to the Alaska Department of Environmental Conservation's Contaminated Sites Laboratory Approval Program (CS-LAP), on January 27, 2022. Based on your lab's National Environmental Laboratory Accreditation Program (NELAP) approval through the Oregon Environmental Laboratory Accreditation Program (ORELAP) and Department of Defense Environmental Laboratory Accreditation Program (DoD-ELAP) approval through the ANSI-ASQ National Accreditation Board (ANAB), Eurofins - Seattle, located at the above address, is granted ***Approved*** status to perform the analyses listed in the attached *Scope of Approval*, for Alaska contaminated sites projects, including underground storage tanks and leaking underground storage tank sites (UST/LUST), under the July 1, 2017 amendments to 18 AAC 78. This approval is effective February 7, 2022 and expires on ***February 19, 2025***.

Be aware that in accordance with 18 AAC 78.815(b) and 18 AAC 78.815(e), **any** changes in your lab's NELAP and/or DoD-ELAP approval status for **any** analytes in **any** methods or **any** matrices must be reported to the CS-LAP within 3 business days. This includes any suspension of any analyte(s) due to failed Performance Testing (PT) or Performance Evaluation (PE) samples. **FAILURE TO REPORT A CHANGE IN STATUS WILL RESULT IN REVOCATION OF ALL CS-LAP APPROVALS FOR ALL ANALYTES AND ALL METHODS IN ALL MATRICES FOR A PERIOD OF ONE YEAR.** Notification should be in writing sent to cs.lab.cert@alaska.gov. We recommend also contacting the CS-LAP by telephone to verify that the message was received.

If you have any questions, please contact the CS-LAP at (907) 465-5390, or by email at cs.lab.cert@alaska.gov. Labs are also highly encouraged to join the CS-LAP listserv by going to http://list.state.ak.us/mailman/listinfo/cs.lab.approval.

Respectfully,

Brian Englert

Attachment: Scope of Approval

Scope of Approval

CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
83-32-9	Water	Acenaphthene	625.1	ANAB
83-32-9	Soil	Acenaphthene	8270E	ANAB
83-32-9	Water	Acenaphthene	8270E	ANAB
83-32-9	Soil	Acenaphthene	8270E-SIM	ANAB
83-32-9	Water	Acenaphthene	8270E-SIM	ANAB
208-96-8	Water	Acenaphthylene	625.1	ANAB
208-96-8	Soil	Acenaphthylene	8270E	ANAB
208-96-8	Water	Acenaphthylene	8270E	ANAB
208-96-8	Soil	Acenaphthylene	8270E-SIM	ANAB
208-96-8	Water	Acenaphthylene	8270E-SIM	ANAB
67-64-1	Water	Acetone	624.1	ANAB
67-64-1	Soil	Acetone	8260D	ANAB
67-64-1	Water	Acetone	8260D	ANAB
309-00-2	Water	Aldrin	608.3	ANAB
309-00-2	Soil	Aldrin	8081A	ANAB
309-00-2	Water	Aldrin	8081A	ANAB
309-00-2	Soil	Aldrin	8081B	ANAB
309-00-2	Water	Aldrin	8081B	ANAB
N/A	Soil	Aliphatic Petroleum Hydrocarbons (>C10 – C12)	NW-EPH	ANAB
N/A	Water	Aliphatic Petroleum Hydrocarbons (>C10 – C12)	NW-EPH	ANAB
N/A	Soil	Aliphatic Petroleum Hydrocarbons (>C10 – C12)	NW-VPH	ANAB
N/A	Water	Aliphatic Petroleum Hydrocarbons (>C10 – C12)	NW-VPH	ANAB
N/A	Soil	Aliphatic Petroleum Hydrocarbons (>C12 – C16)	NW-EPH	ANAB
N/A	Water	Aliphatic Petroleum Hydrocarbons (>C12 – C16)	NW-EPH	ANAB
N/A	Soil	Aliphatic Petroleum Hydrocarbons (>C16 – C21)	NW-EPH	ANAB
N/A	Water	Aliphatic Petroleum Hydrocarbons (>C16 – C21)	NW-EPH	ANAB
N/A	Soil	Aliphatic Petroleum Hydrocarbons (>C21 – C34)	NW-EPH	ANAB
N/A	Water	Aliphatic Petroleum Hydrocarbons (>C21 – C34)	NW-EPH	ANAB
N/A	Soil	Aliphatic Petroleum Hydrocarbons (>C5 – C6)	NW-VPH	ANAB
N/A	Water	Aliphatic Petroleum Hydrocarbons (>C5 – C6)	NW-VPH	ANAB
N/A	Soil	Aliphatic Petroleum Hydrocarbons (>C6 – C8)	NW-VPH	ANAB
N/A	Water	Aliphatic Petroleum Hydrocarbons (>C6 – C8)	NW-VPH	ANAB
N/A	Soil	Aliphatic Petroleum Hydrocarbons (>C8 – C10)	NW-VPH	ANAB

Scope of Approval

CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
N/A	Water	Aliphatic Petroleum Hydrocarbons (>C8 – C10)	NW-VPH	ANAB
N/A	Soil	Aliphatic Petroleum Hydrocarbons (C8 – C10)	NW-EPH	ANAB
N/A	Water	Aliphatic Petroleum Hydrocarbons (C8 – C10)	NW-EPH	ANAB
120-12-7	Water	Anthracene	625.1	ANAB
120-12-7	Soil	Anthracene	8270E	ANAB
120-12-7	Water	Anthracene	8270E	ANAB
120-12-7	Soil	Anthracene	8270E-SIM	ANAB
120-12-7	Water	Anthracene	8270E-SIM	ANAB
7440-36-0	Water	Antimony (metallic)	200.7	ANAB
7440-36-0	Water	Antimony (metallic)	200.8	ANAB
7440-36-0	Soil	Antimony (metallic)	6010D	ANAB
7440-36-0	Water	Antimony (metallic)	6010D	ANAB
7440-36-0	Soil	Antimony (metallic)	6020B	ANAB
7440-36-0	Water	Antimony (metallic)	6020B	ANAB
N/A	Soil	Aromatic Petroleum Hydrocarbons (>C10 – C12)	NW-EPH	ANAB
N/A	Water	Aromatic Petroleum Hydrocarbons (>C10 – C12)	NW-EPH	ANAB
N/A	Soil	Aromatic Petroleum Hydrocarbons (>C10 – C12)	NW-VPH	ANAB
N/A	Water	Aromatic Petroleum Hydrocarbons (>C10 – C12)	NW-VPH	ANAB
N/A	Soil	Aromatic Petroleum Hydrocarbons (>C12 – C13)	NW-VPH	ANAB
N/A	Water	Aromatic Petroleum Hydrocarbons (>C12 – C13)	NW-VPH	ANAB
N/A	Soil	Aromatic Petroleum Hydrocarbons (>C12 – C16)	NW-EPH	ANAB
N/A	Water	Aromatic Petroleum Hydrocarbons (>C12 – C16)	NW-EPH	ANAB
N/A	Soil	Aromatic Petroleum Hydrocarbons (>C16 – C21)	NW-EPH	ANAB
N/A	Water	Aromatic Petroleum Hydrocarbons (>C16 – C21)	NW-EPH	ANAB
N/A	Soil	Aromatic Petroleum Hydrocarbons (>C21 – C34)	NW-EPH	ANAB
N/A	Water	Aromatic Petroleum Hydrocarbons (>C21 – C34)	NW-EPH	ANAB
N/A	Soil	Aromatic Petroleum Hydrocarbons (>C8 – C10)	NW-VPH	ANAB
N/A	Water	Aromatic Petroleum Hydrocarbons (>C8 – C10)	NW-VPH	ANAB
N/A	Soil	Aromatic Petroleum Hydrocarbons (C8 – C10)	NW-EPH	ANAB
N/A	Water	Aromatic Petroleum Hydrocarbons (C8 – C10)	NW-EPH	ANAB
7440-38-2	Water	Arsenic, Inorganic	200.7	ANAB
7440-38-2	Water	Arsenic, Inorganic	200.8	ANAB
7440-38-2	Soil	Arsenic, Inorganic	6010D	ANAB

Scope of Approval

CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
7440-38-2	Water	Arsenic, Inorganic	6010D	ANAB
7440-38-2	Soil	Arsenic, Inorganic	6020B	ANAB
7440-38-2	Water	Arsenic, Inorganic	6020B	ANAB
7440-39-3	Water	Barium	200.7	ANAB
7440-39-3	Water	Barium	200.8	ANAB
7440-39-3	Soil	Barium	6010D	ANAB
7440-39-3	Water	Barium	6010D	ANAB
7440-39-3	Soil	Barium	6020B	ANAB
7440-39-3	Water	Barium	6020B	ANAB
56-55-3	Water	Benz[a]anthracene	625.1	ANAB
56-55-3	Soil	Benz[a]anthracene	8270E	ANAB
56-55-3	Water	Benz[a]anthracene	8270E	ANAB
56-55-3	Soil	Benz[a]anthracene	8270E-SIM	ANAB
56-55-3	Water	Benz[a]anthracene	8270E-SIM	ANAB
71-43-2	Water	Benzene	624.1	ANAB
71-43-2	Soil	Benzene	8260D	ANAB
71-43-2	Water	Benzene	8260D	ANAB
71-43-2	Soil	Benzene	8260D-SIM	ANAB
71-43-2	Water	Benzene	8260D-SIM	ANAB
50-32-8	Water	Benzo[a]pyrene	625.1	ANAB
50-32-8	Soil	Benzo[a]pyrene	8270E	ANAB
50-32-8	Water	Benzo[a]pyrene	8270E	ANAB
50-32-8	Soil	Benzo[a]pyrene	8270E-SIM	ANAB
50-32-8	Water	Benzo[a]pyrene	8270E-SIM	ANAB
205-99-2	Water	Benzo[b]fluoranthene	625.1	ANAB
205-99-2	Soil	Benzo[b]fluoranthene	8270E	ANAB
205-99-2	Water	Benzo[b]fluoranthene	8270E	ANAB
205-99-2	Soil	Benzo[b]fluoranthene	8270E-SIM	ANAB
205-99-2	Water	Benzo[b]fluoranthene	8270E-SIM	ANAB
191-24-2	Water	Benzo[g,h,i]perylene	625.1	ANAB
191-24-2	Soil	Benzo[g,h,i]perylene	8270E	ANAB
191-24-2	Water	Benzo[g,h,i]perylene	8270E	ANAB
191-24-2	Soil	Benzo[g,h,i]perylene	8270E-SIM	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
191-24-2	Water	Benzo[g,h,i]perylene	8270E-SIM	ANAB
207-08-9	Water	Benzo[k]fluoranthene	625.1	ANAB
207-08-9	Soil	Benzo[k]fluoranthene	8270E	ANAB
207-08-9	Water	Benzo[k]fluoranthene	8270E	ANAB
207-08-9	Soil	Benzo[k]fluoranthene	8270E-SIM	ANAB
207-08-9	Water	Benzo[k]fluoranthene	8270E-SIM	ANAB
65-85-0	Water	Benzoic Acid	625.1	ANAB
65-85-0	Soil	Benzoic Acid	8270E	ANAB
65-85-0	Water	Benzoic Acid	8270E	ANAB
100-51-6	Water	Benzyl Alcohol	625.1	ANAB
100-51-6	Soil	Benzyl Alcohol	8270E	ANAB
100-51-6	Water	Benzyl Alcohol	8270E	ANAB
7440-41-7	Water	Beryllium and compounds	200.7	ANAB
7440-41-7	Water	Beryllium and compounds	200.8	ANAB
7440-41-7	Soil	Beryllium and compounds	6010D	ANAB
7440-41-7	Water	Beryllium and compounds	6010D	ANAB
7440-41-7	Soil	Beryllium and compounds	6020B	ANAB
7440-41-7	Water	Beryllium and compounds	6020B	ANAB
111-44-4	Water	Bis(2-chloroethyl)ether	625.1	ANAB
111-44-4	Soil	Bis(2-chloroethyl)ether	8270E	ANAB
111-44-4	Water	Bis(2-chloroethyl)ether	8270E	ANAB
117-81-7	Water	Bis(2-ethylhexyl)phthalate (DEHP)	625.1	ANAB
117-81-7	Soil	Bis(2-ethylhexyl)phthalate (DEHP)	8270E	ANAB
117-81-7	Water	Bis(2-ethylhexyl)phthalate (DEHP)	8270E	ANAB
108-86-1	Water	Bromobenzene	624.1	ANAB
108-86-1	Soil	Bromobenzene	8260D	ANAB
108-86-1	Water	Bromobenzene	8260D	ANAB
75-27-4	Water	Bromodichloromethane	624.1	ANAB
75-27-4	Soil	Bromodichloromethane	8260D	ANAB
75-27-4	Water	Bromodichloromethane	8260D	ANAB
75-27-4	Soil	Bromodichloromethane	8260D-SIM	ANAB
75-27-4	Water	Bromodichloromethane	8260D-SIM	ANAB
75-25-2	Water	Bromoform	624.1	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
75-25-2	Soil	Bromoform	8260D	ANAB
75-25-2	Water	Bromoform	8260D	ANAB
75-25-2	Soil	Bromoform	8260D-SIM	ANAB
75-25-2	Water	Bromoform	8260D-SIM	ANAB
74-83-9	Water	Bromomethane	624.1	ANAB
74-83-9	Soil	Bromomethane	8260D	ANAB
74-83-9	Water	Bromomethane	8260D	ANAB
74-83-9	Soil	Bromomethane	8260D-SIM	ANAB
74-83-9	Water	Bromomethane	8260D-SIM	ANAB
106-99-0	Soil	Butadiene, 1,3-	8260D-SIM	ANAB
106-99-0	Water	Butadiene, 1,3-	8260D-SIM	ANAB
71-36-3	Soil	Butanol, N-	8260D	ANAB
71-36-3	Water	Butanol, N-	8260D	ANAB
85-68-7	Water	Butyl Benzyl Phthalate	625.1	ANAB
85-68-7	Soil	Butyl Benzyl Phthalate	8270E	ANAB
85-68-7	Water	Butyl Benzyl Phthalate	8270E	ANAB
104-51-8	Water	Butylbenzene, n-	624.1	ANAB
104-51-8	Soil	Butylbenzene, n-	8260D	ANAB
104-51-8	Water	Butylbenzene, n-	8260D	ANAB
104-51-8	Soil	Butylbenzene, n-	8260D-SIM	ANAB
104-51-8	Water	Butylbenzene, n-	8260D-SIM	ANAB
135-98-8	Water	Butylbenzene, sec-	624.1	ANAB
135-98-8	Soil	Butylbenzene, sec-	8260D	ANAB
135-98-8	Water	Butylbenzene, sec-	8260D	ANAB
135-98-8	Soil	Butylbenzene, sec-	8260D-SIM	ANAB
135-98-8	Water	Butylbenzene, sec-	8260D-SIM	ANAB
98-06-6	Water	Butylbenzene, tert-	624.1	ANAB
98-06-6	Soil	Butylbenzene, tert-	8260D	ANAB
98-06-6	Water	Butylbenzene, tert-	8260D	ANAB
98-06-6	Soil	Butylbenzene, tert-	8260D-SIM	ANAB
98-06-6	Water	Butylbenzene, tert-	8260D-SIM	ANAB
7440-43-9	Water	Cadmium	200.7	ANAB
7440-43-9	Water	Cadmium	200.8	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
7440-43-9	Soil	Cadmium	6010D	ANAB
7440-43-9	Water	Cadmium	6010D	ANAB
7440-43-9	Soil	Cadmium	6020B	ANAB
7440-43-9	Water	Cadmium	6020B	ANAB
75-15-0	Water	Carbon Disulfide	624.1	ANAB
75-15-0	Soil	Carbon Disulfide	8260D	ANAB
75-15-0	Water	Carbon Disulfide	8260D	ANAB
56-23-5	Water	Carbon Tetrachloride	624.1	ANAB
56-23-5	Soil	Carbon Tetrachloride	8260D	ANAB
56-23-5	Water	Carbon Tetrachloride	8260D	ANAB
5103-71-9	Water	Chlordane, α-	608.3	ANAB
5103-71-9	Soil	Chlordane, α-	8081A	ANAB
5103-71-9	Water	Chlordane, α-	8081A	ANAB
5103-71-9	Soil	Chlordane, α-	8081B	ANAB
5103-71-9	Water	Chlordane, α-	8081B	ANAB
5103-74-2	Water	Chlordane, γ-	608.3	ANAB
5103-74-2	Soil	Chlordane, γ-	8081A	ANAB
5103-74-2	Water	Chlordane, γ-	8081A	ANAB
5103-74-2	Soil	Chlordane, γ-	8081B	ANAB
5103-74-2	Water	Chlordane, γ-	8081B	ANAB
12789-03-6	Water	Chlordane, Total	608.3	ANAB
12789-03-6	Soil	Chlordane, Total	8081A	ANAB
12789-03-6	Water	Chlordane, Total	8081A	ANAB
12789-03-6	Soil	Chlordane, Total	8081B	ANAB
12789-03-6	Water	Chlordane, Total	8081B	ANAB
106-47-8	Water	Chloroaniline, p-	625.1	ANAB
106-47-8	Soil	Chloroaniline, p-	8270E	ANAB
106-47-8	Water	Chloroaniline, p-	8270E	ANAB
108-90-7	Water	Chlorobenzene	624.1	ANAB
108-90-7	Soil	Chlorobenzene	8260D	ANAB
108-90-7	Water	Chlorobenzene	8260D	ANAB
67-66-3	Water	Chloroform	624.1	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
67-66-3	Soil	Chloroform	8260D	ANAB
67-66-3	Water	Chloroform	8260D	ANAB
67-66-3	Soil	Chloroform	8260D-SIM	ANAB
67-66-3	Water	Chloroform	8260D-SIM	ANAB
74-87-3	Water	Chloromethane	624.1	ANAB
74-87-3	Soil	Chloromethane	8260D	ANAB
74-87-3	Water	Chloromethane	8260D	ANAB
91-58-7	Water	Chloronaphthalene, Beta	625.1	ANAB
91-58-7	Soil	Chloronaphthalene, Beta-	8270E	ANAB
91-58-7	Water	Chloronaphthalene, Beta-	8270E	ANAB
91-58-7	Water	Chlorophenol, 2-	625.1	ANAB
95-57-8	Soil	Chlorophenol, 2-	8270E	ANAB
95-57-8	Water	Chlorophenol, 2-	8270E	ANAB
7440-47-3	Water	Chromium (Total)	200.7	ANAB
7440-47-3	Water	Chromium (Total)	200.8	ANAB
7440-47-3	Soil	Chromium (Total)	6010D	ANAB
7440-47-3	Water	Chromium (Total)	6010D	ANAB
7440-47-3	Soil	Chromium (Total)	6020B	ANAB
7440-47-3	Water	Chromium (Total)	6020B	ANAB
218-01-9	Water	Chrysene	625.1	ANAB
218-01-9	Soil	Chrysene	8270E	ANAB
218-01-9	Water	Chrysene	8270E	ANAB
218-01-9	Soil	Chrysene	8270E-SIM	ANAB
218-01-9	Water	Chrysene	8270E-SIM	ANAB
7440-50-8	Water	Copper	200.7	ANAB
7440-50-8	Water	Copper	200.8	ANAB
7440-50-8	Soil	Copper	6010D	ANAB
7440-50-8	Water	Copper	6010D	ANAB
7440-50-8	Soil	Copper	6020B	ANAB
7440-50-8	Water	Copper	6020B	ANAB
N/A	Water	Cresol, m- (3-Methylphenol) + Cresol, p- (4-Methylphenol)	625.1	ANAB
N/A	Soil	Cresol, m- (3-Methylphenol) + Cresol, p- (4-Methylphenol)	8270E	ANAB
N/A	Water	Cresol, m- (3-Methylphenol) + Cresol, p- (4-Methylphenol)	8270E	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
95-48-7	Water	Cresol, o- (2-Methylphenol)	625.1	ANAB
95-48-7	Soil	Cresol, o- (2-Methylphenol)	8270E	ANAB
95-48-7	Water	Cresol, o- (2-Methylphenol)	8270E	ANAB
98-82-8	Water	Cumene (Isopropylbenzene)	624.1	ANAB
98-82-8	Soil	Cumene (Isopropylbenzene)	8260D	ANAB
98-82-8	Water	Cumene (Isopropylbenzene)	8260D	ANAB
98-82-8	Soil	Cumene (Isopropylbenzene)	8260D-SIM	ANAB
98-82-8	Water	Cumene (Isopropylbenzene)	8260D-SIM	ANAB
57-12-5	Soil	Cyanide (CN-)	9012B	ANAB
57-12-5	Water	Cyanide (CN-)	9012B	ANAB
110-82-7	Water	Cyclohexane	624.1	ANAB
110-82-7	Soil	Cyclohexane	8260D	ANAB
110-82-7	Water	Cyclohexane	8260D	ANAB
72-54-8	Water	DDD, 4,4’-	608.3	ANAB
72-54-8	Soil	DDD, 4,4’-	8081A	ANAB
72-54-8	Water	DDD, 4,4’-	8081A	ANAB
72-54-8	Soil	DDD, 4,4’-	8081B	ANAB
72-54-8	Water	DDD, 4,4’-	8081B	ANAB
72-55-9	Water	DDE, 4,4’-	608.3	ANAB
72-55-9	Soil	DDE, 4,4’-	8081A	ANAB
72-55-9	Water	DDE, 4,4’-	8081A	ANAB
72-55-9	Soil	DDE, 4,4’-	8081B	ANAB
72-55-9	Water	DDE, 4,4’-	8081B	ANAB
50-29-3	Water	DDT, 4,4’-	608.3	ANAB
50-29-3	Soil	DDT, 4,4’-	8081A	ANAB
50-29-3	Water	DDT, 4,4’-	8081A	ANAB
50-29-3	Soil	DDT, 4,4’-	8081B	ANAB
50-29-3	Water	DDT, 4,4’-	8081B	ANAB
53-70-3	Water	Dibenz[a,h]anthracene	625.1	ANAB
53-70-3	Soil	Dibenz[a,h]anthracene	8270E	ANAB
53-70-3	Water	Dibenz[a,h]anthracene	8270E	ANAB
53-70-3	Soil	Dibenz[a,h]anthracene	8270E-SIM	ANAB
53-70-3	Water	Dibenz[a,h]anthracene	8270E-SIM	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
132-64-9	Water	Dibenzofuran	625.1	ANAB
132-64-9	Soil	Dibenzofuran	8270E	ANAB
132-64-9	Water	Dibenzofuran	8270E	ANAB
124-48-1	Water	Dibromochloromethane	624.1	ANAB
124-48-1	Soil	Dibromochloromethane	8260D	ANAB
124-48-1	Water	Dibromochloromethane	8260D	ANAB
124-48-1	Soil	Dibromochloromethane	8260D-SIM	ANAB
124-48-1	Water	Dibromochloromethane	8260D-SIM	ANAB
106-93-4	Water	Dibromoethane, 1,2- (Ethylene Dibromide)	624.1	ANAB
106-93-4	Soil	Dibromoethane, 1,2- (Ethylene Dibromide)	8011	ANAB
106-93-4	Water	Dibromoethane, 1,2- (Ethylene Dibromide)	8011	ANAB
106-93-4	Soil	Dibromoethane, 1,2- (Ethylene Dibromide)	8260D	ANAB
106-93-4	Water	Dibromoethane, 1,2- (Ethylene Dibromide)	8260D	ANAB
106-93-4	Soil	Dibromoethane, 1,2- (Ethylene Dibromide)	8260D-SIM	ANAB
106-93-4	Water	Dibromoethane, 1,2- (Ethylene Dibromide)	8260D-SIM	ANAB
74-95-3	Water	Dibromomethane (Methylene Bromide)	624.1	ANAB
74-95-3	Soil	Dibromomethane (Methylene Bromide)	8260D	ANAB
74-95-3	Water	Dibromomethane (Methylene Bromide)	8260D	ANAB
74-95-3	Soil	Dibromomethane (Methylene Bromide)	8260D-SIM	ANAB
74-95-3	Water	Dibromomethane (Methylene Bromide)	8260D-SIM	ANAB
84-74-2	Water	Dibutyl Phthalate	625.1	ANAB
84-74-2	Soil	Dibutyl Phthalate	8270E	ANAB
84-74-2	Water	Dibutyl Phthalate	8270E	ANAB
95-50-1	Water	Dichlorobenzene, 1,2-	624.1	ANAB
95-50-1	Water	Dichlorobenzene, 1,2-	625.1	ANAB
95-50-1	Soil	Dichlorobenzene, 1,2-	8260D	ANAB
95-50-1	Water	Dichlorobenzene, 1,2-	8260D	ANAB
95-50-1	Soil	Dichlorobenzene, 1,2-	8270E	ANAB
95-50-1	Water	Dichlorobenzene, 1,2-	8270E	ANAB
541-73-1	Soil	Dichlorobenzene, 1,3	8260D	ANAB
541-73-1	Water	Dichlorobenzene, 1,3	8260D	ANAB
541-73-1	Soil	Dichlorobenzene, 1,3	8270E	ANAB
541-73-1	Water	Dichlorobenzene, 1,3	8270E	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
541-73-1	Water	Dichlorobenzene, 1,3-	624.1	ANAB
541-73-1	Water	Dichlorobenzene, 1,3-	625.1	ANAB
106-46-7	Water	Dichlorobenzene, 1,4-	624.1	ANAB
106-46-7	Water	Dichlorobenzene, 1,4-	625.1	ANAB
106-46-7	Soil	Dichlorobenzene, 1,4-	8260D	ANAB
106-46-7	Water	Dichlorobenzene, 1,4-	8260D	ANAB
106-46-7	Soil	Dichlorobenzene, 1,4-	8260D-SIM	ANAB
106-46-7	Water	Dichlorobenzene, 1,4-	8260D-SIM	ANAB
106-46-7	Soil	Dichlorobenzene, 1,4-	8270E	ANAB
106-46-7	Water	Dichlorobenzene, 1,4-	8270E	ANAB
91-94-1	Water	Dichlorobenzidine, 3,3'-	625.1	ANAB
91-94-1	Soil	Dichlorobenzidine, 3,3'-	8270E	ANAB
91-94-1	Water	Dichlorobenzidine, 3,3'-	8270E	ANAB
75-71-8	Water	Dichlorodifluoromethane (Freon-12)	624.1	ANAB
75-71-8	Soil	Dichlorodifluoromethane (Freon-12)	8260D	ANAB
75-71-8	Water	Dichlorodifluoromethane (Freon-12)	8260D	ANAB
75-34-3	Water	Dichloroethane, 1,1-	624.1	ANAB
75-34-3	Soil	Dichloroethane, 1,1-	8260D	ANAB
75-34-3	Water	Dichloroethane, 1,1-	8260D	ANAB
107-06-2	Water	Dichloroethane, 1,2-	624.1	ANAB
107-06-2	Soil	Dichloroethane, 1,2-	8260D	ANAB
107-06-2	Water	Dichloroethane, 1,2-	8260D	ANAB
107-06-2	Soil	Dichloroethane, 1,2-	8260D-SIM	ANAB
107-06-2	Water	Dichloroethane, 1,2-	8260D-SIM	ANAB
75-35-4	Water	Dichloroethylene, 1,1-	624.1	ANAB
75-35-4	Soil	Dichloroethylene, 1,1-	8260D	ANAB
75-35-4	Water	Dichloroethylene, 1,1-	8260D	ANAB
75-35-4	Soil	Dichloroethylene, 1,1-	8260D-SIM	ANAB
75-35-4	Water	Dichloroethylene, 1,1-	8260D-SIM	ANAB
156-59-2	Water	Dichloroethylene, 1,2-cis-	624.1	ANAB
156-59-2	Soil	Dichloroethylene, 1,2-cis-	8260D	ANAB
156-59-2	Water	Dichloroethylene, 1,2-cis-	8260D	ANAB
156-60-5	Water	Dichloroethylene, 1,2-trans-	624.1	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
156-60-5	Soil	Dichloroethylene, 1,2-trans-	8260D	ANAB
156-60-5	Water	Dichloroethylene, 1,2-trans-	8260D	ANAB
120-83-2	Water	Dichlorophenol, 2,4-	625.1	ANAB
120-83-2	Soil	Dichlorophenol, 2,4-	8270E	ANAB
120-83-2	Water	Dichlorophenol, 2,4-	8270E	ANAB
78-87-5	Water	Dichloropropane, 1,2-	624.1	ANAB
78-87-5	Soil	Dichloropropane, 1,2-	8260D	ANAB
78-87-5	Water	Dichloropropane, 1,2-	8260D	ANAB
542-75-6	Water	Dichloropropene, 1,3- (cis + trans)	624.1	ANAB
542-75-6	Soil	Dichloropropene, 1,3- (cis + trans)	8260D	ANAB
542-75-6	Water	Dichloropropene, 1,3- (cis + trans)	8260D	ANAB
10061-01-5	Water	Dichloropropene, cis-1,3-	624.1	ANAB
10061-01-5	Soil	Dichloropropene, cis-1,3-	8260D	ANAB
10061-01-5	Water	Dichloropropene, cis-1,3-	8260D	ANAB
10061-01-5	Soil	Dichloropropene, cis-1,3-	8260D-SIM	ANAB
10061-01-5	Water	Dichloropropene, cis-1,3-	8260D-SIM	ANAB
10061-02-6	Water	Dichloropropene, trans-1,3-	624.1	ANAB
10061-02-6	Soil	Dichloropropene, trans-1,3-	8260D	ANAB
10061-02-6	Water	Dichloropropene, trans-1,3-	8260D	ANAB
10061-02-6	Soil	Dichloropropene, trans-1,3-	8260D-SIM	ANAB
10061-02-6	Water	Dichloropropene, trans-1,3-	8260D-SIM	ANAB
60-57-1	Water	Dieldrin	608.3	ANAB
60-57-1	Soil	Dieldrin	8081A	ANAB
60-57-1	Water	Dieldrin	8081A	ANAB
60-57-1	Soil	Dieldrin	8081B	ANAB
60-57-1	Water	Dieldrin	8081B	ANAB
N/A	Soil	Diesel Range Organics (C10 – C25)	AK 102	ANAB
N/A	Water	Diesel Range Organics (C10 – C25)	AK 102	ANAB
84-66-2	Water	Diethyl Phthalate	625.1	ANAB
84-66-2	Soil	Diethyl Phthalate	8270E	ANAB
84-66-2	Water	Diethyl Phthalate	8270E	ANAB
105-67-9	Water	Dimethylphenol, 2,4-	625.1	ANAB
105-67-9	Soil	Dimethylphenol, 2,4-	8270E	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
105-67-9	Water	Dimethylphenol, 2,4-	8270E	ANAB
131-11-3	Water	Dimethylphthalate	625.1	ANAB
131-11-3	Soil	Dimethylphthalate	8270E	ANAB
131-11-3	Water	Dimethylphthalate	8270E	ANAB
51-28-5	Water	Dinitrophenol, 2,4-	625.1	ANAB
51-28-5	Soil	Dinitrophenol, 2,4-	8270E	ANAB
51-28-5	Water	Dinitrophenol, 2,4-	8270E	ANAB
121-14-2	Water	Dinitrotoluene, 2,4- (2,4-DNT)	625.1	ANAB
121-14-2	Soil	Dinitrotoluene, 2,4- (2,4-DNT)	8270E	ANAB
121-14-2	Water	Dinitrotoluene, 2,4- (2,4-DNT)	8270E	ANAB
606-20-2	Water	Dinitrotoluene, 2,6- (2,6-DNT)	625.1	ANAB
606-20-2	Soil	Dinitrotoluene, 2,6- (2,6-DNT)	8270E	ANAB
606-20-2	Water	Dinitrotoluene, 2,6- (2,6-DNT)	8270E	ANAB
123-91-1	Soil	Dioxane, 1,4-	8270E-SIM	ANAB
123-91-1	Water	Dioxane, 1,4-	8270E-SIM	ANAB
959-98-8	Water	Endosulfan I	608.3	ANAB
959-98-8	Water	Endosulfan I	8081A	ANAB
959-98-8	Water	Endosulfan I	8081A	ANAB
959-98-8	Soil	Endosulfan I	8081B	ANAB
959-98-8	Soil	Endosulfan I	8081B	ANAB
33213-65-9	Water	Endosulfan II	608.3	ANAB
33213-65-9	Soil	Endosulfan II	8081A	ANAB
33213-65-9	Water	Endosulfan II	8081A	ANAB
33213-65-9	Soil	Endosulfan II	8081B	ANAB
33213-65-9	Water	Endosulfan II	8081B	ANAB
1031-07-8	Water	Endosulfan sulfate	608.3	ANAB
1031-07-8	Soil	Endosulfan sulfate	8081A	ANAB
1031-07-8	Water	Endosulfan sulfate	8081A	ANAB
1031-07-8	Soil	Endosulfan sulfate	8081B	ANAB
1031-07-8	Water	Endosulfan sulfate	8081B	ANAB
72-20-8	Water	Endrin	608.3	ANAB
72-20-8	Soil	Endrin	8081A	ANAB
72-20-8	Water	Endrin	8081A	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
72-20-8	Soil	Endrin	8081B	ANAB
72-20-8	Water	Endrin	8081B	ANAB
75-00-3	Water	Ethyl Chloride	624.1	ANAB
75-00-3	Soil	Ethyl Chloride	8260D	ANAB
75-00-3	Water	Ethyl Chloride	8260D	ANAB
100-41-4	Water	Ethylbenzene	624.1	ANAB
100-41-4	Soil	Ethylbenzene	8260D	ANAB
100-41-4	Water	Ethylbenzene	8260D	ANAB
100-41-4	Soil	Ethylbenzene	8260D-SIM	ANAB
100-41-4	Water	Ethylbenzene	8260D-SIM	ANAB
206-44-0	Water	Fluoranthene	625.1	ANAB
206-44-0	Soil	Fluoranthene	8270E	ANAB
206-44-0	Water	Fluoranthene	8270E	ANAB
206-44-0	Soil	Fluoranthene	8270E-SIM	ANAB
206-44-0	Water	Fluoranthene	8270E-SIM	ANAB
86-73-7	Water	Fluorene	625.1	ANAB
86-73-7	Soil	Fluorene	8270E	ANAB
86-73-7	Water	Fluorene	8270E	ANAB
86-73-7	Soil	Fluorene	8270E-SIM	ANAB
86-73-7	Water	Fluorene	8270E-SIM	ANAB
N/A	Soil	Gasoline Range Organics (C6 – C10)	AK 101	ANAB
N/A	Water	Gasoline Range Organics (C6 – C10)	AK 101	ANAB
N/A	Soil	Gasoline Range Organics (C6 – C10)	AK 101-MS	ANAB
N/A	Water	Gasoline Range Organics (C6 – C10)	AK 101-MS	ANAB
76-44-8	Water	Heptachlor	608.3	ANAB
76-44-8	Soil	Heptachlor	8081A	ANAB
76-44-8	Water	Heptachlor	8081A	ANAB
76-44-8	Soil	Heptachlor	8081B	ANAB
76-44-8	Water	Heptachlor	8081B	ANAB
1024-57-3	Water	Heptachlor Epoxide	608.3	ANAB
1024-57-3	Soil	Heptachlor Epoxide	8081A	ANAB
1024-57-3	Water	Heptachlor Epoxide	8081A	ANAB
1024-57-3	Soil	Heptachlor Epoxide	8081B	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
1024-57-3	Water	Heptachlor Epoxide	8081B	ANAB
118-74-1	Water	Hexachlorobenzene	625.1	ANAB
118-74-1	Soil	Hexachlorobenzene	8081A	ANAB
118-74-1	Water	Hexachlorobenzene	8081A	ANAB
118-74-1	Soil	Hexachlorobenzene	8081B	ANAB
118-74-1	Water	Hexachlorobenzene	8081B	ANAB
118-74-1	Soil	Hexachlorobenzene	8270E	ANAB
118-74-1	Water	Hexachlorobenzene	8270E	ANAB
87-68-3	Water	Hexachlorobutadiene	624.1	ANAB
87-68-3	Water	Hexachlorobutadiene	625.1	ANAB
87-68-3	Soil	Hexachlorobutadiene	8081A	ANAB
87-68-3	Water	Hexachlorobutadiene	8081A	ANAB
87-68-3	Soil	Hexachlorobutadiene	8081B	ANAB
87-68-3	Water	Hexachlorobutadiene	8081B	ANAB
87-68-3	Soil	Hexachlorobutadiene	8260D	ANAB
87-68-3	Water	Hexachlorobutadiene	8260D	ANAB
87-68-3	Soil	Hexachlorobutadiene	8260D-SIM	ANAB
87-68-3	Water	Hexachlorobutadiene	8260D-SIM	ANAB
87-68-3	Soil	Hexachlorobutadiene	8270E	ANAB
87-68-3	Water	Hexachlorobutadiene	8270E	ANAB
319-84-6	Water	Hexachlorocyclohexane, Alpha- (α-BHC)	608.3	ANAB
319-84-6	Soil	Hexachlorocyclohexane, Alpha- (α-BHC)	8081A	ANAB
319-84-6	Water	Hexachlorocyclohexane, Alpha- (α-BHC)	8081A	ANAB
319-84-6	Soil	Hexachlorocyclohexane, Alpha- (α-BHC)	8081B	ANAB
319-84-6	Water	Hexachlorocyclohexane, Alpha- (α-BHC)	8081B	ANAB
319-85-7	Water	Hexachlorocyclohexane, Beta- (β-BHC)	608.3	ANAB
319-85-7	Soil	Hexachlorocyclohexane, Beta- (β-BHC)	8081A	ANAB
319-85-7	Water	Hexachlorocyclohexane, Beta- (β-BHC)	8081A	ANAB
319-85-7	Soil	Hexachlorocyclohexane, Beta- (β-BHC)	8081B	ANAB
319-85-7	Water	Hexachlorocyclohexane, Beta- (β-BHC)	8081B	ANAB
319-86-8	Water	Hexachlorocyclohexane, Delta- (δ-BHC)	608.3	ANAB
319-86-8	Soil	Hexachlorocyclohexane, Delta- (δ-BHC)	8081A	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
319-86-8	Water	Hexachlorocyclohexane, Delta- (δ-BHC)	8081A	ANAB
319-86-8	Soil	Hexachlorocyclohexane, Delta- (δ-BHC)	8081B	ANAB
319-86-8	Water	Hexachlorocyclohexane, Delta- (δ-BHC)	8081B	ANAB
58-89-9	Water	Hexachlorocyclohexane, Gamma- (Lindane)	608.3	ANAB
58-89-9	Soil	Hexachlorocyclohexane, Gamma- (Lindane)	8081A	ANAB
58-89-9	Water	Hexachlorocyclohexane, Gamma- (Lindane)	8081A	ANAB
58-89-9	Soil	Hexachlorocyclohexane, Gamma- (Lindane)	8081B	ANAB
58-89-9	Water	Hexachlorocyclohexane, Gamma- (Lindane)	8081B	ANAB
77-47-4	Water	Hexachlorocyclopentadiene	625.1	ANAB
77-47-4	Soil	Hexachlorocyclopentadiene	8270E	ANAB
77-47-4	Water	Hexachlorocyclopentadiene	8270E	ANAB
67-72-1	Water	Hexachloroethane	625.1	ANAB
67-72-1	Soil	Hexachloroethane	8270E	ANAB
67-72-1	Water	Hexachloroethane	8270E	ANAB
110-54-3	Water	Hexane, N-	624.1	ANAB
110-54-3	Soil	Hexane, N-	8260D	ANAB
110-54-3	Water	Hexane, N-	8260D	ANAB
591-78-6	Water	Hexanone, 2-	624.1	ANAB
591-78-6	Soil	Hexanone, 2-	8260D	ANAB
591-78-6	Water	Hexanone, 2-	8260D	ANAB
591-78-6	Soil	Hexanone, 2-	8260D-SIM	ANAB
591-78-6	Water	Hexanone, 2-	8260D-SIM	ANAB
193-39-5	Water	Indeno[1,2,3-cd]pyrene	625.1	ANAB
193-39-5	Soil	Indeno[1,2,3-cd]pyrene	8270E	ANAB
193-39-5	Water	Indeno[1,2,3-cd]pyrene	8270E	ANAB
193-39-5	Soil	Indeno[1,2,3-cd]pyrene	8270E-SIM	ANAB
193-39-5	Water	Indeno[1,2,3-cd]pyrene	8270E-SIM	ANAB
78-59-1	Water	Isophorone	625.1	ANAB
78-59-1	Soil	Isophorone	8270E	ANAB
78-59-1	Water	Isophorone	8270E	ANAB
67-63-0	Soil	Isopropanol	8260D-SIM	ANAB
67-63-0	Water	Isopropanol	8260D-SIM	ANAB
7439-92-1	Water	Lead, Total	200.7	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
7439-92-1	Water	Lead, Total	200.8	ANAB
7439-92-1	Soil	Lead, Total	6010D	ANAB
7439-92-1	Water	Lead, Total	6010D	ANAB
7439-92-1	Soil	Lead, Total	6020B	ANAB
7439-92-1	Water	Lead, Total	6020B	ANAB
7439-96-5	Water	Manganese	200.7	ANAB
7439-96-5	Water	Manganese	200.8	ANAB
7439-96-5	Soil	Manganese	6010D	ANAB
7439-96-5	Water	Manganese	6010D	ANAB
7439-96-5	Soil	Manganese	6020B	ANAB
7439-96-5	Water	Manganese	6020B	ANAB
7439-97-6	Water	Mercury (elemental)	245.1	ANAB
7439-97-6	Soil	Mercury (elemental)	1631B	ANAB
7439-97-6	Water	Mercury (elemental)	1631E	ANAB
7439-97-6	Soil	Mercury (elemental)	7470A	ORELAP
7439-97-6	Water	Mercury (elemental)	7470A	ANAB
7439-97-6	Soil	Mercury (elemental)	7471A	ANAB
72-43-5	Water	Methoxychlor	608.3	ANAB
72-43-5	Soil	Methoxychlor	8081A	ANAB
72-43-5	Water	Methoxychlor	8081A	ANAB
72-43-5	Soil	Methoxychlor	8081B	ANAB
72-43-5	Water	Methoxychlor	8081B	ANAB
78-93-3	Water	Methyl Ethyl Ketone (2-Butanone)	624.1	ANAB
78-93-3	Soil	Methyl Ethyl Ketone (2-Butanone)	8260D	ANAB
78-93-3	Water	Methyl Ethyl Ketone (2-Butanone)	8260D	ANAB
108-10-1	Water	Methyl Isobutyl Ketone (4-methyl-2-pentanone)	624.1	ANAB
108-10-1	Soil	Methyl Isobutyl Ketone (4-methyl-2-pentanone)	8260D	ANAB
108-10-1	Water	Methyl Isobutyl Ketone (4-methyl-2-pentanone)	8260D	ANAB
22967-92-6	Soil	Methyl Mercury	1630	ANAB
22967-92-6	Water	Methyl Mercury	1630	ANAB
1634-04-4	Water	Methyl tert-Butyl Ether (MTBE)	624.1	ANAB
1634-04-4	Soil	Methyl tert-Butyl Ether (MTBE)	8260D	ANAB
1634-04-4	Water	Methyl tert-Butyl Ether (MTBE)	8260D	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
1634-04-4	Soil	Methyl tert-Butyl Ether (MTBE)	8260D-SIM	ANAB
1634-04-4	Water	Methyl tert-Butyl Ether (MTBE)	8260D-SIM	ANAB
75-09-2	Water	Methylene Chloride	624.1	ANAB
75-09-2	Soil	Methylene Chloride	8260D	ANAB
75-09-2	Water	Methylene Chloride	8260D	ANAB
90-12-0	Water	Methylnaphthalene, 1-	625.1	ANAB
90-12-0	Soil	Methylnaphthalene, 1-	8270E	ANAB
90-12-0	Water	Methylnaphthalene, 1-	8270E	ANAB
90-12-0	Soil	Methylnaphthalene, 1-	8270E-SIM	ANAB
90-12-0	Water	Methylnaphthalene, 1-	8270E-SIM	ANAB
91-57-6	Water	Methylnaphthalene, 2-	625.1	ANAB
91-57-6	Soil	Methylnaphthalene, 2-	8270E	ANAB
91-57-6	Water	Methylnaphthalene, 2-	8270E	ANAB
91-57-6	Soil	Methylnaphthalene, 2-	8270E-SIM	ANAB
91-57-6	Water	Methylnaphthalene, 2-	8270E-SIM	ANAB
91-20-3	Water	Naphthalene	624.1	ANAB
91-20-3	Water	Naphthalene	625.1	ANAB
91-20-3	Soil	Naphthalene	8260D	ANAB
91-20-3	Water	Naphthalene	8260D	ANAB
91-20-3	Soil	Naphthalene	8260D-SIM	ANAB
91-20-3	Water	Naphthalene	8260D-SIM	ANAB
91-20-3	Soil	Naphthalene	8270E	ANAB
91-20-3	Water	Naphthalene	8270E	ANAB
91-20-3	Soil	Naphthalene	8270E-SIM	ANAB
91-20-3	Water	Naphthalene	8270E-SIM	ANAB
7440-02-0	Water	Nickel, Total	200.7	ANAB
7440-02-0	Water	Nickel, Total	200.8	ANAB
7440-02-0	Soil	Nickel, Total	6010D	ANAB
7440-02-0	Water	Nickel, Total	6010D	ANAB
7440-02-0	Soil	Nickel, Total	6020B	ANAB
7440-02-0	Water	Nickel, Total	6020B	ANAB
98-95-3	Water	Nitrobenzene	625.1	ANAB
98-95-3	Soil	Nitrobenzene	8270E	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
98-95-3	Water	Nitrobenzene	8270E	ANAB
62-75-9	Water	Nitrosodimethylamine, N-	625.1	ANAB
62-75-9	Soil	Nitrosodimethylamine, N-	8270E	ANAB
62-75-9	Water	Nitrosodimethylamine, N-	8270E	ANAB
621-64-7	Water	Nitroso-di-N-propylamine, N-	625.1	ANAB
621-64-7	Soil	Nitroso-di-N-propylamine, N-	8270E	ANAB
621-64-7	Water	Nitroso-di-N-propylamine, N-	8270E	ANAB
86-30-6	Water	Nitrosodiphenylamine, N-	625.1	ANAB
86-30-6	Soil	Nitrosodiphenylamine, N-	8270E	ANAB
86-30-6	Water	Nitrosodiphenylamine, N-	8270E	ANAB
117-84-0	Water	Octyl Phthalate, di-N-	625.1	ANAB
117-84-0	Soil	Octyl Phthalate, di-N-	8270E	ANAB
117-84-0	Water	Octyl Phthalate, di-N-	8270E	ANAB
12674-11-2	Water	PCB - Aroclor-1016	608.3	ANAB
12674-11-2	Soil	PCB - Aroclor-1016	8082A	ANAB
12674-11-2	Water	PCB - Aroclor-1016	8082A	ANAB
11104-28-2	Water	PCB - Aroclor-1221	608.3	ANAB
11104-28-2	Soil	PCB - Aroclor-1221	8082A	ANAB
11104-28-2	Water	PCB - Aroclor-1221	8082A	ANAB
11141-16-5	Water	PCB - Aroclor-1232	608.3	ANAB
11141-16-5	Soil	PCB - Aroclor-1232	8082A	ANAB
11141-16-5	Water	PCB - Aroclor-1232	8082A	ANAB
53469-21-9	Water	PCB - Aroclor-1242	608.3	ANAB
53469-21-9	Soil	PCB - Aroclor-1242	8082A	ANAB
53469-21-9	Water	PCB - Aroclor-1242	8082A	ANAB
12672-29-6	Water	PCB - Aroclor-1248	608.3	ANAB
12672-29-6	Soil	PCB - Aroclor-1248	8082A	ANAB
12672-29-6	Water	PCB - Aroclor-1248	8082A	ANAB
11097-69-1	Water	PCB - Aroclor-1254	608.3	ANAB
11097-69-1	Soil	PCB - Aroclor-1254	8082A	ANAB
11097-69-1	Water	PCB - Aroclor-1254	8082A	ANAB
11096-82-5	Water	PCB - Aroclor-1260	608.3	ANAB
11096-82-5	Soil	PCB - Aroclor-1260	8082A	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
11096-82-5	Water	PCB - Aroclor-1260	8082A	ANAB
37324-23-5	Water	PCB - Aroclor-1262	608.3	ANAB
37324-23-5	Soil	PCB - Aroclor-1262	8082A	ANAB
37324-23-5	Water	PCB - Aroclor-1262	8082A	ANAB
11100-14-4	Water	PCB - Aroclor-1268	608.3	ANAB
11100-14-4	Soil	PCB - Aroclor-1268	8082A	ANAB
11100-14-4	Water	PCB - Aroclor-1268	8082A	ANAB
87-86-5	Water	Pentachlorophenol	625.1	ANAB
87-86-5	Soil	Pentachlorophenol	8270E	ANAB
87-86-5	Water	Pentachlorophenol	8270E	ANAB
87-86-5	Soil	Pentachlorophenol	8270E-SIM	ANAB
87-86-5	Water	Pentachlorophenol	8270E-SIM	ANAB
85-01-8	Water	Phenanthrene	625.1	ANAB
85-01-8	Soil	Phenanthrene	8270E	ANAB
85-01-8	Water	Phenanthrene	8270E	ANAB
85-01-8	Soil	Phenanthrene	8270E-SIM	ANAB
85-01-8	Water	Phenanthrene	8270E-SIM	ANAB
108-95-2	Water	Phenol	625.1	ANAB
108-95-2	Soil	Phenol	8270E	ANAB
108-95-2	Water	Phenol	8270E	ANAB
103-65-1	Water	Propyl benzene	624.1	ANAB
103-65-1	Soil	Propyl benzene	8260D	ANAB
103-65-1	Water	Propyl benzene	8260D	ANAB
129-00-0	Water	Pyrene	625.1	ANAB
129-00-0	Soil	Pyrene	8270E	ANAB
129-00-0	Water	Pyrene	8270E	ANAB
129-00-0	Soil	Pyrene	8270E-SIM	ANAB
129-00-0	Water	Pyrene	8270E-SIM	ANAB
N/A	Soil	Residual Range Organics (C25 – C36)	AK 103	ANAB
N/A	Water	Residual Range Organics (C25 – C36)	AK 103	ANAB
7782-49-2	Water	Selenium	200.7	ANAB
7782-49-2	Water	Selenium	200.8	ANAB
7782-49-2	Soil	Selenium	6010D	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
7782-49-2	Water	Selenium	6010D	ANAB
7782-49-2	Soil	Selenium	6020B	ANAB
7782-49-2	Water	Selenium	6020B	ANAB
7440-22-4	Water	Silver	200.7	ANAB
7440-22-4	Water	Silver	200.8	ANAB
7440-22-4	Soil	Silver	6010D	ANAB
7440-22-4	Water	Silver	6010D	ANAB
7440-22-4	Soil	Silver	6020B	ANAB
7440-22-4	Water	Silver	6020B	ANAB
7440-24-6	Water	Strontium	200.7	ANAB
7440-24-6	Water	Strontium	200.8	ORELAP
7440-24-6	Soil	Strontium	6010D	ANAB
7440-24-6	Water	Strontium	6010D	ANAB
7440-24-6	Soil	Strontium	6020B	ORELAP
7440-24-6	Water	Strontium	6020B	ORELAP
100-42-5	Water	Styrene	624.1	ANAB
100-42-5	Soil	Styrene	8260D	ANAB
100-42-5	Water	Styrene	8260D	ANAB
630-20-6	Water	Tetrachloroethane, 1,1,1,2-	624.1	ANAB
630-20-6	Soil	Tetrachloroethane, 1,1,1,2-	8260D	ANAB
630-20-6	Water	Tetrachloroethane, 1,1,1,2-	8260D	ANAB
630-20-6	Soil	Tetrachloroethane, 1,1,1,2-	8260D-SIM	ANAB
630-20-6	Water	Tetrachloroethane, 1,1,1,2-	8260D-SIM	ANAB
79-34-5	Water	Tetrachloroethane, 1,1,2,2-	624.1	ANAB
79-34-5	Soil	Tetrachloroethane, 1,1,2,2-	8260D	ANAB
79-34-5	Water	Tetrachloroethane, 1,1,2,2-	8260D	ANAB
79-34-5	Soil	Tetrachloroethane, 1,1,2,2-	8260D-SIM	ANAB
79-34-5	Water	Tetrachloroethane, 1,1,2,2-	8260D-SIM	ANAB
127-18-4	Water	Tetrachloroethylene	624.1	ANAB
127-18-4	Soil	Tetrachloroethylene	8260D	ANAB
127-18-4	Water	Tetrachloroethylene	8260D	ANAB
127-18-4	Soil	Tetrachloroethylene	8260D-SIM	ANAB
127-18-4	Water	Tetrachloroethylene	8260D-SIM	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
7440-28-0	Water	Thallium, Total	200.7	ANAB
7440-28-0	Water	Thallium, Total	200.8	ANAB
7440-28-0	Soil	Thallium, Total	6010D	ANAB
7440-28-0	Water	Thallium, Total	6010D	ANAB
7440-28-0	Soil	Thallium, Total	6020B	ANAB
7440-28-0	Water	Thallium, Total	6020B	ANAB
108-88-3	Water	Toluene	624.1	ANAB
108-88-3	Soil	Toluene	8260D	ANAB
108-88-3	Water	Toluene	8260D	ANAB
108-88-3	Soil	Toluene	8260D-SIM	ANAB
108-88-3	Water	Toluene	8260D-SIM	ANAB
N/A	Soil	Total Organic Carbon	9060	ANAB
N/A	Water	Total Organic Carbon	9060	ANAB
N/A	Soil	Total Organic Carbon	9060A	ANAB
N/A	Water	Total Organic Carbon	9060A	ANAB
N/A	Water	Total Organic Carbon	SM 5310 B	ANAB
8001-35-2	Water	Toxaphene	608.3	ANAB
8001-35-2	Soil	Toxaphene	8081A	ANAB
8001-35-2	Water	Toxaphene	8081A	ANAB
8001-35-2	Soil	Toxaphene	8081B	ANAB
8001-35-2	Water	Toxaphene	8081B	ANAB
76-13-1	Water	Trichloro-1,2,2-trifluoroethane, 1,1,2- (Freon 113)	624.1	ANAB
76-13-1	Soil	Trichloro-1,2,2-trifluoroethane, 1,1,2- (Freon 113)	8260D	ANAB
76-13-1	Water	Trichloro-1,2,2-trifluoroethane, 1,1,2- (Freon 113)	8260D	ANAB
87-61-6	Water	Trichlorobenzene, 1,2,3-	624.1	ANAB
87-61-6	Soil	Trichlorobenzene, 1,2,3-	8260D	ANAB
87-61-6	Water	Trichlorobenzene, 1,2,3-	8260D	ANAB
120-82-1	Water	Trichlorobenzene, 1,2,4-	624.1	ANAB
120-82-1	Water	Trichlorobenzene, 1,2,4-	625.1	ANAB
120-82-1	Soil	Trichlorobenzene, 1,2,4-	8260D	ANAB
120-82-1	Water	Trichlorobenzene, 1,2,4-	8260D	ANAB
120-82-1	Soil	Trichlorobenzene, 1,2,4-	8270E	ANAB
120-82-1	Water	Trichlorobenzene, 1,2,4-	8270E	ANAB

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CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
71-55-6	Water	Trichloroethane, 1,1,1-	624.1	ANAB
71-55-6	Soil	Trichloroethane, 1,1,1-	8260D	ANAB
71-55-6	Water	Trichloroethane, 1,1,1-	8260D	ANAB
79-00-5	Water	Trichloroethane, 1,1,2-	624.1	ANAB
79-00-5	Soil	Trichloroethane, 1,1,2-	8260D	ANAB
79-00-5	Water	Trichloroethane, 1,1,2-	8260D	ANAB
79-00-5	Soil	Trichloroethane, 1,1,2-	8260D-SIM	ANAB
79-00-5	Water	Trichloroethane, 1,1,2-	8260D-SIM	ANAB
79-01-6	Water	Trichloroethylene	624.1	ANAB
79-01-6	Soil	Trichloroethylene	8260D	ANAB
79-01-6	Water	Trichloroethylene	8260D	ANAB
79-01-6	Soil	Trichloroethylene	8260D-SIM	ANAB
79-01-6	Water	Trichloroethylene	8260D-SIM	ANAB
75-69-4	Water	Trichlorofluoromethane	624.1	ANAB
75-69-4	Soil	Trichlorofluoromethane	8260D	ANAB
75-69-4	Water	Trichlorofluoromethane	8260D	ANAB
95-95-4	Water	Trichlorophenol, 2,4,5-	625.1	ANAB
95-95-4	Soil	Trichlorophenol, 2,4,5-	8270E	ANAB
95-95-4	Water	Trichlorophenol, 2,4,5-	8270E	ANAB
88-06-2	Water	Trichlorophenol, 2,4,6-	625.1	ANAB
88-06-2	Soil	Trichlorophenol, 2,4,6-	8270E	ANAB
88-06-2	Water	Trichlorophenol, 2,4,6-	8270E	ANAB
96-18-4	Water	Trichloropropane, 1,2,3-	624.1	ANAB
96-18-4	Soil	Trichloropropane, 1,2,3-	8011	ANAB
96-18-4	Water	Trichloropropane, 1,2,3-	8011	ANAB
96-18-4	Soil	Trichloropropane, 1,2,3-	8260D	ANAB
96-18-4	Water	Trichloropropane, 1,2,3-	8260D	ANAB
95-63-6	Water	Trimethylbenzene, 1,2,4-	624.1	ANAB
95-63-6	Soil	Trimethylbenzene, 1,2,4-	8260D	ANAB
95-63-6	Water	Trimethylbenzene, 1,2,4-	8260D	ANAB
95-63-6	Soil	Trimethylbenzene, 1,2,4-	8260D-SIM	ANAB
95-63-6	Water	Trimethylbenzene, 1,2,4-	8260D-SIM	ANAB
108-67-8	Water	Trimethylbenzene, 1,3,5-	624.1	ANAB

Scope of Approval

CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
108-67-8	Soil	Trimethylbenzene, 1,3,5-	8260D	ANAB
108-67-8	Water	Trimethylbenzene, 1,3,5-	8260D	ANAB
108-67-8	Soil	Trimethylbenzene, 1,3,5-	8260D-SIM	ANAB
108-67-8	Water	Trimethylbenzene, 1,3,5-	8260D-SIM	ANAB
688-73-3	Soil	Tri-n-butyltin	Organotins, PSEP (GC/MS)	ANAB
688-73-3	Water	Tri-n-butyltin	Organotins, PSEP (GC/MS)	ANAB
7440-62-2	Water	Vanadium, Total	200.7	ANAB
7440-62-2	Water	Vanadium, Total	200.8	ANAB
7440-62-2	Soil	Vanadium, Total	6010D	ANAB
7440-62-2	Water	Vanadium, Total	6010D	ANAB
7440-62-2	Soil	Vanadium, Total	6020B	ANAB
7440-62-2	Water	Vanadium, Total	6020B	ANAB
108-05-4	Water	Vinyl Acetate	624.1	ANAB
108-05-4	Soil	Vinyl Acetate	8260D	ANAB
108-05-4	Water	Vinyl Acetate	8260D	ANAB
75-01-4	Water	Vinyl Chloride	624.1	ANAB
75-01-4	Soil	Vinyl Chloride	8260D	ANAB
75-01-4	Water	Vinyl Chloride	8260D	ANAB
75-01-4	Soil	Vinyl Chloride	8260D-SIM	ANAB
75-01-4	Water	Vinyl Chloride	8260D-SIM	ANAB
N/A	Water	Xylene, m+p-	624.1	ANAB
N/A	Soil	Xylene, m+p-	8260D	ANAB
N/A	Water	Xylene, m+p-	8260D	ANAB
N/A	Soil	Xylene, m+p-	8260D-SIM	ORELAP
179601-23-1	Soil	Xylene, m+p-	8260D-SIM	ANAB
N/A	Water	Xylene, m+p-	8260D-SIM	ORELAP
179601-23-1	Water	Xylene, m+p-	8260D-SIM	ANAB
95-47-6	Water	Xylene, o-	624.1	ANAB
95-47-6	Soil	Xylene, o-	8260D	ANAB
95-47-6	Water	Xylene, o-	8260D	ANAB
95-47-6	Soil	Xylene, o-	8260D-SIM	ANAB
95-47-6	Water	Xylene, o-	8260D-SIM	ANAB
1330-20-7	Water	Xylene, Total	624.1	ANAB

Scope of Approval

CAS Number	Matrix	Hazardous Substance	Method	Accreditation Body
1330-20-7	Soil	Xylene, Total	8260D	ANAB
1330-20-7	Water	Xylene, Total	8260D	ANAB
7440-66-6	Water	Zinc, Total	200.7	ANAB
7440-66-6	Water	Zinc, Total	200.8	ANAB
7440-66-6	Soil	Zinc, Total	6010D	ANAB
7440-66-6	Water	Zinc, Total	6010D	ANAB
7440-66-6	Soil	Zinc, Total	6020B	ANAB
7440-66-6	Water	Zinc, Total	6020B	ANAB

Appendix B. Health and Safety Plan

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FINAL
HEALTH AND SAFETY PLAN
Version 0

Health and Safety Plan
Red Devil Mine, Alaska

Contract Number: 140L63-22-P-0046

Submitted to:

U.S. DEPARTMENT OF INTERIOR
BUREAU OF LAND MANAGEMENT
Anchorage Field Office
222 West 7th Ave #13
Anchorage, Alaska 99513

Submitted by:

Sundance Consulting Inc.
333 S Main Street, Suite 20
Pocatello ID 83204

March 2023

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Revision Log

Revision	Date	Reason for Revision
0	3/13/2023	BLM Approved

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Contents

List of Attachments

United States Department of the Interior - Bureau of Land Management

Risk Management Worksheets

- All-Terrain Vehicles
- Working in Remote Areas
- Hazardous Materials
- Hunter Encounters
- Wildlife Encounters
- Heat/Cold Stress
- Hazardous Weather Changes
- COVID-19 Related Hazards
- Mine Site Hazards

List of Acronyms and Abbreviations

ACGIH	American Conference of Governmental Industrial Hygienists
ANSI	American National Standards Institute
ATV	all-terrain vehicle
CFR	Code of Federal Regulations
CDC	Centers for Disease Control and Prevention
CPR	cardiopulmonary resuscitation
CoC	contaminant of concern
dBA	decibels average
DEET	N, N-diethyl-m-toluamide
GDA	government designated authority
GFCI	ground fault circuit interrupters
HAZWOPER	Hazardous Waste Operations and Emergency Response
HEPA	high-efficiency particulate air
ISEA	International Safety Equipment Association
OSHA	Occupational Safety and Health Administration
PPE	personal protective equipment
RDM	Red Devil Mine
SSHO	Site Safety and Health Officer
Sundance	Sundance Consulting Inc.
TLV	threshold limit value
TWA	time-weighted average
V	volt
Work Plan	*Final Work Plan, Groundwater and Surface Water Baseline Monitoring, Red Devil Mine, Alaska*

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1.0 Site Description and Contamination Characterization

The Red Devil Mine (RDM) site is located approximately 250 miles west of Anchorage, Alaska. Located on the southwest bank of the Kuskokwim River, approximately 2 miles southeast of the village of Red Devil, the site is 8 miles northwest of the village of Sleetmute, and 75 miles northeast of Aniak, the largest village in the region. Fifteen villages are located downstream of Red Devil on the Kuskokwim River. The legal description for the RDM site is Township 19 North, Range 44 West, Southeast Quarter of Section 6, Sleetmute D-4 Quadrangle, Seward Meridian. The RDM site's approximate coordinates are 61° 45' 38.1" north latitude and 157° 18' 42.7" west longitude (North American Datum 1927). The RDM site is in a remote location, and access to the site is available by boat or barge on the Kuskokwim River or by means of an airstrip at the nearby village of Red Devil. An unimproved road leads from the airstrip through the village of Red Devil to the RDM site. Access to the RDM site is restricted by two locked gates, one on the unimproved road and a boat landing along the Kuskokwim River.

1.1 List of Field Activities

All field activities will be performed in accordance with the *Final Work Plan, Groundwater and Surface Water Baseline Monitoring, Red Devil Mine, Alaska* (*Work Plan*; BLM, 2019), and the addendum to the *Work Plan* (Sundance, 2023). Field activities will include tailgate safety meetings, the assessment of the operational status of the monitoring well network, synaptic measurement of groundwater elevations, downloading of transducer data, groundwater sampling, Red Devil Creek and seep discharge gauging, and surface water sampling.

2.0 Staff Organization, Qualifications, and Responsibilities

Personnel Name:	Role:	Contact:
Colleen Rust	Project Manager	(505) 321-4515
George Garner	Task Manager/Field Team Lead/Site Safety and Health Officer (SSHO)	(251) 605-1931
Nicholas Potter	Geologist	(262) 497-2422
Riley Wittler	Alternate Geologist	(505) 235-8685

Sundance personnel are certified with Occupational Safety and Health Administration (OSHA) 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) certification. Additionally, the SSHO holds an OSHA 30-Hour Construction Safety qualification.

3.0 Emergency Information

3.1 Standard Emergency Procedures

The following facilities and services will be utilized in case of an emergency. Minor incidents will be addressed on site by first aid trained personnel. For more serious medical emergencies requiring medical evacuation, LifeMED Alaska has been contracted to provide emergency medical evacuation to the Anchorage Regional Hospital.

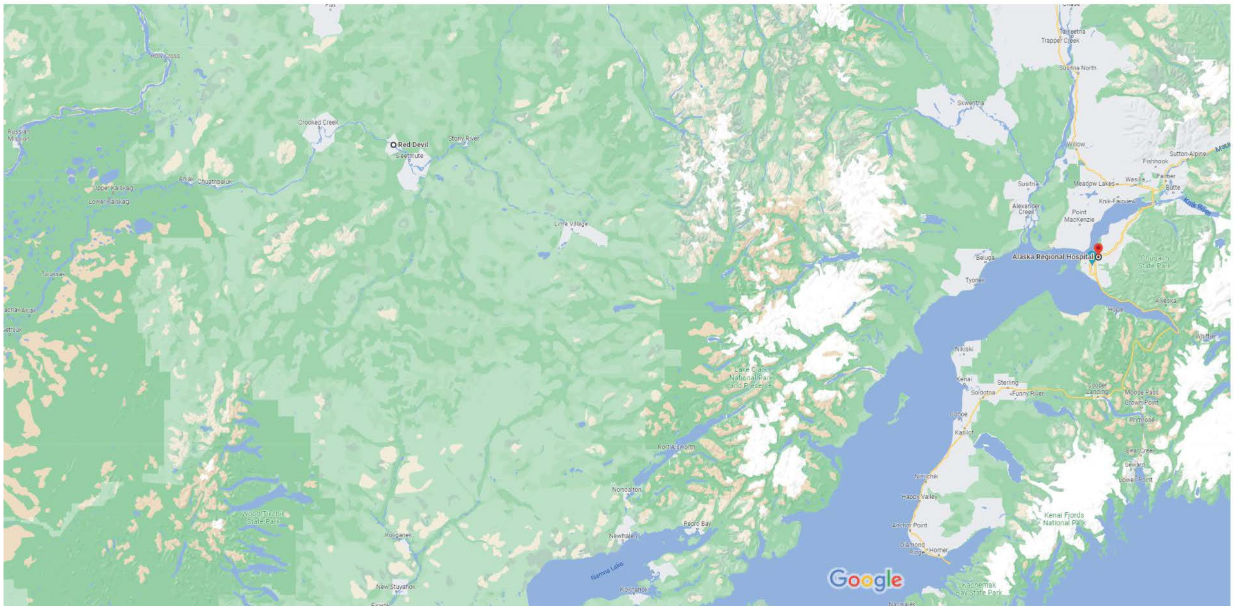
Hospital Name:	**Alaska Regional Hospital**
Hospital Address:	2801 Debarr Road, Anchorage, AK 99508
LifeMED Alaska	1-855-907-5433

All site emergencies will be reported immediately to the SSHO who will determine the best immediate course of action and reporting. If further evaluation is required, the Project Manager will be notified for direction and guidance.

All emergency communications will be conducted by direct vocal communication via satellite phone, InReach, two-way radios, or other acceptable means, as necessary.

3.2 Hospital Maps

Google Maps Red Devil, Alaska to Alaska Reg'l Hospital, 2801 Debarr Road, Anchorage, AK 99508



Note: Red Devil is not connected with the United States National Highway System.

In the event of a medical emergency, personnel must be airlifted from Red Devil to the Alaska Regional Hospital (Anchorage) or other medical facility. LifeMED Alaska has been contracted to provide any emergency medical evacuation flights.

4.0 Hazard/Risk Analysis

The following section evaluates the potential hazards that may be encountered during project field activities.

4.1 General Hazards

Hazards anticipated at the sites are listed below. Chemical and equipment hazards, considered to be more project-specific, are discussed below. Discussions of the more general hazards are presented in Attachment 1. Hazards will be discussed during safety and health meetings.

- Physical hazards
- Biological and Wildlife-Related Hazards
- Equipment hazards
- Chemical hazards
- Environmental hazards

4.2 Physical Hazards

Physical hazards will be present during all activities. The SSHO will screen the area for physical hazards prior to beginning work. Physical hazards include the following:

- Heat and cold stress
- Slips, trips, and falls
- Noise
- Handling heavy objects
- Fire protection
- Electrical hazards

4.2.1 Heat and Cold Stress

The following guidelines will be followed to prevent heat/cold-related injury:

- Drinking water
    - Potable drinking water will be always made available to employees, and employees are encouraged to consume water frequently.
- Employee work breaks
    - Employees will be encouraged to take breaks in a cooler or warmer location, depending on the needs of the day. A buddy system will be used to encourage fluid intake and to watch for symptoms of heat- or cold-related injuries.
- Solar radiation
    - If employees are exposed to solar radiation for short periods and there is the potential for sunburn, sunscreen and appropriate sun protection factor for their skin type will be applied.
- Wind Chill
    - If wind chill is a factor, the cooling effect of wind will be reduced by shielding the work area or requiring employees to wear an outer windbreak garment.
- Evaporative Liquids

  - Employees handling evaporative liquids at air temperatures below 40 degrees Fahrenheit will take precautions to avoid soaking of clothing or contact with skin.

4.2.2 Slips, Trips, and Falls

Slips, trips, and falls can be prevented easily by using common sense practices such as good housekeeping procedures, identifying tripping hazards and rectifying or avoiding them, and walking slowly with proper footwear on slippery surfaces. If a hazard cannot be immediately corrected, it will be cordoned off or marked (i.e., bright flagging tape) accordingly until it can be rectified.

4.2.3 Noise

Use of equipment (e.g., generators, all-terrain vehicles [ATVs], etc.) may expose the field team to noise levels that exceed the American Conference of Governmental Industrial Hygienists (ACGIH) threshold limit value (TLV) of 85 decibels average (dBA) for an 8-hour day. Exposure noise can result in:

- Temporary hearing losses where normal hearing returns after a rest period.
- Interference with speech communication and the perception of auditory signals.
- Interference with the performance of complicated tasks.
- Permanent hearing loss due to repeated exposure, resulting in nerve destruction in the hearing organ.

4.2.4 Handling Heavy Objects

Field personnel will observe proper lifting techniques and obey sensible lifting limits (50-pound maximum per person manual lifting unless reviewed by the SSHO to determine the lift is safe for the affected employee[s]).

4.2.5 Fire Protection

Fire safety is everyone’s responsibility on site. All employees should know how to prevent and respond to fires and are responsible for adhering to company policy regarding fire emergencies. Management will provide adequate controls to provide a safe workplace and will provide adequate resources and training to its employees to encourage fire prevention and the safest possible response in the event of a fire emergency.

4.2.6 Electrical Hazards

Field personnel should be aware of potential electrical shock dangers such as faulty field equipment. Field personnel will try to keep energized equipment as dry as possible by covering it with a tent, tarps, trash bags, or by other means.

Use of ground fault circuit interrupters (GFCI) is required for all project activities.

Per OSHA electrical regulations (29 Code of Federal Regulations [CFR] 1910.333), only qualified persons may work on energized electrical circuit parts or equipment or perform testing work on energized electrical circuits or equipment.

The OSHA standard further states that even qualified persons working near exposed energized electrical parts will not approach closer than 1-foot of a system that is over 300 volts (V) but not over 750 V. The standard does allow for closer approaches by qualified personnel if personnel are wearing insulated gloves with the proper voltage rating. Qualified personnel will wear rubber insulated gloves with a voltage rating of up to 1,000 V (Class O glove). Leather protector gloves will be worn over the rubber insulating gloves as needed.

4.3 Biological and Wildlife-Related Hazards

The SSHO will survey the work location(s) for biological hazards prior to beginning work. Care should be taken during field activities to prevent contact with biological hazards. Biological hazards may include insects, moose, or bears.

4.3.1 Insects

Mosquitoes, flies, and stinging insects present a potential hazard on this project. Protection against insects, such as protective clothing and insect repellents, will be used by field personnel.

4.3.2 Moose

Moose are common in the RDM area. Moose pose a risk in early spring due to calving season and during fall due to mating season. Mother moose can be very protective of calves especially if surprised by field personnel. Field personnel will be trained in the proper way to respond to aggressive moose.

4.3.3 Bears

Black bears and brown bears are common in the RDM area. Bears with cubs pose a significant risk to field personnel if encountered. Field personnel will be required to take bear awareness training prior to conducting field activities. Personnel will receive training in the use of bear spray.

4.4 Equipment Hazards

Following are a few common safety rules for field personnel:

1. ***Wear hearing protection when required***—If it has been determined that noise levels around the equipment could potentially cause hearing loss, always use protective plugs or muffs when working on or around the equipment.
2. ***Inspect and service motorized field equipment regularly***—Complete equipment service in accordance with the manufacturer’s recommendation. Qualified personnel will do periodic safety inspections on all components of the equipment regularly. Inspect the steering system and brake systems carefully. A pre-shift walk-around inspection will be performed by the operator.

Electrical-related repairs shall be limited to properly trained individuals. When performing field work with generators and electrical pumps, cords and electrical lines will be inspected for cracks and loose connections. The generator shall be properly secured for transport to avoid any unnecessary movement. During use, cords shall be kept away from standing water (if present) and protected from vehicular traffic.

When utilizing air compressors and blowers, the fluid lines in the remediation systems can sustain high pressures and temperatures. Care must be exercised when disconnecting fluid hoses to ensure that they are not under pressure or at high temperatures. Sufficient time must be allotted for lines to de-pressurize and cool prior to disconnection.

4.5 Chemical Hazards

There are several known chemical hazards at the RDM. These include petroleum products, arsenic, mercury, and other heavy metals.

4.5.1 Heavy metals

Heavy metal containing materials are found naturally in the rock of RDM site. Mercury and antimony ores are found in the tailing piles, in Red Devil Creek, and the creek delta. Additionally, waste rock present at the site contains elevated levels of arsenic, antimony, and mercury. While working around the waste rock staff should wear gloves, field staff should wear appropriate personal protective equipment (PPE), including nitrile gloves to prevent exposure. Additionally, field staff should wash their hands before eating and avoid exposure to the Red Devil Creek water and sediment. Masks should be worn if high levels of dust are present due to dry conditions. The overall exposure risk is low.

4.5.2 Hydrocarbons

Aromatic hydrocarbons (e.g., benzene) are locally present in certain areas on site due to historical fuel spills and during refueling equipment. The risk to exposure can be mitigated by avoiding the fuel spill area, using PPE when refueling, and washing hands regularly. Exposure to petroleum byproducts can lead to blood, liver, or nerve toxicity; decreased body weight; and liver or blood changes. However, human exposure to these chemicals during field activities is considered extremely low.

4.5.2.1 Skin Contact

Acute exposure to the anticipated on-site chemicals can produce skin irritation. Nitrile disposable gloves will be worn by personnel participating in sampling activities to protect against skin contact. Work gloves will be worn during manual material handling activities.

4.5.2.2 Eye Contact

In accordance with American National Standards Institute (ANSI) Z87.1, safety glasses will be worn for all site activities. A portable eyewash station will be present on site during all activities.

4.5.2.3 Inhalation

Precautions will be taken to prevent employee contact and exposure to dust and chemical inhalation. Dust masks will be deployed by field personnel if drought conditions exist on site.

4.5.2.4 Ingestion

All on-site personnel will wash their hands frequently. Food and drink will only be consumed once their hands are clean and in assigned areas that are safe for consumption.

4.5.3 Acids

Acids (e.g., HCl) are common preservatives found in sample bottles used for this project. Exposure to acids can cause chemical burns and serious injury. Exposure will be mitigated by wearing proper PPE (gloves) and practicing safe handling of the containers.

4.5.4 First Aid

In general, if direct skin contact with any of these chemicals occurs, immediately wash the contaminated skin with water. If these chemicals penetrate the clothing, immediately remove the clothing, and wash the skin with water. If symptoms occur after washing, get medical attention immediately.

When these chemicals contact the eyes, immediately wash the eyes with large amounts of water, occasionally lifting the lower and upper lids. Get medical attention immediately.

If a person breathes large amounts of these chemicals, move them to fresh air at once. Get medical attention as soon as possible and keep the affected person warm and at rest. Should the person lose consciousness, monitor their breathing and pulse, and if needed, assist breathing, or give cardiopulmonary resuscitation (CPR) until help arrives or until physical exhaustion. If these chemicals are swallowed, get medical attention immediately.

4.6 Environmental Hazard Monitoring

Should field personnel experience changes in health while conducting sampling (i.e., nausea, dizziness, or headache), these symptoms will be immediately reported to the SSHO, and field personnel will stand by until the situation is evaluated. The SSHO will report the incident to the Project Manager when communication becomes available.

4.6.1 Wildfires

Wildfires are a common occurrence in Alaska during the dry summer months. Wildfires may present hazards to field personnel. Wildfires have direct and indirect impacts on field personnel. Wildfires directly endanger the lives of staff and indirectly through negative impact on air quality.

4.6.2 Health Threat from Wildfire Smoke

Smoke from wildfires is a mixture of gases and fine particles from burning trees and other plant materials. Smoke can irritate the eyes and respiratory system and worsen chronic heart and lung diseases.

4.6.3 How to Tell If Smoke Is Affecting You

Smoke can cause coughing, scratchy throat, irritated sinuses, shortness of breath, chest pain, headaches, stinging eyes, and runny nose. Smoke may worsen symptoms for people who have pre-existing respiratory conditions, such as respiratory allergies, asthma, and chronic obstructive pulmonary disease, in the following ways:

- Inability to breathe normally
- Cough with or without mucus

- Chest discomfort
- Wheezing and shortness of breath.

When smoke levels are high enough, even healthy people may experience some of these symptoms.

4.6.4 Know Whether You Are at Risk

Those with a heart or lung disease (i.e., congestive heart failure, angina, chronic obstructive pulmonary disease, emphysema, or asthma) are at higher risk of having health problems than healthy people. Older adults are more likely to be affected by smoke, possibly because they are more likely to have heart or lung diseases than younger people.

4.6.5 Protect Yourself

Exposure to smoke should be limited. Following are some ways to limit such exposure:

- Pay attention to local air quality reports
- Listen and watch for news or health warnings about smoke
- Find out if the local community provides reports about the Environmental Protection Agency's Air Quality Index
- Pay attention to public health messages about taking additional safety measures
- Refer to visibility guides if they are available.

If advised to stay indoors, keep indoor air as clean as possible. Keep windows and doors closed unless it is extremely hot outside. Run an air conditioner if available but keep the fresh-air intake closed and the filter clean to prevent outdoor smoke from getting inside. If no air conditioner is available and if it is too warm to stay inside with the windows closed, seek shelter elsewhere.

Do not add to indoor pollution. When smoke levels are high, do not use anything that burns, i.e., candles, fireplaces, or gas stoves. Do not vacuum because vacuuming stirs up particles already inside a building. Do not smoke because smoking puts even more pollution into the air.

For those with asthma or another lung disease, a doctor's advice should be followed about medicines and a respiratory management plan. Dust masks are not enough. Paper "comfort" or "dust" masks commonly found at hardware stores are designed to trap large particles, i.e., sawdust. These masks will not protect the lungs from smoke.

4.6.6 Evacuation

If a forest fire is determined to endanger the lives of the field team, they will be evacuated from Red Devil as soon as possible. This decision to evacuate will be made by the Project Manager or the SSHO. If the Project Manager and the SSHO are unavailable, the responsibility for the decision to evacuate will be next most senior staff's responsibility.

5.0 Risk Mitigation

5.1 Medical and Environmental Hazard Monitoring

Prior to commencing field operations for each season, personnel involved with hazardous waste activities will be required to have completed their annual 8-Hour HAZWOPR refresher course and be cleared for work during annual medical monitoring physical.

5.2 Safety Equipment and Safe Work Practice

This section describes the safety equipment necessary and work practices that will be followed for the project. The SSHO will periodically evaluate site conditions and based on changes to the expected site conditions may recommend modifications to PPE requirements. The SSHO will coordinate all changes with the Project Manager.

5.2.1 Safety Equipment

The following safety equipment must be present at each site:

- First aid kit
- Eye wash bottle or eyewash kit
- Drinking water
- Personal location beacon (Garmin InReach or equivalent)
- Bear spray

5.3 Communication

The SSHO(s) will conduct daily tailgate meetings throughout the field portion of the project. Topics covered will include general site safety practices and safety concerns for anticipated field activities for the day. Additional meetings throughout the day will be held for unanticipated field activities. The SSHO(s) will review this HASP, current safety topics, and hazardous communications with any new onsite personnel (including facility staff). A cellular telephone or satellite phone will be available at the site for additional communication with the Project Manager.

5.3.1 General Safe Work Practices

The following safe work practices will be followed throughout the duration of the project:

- The buddy system must be used (at minimum two people working together/in communication with each other) when sampling in remote areas.
- Field personnel should avoid skin contact with permanone and N, N-diethyl-m-toluamide (DEET) bug repellant; it should be applied only on articles of clothing. These compounds may cause allergic and toxic effects with repeated use.
- Smoking, eating, drinking, and chewing either tobacco or gum is prohibited in work areas.
- Prior to initiating work at the site, the SSHO must identify the nearest sanitation facility for site personnel in each area.

- Site personnel should wash their hands and face upon leaving the site.
- Prescription drugs must not be taken unless specifically approved by a physician who understands the nature of the work exposure.
    - First aid treatment will be administered only by trained personnel.
- During hot or cold weather, regular rest breaks should be taken to avoid temperature-related stress. Non-alcoholic beverages, preferably water, should be consumed regularly to avoid dehydration.
- Prior to work start-up the SSHO must identify and demarcate work zones if necessary. Unauthorized persons will be excluded from the work area.
- Avoid skin contact with water when collecting samples.
- Be alert for hazards related to moving equipment and pressurized lines.

5.4 Work Practices

Work practice controls will be used when engineering controls are not feasible or are not sufficient to limit exposure to hazardous or toxic agents and environments within acceptable limits.

5.5 Personal Protective Equipment

Appropriate PPE and associated programs will be instituted when engineering or work practice controls are not feasible or are not sufficient to limit exposure to hazardous or toxic agents. Level D PPE is anticipated to be sufficient for the duration of all field activities. The following equipment will be considered mandatory:

- Gloves rated for full protection from the contaminants of concern (CoCs) when sampling.
- Safety glasses or other eye protection while driving ATVs and operating pressurized equipment.
- Orange traffic vest when working near traffic or heavy equipment operations.
- Hearing protection, either ear plugs or earmuffs, when working near heavy equipment, and other loud locations.
- Helmets while driving ATVs.
- Life jackets while operating boats.

5.5.1 Eye and Face Protection

Personnel will be provided with eye and face protection equipment when machines or operations present potential eye or face injury from physical, chemical, or radiological agents. Safety glasses will be always worn except while in vehicles with enclosed cabs or where additional eye protection is required. Eye and face protection equipment will meet the requirement of ANSI Z87.1 and bear a legible and permanent “Z87” logo to indicate compliance with the standard.

Operations that require the handling of harmful substances and operations where protection from gases, fumes, and liquids is necessary will require the wearing of goggles with cups of pliable rubber and suitable face shields, masks, or hoods that cover the head and neck and other protective clothing appropriate to the hazards involved.

5.5.2 Hearing Protection

Practical engineering or administrative controls will be used but are not limited to moving workers away from the noise source, restricting access to areas, rotating workers performing noisy tasks, and shutting down noisy equipment when not needed.

If hearing protection is required, the provided protection will be capable of reducing worker noise exposure below an 8-hour time-weighted average (TWA) of 85 dBA. If the provided hearing protection is not capable of achieving the required noise reduction level, additional engineering or administrative controls are necessary.

Whenever sound-pressure levels equal or exceed 85 dBA (measured as an 8-hour TWA), a continuing, effective hearing conservation program will be administered that is in accordance with 29 CFR 1910.95. When sound-pressure levels exceed 115 dBA, steady state, personal hearing protection equivalent to the combination of ear plugs and earmuffs will be required.

5.5.3 High-Visibility Apparel

High-visibility apparel will meet ANSI/International Safety Equipment Association (ISEA) 07-2004 Performance Class 2 requirements and will be used whenever working at RDM.

5.5.4 COVID-19 Precautions

Field team personnel will follow the most up to date Centers for Disease Control and Prevention (CDC) COVID-19 guidelines. General precautions the field team will follow include maintaining a minimum distance of 6 feet from other personnel unless otherwise necessary to perform job function. All personnel will abide by federal, state, and village mandates regarding the use of face coverings/masks. The field team will maintain diligent use of PPE, including safety glasses and nitrile gloves.

Staff will wash hands often with soap and water for at least 20 seconds especially if in a public place, or after blowing your nose, coughing, or sneezing. If soap and water are not readily available, use a hand sanitizer that contains at least 66% alcohol. Cover all surfaces of hands and rub them together until they feel dry.

The following questions will be asked of each employee and any potential site/office visitors to help identify people that should be excluded from the site. If an employee or subcontractor answers yes to any of the questions, they will be asked to leave the job site immediately and contact their supervisor and/or appropriate Human Resources Department for further instruction.

- Do you currently have a fever, chills, a cough, sore throat, or shortness of breath?
- Have you lost your sense of smell or taste?
- Have you been in contact with someone who has been medically diagnosed with COVID-19?

Staff will comply with all issued federal, state, and local requirements and mandates.

Attachment 1. BLM Risk Management Worksheets

Form 1112-5
(May 2001)

BLM

UNITED STATES
DEPARTMENT OF THE INTERIOR
BUREAU OF LAND MANAGEMENT
RISK MANAGEMENT WORKSHEET

1. Organization and Location BLM, Alaska State Office, Division of Renewable Resources (AK-931)			**2. Page** 1 of 8
3. Operation / Task Field Operations - Red Devil Mine, Alaska	**4. Beginning Date:** 1 May 2023	**5. Ending Date:** 31 October 2023	**6. Date Prepared** 13 March 2023
7. Prepared by (*Name / Duty Position*): Colleen Rust – Project Manger			

8. Identified Hazards: **PPE Required:** Field Going attire for operations in Border Regions of Southern Arizona	**9. Assess the Hazards Initial**				**10. Control Measures Developed for Identified Hazards: *(Specific measures taken to reduce the probability of a hazard.)***	**11. Assess the Hazard's Residual's Risk**				**12. How to Implement the Controls: *(Include SOP's, references,***	**13. Supervision and Evaluation Method: *(Continuous Leader Checks, Buddy System, etc.)***
(Be Specific)	**L**	**M**	**H**	**E**	**(Be Specific)**	**L**	**M**	**H**	**E**	**(Be Specific)**	**(Be Specific)**

All-terrain vehicle (ATV) - Collisions with other machines and/or stationary objects, loss of control/roll over		**X**			• All drivers must be trained to operate ATV prior to operation.
					• All riders will wear BLM prescribed riding PPE always when riding:
					  • Helmet
					  • Eye protection
					  • Long Sleeve and pants
					  • Gloves
					  • Boots.
					• Speeds will be kept to minimum levels (Not to exceed 25 Mph)
						X				Riders will complete a course in ATV operation.
										Required PPE will be provided on site
											FOR ALL: All the following will be used:
											• Continuous team checks.
											• Buddy system of constant observation and equipment checks.
											• Low speeds

14. Remaining Risk Level After Control Measures Are Implemented: (CIRCLE **HIGHEST** REMAINING RISK LEVEL)	**LOW** **(Line Supervisor)**	**MEDIUM** **(Branch Chief)**	**HIGH** **(District Manager)**	**EXTREMELY HIGH** **(Must be State Director/Associate)**

15. RISK DECISION AUTHORITY: (Approval/Authority Signature Block) (If Initial Risk Level is Medium, High, or Extremely High, Brief Risk Decision Authority at that level on Controls and Control Measures used to reduce risks. NOTE: if the person preparing the form signs this block, the signature indicates only that the appropriate risk decision authority was notified of the initial risk level, control measures taken, and appropriate resources requested; and that the risk was accepted by the decision authority.)

_______________ 13 March 2023
(Signature)

8. Identified Hazards:	**9. Assess the Hazards Initial**				**10. Control Measures Developed for Identified Hazards:** ***(Specific measures taken to reduce the probability of a hazard.)***	**11. Assess the Hazard's Residual's Risk**				**12. How to Implement the Controls:** ***(Include SOP's, references, etc.)***	**13. Supervision and Evaluation Method:** ***(Continuous Leader Checks, Buddy System, etc.)***
(Be Specific)	**L**	**M**	**H**	**E**	**(Be Specific)**	**L**	**M**	**H**	**E**	**(Be Specific)**	**(Be Specific)**
WORKING IN REMOTE AREAS Encounters with people conducting illegal activity in remote areas		**X**			1. Be especially alert in remote areas. The more remote the area, the more likely you are to find persons smuggling drugs on foot, on horseback, or in a vehicle. It is likely that you will encounter people entering the United States illegally. 2. Consult with BLM Law Enforcement Officers to see if the remote area you wish to travel/work in is now an area of concern. 3. Always know your location and be able to describe it. Let Dispatch know where you are going, when you expect to return, and check in at predetermined times. 4. Avoid working alone in remote areas. For safety, use the buddy system. 5. Expect surprise encounters in areas where your options for avoiding contact are limited. 6. Develop a plan for what you will do in the event of such an encounter. 7. Making noise as you would do in bear country is one way to avoid a surprise encounter by giving smugglers and migrants a chance to scatter before you come face to face. 8. Do not do anything to give the impression you are a Law Enforcement Officer or other person with the authority to interfere with illegal activity such as drug smuggling. Wear a subdued BLM uniform – one that does not give the impression of a law enforcement officer; but one that does give the impression that you work for the BLM.	**X**				Inform field team of the hazards that they may encounter when working in remote locations. Ensure the field team knows the proper check in /out procedures and how to use a satellite phone and two-way radio.	

Continued

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(Be Specific)	**L**	**M**	**H**	**E**	**(Be Specific)**	**L**	**M**	**H**	**E**	**(Be Specific)**	**(Be Specific)**
					9. Do not act in a threatening manner. 10. Leave the area as quickly as possible and avoid using your radio or cell phone until you are clear of the area and out of sight. 11. Always lock your vehicle when leaving it unattended and store valuable items, food, and water out of sight to avoid break ins. 12. Do not leave packs, equipment, food, and water unattended.						
HAZARDOUS MATERIALS - Exposure to various chemicals		**X**			1. Project areas are to be surveyed for the presence of hazardous materials prior to project implementation. However, other hazardous materials might still be found in the project areas. If suspicious materials are found, participants will NOT TOUCH, handle, or move the materials. 2. Note the location of the materials and, if possible, flag the location. 3. Notify other workers to avoid the materials and inform crew boss or project leader. If it is not certain whether a material is hazardous, consider it hazardous. 4. Examples of materials that should be considered hazardous include: • Unidentifiable liquids and powders • Materials with strong petroleum or unwholesome (chemical) odors including motor oils and coolants • Needles, syringes, or other identifiable medical wastes	**X**				Inform field team of what to do if they encounter hazardous materials.	

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(Be Specific)	**L**	**M**	**H**	**E**	**(Be Specific)**	**L**	**M**	**H**	**E**	**(Be Specific)**	**(Be Specific)**
					• Pesticide, chemical, and solvent containers • Drums and five-gallon buckets (do not open, move, bang on, or stick your head in any drum) • Asbestos, including insulation and ceiling and floor tiles • Automobile and motorcycle						
HUNTER ENCOUNTERS - hunters not being aware of field team activities in the area. This could result in the hunters' inattention to their surroundings. An employee may become a victim.		**X**			1. Field team should be made aware of current hunting seasons. 2. Field team should wear proper clothing (blaze orange if needed). 3. Field team should have an awareness of proper hunting etiquette. 3. Alert hunters to nature of project and presence of employees in area. 4. Reschedule activity if necessary.	**X**				Inform field team about BLM's hunting regulations. Inform field team about the hazards they might encounter during hunting season.	
WILDLIFE ENCOUNTERS – Field team becoming bitten or stung or attacked causing injury and illness		**X**			1. Use insect repellent when needed. 2. Carry epi pen if prescribed by a medical provider and prompt medical attention if necessary. 3. Bears and other wildlife are attracted to the trash. Avoid any wildlife encounters. 4. All persons must be aware of bear/wildlife interactions and how to handle the situation 5. Be aware of surroundings and use buddy system • Use of whistle or other noise device to deter an attack • Use of bear spray to deter attack	**X**				Inform field team of hazards. Ensure employees are trained in 1st Aid/CPR/AED. Attend bear and wildlife awareness training. Bear Guard must be trained on use of bear non-lethal deterrence	Buddy system of constant observation

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(Be Specific)	**L**	**M**	**H**	**E**	**(Be Specific)**	**L**	**M**	**H**	**E**	**(Be Specific)**	**(Be Specific)**
HEAT/COLD STRESS – serious medical emergencies can occur due to these conditions.		**X**			1. Maintain adequate body fluids; always pack extra food/water. 2. Be aware of weather. 3. Use appropriate sunscreen and clothing. 4. Take frequent breaks. 5. Avoid fatigue. 6. Know signs and symptoms of heat stress and cold stress.	**X**					
Hazardous Weather Changes		**X**			All persons will have on them at all times: • Equipment Clothing for hot weather such as a hat, long sleeves, sunscreen, extra water • Equipment Clothing for cold weather such as heavy windproof jacket, insulated gloves, rain pants, stocking cap, extra socks • Survival equipment will include space blankets and fire starter material	**X**				All persons will be have basic wilderness survival training and will carry with them equipment required for conditions.	Continuous team checks. Buddy system of constant observation and equipment checks.

Continued

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(Be Specific)	**L**	**M**	**H**	**E**	**(Be Specific)**	**L**	**M**	**H**	**E**	**(Be Specific)**	**(Be Specific)**
COVID-19-related hazards Charter Flight Field Work Common/living area camp		**X**			1) Less than 3 passengers plus Pilot in Aircraft. Masks worn during flight. 2) Outdoor work: socially distance, wear masks, use hand sanitizer, avoid touching face 3) Each project member will maintain their own area as much as possible – reduce number and use of common areas, wear masks when needed, hand sanitizer, use disinfecting wipes, social distance 4) Follow all agency, federal, state, local, and airline/airport safety guidelines 5) COVID vaccinations	**X**				Check in with project coordinator regularly. Research agency, federal, state, local, and flight/airport safety guidelines immediately prior to departure. Follow pilot's aircraft requirements. Daily project safety Briefings, reminders for mask use and social distancing Continual safety check-ins and vigilance during field work.	Check in with supervisor at the end of each workday. Self-evaluation and identification of improvement for next session.

Continued

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(Be Specific)	**L**	**M**	**H**	**E**	**(Be Specific)**	**L**	**M**	**H**	**E**	**(Be Specific)**	**(Be Specific)**
Mine Site Hazards Open pits, shafts, holes Overhead/hanging objects Oil/Fuel and HAZMAT storage Old and deteriorated structures Non-operable equipment		X			1) Use situation awareness around the mine site, especially near equipment and processing plants 2) avoid entering unsafe structures 3) Observe and record/annotate hazmat or fuel issues for notification to appropriate staff	X				Follow hazards awareness training within policy.	Continuous team checks. Buddy system of constant observation and equipment checks.

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(Be Specific)	**L**	**M**	**H**	**E**	**(Be Specific)**	**L**	**M**	**H**	**E**	**(Be Specific)**	**(Be Specific)**
Boat Operations Slips, trips, and falls, including falls overboard) Drowning Boat crash or collision		**X**			1) Monitor radio, inReach, and/or satellite phone for up-to-date severe weather forecasts. 2) Boat operators will be trained by the site supervisor and/or the senior boat operator. 3)Discontinue work during thunderstorms and severe weather events. 2) Boat operation will be avoided when the river conditions are unfavorable or deemed unsafe by the operator 4) Boat operator will inspect the boat prior to operation. The operator will ensure the number of personal flotation devices is equal to or greater than the number of passengers onboard. 3) The will boat will be packed in a stable way 5) No personnel will embark or disembark the vessel without the direction of the vessel operator. The vessel operator will ensure passengers are wearing personal flotation devices while on deck. At the request of the operator, personnel will be seated. 6) Passengers will stay seated until the boat is docked. Ensure three-point contact whenever possible or practical. 7) A Type IV throwable device will be readily available onboard.	**X**				Have only competent staff act as the boat operator Have a safety moment before operating a boat to check weather and river conditions Inform the field team of hazards. Ensure employees are trained in 1st Aid/CPR/AED. Required will be provided on site.	Continuous team checks. Buddy system of constant observation and equipment checks.