Pace Analytical®
National Center for Testing & Innovation

STANDARD OPERATING PROCEDURE
TITLE: Volatile Organic Compounds by GC/MS
(EPA 8260B, 8260C, 8260D, 624, 624.1, and SM6200 B)
ISSUER: Pace National – Mt. Juliet, Tennessee

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
460-00-4	4-Bromofluorobenzene	9971	99.7	4.9	85	114
106-43-4	4-Chlorotoluene	23616	99.9	7.4	78	122
108-10-1	4-Methyl-2-pentanone [MIBK]	25796	98.5	10.6	67	130
67-64-1	Acetone	25006	99.5	20.1	39	160
75-05-8	Acetonitrile	13308	95.8	15.2	50	142
106-99-0	1,3-Butadiene	1202	100.6	19.2	43	158
541-73-1	1,3-Dichlorobenzene	26951	99.7	6.5	80	119
142-28-9	1,3-Dichloropropane	23811	99.1	6.5	80	119
542-75-6	1,3-Dichloropropene	9784	99.9	7.6	77	123
106-46-7	1,4-Dichlorobenzene	27715	98.3	6.5	79	118
105-05-5	1,4-Diethylbenzene	1980	98.4	6.4	79	118
123-91-1	1,4-Dioxane	17866	99	13.4	59	139
544-10-5	1-Chlorohexane	5790	99.6	8	76	124
540-84-1	2,2,4-Trimethylpentane [Isooctane]	5432	95.2	12.3	58	132
594-20-7	2,2-Dichloropropane	23775	99.7	13.2	60	139
75-85-4	2-Butanol	4332	92.7	9.1	66	120
78-93-3	2-Butanone [MEK]	26659	99.6	14.6	56	143
126-99-8	2-Chloro-1,3-butadiene	15673	100	11.7	65	135
110-75-8	2-Chloroethyl vinyl ether	18225	94.7	14.7	51	139
95-49-8	2-Chlorotoluene	23750	100	7.2	79	122
591-78-6	2-Hexanone	25368	97.9	13.5	57	139
91-57-6	2-Methylnaphthalene	3754	79.4	20.9	17	142
79-46-9	2-Nitropropane	10213	92.6	14.5	49	136
67-63-0	2-Propanol [Isopropyl alcohol]	2034	98.8	14.4	56	142
624-95-3	3,3-Dimethyl-1-butanol	6491	90.9	13.9	49	133
460-00-4	4-Bromofluorobenzene	9971	99.7	4.9	85	114
106-43-4	4-Chlorotoluene	23616	99.9	7.4	78	122
108-10-1	4-Methyl-2-pentanone [MIBK]	25796	98.5	10.6	67	130
67-64-1	Acetone	25006	99.5	20.1	39	160
75-05-8	Acetonitrile	13308	95.8	15.2	50	142
107-02-8	Acrolein [Propenal]	16380	96.8	19.3	39	155
107-13-1	Acrylonitrile	20173	99	11.9	63	135
107-05-1	Allyl chloride	15758	99	10.4	68	130
71-43-2	Benzene	34376	99.4	6.9	79	120
100-44-7	Benzyl chloride	10675	90.1	15.9	42	138
108-86-1	Bromobenzene	23762	99.7	6.7	80	120
74-97-5	Bromochloromethane	24356	100.8	7.5	78	123
75-27-4	Bromodichloromethane	26888	101.8	7.8	79	125
75-25-2	Bromoform	27675	97.8	10.8	66	130
74-83-9	Bromomethane	26717	97	14.7	53	141
75-15-0	Carbon disulfide	25719	98.8	11.5	64	133
56-23-5	Carbon tetrachloride	28870	103.8	10.7	72	136
108-90-7	Chlorobenzene	29802	100	6.1	82	118
124-48-1	Chlorodibromomethane	27424	100	8.5	74	126
75-45-6	Chlorodifluoromethane	7197	84.4	14.9	40	129

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
75-00-3	Chloroethane	27069	99	13	60	138
67-66-3	Chloroform	29373	101.1	7.5	79	124
74-87-3	Chloromethane	27697	94.5	15	50	139
156-59-2	cis-1,2-Dichloroethene	27935	100.1	7.5	78	123
10061-01-5	cis-1,3-Dichloropropene	27197	99.5	8	75	124
1476-11-5	cis-1,4-Dichloro-2-butene	1524	101.5	14.9	57	146
110-82-7	Cyclohexane	20438	100.4	10	71	130
1868-53-7	Dibromofluoromethane	5702	99.1	6.5	80	119
74-95-3	Dibromomethane	24473	101.1	7.3	79	123
75-71-8	Dichlorodifluoromethane [Freon-12]	25410	92	20.1	32	152
107-02-8	Acrolein [Propenal]	16380	96.8	19.3	39	155
107-13-1	Acrylonitrile	20173	99	11.9	63	135
107-05-1	Allyl chloride	15758	99	10.4	68	130
71-43-2	Benzene	34376	99.4	6.9	79	120
100-44-7	Benzyl chloride	10675	90.1	15.9	42	138
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75-00-3	Chloroethane	27069	99	13	60	138
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74-87-3	Chloromethane	27697	94.5	15	50	139
156-59-2	cis-1,2-Dichloroethene	27935	100.1	7.5	78	123
10061-01-5	cis-1,3-Dichloropropene	27197	99.5	8	75	124
1476-11-5	cis-1,4-Dichloro-2-butene	1524	101.5	14.9	57	146
110-82-7	Cyclohexane	20438	100.4	10	71	130
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74-95-3	Dibromomethane	24473	101.1	7.3	79	123
75-71-8	Dichlorodifluoromethane [Freon-12]	25410	92	20.1	32	152
75-43-4	Dichlorofluoromethane	1504	101.5	9.8	72	131
60-29-7	Diethyl ether	17189	98.6	10.2	68	129
108-20-3	Diisopropyl ether	22989	97.5	10.3	67	128
64-17-5	Ethanol	9543	99.2	17.1	48	151
141-78-6	Ethyl acetate	9208	96.8	13.9	55	138
97-63-2	Ethyl methacrylate	16674	98.7	9	72	126
637-92-3	Ethyl tert-butyl ether	19841	98.3	9.4	70	127
100-41-4	Ethylbenzene	33325	99.8	7	79	121
462-06-6	Fluorobenzene	1373	97.9	6.1	80	116

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
142-82-5	Heptane	11878	94.4	15	49	140
87-68-3	Hexachlorobutadiene	23535	100.1	11.3	66	134
67-72-1	Hexachloroethane	8718	102.9	10.3	72	134
110-54-3	Hexane	15545	95.5	15.9	48	143
74-88-4	Iodomethane	20229	100	10.4	69	131
78-83-1	Isobutyl alcohol	14123	97.7	11.7	63	133
108-21-4	Isopropyl acetate [Acetic acid]	7216	97.8	11.6	63	133
98-82-8	Isopropylbenzene	28636	101.5	9.9	72	131
179601-23-1	m/p-Xylene [3/4-Xylene]	28168	100.5	6.9	80	121
126-98-7	Methacrylonitrile	15982	97.9	11.6	63	133
79-20-9	Methyl acetate	19698	96	13.2	56	136
80-62-6	Methyl methacrylate	16524	97.7	10.2	67	128
1634-04-4	Methyl tert-butyl ether [MTBE]	29660	97.3	8.8	71	124
108-87-2	Methylcyclohexane	20025	101.8	10.1	72	132
75-09-2	Methylene chloride	27659	99.4	8.3	74	124
123-86-4	n-Butyl acetate	7247	96.8	9.4	69	125
75-43-4	Dichlorofluoromethane	1504	101.5	9.8	72	131
60-29-7	Diethyl ether	17189	98.6	10.2	68	129
108-20-3	Diisopropyl ether	22989	97.5	10.3	67	128
64-17-5	Ethanol	9543	99.2	17.1	48	151
141-78-6	Ethyl acetate	9208	96.8	13.9	55	138
97-63-2	Ethyl methacrylate	16674	98.7	9	72	126
637-92-3	Ethyl tert-butyl ether	19841	98.3	9.4	70	127
100-41-4	Ethylbenzene	33325	99.8	7	79	121
462-06-6	Fluorobenzene	1373	97.9	6.1	80	116
142-82-5	Heptane	11878	94.4	15	49	140
87-68-3	Hexachlorobutadiene	23535	100.1	11.3	66	134
67-72-1	Hexachloroethane	8718	102.9	10.3	72	134
110-54-3	Hexane	15545	95.5	15.9	48	143
74-88-4	Iodomethane	20229	100	10.4	69	131
78-83-1	Isobutyl alcohol	14123	97.7	11.7	63	133
108-21-4	Isopropyl acetate [Acetic acid]	7216	97.8	11.6	63	133
98-82-8	Isopropylbenzene	28636	101.5	9.9	72	131
179601-23-1	m/p-Xylene [3/4-Xylene]	28168	100.5	6.9	80	121
126-98-7	Methacrylonitrile	15982	97.9	11.6	63	133
79-20-9	Methyl acetate	19698	96	13.2	56	136
80-62-6	Methyl methacrylate	16524	97.7	10.2	67	128
1634-04-4	Methyl tert-butyl ether [MTBE]	29660	97.3	8.8	71	124
108-87-2	Methylcyclohexane	20025	101.8	10.1	72	132
75-09-2	Methylene chloride	27659	99.4	8.3	74	124
123-86-4	n-Butyl acetate	7247	96.8	9.4	69	125
71-36-3	n-Butyl alcohol	10122	95.1	12	59	131
104-51-8	n-Butylbenzene	24088	101.1	8.8	75	128
109-60-4	n-Propyl acetate	602	100.8	8.3	76	126
103-65-1	n-Propylbenzene	24419	101	8.5	76	126
91-20-3	Naphthalene	27847	94.6	11.3	61	128

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
95-47-6	o-Xylene	31776	100	7.2	78	122
99-87-6	p-Isopropyltoluene [p-Cymene]	24335	102	8.5	77	127
76-01-7	Pentachloroethane	11688	101.1	10.7	69	133
109-66-0	Pentane	3915	74.8	19.7	16	134
107-12-0	Propionitrile [Ethyl cyanide]	15701	99.9	12	64	136
135-98-8	sec-Butylbenzene	24191	101.1	8.1	77	126
100-42-5	Styrene	26985	100.5	7.6	78	123
994-05-8	tert-Amyl methyl ether [TAME]	19726	98.1	10.1	68	128
75-65-0	tert-Butyl alcohol	21112	98.6	10.1	68	129
762-75-4	tert-Butyl formate	6651	98.1	11.1	65	132
98-06-6	tert-Butylbenzene	23919	101	7.7	78	124
127-18-4	Tetrachloroethene	29017	101.3	9.3	74	129
109-99-9	Tetrahydrofuran	18021	95	12.8	57	133
108-88-3	Toluene	33510	100.1	6.8	80	121
2037-26-5	Toluene-d8	9809	100.4	3.8	89	112
156-60-5	trans-1,2-Dichloroethene	27663	99.5	8.2	75	124
10061-02-6	trans-1,3-Dichloropropene	27134	100	8.9	73	127
110-57-6	trans-1,4-Dichloro-2-butene	19320	91.5	16.1	43	140
79-01-6	Trichloroethene	30150	101.1	7.3	79	123
75-69-4	Trichlorofluoromethane	26108	103	12.8	65	141
71-36-3	n-Butyl alcohol	10122	95.1	12	59	131
104-51-8	n-Butylbenzene	24088	101.1	8.8	75	128
109-60-4	n-Propyl acetate	602	100.8	8.3	76	126
103-65-1	n-Propylbenzene	24419	101	8.5	76	126
91-20-3	Naphthalene	27847	94.6	11.3	61	128
95-47-6	o-Xylene	31776	100	7.2	78	122
99-87-6	p-Isopropyltoluene [p-Cymene]	24335	102	8.5	77	127
76-01-7	Pentachloroethane	11688	101.1	10.7	69	133
109-66-0	Pentane	3915	74.8	19.7	16	134
107-12-0	Propionitrile [Ethyl cyanide]	15701	99.9	12	64	136
135-98-8	sec-Butylbenzene	24191	101.1	8.1	77	126
100-42-5	Styrene	26985	100.5	7.6	78	123
994-05-8	tert-Amyl methyl ether [TAME]	19726	98.1	10.1	68	128
75-65-0	tert-Butyl alcohol	21112	98.6	10.1	68	129
762-75-4	tert-Butyl formate	6651	98.1	11.1	65	132
98-06-6	tert-Butylbenzene	23919	101	7.7	78	124
127-18-4	Tetrachloroethene	29017	101.3	9.3	74	129
109-99-9	Tetrahydrofuran	18021	95	12.8	57	133
108-88-3	Toluene	33510	100.1	6.8	80	121
2037-26-5	Toluene-d8	9809	100.4	3.8	89	112
156-60-5	trans-1,2-Dichloroethene	27663	99.5	8.2	75	124
10061-02-6	trans-1,3-Dichloropropene	27134	100	8.9	73	127

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Table 4. LCS Control Limits – Method 8260 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
110-57-6	trans-1,4-Dichloro-2-butene	19320	91.5	16.1	43	140
79-01-6	Trichloroethene	30150	101.1	7.3	79	123
75-69-4	Trichlorofluoromethane [Freon-11]	26108	103	12.8	65	141
108-05-4	Vinyl acetate	18941	100.2	15.3	54	146
75-01-4	Vinyl chloride	29472	97.4	13.2	58	137
1330-20-7	Xylenes [total]	23426	100.1	7	79	121

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB from method.	Retune instrument and verify.	Flagging is not appropriate.	No samples shall be analyzed without a valid tune.
Initial calibration (ICAL) for all analytes (including surrogates)	At instrument set-up, prior to sample analysis	Each analyte must meet one of the three options below: **Option 1:** RSD for each analyte ≤ 15%; **Option 2:** linear least squares regression for each analyte: $r^2 \geq 0.99$; **Option 3**: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. No samples shall be analyzed until ICAL has passed. If the specific version of a method requires additional evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL s performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and surrogate.
Evaluation of Relative Retention Times (RRT)	With each sample.	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	NA	After maintenance is performed which may affect retention times, RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat the CCV and all associated samples since the last successful CCV. Alternately, Recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.
Internal standards (IS)	Every field sample, standard and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within - 50% to +100% of ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, data must be qualified and explained in the case narrative. Apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards.	

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the limits in Tables 3 and 4 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Must contain all surrogates and all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use the limits in Tables 3 and 4 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	Must contain all surrogates and all analytes to be reported. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.

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Table 5. Quality Control Requirements – Organic Analysis by Gas Chromatography/Mass Spectrometry

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use the limits in Tables 3 and 4 for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	MSD: Must contain all surrogates and all analytes to be reported. The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use limits in Tables 3 and 4 or in-house LCS limits (see the LIMS) if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the case narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

1.0 SCOPE AND APPLICATION

NOTE: For clients, whose environment laboratory quality program is administered by Environmental Standards Inc. (ESI), see controlled document QUA-25 AIR. \\FAP\NovDishH\QAQC\Controlled Docs.

This standard operating procedure applies to the analysis of methane, ethane, ethene, propane, acetylene, butane, and isobutane in ground water samples and air samples from various sources, including fuel and chlorinated solvent spills.

In water matrix, headspace is generated in each sample using ultra-pure helium. A portion of the headspace created is injected into a gas chromatograph (GC) and the methane, ethane, ethene, propane, and acetylene concentrations are determined by using a flame ionization detector (FID) and integrator. Air samples are direct injected into the GC inlet.

The sensitivity limit for a compound is defined as the minimum detectable concentration of that compound, or the concentration that produces a signal-to-noise ratio of 3 to 1. For methane, ethane, ethene, propane, acetylene, butane, and isobutane, the sensitivity limit is in the mid ppbv range.

Method reporting limits:

Compound	RL* ppbv (water)	RL of Low Level* ppbv (water)	RL* ppmv (air)
Methane	10	0.678	10
Ethane	13	1.29	10
Ethene	13	1.27	10
Propane	18.6	n/a	10
Acetylene	20.8	n/a	10
Butane	1	.0024	1
IsoButane	1	.0024	1

*See Appendix A.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes that can be determined by this SOP and the associated LOQ is provided in Table 1, Appendix A.

2.0 SUMMARY OF METHOD

A portion of the sample is injected into a gas chromatograph (GC) and the methane, ethane ethene propane, acetylene, butane and isobutane concentrations are determined by using a Flame Ionization Detector (FID) and integrator. Calibration for this method is accomplished using external calibration.

3.0 INTERFERENCES

Since the FID exhibits universal response and detects most gas components except the carrier, interferences may occur. Choosing the appropriate GC retention times by changing the column flow rate may help to eliminate resolution interferences.

High purity reagents must be used to minimize interference problems.

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If the retention time of the methane peak is off by 0.02 or has a different peak shape than the low point of the calibration it is deleted to prevent reporting air/moisture contamination as methane.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

Contact your supervisor or local safety coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure

The following sections provide general health and safety information about chemicals and materials that may be present in the laboratory.

- The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.
- The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (EHS) policies and procedures specified in this SOP and in the Pace® Chemical Hygiene / Safety Manual (COR-MAN-0001)
- Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.
- Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. For procedures that require use of acids, use acids in a fume hood whenever possible with PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. For procedures that that emit large volumes of solvents (evaporation/concentration processes), these activities must be performed in a fume hood or apparatus that reduces exposure.

6.0 .SAMPLE COLLECTION, PRESERVATION, HOLDING TIME & STORAGE

The laboratory provides containers for the collection of samples upon client request. Refer to laboratory SOP ENV-SOP-MTJL-0064 *Sample Shipping* for procedures related to preparation of bottle kits for the test method(s) associated with this SOP.

The laboratory does not perform sample collection or field measurements for this test method. Samples should be collected in accordance with a sampling plan and sampling procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

Container Type, Minimum Sample Amount, Preservation, and Holding Time Requirements:

Matrix	Container Size & Type	Required Sample Amount[1]	Preservation	Holding Time
Water	40mL amber vials	Filled completely with zero headspace or bubbles.	Thermal: -<6°C Chemical: <2 pH with HCl	Collection to Prep: N/A Prep to Analysis: 14 days
Air	ALTEF Tedlar bags or summa canisters	N/A	Thermal: ambient temperature Chemical: N/A	Collection to Prep: N/A Prep to Analysis: 14 days

[1] *Amount of sample required for each discrete test.*

Thermal preservation is checked and recorded on receipt in accordance with laboratory SOP ENV-SOP-MTJL-0060 *Sample Receiving.* Chemical preservation is checked and recorded at time of receipt or prior to sample preparation. Samples that do not have a pH<2 are qualified with a G1 if analyzed past 7 days of collection.

In some cases, exceeding the appropriate preservation and storage criteria can cause significant bias in the resulting data. Clients may need to resubmit samples where the conditions during shipment cause uncertainty regarding sample integrity. If samples do not meet the requirements for preservation, sampling, shipment and storage and the client approves the completion of the analytical process, sample results can be qualified per the ENV-SOP-MTJL-0014, *Data Handling and Reporting.*

After analysis, samples are retained as stated in the Pace® standard terms and conditions, unless otherwise specified in the analytical services contract. Samples are then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT & SUPPLIES

7.1 Equipment

- Gas chromatograph. GC having at least the following components:
  - Separation column. A 30m X 0.320mm CS-Gaspro column made by J&W scientific or equivalent. Part #113-4332.
  - Direct injection inlet
  - Flame ionization detector
  - Headspace autosampler, EST analytical LGX50, or equivalent
- Integration system- PC controlled software Chemstation HPCHEM
- Regulators- to control gas cylinder pressures and flow rates
- Water bath or incubator capable of 25°C

7.2 Supplies

- 10uL, 25uL, 100uL, 50cc, 100cc, and 100mL gas tight syringes

8.0 REAGENTS & STANDARDS

All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standard Logger–Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. For Air standards, see CoA for expiration dates provided by the vendor. All intermediate spiking solutions and surrogate standard solutions should be replaced at least every 1 year or sooner if a problem is detected unless otherwise noted.

8.1 Reagents

- Carrier gas is helium, from Volunteer Welding – UHP UN1002
- High-purity- FID gases are ultra-high purity grade air and hydrogen, from Volunteer Welding – UHP 1046.
- Calibration verification gas. Standard cylinder gas mixtures for each compound of interest; prepared in nitrogen. 0.01% (100ppm) custom mix from Linde, or equivalent.
- Secondary Source Calibration Verification. Standard cylinder gas mixtures for each compound of interest; prepared in nitrogen. 0.1% (1000ppm) custom mix from Matheson, or equivalent.
- Standards are transferred directly to tedlar bags which are used for ICALs and calibration verification.

8.2 Standards

Not applicable.

8.3 Formulations

Not applicable.

9.0 PROCEDURE

9.1 Equipment Preparation

9.1.1 Support Equipment

Below are the typical conditions for instrument and column:

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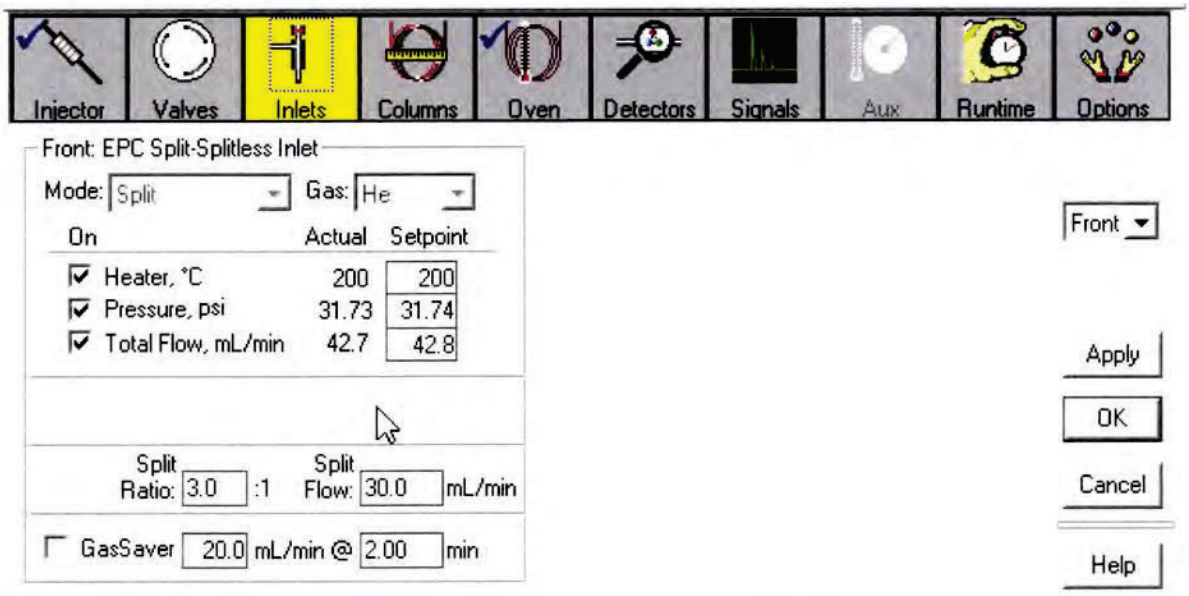
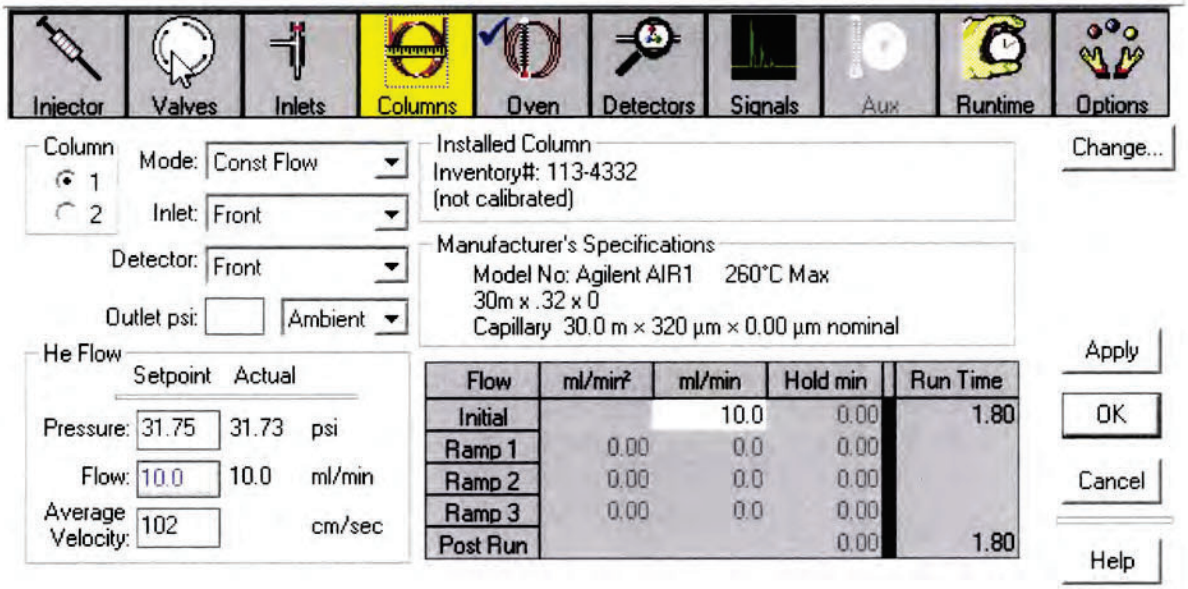
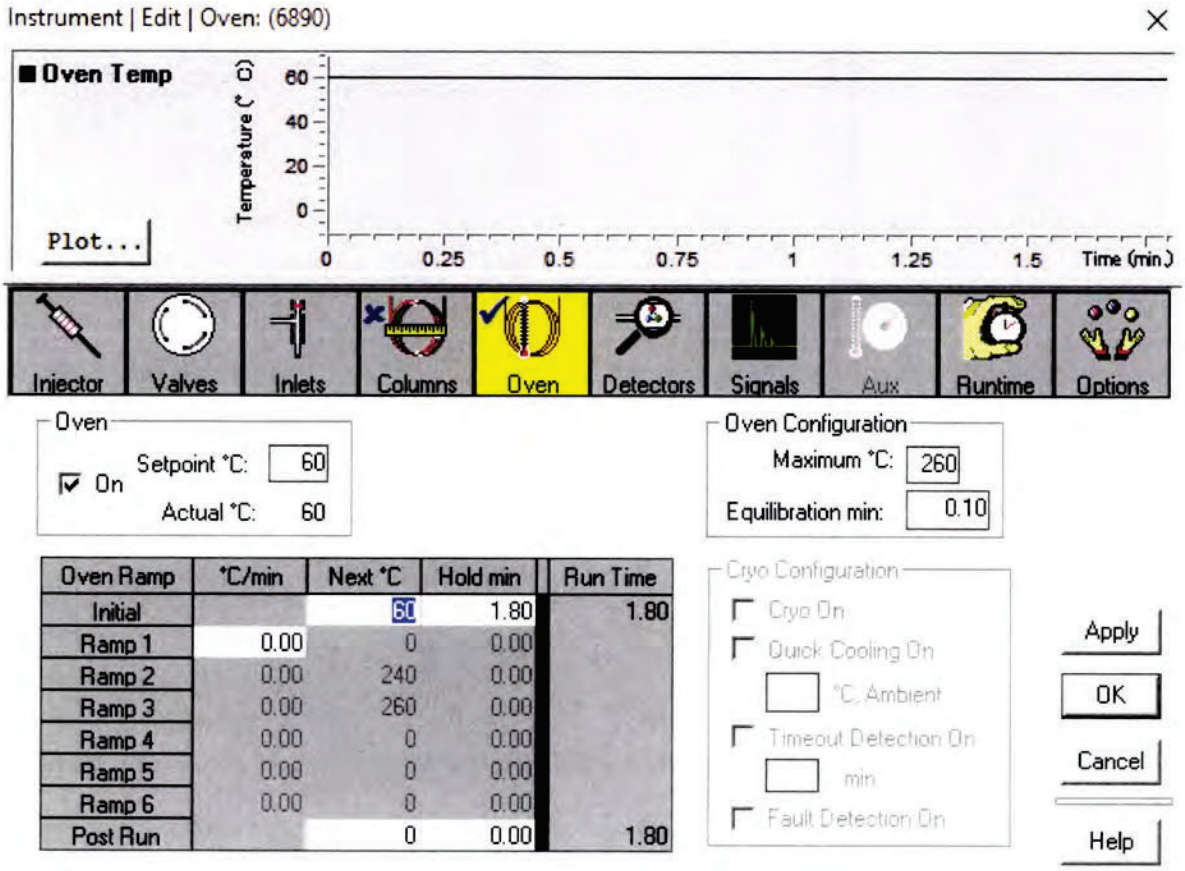
9.1.2 Instrument Set Up

9.1.2.1 Routine Instrument Operating Conditions







9.2 Calibration

9.2.1 Calibration Frequency

As needed.

9.2.2 Calibration Levels

RSK Water Matrix (Headspace Analyzer Calibration)						
Target Parameters	STD 1 Stock Amount Used - 0.02mL	STD 2 Stock Amount Used - 0.2mL	STD 3 Stock Amount Used - 2.0mL	STD 4 Stock Amount Used - 10mL	STD 5 Stock Amount Used - 20mL	STD 6 Stock Amount Used - 200mL
Methane (in ppm)	0.000678	0.00678	0.0678	0.339	0.678	6.78
Ethane (in ppm)	0.00129	0.0129	0.129	0.645	1.29	10.29
Ethene (in ppm)	0.00127	0.0127	0.127	0.634	1.27	10.27
Propane (in ppm)	n/a	0.0186	0.186	0.93	1.86	10.86
Acetylene (in ppm)	n/a	0.0208	0.208	1.04	2.08	20.8

RSK water matrix (manual calibration)

0.01% or 100ppm std for additional compounds

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Target Parm	Std amt 5uL	Std amt 10uL	Std amt 25uL	Std amt 50uL	Std amt 100uL
Butane	0.0122	0.0243	0.0608	0.122	0.243
Isobutane	0.0121	0.0242	0.0605	0.121	0.242

RSK Water Matrix (Manual Calibration)						
Target Parameters	STD 1 Stock Amount Used - 0.1µL	STD 2 Stock Amount Used - 1.0µL	STD 3 Stock Amount Used - 10.0µL	STD 4 Stock Amount Used - 50.0µL	STD 5 Stock Amount Used - 100.0µL	STD 6 Stock Amount Used - 1.0mL
Methane (in ppm)	0.000678	0.00678	0.0678	0.339	0.678	6.78
Ethane (in ppm)	0.00129	0.0129	0.129	0.645	1.29	10.29
Ethene (in ppm)	0.00127	0.0127	0.127	0.634	1.27	10.27
Propane (in ppm)	n/a	0.0186	0.186	0.93	1.86	10.86
Acetylene (in ppm)	n/a	0.0208	0.208	1.04	2.08	20.8

EEM Air Matrix (Manual Calibration)							
Target Parameters	STD 1 Stock Amount Used - 0.1µL	STD 2 Stock Amount Used - 1.0µL	STD 3 Stock Amount Used - 10.0µL	STD 4 Stock Amount Used - 50.0µL	STD 5 Stock Amount Used - 100.0µL	STD 6 Stock Amount Used - 5.0mL	STD 7 Stock Amount Used - 10.0mL
Methane (in ppmv)	1	10	100	500	1000	5000	10000
Ethane (in ppmv)	1	10	100	500	1000	5000	10000
Ethene (in ppmv)	1	10	100	500	1000	5000	10000
Propane (in ppmv)	1	10	100	500	1000	5000	10000
Butane (in ppmv)	1	10	100	500	1000	5000	10000
Isobutane (in ppmv)	1	10	100	500	1000	5000	10000
Acetylene (in ppmv)	1	10	100	500	1000	5000	10000

- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD)- 100ppm air liquid custom mix, or equivalent. Standard is transferred to a Tedlar bag until partially inflated for ease of use. 10uL of this standard is injected into the GC for an LCS analysis.

9.2.3 Calibration Sequence

Example calibration sequence includes an instrument blank, ICAL, and SSCV. Sequence may continue with a batch of samples beginning with batch QC (Blank/LCS/D).

9.2.4 Calibration Evaluation

9.2.4.1 Curve Fit

A minimum of five (5) points must be used for an initial calibration curve, using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. Acceptance criteria for initial calibration verification (ICV) is ±15%.

RL checks are evaluated but are currently applicable to Minnesota samples only.

All compounds must pass with <15% RSD or a correlation coefficient of 0.990 or better using the most appropriate curve fitting model from among the following choices below (given in order of preference):

Average Response Factor

- Must be <15% RSD

Linear- No Weighting, 1/x Weighting, $1/x^2$ Weighting

- Correlation coefficient of 0.990 or better.
- **STATE NOTE:** For samples analyzed from the state of Minnesota, the correlation coefficient must be 0.995 or greater. All calibration points must be re-processed following calibration and must have a %recovery within +30% of the target concentration for all analytes in all standards, except for the lowest calibration standard which must recover within +40%. If the criterion is not met the reporting limit must be amended to reflect the increased concentration of the standard utilized.

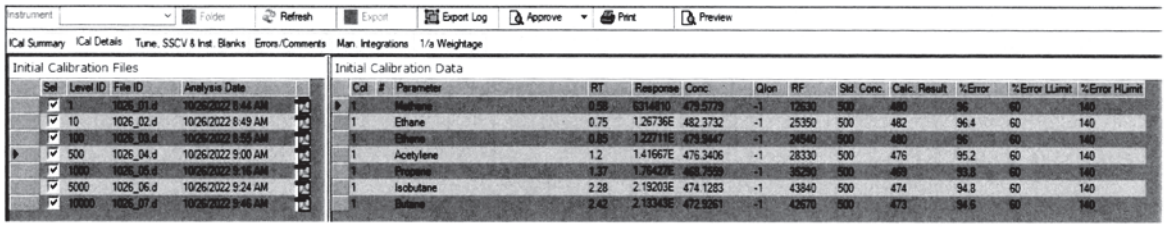
High order polynomial curves (i.e. second order, third order and greater) are not allowed at Pace National.

9.2.4.2 Relative Error

For Multi-point calibration that use average response factor, RSD is the measure of relative error and not additional calculation is required.

For multi-point calibration that use linear regression, the Relative Standard Error (RSE) must be evaluated at the low standard and the midpoint of the calibration. Place see the Pace calibration policy, ENV-POL-CORQ-0005 *Acceptable Calibration Practices*, for the acceptance limits if not explicitly stated in the method.

RSE is automatically calculated in Organics TREE when the calibration is loaded into the initial calibration window. Tree Calculates RSE for each analyte and each level based on the updated ICAL summary from Chemstation.



9.2.4.3 Initial Calibration Verification

Daily verification of the calibration curve can be used in lieu of a full calibration. Prior to analysis of samples, rinse the appropriate syringe a minimum of 3 times.

9.2.4.4 Continuing Calibration Verification

For routine analysis, a single point continuing calibration verification sample or CCV is evaluated at the beginning of the run to ensure on-going calibration stability, every 10 client samples, and at the conclusion of analysis.

9.3 Sample Preparation

- When requested, MS/MSD samples can be prepared. Then using a syringe, 100µL of sample headspace is pulled into a syringe along with 10µL of the LCS spiking standard. This is directly injected into the GC for analysis.
- If sample is received in a Snap Sampler vial and there is headspace present, then the amount of headspace is estimated and recorded. The total headspace created is equal to the estimated headspace plus the added headspace to create a total headspace in the vial of 20cc.
- No sample preparation is needed for air samples.
- Blank preparation: Water (RSK175) 1 40mL VOA vial is filled with volatile organic free water and is prepped with the batch of samples following the same prep steps as the samples. For Air matrix (8015/TO-3) a tedlar bag is filled with Helium gas.
- Water samples: A 100uL aliquot of the headspace from each sample is injected into the GC using a 100uL gas-tight syringe for direct inject (manual) samples only. For Air samples: 100ul of sample is pulled from the tedlar bag or canister and injected into the GC inlet. Between samples the syringe is rinsed to eliminate sample carry-over. Lab air syringe 'rinses' are sufficient for Air analysis.

9.4 Analysis

9.4.1 Preparation

- Samples are pulled into batches of at most 20. Water samples are removed from the refrigerator for RSK175 and are placed in the water bath or incubator and equilibrated to 25°C–approximately 1 hour. Water Bath/Incubator temperature is documented with each batch in the PrepData program. Air samples in Tedlar bags or summa canisters are stored at room temperature in the laboratory.
- There are 2 options for analysis of samples:
    - Manual Direct Injection-

- RSK175: After approximately 1 hour, samples are prepared by creating 20cc of headspace in each sample vial. The headspace is generated using a 100cc syringe of helium to force 20cc of headspace into a 50cc syringe. Samples are to be mechanically shaken at 1100rpm (set at 55 on the Glass-Col Large Capacity Mixer) for 5 minutes. They are then analyzed approximately 2 hours after the samples have reached equilibrium.
- 8015/TO-3. No preparatory steps. 100ul of sample is pulled from the tedlar bag or canister and injected into the GC inlet.

- Headspace Analyzer – (RSK175 only) After approximately 1 hour after samples have been removed from the refrigerator, samples are placed on and prepared in sequential order by the headspace analyzer using the following typical conditions:

Heating sample	10 minutes at 100°C
Vial pressure Time	12 seconds
Loop fill time and Loop Equil.Time	2 seconds
Valve Temp	85°C
GC Line Temp	130°C

9.4.2 Example Analytical Sequence

Batches are defined as sets of 1 - 20 samples. Batch analysis must include the following: 1 Method Blank, 1 Continuing Calibration Verification (CCV) or 5-Level Calibration Curve, 1 Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD), 1 Continuing Calibration Verification (CCV) following every 10 field samples, and 1 CCV at end of run.

10.0 Data Analysis & Calculations

Carry out calculations retaining at least 1 extra decimal figure beyond that of the acquired data. Round off results only after the final calculation.

Sample multipliers are calculated by the equation below. All samples are considered to achieve equilibrium due to 20mL of sample and 20cc of headspace. These multipliers are applied to each of the calibration standard levels, so the instrument quantitation reports reflect these calculations, and no further calculations are needed in LIMS or by the end user.

EPA RSK 175 Calculation Validation:

$TC = A_i + C$ *EQ 10*

$TC = N_g * MW * 1000 + A_h * D/L_w$ *EQ 6 and 9*

$TC = P_g * MW * 55000/Hc + mL_{hs} * P_g * D/L_w$ *EQ 5*

$TC = P_g * MW * 55000/Hc + mL_{hs} * P_g * MW/ (L_w * 24.45)$ *EQ 7*

Headspace = 20mL

Water Extracted = 0.02L

$TC = P_g * MW * 55000/Hc + 20mL * P_g * MW/ (0.02 * 24.45)$

$TC = P_g * MW * 55000/Hc + 40.9 * P_g * MW$

Methane: MW = 16, Hc = 37000

$TC = P_g * 16 * 55000/37000 + 40.9 * P_g * 16$

$TC = P_g * 23.8 + P_g * 654.4 = P_g * 678.2$, which is a multiplier of 0.000678

Ethane: MW = 30, Hc = 28000

$TC = P_g * 30 * 55000/28000 + 40.9 * P_g * 30$
$TC = P_g * 43.4 + P_g * 1227 = P_g * 1270$, which is a multiplier of 0.00129

Ethene: MW = 28, Hc = 12000
$TC = P_g * 28 * 55000/12000 + 40.9 * P_g * 28$
$TC = P_g * 128.3 + P_g * 1145.2 = P_g * 1273$, which is a multiplier of 0.00127

Propane: MW = 44, Hc = 40000
$TC = P_g * 44 * 55000/40000 + 40.9 * P_g * 44$
$TC = P_g * 60.5 + P_g * 1800 = P_g * 1860$, which is a multiplier of 0.00186

Acetylene: MW = 26. Hc = 1400
$TC = P_g * 26 * 55000/1400 + 40.9 * P_g * 26$
$TC = P_g * 1021.4 + P_g * 1063 = P_g * 2085$, which is a multiplier of 0.00208

Butane: MW = 58, Hc = 51000

$TC = P_g*58*55000/51000 + 40.9*P_g*58$

$TC = P_g*62.5 + P_g*2372.2 = P_g*2434.7$, which is a multiplier of 0.00243

Isobutane: MW = 58, Hc = 65000

$TC = P_g*58*55000/65000 + 40.9* P_g*58$

$TC = P_g*49.1 + P_g*2372.2 = P_g*2421.3$, which is a multiplier of 0.00242

Where:

TC= total concentration

A= analyte in liquid phase

C= concentration

Ng= moles of gas

MW= molecular weight

Ah= analyte headspace

D= density at 25°C

Lw= amount of sample in vials in liters

Pg= partial pressure of target analyte

Hc= Henry's constant (see below)

mLhs= milliliters of headspace

Henry's Constants at 25°C

Name	MW	K	1/K	Calculated Henry	Round Henry
Methane	16.04	0.0015	666.67	37481.00	37000
Ethane	30.07	0.002	500.00	28110.75	28000
Ethene	28.05	0.0048	208.33	11712.81	12000
Propane	44.06	0.0014	714.29	40158.45	40000
Acetylene	26.04	0.039	25.64	1441.52	1400
Butane	58.12	.0011	909.09	51110.45	51000
IsoButane	58.12	.00086	162.79	65373.84	65000

Henry = 1/K * 55.5 * 1.013

K values from www.webbook.nist.gov using Yaws and Yang, 1992

10.1 Qualitative Identification

10.1.1 Tentatively Identified Compounds (TICs)

Not applicable

10.1.2 Manual Integration

Manual integration is sometimes necessary to correct inaccurate automated integrations but must never be used to meet QC criteria or to substitute for proper instrument maintenance and/or method set-up. To assure that all manual integrations are justified and proper all manual integrations must be performed, documented, reviewed, and approved in accordance with corporate SOP ENV-SOP-CORQ-0006, *Manual Integration.* Refer to this SOP for guidance on manual integration techniques and required procedures.

11.0 Quality Control & Method Performance

11.1 Quality Control

No QC failures are allowed when reporting this analysis with the exception of high failures. If the QC fails high only non-detect target parameters may be reported. Continuous re-analysis of QC is not permitted. If samples are to be reported using low failing instrument or batch QC, appropriate qualification must be used. This could occur if no additional sample is remaining for re-injection or failures are matrix related.

Prepare the following QC samples with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action(s).

QC Check	Acronym	Frequency
Method Blank	MB	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
Laboratory Control Sample	LCS	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
LCS Duplicate	LCSD	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
Matrix Spike	MS	Upon client request.
Matrix Spike Duplicate	MSD	Upon client request.
Sample Duplicate	SD	Every 10 samples.

11.2 Instrument QC

Perform the following checks to verify instrument performance. Refer to Appendix B for acceptance criteria and required corrective action.

Instrument Check	Acronym	Frequency
Initial Calibration Verification	ICV	Before any samples are ran.
Continuing Calibration Verification	CCV	Every 1 injections and at the end of run.

11.3 Method Performance

11.3.1 Method Validation

Refer to corporate SOP ENV-SOP-CORQ-0011 for general requirements and procedures for method validation.

Establish detection limits (DL) and limits of quantitation (LOQ) at initial method set up and verify the DL and LOQ on an on-going basis thereafter. Refer to corporate policy and/or SOP for DL and LOQ requirements and procedures.

12.0 Data Review & Corrective Action

12.1 Data Review

The data review process of Pace® Analytical Services includes a series of checks performed at different stages of the process by different people to ensure that SOPs were followed, the analytical record is complete, and properly documented, QC criteria were met, proper corrective actions were taken for QC failure and other nonconformance(s), and test results are reported with proper qualification, when necessary.

The review and checks that are performed by the employee performing the task is called primary review.

All data and test results are also peer reviewed.

This process, known as secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented, and approved in accordance with the Pace® Analytical Services SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

Lastly, a third-level review, called a completeness check, is performed by reporting or project management staff to verify the test report is complete.

Refer to laboratory SOP ENV-SOP-MTJL-0038_*Data* Review for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is required when QC or sample results are not within acceptance criteria.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 Pollution Prevention & Waste Management

Pace® proactively seeks ways to minimize waste generated during work processes. Some examples of pollution prevention include but are not limited to reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practices comply with all applicable federal and state laws and regulations. Excess reagents, samples, and method process wastes are characterized and disposed of in an acceptable manner in accordance with the Pace® Chemical Hygiene Plan / Safety Manual. Refer to this manual for these procedures.

14.0 MODIFICATIONS

The procedures in this SOP have been modified from the reference test method as follows:

Modification	Test Method Procedure	Justification for Modification
Adjustment to the concentrations of standards/spiking solutions, standards providers, and quality control	N/A	These are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.
Modifications to this method are noted in the body of the text as state notes.	N/A	Compliance analyses performed in conjunction with specific state requirements must be performed as noted within specific state(s) note listed.

When applicable, comparability and/or equivalency studies necessary to validate the modification as required per corporate SOP ENV-SOP-CORQ-0011 are retained by local quality personnel for historical reference.

Method TO-3 discusses the use of cryogenic concentration to assist with sample delivery to the GC system. Due to the compounds of concern and concentration or reporting limits, the cryogenic concentration is bypassed in lieu of a direct injection method being used for Air samples.

15.0 RESPONSIBILITIES

- All employees of Pace® Analytical Services that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement (R&A) in their training file for the version(s) of the SOP that were in effect during the time the employee performed the activity.
- Local quality personnel are responsible for tracking the currency of the R&A on this SOP for employees at the locations they are assigned to and for notifying the General Manager (GM), however named, when R&A are overdue or outstanding. The GM and the employee's direct supervisor are responsible for ensuring the employee completes the R&A assignments as required.
- The supervisors and managers of Pace® Analytical Services, however named, are responsible for training employees on the procedures in this SOP, implementing the SOP in the work area, and monitoring on-going adherence to the SOP the work area(s) they oversee.
- All employees of Pace® Analytical Services are responsible for following the procedures in this SOP. Unauthorized deviations or departures from this SOP are not allowed except with documented approval from the local Quality Manager and only when those deviations do not violate the Pace® Code of Ethics or Professional Conduct (COR-POL-0004) or associated policy and procedure(s). Hand-edits or manual change to the SOP are not permitted. If a change is desired or necessary, Pace® employees must follow the procedures for document revision specified in corporate SOPs ENV-SOP-CORQ-0015 *Document Management* and ENV-SOP-CORQ-0016 *SOP for Creation of SOP and SWI*.
- Local quality personnel are responsible for monitoring conformity to this SOP during routine internal audits of work areas that utilize this SOP and for communicating gaps and deviations found during monitoring to the work area supervisor, who is responsible for correction of the situation.

16.0 ATTACHMENTS

- Appendix A: Routine Analyte List and LOQ
- Appendix B: QC Summary & Corrective Action Table
- Appendix C: DoD Requirements

17.0 REFERENCES

- ENV-POL-CORQ-0005, *Acceptable Calibration Practices,* current version.
- ENV-SOP-CORQ-0006, *Manual Integration*, current version.
- ENV-SOP-CORQ-0011, *Method Validation*, current version.
- ENV-SOP-CORQ-0015, *Document Management*, current version.
- ENV-SOP-CORQ-0016, *SOP for SOP and SWI,* current version.
- ENV-MAN-MTJL-0001, *Quality Manual*, current version.
- COR-POL-0004, *Code of Ethics and Professional Conduct*, current version.
- COR-MAN-001, *Pace® Safety Manual,* current version.
- *RSK SOP-175*, U.S. EPA, Ground Water and Ecosystems Restoration Division, Revision 2, May 2004.
- Method TO-3, U.S. EPA, *Method for the Determination of Volatile Organic Compounds in Ambient Air Using Cryogenic Preconcentration Techniques and Gas Chromatography with Flame Ionization and Electron Capture Detection,* Revision 1, April, 1984.
- *Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique*, Don Kampbell and Steve Vandegrift, Journal of Chromatographic Science, Volume 36, May 1998.

18.0 REVISION HISTORY

Authorship

Primary Author[1]	Job Title	Date Complete
Matt Ferrell	Air Manager	3/31/2023

[1]The primary author is the individual / role responsible for the content of this SOP. Send questions or suggestions for content to the primary author. See the Quality Manager for questions or concerns related to implementation of this SOP.

Revisions Made from Prior Version

Section	Description of Change
All	Formatted to corporate template; Annual review and addition of TO-3 criteria.

Document Succession: This version replaces the following documents:

Document Number & Version	Document Title	Effective Date:
ENV-SOP-MTJL-0106 v03	RSK175	2/21/2022
ENV-SOP-MTJL-0106 v02	RSK175	7/9/2020
ENV-SOP-MTJL-0106 v01	RSK175	7/24/2019

Appendix A: Target Analyte List and LOQ

Table 1: Standard Analyte List and LOQ

Analyte	CAS #	LOQ[1] Matrix (Air/ppmv)	LOQ[1] Matrix (water/mg/l)	LOQ[1] Matrix (units)
ACETYLENE	74-86-2	10	.0208	
BUTANE	106-97-8	1	N/A	
ETHANE	74-84-0	10	.00129	
ETHENE	54509-73-8	10	.00127	
ISOBUTANE	75-28-5	1	N/A	
METHANE	74-82-8	10	.000678	
PROPANE	74-98-6	10	.0186	

[1] *Values as of effective date of this SOP. LOQ are subject to change, contact quality personnel for most current information.*

Appendix B: QC Summary and Corrective Action Table

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICV	Before any samples are ran.	Must perform within +15%	The laboratory shall demonstrate acceptable performance after corrective action with 2 consecutive calibration verifications, or a new initial instrument calibration shall be performed.	N/A
CCV	Every 10 samples and at the end of the analytical run.	Must perform within +15%	The samples between the CCV bracket should be re-analyzed if failures are low, any samples that are non-detected with a high failure are reportable. All samples containing target analytes above the RL that are not bracketed by acceptable calibration/verification must be re-analyzed.	N/A
Method Blank	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.	Must be free of interferences and below the MDL for all target compounds.	The instrument must be checked for problems before continuing analysis. If the MB concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.	If the MB concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, qualify associated sample results to indicate that analyte was detected in the MB.
LCS/LCSD	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.	Must pass within 85-115% (70-130% for 8015-TO-3) with an RPD of <20%	Re-analyze once. If failure persists, the instrument should be checked for performance and re-calibration should be performed if needed.	N/A
MS/MSD	Upon client request.	+15% (70-130% for 8015-TO-3) with an RPD of <20%	Failures are considered to be matrix specific unless analyst error is suspected, and all other QC meets the method criteria.	The parent sample will be qualifiec appropriately for failures.
Sample Duplicate	Every 10 samples.	RPD of <20%	Re-analyze once. If failure persists, qualify the data where allowable.	When the sample and duplicate results are less than 5x the RL, use the J3 qualifier. When the sample and duplicate results are greater than 5x the RL, use the P1 qualifier.

Appendix C: DoD Requirements

1.0 Equipment/Instrument Maintenance

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking traps and columns, changing injection port liners, replacing columns, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

2.0 Computer Hardware and Software

Software name and version: HP Chemstation G1701BA Version B.01.00 or equivalent

3.0 Troubleshooting

Table 1. GC Troubleshooting Guide

Problem	Cause	Treatment
No Peaks	Syringe clogged	Clean or replace syringe
	Detector/Software/Computer failure	Check cables. Restart computer.
	Column Leaks	Use new ferrules.
	Broken Column	If at ends, clip column. If in the middle or multiple sites, replace column.
Peaks too Small	Split too high	Reduce split
	Column connection leaks	Check column installation. Search for leaks. Replace ferrules.
	Injector temperature too low	Check temperature program. Increase injector temperature.
	Dirty ECD	Clean ECD.
Retention Times Change	Gas flow too low or too high	Replace septum. Check gas regulator.
	Oven temperature unstable	Check temperature program. Check temperature with external thermometer.
	Column blocked	Compare flow at column entrance to outlet. Replace column.
Constantly Rising Baseline	Leak at column entrance or injection septum.	Check column installation; search for leaks; replace ferrules.
	Injector contaminated.	Make a run at lower injector temperature; if the baseline improves, replace liner, use low bleed or high temperature septa.
	Column contaminated.	Cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column.
	Detector contaminated.	Clean detector.
	Increase of temperature too fast.	Decrease temperature gradient and end temperature.
	Poor gas quality.	Use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC.

Table 1. GC Troubleshooting Guide		
Problem	**Cause**	**Treatment**
Increasing Baseline at High Temperatures	Decomposition of the stationary phase.	Check for leaks; matrix check for compatibility with the column.
	Column contaminated.	Cut two turns from column entrance; rinse column with solvent (only chemically bonded phases); otherwise replace column or use guard column.
	Increase of temperature too fast / end temperature too high.	Decrease temperature gradient and end temperature.
	Column not properly conditioned.	Condition column according to manufacturers' instructions (while column is not connected to the detector).
	Detector contaminated	Clean detector according to manufacturers' instructions.
	Poor gas quality.	Use gas grades recommended for GC; for longer supply lines from gas source to GC use gas purification cartridges directly connected to the GC.
Plateaus at Certain Temperatures	Steps in temperature program too drastic.	Avoid very short and strong heating periods.
Fronting	Column overload.	Decrease injection volume; dilute sample.
	Sample vaporizes too slowly, not evenly or condenses.	Increase injector temperature (consider max. temperature limits of the column).
	Analytes coelute.	Change temperature program or use column with different selectivity.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner.
	Column absorbs or decomposes analytes.	Check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; and replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity.
Tailing	Sample vaporizes too slowly, not evenly or condenses.	Increase injector temperature (consider max. temperature limits of the column).
	System leaks.	Check column installation; search for leaks; replace ferrules.
	Analytes coelute.	Change temperature program or use column with different selectivity.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one.
	Column absorbs or decomposes analytes.	Check capillary ends; check intact deactivation using the test mixture; for poor results shorten both column ends by about 10 cm; and replace column; if column test does not show any defects: a) use a column with thicker film b) use phase with better deactivation c) use column with special selectivity.
	Split rate too low.	Increase split rate.
	Column overload.	Decrease injection volume; dilute sample.
Split Peaks	Solvent and column not compatible.	Change solvent or use guard column.
	Solvent mixtures with large differences in boiling point and polarity.	Use just one solvent.
	Sample decomposes.	Check temperature program, oven temperature (external thermometer); if analytes are not temperature-stable, reduce injector temperature; replace liner by a deactivated one.

Table 1. GC Troubleshooting Guide		
Problem	**Cause**	**Treatment**
	Analytes coelute.	Modify temperature program or use longer column; possibly change column polarity.
	Detector overload.	Inject less; control make-up flow.

4.0 Other Requirements

4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.

4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.

4.3 A storage blank must be stored with all volatile organic samples, regardless of suspected concentration levels.

4.4 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

4.5 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used, or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

Table 2. Support Equipment Checks		
Performance Check	**Frequency**	**Acceptance Criteria**
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: ±2% or ±0.02g, whichever is greater Analytical balance: ±0.1% or ±0.5 mg, whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through an NMI	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e., 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out-of-control event capabilities if personnel not available to record daily]	Refrigerators: 0°C to 6°C Freezers: ≤-10°C
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use Class A and B: Upon evidence of deterioration	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within ±3% of nominal volume Precision: RSD ≤3% of nominal volume (based on 10 replicate measurements)

Table 2. Support Equipment Checks		
Performance Check	**Frequency**	**Acceptance Criteria**
Mechanical volumetric pipette	Quarterly	Bias: Mean within ±2% of nominal volume Precision: RSD ≤1% of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within ±5% of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

4.6 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.

4.7 To avoid preparing non-representative samples, the laboratory shall not "target" within a relatively small mass range (e.g., 1.00 ± 0.01g) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material, so the sample aliquot weighs exactly 1.00g ± 0.01g, as an example.

4.8 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:

U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.

J The reported result is an estimated value (e.g., matrix interference was observed, or the analyte was detected at a concentration outside the quantitation range).

B Blank contamination. The recorded result is associated with a contaminated blank.

N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.

Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).

Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).

4.9 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.

4.10 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the LOD/LOQ verifications on the worst-case basis (preparation method with all applicable cleanup steps).

4.11 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least 2 times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:

- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
- If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
- The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
- The DL and LOD must be reported for all analyte-matrix-methods suites unless it is not applicable to the test or specifically excluded by project requirements.

4.12 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.

4.13 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.

4.14 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.

4.15 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.

4.16 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.

4.17 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.

- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.
- Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
- Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

4.18 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Addendum Table 3) or project limits, if specified. If the specific analyte(s) are not available in the Addendum Table 3, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.

4.19 Surrogate spike results shall be compared with DoD LCS limits (see Addendum Table 3), or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).

4.20 The method blank shall be considered to be contaminated if:

- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/0th the regulatory limit, whichever is greater.
- The concentration of any common laboratory contaminant in the blank exceeds the LOQ.
- If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.

4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.

4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

Table 3. LCS Control Limits – Method RSK-175 Water Matrix

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
74-86-2	Acetylene	719	99.6	9.8	70	129
106-97-8	Butane	262	97.3	7.3	75	119
124-38-9	Carbon dioxide	441	100.8	6.9	80	122
74-84-0	Ethane	2240	102.6	9.6	74	131
74-85-1	Ethylene	2284	102.5	10.2	72	133
75-28-5	Isobutane	267	98.6	6.6	78	117
74-82-8	Methane	2459	99.2	8.1	73	125
74-98-6	Propane	900	98.1	8.2	74	123

Table 4. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: **Option 1:** RSD for each analyte ≤ 20%; **Option 2:** linear least squares regression for each analyte: r2 ≥ 0.99; **Option 3:** non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.	Correct problem then repeat ICAL.	Flagging is not appropriate.	Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA	NA	Calculated for each analyte and surrogate.
Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study or 0.03 minutes, whichever is greater.	NA	NA	Calculated for each analyte and surrogate. Only applicable if internal standard calibration is not used.

Table 4. Quality Control Requirements – Organic Analysis by Gas Chromatography					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified with a second source.
Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticides multi-component analytes (i.e., Toxaphene, Chlordane), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 20% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action(s), and reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	No analytes detected >1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, re-prep and reanalyze MB and all QC samples and field samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table 4. Quality Control Requirements – Organic Analysis by Gas Chromatography

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use Table 3 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch.	A laboratory must use Table 3 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	One per preparatory batch.	A laboratory must use Table 3 limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits (see the LIMS) if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ.

Table 4. Quality Control Requirements – Organic Analysis by Gas Chromatography					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Surrogate Spike	All field and QC samples.	QC acceptance criteria specified by the project if available; otherwise use Table 3 limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then re-prep and reanalyze all failed samples for all surrogates in the associated preparatory batch if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data, and the failures must be discussed in the Case Narrative.	Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the Case Narrative.	Alternative surrogates are recommended when there is obvious chromatographic interference.

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ENVIRONMENTAL SCIENCES

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QM Approval

Name/Signature	Title	Date	Meaning/Reason
Rebecca King (010125)	Manager - Quality	21 Feb 2022, 05:36:05 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Heidi Ferrell (006481)	Manager - Operations	04 Nov 2021, 08:42:17 AM	Approved

Pace Analytical®
National Center for Testing & Innovation

STANDARD OPERATING PROCEDURE
TITLE: Total Alkalinity, Manual and Automated (Titration)(SM 2320B)
ISSUER: Pace National – Mt. Juliet, Tennessee

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1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to drinking, surface, saline, and ground waters, domestic and industrial wastes.

1.2 The nominal Reporting Limit (RL) is 20mg/L as CaCO3. See LIMS for active limits.

2.0 METHOD SUMMARY AND DEFINITIONS

2.1 The acid-neutralizing capacity of a water sample Is determined by titration to a pre-determined endpoint pH using 0.1N Sulfuric Acid. For routine analysis, the endpoint pH is 4.5su.

2.2 Alternate LCS (also called Independent Calibration Standard, ICV)– A control of known composition, at a different concentration than the LCS, prepared from a different stock solution than the LCS. The concentration of the alternate LCS may be changed periodically.

2.3 See the current Quality Assurance Manual for definitions associated with terms found in this document.

3.0 HEALTH AND SAFETY

3.1 The toxicity or carcinogenicity of each chemical or sample being diluted in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

3.2 The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets (SDS) for all hazardous chemicals are available to all personnel. Employees must abide by the environmental, health, and safety (EHS) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.

3.3 Personal protective equipment (PPE) such as safety glasses and/or side shields, gloves, a laboratory coat, and shoes that are not cloth, canvas, and/or perforated must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure. When handling glass, needles, knives, or any material with a potential sharp edge, employees must use cut-resistant gloves.

3.4 Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids, bases, or oxidizers in a fume hood whenever possible with the appropriate PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions.

3.5 Spill kits are located in each laboratory department. Employees are to familiarize themselves with the location and contents of each spill kit in their area.

3.6 Universal precautions should be observed when performing any tests or procedures. Hard surfaces, instrument surfaces may be contaminated and should be handled according to good laboratory practices.

3.7 Contact your supervisor or local EHS coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure. Any accidents involving

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personnel or sample supplies are to be reported immediately to either the Manager and/or to the Safety Officer.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 All samples must have been collected using a sampling plan that addresses the considerations of this method.

4.2 Sample bottles may be either polyethylene or glass.

4.3 Sample bottles should be stored at ≤6°C (not frozen) and analysis performed as soon as practical. Maximum holding time is 14 days.

5.0 INTERFERENCES

5.1 Soaps, oily matter, suspended solids, or precipitates may coat the glass electrode and cause a sluggish response. Allow additional time between titrant additions to let electrode come to equilibrium or clean the electrodes.

5.2 Do not filter, dilute, concentrate, or alter the original sample.

6.0 EQUIPMENT AND SUPPLIES

–

Metrohm Model 855 Autosampler

- Model 772 Pump Unit
- Model 800 Dosing Unit(s)
-
- Model 6.0257.00 Combination pH electrode or equivalent
- 120mL Autosampler Vessels (Metrohm 020211903 or equivalent)
- Note - Equivalents systems are acceptable
- Volumetric glassware, Class A

7.0 REAGENTS AND STANDARDS

7.1 All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standard Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six (6) months or sooner if a problem is detected unless otherwise noted.

7.2 Sodium Carbonate solution, 0.05N – Dry 3-5g primary standard Na_2CO_3 for four (4) hours at 250 ± 10°C, cool and store in a desiccator. Weigh 2.5g +/- 0.2 g (to the nearest mg). Transfer to a 1L volumetric flask containing approximately 500mL reagent water to dissolve and mix reagent. Fill to the 1L mark with reagent water. Expiration – one week.

7.3 Standard Sulfuric Acid, 0.1N – Add 2.8mL of concentrated Sulfuric Acid to a 1L volumetric flask containing 500mL reagent water. Fill to the mark with reagent water and mix. Standardize against the 0.05N Na_2CO_3: Add 40mL of 0.05N Na_2CO_3 to a beaker. Add 60mL reagent water. Amount may vary. Record.Titrate potentiometrically to a pH of about 5.0. Lift out electrodes and rinse into beaker. Boil gently for 3-5 minutes under a watch glass. Cool to room temperature and rinse watch glass into beaker. Finish titrating to pH 4.5. See the equation in section 9.1 to calculate the exact normality of the acid solution. Standardize this solution monthly. Commercially certified titrant is acceptable.

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For samples less than 100 ug/ml, the use of a 0.02 N standard is acceptable. Prepare by dilution of the 0.1 N or prepare acid. Standardization is required.

7.4 1000mg/L Alkalinity Primary Stock standard – Purchase prepared standard, Sigma-Aldrich Alk1000, or equivalent. Expiration – follow manufacturer's instructions. ISO 34, if available.

7.5 100mg/L LCS – Dilute 100mL of the 1000mg/L standard to 1000mL with reagent water using volumetric glassware. Expiration – one month.

7.6 Alternate LCS – Dilute 500mg/L Secondary Stock standard to make a standard of different concentration and from a different source than the LCS. For a 50 mg/L standard, dilute 100 ml stock to 1000ml reagent water. Expiration – one month. A commercial 500µg/mL standard is acceptable (Reagent Chemicals 500 ug/ml CA017700-1A). ISO 34, if available.

7.7 RLV (20 mg/L) - If necessary, an RLV must be analyzed to demonstrate accurate quantitation at the reporting limit being used. Dilute 10mL of the 1000mg/L standard to 500mL using volumetric glassware.

7.8 pH 1 buffer (Ricca 1489-32 or equivalent), optional

7.9 pH 4 buffer (Inorganic Ventures PHRED-4 or equivalent)

7.10 pH 7 buffer (Inorganic Ventures PHYellow-7 or equivalent)

7.11 pH 10 buffer (Inorganic Ventures PHBLUE-10 or equivalent)

7.12 pH 13 buffer (Ricca 1625-16 or equivalent), optional

NOTE: Bulk buffers are valid for one month after opening.

7.13 Reagent water – ASTM Type II or equivalent. Target less than ½ RL. Boiling and/or purging with Nitrogen or Helium is allowed.

8.0 PROCEDURE

8.1 The pH meter within the Titrando unit is calibrated each day it is in use. It is standardized using at a minimum 4,7 and 10 buffers. Additional pH buffers may be used.

8.1.1 Buffers transferred to beaakers/vessels can be used only once. Use the smallest possible aliquots of each buffer that provides sufficient probe immersion and discard remainders of the aliquots when completed.

8.1.2 A calibration curve must be performed daily. The Nernst slope must be 95-105%.

8.2 Fill out the sample table with the QA checks and samples to be analyzed. Make sure the alkalinity method is specified and include all other pertinent information, including the RLV if required.

A typical workgroup will be analyzed in the following order:

Startup/sequencing Blank (INSTBLK)

ALT (second-source) (50 mg/Lfor 0.02 N or 500 mg/L for 0.1 N) (once per sequence)
LCS
Method Blank

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RLV (20 ug/ml), if necessary (once per sequence)
10 samples
Duplicate
CCV (100 mg/L) Primary Stock Standard)
CCB
10 samples
Duplicate
CCV (100mg/L)

CCB

NOTE – if using two titrants of different Normality, at least one check standard must be run with each.

8.3 Pour samples and QA checks into autosampler vessels and place in the autosampler positions specified in the sample table.Include Worklist ID, Sample ID, titrant info and sample volume.

8.4 Click on [Start]. ; Samples will be automatically titrated to an endpoint of 4.5su for Total Alkalinity.

8.5 If a sample requires more than 20mL of titrant, the Titrando will record a value of "Invalid." Re-titrate these samples using less sample.

8.6 If the initial pH is less than 4.5, the Titrando will titrate using 20mL of titrant but record a value of "Invalid." This will be reported as <20mg/L alkalinity. Samples with low initial pH may cause carryover in the titration cup. If necessary, rinse the titration cup with reagent water until carryover contamination is eliminated.

8.7 When the analysis is finished, export the data to Tree and capture. Tree will calculate Carbonate, Bicarbonate, and Hydroxide alkalinities using the volume of titrant needed to reach a pH of 8.3 and 4.5. Instrument software may calculate results.

9.0 DATA ANALYSIS AND CALCULATIONS

9.1 Calculate the exact Normality of the H_2SO_4 using the following equation:

$$\text{Normality of } H_2SO_4 = \frac{(\text{g } Na_2CO_3 \text{ weighed into flask}) \times (\text{mL } Na_2CO_3 \text{ solution used for titration})}{(53.00) \times (\text{mL acid used})}$$

9.2 Calculate Alkalinity as mg $CaCO_3$/L using the following equation:

$$\text{Total Alkalinity mg/L as CaCO3 to pH xx} = \frac{(\text{mL std acid})(\text{N std acid})(50{,}000)}{\text{mL of sample}}$$

9.3 If Carbonate Alkalinity, Bicarbonate Alkalinity, or Hydroxide Alkalinity are requested, . If the initial sample pH is <8.3, P=0. Calculate the component alkalinities from the following table (T=total alkalinity):

Result of Titration	Hydroxide	Carbonate	Bicarbonate
P=0	0	0	T
P<0.5T	0	2P	T-2P
P=0.5T	0	2P	0
P>0.5T	2P-T	2(T-P)	0
P+T	T	0	0

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9.4 See the current Quality Assurance Manual for other equations associated with common calculations.

10.0 QUALITY CONTROL AND METHOD PERFORMANCE

10.1 All analysts must meet the qualifications specified in ENV-SOP-MTJL-0015, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency before being approved to perform this method. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department.

10.2 Use the designated Run log to record batch order and standards/reagents used during analysis. See ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

10.3 Batches are defined as sets of 1 - 20 samples. Batch analysis must include the following: 1 Method Blank, 1 Laboratory Control Sample (LCS) and 1 Alt per sequence, 1 Sample Duplicate for every 10 samples, 1 CCV/CCB every 10 samples. All batch information must be maintained in the preparation documentation assigned to the department.

10.4 Method Blank - A Method Blank must be analyzed at the beginning of every workgroup. The concentration of target analyte in the blank must be less than the MDL or ½ RL, as needed.

10.5 Laboratory Control Sample (LCS): One LCS must be analyzed with every batch of 20 samples. An LCS is performed by prepping and analyzing a standard of known concentration and is treated the same as a sample. The recoveries for the LCS must be within ±10% of the true value.

10.6 RLV – The reporting limit verification when analyzed must recover within ±50% of the target concentration for the standard. Required once per sequence.

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly. The reporting limit verification (RLV) must recover within ±40% of the expected concentration. If this criterion is not met, the RLV may be re-analyzed once, instrument maintenance can be performed or a higher concentration standard can be analyzed. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

10.7 Sample Duplicate: One sample duplicate must be analyzed with every set of 10 samples. A sample duplicate is an additional aliquot of the original sample that has been taken through the entire procedure. The RPD must be ≤20%.

10.8 Alternate LCS: One alternate standard must be analyzed with every sequence. An alternate standard is a standard prepared from a different source that the LCS. The percent recovery must be ±15% of the expected value.

10.10 Continuing Calibration Verification (CCV and CCB) -Same source and concentration as LCS. Run every 10 samples and at end of run. Recovery must be within 10% or rerun samples affected. CCB less than ½ RL.

11.0 DATA VALIDATION AND CORRECTIVE ACTION

11.1 All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard and continuing

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calibrations to ensure that they meet the criteria of the method. The analyst should review any sample that has quantifiable compounds and make sure that they have been confirmed. The analyst must also verify that reported results are derived from quantitation between the RL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments are noted when data is flagged.

11.2 All data must then undergo a second analyst review. This review must be performed according to ENV-SOP-MTJL-0014, *Data Handling and Reporting.*

11.3 Method Blank - If a blank fails, it may be re-analyzed once. If the failure persists, the cause of the failure must be identified and corrected.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.
- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
    - **STATE NOTE:** For blanks associated with samples originating in West Virginia, any constituent(s) recovered in a method blank must generally be less than or equal to one-half the reporting level (unless the method specifies otherwise). If any MB measurements are at or above the reporting level, take the following immediate corrective action:If the reagent blank is less than the MDL and the sample results are greater than the MQL, then no qualification is required.
- If the reagent blank is greater than the MDL but less than the MQL and sample results are greater than the MQL, then qualify the results to indicate that the analyte was detected in the reagent blank. If samples are greater then the RL but less than ten times the blank, rerun.
- If the reagent blank is greater than the MQL, further corrective action and qualification is required. This may include re-analyzing the sample batch. An analyst should consult their supervisor for further instruction. If samples are less than ½ RL but the blanks isn't it is acceptable to report and qualify.

11.4 Laboratory Control Sample (LCS): If failure occurs, run one more time. If it still fails, re-prep and re-standardize the titrant and re-analyze all samples.

11.5 RLV: If failure occurs, re-analyze once. If failure persists, the cause of the failure must be identified and corrected. Corrections can include re-preparing the RLV standard, instrument maintenance, or raising the reporting limit being utilized, if acceptable to client and project requirements, and analyzing a higher concentration RLV, etc. A passing RLV must accompany each analytical sequence containing drinking water samples.

11.6 Sample Duplicate: If the RPD is >20%, the result may be reported with a J3 qualifier. If the RPD is beyond acceptance limits and the sample concentration is <5X the RL, then the value can be flagged with a "P1" qualifier indicating that the RPD calculation is not applicable at that concentration.

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11.7 The analyst must verify all reported results are above the reporting limit and that appropriate adjustments have been made to either sample volume or titrant concentration to produce a titrant volume of less than 20mL.

STATE NOTE: Drinking water samples analyzed using this procedure for compliance cannot be qualified.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 The EPA requires that laboratory waste management practices be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *Pace National Waste Management Plan.*

12.2 See ENV-SOP-MTJL-0046, *Environmental Sustainability & Pollution Prevention.*

13.0 METHOD MODIFICATIONS/CLARIFICATIONS

13.1 Analyses of Alternate LCS have been added to comply with Pace National internal QA policy.

13.2 Method modifications are not allowed for drinking water samples.

14.0 REFERENCES

14.1 *Required Containers, Preservation Techniques, and Holding Times*, 40 CFR PART 136.3, Table II and Standard Methods table 1060:I.

14.2 *Alkalinity by the Titration Method*, Standard Methods 2320B, 22 nd Editionor on-line version.

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Attachment I: Revision History

Current Version (Pace National):

Date	Description of Revisions
8/22/2020	Technical and quality review and update. Revised sections 7.4, 7.6, 7.7 and 11.3.

Superseded Versions (ESC Lab Sciences SOP #340301):

Version	Date	Description of Revisions
0	11/13/92	Origination
1	5/2/06	Technical and Quality Review and update.
2	2/12/08	Technical and Quality Review and update. EPA Method 310.1 was removed per MUR.
3	5/21/09	Annual Technical and Quality Review. Added sections 9.4 & 13.1. Updated sections 7.7, 10.3, 10.8, 11.4 & 11.5. Reformatted sections 10 & 11 and added State Note.
4	10/12/10	Annual Technical and Quality Review. Added sections 1.3, 2.7 through 2.13, 8.12, state note #2 following section 11.8 and 13.2; Revised sections 2.6, 7.1, 7.3, 7.4, 7.7, 9.1, 9.2, 9.4, 9.5, 10.8, 11.5, 11.6, and 12.1.
	11/3/11	Reviewed with no changes per K. Garrett/D. Marlin
5	11/7/13	Annual Technical and Quality Review. Consolidated ESC SOP# 340301 and 340301A into this procedure; Added sections 2.9, 6.2, 8.3, 9.6, 11.9, 14.3 and 14.4; Added state note to section 10.6; Revised title and sections 1.1, 4.5, 6.1, 7.1, 7.3, 7.8, 8.1, 8.2, 10.3, 10.6, 11.5 and 12.1; Deleted second state note in section 11.8 and removed sections 1.3, 2.5, 2.8 through 2.10.
6	11/7/2015	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 7.1 and 12.2.
7	11/17/2015	Grammatical corrections and update to add Titrando volume verification. Revised Sections 7.1, 10.6, 10.9, 10.9.1 through 10.9.7, and 13.1.
	1/18/2016	Revised a typographical error in the header. Changed the revision date from 11/18/13 to 11/18/15.
8	11/11/2016	Technical and quality review and update. Header and signature block re-formatting. Revised Sections 1.1, 2.2, 2.3, 3.0, 6.0, 7.8, 8.3, 8.5, 9.4, 9.5, 10.4, 10.8, 11.8, 14.1, 14.2, 14.3, and 14.4. Deleted Sections 2.2 through 2.4, 2.6 through 2.9, 6.1 and all subsections, 8.2 and all subsections, 9.4, 9.5, 11.7, 11.9.1, and 11.9.2. Added Sections 7.9, 8.6, 8.7, 8.8, and 9.6.
9	10/11/2017	Review per SC DHEC 9/27/17 letter. Revised Sections 7.4, 8.3, 8.6, and 10.3. Deleted Sections 7.9, 9.2, 10.4, 11.3, 11.8, and re-numbered as necessary. Added Section 13.3. Also deleted Section 9.4.

Superseded Versions (Pace National):

Date	Description of Revisions
12/22/2018	Technical and quality review and update. Deleted header, footer and signature bar. Revised sections 3.0, 4.5, 7.1, 7.2, 7.5, 7.6, 7.7, 8.3, 10.1, 10.2, 10.4, 10.5, 10.7, 11.1, 11.2, 11.3, 11.5, 11.6, 12.1, 12.2, 13.1, 14.1, 14.3 and 14.4. Deleted sections 4.3, 4.4, 8.1, 9.3, 13.2 and renumbered as necessary. Added sections 7.9, 7.10, 7.11, 7.12, 7.13, 8.1, 8.1.1, 8.1.2, 10.4, 11.3 and renumbered as necessary. Revised Attachment I.

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Date	Description of Revisions
9/19/2019	Update in response to SC DHEC 8/29/2019 technical review letter. Added corporate header and footer. Added Sections 8.7.1, 8.7.2, and 9.4.
4/2/2020	Added West Virginia state note to Section 11.3.
5/31/2020	Update in response to internal audit. Revised Sections 1.1, 1.2, 2.1, 2.2, all subsections of 3.0, 4.2, 5.2, 6.0, 7.2, 7.3, 7.5, 7.6, 7.7, 7.8, 7.9, 7.13, 8.1, 8.4, 8.5, 9.2, 10.3, 10.4, 10.5, 10.9.1, 10.9.2, 11.3, and 11.4. Added Sections 2.4, 7.14, 10.10, and 13.2. Deleted Sections 4.4, 7.4, 8.2, 8.7.1, 8.7.2, 9.4, 14.1, 14.3, and re-numbered as appropriate.

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of Nitrogen as Nitrite, Fluoride, Sulfate, Chloride, Nitrogen as Nitrate, Chlorate, Chlorite, and Bromide in aqueous matrices (drinking, surface, ground, effluent and influent water), soil extracts and combustates. by IC.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes that can be determined by this SOP and the associated LOQ is provided in Table 1, Appendix A.

2.0 SUMMARY OF METHOD

Aqueous samples, soil extracts and bomb combustates are injected into the Ion Chromatograph (IC) and separated on an analytical column equipped with a guard column. A Potassium Hydroxide or Carbonate/Bicarbonate solution serves as the eluent (mobile phase). Once separated, the anions pass through an anion suppressor where they are converted to their highly conductive forms. The separated anions are measured by conductivity and are identified by retention time as compared to known targets.

Ion Chromatography eliminates the need to use hazardous reagents and it effectively distinguishes among the halides (Br^-, Cl^-, F^-) and the oxy-anions (SO_3^{-}/SO_4^{-}, NO_2^{-}/NO_3^{-}, ClO_2/ ClO_3^{-}, PO_4^{-3}).

3.0 INTERFERENCES

- Any species with a retention time similar to that of the desired ion will interfere. Large quantities of ions eluting near the ion of interest will also result in interference.
    - Separation may be improved by adjusting the eluent concentration and/or flow rate.
    - Sample dilution and/or the use of the method of standard additions can also be used. For example, high levels of organic acids may be present in industrial wastes, which may interfere with inorganic anion analysis. Two common species, Formate and Acetate, may elute between Fluoride and Chloride depending on the column and eluent used.
- Interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to artifacts or elevated baselines.
- Targets, by definition, are "dissolved". Samples may be filtered using either 0.45/0.2 um pore filters/frits.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

Contact your supervisor or local safety coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure

The following sections provide general health and safety information about chemicals and materials that may be present in the laboratory.

- The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.
- The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (EHS) policies and procedures specified in this SOP and in the Pace® Chemical Hygiene / Safety Manual (COR-MAN-0001)
- Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.
- Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. For procedures that require use of acids, use acids in a fume hood whenever possible with PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. For procedures that that emit large volumes of solvents (evaporation/concentration processes), these activities must be performed in a fume hood or apparatus that reduces exposure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME & STORAGE

The laboratory provides containers for the collection of samples upon client request. Refer to laboratory SOP ENV-SOP-MTJL-0064, Sample Shipping for procedures related to preparation of bottle kits for the test method(s) associated with this SOP.

The laboratory does not perform sample collection or field measurements for this test method. Samples should be collected in accordance with a sampling plan and sampling procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

NOTE: The preservation and the holding times for samples analyzed by this procedure is determined by the anion(s) of interest. In a given sample, the anion that requires the preservation treatment and the shortest holding time will determine the preservation treatment utilized within the laboratory.

Container Type, Minimum Sample Amount, Preservation, and Holding Time Requirements:

Matrix	Container Size & Type	Required Sample Amount[1]	Preservation	Holding Time
Aqueous	plastic or glass bottles. Note: Samples for Chlorite and recommended	Volume collected must be sufficient to ensure a representative sample, allow for replicate	Thermal: See table below for analyte specific requirements Chemical: See table below for analyte specific requirements	Collection Analysis: See table below for analyte specific requirements

	for Chlorate must be protected from light .i.e. containers must be opaque or covered.	analysis, if required, and minimize waste disposal		
Leachates/Combustables	plastic or glass bottles. Note: Samples for Chlorite and recommended for Chlorate must be protected from light .i.e. containers must be opaque or covered.	Volume collected must be sufficient to ensure a representative sample, allow for replicate analysis, if required, and minimize waste disposal	Thermal: See table below for analyte specific requirements Chemical: See table below for analyte specific requirements	Extraction or bombing to analysis: See table below for analyte specific requirements

[1] *Amount of sample required for each discrete test.*

Analyte	Preservation	Holding Time
Bromide	None required	28 days
Chloride	None required	28 days
Fluoride	None required	28 days
Nitrate*	Cool to ≤6°C (not frozen)	48 hours*
Nitrite	Cool to ≤6°C (not frozen)	48 hours
Orthophosphate	Cool to ≤6°C (not frozen)	48 hours
Sulfate	Cool to ≤6°C (not frozen)	28 days
Chlorate	None required 300, EDA if 300.1	28 days
Chlorite**	Cool to 4°C (50 ppm EDA)	14 days

*For chlorinated drinking water samples, the holding time for Nitrate is extended to 14 days if kept at ≤6°C (not frozen).

**Since the sample cannot be analyzed for Chlorite within 10 minutes, the sample must be preserved by adding 1 mL of the Ethylenediamine (EDA) preservation solution to 1 L of sample or equivalent.

Thermal preservation is checked and recorded on receipt in accordance with laboratory SOP ENV-SOP-MTJL-0060, *Sample Receiving*. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at ≤6°C (not frozen) until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at ≤6°C (not frozen) C until sample analysis.

After analysis, samples are retained as stated in the Pace® standard terms and conditions, unless otherwise specified in the analytical services contract. Samples are then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT & SUPPLIES

Use of equivalent instrumentation, support equipment, and supplies from alternative vendors is acceptable so long as they meet the specifications as stated in the reference method(s).

7.1 Equipment

- Ion Chromatograph: Dionex ICS 2100, or equivalent.
- Detector: Dionex ED40, CD20 or equivalent.
- Automated sampler: Dionex AS-DV, or equivalent.
- Eluent Pump: Dionex GP40, GP50 or equivalent.
- Balance – Analytical, capable of accurately weighing to the nearest 0.0001 grams: Mettler AG204 Delta Range or equivalent.
- Eluent generator: Dionex EG50 or equivalent. Manual eluent preparation may be used.

7.2 Supplies

- Column: AS-18 (Dionex P/N 060549), or equivalent.
- Guard column: AG-18 (Dionex P/N 060551), or equivalent.
- Automatic Self-Regenerating Suppressor: ASRS 500 (Dionex P/N 082540), or equivalent.
- 5.0mL autosampler vials with filter caps: Environmental Express K1250 or equivalent.
- 0.20µm or 0.45 um pore filters (Acrodisc and Gelman): Fisher SLGN033NK, or equivalent.
- 10mL syringes (sterile polypropylene or glass): Fisher 1482316E or equivalent.
- Software package for control of instrument operations and conditions and for data evaluation and reporting: Chromeleon™, or equivalent.
- Eluent generator cartridge (if eluent generation is used): Dionex EGCIIKOH.
- Sulfate test strips: EMD 10019-1, or equivalent.
- Nitrate/Nitrite test strips: HACH 27454-25, or equivalent.
- Chloride test strips: HACH 27513-40, or equivalent.
- Serological Pipettes for Dilutions: VWR™ Part #89130 or equivalent. Volumes will vary. Each lot must be verified for accuracy. Pipettes used for dilution must be replaced daily.
- Pretreatment cartridge(s) for removal of interferences.

8.0 Reagents & Standards

All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six (6) months, or sooner if a problem is detected unless otherwise noted.

Use the designated Run log to record batch order and standards/reagents used during analysis. See ENV-SOP-MTJL-0014, *Data Handling and Reporting*.

8.1 Reagents

- Sodium Chloride ACS Grade (VWR™ 200004-248 or equivalent) – Used for the Primary Source Stock Standard in the Fast Chloride Method for Soil. Dry at 105°C for at least 30 minutes.
- Ethylenediamine (EDA) 99 %
- Ottawa Sand or equivalent.

8.2 Standards

- Stock Standards: Stock standard solutions are purchased as certified solutions (Inorganic Ventures ESC-IC-2A) with Fluoride, Nitrite (N) and Nitrate (N) at 100mg/mL and Chloride, Bromide, and Sulfate at 1000mg/mL A 50mg/mL phosphate as P may be purchased as a certified standard (RICCA 5830-16). ISO 34, if available.

  NOTE: The manufacturer provides an expiration date of the stock standards, and stock standards are stored according to manufacturer's recommendations. The Primary and Secondary Source Standards must be from the different vendors or lot numbers. Dilute working standards are prepared fresh weekly, except those that contain Nitrite, which must be prepared fresh daily or sooner if a problem is detected.

- Fast Chloride Primary Source Stock Standard
- Chlorate Stock primary standard (1000mg/L): Inorganic Ventures ICCLO31 Exp.: 1year from time of opening or the manufactures expiration whichever is sooner. ISO 34, if available.
- 100mg/L Chlorate primary intermediate standard
- Chlorite Stock Primary Standard (1000mg/L): Inorganic Ventures 1CCL021 Exp.: 1 year from time of opening or the manufactures expiration whichever is sooner. ISO 34, if available.
- 100mg/L Chlorite Primary Intermediate Standard
- Combined Working Standards
- Laboratory Control Sample (LCS) - secondary source.
- Surrogate

8.3 Formulations

- Fast Chloride Primary Source Stock Standard: Prepare a solution containing Chloride at an appropriate concentration such that the solution may be diluted to create a range of calibration standards. An appropriate example follows. Dissolve the following amounts in reagent water and dilute to 1000mL. This stock standard is stable for one month. Use of commercial standard is acceptable.

Anion	Salt	Amount (g)	Final conc. (mg/L)
Chloride	Sodium Chloride	1.6485	1000

- 100mg/L Chlorate primary intermediate standard: 10mL of 1000mg/L Chlorate primary Stock to 100mL. Exp.: 6months
- 100mg/L Chlorite Primary Intermediate Standard: 10mL of 1000mg/L Chlorite Primary Stock to 100mL. Exp.: 6months
- Combined Working Standards: Prepare serial dilutions of the Primary Source Stock Standard Solution(s) to create calibration standards over a range of concentration values

with the lowest level of the curve being at or below the reporting limit. Use volumetric flasks and Class A pipettes for all dilutions. Examples of appropriate sets of calibration standards for water and soil samples follow. Concentrations may be adjusted as needed to meet expected sample ranges or as instrument conditions change. Cal Std #1 as listed in the water calibration standards is also used for the RL 1 standard for drinking water analysis.

Water Calibration Standards:

Water Calibration Standards:

Cal Std #	Chlorate Intermediate (mL)	Anion Stock Std (mL)*	Final Volume (mL)*	Final Concentration (µg/mL) *
Zero	0.00	0.0	100	0.00
STD 1 (RL 1)	0.05	0.1	100	0.05 / 0.1 / 1.0
STD 2	0.25	0.5	100	0.25 / 0.5 / 5.0
STD 3	0.5	1.0	100	0.5 / 1.0 / 10.0
STD 4	1.0	2.5	100	1.0 / 2.5 / 25.0
STD 5	2.5	5.0	100	2.5 / 5.0 / 50.0
STD 6	10	10	100	10 / 10.0 / 100
STD 7	20	20	100	20/20/200

** Spiking volumes/concentrations and final volumes may be revised to better meet client/project/regulatory needs or to improve laboratory method performance. First value is Chlorate concentration. Second value is Nitrate, Nitrite, and Fluoride concentration. Third value is Bromide, Chloride, and Sulfate concentration.*

Soil Calibration Standards:

Cal Std #	Anion Stock Std (mL)*	Chlorate/Chlorite Intermediate (mL)	Final Volume (mL)*	Final Conc. (µg/mL) *
Zero	0.00	0.0	100	0.00
STD 1 (RL1)	0.1	0.05	100	0.05 / 0.1 / 1.0
STD 2	0.5	0.25	100	0.25 / 0.5 / 5.0
STD 3	1.0	0.5	100	0.5 / 1.0 / 10.0
STD 4	2.5	1.0	100	1.0 / 2.5 / 25.0
STD 5	5.0	2.5	100	2.5 / 5.0 / 50.0
STD 6	10	10	100	10 / 10.0 / 100
STD 7	20	20	100	20/20/200

** Spiking volumes/concentrations and final volumes may be revised to better meet client, project, and regulatory needs or to improve laboratory method performance. First value is Chlorate concentration. Second value is Nitrate, Nitrite, and Fluoride concentration. Third value is Bromide, Chloride, and Sulfate concentration.*

NOTE: Dilute working standards are prepared fresh daily.

NOTE: If EDA used as preservative, add to calibration for Chlorite/Chlorate.

NOTE: Concentrations may vary but one must be at or below the RL.

NOTE: when used, add constant amount of surrogate to all calibration standards for 1.0 ug/ml concentration.

NOTE: Limit calibration range to two (2) orders of magnitude.

Low Level Bromide Water Calibration Standards:
Make a 10mg/L Br Standard using 1mL 1000ppm ICV Standard to 100mL.

Cal Std #	Anion Stock Std (mL)*	Final Volume (mL)*	Final Concentration (µg/mL)*
Zero	0.00	100	0.00
STD 1 (RL 1)	0.5	100	0.05
STD 2	1.0	100	0.1
STD 3	2.5	100	0.25
STD 4	5.0	100	0.5
STD 5	10	100	1.0

** Spiking volumes/concentrations and final volumes may be revised to better meet client/project/regulatory needs or to improve laboratory method performance.*

- Working LCS: 20mL Anions water LCS + 5mL 100mg/L Secondary Chlorate intermediate + 5mL 100mg/L Chlorite Secondary Intermediate Standard + 0.1mL EDA to 100mL.
    - Concentrations are: F, NO2, NO3 8mg/L, Cl, Br, SO4: 40mg/L; ClO3/ ClO_2: 5mg/L
    - Solution MUST be obtained from different source than the Calibration standard solution.
    - This standard is made daily.
    - **NOTE:** concentration may vary but should be near mid-level.
- Ethylenediamine (EDA) 99 %: Dilute 10 ml EDA to 200 ml reagent water. Use 1 ml per 1000 ml sample.
- Surrogate, Dichloroacetate as Potassium salt, commercial, various. Prepare 500 ug/ml solution by dissolving 0.065 g to 100 ml reagent water. The surrogate is used for method 300.1 only.

9.0 PROCEDURE

9.1 Equipment Preparation

9.1.1 Support Equipment

Refer to laboratory SOP ENV-SOP-MTJL-0374, *Support Equipment*, or however named, for additional information on calibration requirements for support equipment that may be used in this procedure.

9.1.2 Instrument Set Up

9.1.2.1 Routine Instrument Operating Conditions

The retention time window used to make identifications must be based on measurements of actual retention time variations of standards during the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for a compound. However, the experience of the analyst should weigh heavily in chromatogram interpretations.

9.2 Calibration

9.2.1 Calibration Frequency

The calibration curve is analyzed weekly

9.2.2 Calibration Levels

Prepare at least six calibration standards (as described in 7.3). The calibration model is not forced through zero.

- If the working range exceeds the linear range of the system, a sufficient number of standards must be analyzed to allow an accurate calibration curve to be established.
- One of the standards must be representative of a concentration at or below the reporting limit.
- The other standards should correspond to the range of concentrations expected in the sample or should define the working range of the detector.

9.2.3 Calibration Sequence

Begin to inject standards starting with the lowest concentration standard and increasing in concentration.

9.2.4 Calibration Evaluation

9.2.4.1 Curve Fit

The calibration correlation coefficient, r, should be 0.995 or better or if r2 at least 0.990. Use of average RF is recommended with </= RSD of 15. When appropriate, select calibration model that minimizes residuals and intercept.

Compare these values with those obtained in the past. If they are significantly different, stop the analysis and look for the cause.

Nonlinear response can result when the separator column capacity is exceeded (overloading). Maximum column loading (all anions) should not typically exceed about 400 ug/ml

STATE NOTE: When analyzing samples in conjunction with TN Drinking Water, and South Carolina programs, the calibration model must be linear. Quadratic curve modeling is not permitted.

9.2.4.2 Relative Error

9.2.4.3 Initial Calibration Verification

Calibration is required when initial QC fails or if major changes are made to the instrument.

9.2.4.4 Continuing Calibration Verification

Daily, in lieu of an entire initial calibration, a mid-level initial calibration verification (ICV) standard may be analyzed every 10 samples for 300.0 or a low, mid, and high for 4110. Standards are analyzed prior to any QC or field samples as well as at a frequency of every ten injections (including LCS, MS, MSD, duplicates, and blanks in the count) during an analytical sequence and after the last analytical

sample. These standards must recover within ±10% of the expected concentration except for the low check which is 25 % true. If applicable, EDA must be added at 50 ug/ml. For 300.1, calculate Peak Gaussian Factor using the low point check standard.

$$PGF = 1.83 \times W / w$$

Where W = peak width at ½ height
w = peak width at 1/10 height

NOTE: For 4110, CCV criteria for low, mid, and high-level verification based on concentration as follows: low 75-125 %, mid and high ±10% true.

STATE NOTE: Recoveries of all standards, including the low check, must be ±10% of the true value for South Carolina samples.

Following each ICV/CCV, an initial (ICB) or continuing calibration blank (CCB) must be analyzed. The ICB/CCB are aliquots of the Method Blank that are analyzed to demonstrate a continued lack of analyte carryover in the instrument. The target analyte concentration in the ICB/CCB must be less than the MDL or ½ RL, as applicable. As needed, add 50 ug/ml EDA.

9.3 Sample Preparation

9.3.1 Homogenization & Subsampling

- **Water Sample preparation**
  - All samples and quality control checks must be filtered.
  - The Method Blank/ICB/CCB is prepared using approximately 5mL of reagent water and the LCS is prepared using approximately 5mL of the solution found in section 8.3.3. For each MS/MSD, take duplicate 9.5mL aliquots of a randomly selected field sample and add 500µL of Anion Stock Solution + 50uL of 1000mg/L Chlorate Primary stock standard +50uL 1000mg/L Chlorite Stock Primary.
- Soil Sample Preparation
  - Sample Prep: Place 5g of sample into a 50mL centrifuge tube and using a Class A graduated cylinder dilute to 50mL with reagent water.
  - QC Prep:
    - MS/MSD: 5g of sample + 2.5mL of Anion/Chloride Stock Solution + 5mL of Phosphate Intermediate Standard+ 0.25mL 1000mg/L Chlorate Primary Stock standard + 0.25mL 1000mg/L Chlorite Primary Stock Standard into 50mL centrifuge tube, diluted to 50mL using a Class A graduated cylinder with reagent water.
    - Method Blank: 5g of sand into a 50mL centrifuge tube, diluted to 50mL with reagent water using a Class A graduated cylinder.
    - ICV solution: 5mL of Anion/Chloride Primary Source Stock Standard + 10mL of Phosphate Primary Intermediate Standard +5mL 100mg/L Chlorate Primary Intermediate standard to 100mL reagent water.
    - LCS: Weigh 5.0g of sand and into a 50mL centrifuge tube, spike with 1.0mL of Inorganic Ventures IC Secondary Source Stock standard, 2.5mL 100mg/L Chlorate Secondary Intermediate, and 2.0mL of RICCA Orthophosphate

Secondary standard. Dilute to 50mL with reagent water, using a Class A graduated cylinder.

- NOTE: May vary but attempt to spike at mid-level.

- Place all samples on a vortex agitator for at least 10 minutes, and then centrifuge.
- Filter each sample and quality control checks through a 0.2 or 0.45 um pore Acro disc filter or equivalent.

9.4 Analysis

9.4.1 Preparation

- **Screening procedure** – Screen water samples and soil extracts using the test strips listed in section 7.2. Use according to manufacturer's instructions. Determine appropriate dilutions for each sample or extract based on the screening results.
  - Test strips must never be inserted directly into a sample undergoing preparation prior to analysis. Instead, remove sample material from the original container with an appropriate non-contaminated implement (e.g., disposable pipette, glass rod) and place on the test strip.
- Sample analysis
  - Type each sample and QC check, its position, and dilution factor (if applicable into the Chromeleon™ sample table. Make sure that the correct instrument program and quantitation method are selected.
  - Filter and/or use pretreatment cartridge(s) samples and standards into vials. Using 10 of sample, spike with 0.02 ml surrogate (required for method 300).1 for a 1 ug/ml concentration. Other volumes allowed. Adjust surrogate, as needed ~~L~~oad the vials onto the autosampler.
    NOTE – some vials may use filter frits. This is acceptable.
  - Start instrument. The Chromeleon™ software will record retention times, peak areas, and concentrations.
  - The retention time window used to make identifications must be based on measurements of actual retention time variations of standards during the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for a compound. However, the experience of the analyst should weigh heavily in chromatogram interpretations.
  - If the response for the peak exceeds the upper calibration standard, dilute the sample with an appropriate amount of reagent water and reanalyze.
  - If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, spike the sample with an appropriate amount of standard and reanalyze.
    NOTE: Nitrate and Sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases, this peak migration can produce poor resolution or misidentification.

- The laboratory has developed a "fast chloride method" for the analysis of chloride in soil samples. This method utilizes the quality control parameters that are listed in this SOP, but the flow rate of the instrument to approximately double of the regular analysis. This results in a run time that is approximately half of the regular analysis.

9.4.2 Example Analytical Sequence

Extraction Batches - defined as sets of 1 - 20 samples. Must include the following: 1 Method Blank, 1 Laboratory Control Sample (LCS), 1 Matrix Spike and Duplicate per 10 samples for 300.0/300.1 or one MS/MSD part per 20 samples for 4110/9056A.

Analytical Batches (sequences) - must include the following: Opening ICV or CCV, Calibration Blank (ICB/CCB) following each ICV or CCV. A low, mid, and high CCV for method 4110 All samples must be bracketed by acceptable QA/QC.

10.0 Data Analysis & Calculations

10.1 Qualitative Identification

Calculations are performed by PeakNet 6 (Chromeleon™) software provided by Dionex. The software calculates results using linear regression or RF using peak area vs. concentration (Sample concentration water (mg/L) = result X dilution factor). For soils ug/g = instrument result ug/ml x extract/combustate volume in ml x dilution / sample weight in g.

10.1.1 Tentatively Identified Compounds (TICs)

Not applicable

10.1.2 Manual Integration

Manual integration is sometimes necessary to correct inaccurate automated integrations but must never be used to meet QC criteria or to substitute for proper instrument maintenance and/or method set-up. To assure that all manual integrations are justified and proper all manual integrations must be performed, documented, reviewed, and approved in accordance with corporate SOP ENV-SOP-CORQ-0006, *Manual Integration.* Refer to this SOP for guidance on manual integration techniques and required procedures.

11.0 Quality Control & Method Performance

11.1 Quality Control

Prepare the following QC samples with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action(s).

QC Check	Acronym	Frequency
Method Blank	MB	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
Laboratory Control Sample	LCS	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.
LCS Duplicate	LCSD	As Required.

Matrix Spike/ Matrix Spike Duplicate	MS/MSD	One Matrix Spike and Duplicate per 10 samples for 300.0/300.1 or one MS/MSD part per 20 samples for 4110/9056A.
Surrogate	SSTD	Every field sample, standard and QC sample
Quality Control Sample	QCS	On a quarterly basis (or as needed to meet data quality needs)
Reporting Limit Verification	RLV	For EPA Method 9056A, the state of Arizona, and any batch of samples containing drinking water: Must be analyzed following each calibration

STATE NOTE: For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly, with each new initial calibration, or when there has been significant change to the instrument (column replacement, cleaning source, etc.) whichever is more frequent. The reporting limit verification can be performed by either re-injecting the low standard or by re-processing the low standard that was analyzed in the calibration curve. The reporting limit verification (RLV) must recovery within ±40% of the expected concentration. If these criteria are not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

STATE NOTE: Per West Virginia DEP compliance for EPA Method 300, all WV samples require two (2) matrix spikes per batch of 20 samples. MSDs are not required, and minimal precision can be determined and demonstrated by the LCS/LCSD. Field sample precision can be demonstrated by the parent sample duplicate. For all other state regulatory programs, the performance of the MS/MSD meets the requirements of the published method.

11.2 Instrument QC

Perform the following checks to verify instrument performance. Refer to Appendix B for acceptance criteria and required corrective action.

Instrument Check	Acronym	Frequency
Initial Calibration Verification	ICV	Immediately following the Calibration
Initial Calibration Blank	ICB	Immediately following the ICV
Continuing Calibration Verification	CCV	After every 10 samples and at the end of the analytical sequence
Continuing Calibration Blank	CCB	After every 10 samples and at the end of the analytical sequence
Linear Calibration Range	LCR	LCR studies must be established at least semi-annually or when instrumentation change occurs

11.3 Method Performance

11.3.1 Method Validation

Refer to corporate SOP ENV-SOP-CORQ-0011 for general requirements and procedures for method validation.

Establish detection limits (DL) and limits of quantitation (LOQ) at initial method set up and verify the DL and LOQ on an on-going basis thereafter. Refer to corporate policy and/or SOP for DL and LOQ requirements and procedures.

12.0 Data Review & Corrective Action

12.1 Data Review

The data review process of Pace® Analytical Services includes a series of checks performed at different stages of the process by different people to ensure that SOPs were followed, the analytical record is complete, and properly documented, QC criteria were met, proper corrective actions were taken for QC failure and other nonconformance(s), and test results are reported with proper qualification, when necessary.

The review and checks that are performed by the employee performing the task is called primary review.

All data and test results are also peer reviewed.

This process, known as secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented, and approved in accordance with the Pace® Analytical Services SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

Lastly, a third-level review, called a completeness check, is performed by reporting or project management staff to verify the test report is complete.

Refer to laboratory SOP ENV-SOP-MTJL-0014, *Data Handling and Reporting* and ENV-SOP-MTJL-0038, *Data Review* for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is required when QC or sample results are not within acceptance criteria.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted

and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 Pollution Prevention & Waste Management

Pace® proactively seeks ways to minimize waste generated during work processes. Some examples of pollution prevention include but are not limited to reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practices comply with all applicable federal and state laws and regulations. Excess reagents, samples, and method process wastes are characterized and disposed of in an acceptable manner in accordance with the Pace® Chemical Hygiene Plan / Safety Manual. Refer to this manual for these procedures.

14.0 Modifications

Modifications have been made to the Eluent solution per the instrument manufacturer's conditions for the column utilized and is allowed by method.

When applicable, comparability and/or equivalency studies necessary to validate the modification as required per corporate SOP ENV-SOP-CORQ-0011 are retained by local quality personnel for historical reference.

15.0Responsibilities

- All employees of Pace® Analytical Services that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement (R&A) in their training file for the version(s) of the SOP that were in effect during the time the employee performed the activity.
- Local quality personnel are responsible for tracking the currency of the R&A on this SOP for employees at the locations they are assigned to and for notifying the General Manager (GM), however named, when R&A are overdue or outstanding. The GM and the employee's direct supervisor are responsible for ensuring the employee completes the R&A assignments as required.
- The supervisors and managers of Pace® Analytical Services, however named, are responsible for training employees on the procedures in this SOP, implementing the SOP in the work area, and monitoring on-going adherence to the SOP the work area(s) they oversee.
- All employees of Pace® Analytical Services are responsible for following the procedures in this SOP. Unauthorized deviations or departures from this SOP are not allowed except with documented approval from the local Quality Manager and only when those deviations do not violate the Pace® Code of Ethics or Professional Conduct (COR-POL-0004) or associated policy and procedure(s). Hand-edits or manual change to the SOP are not permitted. If a change is desired or necessary, Pace® employees must follow the procedures for document revision specified in corporate SOPs ENV-SOP-CORQ-0015 *Document Management* and ENV-SOP-CORQ-0016 *SOP for Creation of SOP and SWI*.
- Local quality personnel are responsible for monitoring conformity to this SOP during routine internal audits of work areas that utilize this SOP and for communicating gaps and deviations found during monitoring to the work area supervisor, who is responsible for correction of the situation.

16.0 ATTACHMENTS

- Appendix A: Routine Analyte List and LOQ
- Appendix B: QC Summary & Corrective Action Table

17.0 REFERENCES

- ENV-SOP-CORQ-0006, *Manual Integration*, current version.
- ENV-SOP-CORQ-0011, *Method Validation*, current version.
- ENV-SOP-CORQ-0015, *Document Management*, current version.
- ENV-SOP-CORQ-0016, *SOP for SOP and SWI,* current version.
- ENV-TMP-CORQ-0007, *Quality Manual Template*, current version.
- COR-POL-0004, *Code of Ethics and Professional Conduct*, current version.
- COR-MAN-001, *Pace® Safety Manual*, current version.
- Installation Instructions and Troubleshooting Guide for the IonPac AS14 Guard Column and IonPac AS14 Analytical Column, DIONEX Corporation.
- U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, Method 300.1, Revision 1.0, 1997.
- U.S. Environmental Protection Agency, Manual for the Certification of Laboratories Analyzing Drinking Water, January 2005
- Standard Methods for Examination of Water and Wastewater, 23rd Ed. or online version.
- US EPA, SW846, Update IV, 9056A 2/07.

18.0 REVISION HISTORY

Authorship

Primary Author[1]	Job Title	Date Complete
Mike Dunn	Technical Supervisor-Wetchemistry	7/26/2022

[1]The primary author is the individual / role responsible for the content of this SOP. Send questions or suggestions for content to the primary author. See the Quality Manager for questions or concerns related to implementation of this SOP.

Revisions Made from Prior Version

Section	Description of Change
8.3	Updated calibration table

Document Succession: This version replaces the following documents:

Document Number & Version	Document Title	Effective Date:
ENV-SOP-MTJL-0155 V02	Inorganic Anions by IC	7/27/22

Appendix A: Target Analyte List and LOQ

Table 1: Standard Analyte List and LOQ

Analyte	CAS #	LOQ[1] Matrix (mg/L)	LOQ[1] Matrix (mg/kg)
Bromide	24959-67-9	1	10
Chloride	16887-00-6	1	10
Fluoride	16984-48-8	0.1	1
Nitrate (N)	14797-55-8	0.1	1
Nitrite (N)	14797-65-0	0.1	1
Orthophosphate	14265-44-2	0.1	1
Sulfate	14808-79-8	5	50
Chlorate	14866-68-3	0.05	--
Chlorite	14998-27-7	0.05	--

[1] *Values as of effective date of this SOP. LOQ are subject to change, contact quality personnel for most current information.*

Appendix B: QC Summary and Corrective Action Table

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICAL	At instrument set up, Weekly	Must meet one of curve fit options presented in Section 9.0. For any curve fit other than Average RF (RSD), curve must also pass RSE test at the low and midpoint calibration standard.	Identify and correct source of problem, repeat	None. Do not proceed with analysis
ICV	After Each ICAL	All analytes must be within ± 10% of the true value. (%R)	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
CCV	Daily, before sample analysis, after every 10, and at end of analytical window.	Opening CCV: All analytes within ± 10 %D Ending CCV: **NOTE:** For 4110, CCV criteria for low, mid, and high-level verification based on concentration as follows: low 75-125 %, mid and high ±10% true. **STATE NOTE:** Recoveries of all standards, including the low check, must be ±10% of the true value for South Carolina samples.	See Section 9.2.4.4 for required corrective actions based on circumstance.	Qualify analytes with CCV out of of criteria.
Surrogate	Every field sample, standard and QC sample	Surrogate for 300.1 – recovery is 90 – 115 %. Retention time shift less than 2 % for ICAL and less than 5 % for samples.	If failure occurs, re-analyze the sample, standard, and QC samples once. If failure(s) persists, determine the cause of the failure and re-prep. The cause of the failure could be improperly prepared standards, samples, or QC. If failure(s) still occur, dilute the sample and qualified data.	Surrogate failure must be flag as J1.
Method Blank	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.	The Method Blank/ICB/CCB concentration must be less than the Method Detection Limit or ½ RL, as needed	If the Method Blank fails, rinse and re-analyze once. If the contamination still occurs, corrective actions can include instrument maintenance, reviewing data for errors and	General guidelines for qualifying sample results with regard to method blank

			review of the calibration curve. A Method Blank must meet the acceptance criteria prior to the analysis of any field samples. Re-analyze all sample analyses not bracketed by acceptable ICB/CCBs, except as noted in section 11.11. If the contamination still occurs after maintenance has been performed, the batch must be re-prepared and re-analyzed.	quality are as follows: If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required. No qualification is necessary when an analyte is detected in the method blank but not in the associated samples. If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required. If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank. If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for

				further instruction.
LCS	1 per batch of 20 or fewer samples. If batch exceeds 20 samples, every 20 samples.	The recovery of the LCS must be within +10% for 300.0/300.1 and at 15% for other methods.	If failure occurs, re-analyze the LCS once. If failure(s) persists, determine the cause of the failure. The cause of the failure could be improperly prepared standards, the instrument or one of its components may require maintenance or replacement, the instrument needs to be re-calibrated, the reagents may need to be re-prepped, etc. (Check instrument manual for additional information). When the cause of the failure has been corrected, re-calibrate or re-verify the calibration and begin the analysis again. If the failure still occurs after maintenance has been performed the batch must be re-prepared and re-analyzed. A passing LCS must be analyzed with each analytical batch; however, if LCS is high and samples are ND, they may be reported.	N/A
MS/MSD	One Matrix Spike and Duplicate per 10 samples for 300.0/300.1 or one MS/MSD part per 20 samples for 4110/9056A. **STATE NOTE**: Per West Virginia DEP compliance for EPA Method 300, all WV samples require two (2) matrix spikes per batch of 20 samples.	Recovery must be within ±10% of the true value for each MS and MSD and the relative percent difference (RPD) must not exceed <20%.	N/A	Matrix Spike failures must be flagged with a J5 (high) or J6 (low). If there is an RPD failure, report the result with a J3 qualifier. Matrix spikes that are over the calibration range shall be qualified with an E.
Reporting Limit Verification	For EPA Method 9056A, the state of Arizona, and any batch of samples containing drinking water: Must be analyzed following each calibration	RLV must recover within +50% of the expected value. *State Note*: The reporting limit verification (RLV) must recovery within +40% of the expected concentration for all samples from Minnesota State Note: The reporting limit verification (RLV) must recovery within +10% of the expected concentration for all	State Note: If these criteria are not met, the RLV may be re-analyzed once, instrument maintenance can be performed, a higher concentration standard can be injected, or a new calibration curve must be generated. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.	N/A

		samples from South Carolina		
Linear Calibration Range	LCR studies must be established at least semi-annually or when instrumentation change occurs	The recovery must be within ± 10% for 300. 300.1 should cover the expected concentration range of the field samples and should not extend over more than 2 orders of magnitude in concentration.	If failure occurs, re-analyze the calibration. If failure(s) persists, determine the cause of the failure, and conduct instrument maintenance.	N/A
Quality Control Sample	On a quarterly basis (or as needed to meet data quality needs)	The recovery of the LCS must be within +10% for 300.0/300.1 and at 15% for other methods.	If failure occurs, re-analyze the LCS once. If failure(s) persists, determine the cause of the failure. The cause of the failure could be improperly prepared standards, the instrument or one of its components may require maintenance or replacement, the instrument needs to be re-calibrated, the reagents may need to be re-prepped, etc. (Check instrument manual for additional information). When the cause of the failure has been corrected, re-calibrate or re-verify the calibration and begin the analysis again. If the failure still occurs after maintenance has been performed the batch must be re-prepared and re-analyzed. A passing LCS must be analyzed with each analytical batch; however, if LCS is high and samples are ND, they may be reported.	N/A
Sample Duplicate	Every 10 samples	RPD < 20%	N/A	If the RPD is >20%, the sample and duplicate must be reported with a "J3" qualifier. If a sample duplicate is above the acceptable range for the RPD and the sample concentration is <5X the RL, then the value can be flagged with a "P1" qualifier indicating that the RPD calculation is not applicable at that concentration.

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Pace ANALYTICAL SERVICES	**ENV-SOP-MTJL-0197 v04_Carbon Dioxide by Calculation**	
	Effective Date: 09/06/2022	COPYRIGHT© 2019, 2021, 2022 Pace®

Management Approval:
John Davis Approved on 9/1/2022 12:55:21 PM
Kayla Coble Approved on 9/2/2022 2:04:05 PM
Robert Johnson Approved on 9/6/2022 8:59:27 AM
Rebecca King Approved on 9/6/2022 1:36:46 PM

1.0 Scope and Application

This procedure is applicable to drinking, surface, and saline waters, domestic, and industrial waste. The Reporting Limit (RL) is based on a final calculated Alkalinity result. See LIMS for active limits.

2.0 Summary of Method

When Alkalinity is due almost entirely to hydroxides, carbonates, or bicarbonates, total and free CO2 can be calculated from the sample pH and Total Alkalinity. The sample is analyzed for Alkalinity (ENV-SOP-MTJL-0138, *Total Alkalinity, Manual and Automated (Titration)(SM 2320B).*) and pH (ENV-SOP-MTJL-0163, *pH, Manual or Automated (EPA Methods 150.1, 9040C and 9045D; SM 4500H+ B) Including Corrosivity for Solids and Liquids Using These Methods.*).

3.0 Interferences

Carbon Dioxide may be lost during shipment and storage. Fill sample containers completely with no headspace to minimize this loss. Samples containing more than approximately 500mg/L Total Dissolved Solids may bias the results.

4.0 Definitions

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 Health And Safety

Contact your supervisor or local safety coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure

The following sections provide general health and safety information about chemicals and materials that may be present in the laboratory.

- The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.
- The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (EHS) policies and procedures specified in this SOP and in the Pace® Chemical Hygiene / Safety Manual (COR-MAN-0001)
- Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

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- Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. For procedures that require use of acids, use acids in a fume hood whenever possible with PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. For procedures that that emit large volumes of solvents (evaporation/concentration processes), these activities must be performed in a fume hood or apparatus that reduces exposure.

6.0 Sample Collection, Preservation, Holding Time & Storage

The laboratory provides containers for the collection of samples upon client request. Refer to laboratory SOP ENV-SOP-MTJL-0064, Sample Shipping for procedures related to preparation of bottle kits for the test method(s) associated with this SOP.

The measurements of pH and alkalinity should be made at the time of sampling in the field. When that is the case, Carbon Dioxide (CO2) can be calculated. However, if the sample is shipped to the lab for analysis, the result should be flagged with a T8 (sample received out of holding time of the associated pH test). See the determinative procedures for pH and alkalinity for more information regarding sample collection, containers, handling, and storage.

After analysis, samples are retained as stated in the Pace® standard terms and conditions, unless otherwise specified in the analytical services contract. Samples are then disposed of in accordance with Federal, State, and Local regulations.

7.0 Equipment & Supplies

7.1 Equipment and Supplies

- All equipment required by ENV-SOP-MTJL-0138, *Total Alkalinity, Manual and Automated (Titration) (SM 2320B).*
- All equipment required by ENV-SOP-MTJL-0163, *pH, Manual or Automated (EPA Methods 150.1, 9040C and 9045D; SM 4500H+ B) Including Corrosivity for Solids and Liquids Using These Methods.*

8.0 Reagents & Standards

All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standard Logger–Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital archive system. All spiking solutions and surrogate standard solutions should be replaced at least every 6 months or sooner if a problem is detected unless otherwise noted.

8.1 Reagents and Standards

- All reagents and standards as required by ENV-SOP-MTJL-0138, Total Alkalinity, Manual and Automated (Titration) (SM 2320B).
- All reagents and standards as required by ENV-SOP-MTJL-0163, *pH, Manual or Automated (EPA Methods 150.1, 9040C and 9045D; SM 4500H+ B) Including Corrosivity for Solids and Liquids Using These Methods.*

8.2 Formulations

Not applicable

9.0 PROCEDURE

Determine Total Alkalinity using ENV-SOP-MTJL-0138, *Total Alkalinity, Manual and Automated (Titration) (SM 2320B).*

Determine pH using the ENV-SOP-MTJL-0163, *pH, Manual or Automated (EPA Methods 150.1, 9040C and 9045D; SM 4500H+ B) Including Corrosivity for Solids and Liquids Using These Methods.*

10.0 DATA ANALYSIS & CALCULATIONS

10.1 Qualitative Identification

Calculate Bicarbonate Alkalinity (B) from the Total Alkalinity determined using the following equation:

$$B \text{ as mg } CaCO_3/L = \frac{T - 5.0 * 10^{(pH-10)}}{1 + 0.94 * 10^{(pH-10)}}$$

where: T = Total Alkalinity in mg $CaCO_3$/L
pH = pH value for sample in field, if available.

Calculate Carbonate Alkalinity (CO_3) i.e., (C) using Bicarbonate Alkalinity (B):

$$CO_3^{2-} \text{ as mg } CaCO_3/L = 0.94 \text{ X B X } 10^{(pH-10)}$$

Calculate free carbon dioxide (A) as follows:

$$\text{mg free } CO_2/L = 2.0 \text{ X B X } 10^{(6-pH)}$$

Calculate total carbon dioxide as follows:

$$\text{mg total } CO_2/L = A + 0.44 (2B + C)$$

Where: A = mg free CO_2/L
B = Bicarbonate Alkalinity
C = Carbonate Alkalinity

NOTE: forms/relationships may be determined by instrument software.

10.1.1 Manual Integration

Manual integration is sometimes necessary to correct inaccurate automated integrations but must never be used to meet QC criteria or to substitute for proper instrument maintenance and/or method set-up. To assure that all manual integrations are justified and proper all manual integrations must be performed, documented, reviewed, and approved in accordance with corporate SOP ENV-SOP-CORQ-0006, *Manual Integration.* Refer to this SOP for guidance on manual integration techniques and required procedures.

11.0 QUALITY CONTROL & METHOD PERFORMANCE

All analysts must meet the qualifications specified in ENV-SOP-MTJL-0015, *Technical Training and Personnel Qualifications* before approval to perform this method. Analysts must complete an initial demonstration of proficiency using the parent tests before being approved to perform these methods. Continuing proficiency must be demonstrated using proficiency testing, laboratory control sample analysis and/or MDL studies for parent analytical tests. Method performance is assessed per analyst. Updated method performance records are filed and stored in a central location within the department. Use the designated Run log batch order and standards/reagents used during analysis. See ENV-SOP-MTJL-0014, *Data Handling and Reporting.* Batches are defined as sets of 1-20 samples. Batch analysis must include the quality control samples required by the parent tests: alkalinity and pH. All batch information must be maintained in the preparation documentation assigned to the department.

NOTE: Drinking water samples for compliance may not be analyzed using this procedure unless done at time of sampling.

12.0 Data Review & Corrective Action

12.1 Data Review

The data review process of Pace® Analytical Services includes a series of checks performed at different stages of the process by different people to ensure that SOPs were followed, the analytical record is complete, and properly documented, QC criteria were met, proper corrective actions were taken for QC failure and other nonconformance(s), and test results are reported with proper qualification, when necessary.

The review and checks that are performed by the employee performing the task is called primary review.

All data and test results are also peer reviewed.

This process, known as secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented, and approved in accordance with the Pace® Analytical Services SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

Lastly, a third-level review, called a completeness check, is performed by reporting or project management staff to verify the test report is complete.

Refer to laboratory SOP ENV-SOP-MTJL-0014, Data Handling and ENV-SOP-MTJL-0038, Data Review for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Refer to Corrective Action as required by ENV-SOP-MTJL-0138, *Total Alkalinity, Manual and Automated (Titration) (SM 2320B)* and ENV-SOP-MTJL-0163, *pH, Manual or Automated (EPA Methods 150.1, 9040C and 9045D; SM 4500H+ B) Including Corrosivity for Solids and Liquids Using These Methods.*

13.0 Pollution Prevention & Waste Management

Pace® proactively seeks ways to minimize waste generated during work processes. Some examples of pollution prevention include but are not limited to reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practices comply with all applicable federal and state laws and regulations. Excess reagents, samples, and method process wastes are characterized and disposed of in an acceptable manner in accordance with the Pace® Chemical Hygiene Plan / Safety Manual. Refer to this manual for these procedures.

14.0 MODIFICATIONS

The procedures in this SOP have not been modified from the reference test method(s) cited.

15.0 RESPONSIBILITIES

- All employees of Pace® Analytical Services that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement (R&A) in their training file for the version(s) of the SOP that were in effect during the time the employee performed the activity.
- Local quality personnel are responsible for tracking the currency of the R&A on this SOP for employees at the locations they are assigned to and for notifying the General Manager (GM), however named, when R&A are overdue or outstanding. The GM and the employee's direct supervisor are responsible for ensuring the employee completes the R&A assignments as required.
- The supervisors and managers of Pace® Analytical Services, however named, are responsible for training employees on the procedures in this SOP, implementing the SOP in the work area, and monitoring on-going adherence to the SOP the work area(s) they oversee.
- All employees of Pace® Analytical Services are responsible for following the procedures in this SOP. Unauthorized deviations or departures from this SOP are not allowed except with documented approval from the local Quality Manager and only when those deviations do not violate the Pace® Code of Ethics or Professional Conduct (COR-POL-0004) or associated policy and procedure(s). Hand-edits or manual change to the SOP are not permitted. If a change is desired or necessary, Pace® employees must follow the procedures for document revision specified in corporate SOPs ENV-SOP-CORQ-0015 *Document Management* and ENV-SOP-CORQ-0016 *SOP for Creation of SOP and SWI*.
- Local quality personnel are responsible for monitoring conformity to this SOP during routine internal audits of work areas that utilize this SOP and for communicating gaps and deviations found during monitoring to the work area supervisor, who is responsible for correction of the situation.

16.0 ATTACHMENTS

Not applicable

17.0 REFERENCES

- ENV-SOP-CORQ-0006, *Manual Integration*, current version.
- ENV-SOP-CORQ-0011, *Method Validation*, current version.
- ENV-SOP-CORQ-0015, *Document Management*, current version.

- ENV-SOP-CORQ-0016, *SOP for SOP and SWI,* current version.
- ENV-TMP-CORQ-0007, *Quality Manual Template*, current version.
- COR-POL-0004, *Code of Ethics and Professional Conduct*, current version.
- COR-MAN-001, *Pace® Safety Manual*, current version.
- *Carbon Dioxide and Forms of Alkalinity by Calculation*, Standard Methods 4500-CO_2 D-2011, 23rd Edition or on-line version.

18.0 REVISION HISTORY

Authorship

Primary Author[1]	Job Title	Date Complete
Mike Dunn; Kayla Coble	Technical Specialist; Wet Chemistry Supervisor	2/12/2021

[1]The primary author is the individual / role responsible for the content of this SOP. Send questions or suggestions for content to the primary author. See the Quality Manager for questions or concerns related to implementation of this SOP.

Revisions Made from Prior Version

Section	Description of Change
All	Formatted to corporate template

Document Succession: This version replaces the following documents:

Document Number & Version	Document Title	Effective Date:
ENV-SOP-MTJL-0197 v04	CO2 Calculation	8/12/2022

Groundwater Sampling

A8.1 Purpose and Applicability

This procedure conforms to the EPA Quality Assurance Requirements and describes methods for purging and sampling a groundwater monitoring well to ensure that the sample collected is representative of the formation groundwater. The procedure follows EPA guidance detailed in *Region 4 Science and Ecosystem Support Division (SESD) Operating Procedure (OP) for Groundwater Sampling, SESDPROC-301-R3*. (EPA, 2013).

A8.2 Definitions

Bailer: A hollow tube constructed of stainless steel or Teflon® that is used to collect groundwater samples. A dedicated bailer remains in the well casing.

A8.3 Procedures

A8.3.1 Purging

The following equipment is required for well purging:

- Bailer or pump. The device used depends upon aquifer properties, individual well construction, well yield, and data quality objectives (DQOs).
- Water level measuring device.
- Tape measuring device.
- pH, specific conductance, turbidity, and temperature measuring device.

Well purging is performed as follows:

- For the well to be purged/sampled, the following information is obtained and recorded in the groundwater purging/sampling data sheet or the field log book: date, field conditions, well location, well ID, well diameter, groundwater elevation, total well depth, screened interval, water quality field measurements (pH, specific conductance, turbidity, and temperature), and the method for disposal of purged water.
- Field instruments are calibrated prior to use and according to manufacturers' instructions.
- Prior to opening the well, plastic sheeting is placed on the ground surrounding the well head to prevent contamination by sample spillage;
- The well is unlocked and opened and an FID/PID reading is immediately taken.
- The water level and the total depth of the well are measured.
- The volume in gallons of water in the well casing or sections of telescoping well casing is calculated as follows:

  $(\Pi\ r^2h)\ 7.48$ = gallons
  where: $\Pi = 3.142$
  r = Radius of the well pipe in feet

h = Linear feet of water in well
7.48 = Gallons per cubic foot of water

The volume of water in typical well casings may be calculated as follows:

gallons/feet x __ (linear feet of water) = total gallons

where:
2-inch well = 0.163 gallons/foot
3-inch well = 0.367 gallons/foot
4-inch well = 0.653 gallons/foot
5-inch well = 1.02 gallons/foot
6-inch well = 1.469 gallons/foot
7-inch well = 1.999 gallons/foot
8-inch well = 2.611 gallons/foot
10-inch well = 4.28 gallons/foot
12-inch well = 5.87 gallons/foot

- Purging the well will begin by lowering the decontaminated purging apparatus (pump or bailer) to the standing water column so that the water will be pulled through the casing and the entire static volume will be removed. A bailer is used when the well does not yield sufficient water for pumping; otherwise, a pump is preferred. For low- flow sampling techniques, see section A8.3.4.
- The initial pH, specific conductance, turbidity, and temperature of water are measured and recorded in the field logbook along with the odor, color, clarity, silt concentrations and general water condition. During purging, field parameters are measured at least once during each well volume (more often is preferable). Record changes in the physical condition of the monitoring wells that could affect the well integrity.
- For purging to be complete, a total of at least 3-5 volumes of groundwater should be removed from the well, and the field parameters must stabilize. The amount of purged fluid will be measured by filling a graduated bucket or by using a stopwatch and noting the flow rate of the pump versus elapsed time. Field parameter stabilization is as follows: pH measurements ± 0.1 units, temperature measurements ±1°C, specific conductance measurements ± 10%, and ± 10% for turbidity).
- Wells with little or no recharge will be purged to near dryness, and the well is allowed to recover before sampling.
- When using a pump, prior to the completion of purging activities, the pump will be brought to the water surface to ensure complete removal of stagnant water.
- Purge water will be placed in a storage tank and disposed of as IDW

Wells will be sampled immediately after purging, if possible, but generally no later than 6 hours after purging. Wells that recharge slowly will be purged dry and allowed to recharge before sampling. If excessive time (greater than 10 hours) is required for the slow recharging wells to recharge, it will be documented in the field log.

A8.3.2 Sample Collection

Following are the general procedures for groundwater sampling along with methods for utilizing specific sampling devices and techniques.

A8.3.2.1 General

- With the exception of low-flow sampling (Section A8.3.4) and open borehole sampling, before samples are taken, the well is purged as described in Section A8.3.1.

- Sampling equipment will be cleaned and decontaminated prior to the commencement of sampling activities. A new pair of disposable gloves will be worn at each location by sampling personnel.
- Prelabeled, precleaned, sample bottles with preservative added, are used to contain the groundwater samples. VOA samples will be collected first followed by other organic analyses. Inorganic analyses are collected last accept in the case where the influences of turbidity on metals concentrations is a concern. In this case metals samples will be collected immediately following the volatile organics.
- As the sample is taken, the sample container is tilted slightly allowing the water to run down the inside of the sample bottle with a minimum of splashing
- Adequate space is left in the bottle to allow for expansion, except for volatile organic analysis (VOA) vials, which are filled to overflowing and capped. VOAs vials are checked for air bubbles and if detected, more sample is carefully added to the vial (care must be taken to minimize the loss of preservative).
- Samples are placed in appropriate containers, and packed with ice in coolers immediately after the sample is collected.

Measure pH, conductivity, temperature, and turbidity after sample bottles have been filled and record the measurements in logbook.

A8.3.2.2 Bailer

A decontaminated Teflon® bailer can be used to remove groundwater samples from a well as follows:

- A decontaminated and properly secured, bailer is lowered to the sampling interval from which the sample is to be collected.
- The bailer is allowed to fill with a minimum of surface disturbance to prevent sample water aeration. When the bailer is raised, the bailer cord must not be allowed to touch the ground.
- The sample is slowly poured from the bailer and the bottle is tilted slightly allowing the water to runs down the inside of the sample bottle with a minimum of splashing
- If the bailer is dedicated, it is returned to the well and the well is capped and locked. Non-dedicated samplers are cleaned and decontaminated after use.

A8.3.3 Purging/Sampling Using a Small Diameter, Electric Submersible Pump

Small Diameter Electric Submersible Pumps includes a range of small diameter, variable speed pumps capable of pumping rates ranging from 0.5 ml./min. to in excess of 9 gallons per minute. The power source for these pumps can be provided directly from an automobile battery or from a generator. While small diameter pumps are generally light-weight and easily handled by one person when lowering into a well, two people are generally needed when removing the pump, one to pull and another to reel the hose and power lead. Groundwater monitoring wells can be purged utilizing a decontaminated pump and clean flexible tubing as follows:

- Slowly lower the pump to the middle of the screened interval. This minimizes excessive mixing of the stagnant water in the casing above the screen with the screened interval zone water, and to minimize re-suspension of solids, which will have collected at the bottom of the well (EPA, 1996).
- Follow the manufacturer's procedures, and begin pump-purging the monitoring well.
- If the recovery rate of the well is faster than the pump rate, the pump may be left hanging at the initial level. If the pump rate exceeds the recovery rate, the pump must be lowered to accommodate the drawdown, or the pump rate can be decreased.

- Once 3-5 well volumes have been removed from the well, and the field parameters have stabilized, remove the pump from the well, and sample utilizing a Teflon® bailer.

A8.3.4 Purging/Sampling Using Modified Low-Flow Techniques

Low-flow techniques are utilized to obtain a more representative sample from the aquifer formation. Jacobs is currently using this procedure for its quarterly and semiannual groundwater sample collection at Sites B-10 and B-90. In general, the advantages of low-flow purging include (EPA, 1996):

- Samples which are representative of the mobile load of contaminants present (dissolved and colloid-associated);
- Minimal disturbance of the sampling point thereby minimizing sampling artifacts (i.e. less turbidity);
- Less operator variability, greater operator control;
- Reduced stress on the formation (minimal drawdown);
- Less mixing of stagnant casing water with formation water;
- Reduce the need for filtration and, therefore, less time required for sampling;
- Smaller purging volume which decreases IDW disposal costs;
- Better sample consistency; reduced artificial sample variability.

The pumps selected for perform low-flow sampling, should be capable of producing purge rates sufficient to allow for the modified low-flow sampling technique. Pumps, which meet these requirements include but are not limited to, bladder-type pumps (provided that reagent grade nitrogen is used for bladder inflation) and the Grundfos Redi-Flow2 pump.

Following are the procedures for modified low-flow groundwater sampling. These procedures include adaptations from EPA's paper entitled "Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures" (EPA, 1996):

- Slowly lower the decontaminated pump to the middle of the screened interval. This is to minimize excessive mixing of the stagnant water in the casing above the screen with the screened interval zone water, and to minimize re-suspension of solids, which will have collected at the bottom of the well.
- Once the pump is positioned in the well, an airtight flow-through cell (equipped with a YSI or Horiba-type water quality meter) is plumbed to the water discharge line.
- Lower a decontaminated water level gauge into the well to monitor the water table.
- Once purging is initiated, water level measurements should be continuously monitored, and pumping rates adjusted as necessary (e.g., 0.1 - 0.3 L/min) to maintain minimal drawdown. Modified low-flow techniques should cause less than three feet of drawdown during purging.
- While purging, the groundwater field parameters (including water level) should be continuously monitored every 3-5 minutes until all parameters have stabilized for 3 consecutive readings.
- Stabilization for each parameter is defined as follows: ±0.1 for pH, ±3% for conductivity, ±10 mv for redox potential, ±10% for turbidity, ±10% for dissolved oxygen (DO), and ± 3ft. for drawdown.

Once field parameters have stabilized for 3 consecutive readings, samples may be taken. The same device used for purging should be used for sampling (remove flow-through cell).

Water-Level Measurements

A9.1 Purpose and Scope

The purpose of this procedure is to provide a guideline for the measurement of the depth to groundwater in piezometers and monitoring wells, even where a second phase of floating liquid (e.g., gasoline) is encountered, and on staff gages in surface-water bodies. This SOP includes guidelines for discrete measurements of static water levels and does not cover the use of continuously recording loggers (see SOP Use of Data Loggers and Pressure Transducers).

A9.2 Equipment and Materials

- Electronic water-level meter (Solinst® or equivalent) with a minimum 100-foot tape; the tape should have graduations in increments of 0.01 feet or less
- Interface probe (Solinst® Model 122 Interface Meter or equivalent)

A9.3 Procedures and Guidelines

Verify that the unit is turned on and functioning properly. Slowly lower the probe on its cable into the piezometer or well until the probe just contacts the water surface; the unit will respond with a tone or light signal. Note the depth from a reference point indicated on the piezometer or well riser. Typically this is the top of the PVC casing. If no reference is clearly visible, measure the depth to water from the northern edge of the PVC casing. If access to the top of the PVC casing is difficult, sight across the top of the locking casing adjacent to the measuring point, recording the position of the cable when the probe is at the water surface.

Measure the distance from this point to the closest interval marker on the tape, and record the water level reading in the logbook. Water levels will be measured to the nearest 0.01-foot. Also when specified in the project plans, measure and record the depth of the piezometer or well. The depth of the piezometer or well may be measured using the water-level probe with the instrument turned off.

Free product light or dense nonaqueous phase liquid may be present in the piezometer or well. If the presence of free product is suspected, the thickness of the product should be determined using appropriate equipment (e.g., Solinst® Model 122 Interface Meter). The depth to water also is determined with this equipment and the water-level meter should not be used in the piezometer or well as long as product is present. Typically, a constant sound is emitted from the device when free product is encountered and an alternating on/off beep sound is emitted when water is encountered.

The apparent elevation of the water level in the well or piezometer is determined by measuring both the apparent depth to water and the thickness of free product. The corrected water-level elevation is calculated by the following equation:

$$WL_c = Wl_a + (\text{Free-product thickness} \times 0.80)$$

Where WL_c = Corrected water-level elevation

Wl_a = Apparent water-level elevation

0.80 = Typical value for the density of petroleum hydrocarbon products.

If free product is detected on the surface of the water in the piezometer or well, the value of sampling should be reconsidered because of the potential for contaminating the sampling equipment.

Staff gages may be installed in some surface-water bodies. These facilities typically are constructed by attaching a calibrated, marked staff gage to a wood or metal post, driving the post into the bottom of the surface-water body, and surveying the elevation of the top of the post to a resolution of 0.01-foot. The elevation of the water in the surface-water body then can be determined by reading off the distance the water level is from the top of the post. A shield or other protection may be needed to calm the fluctuations in water level if the gage is installed at a location exposed to wind or wave.

A9.4 Attachments

None.

A9.5 Key Checks

- Before each use, verify that the battery is charged by pressing the test button on the water-level meter.
- Verify that the unit is operating correctly by testing the probe in distilled or de-ionized water. Leave the unit turned off when not in use.

STANDARD OPERATING PROCEDURE A10

Use and Calibration of Field Instruments

A10.1 Purpose and Applicability

This procedure conforms to the applicable EPA QA requirements and the QAPP, and it establishes standard methodologies for the use, calibration, and maintenance of field instruments. The procedure applies to all field investigations.

A10.2 Definitions

None.

A10.3 Procedure

Field instruments will be used for the collection of field data and measurement of various conditions observed during the site investigations. In general, take the following steps when using field instruments:

- Before field use, remove the instrument from its container and assemble and clean it according to the manufacturer's instructions.
- Before commencement of field activities each day, calibrate the instrument according to the manufacturer's instructions. Record the instrument's ID and serial number, along with the calibration process/results, in the field logbook and/or calibration logbook.
- Use the instrument to make the appropriate physical/chemical measurements and clean/decontaminate the instrument, if necessary, after each measurement.
- If erroneous measurements are observed or if changes in environmental conditions warrant recalibration, recalibrate the instrument as specified by the manufacturer. Record the recalibration information in the field logbook and/or the calibration logbook.
- At the end of each day, clean and decontaminate the instrument then return it to the storage location. Recharge instrument as necessary.
- Perform factory maintenance and calibration at the intervals specified by the manufacturer. Have repairs, maintenance, and calibration performed by trained individuals according to the manufacturer's requirements. Record repairs, maintenance, and calibration in the field logbook or instrument calibration/maintenance logbook.

Decontamination of Equipment

A11.1 Purpose and Applicability

To provide general guidelines for the decontamination of personnel, sampling equipment, and monitoring equipment used in potentially contaminated environments.

A11.2 Scope

This is a general description of decontamination procedures.

A11.3 Equipment and Materials

- Demonstrated analyte-free, deionized ("DI") water (specifically, ASTM Type II water or lab-grade DI water)
- Potable water; must be from a municipal water supplier, otherwise an analysis must be run for appropriate volatile and semivolatile organic compounds and inorganic chemicals (e.g., Target Compound List and Target Analyte List chemicals)
- 2.5% (W/W) Liquinox® and water solution
- Concentrated (V/V) pesticide grade isopropanol (DO NOT USE ACETONE)
- Large plastic pails or tubs for Liquinox® and water, scrub brushes, squirt bottles for Liquinox® solution, methanol and water, plastic bags and sheets
- DOT approved 55-gallon drum for disposal of waste
- Personal Protective Equipment as specified by the Health and Safety Plan
- Decontamination pad and steam cleaner/high pressure cleaner for large equipment

A11.4 Procedures and Guidelines

A11.4.1 Sampling Equipment Decontamination – Groundwater Sampling Pumps

Sampling pumps are decontaminated after each use as follows.

- Don phthalate-free gloves.
- Spread plastic on the ground to keep equipment from touching the ground
- Turn off pump after sampling. Remove pump from well and remove and dispose of tubing. Place pump in decontamination tube.
- Turn pump back on and pump 1 gallon of Liquinox® solution through the sampling pump.
- Rinse with 1 gallon of 10% isopropanol solution pumped through the pump. (DO NOT USE ACETONE). (Optional)
- Rinse with 1 gallon of tap water.
- Rinse with 1 gallon of deionized water.

- Keep decontaminated pump in decontamination tube or remove and wrap in aluminum foil or clean plastic sheeting.
- Collect all rinsate and dispose of in a DOT-approved 55-gallon drum.
- Decontamination materials (e.g., plastic sheeting, tubing, etc.) that have come in contact with used decontamination fluids or sampling equipment will be disposed of in either DOT-approved 55-gallon drums or with solid waste in garbage bags, dependent on Facility/project requirements.

A11.4.2 Sampling Equipment Decontamination – Other Equipment

Reusable sampling equipment is decontaminated after each use as follows.

- Don phthalate-free gloves.
- Before entering the potentially contaminated zone, wrap soil contact points in aluminum foil (shiny side out).
- Rinse and scrub with potable water.
- Wash all equipment surfaces that contacted the potentially contaminated soil/water with Liquinox solution.
- Rinse with potable water.
- Rinse with distilled or potable water and isopropanol solution (DO NOT USE ACETONE). (Optional)
- Air dry.
- Rinse with deionized water.
- Completely air dry and wrap exposed areas with aluminum foil (shiny side out) for transport and handling if equipment will not be used immediately.
- Collect all rinsate and dispose of in a DOT-approved 55-gallon drum.
- Decontamination materials (e.g., plastic sheeting, tubing, etc.) that have come in contact with used decontamination fluids or sampling equipment will be disposed of in DOT-approved 55-gallon drums or with solid waste in garbage bags, dependent on Facility/project requirements.

A11.4.3 Sampling Container Decontamination

The outsides of sample bottles or containers filled in the field may need to be decontaminated before being packed for shipment or handled by personnel without hand protection. The procedure is:

- Wipe container with a paper towel dampened with Liquinox solution or immerse in the solution AFTER THE CONTAINERS HAVE BEEN SEALED. Repeat the above steps using potable water.
- Dispose of all used paper towels in a DOT-approved 55-gallon drum or with solid waste in garbage bags, dependent on Facility/project requirements.

A11.5 Attachments

None

A11.6 Key Checks and Items

- Clean with solutions of Liquinox and distilled water.
- Do not use acetone for decontamination.
- Drum all contaminated rinsate and materials.
- Decontaminate filled sample bottles before relinquishing them to anyone.

Appendix B
List of Site Features

Appendix B. List of Site Features

Products (SE) Pipe Line Corporation

Lewis Drive Remediation Site, Belton, South Carolina

Site ID #18693 "Kinder Morgan Belton Pipeline Release"

Monitoring Wells
MW-01
MW-01B
MW-02
MW-02B
MW-03
MW-04
MW-05
MW-06
MW-06B
MW-07
MW-08
MW-09
MW-09B
MW-10
MW-11
MW-12
MW-12B
MW-13
MW-13B
MW-14
MW-14B
MW-15
MW-15B
MW-16
MW-17
MW-17B
MW-18
MW-19
MW-20
MW-21
MW-22
MW-23
MW-23B
MW-24
MW-24B
MW-25
MW-25B
MW-26
MW-26B
MW-27
MW-27B
MW-28
MW-29
MW-30
MW-31
MW-31B
MW-32
MW-33
MW-33T
MW-34
MW-35
MW-36
MW-36B
MW-37

Appendix B. List of Site Features

Products (SE) Pipe Line Corporation
Lewis Drive Remediation Site, Belton, South Carolina
Site ID #18693 "Kinder Morgan Belton Pipeline Release"

MW-38
MW-39
MW-40
MW-41
MW-42
MW-43
MW-43B
MW-44
MW-44B
MW-45
MW-45B
MW-46
MW-47
MW-48B
MW-49
MW-50B
MW-51
MW-52
MW-53
MW-54
MW-55
MW-56
MW-57
MW-58
MW-59
MW-60
MW-61B
MW-62
MW-63

Recovery Wells
RW-01
RW-02
RW-03
RW-04
RW-05
RW-06
RW-07
RW-08
RW-09
RW-10
RW-11
RW-12
RW-14
RW-15

Recovery Sumps
RS-01
RS-02
RS-04
RS-05
RS-06
RS-07
RS-08
RS-09
RS-10
RS-11

Appendix B. List of Site Features

Products (SE) Pipe Line Corporation
Lewis Drive Remediation Site, Belton, South Carolina
Site ID #18693 "Kinder Morgan Belton Pipeline Release"

RS-12
RS-13
RS-14
RS-15
RS-16
RS-17
RS-18
RS-20
Recovery Trench Sumps
RT-1A
RT-1B
RT-1C
Piezometers
TW-55
TW-64
TW-66
TW-67
TW-94
Vertical Air Sparging Wells
VAS-01
VAS-02
VAS-03
VAS-04
VAS-05
VAS-06
VAS-07
VAS-08
VAS-09
VAS-10
VAS-11
VAS-12
VAS-13
VAS-14
VAS-15
VAS-16
VAS-17
VAS-18
VAS-19
VAS-20
VAS-21
VAS-22
VAS-23
VAS-24
VAS-25
VAS-26
VAS-27
VAS-28
VAS-29
VAS-30
VAS-31
VAS-32
VAS-33
VAS-34

Appendix B. List of Site Features

Products (SE) Pipe Line Corporation

Lewis Drive Remediation Site, Belton, South Carolina

Site ID #18693 "Kinder Morgan Belton Pipeline Release"

VAS-35
VAS-36
VAS-37
VAS-38
VAS-39
VAS-40
VAS-41
VAS-42A
VAS-43A
VAS-44A
VAS-46
VAS-47
VAS-48
VAS-49
VAS-50
VAS-51
VAS-52
VAS-53
VAS-54
VAS-55
VAS-56
VAS-57
VAS-58
VAS-59
Horizontal Air Sparging Wells
HAS-1
HAS-2
HAS-3
HAS-4
HAS-5
HAS-6
Vertical Bedrock Sparging Wells
VBS-01
Surface Water Sampling Locations
SW-01
SW-02
SW-03
SW-04
SW-05
SW-07
SW-08
SW-09
SW-10
SW-11
SW-12
SW-13
SW-14

Appendix C
Lab Certification

dhec

Healthy People. Healthy Communities.

S.C. Department of Health and Environmental Control

Environmental Laboratory Certification Program

In accordance with the provisions of Regulation 61-81, entitled "State Environmental Laboratory Certification Regulations"

PACE ANALYTICAL NATIONAL CENTER
12065 LEBANON RD
MT JULIET, TENNESSEE 37122-2508

is hereby certified to perform analyses as documented on the attached parameter list(s). This certification does not guarantee validity of the data generated, but indicates the laboratory's adherence to prescribed methodology, quality control, records keeping, and reporting procedures. This certificate is the property of S.C. DHEC and must be surrendered upon demand. This certificate is non-transferable and is valid only for the parameters and methodology listed on the attached parameter list(s).

Laboratory Director: ERIC JOHNSON
Certifying Authority: LA
Date of Issue: December 09, 2022
Date of Expiration: June 30, 2023
Certificate Number: 84004002

Program Manager
Office of Environmental Laboratory Certification

CR-010021 (09/2016)

SOUTH CAROLINA DEPARTMENT OF HEALTH AND ENVIRONMENTAL CONTROL
ENVIRONMENTAL LABORATORY CERTIFICATION PROGRAM

PACE ANALYTICAL NATIONAL CENTER (Laboratory ID 84004)
Laboratory Director: ERIC JOHNSON
Certifying Authority: LA
Certificate Number: 84004002

Date of Issue: December 09, 2022
Expiration Date: June 30, 2023

CLEAN WATER ACT

INORGANIC - DEMAND

CHEMICAL OXYGEN DEMAND (COD)	EPA 410.4 (1993)	SPECTROPHOTOMETRIC, MANUAL OR AUTOMATED
TOTAL ORGANIC CARBON (TOC)	SM 5310 B-2014	HIGH TEMPERATURE COMBUSTION (TOC)

INORGANIC - MINERAL

ALKALINITY	SM 2320 B-2011	TITRIMETRIC
CHLORIDE	EPA 300.0 (1993)	ION CHROMATOGRAPHY
FLUORIDE	EPA 300.0 (1993)	ION CHROMATOGRAPHY
HARDNESS, TOTAL (AS CACO3)	EPA 130.1 (1971)	AUTOMATED COLORIMETRIC
HYDROGEN-ION CONC. (PH)	SM 4500-H B-2011	ELECTROMETRIC MEASUREMENT
SPECIFIC CONDUCTANCE	EPA 120.1 (1982)	WHEATSTONE BRIDGE
SULFATE	EPA 300.0 (1993)	ION CHROMATOGRAPHY

INORGANIC - MISCELLANEOUS

BROMIDE	EPA 300.0 (1993)	ION CHROMATOGRAPHY
CYANIDE, AMEN. TO CHLORINATION	SM 4500-CN G-2016	AMENABLE TO CHLORINATION (AFTER DISTILLATION)
CYANIDE, TOTAL	EPA 335.4 (1993)	SEMI-AUTOMATED COLORIMETRY
OIL & GREASE	EPA 1664B (2010)	OIL & GREASE - HEM/SGT-HEM
PHENOLICS, TOTAL RECOVERABLE	EPA 420.4 (1993)	AUTOMATED COLORIMETRIC (4AAP)
SULFIDE	SM 4500-S2 B-2011	SEPARATION OF SOLUBLE AND INSOLUBLE SULFIDES
SULFIDE	SM 4500-S2 C-2011	SAMPLE PRETREATMENT OR CONCENTRATION
SULFIDE	SM 4500-S2 D-2011	COLORIMETRIC (METHYLENE BLUE)
TURBIDITY	EPA 180.1 (1993)	NEPHELOMETRIC

INORGANIC - NUTRIENT

AMMONIA-NITROGEN	EPA 350.1 (1993)	MANUAL DISTILLATION WITH AUTOMATED PHENATE
AMMONIA-NITROGEN	SM 4500-NH3 B-2011	DISTILLATION
AMMONIA-NITROGEN	SM 4500-NH3 G-2011	AUTOMATED PHENATE
KJELDAHL-NITROGEN	EPA 351.2 (1993)	SEMI-AUTOMATED BLOCK DIGESTER COLORIMETRIC
NITRATE-NITRITE (N02&N03)	SM 4500-NO3 F-2016	CADMIUM REDUCTION (AUTOMATED)
ORTHOPHOSPHATE	SM 4500-P E-2011	ASCORBIC ACID (MANUAL SINGLE REAGENT)
TOTAL ORGANIC NITROGEN	EPA TKN-NH3-N	TOTAL KJELDAHL-N MINUS AMMONIA-N

INORGANIC - RESIDUE

RESIDUE, FILTERABLE (TDS)	SM 2540 C-2015	GRAVIMETRIC (180)
RESIDUE, NONFILTERABLE (TSS)	SM 2540 D-2015	GRAVIMETRIC 103-105
RESIDUE, SETTLEABLE (SS)	SM 2540 F-2015	VOLUMETRIC (IMHOFF CONE) OR GRAVIMETRIC

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CLEAN WATER ACT

INORGANIC - RESIDUE

RESIDUE, TOTAL (TS)	SM 2540 B-2015	GRAVIMETRIC 103-105

INORGANIC - TRACE METAL

ALUMINUM	EPA 200.7 (1994)	ICP/AES
ANTIMONY	EPA 200.7 (1994)	ICP/AES
ARSENIC	EPA 200.7 (1994)	ICP/AES
BARIUM	EPA 200.7 (1994)	ICP/AES
BERYLLIUM	EPA 200.7 (1994)	ICP/AES
BORON	EPA 200.7 (1994)	ICP/AES
CADMIUM	EPA 200.7 (1994)	ICP/AES
CALCIUM	EPA 200.7 (1994)	ICP/AES
CHROMIUM	EPA 200.7 (1994)	ICP/AES
CHROMIUM, HEXAVALENT	SM 3500-CR B-2011	COLORIMETRIC (DIPHYENYLCARBAZIDE)
COBALT	EPA 200.7 (1994)	ICP/AES
COPPER	EPA 200.7 (1994)	ICP/AES
IRON	EPA 200.7 (1994)	ICP/AES
LEAD	EPA 200.7 (1994)	ICP/AES
MAGNESIUM	EPA 200.7 (1994)	ICP/AES
MANGANESE	EPA 200.7 (1994)	ICP/AES
MERCURY	EPA 245.1 (1994)	COLD VAPOR (MANUAL)
MOLYBDENUM	EPA 200.7 (1994)	ICP/AES
NICKEL	EPA 200.7 (1994)	ICP/AES
POTASSIUM	EPA 200.7 (1994)	ICP/AES
SELENIUM	EPA 200.7 (1994)	ICP/AES
SILVER	EPA 200.7 (1994)	ICP/AES
SODIUM	EPA 200.7 (1994)	ICP/AES
THALLIUM	EPA 200.7 (1994)	ICP/AES
VANADIUM	EPA 200.7 (1994)	ICP/AES
ZINC	EPA 200.7 (1994)	ICP/AES

PCBS AND PESTICIDES

ORGANOCHLORINE PEST. & PCBS - GC/ECD	EPA 608.3 (2016)
ORGANOCHLORINE PEST. & PCBS - GC/ECD	EPA 608.3-RVE (2016)

SEMI-VOLATILES

BASE/NEUTRALS AND ACIDS - GC/MS	EPA 625.1 (2016)
BASE/NEUTRALS AND ACIDS - GC/MS - REDUC	EPA 625.1-RVE (2016)

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CLEAN WATER ACT

VOLATILES (VOCS)

PURGEABLES - GC/MS	EPA 624.1 (2016)	

SOLID & HAZARDOUS WASTES

INORGANIC - DEMAND

TOTAL ORGANIC CARBON (TOC)	EPA 9060A (2004)	CARBONACEOUS ANALYZER

INORGANIC - HAZARDOUS WASTE CHARACTERISTICS

IGNITABILITY	EPA 1010B (2018)	PENSKY-MARTENS CLOSED-CUP
PAINT FILTER LIQUIDS TEST	EPA 9095B (2004)	FILTRATION
SPLP - BOTTLE EXTRACTION	EPA 1312 (1994)	SYNTHETIC PRECIPITATION LEACHING PROCEDURE
SPLP - ZERO HEADSPACE	EPA 1312 (1994)	SYNTHETIC PRECIPITATION LEACHING PROCEDURE
TCLP - BOTTLE EXTRACTION	EPA 1311 (1992)	TOXICITY CHARACTERISTIC LEACHING PROCEDURE
TCLP - ZERO HEADSPACE	EPA 1311 (1992)	TOXICITY CHARACTERISTIC LEACHING PROCEDURE

INORGANIC - MINERAL

CHLORIDE	EPA 9056A (2007)	ION CHROMATOGRAPHY
FLUORIDE	EPA 9056A (2007)	ION CHROMATOGRAPHY
HYDROGEN-ION CONC. (PH)	EPA 9040C (2004)	ELECTROMETRIC
HYDROGEN-ION CONC. (PH) (SOIL & WASTE)	EPA 9045D (2004)	SOIL AND WASTE
SPECIFIC CONDUCTANCE	EPA 9050A (1996)	WHEATSTONE BRIDGE
SULFATE	EPA 9056A (2007)	ION CHROMATOGRAPHY

INORGANIC - MISCELLANEOUS

BROMIDE	EPA 9056A (2007)	ION CHROMATOGRAPHY
CYANIDE DISTILLATION	EPA 9010C (2004)	DISTILLATION FOR TOTAL & AMENABLE CYANIDE
CYANIDE, TOTAL	EPA 9012B (2004)	TOTAL AND AMENABLE (COLORIMETRIC, AUTOMATED UV)
EXTRACT. ORGANIC HALIDES IN SOLIDS (EOX	EPA 9023 (1996)	EXTRACTRABLE ORGANIC HALIDES IN SOLIDS (EOX)

INORGANIC - NUTRIENT

NITRATE-NITROGEN	EPA 9056A (2007)	ION CHROMATOGRAPHY
NITRITE-NITROGEN	EPA 9056A (2007)	ION CHROMATOGRAPHY

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SOLID & HAZARDOUS WASTES

INORGANIC - TRACE METAL

ALUMINUM	EPA 6010D (2018)	ICP/AES
ALUMINUM	EPA 6020B (2014)	ICP/MS
ANTIMONY	EPA 6010D (2018)	ICP/AES
ANTIMONY	EPA 6020B (2014)	ICP/MS
ARSENIC	EPA 6010D (2018)	ICP/AES
ARSENIC	EPA 6020B (2014)	ICP/MS
BARIUM	EPA 6010D (2018)	ICP/AES
BERYLLIUM	EPA 6010D (2018)	ICP/AES
BERYLLIUM	EPA 6020B (2014)	ICP/MS
CADMIUM	EPA 6010D (2018)	ICP/AES
CADMIUM	EPA 6020B (2014)	ICP/MS
CALCIUM	EPA 6010D (2018)	ICP/AES
CALCIUM	EPA 6020B (2014)	ICP/MS
CHROMIUM	EPA 6010D (2018)	ICP/AES
CHROMIUM	EPA 6020B (2014)	ICP/MS
CHROMIUM, HEXAVALENT	EPA 7196A (1992)	COLORIMETRIC
COBALT	EPA 6010D (2018)	ICP/AES
COPPER	EPA 6010D (2018)	ICP/AES
COPPER	EPA 6020B (2014)	ICP/MS
IRON	EPA 6010D (2018)	ICP/AES
IRON	EPA 6020B (2014)	ICP/MS
LEAD	EPA 6010D (2018)	ICP/AES
LEAD	EPA 6020B (2014)	ICP/MS
MAGNESIUM	EPA 6010D (2018)	ICP/AES
MAGNESIUM	EPA 6020B (2014)	ICP/MS
MANGANESE	EPA 6010D (2018)	ICP/AES
MERCURY	EPA 7470A (1994)	COLD VAPOR TECHNIQUE LIQUID
MERCURY	EPA 7471B (2007)	COLD VAPOR TECHNIQUE SOLID
METALS DIGESTION	EPA 3015A (2007)	AQUEOUS MICROWAVE ACID DIGESTION TOTAL METALS FLAA OR ICP
METALS DIGESTION	EPA 3050B (1996)	SOLID ACID DIGESTION
METALS DIGESTION	EPA 3051A (2007)	NON-AQUEOUS MICROWAVE ACID DIGESTION
METALS DIGESTION	EPA 3060A (1996)	ALKALINE DIGESTION HEX CHROM
MOLYBDENUM	EPA 6010D (2018)	ICP/AES
NICKEL	EPA 6010D (2018)	ICP/AES
NICKEL	EPA 6020B (2014)	ICP/MS
POTASSIUM	EPA 6010D (2018)	ICP/AES
POTASSIUM	EPA 6020B (2014)	ICP/MS
SELENIUM	EPA 6010D (2018)	ICP/AES
SILVER	EPA 6010D (2018)	ICP/AES
SILVER	EPA 6020B (2014)	ICP/MS

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SOLID & HAZARDOUS WASTES

INORGANIC - TRACE METAL

SODIUM	EPA 6010D (2018)	ICP/AES
SODIUM	EPA 6020B (2014)	ICP/MS
THALLIUM	EPA 6010D (2018)	ICP/AES
THALLIUM	EPA 6020B (2014)	ICP/MS
VANADIUM	EPA 6010D (2018)	ICP/AES
ZINC	EPA 6010D (2018)	ICP/AES
ZINC	EPA 6020B (2014)	ICP/MS

PCBS AND PESTICIDES

ORGANOCHLORINE PESTICIDES BY GC	EPA 8081B (2007)	EPA 3510C-RVE (1996)
ORGANOCHLORINE PESTICIDES BY GC	EPA 8081B (2007)	EPA 3546 (2007)
ORGANOCHLORINE PESTICIDES BY GC	EPA 8081B (2007)	EPA 3580A (1992)
ORGANOCHLORINE PESTICIDES BY GC	EPA 8081B (2007)	EPA 3510C (1996)
POLYCHLORINATED BIPHENYLS BY GC	EPA 8082A (2007)	EPA 3546 (2007)
POLYCHLORINATED BIPHENYLS BY GC	EPA 8082A (2007)	EPA 3580A (1992)
POLYCHLORINATED BIPHENYLS BY GC	EPA 8082A (2007)	EPA 3510C-RVE (1996)
POLYCHLORINATED BIPHENYLS BY GC	EPA 8082A (2007)	EPA 3510C (1996)

SEMI-VOLATILES

EDB & DBCP BY MICROEXTRACTION AND GC	EPA 8011 (1992)	
SEMIVOLATILE ORGANICS BY GC/MS	EPA 8270E (2018)	EPA 3546 (2007)
SEMIVOLATILE ORGANICS BY GC/MS	EPA 8270E (2018)	EPA 3580A (1992)
SEMIVOLATILE ORGANICS BY GC/MS	EPA 8270E (2018)	EPA 3510C-RVE (1996)
SEMIVOLATILE ORGANICS BY GC/MS (SIM)	EPA 8270E (SIM) (2018)	EPA 3510C-RVE (1996)
SEMIVOLATILE ORGANICS BY GC/MS (SIM)	EPA 8270E (SIM) (2018)	EPA 3546 (2007)
SEMIVOLATILE ORGANICS BY GC/MS (SIM)	EPA 8270E (SIM) (2018)	EPA 3510C (1996)

VOLATILES (VOCS)

OXYGENATE VOLATILE ORGANICS BY GC/MS	EPA 8260D-OXY (2018)	EPA 5030B (1996)
VOLATILE ORGANICS BY GC/MS	EPA 8260D (2018)	EPA 5035 (1996)
VOLATILE ORGANICS BY GC/MS	EPA 8260D (2018)	EPA 5030B (1996)

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SOLID & HAZARDOUS WASTES

----------VOLATILES (VOCS)----------

EPA 8260D (2018)
EPA 5035 (1996)

4-METHYL-2-PENTANONE
ACETONE
ACETONITRILE
ACROLEIN
ACRYLONITRILE
ALLYL CHLORIDE
BENZENE
BROMOBENZENE
BROMOCHLOROMETHANE
BROMODICHLOROMETHANE
BROMOFORM
BROMOMETHANE
CARBON DISULFIDE
CARBON TETRACHLORIDE
CHLOROBENZENE
CHLORODIBROMOMETHANE
CHLOROETHANE
CHLOROFORM
CHLOROMETHANE
CHLOROPRENE
CIS-1,2-DICHLOROETHENE
CIS-1,3-DICHLOROPROPENE
CIS-1,4-DICHLORO-2-BUTENE
DIBROMOMETHANE
DICHLORODIFLUOROMETHANE
DIETHYL ETHER
ETHYL ACETATE
ETHYL METHACRYLATE
ETHYLBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROETHANE
IODOMETHANE
ISOBUTYL ALCOHOL
ISOPROPYLBENZENE
METHACRYLONITRILE
METHYL ETHYL KETONE (MEK)
METHYL METHACRYLATE
METHYL TERT BUTYL ETHER (MTBE)
METHYLENE CHLORIDE
N-BUTANOL
N-BUTYLBENZENE
N-PROPYLBENZENE
NAPHTHALENE

EPA 8260D (2018)
EPA 5035 (1996)

P-ISOPROPYLTOLUENE
PENTACHLOROETHANE
PROPIONITRILE
SEC-BUTYLBENZENE
STYRENE
TERT-BUTYLBENZENE
TETRACHLOROETHENE
TOLUENE
TRANS-1,2-DICHLOROETHENE
TRANS-1,3-DICHLOROPROPENE
TRANS-1,4-DICHLORO-2-BUTENE
TRICHLOROETHENE
TRICHLOROFLUOROMETHANE
VINYL ACETATE
VINYL CHLORIDE
XYLENE, TOTAL

EPA 8260D-OXY (2018)
EPA 5030B (1996)

3,3-DIMETHYL-1-BUTANOL
DIISOPROPYL ETHER
ETHANOL
ETHYL TERT BUTYL ETHER
T-AMYL ALCOHOL
T-AMYL METHYL ETHER
T-BUTYL ALCOHOL
T-BUTYL FORMATE

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----------PCBS AND PESTICIDES----------

EPA 608.3 (2016)

4,4'-DDD
4,4'-DDE
4,4'-DDT
ALDRIN
ALPHA-BHC
BETA-BHC
CHLORDANE
DELTA-BHC
DIELDRIN
ENDOSULFAN I
ENDOSULFAN II
ENDOSULFAN SULFATE
ENDRIN
ENDRIN ALDEHYDE
GAMMA-BHC (LINDANE)
HEPTACHLOR
HEPTACHLOR EPOXIDE
METHOXYCHLOR
PCB-1016 (AROCLOR-1016)
PCB-1221 (AROCLOR-1221)
PCB-1232 (AROCLOR-1232)
PCB-1242 (AROCLOR-1242)
PCB-1248 (AROCLOR-1248)
PCB-1254 (AROCLOR-1254)
PCB-1260 (AROCLOR-1260)
TOXAPHENE

EPA 608.3-RVE (2016)

4,4'-DDD
4,4'-DDE
4,4'-DDT
ALDRIN
ALPHA-BHC
BETA-BHC
CHLORDANE
DELTA-BHC
DIELDRIN
ENDOSULFAN I
ENDOSULFAN II
ENDOSULFAN SULFATE
ENDRIN
ENDRIN ALDEHYDE
GAMMA-BHC (LINDANE)
HEPTACHLOR
HEPTACHLOR EPOXIDE
METHOXYCHLOR
PCB-1016 (AROCLOR-1016)
PCB-1221 (AROCLOR-1221)
PCB-1232 (AROCLOR-1232)
PCB-1242 (AROCLOR-1242)
PCB-1248 (AROCLOR-1248)
PCB-1254 (AROCLOR-1254)
PCB-1260 (AROCLOR-1260)
TOXAPHENE

----------SEMI-VOLATILES----------

EPA 625.1 (2016)

1,2,4-TRICHLOROBENZENE
2,4,6-TRICHLOROPHENOL
2,4-DICHLOROPHENOL
2,4-DIMETHYLPHENOL
2,4-DINITROPHENOL
2,4-DINITROTOLUENE (2,4-DNT)
2,6-DINITROTOLUENE (2,6-DNT)
2-CHLORONAPHTHALENE
2-CHLOROPHENOL
2-METHYL-4,6-DINITROPHENOL
2-NITROPHENOL
3,3-DICHLOROBENZIDINE
4-BROMOPHENYLPHENYL ETHER
4-CHLORO-3-METHYLPHENOL
4-CHLOROPHENYL PHENYL ETHER
4-NITROPHENOL
ACENAPHTHENE
ACENAPHTHYLENE
ANTHRACENE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
BENZYL BUTYL PHTHALATE
BIS(2-CHLORO-1-METHYLETHYL)ETHER
BIS(2-CHLOROETHOXY)METHANE
BIS(2-CHLOROETHYL)ETHER
BIS(2-ETHYLHEXYL)PHTHALATE
CHRYSENE
DI-N-BUTYL PHTHALATE
DI-N-OCTYL PHTHALATE
DIBENZO(A,H)ANTHRACENE
DIETHYL PHTHALATE
DIMETHYL PHTHALATE
FLUORANTHENE
FLUORENE
HEXACHLOROBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROCYCLOPENTADIENE
HEXACHLOROETHANE
INDENO(1,2,3-CD)PYRENE
ISOPHORONE
N-NITROSODI-N-PROPYLAMINE
NAPHTHALENE
NITROBENZENE (NB)
PENTACHLOROPHENOL
PHENANTHRENE
PHENOL
PYRENE

EPA 625.1-RVE (2016)

1,2,4-TRICHLOROBENZENE
2,4,6-TRICHLOROPHENOL
2,4-DICHLOROPHENOL
2,4-DIMETHYLPHENOL
2,4-DINITROPHENOL
2,4-DINITROTOLUENE (2,4-DNT)
2,6-DINITROTOLUENE (2,6-DNT)
2-CHLORONAPHTHALENE
2-CHLOROPHENOL
2-METHYL-4,6-DINITROPHENOL
2-NITROPHENOL
3,3-DICHLOROBENZIDINE
4-BROMOPHENYLPHENYL ETHER
4-CHLORO-3-METHYLPHENOL
4-CHLOROPHENYL PHENYL ETHER
4-NITROPHENOL
ACENAPHTHENE
ACENAPHTHYLENE
ANTHRACENE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
BENZYL BUTYL PHTHALATE
BIS(2-CHLORO-1-METHYLETHYL)ETHER
BIS(2-CHLOROETHOXY)METHANE
BIS(2-CHLOROETHYL)ETHER
BIS(2-ETHYLHEXYL)PHTHALATE
CHRYSENE
DI-N-BUTYL PHTHALATE
DI-N-OCTYL PHTHALATE
DIBENZO(A,H)ANTHRACENE
DIETHYL PHTHALATE
DIMETHYL PHTHALATE
FLUORANTHENE
FLUORENE
HEXACHLOROBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROCYCLOPENTADIENE
HEXACHLOROETHANE
INDENO(1,2,3-CD)PYRENE
ISOPHORONE
N-NITROSODI-N-PROPYLAMINE
NAPHTHALENE
NITROBENZENE (NB)
PENTACHLOROPHENOL
PHENANTHRENE
PHENOL
PYRENE

----------VOLATILES (VOCS)----------

EPA 624.1 (2016)

1,1,1-TRICHLOROETHANE
1,1,2,2-TETRACHLOROETHANE
1,1,2-TRICHLOROETHANE
1,1-DICHLOROETHANE
1,1-DICHLOROETHENE
1,2-DICHLOROBENZENE
1,2-DICHLOROETHANE
1,2-DICHLOROPROPANE
1,3-DICHLOROBENZENE
1,4-DICHLOROBENZENE
2-CHLOROETHYLVINYL ETHER
BENZENE
BROMODICHLOROMETHANE
BROMOFORM

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----------VOLATILES (VOCS)----------

EPA 624.1 (2016)

BROMOMETHANE
CARBON TETRACHLORIDE
CHLOROBENZENE
CHLORODIBROMOMETHANE
CHLOROETHANE
CHLOROFORM
CHLOROMETHANE
CIS-1,3-DICHLOROPROPENE
DICHLORODIFLUOROMETHANE
ETHYLBENZENE
METHYL TERT BUTYL ETHER (MTBE)
METHYLENE CHLORIDE
TETRACHLOROETHENE
TOLUENE
TRANS-1,2-DICHLOROETHENE
TRANS-1,3-DICHLOROPROPENE
TRICHLOROETHENE
TRICHLOROFLUOROMETHANE
VINYL CHLORIDE
XYLENE, TOTAL

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----------PCBS AND PESTICIDES----------

EPA 8081B (2007)
EPA 3510C (1996)

4,4'-DDD
4,4'-DDE
4,4'-DDT
ALDRIN
ALPHA-BHC
ALPHA-CHLORDANE
BETA-BHC
CHLORDANE
DELTA-BHC
DIELDRIN
ENDOSULFAN I
ENDOSULFAN II
ENDOSULFAN SULFATE
ENDRIN
ENDRIN ALDEHYDE
ENDRIN KETONE
GAMMA-BHC (LINDANE)
GAMMA-CHLORDANE
HEPTACHLOR
HEPTACHLOR EPOXIDE
HEXACHLOROBENZENE
METHOXYCHLOR
TOXAPHENE

EPA 8081B (2007)
EPA 3510C-RVE (1996)

4,4'-DDD
4,4'-DDE
4,4'-DDT
ALDRIN
ALPHA-BHC
ALPHA-CHLORDANE
BETA-BHC
CHLORDANE
DELTA-BHC
DIELDRIN
ENDOSULFAN I
ENDOSULFAN II
ENDOSULFAN SULFATE
ENDRIN
ENDRIN ALDEHYDE
ENDRIN KETONE

EPA 8081B (2007)
EPA 3510C-RVE (1996)

GAMMA-BHC (LINDANE)
GAMMA-CHLORDANE
HEPTACHLOR
HEPTACHLOR EPOXIDE
HEXACHLOROBENZENE
METHOXYCHLOR
TOXAPHENE

EPA 8081B (2007)
EPA 3546 (2007)

4,4'-DDD
4,4'-DDE
4,4'-DDT
ALDRIN
ALPHA-BHC
ALPHA-CHLORDANE
BETA-BHC
CHLORDANE
DELTA-BHC
DIELDRIN
ENDOSULFAN I
ENDOSULFAN II
ENDOSULFAN SULFATE
ENDRIN
ENDRIN ALDEHYDE
ENDRIN KETONE
GAMMA-BHC (LINDANE)
GAMMA-CHLORDANE
HEPTACHLOR
HEPTACHLOR EPOXIDE
HEXACHLOROBENZENE
METHOXYCHLOR
TOXAPHENE

EPA 8081B (2007)
EPA 3580A (1992)

4,4'-DDD
4,4'-DDE
4,4'-DDT
ALDRIN
ALPHA-BHC
ALPHA-CHLORDANE
BETA-BHC
CHLORDANE
DELTA-BHC
DIELDRIN
ENDOSULFAN I
ENDOSULFAN II
ENDOSULFAN SULFATE
ENDRIN
ENDRIN ALDEHYDE
ENDRIN KETONE
GAMMA-BHC (LINDANE)
GAMMA-CHLORDANE
HEPTACHLOR
HEPTACHLOR EPOXIDE
HEXACHLOROBENZENE
METHOXYCHLOR
TOXAPHENE

EPA 8082A (2007)
EPA 3510C (1996)

PCB-1016 (AROCLOR-1016)
PCB-1221 (AROCLOR-1221)
PCB-1232 (AROCLOR-1232)
PCB-1242 (AROCLOR-1242)
PCB-1248 (AROCLOR-1248)
PCB-1254 (AROCLOR-1254)
PCB-1260 (AROCLOR-1260)

EPA 8082A (2007)
EPA 3510C-RVE (1996)

PCB-1016 (AROCLOR-1016)
PCB-1221 (AROCLOR-1221)
PCB-1232 (AROCLOR-1232)
PCB-1242 (AROCLOR-1242)
PCB-1248 (AROCLOR-1248)
PCB-1254 (AROCLOR-1254)
PCB-1260 (AROCLOR-1260)

EPA 8082A (2007)
EPA 3546 (2007)

PCB-1016 (AROCLOR-1016)
PCB-1221 (AROCLOR-1221)
PCB-1232 (AROCLOR-1232)

EPA 8082A (2007)
EPA 3546 (2007)

PCB-1242 (AROCLOR-1242)
PCB-1248 (AROCLOR-1248)
PCB-1254 (AROCLOR-1254)
PCB-1260 (AROCLOR-1260)

EPA 8082A (2007)
EPA 3580A (1992)

PCB-1016 (AROCLOR-1016)
PCB-1221 (AROCLOR-1221)
PCB-1232 (AROCLOR-1232)
PCB-1242 (AROCLOR-1242)
PCB-1248 (AROCLOR-1248)
PCB-1254 (AROCLOR-1254)
PCB-1260 (AROCLOR-1260)

----------SEMI-VOLATILES----------

EPA 8011 (1992)

1,2-DIBROMO-3-CHLOROPROPANE(DBCP)
1,2-DIBROMOETHANE (EDB)

EPA 8270E (2018)
EPA 3510C-RVE (1996)

1,2,4,5-TETRACHLOROBENZENE
1,2,4-TRICHLOROBENZENE
1,2-DICHLOROBENZENE
1,3,5-TRINITROBENZENE (1,3,5-TNB)
1,3-DICHLOROBENZENE
1,3-DINITROBENZENE (1,3-DNB)
1,4-DICHLOROBENZENE
1,4-NAPHTHOQUINONE
1-CHLORONAPHTHALENE
1-NAPHTHYLAMINE
2,3,4,6-TETRACHLOROPHENOL
2,4,5-TRICHLOROPHENOL
2,4,6-TRICHLOROPHENOL
2,4-DICHLOROPHENOL
2,4-DIMETHYLPHENOL
2,4-DINITROPHENOL
2,4-DINITROTOLUENE (2,4-DNT)
2,6-DICHLOROPHENOL
2,6-DINITROTOLUENE (2,6-DNT)

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----------SEMI-VOLATILES----------

EPA 8270E (2018)
EPA 3510C-RVE (1996)

2-ACETYLAMINOFLUORENE
2-CHLORONAPHTHALENE
2-CHLOROPHENOL
2-METHYLNAPHTHALENE
2-METHYLPHENOL
2-NAPHTHYLAMINE
2-NITROANILINE
2-NITROPHENOL
2-PICOLINE (2-METHYLPYRIDINE)
3,3-DICHLOROBENZIDINE
3,3-DIMETHYLBENZIDINE
3-METHYLCHOLANTHRENE
3-NITROANILINE
4,6-DINITRO-2-METHYLPHENOL
4-AMINOBIPHENYL
4-BROMOPHENYLPHENYL ETHER
4-CHLORO-3-METHYLPHENOL
4-CHLOROANILINE
4-CHLOROPHENYL PHENYL ETHER
4-METHYLPHENOL
4-NITROANILINE
4-NITROPHENOL
4-NITROQUINOLINE-1-OXIDE
5-NITRO-O-TOLUIDINE
7,12-DIMETHYLBENZ(A)ANTHRACENE
ACENAPHTHENE
ACENAPHTHYLENE
ACETOPHENONE
ANILINE
ANTHRACENE
BENZIDINE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
BENZYL ALCOHOL
BIS(2-CHLORO-1-METHYLETHYL)ETHER
BIS(2-CHLOROETHOXY)METHANE
BIS(2-CHLOROETHYL)ETHER
BIS(2-ETHYLHEXYL)PHTHALATE
BUTYL BENZYL PHTHALATE
CHLOROBENZILATE

EPA 8270E (2018)
EPA 3510C-RVE (1996)

CHRYSENE
DI-N-BUTYL PHTHALATE
DI-N-OCTYL PHTHALATE
DIALLATE
DIBENZ(A,J)ACRIDINE
DIBENZO(A,E)PYRENE
DIBENZO(A,H)ANTHRACENE
DIBENZOFURAN
DIETHYL PHTHALATE
DIMETHOATE
DIMETHYL PHTHALATE
DIMETHYLAMINOAZOBENZENE
DINOSEB
DIPHENYLAMINE
DISULFOTON
ETHYL METHANESULFONATE
FAMPHUR
FLUORANTHENE
FLUORENE
HEXACHLOROBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROCYCLOPENTADIENE
HEXACHLOROETHANE
INDENO(1,2,3-CD)PYRENE
ISODRIN
ISOPHORONE
ISOSAFROLE
KEPONE
METHYL METHANESULFONATE
METHYL PARATHION
N-NITROSODI-N-BUTYLAMINE
N-NITROSODI-N-PROPYLAMINE
N-NITROSODIETHYLAMINE
N-NITROSODIMETHYLAMINE
N-NITROSODIPHENYLAMINE
N-NITROSOMETHYLETHYLAMINE
N-NITROSOMORPHOLINE
N-NITROSOPIPERIDINE
N-NITROSOPYRROLIDINE
NAPHTHALENE
NITROBENZENE (NB)
O,O,O-TRIETHYLPHOSPHOROTHIOATE
O-TOLUIDINE
PARATHION
PENTACHLOROBENZENE

EPA 8270E (2018)
EPA 3510C-RVE (1996)

PENTACHLORONITROBENZENE
PENTACHLOROPHENOL
PHENACETIN
PHENANTHRENE
PHENOL
PHORATE
PRONAMIDE
PYRENE
PYRIDINE
SAFROLE
THIONAZIN

EPA 8270E (2018)
EPA 3546 (2007)

1,2,4,5-TETRACHLOROBENZENE
1,2,4-TRICHLOROBENZENE
1,2-DICHLOROBENZENE
1,3,5-TRINITROBENZENE (1,3,5-TNB)
1,3-DICHLOROBENZENE
1,3-DINITROBENZENE (1,3-DNB)
1,4-DICHLOROBENZENE
1,4-NAPHTHOQUINONE
1-CHLORONAPHTHALENE
1-NAPHTHYLAMINE
2,3,4,6-TETRACHLOROPHENOL
2,4,5-TRICHLOROPHENOL
2,4,6-TRICHLOROPHENOL
2,4-DICHLOROPHENOL
2,4-DIMETHYLPHENOL
2,4-DINITROPHENOL
2,4-DINITROTOLUENE (2,4-DNT)
2,6-DICHLOROPHENOL
2,6-DINITROTOLUENE (2,6-DNT)
2-ACETYLAMINOFLUORENE
2-CHLORONAPHTHALENE
2-CHLOROPHENOL
2-METHYLNAPHTHALENE
2-METHYLPHENOL
2-NAPHTHYLAMINE
2-NITROANILINE
2-NITROPHENOL
2-PICOLINE (2-METHYLPYRIDINE)
3,3-DICHLOROBENZIDINE
3,3-DIMETHYLBENZIDINE

EPA 8270E (2018)
EPA 3546 (2007)

3-METHYLCHOLANTHRENE
3-NITROANILINE
4,6-DINITRO-2-METHYLPHENOL
4-AMINOBIPHENYL
4-BROMOPHENYLPHENYL ETHER
4-CHLORO-3-METHYLPHENOL
4-CHLOROANILINE
4-CHLOROPHENYL PHENYL ETHER
4-METHYLPHENOL
4-NITROANILINE
4-NITROPHENOL
4-NITROQUINOLINE-1-OXIDE
5-NITRO-O-TOLUIDINE
7,12-DIMETHYLBENZ(A)ANTHRACENE
A,A-DIMETHYLPHENETHYLAMINE
ACENAPHTHENE
ACENAPHTHYLENE
ACETOPHENONE
ANILINE
ANTHRACENE
BENZIDINE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
BENZYL ALCOHOL
BIS(2-CHLORO-1-METHYLETHYL)ETHER
BIS(2-CHLOROETHOXY)METHANE
BIS(2-CHLOROETHYL)ETHER
BIS(2-ETHYLHEXYL)PHTHALATE
BUTYL BENZYL PHTHALATE
CHLOROBENZILATE
CHRYSENE
DI-N-BUTYL PHTHALATE
DI-N-OCTYL PHTHALATE
DIALLATE
DIBENZ(A,J)ACRIDINE
DIBENZO(A,H)ANTHRACENE
DIBENZOFURAN
DIETHYL PHTHALATE
DIMETHOATE
DIMETHYL PHTHALATE
DIMETHYLAMINOAZOBENZENE
DINOSEB

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----------SEMI-VOLATILES----------

EPA 8270E (2018)
EPA 3546 (2007)

DIPHENYLAMINE
DISULFOTON
ETHYL METHANESULFONATE
FAMPHUR
FLUORANTHENE
FLUORENE
HEXACHLOROBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROCYCLOPENTADIENE
HEXACHLOROETHANE
HEXACHLOROPHENE
HEXACHLOROPROPENE
INDENO(1,2,3-CD)PYRENE
ISODRIN
ISOPHORONE
ISOSAFROLE
KEPONE
METHAPYRILENE
METHYL METHANESULFONATE
METHYL PARATHION
N-NITROSODI-N-BUTYLAMINE
N-NITROSODI-N-PROPYLAMINE
N-NITROSODIETHYLAMINE
N-NITROSODIMETHYLAMINE
N-NITROSODIPHENYLAMINE
N-NITROSOMETHYLETHYLAMINE
N-NITROSOMORPHOLINE
N-NITROSOPIPERIDINE
N-NITROSOPYRROLIDINE
NAPHTHALENE
NITROBENZENE (NB)
O,O,O-TRIETHYLPHOSPHOROTHIOATE
O-TOLUIDINE
PARATHION
PENTACHLOROBENZENE
PENTACHLORONITROBENZENE
PENTACHLOROPHENOL
PHENACETIN
PHENANTHRENE
PHENOL
PHORATE
PYRENE
PYRIDINE

EPA 8270E (2018)
EPA 3546 (2007)

SAFROLE
THIONAZIN

EPA 8270E (2018)
EPA 3580A (1992)

1,2,4,5-TETRACHLOROBENZENE
1,2,4-TRICHLOROBENZENE
1,2-DICHLOROBENZENE
1,3,5-TRINITROBENZENE (1,3,5-TNB)
1,3-DICHLOROBENZENE
1,3-DINITROBENZENE (1,3-DNB)
1,4-DICHLOROBENZENE
1,4-NAPHTHOQUINONE
1-CHLORONAPHTHALENE
1-NAPHTHYLAMINE
2,3,4,6-TETRACHLOROPHENOL
2,4,5-TRICHLOROPHENOL
2,4,6-TRICHLOROPHENOL
2,4-DICHLOROPHENOL
2,4-DIMETHYLPHENOL
2,4-DINITROPHENOL
2,4-DINITROTOLUENE (2,4-DNT)
2,6-DICHLOROPHENOL
2,6-DINITROTOLUENE (2,6-DNT)
2-ACETYLAMINOFLUORENE
2-CHLORONAPHTHALENE
2-CHLOROPHENOL
2-METHYLNAPHTHALENE
2-METHYLPHENOL
2-NAPHTHYLAMINE
2-NITROANILINE
2-NITROPHENOL
2-PICOLINE (2-METHYLPYRIDINE)
3,3-DICHLOROBENZIDINE
3,3-DIMETHYLBENZIDINE
3-METHYLCHOLANTHRENE
3-NITROANILINE
4,6-DINITRO-2-METHYLPHENOL
4-AMINOBIPHENYL
4-BROMOPHENYLPHENYL ETHER
4-CHLORO-3-METHYLPHENOL
4-CHLOROANILINE
4-CHLOROPHENYL PHENYL ETHER
4-METHYLPHENOL

EPA 8270E (2018)
EPA 3580A (1992)

4-NITROANILINE
4-NITROPHENOL
4-NITROQUINOLINE-1-OXIDE
5-NITRO-O-TOLUIDINE
7,12-DIMETHYLBENZ(A)ANTHRACENE
A,A-DIMETHYLPHENETHYLAMINE
ACENAPHTHENE
ACENAPHTHYLENE
ACETOPHENONE
ANILINE
ANTHRACENE
BENZIDINE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
BENZYL ALCOHOL
BIS(2-CHLORO-1-METHYLETHYL)ETHER
BIS(2-CHLOROETHOXY)METHANE
BIS(2-CHLOROETHYL)ETHER
BIS(2-ETHYLHEXYL)PHTHALATE
BUTYL BENZYL PHTHALATE
CHLOROBENZILATE
CHRYSENE
DI-N-BUTYL PHTHALATE
DI-N-OCTYL PHTHALATE
DIALLATE
DIBENZ(A,J)ACRIDINE
DIBENZO(A,E)PYRENE
DIBENZO(A,H)ANTHRACENE
DIBENZOFURAN
DIETHYL PHTHALATE
DIMETHOATE
DIMETHYL PHTHALATE
DIMETHYLAMINOAZOBENZENE
DINOSEB
DIPHENYLAMINE
DISULFOTON
ETHYL METHANESULFONATE
FAMPHUR
FLUORANTHENE
FLUORENE
HEXACHLOROBENZENE
HEXACHLOROBUTADIENE

EPA 8270E (2018)
EPA 3580A (1992)

HEXACHLOROCYCLOPENTADIENE
HEXACHLOROETHANE
HEXACHLOROPHENE
HEXACHLOROPROPENE
INDENO(1,2,3-CD)PYRENE
ISODRIN
ISOPHORONE
ISOSAFROLE
KEPONE
METHAPYRILENE
METHYL METHANESULFONATE
METHYL PARATHION
N-NITROSODI-N-BUTYLAMINE
N-NITROSODI-N-PROPYLAMINE
N-NITROSODIETHYLAMINE
N-NITROSODIMETHYLAMINE
N-NITROSODIPHENYLAMINE
N-NITROSOMETHYLETHYLAMINE
N-NITROSOMORPHOLINE
N-NITROSOPIPERIDINE
N-NITROSOPYRROLIDINE
NAPHTHALENE
NITROBENZENE (NB)
O,O,O-TRIETHYLPHOSPHOROTHIOATE
O-TOLUIDINE
PARATHION
PENTACHLOROBENZENE
PENTACHLORONITROBENZENE
PENTACHLOROPHENOL
PHENACETIN
PHENANTHRENE
PHENOL
PHORATE
PRONAMIDE
PYRENE
PYRIDINE
SAFROLE
THIONAZIN

EPA 8270E (SIM) (2018)
EPA 3510C (1996)

2-METHYLNAPHTHALENE
ACENAPHTHENE
ACENAPHTHYLENE

SOUTH CAROLINA DEPARTMENT OF HEALTH AND ENVIRONMENTAL CONTROL
ENVIRONMENTAL LABORATORY CERTIFICATION PROGRAM

PACE ANALYTICAL NATIONAL CENTER (Laboratory ID 84004)
Certifying Authority: LA
Certificate Number: 84004002

Date of Issue: December 09, 2022
Expiration Date: June 30, 2023

SOLID & HAZARDOUS WASTES

----------SEMI-VOLATILES----------

EPA 8270E (SIM) (2018)
EPA 3510C (1996)

ANTHRACENE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
CHRYSENE
DIBENZO(A,H)ANTHRACENE
FLUORANTHENE
FLUORENE
INDENO(1,2,3-CD)PYRENE
NAPHTHALENE
PHENANTHRENE
PYRENE

EPA 8270E (SIM) (2018)
EPA 3510C-RVE (1996)

2-METHYLNAPHTHALENE
ACENAPHTHENE
ACENAPHTHYLENE
ANTHRACENE
BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
CHRYSENE
DIBENZO(A,H)ANTHRACENE
FLUORANTHENE
FLUORENE
INDENO(1,2,3-CD)PYRENE
NAPHTHALENE
PHENANTHRENE
PYRENE

EPA 8270E (SIM) (2018)
EPA 3546 (2007)

2-METHYLNAPHTHALENE
ACENAPHTHENE
ACENAPHTHYLENE
ANTHRACENE

EPA 8270E (SIM) (2018)
EPA 3546 (2007)

BENZO(A)ANTHRACENE
BENZO(A)PYRENE
BENZO(B)FLUORANTHENE
BENZO(G,H,I)PERYLENE
BENZO(K)FLUORANTHENE
CHRYSENE
DIBENZO(A,H)ANTHRACENE
FLUORANTHENE
FLUORENE
INDENO(1,2,3-CD)PYRENE
NAPHTHALENE
PHENANTHRENE
PYRENE

----------VOLATILES (VOCS)----------

EPA 8260D (2018)
EPA 5030B (1996)

1,1,1,2-TETRACHLOROETHANE
1,1,1-TRICHLOROETHANE
1,1,2,2-TETRACHLOROETHANE
1,1,2-TRICHLOROETHANE
1,1-DICHLOROETHANE
1,1-DICHLOROETHENE
1,1-DICHLOROPROPENE
1,2,3-TRICHLOROBENZENE
1,2,3-TRICHLOROPROPANE
1,2,4-TRICHLOROBENZENE
1,2,4-TRIMETHYLBENZENE
1,2-DIBROMO-3-CHLOROPROPANE(DBCP)
1,2-DIBROMOETHANE (EDB)
1,2-DICHLOROBENZENE
1,2-DICHLOROETHANE
1,2-DICHLOROPROPANE
1,3,5-TRIMETHYLBENZENE
1,3-DICHLOROBENZENE
1,3-DICHLOROPROPANE
1,4-DICHLOROBENZENE
1,4-DIOXANE
2,2-DICHLOROPROPANE
2-CHLOROETHYLVINYL ETHER
2-CHLOROTOLUENE
2-HEXANONE
2-NITROPROPANE

EPA 8260D (2018)
EPA 5030B (1996)

4-CHLOROTOLUENE
4-METHYL-2-PENTANONE
ACETONE
ACETONITRILE
ACROLEIN
ACRYLONITRILE
ALLYL CHLORIDE
BENZENE
BROMOBENZENE
BROMOCHLOROMETHANE
BROMODICHLOROMETHANE
BROMOFORM
BROMOMETHANE
CARBON DISULFIDE
CARBON TETRACHLORIDE
CHLOROBENZENE
CHLORODIBROMOMETHANE
CHLOROETHANE
CHLOROFORM
CHLOROMETHANE
CHLOROPRENE
CIS-1,2-DICHLOROETHENE
CIS-1,3-DICHLOROPROPENE
CIS-1,4-DICHLORO-2-BUTENE
DIBROMOMETHANE
DICHLORODIFLUOROMETHANE
DIETHYL ETHER
ETHYL ACETATE
ETHYL METHACRYLATE
ETHYLBENZENE
HEXACHLOROBUTADIENE
HEXACHLOROETHANE
IODOMETHANE
ISOBUTYL ALCOHOL
ISOPROPYLBENZENE
METHACRYLONITRILE
METHYL ETHYL KETONE (MEK)
METHYL METHACRYLATE
METHYL TERT BUTYL ETHER (MTBE)
METHYLENE CHLORIDE
N-BUTANOL
N-BUTYLBENZENE
N-PROPYLBENZENE
NAPHTHALENE
P-ISOPROPYLTOLUENE

EPA 8260D (2018)
EPA 5030B (1996)

PENTACHLOROETHANE
PROPIONITRILE
SEC-BUTYLBENZENE
STYRENE
TERT-BUTYLBENZENE
TETRACHLOROETHENE
TOLUENE
TRANS-1,2-DICHLOROETHENE
TRANS-1,3-DICHLOROPROPENE
TRANS-1,4-DICHLORO-2-BUTENE
TRICHLOROETHENE
TRICHLOROFLUOROMETHANE
VINYL ACETATE
VINYL CHLORIDE
XYLENE, TOTAL

EPA 8260D (2018)
EPA 5035 (1996)

1,1,1,2-TETRACHLOROETHANE
1,1,1-TRICHLOROETHANE
1,1,2,2-TETRACHLOROETHANE
1,1,2-TRICHLOROETHANE
1,1-DICHLOROETHANE
1,1-DICHLOROETHENE
1,1-DICHLOROPROPENE
1,2,3-TRICHLOROBENZENE
1,2,3-TRICHLOROPROPANE
1,2,4-TRICHLOROBENZENE
1,2,4-TRIMETHYLBENZENE
1,2-DIBROMO-3-CHLOROPROPANE(DBCP)
1,2-DIBROMOETHANE (EDB)
1,2-DICHLOROBENZENE
1,2-DICHLOROETHANE
1,2-DICHLOROPROPANE
1,3,5-TRIMETHYLBENZENE
1,3-DICHLOROBENZENE
1,3-DICHLOROPROPANE
1,4-DICHLOROBENZENE
1,4-DIOXANE
2,2-DICHLOROPROPANE
2-CHLOROTOLUENE
2-HEXANONE
2-NITROPROPANE
4-CHLOROTOLUENE

dhec

Healthy People. Healthy Communities.

December 09, 2022

ERIC JOHNSON
PACE ANALYTICAL NATIONAL CENTER
12065 LEBANON RD
MT. JULIET, TENNESSEE 37122-2508

Laboratory ID # 84004
Certificate # 84004002

Dear Eric Johnson:

Your current certificate and associated parameter/method list(s) are enclosed. These documents now represent the certificate of record for your laboratory. All previous certificate(s) and associated parameter list(s) received are now null and void and should be destroyed. Please be reminded that all environmental data submitted to the Department is reviewed to ensure that the reporting laboratory possesses the necessary certification. Data reported by laboratories without the proper certification will be addressed by the affected enforcement programs.

If you have any questions or problems are detected concerning your certificate, please contact our office within ten (10) working days at (803)896-0970 or by e-mail at labcerthelp@dhec.sc.gov.

Sincerely,

Bennie L. Cockerel, Jr., Program Manager
Office of Environmental Laboratory Certification
Bureau of Environmental Health Services

Enclosures

Register on our website at www.scdhec.gov/labcert to receive e-mail updates for the Laboratory Certification Program. Subscribing is easy and you'll automatically receive new posts to our website.