Vacuum Distillation Unit Interlaboratory Study Evaluation

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Method 8261 Vacuum Distillation Background

- USEPA SW-846 Method 8261 Developed by USEPA National Exposure Research Laboratory (NERL) in Las Vegas, Nevada
- Method 8261 Uses a Vacuum Distillation Unit (VDU) to Extract VOCs and Select SVOCs from Environmental Samples
- VDU Developed and Patented by USEPA NERL
- Cincinnati Analytical Instruments (CAI) Licensed by USEPA to Manufacture VDU
- Additional Information Can be Obtained on EPA Website
  - www.epa.gov/nerlesd1/chemistry/vacuum/default.htm
- QATS Laboratory Tasked to Evaluate Method 8261 for Applicability to the Contract Laboratory Program (CLP)

Method 8261 Vacuum Distillation Procedure

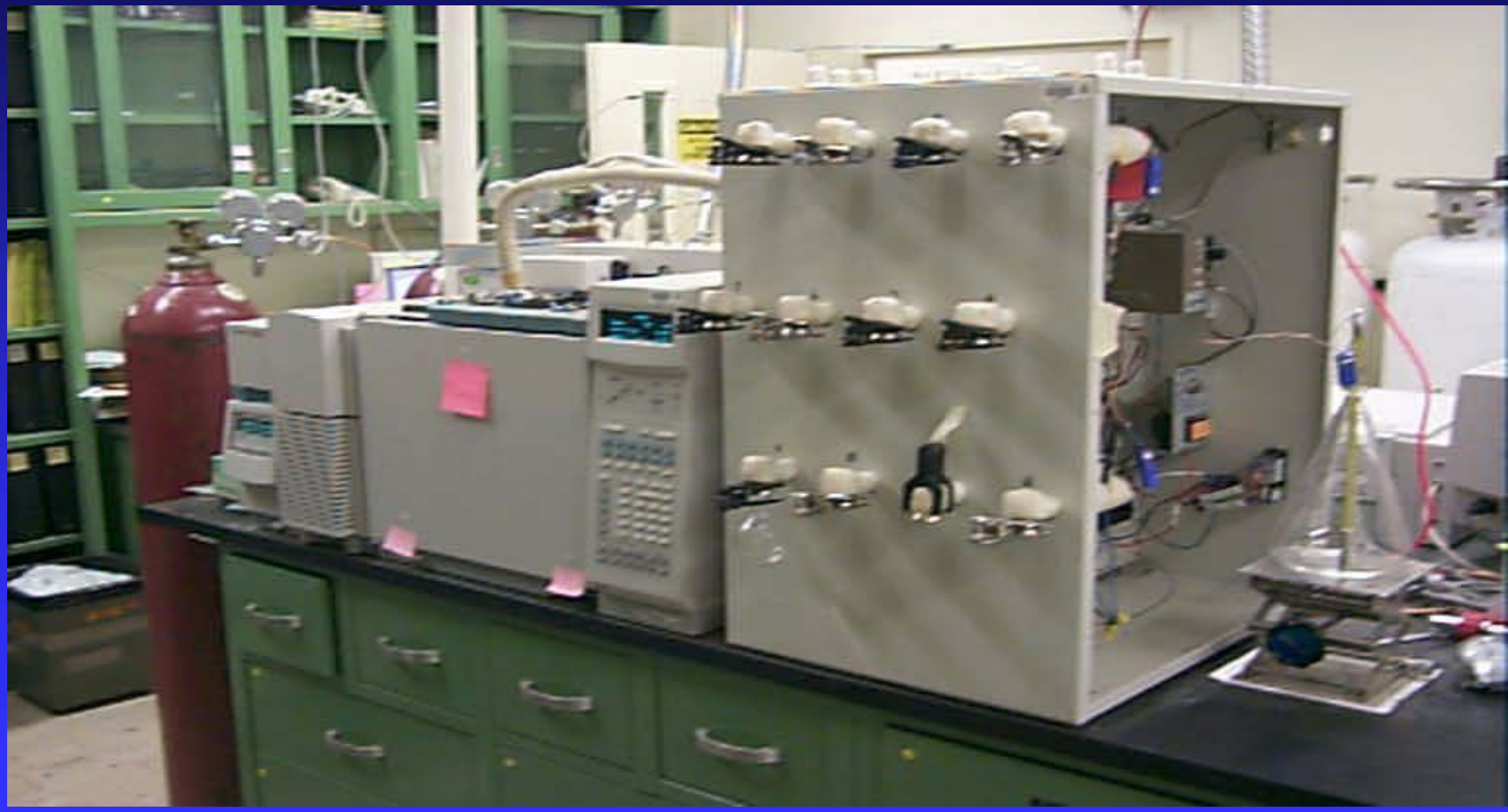
- Matrices Include Water, Soil, Sediment, Sludge, Oily Waste, and Animal and Plant Tissue
- Sample Size is Typically 5 mL or 5 Grams Depending on Matrix
  - High Level Samples or Difficult Matrices Can Use Less
- Sample is Transferred to a Distillation Flask, 5 mL of Water is Added to Solid and Oily Matrices, and Surrogates are Added
- Flask is Attached to the VDU Sample Mount – Current VDU Autosampler has 12 Sample Positions
- Sample Chamber Pressure is Reduced to Approximately the Vapor Pressure of Water Using Vacuum
- Vapor Passes Over a Chilled Condenser Coil to Condense Water Removed from the Sample

Method 8261 Vacuum Distillation Procedure (Cont'd)

- Uncondensed Distillate is Cryogenically Trapped in a Stainless Steel Loop Chilled With Liquid Nitrogen (-196^{o}C)
- After Vacuum Distillation, Cryotrap is Thermally Desorbed and Analytes are Transferred to a Gas Chromatograph by Carrier Gas
- Analytes are Separated on the GC and Flow into a Mass Spectrometer for Detection
- Target Analytes are Quantitated Using Surrogate-Based Matrix Correction
- System Performance is Monitored and Evaluated Using Recoveries of a Series of Check Surrogates

Method 8261 Vacuum Distillation Unit Prototype

Method 8261 Current Vacuum Distillation Unit



Method 8261 VDU Distillation Vessel

Method 8261 VDU Analyte Quantitation Procedure Description

- Quantitation is a Three Step Process
  - Analyte Amount is Calculated Using External Standard Procedures Using Area Response and Response Factors from Initial Calibration
  - Surrogate Recovery is Determined and Used to Predict the Recoveries of Analytes Within Surrogate Groups Based on Physical Properties – Relative Volatility and Boiling Point
  - Analyte Concentration is Calculated Using the Predicted Recovery, Sample Size, and Amount of Analyte Detected Using External Standard Calculation
- Quantitation and Recovery of a Known Amount of Spiked Check Surrogates are Used to Evaluate System Performance

Method 8261 VDU Analyte Quantitation Procedure Description (Cont'd)

- Three Types of Surrogates are Used
  - alpha-Surrogates are Used to Measure Recovery of an Analyte Relative to the Gas/Liquid Partition Characteristics of the Analyte
  - beta-Surrogates are Used to Measure the Recovery of an Analyte Relative to the Condensation Characteristics of the Analyte
  - Check Surrogates are used to Monitor and Evaluate the Overall System Performance
- SMCReporter Stand-Alone Software has Been Developed to Perform All Quantitation Calculations and is Available on the USEPA Vacuum Distillation Website

Method 8261 Interlaboratory Study

- Four Laboratories Participated in the Interlaboratory Study
- Two Laboratories Have Completed the Study and Results from Two Laboratories are Pending
- None of the Laboratories Had Previous Experience With the Method or the VDU
- VDUs and All Ampulated Standards and Blinds Were Supplied to the Laboratories
- All Laboratories Requested to Perform:
  - Initial Calibration Studies
  - Method Detection Limit (MDL) Studies
  - Appropriate Continuing Calibration Verification (CCV) Analyses
  - Appropriate Blank Analyses
  - Performance Spike Analyses
  - Blind Sample Analyses

Method 8261 Interlaboratory Study (Cont'd)

- Initial Calibration
  - Laboratories Performed Initial Calibrations as Required
  - Five Point Initial Calibrations Were Analyzed
  - Range for Most Compounds is 1 to 50 ug/L (ppb)
  - Select Compounds Have Other Ranges
  - External Standard Quantitation is Performed Using Average Response Factors from Initial Calibration
- Representative Compounds are Presented Here for Evaluation (Method 8261 Has Over 90 Target Analytes
- Most Compounds Exhibit RSD Values Less Than 20 Percent

Laboratory #1 Initial Calibration Summary

Analyte	Concentration Range (ug/L)	n	RSD
Vinyl chloride	1 – 50	5	11.4
Chloroform	1 – 50	5	10.6
Trichloroethene	1 – 50	5	15.1
Toluene	1 – 50	5	13.7
4-Methyl-2-pentanone	4 – 200	5	7.6
1,4-Dioxane	10 - 500	5	19.6
Pyridine	5 – 250	5	91.7
DBCP	1 – 50	5	10.8
1,4-Dichlorobenzene	1 – 50	5	13.8
Naphthalene	1 – 50	5	9.6
Nitrobenzene	2 – 100	5	31.6
Aniline	20 - 1000	5	7.1

Laboratory #2 Initial Calibration Summary

Analyte	Concentration Range (ug/L)	n	RSD
Vinyl chloride	1 – 50	5	6.2
Chloroform	1 – 50	5	3.6
Trichloroethene	1 – 50	5	3.7
Toluene	1 – 50	5	6.1
4-Methyl-2-pentanone	4 – 200	5	18.7
1,4-Dioxane	10 - 500	5	23.0
Pyridine	5 – 250	5	90.2
DBCP	1 – 50	5	16.1
1,4-Dichlorobenzene	1 – 50	5	9.8
Naphthalene	1 – 50	5	12.9
Nitrobenzene	2 – 100	5	39.5
Aniline	20 - 1000	5	23.8

Method 8261 Interlaboratory Study (Cont'd)

- Method Detection Limit (MDL) Study
  - Laboratory #1 and Laboratory #2 Performed MDL Studies on Both 5 mL Water and 5 gram Soil Samples
  - Laboratory #2 Also Performed MDL Study Using Low Concentration 25 mL Samples
  - Most Compounds Exhibited Good Percent Recoveries
  - Most Compounds Exhibited MDLs Less Than 1 ug/L (ppb)
  - Most Compounds Exhibited Good Precision Indicated by Low RSD Values
  - MDL Values are Comparable to Previously Reported Method 8261 MDL Values

Laboratory #1 Water MDL Sample Set Summary (5mL Sample; n=8)

Analyte	Spike Conc. (ug/L)	Avg % Rec.	SD	RSD	EST. MDL (ug/L)
Vinyl chloride	2	99	0.24	12.1	0.72
Chloroform	2	101	0.11	5.4	0.33
Trichloroethene	2	99	0.13	6.7	0.39
Toluene	2	102	0.24	11.7	0.72
4-Methyl-2-pentanone	8	94	0.76	10.1	2.3
1,4-Dioxane	20	79	3.9	24.6	11.6
Pyridine	10	9.2	0.71	77.2	2.1
DBCP	2	94	0.18	9.8	0.55
1,4-Dichlorobenzene	2	99	0.092	4.6	0.28
Naphthalene	2	98	0.086	4.4	0.26
Nitrobenzene	4	77	0.52	16.9	1.6
Aniline	40	129	19.6	38.0	58.8

Laboratory #1 Soil MDL Sample Set Summary (5 gram Sample; n=8)

Analyte	Spike Conc. (ug/Kg)	Avg % Rec.	SD	RSD	EST. MDL (ug/Kg)
Vinyl chloride	1	124	0.12	9.4	0.35
Chloroform	1	108	0.057	5.3	0.17
Trichloroethene	1	114	0.041	3.6	0.12
Toluene	1	106	0.039	3.7	0.12
4-Methyl-2-pentanone	4	100	0.46	11.5	1.4
1,4-Dioxane	10	186	3.1	16.7	9.4
Pyridine	5	1390	48.2	69.4	145
DBCP	1	108	0.072	6.7	0.22
1,4-Dichlorobenzene	1	100	0.067	6.8	0.20
Naphthalene	1	118	0.071	5.9	0.21
Nitrobenzene	2	110	0.39	17.9	1.2
Aniline	20	144	5.8	20.0	17.2

Laboratory #2 Water MDL Sample Set Summary (5 mL Sample; n=7)

Analyte	Spike Conc. (ug/L)	Avg % Rec.	SD	RSD	EST. MDL (ug/L)
Vinyl chloride	1	83	0.036	4.4	0.11
Chloroform	1	91	0.037	4.1	0.12
Trichloroethene	1	87	0.036	4.2	0.12
Toluene	1	100	0.076	7.6	0.24
4-Methyl-2-pentanone	4	82	0.15	4.5	0.46
1,4-Dioxane	10	113	2.0	17.9	6.3
Pyridine	30	93	9.2	33.1	29.1
DBCP	1	61	0.077	12.6	0.24
1,4-Dichlorobenzene	1	90	0.061	6.8	0.19
Naphthalene	1	100	0.11	10.6	0.33
Nitrobenzene	2	26	0.30	58.1	0.95
Aniline	120	126	39.3	26.4	124

Laboratory #2 Soil MDL Sample Set Summary (5 gram Sample; n=7)

Analyte	Spike Conc. (ug/Kg)	Avg % Rec.	SD	RSD	EST. MDL (ug/Kg)
Vinyl chloride	1	75	0.041	5.4	0.13
Chloroform	1	87	0.029	3.3	0.09
Trichloroethene	1	88	0.042	4.8	0.13
Toluene	1	100	0.037	3.7	0.12
4-Methyl-2-pentanone	4	83	0.24	7.2	0.76
1,4-Dioxane	10	87	1.82	20.9	5.7
Pyridine	30	59	1.03	5.8	3.2
DBCP	1	71	0.14	19.1	0.43
1,4-Dichlorobenzene	1	91	0.057	6.3	0.18
Naphthalene	1	106	0.12	10.9	0.36
Nitrobenzene	2	38	0.29	38.9	0.92
Aniline	120	ND	ND	ND	ND

Laboratory #2 Low Concentration Water MDL Sample Set Summary (25 mL Sample; n=7)

Analyte	Spike Conc. (ug/L)	Avg % Rec.	SD	RSD	EST. MDL (ug/L)
Vinyl chloride	0.4	83	0.014	4.4	0.05
Chloroform	0.4	91	0.015	4.1	0.05
Trichloroethene	0.4	87	0.015	4.2	0.05
Toluene	0.4	100	0.03	7.6	0.10
4-Methyl-2-pentanone	1.6	82	0.058	4.5	0.18
1,4-Dioxane	4	113	0.80	17.7	2.5
Pyridine	12	93	3.69	33.1	11.6
DBCP	0.4	61	0.03	12.6	0.10
1,4-Dichlorobenzene	0.4	90	0.024	6.7	0.08
Naphthalene	0.4	100	0.042	10.6	0.13
Nitrobenzene	0.8	26	0.12	58.1	0.38
Aniline	48	126	15.7	26.1	49.4

Method 8261 Interlaboratory Study (Cont'd)

- Performance Spike Analysis
  - Laboratories Requested to Analyze Replicate Performance Spike Samples at Three Concentrations in Water and Soil
  - Laboratories Requested to Analyze Replicate Performance Spike Samples at a Single Concentration in Salt Water
  - Laboratories Requested to Analyze Replicate Performance Spike Samples at a Single Concentration in Glycerol/Water
  - Average Percent Recoveries Indicate High Level of Accuracy for Most Analytes
  - Average RSD Values Indicate Good Precision for Most Analytes

Laboratory #1 Water Performance Spike Summary 1, 10, & 50 ppb Sample Sets (n=9)

Analyte	Low Conc. Avg % Rec.	Med. Conc. Avg % Rec.	High Conc. Avg % Rec.	Avg % Rec.	RSD
Vinyl chloride	113	116	107	112	4.2
Chloroform	96	105	99	100	4.4
Trichloroethene	95	107	102	101	3.2
Toluene	88	106	104	99	4.3
4-Methyl-2-pentanone	64	103	85	84	17.7
1,4-Dioxane	128	103	114	115	6.4
Pyridine	965	527	119	564	57.6
DBCP	77	100	81	86	19.1
1,4-Dichlorobenzene	82	114	97	97	5.6
Naphthalene	101	106	89	99	13.1
Nitrobenzene	75	84	87	82	35.8
Aniline	250	135	101	162	18.4

Laboratory #2 Water Performance Spike Summary 1, 3, & 5 ppb Sample Sets (n=12)

Analyte	Low Conc. Avg % Rec.	Med. Conc. Avg % Rec.	High Conc. Avg % Rec.	Avg % Rec.	RSD
Vinyl chloride	75	76	116	89	20.2
Chloroform	89	83	99	90	7.0
Trichloroethene	92	82	96	90	6.7
Toluene	119	84	96	100	5.7
4-Methyl-2-pentanone	75	84	88	82	10.4
1,4-Dioxane	327	145	178	217	13.9
Pyridine	ND	55	48	51	55.6
DBCP	64	82	87	78	16.6
1,4-Dichlorobenzene	93	85	96	91	6.4
Naphthalene	98	93	103	98	9.5
Nitrobenzene	ND	60	77	69	33.0
Aniline	ND	125	179	152	49.3

Laboratory #1 Soil Performance Spike Summary 1, 10, & 50 ppb Sample Sets (n=9)

Analyte	Low Conc. Avg % Rec.	Med. Conc. Avg % Rec.	High Conc. Avg % Rec.	Avg % Rec.	RSD
Vinyl chloride	99	98	98	98	6.3
Chloroform	97	103	100	100	5.3
Trichloroethene	100	104	102	102	5.0
Toluene	91	103	101	98	6.1
4-Methyl-2-pentanone	60	96	119	92	14.6
1,4-Dioxane	137	103	122	121	21.0
Pyridine	16	45	239	100	78.0
DBCP	81	92	135	102	13.2
1,4-Dichlorobenzene	86	105	100	97	7.3
Naphthalene	93	91	130	105	15.7
Nitrobenzene	80	79	179	112	29.5
Aniline	230	141	76	149	23.7

Laboratory #2 Soil Performance Spike Summary 1, 3, & 5 ppb Sample Sets (n=12)

Analyte	Low Conc. Avg % Rec.	Med. Conc. Avg % Rec.	High Conc. Avg % Rec.	Avg % Rec.	RSD
Vinyl chloride	74	74	101	83	6.9
Chloroform	83	82	104	90	6.3
Trichloroethene	83	81	99	88	6.7
Toluene	100	89	101	97	10.2
4-Methyl-2-pentanone	82	82	99	88	7.9
1,4-Dioxane	156	127	166	150	21.0
Pyridine	134	67	38	80	57.5
DBCP	85	85	98	89	14.5
1,4-Dichlorobenzene	82	82	100	88	6.0
Naphthalene	101	90	109	100	7.3
Nitrobenzene	43	60	83	62	13.7
Aniline	ND	ND	189	189	25.2

Laboratory #1 Salt Water Performance Spike Summary

Analyte	Spike Conc. (ug/L)	Avg Conc. (ug/L)	Avg % Recovery	RSD	n
Vinyl chloride	50	48	95	2.2	3
Chloroform	50	49	98	1.2	3
Trichloroethene	50	51	102	1.4	3
Toluene	50	50	100	0.9	3
4-Methyl-2-pentanone	200	198	99	5.7	3
1,4-Dioxane	500	379	76	14.2	3
Pyridine	250	249	99	18.0	3
DBCP	50	48	95	8.1	3
1,4-Dichlorobenzene	50	49	97	3.4	3
Naphthalene	50	46	92	6.4	3
Nitrobenzene	100	86	86	18.1	3
Aniline	1000	744	74	23.0	3

Laboratory #2 Salt Water Performance Spike Summary

Analyte	Spike Conc. (ug/L)	Avg Conc. (ug/L)	Avg % Recovery	RSD	n
Vinyl chloride	5	4.2	83	8.2	3
Chloroform	5	4.7	94	4.2	3
Trichloroethene	5	4.4	87	6.5	3
Toluene	5	4.4	88	5.3	3
4-Methyl-2-pentanone	20	20	98	5.9	3
1,4-Dioxane	50	82	163	6.9	3
Pyridine	25	29	115	12.4	3
DBCP	5	5.2	103	14.2	3
1,4-Dichlorobenzene	5	4.6	92	5.8	3
Naphthalene	5	5.4	109	10.4	3
Nitrobenzene	10	94	94	6.8	3
Aniline	200	180	90	14.1	3

Laboratory #1 Glycerol/Water Performance Spike Summary

Analyte	Spike Conc. (ug/L)	Avg Conc. (ug/L)	Avg % Recovery	RSD	n
Vinyl chloride	50	51	101	9.3	3
Chloroform	50	51	101	4.1	3
Trichloroethene	50	51	103	4.8	3
Toluene	50	50	101	5.1	3
4-Methyl-2-pentanone	200	206	103	1.6	3
1,4-Dioxane	500	732	147	19.2	3
Pyridine	250	58	23	31.6	3
DBCP	50	49	98	0.75	3
1,4-Dichlorobenzene	50	51	103	3.0	3
Naphthalene	50	51	101	2.9	3
Nitrobenzene	100	72	72	7.5	3
Aniline	1000	2175	218	15.4	3

Laboratory #2 Glycerol/Water Performance Spike Summary

Analyte	Spike Conc. (ug/L)	Avg Conc. (ug/L)	Avg % Recovery	RSD	n
Vinyl chloride	5	4.1	82	6.1	3
Chloroform	5	4.9	98	3.1	3
Trichloroethene	5	4.4	87	5.5	3
Toluene	5	4.8	96	5.6	3
4-Methyl-2-pentanone	20	16	78	7.8	3
1,4-Dioxane	50	84	169	8.5	3
Pyridine	25	19	76	26.9	3
DBCP	5	3.6	72	18.3	3
1,4-Dichlorobenzene	5	4.4	89	2.9	3
Naphthalene	5	4.5	90	7.9	3
Nitrobenzene	10	5.8	58	17.6	3
Aniline	200	376	188	18.1	3

Method 8261 Interlaboratory Study (Cont'd)

- Blind Sample Analysis
  - Laboratories Were Supplied With Five Blind Ampulated Samples for Replicate Analysis in Both Water and Soil Matrix
  - Each Blind Sample Contained Fifteen Target Analytes at Various Concentrations
  - Recoveries Were Compared to Historically Based Acceptance Limits (HBAL) Database Maintained by QATS
  - Results Indicate That Most Analytes Exhibit Comparable Recovery and Precision to HBAL Statistics
  - Results Indicate That Most Analyte Recoveries Fall Within the HBAL Acceptance Limits
  - Blind Soil Spike Analysis Results (Not Presented) are Comparable to the Blind Water Spike Results

Method 8261 Blind #1 Composite Results Summary 5 mL Water Samples (n=5)

Target Analyte	Spike (ug/L)	Avg % Rec.	HBAL % Rec.	Avg RSD	HBAL RSD
Vinyl Chloride	45	118	83	32.5	20.2
1,1-Dichloroethane	48	105	97	5.5	10.7
Benzene	1	132	100	8.9	9.6
1,4-Dioxane	375	95	NA	11.4	NA
4-Methyl-2-pentanone	175	105	99	9.6	18.5
N-Nitrosodimethylamine	90	NR	NA	NR	NA
1,2-Dibromoethane	43	103	107	9.4	7.7
N-Nitrosodiethylamine	940	56	NA	47.5	NA

Method 8261 Blind #1 Composite Results Summary 5 mL Water Samples (n=5) (Cont'd)

Target Analyte	Spike (ug/L)	Avg % Rec.	HBAL % Rec.	Avg RSD	HBAL RSD
1,2,4-Trimethylbenzene	13	90	NA	10.6	NA
Aniline	225	18	NA	30.2	NA
1,4-Dichlorobenzene	12	94	98	6.7	11.2
Acetophenone	95	70	77	29.2	11.7
DBCP	1	97	104	19.0	12.2
Hexachlorobutadiene	9	94	56	8.0	26.7
Naphthalene	45	98	70	5.3	17.8

Method 8261 Blind #2 Composite Results Summary 5 mL Water Samples (n=5)

Target Analyte	Spike (ug/L)	Avg % Rec.	HBAL % Rec.	Avg RSD	HBAL RSD
Trichloroethene	35	108	102	3.5	10.5
Methyl methacrylate	18	103	NA	9.4	NA
1,4-Dioxane	55	102	NA	13.6	NA
Toluene	12	100	99	5.8	9.8
Pyridine	235	154	NA	151.3	NA
N-Nitrosodimethylamine	310	37	NA	3.5	NA
2-Hexanone	29	112	113	19.9	32.8
Chlorobenzene	8	103	99	5.8	8.5

Method 8261 Blind #2 Composite Results Summary 5 mL Water Samples (n=5) (Cont'd)

Target Analyte	Spike (ug/L)	Avg % Rec.	HBAL % Rec.	Avg RSD	HBAL RSD
Isopropylbenzene	33	102	82	7.6	6.5
Bromoform	45	113	97	6.2	13.1
1,1,2,2-Tetrachloroethane	12	95	98	9.6	11.0
N-Nitroso-di-n-propylamine	70	71	75	6.8	18.0
DBCP	5	100	104	14.1	12.2
Nitrobenzene	78	112	78	25.5	17.4
2-Methylnaphthalene	9	82	71	13.7	19.7

Method 8261 Blind #3 Composite Results Summary 5 mL Water Samples (n=5)

Target Analyte	Spike (ug/L)	Avg % Rec.	HBAL % Rec.	Avg RSD	HBAL RSD
Vinyl chloride	15	107	83	8.3	20.2
Trichloroethene	4	154	102	29.2	10.5
1,4-Dioxane	125	111	NA	7.8	NA
4-Methyl-2-pentanone	75	118	99	20.9	18.5
Tetrachloroethene	24	101	100	11.0	11.5
1,2-Dibromoethane	11	107	107	14.2	7.7
N-Nitrosodiethylamine	85	32	NA	3.9	NA
1,1,2,2-Tetrachloroethane	45	105	98	23.1	11.0

Method 8261 Blind #3 Composite Results Summary 5 mL Water Samples (n=5) (Cont'd)

Target Analyte	Spike (ug/L)	Avg % Rec.	HBAL % Rec.	Avg RSD	HBAL RSD
Aniline	800	18	NA	30.3	NA
1,4-Dichlorobenzene	1	127	98	44.9	11.3
N-Nitroso-di-n-propylamine	230	90	75	19.6	18.0
Nitrobenzene	9	113	78	31.6	17.4
1,2,4-Trichlorobenzene	44	99	97	16.5	12.5
Naphthalene	27	104	70	22.3	17.8
2-Methylnaphthalene	38	135	71	14.3	19.7

Method 8261 Blind #4 Composite Results Summary 5 mL Water Samples (n=5)

Target Analyte	Spike (ug/L)	Avg % Rec.	HBAL % Rec.	Avg RSD	HBAL RSD
Benzene	38	101	100	7.1	9.6
Methyl methacrylate	88	108	NA	11.8	NA
1,4-Dioxane	1	660	NA	24.0	NA
Toluene	43	106	99	7.2	9.8
Pyridine	95	246	NA	101.2	NA
N-Nitrosodimethylamine	740	25	NA	31.6	NA
2-Hexanone	85	115	114	22.6	32.9
1,2-Dibromoethane	0.5	114	107	5.0	7.7

Method 8261 Blind #4 Composite Results Summary 5 mL Water Samples (n=5) (Cont'd)

Target Analyte	Spike (ug/L)	Avg % Rec.	HBAL % Rec.	Avg RSD	HBAL RSD
Chlorobenzene	35	106	99	7.9	8.5
Bromoform	17	97	97	7.0	13.1
N-Nitrosodiethylamine	550	25	NA	37.8	NA
1,4-Dichlorobenzene	38	101	98	6.8	11.3
DBCP	46	116	104	19.5	12.2
Nitrobenzene	27	92	78	21.0	17.4
Naphthalene	6	103	70	9.5	17.8

Method 8261 Blind #5 Composite Results Summary 5 mL Water Samples (n=5)

Target Analyte	Spike (ug/L)	Avg % Rec.	HBAL % Rec.	Avg RSD	HBAL RSD
1,1-Dichloroethane	6	102	97	9.0	10.7
1,4-Dioxane	7	196	NA	24.2	NA
4-Methyl-2-pentanone	8	85	99	10.0	18.5
Toluene	1	131	99	43.9	9.8
Pyridine	21	31	NA	8.1	NA
Tetrachloroethene	42	99	100	7.2	11.5
1,2-Dibromoethane	1	92	107	0.8	7.7
Isopropylbenzene	3	67	82	15.7	6.5

Method 8261 Blind #5 Composite Results Summary 5 mL Water Samples (n=5) (Cont'd)

Target Analyte	Spike (ug/L)	Avg % Rec.	HBAL % Rec.	Avg RSD	HBAL RSD
1,2,4-Trimethylbenzene	22	95	NA	11.9	NA
Aniline	65	NR	NA	NR	NA
N-Nitroso-di-n-propylamine	860	107	75	39.6	18.0
Acetophenone	22	48	77	14.5	11.7
Hexachlorobutadiene	36	89	56	8.2	26.7
1,2,4-Trichlorobenzene	15	94	97	8.5	12.5
2-Methylnaphthalene	84	134	71	17.4	19.7

Method 8261 Interlaboratory Study (Cont'd)

- *Method 8261 VDU Study Check Surrogate Recovery Summary*
  - Twelve Check Surrogates Used to Monitor Performance of the Analytical System
  - Recoveries are Calculated in Same Manner as Target Analytes
  - Check Surrogate Recoveries from Performance Spike and Blind Sample Analyses Evaluated
  - Average Water Sample Recoveries are Between 78 and 111 Percent
  - Average Soil Sample Recoveries are Between 86 and 106 Percent
  - Most RSD Values are Below 20% Indicating Good Precision
  - No Acceptance Limits in Method 8261 – Composite Interlaboratory Results Can be Used to Create Advisory Limits

Interlaboratory Study Check Surrogate Recoveries Composite Blind & Performance Spike Water Samples

Surrogate	n	Min % Rec	Max % Rec	Avg % Rec	SD	RSD
Methylene chloride-d2	46	91	121	103	4.6	5.4
Benzene-d6	46	93	121	98	4.5	4.6
1,2-Dichloropropane-d6	46	86	109	102	4.2	4.1
1,1,2-Trichloroethane-d3	46	85	119	101	6.5	6.4
4-Bromofluorobenzene	46	92	112	98	4.0	4.1
Nitromethane-^{13}C	46	53	110	97	9.2	9.5
Ethyl acetate-^{13}C	46	74	113	97	8.9	9.1
Pyridine-d5	46	1.4	196	84	52.2	61.9
Decafluorobiphenyl	46	82	166	111	17.5	15.7
Nitrobenzene-d5	46	53	150	83	19.6	23.6
Acetophenone-d5	46	40	129	78	21.6	27.7
Naphthalene-d8	46	82	118	99	7.7	7.8

Interlaboratory Study Check Surrogate Recoveries Composite Blind & Performance Spike Soil Samples

Surrogate	n	Min % Rec	Max % Rec	Avg % Rec	SD	RSD
Methylene chloride-d2	46	75	176	102	13.3	13.0
Benzene-d6	46	93	110	99	3.0	3.0
1,2-Dichloropropane-d6	46	90	118	103	5.7	5.6
1,1,2-Trichloroethane-d3	46	77	160	106	17.7	16.7
4-Bromofluorobenzene	46	93	109	100	3.3	3.2
Nitromethane-^{13}C	46	77	110	96	6.7	7.0
Ethyl acetate-^{13}C	46	85	136	103	9.4	9.1
Pyridine-d5	46	24	278	99	62.4	62.7
Decafluorobiphenyl	46	78	164	104	20.3	19.5
Nitrobenzene-d5	46	54	230	98	34.3	34.9
Acetophenone-d5	46	34	238	86	36.7	42.6
Naphthalene-d8	46	86	149	106	12.2	11.5

Method 8261 Interlaboratory Study Summary

- Preliminary Results Indicate:
  - Acceptable Precision for Initial Calibration Analysis With RSD Values for Most Compounds Within Method Criteria
  - Acceptable Precision for CCV Analysis With Percent Difference Values for Most Compounds Within Method Criteria
  - Acceptable MDLs With Most Compound MDL Values Below 1 ug/L (ppb)
  - Acceptable Recovery and Precision in Performance Spike Analysis Using Various Matrices
  - Acceptable Recovery and Precision of Blind Sample Analytes Compared to HBAL Statistics

Method 8261 Interlaboratory Study Summary

- Results from Two Additional Laboratories Will be Processed to Help Evaluate Method and Instrumentation
- Applicability to CLP
  - Method 8261 May be Included for Non-Routine CLP Analysis in Near Future
  - Method 8261 May be Included as the CLP Expands Beyond the Realm of Superfund
  - Method 8261 May be Included as the CLP Continually Reviews the Needs of the USEPA Regions

Vacuum Distillation Unit Interlaboratory Study Evaluation

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