Supplement to:

The Role of Internal Standards and their Interaction with Soils Impact Accuracy of Volatile Organics Determinations

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Supplementary Information

Data that was too detailed for "The Role of Internal Standards and their Interaction with Soils Impact Accuracy of Volatile Organics Determinations" is presented as supplemental information. This information includes each analyte by matrix and includes the accuracy evaluation, analyte results impacted by criteria, and results from varying spike equilibration times. The list of internal standards and their chemical properties are presented in Table S1 while the surrogates are listed in Table S2.

Tables of results by analyte are included as Tables S3-8. These tables include the data by matrix and combined. A discussion of observations by matrix follows.

Sand samples

The acid washed sand samples (100% silicon dioxide from Research Chemicals Ltd, Heysham, Lancashire, England) were spiked by addition of the solutions to the glass vessel surface under the sand and the vessel was quickly attached to the vacuum distiller sealing the sample. After all samples in a batch were spiked, each sample vessel was removed from the distiller and 5 mL water was added. This was done quickly to limit loss of gases and there was no additional mixing of the sand.

Only the results for two analytes (dichlorofluoromethane, and hexachlorobutadiene) were outliers. Qualifying the gas analyte class when the surrogate vinyl chloride-d_3 recovery fell below 42.1 % eliminated the outlier, dichlorodifluoromethane.

There was not a QC parameter that related to hexachlorobutadiene being an outlier. Hexachlorobutadiene appears to be a compound that slowly equilibrated with the sand matrix. By extending the equilibration time to 20 hours, the compound met requirements with a recovery of 102 +/- 6%. The compound was not an outlier when analytes were added to sand after water was added.

There was one biased analyte, 2-hexanone, with an average recovery of 71 ± 9%. By adding the analytes to sand after water was added, the compound recovery was 93 ± 4%.

The analyte recoveries for the sand samples are presented in Table S4. The average ambient concentration of analyte in the sand is presented. The results that failed mixing criteria and the results that failed other QC criteria are presented along with those results that passed all criteria. Also presented are the data resulting from spiking the sand after the diluting water was added.

Nevada mountain soil (NV)

The NV is a mountain topsoil (61 % sand, 31% silt, 8% clay and 3.8% organic carbon) had absorbed volatiles present in the sample storage environment. A criterion for not including analytes for a matrix due to contamination was to exclude an analyte when its concentration (or the presence of an interference) in the matrix was more than 5% of the medium spike amount (based on 5 g samples).

The spiking of the Nevada mountain soil was done as for sand but with an additional shaking of the soil to disperse the spikes through the soil before adding water.

Applying the initial quality controls, the mixing criteria, the vinyl chloride-d_3 minimum recovery of 42.1% found for sand matrix, and eliminating determinations that did not meet these criteria, there were no outliers. There were 7 biased analyte recoveries after criteria met (Table S5).

Spiking the water phase after adding 10 mL water to 5 g NV soil yielded more uniform data with only 5 biased recovery averages and all but one were semivolatile (the other being dichlorodifluoromethane). Spiking the water phase and allowing equilibration overnight improved the results marginally and some analytes apparently began to degrade.

Table S5 presents the results by analyte for the NV soil as for sand with results for overnight spiking and degradation over an extended time.

Georgia clay samples (GA)

The GA soil is a subsurface soil from Hayesville (46% sand, 22% silt, 32% clay, and 0.2% organic carbon). After observing potential for an uneven mixing of internal standards and analytes in the Nevada samples, there was a more vigorous mixing of the clay samples. While the samples were spiked as sand, the dry clay samples were shaken lightly while dry to mix the spike throughout the sample and then attached to the vacuum distiller. An amount of water equal to the sample size was added to the clay and more manual mixing was done vigorously. Mixing was done by hand to ensure the clumping clay was dispersed and slurry resulted.

After applying the mix criteria (analytes and labeled analogs recovery must be within 25% of each other) and basic QC the volatile gases, 1,1-dichloroethene, and carbon disulfide were outliers. There were also 5 outliers and all but one semivolatile.

The difficulty preparing spikes in the clay samples did cause loss of the gases. The gas outliers were eliminated by providing a lower recovery limit for vinylchloride-d_3 at 88.1% which also eliminated 34 of 36 analyses. A study was conducted to verify that gases would perform well if mixing was done while the system sealed. For this study, spikes (high concentration) were added to 5 g of the matrix as was done with the sand (no mixing) and then 10 ml water and a magnetic stir bar (12.7 by 3mm) added and the sample connected to the distiller. Mixing was performed using a micro-thin stirrer held under the sample while connected to the distiller. With the precaution of a closed system during mixing, the gases were effectively recovered. Only dichlorodifluoromethane was not characterized well with the vinyl chloride surrogate. For the closed system mixing, the vinyl chloride recovery range narrowed greatly (85.9 to 98.6% recovery).

When the Georgia clay was spiked after water was added carbon disulfide and 1,1-dichloroethene behaved well initially and were not outliers. However, the response of 1,2-dichloroethene dropped after 6 hours and became an outlier. Also if the spikes were added to the dry clay and water added with an overnight equilibration period, carbon disulfide was still not an outlier while 1,1-dichloroethene was an outlier.

Table S6 lists analyte results for the Georgia soil. Results documenting the improvement due to attaching the sample vessel to the distiller prior to mixing are also present. In addition results for adding spike to soil after diluting with water is presented.

Oregon soil (OR)

The OR soil is a topsoil from Willamette Valley (66% sand, 8% silt, 27% clay, and 4.9% organic carbon). The Oregon soil samples were spiked as the Georgia clay was spiked. As with the mountain soils, there was elevated organic content and the samples was contaminated with some of the analytes during storage.

There was only one outlier, dibromomethane but there were 14 biased analytes (average results greater than 125% or less than 75% of true values. There was no QC criterion that could be related to the outlier status. There was no clear difference in results for the different sample sizes (5, 10 and 15 g). As there were discrepancies in bias for the results and the extended equilibration times for the reactivity studies a look at the various means of introducing spikes was required.

When the Oregon soil was spiked after adding water with same-day analyses, dibromomethane was no longer an outlier nor were its average biased (81±3%). Also the analytes with biased results were far fewer (and of 6 remaining, 4 were semivolatile, Table S7). When the Oregon soil was spiked after adding water with an overnight equilibration before analyses, dibromomethane was no longer an outlier. Also the number of analytes with biased results was less (5 of which 4 were of the semivolatile

class). Extending the equilibration time with water added yielded results very similar to adding spikes to the water phase with extended equilibration.

Greenwich Bay sediment (SD)

The wet sediment (46% water) was very viscous and heterogeneous. Taking an aliquot, spiking, and mixing were difficult due to the sticky nature of the matrix. Spikes were added after adding an equal volume of water to the sample in the distilling flask followed by mixing. The mixing consisted of swirling the sample vigorously while immersing the flask in a sonic bath until clumps of sediment were no longer visible. The sediment was quite dark and the samples could not be verified as mixed thoroughly.

Whereas all of the soil samples had been spiked before adding water, the sediment was spiked by adding the spike to the slurry.

Utilizing the QC derived from the soil samples (sand, GA, NV and OR) there were six outliers, vinyl chloride, acetonitrile, bromochloromethane, methyl cyclohexane, dibromomethane, and 1,1,2-trichloroethane.

Vinyl chloride could be eliminated as an outlier if gases determinations were omitted when the surrogate vinyl chloride-d_3 fell below 55.8% recovery. The lower recovery for vinyl chloride-d_3 for spikes added to the soils after water was added was 76.8%.

Methyl cyclohexane behaved well with a more thorough mixing or an overnight equilibration.

Surprisingly the surrogate 1,1,2-trichloroethane-d3 recovery could not be limited and ensure that its analog was not an outlier.

The non-purgeable acetonitrile could not be linked to an internal standard or surrogate QC parameter. It was observed that the check standards for acetonitrile varied more than for any other analyte in the study (70% during the sediment analyses and 45% over the entire study). A 25% limit on the continuing calibration difference from the initial calibration would remove the analyte from being an outlier.

Table S8 presents the results by analyte for the sediment. Also included are results from spiking the matrix with the improved mixing and overnight equilibration.

Reviewing the recovery of relative volatility internal standards that bracketed the compound it was observed that there was a great disparity between the internal standards diethyl ether-d_{10} (relative volatility 32.5) and 1,2-dibromoethane-d_4 (relative volatility 26). Normally there is little difference and this was directly related to the outlier status, but when the recovery of diethyl ether-d_{10} is more than 162% of 1,2-dibromoethane-d_4 there was an outlier. Therefore not including results for compounds associated with these

internal standards when their relative recovery exceeded 162% eliminated the outlier status (and eliminated 30 of 36 analyses). Therefore, noting that the variation between the two internal standards is likely linked to the outlier status the control was not used.

The relationship of organic content and equilibration time on monitoring internal standards is presented in Table S9. Only the 1,2-dichlorobenzene-d_4 data from spiking soil prior to adding water was discussed in the article as an example of the general trend.

Table S1. Internal Standards used for Method 8261A Quantitation

Range		Grouping	
Type	Values	Internal standards	Value[a]
Relative volatility	0.62 to 3.72	methyl cyclohexane- d_{14}	.62
		hexafluorobenzene	.86
		ethylbenzene-d_{10}	3.6
		1,4-difluorobenzene[b]	3.83
	3.72 to 6.20	ethylbenzene-d_{10}	3.6
		1,4-difluorobenzene	3.83
		o-xylene-d_{10}	6.14
		chlorobenzene-d_5[b]	6.27
	6.2 to 29.2	*o*-xylene-d_{10}	6.14
		chlorobenzene-d_5	6.27
		1,2-dibromoethane-d_4	26.
		diethyl ether-d_{10}	32.5
	29.2 to 478	1,2-dibromoethane-d_4	26.
		diethyl ether-d_{10}	32.5
		tetrahydrofuran-d_8	355
		acetone-d_6	600
	478 to 5800	tetrahydrofuran-d_8	355
		acetone-d_6	600
		1,4-dioxane-d_8	5800
	5800 to 14400	acetone-d_6	600
		1,4-dioxane-d_8	5800
		2-chloroethanol-d_4	13800
Boiling point	85 to 155 (°C)	pentafluorobenzene	85(°C)
		toluene-d_8	111
		bromobenzene-d_5	155
	155 to 181	toluene-d_8	111
		bromobenzene-d_5	155
		1,2-dichlorobenzene-d_4	181
	181 to 218	1,2-dichlorobenzene-d_4[b]	181
		napththalene-d_8	217
		1,2,3-trichlorobenzene-d_3[b]	218
	218 to 243.5	napththalene-d_8	217
		1,2,3-trichlorobenzene-d_3	218
		1-methylnaphthalene-d_{10}	241
		3,5-dibromotoluene	246

[a] Values are from references 11.

[b] Internal standards from Table 1 of Method 8260C [8]. 1,2-Dichlorobenzene-d_4 is a substitute for 1,4-dichlorobenzene-d_4. 1,2,3-Trichlorobenzene-d_3 was added as an internal standard for semivolatile analytes.

Table S2. Surrogate Compounds by Class

	Boiling Point	Relative Volatility[a]
Volatile Class (boiling point less than 159)		
vinyl chloride-d_3	-13	.48
methylene chloride-d_2	40	11.10
benzene-d_6	79	3.92
1,2-dichloropropane-d_6	95	11.00
1,1,2-trichloroethane-d_3	112	26.6
4-bromofluorobenzene	152	8.05
Non-Purgeable Class (rel vol>100)		
nitromethane-^{13}C	101	510
ethylacetate-^{13}C	77	150
pyridine-d_5	115	15000
Semi-Volatile Class (boiling point >= 159		
decafluorobiphenyl	206	3.03
nitrobenzene-d_5	210	87.5
acetophenone-d_5	202	161
1,2,4-trichlorobenzene-d_3	213	7.88
a,*a*-dichloro-*o*-xylene	240	113.5
azulene	242	91
3,5-*di*-*tert*-butyltoluene	244	3.61

[a] Values are from reference 11.

Table S3. Study Analytes

																Methods Comparison[g]				
	Relative Volatility[a]	Boiling Point[b]	Calib Range[c]	Degradation by Soil[d] (% loss)				Soils[e]			Sediment[f]					Method 8261A		Method 8260C		
Compound		°C	ngs	GA	NV	OR	SD	#	avg	dev	#	avg	dev	#	avg	dev	avg	dev		
dichlorodifluoromethane	0.07	-30	5 - 500					28	70	29	9	77	11	11	149	63	132	56		
chloromethane	1.37	-24	5 - 500					12	68	11	0	NA		16	113	22	111	21		
vinylchloride	0.48	-13	5 - 500					63	91	13	9	90	14	28	98	9	87	9		
bromomethane	1.82	4	5 - 500		79	62	100	75	87	14	0			22	91	9	90	10		
chloroethane	1.01	12	5 - 500					35	85	31	0	NA		28	82	20	77	20		
trichlorofluoromethane	0.20	24	5 - 500					46	108	14	9	99	12	28	99	11	89	11		
diethyl_ether	34.9	35	10 - 1000					0	NA		0	NA		0	NA		NA			
1,1,2-trichloro-1,2,2-trifluoroethane	0.40	48	5 - 500					108	113	20	34	102	6	28	101	8	90	8		
acetone	600.	56	10 - 1000					0	NA		0	NA		0	NA		NA			
1,1-dichloroethene	0.63	37	5 - 500					72	95	12	34	102	9	28	89	15	80	14		
iodomethane	2.29	42	10 - 1000		92	84	100	107	94	15	0	NA		22	94	10	94	10		
allylchloride	1.34	45	5 - 500				99	36	96	8	0	NA		17	98	18	95	17		
acetonitrile	545.	82	20 - 2000		100	99		36	104	16	18	61	9	11	77	10	115	10		
methyl acetate	222.	57	5 - 500		97		100	66	109	25	0	NA		5	65	22	107	35		
carbon disulfide	0.31	46	5 - 500		68	82	79	107	88	17	0	NA		22	91	21	82	18		
methylene chloride	10.1	40	5 - 500					143	86	9	0	NA		28	89	16	105	18		
MTBE	33.7	55	5 - 500					72	108	5	34	109	4	28	90	17	124	21		
acrylonitrile	161.	78	10 - 1000		100	100	98	144	98	15	0	NA		22	94	9	138	15		
trans-1,2-dichloroethene	2.30	48	5 - 500				33	143	90	11	34	92	7	28	90	7	90	8		
1,1-dichloroethane	4.12	57	5 - 500					142	90	15	34	95	6	28	91	6	96	8		
2,2-dichloropropane	1.37	69	5 - 500				35	143	115	20	34	103	5	28	99	7	95	8		
propionitrile	1420.	97	10 - 1000		100	100	43	102	108	10	24	133	26	28	114	11	148	27		

2-butanone	770.	80	20 - 2000			32	0	NA		0	NA		6	106	7	169	11
cis-1,2-dichloroethene	5.34	60	5 - 500				143	93	11	34	101	4	28	94	6	102	3
methacrylonitrile	103.	90	10 - 1000	99	100		72	116	6	36	121	17	28	91	14	131	16
chloroform	6.39	62	5 - 500				143	92	10	34	102	4	28	96	4	106	4
bromochloromethane	15.4	68	5 - 500			70	143	94	14	34	142	6	28	99	12	123	16
cyclohexane	0.59	81	5 - 500				143	112	25	34	98	8	28	100	7	89	6
1,1,1-trichloroethane	1.31	74	5 - 500			39	108	116	9	34	104	6	28	102	4	98	6
1,1-dichloropropene	0.88	104	5 - 500				143	115	24	34	107	6	28	103	8	91	3
carbon tetrachloride	0.64	77	5 - 500			88	143	115	24	0	NA		22	103	7	94	6
1,2-dichloroethane	18.7	84	5 - 500				143	89	12	34	99	3	28	91	5	117	9
benzene	3.55	80	5 - 500		84	51	72	92	3	34	97	3	28	94	17	97	18
trichloroethene	2.34	87	5 - 500				72	103	3	34	112	5	28	98	10	99	7
methyl cyclohexane	0.62	101	5 - 500				108	111	12	0	NA		28	100	10	87	9
1,2-dichloropropane	10.9	96	5 - 500				143	96	7	34	102	3	28	92	5	108	5
methylmethacrylate	71.4	101	10 - 1000	100	95	100	140	98	15	34	92	11	28	95	10	130	12
dibromomethane	23.9	97	5 - 500			89	107	111	12	34	155	9	28	99	18	129	26
bromodichloromethane	12.3	90	5 - 500	77	52	100	143	94	18	0	NA		22	91	10	110	4
1,4-dioxane	5750.	101	50 - 5000				107	90	19	36	110	14	28	101	6	97	28
4-methyl-2-pentanone	120.	117	20 - 2000				36	104	12	36	158	32	16	119	25	172	29
trans-1,3-dichloropropene	14.1	112	25 - 2500	82	85	100	107	105	9	0	NA		22	92	6	105	7
toluene	3.88	111	5 - 500				36	90	4	34	98	4	17	102	3	100	3
cis-1,3-dichloropropene	19.6	104	25 - 2500	87	81	100	72	111	5	0	NA		22	86	12	115	8
2-hexanone	131.	128	10 - 1000	97	78		36	71	9	36	113	29	11	110	18	159	15
1,1,2-trichloroethane	26.2	114	5 - 500				143	99	12	34	130	10	28	99	8	133	13
1,3-dichloropropane	24.9	120	5 - 500				143	99	13	34	117	5	28	100	7	130	12
tetrachloroethene	1.43	121	5 - 500				72	102	3	34	117	9	28	104	10	97	12
dibromochloromethane	19.2	120	5 - 500	86	52	100	143	99	16	0	NA		22	95	6	121	7

1,2-dibromoethane	26.7	132	5 - 500				95	143	99	13	34	98	5	28	100	6	125	11
chlorobenzene	6.07	132	5 - 500					143	87	11	34	104	4	28	95	7	97	6
1,1,1,2-tetrachloroethane	11.6	131	5 - 500				100	143	98	7	0	NA		22	98	6	111	8
ethylbenzene	3.60	136	5 - 500					36	96	3	34	102	5	22	101	5	95	4
m,p-xylenes	3.91	138	5 - 500					36	90	4	34	102	5	17	99	3	94	4
o-xylene	5.54	144	5 - 500					36	95	3	34	100	4	22	101	6	97	4
styrene	6.87	145	5 - 500	35		97	82	72	98	6	0	NA		22	91	9	90	11
isopropylbenzene	2.75	152	5 - 500					72	95	4	34	97	8	28	106	16	91	10
bromoform	23.4	150	5 - 500		89		100	143	111	11	0	NA		22	104	7	117	11
cis-1,4-dichloro-2-butene	33.3	152	20 - 2000		100	99	100	143	103	20	0	NA		22	102	7	117	12
1,1,2,2-tetrachloroethane	30.3	146	5 - 500					108	100	11	34	132	10	28	103	7	125	15
1,2,3-trichloropropane	33.6	157	5 - 500					0	NA		34	153	12	16	123	18	206	60
propylbenzene	2.43	159	5 - 500					20	99	4	27	104	9	28	110	19	125	41
bromobenzene	7.89	156	5 - 500					143	86	14	34	107	5	28	96	10	124	21
trans-1,4-dichloro-2-butene	33.8	156	20 - 2000		100	100	100	142	89	14	0	NA		22	102	8	161	43
1,3,5-trimethylbenzene	3.75	165	5 - 500					20	93	4	27	98	7	22	102	10	107	22
2-chlorotoluene	4.04	159	5 - 500					75	98	10	27	102	6	28	101	9	119	28
4-chlorotoluene	4.78	162	5 - 500					105	82	11	27	102	7	28	98	9	113	21
tert-butylbenzene	2.72	169	5 - 500					105	122	28	27	102	9	28	115	27	120	40
sec-butylbenzene	1.91	173	5 - 500					54	93	5	27	100	13	28	117	31	114	36
pentachloroethane	13.20	162	5 - 500		83		100	105	97	12	0	NA		22	96	10	129	28
1,2,4-trimethylbenzene	4.50	169	5 - 500					20	88	5	27	99	7	17	100	6	99	6
p-isopropyltoluene	2.50	183	5 - 500					0	NA		27	103	12	17	105	10	92	6
1,3-dichlorobenzene	5.72	173	5 - 500					103	81	10	27	91	6	28	94	10	99	12
1,4-dichlorobenzene	6.14	174	5 - 500				32	54	91	5	27	91	6	28	93	10	98	9
n-butylbenzene	1.88	183	5 - 500				57	20	89	7	27	95	14	28	118	34	102	30
1,2-dichlorobenzene	7.86	180	5 - 500					102	95	12	27	93	5	28	94	10	97	9

acetophenone	161.	203	10 - 1000		88		8	109	22	27	140	25	11	142	66	164	49
1,2-dibromo-3-chloropropane	38.9	196	5 - 500			99	82	166	50	27	132	14	28	146	27	268	104
nitrobenzene	87.5	211	10 - 1000	88	82	97	54	112	14	0	NA		16	131	22	198	51
1,2,4-trichlorobenzene	7.73	214	5 - 500				73	76	11	27	76	9	28	85	13	97	13
hexachlorobutadiene	2.08	215	5 - 500				66	117	27	27	88	21	28	128	60	125	55
naphthalene	16.70	218	5 - 500				20	106	8	27	117	10	17	107	9	135	16
1,2,3-trichlorobenzene	11.30	218	5 - 500				84	72	12	21	74	9	28	80	13	91	14
2-methylnaphthalene	67.00	245	10 - 1000				20	98	21	27	91	11	17	92	8	111	22
1-methylnaphthalene	67.00	245	10 - 1000				0	NA		27	104	14	28	85	13	92	34

[a] Relative volatility values from reference 11.
[b] Boiling points from reference 11.
[c] Calibration range in amounts of compound.
[d] Degradation of compounds observed during extended period as a percent loss. Compounds that have no entry were not observed to degrade with overnight equilibration, No compounds were observed to degrade in contact with sand. GA equilibration time was 121 hr. NV equilibration time was 119 hr. OR equilibration time of 121 hr. SD equilibration time was 119 hr.
[e] The combined results for each analyte in the soil matrices after criteria met.
[f] The results for each analyte in sediment after criteria met
[g] The results for the optimized spiking in samples (six samples for each matrix) after dilution with water. Results are from processing raw data by Methods 8260C and 8261A.
[h] There were no results that passed criteria.

Table S4 Acid-washed sand results by analyte

	conc[a]		pass criteria[b]			fail mix criteria[c]			fail QC criteria[d]			wet spike[e]				Dry spike[f]	
	% of			% recovery			% recovery			% recovery		3 hr		114 hr		20 hr	
Compound	spike	run	#	avg	dev	#	avg	dev	#	avg	dev	avg	dev	avg	dev	avg	dev
dichlorodifluoromethane	0.3	36	16	75	33	0			20	44	17	NA		118	13	171	30
chloromethane	0.9	0	0	NA[g]		0			0			137	7	113	3	160	5
vinylchloride	0.0	36	22	89	10	0			14	65	5	86	4	89	1	86	11
bromomethane	0.2	36	22	90	9	0			14	70	4	85	3	84	3	71	5
chloroethane	0.2	36	0	NA		0			36	80	23	97	10	107	6	77	8
trichlorofluoromethane	0.1	36	22	97	7	0			14	85	6	92	3	99	3	100	11
diethyl ether	8.9	0	0	NA		0			0			NA		84	5	NA	
1,1,2-trichloro-1,2,2-trifluoroethane	0.0	36	36	95	5	0			0			95	4	100	1	108	10
acetone	38.2	0	0	NA		0			0			NA		108	3	NA	

1,1-dichloroethene	0.3	36	36	87	9	0	0			86	5	98	3	112	11
iodomethane	0.0	36	36	96	7	0	0			98	7	95	3	77	3
allylchloride	0.2	36	36	96	8	0	0			93	19	114	12	39	8
acetonitrile	0.2	36	36	104	16	0	0			68	9	130	50	72	3
methyl acetate	4.7	36	36	109	25	0	0			65	22	117	21	8	1
carbon disulfide	0.0	36	36	93	9	0	0			78	17	103	6	63	9
methylene chloride	1.7	36	36	85	5	0	0			90	5	100	0	88	2
MTBE	0.1	36	36	108	5	0	0			88	2	95	0	94	4
acrylonitrile	0.1	36	36	110	6	0	0			87	5	168	75	72	3
trans-1,2-dichloroethene	0.0	36	36	95	5	0	0			92	1	98	3	92	3
1,1-dichloroethane	0.4	36	35	80	5	0	1	6	0	93	2	97	1	97	3
2,2-dichloropropane	0.0	36	36	104	3	0	0			93	2	88	1	106	5
propionitrile	0.1	36	30	115	9	0	6	115	9	111	3	171	57	85	4
2-butanone	10.1	0	0	NA		0	0			NA		107	1	NA	
cis-1,2-dichloroethene	0.0	36	36	101	2	0	0			101	3	99	1	101	2
methacrylonitrile	0.1	36	36	116	6	0	0			89	3	113	9	15	4
chloroform	0.3	36	36	92	3	0	0			101	2	100	2	100	2
bromochloromethane	0.0	36	36	111	4	0	0			96	1	98	3	100	1
cyclohexane	0.1	36	36	105	3	0	0			93	2	104	2	111	12
1,1,1-trichloroethane	0.0	36	36	109	3	0	0			98	1	104	3	112	6
1,1-dichloropropene	0.0	36	36	110	4	0	0			93	2	102	4	116	8
carbon tetrachloride	0.0	36	36	105	4	0	0			96	2	107	0	102	11
1,2-dichloroethane	0.1	36	36	102	5	0	0			90	2	98	0	91	3
benzene	0.2	36	36	92	3	0	0			98	1	102	2	101	2
trichloroethene	0.0	36	36	103	3	0	0			96	1	100	2	96	3
methyl cyclohexane	0.1	36	36	100	2	0	0			96	3	108	2	113	10
1,2-dichloropropane	0.0	36	36	101	3	0	0			88	3	98	3	97	2
methylmethacrylate	0.1	36	36	112	7	0	0			93	1	96	0	29	3

dibromomethane	0.0	36	36	111	5	0			0	92	2	100	1	99	3
bromodichloromethane	0.1	36	36	114	4	0			0	95	2	102	0	80	5
1,4-dioxane	0.4	36	36	79	7	0			0	92	3	107	1	113	4
4-methyl-2-pentanone	0.1	36	36	104	12	0			0	87	3	99	2	144	5
trans-1,3-dichloropropene	0.0	36	36	105	3	0			0	89	2	92	2	89	4
toluene	0.7	36	36	90	4	0			0	99	1	102	4	119	2
cis-1,3-dichloropropene	0.0	36	36	111	5	0			0	90	1	96	0	76	3
2-hexanone	2.6	36	36	71	9	0			0	93	4	74	5	148	5
1,1,2-trichloroethane	0.0	36	36	111	6	0			0	91	3	97	0	106	3
1,3-dichloropropane	0.0	36	36	111	6	0			0	90	3	99	1	108	3
tetrachloroethene	0.0	36	36	102	3	0			0	92	2	99	1	130	9
dibromochloromethane	0.0	36	36	116	5	0			0	94	2	101	0	83	4
1,2-dibromoethane	0.0	36	36	111	5	0			0	93	2	98	2	115	2
chlorobenzene	0.0	36	36	99	3	0			0	96	1	99	0	93	1
1,1,1,2-tetrachloroethane	0.0	36	36	100	4	0			0	93	2	101	1	101	4
ethylbenzene	0.2	36	36	96	3	0			0	96	1	100	5	99	3
m,p-xylenes	0.7	36	36	90	4	0			0	96	1	100	3	99	3
o-xylene	0.3	36	36	95	3	0			0	95	1	100	2	102	4
styrene	0.5	36	36	98	6	0			0	93	2	94	2	102	3
isopropylbenzene	0.1	36	36	95	4	0			0	94	2	100	4	99	4
bromoform	0.0	36	36	114	8	0			0	95	1	96	3	106	5
cis-1,4-dichloro-2-butene	0.0	36	36	106	13	0			0	91	3	98	4	111	6
1,1,2,2-tetrachloroethane	0.0	36	36	95	8	0			0	98	1	96	2	109	7
1,2,3-trichloropropane	3.4	0	0	NA		0			0	99	2	109	3	112	6
propylbenzene	0.1	36	20	99	4	16	95	5	0	94	1	104	1	100	3
bromobenzene	0.1	36	36	100	5	0			0	95	2	105	3	106	5
trans-1,4-dichloro-2-butene	0.0	36	36	93	11	0			0	98	2	113	2	114	8
1,3,5-trimethylbenzene	0.2	36	20	93	4	16	89	6	0	95	2	102	2	102	4

2-chlorotoluene	0.1	36	20	98	5	16	95	5	0			95	1	103	1	102	4
4-chlorotoluene	0.0	36	20	95	4	16	92	5	0			94	2	102	1	102	3
tert-butylbenzene	0.0	36	20	91	5	16	85	6	0			95	2	107	3	102	4
sec-butylbenzene	0.3	36	20	93	5	16	87	7	0			96	2	106	2	99	4
pentachloroethane	0.0	36	20	101	6	16	93	6	0			91	3	104	1	99	4
1,2,4-trimethylbenzene	0.6	36	20	88	5	16	84	6	0			96	1	103	2	101	3
p-isopropyltoluene	1.2	0	0	NA		0			0			94	1	109	1	101	3
1,3-dichlorobenzene	0.1	36	20	92	5	16	86	6	0			95	3	97	1	101	4
1,4-dichlorobenzene	0.1	36	20	91	5	16	87	5	0			94	2	97	1	103	4
n-butylbenzene	0.1	36	20	89	7	16	78	8	0			94	2	104	2	100	4
1,2-dichlorobenzene	0.1	36	20	91	6	16	83	6	0			93	2	97	2	104	4
acetophenone	2.1	36	8	109	22	16	100	25	12	109	15	74	11	64	8	94	12
1,2-dibromo-3-chloropropane	0.0	36	20	108	10	16	91	11	0			113	3	108	4	117	6
nitrobenzene	0.4	36	20	112	14	16	96	14	0			109	11	100	8	123	10
1,2,4-trichlorobenzene	0.3	36	20	84	10	16	70	6	0			84	3	95	1	98	3
hexachlorobutadiene	0.1	36	0	NA		16	50	8	20	67	17	84	3	116	0	102	6
naphthalene	0.5	36	20	106	8	16	91	6	0			95	3	108	4	109	3
1,2,3-trichlorobenzene	0.5	36	20	79	11	16	62	5	0			84	2	92	1	100	3
2-methylnaphthalene	0.7	36	20	98	21	16	79	11	0			81	3	98	4	100	5
1-methylnaphthalene	0.6	0	0	NA		0			0			85	3	104	2	103	5
Totals		2808	2351	99	10	320	85	12	137	71	33						

[a] The amount of analyte (or interference) observed in 5g sample as a percentage of the medium spike amount.
[b] Analyte results that passed criteria for accuracy study.
[c] Analyte results that failed mixing criteria for accuracy study.
[d] Analyte results that failed quality control criteria for accuracy study.
[e] Analyte results for evaluation of spiking sand after adding water with average equilibration times of 3 and 114 hr.
[f] Internal standards, surrogates, and analytes added to dry sand with overnight equilibration. Some analytes were observed to degrade.
[g] No data used due to background concentration or interference.

Table S5 Nevada mountain soil results by analyte

		Conc[b]	run	meet all criteria[c]			fail mix criteria[d]			fail QC criteria[e]			wet spike[f]				dry spike[g]	
	degrade	as % of											3.5		20.2		19.6	
Compound	120 hr[a]	spike	#	#	avg	dev	#	avg	dev	#	avg	dev	avg	dev	avg	dev	avg	dev
dichlorodifluoromethane		1.7	36	6	92	23	1	14	0	29	60	44	196	41	171	30	92	9
chloromethane		7.0	0	0	NA[h]		0			0			112	13	160	5	88	4
vinylchloride		0.1	36	17	99	14	1	5	0	18	56	13	104	4	86	11	81	2
bromomethane	-79	0.4	36	29	81	17	1	3	0	6	37	11	92	4	71	5	64	2
chloroethane		0.7	36	23	92	32	1	7	0	12	104	37	97	15	77	8	70	5
trichlorofluoromethane		1.0	0	0	NA		0			0			110	5	100	11	98	4
diethyl ether		6.7	0	0	NA		0			0			NA		NA		NA	
1,1,2-trichloro-1,2,2-trifluoroethane		0.5	0	0	NA		0			0			109	4	108	10	104	2
acetone		2065.0	0	0	NA		0			0			NA		NA		NA	
1,1-dichloroethene		3.7	0	0	NA		0			0			108	22	112	11	83	16

iodomethane	-92	0.2	36	35	95	23	1	6	0	0	102	27	77	3	65	9
allylchloride		28.1	0	0	NA		0			0	110	42	39	8	42	9
acetonitrile	-100	8.4	0	0	NA		0			0	75	16	72	3	87	14
methyl acetate	-97	47.6	0	0	NA		0			0	NA		8	1	NA	
carbon disulfide	-68	0.4	36	35	104	21	1	6	0	0	92	32	63	9	83	12
methylene chloride		2.4	36	35	89	12	1	11	0	0	87	22	88	2	89	8
MTBE		2.0	0	0	NA		0			0	91	18	94	4	92	4
acrylonitrile	-100	0.6	36	36	99	17	0			0	95	9	72	3	80	5
trans-1,2-dichloroethene		0.0	36	35	106	10	1	4	0	0	97	10	92	3	87	2
1,1-dichloroethane		0.0	36	35	107	12	1	4	0	0	93	4	97	3	89	4
2,2-dichloropropane		0.1	36	35	147	15	1	6	0	0	110	4	106	5	99	2
propionitrile	-100	0.9	0	0	NA		0			0	123	4	85	4	88	5
2-butanone		175.2	0	0	NA		0			0	NA		NA		NA	
cis-1,2-dichloroethene		0.1	36	35	107	8	1	4	0	0	93	1	101	2	92	3
methacrylonitrile	-99	1.7	0	0	NA		0			0	69	9	15	4	16	2
chloroform		0.7	36	35	108	9	1	7	0	0	93	2	100	2	93	3
bromochloromethane		0.0	36	35	93	6	1	3	0	0	94	6	100	1	92	3
cyclohexane		0.5	36	35	159	13	1	6	0	0	105	6	111	12	110	2
1,1,1-trichloroethane		2.3	0	0	NA		0			0	111	5	112	6	108	2
1,1-dichloropropene		0.1	36	35	161	13	1	5	0	0	111	5	116	8	117	3
carbon tetrachloride		0.0	36	35	159	12	1	6	0	0	109	6	102	11	102	4
1,2-dichloroethane		0.1	36	35	90	5	1	4	0	0	93	6	91	3	89	2
benzene		5.0	0	0	NA	NA	0			0	97	2	101	2	96	3
trichloroethene		1.2	0	0	NA		0			0	88	3	96	3	93	2
methyl cyclohexane		1.6	0	0	NA		0			0	97	11	113	10	119	1
1,2-dichloropropane		0.1	36	35	98	7	1	4	0	0	89	4	97	2	90	2
methylmethacrylate	-100	0.2	36	35	84	9	1	4	0	0	78	6	29	3	30	3
dibromomethane		0.0	36	35	89	6	1	3	0	0	91	6	99	3	97	2

bromodichloromethane	-77	0.1	36	35	84	5	1	3	0	0			84	3	80	5	75	2
1,4-dioxane		0.7	36	35	111	21	0			1	9	0	104	3	113	4	122	3
4-methyl-2-pentanone		6.2	0	0	NA		0			0			121	4	144	5	160	6
trans-1,3-dichloropropene	-82	0.0	36	35	89	4	1	3	0	0			90	5	89	4	84	2
toluene		29.1	0	0	NA		0			0			108	3	119	2	117	3
cis-1,3-dichloropropene	-87	0.0	0	0	NA		0			0			78	3	76	3	75	2
2-hexanone	-97	80.7	0	0	NA		0			0			139	8	148	5	171	6
1,1,2-trichloroethane		0.2	36	35	95	7	1	4	0	0			95	5	106	3	103	3
1,3-dichloropropane		0.0	36	35	107	7	1	3	0	0			97	4	108	3	108	2
tetrachloroethene		2.3	0	0	NA		0			0			108	12	130	9	125	2
dibromochloromethane	-86	0.1	36	35	91	8	1	4	0	0			90	2	83	4	82	5
1,2-dibromoethane		0.1	36	35	113	8	1	3	0	0			106	4	115	2	116	2
chlorobenzene		0.4	36	35	85	6	1	5	0	0			88	4	93	1	92	2
1,1,1,2-tetrachloroethane		0.0	36	35	92	9	1	3	0	0			95	6	98	3	99	2
ethylbenzene		5.7	0	0	NA		0			0			108	7	119	2	118	2
m,p-xylenes		32.9	0	0	NA		0			0			112	9	127	3	124	2
o-xylene		10.0	0	0	NA		0			0			108	7	120	2	116	2
styrene		7.3	0	0	NA		0			0			84	5	67	2	62	3
isopropylbenzene		3.1	0	0	NA		0			0			110	16	131	7	120	1
bromoform	-89	0.2	36	35	123	13	1	3	0	0			107	1	95	6	101	6
cis-1,4-dichloro-2-butene	-100	0.2	36	35	134	16	1	4	0	0			102	12	56	10	56	11
1,1,2,2-tetrachloroethane		0.6	0	0	NA		0			0			95	4	104	3	107	3
1,2,3-trichloropropane		5.2	0	0	NA		0			0			126	5	153	6	168	4
propylbenzene		2.5	0	0	NA		0			0			110	21	134	5	118	1
bromobenzene		0.1	36	35	79	9	1	3	0	0			86	4	88	3	88	2
trans-1,4-dichloro-2-butene	-100	0.0	36	34	110	10	1	3	0	1	67	0	101	11	59	8	60	11
1,3,5-trimethylbenzene		4.1	0	0	NA		0			0			110	19	132	5	118	1
2-chlorotoluene		0.2	36	21	84	9	15	80	25	0			96	13	106	2	97	1

4-chlorotoluene		0.1	36	21	76	9	15	74	24	0			89	11	92	2	88	1
tert-butylbenzene		0.2	36	21	137	27	15	132	49	0			122	27	167	8	138	2
sec-butylbenzene		1.0	0	0	NA		0			0			121	35	175	12	130	3
pentachloroethane	-83	0.0	36	21	78	10	15	70	22	0			83	4	76	5	73	3
1,2,4-trimethylbenzene		26.0	0	0	NA		0			0			123	19	146	6	134	2
p-isopropyltoluene		101.6	0	0	NA		0			0			NA		NA		NA	
1,3-dichlorobenzene		0.3	36	19	77	10	15	80	29	2	77	0	82	12	87	3	78	2
1,4-dichlorobenzene		2.1	0	0	NA		0			0			82	11	85	2	80	2
n-butylbenzene		2.3	0	0	NA		0			0			117	39	173	15	115	3
1,2-dichlorobenzene		0.4	36	18	100	17	15	110	42	3	100	14	84	10	97	7	92	3
acetophenone		79.2	0	0	NA		0			0			155	20	173	21	235	27
1,2-dibromo-3-chloropropane		0.6	0	0	NA		0			0			167	10	219	12	249	13
nitrobenzene	-88	2.0	0	0	NA		0			0			150	14	137	22	178	18
1,2,4-trichlorobenzene		1.0	0	0	NA		0			0			76	16	76	7	63	4
hexachlorobutadiene		0.0	36	11	88	26	15	98	59	10	77	29	136	66	241	35	103	8
naphthalene		29.5	0	0	NA		0			0			111	8	134	7	136	4
1,2,3-trichlorobenzene		0.7	36	11	61	10	15	80	69	10	60	7	71	13	76	6	67	4
2-methylnaphthalene		11.4	0	0	NA		0			0			69	6	72	12	80	5
1-methylnaphthalene		7.1	0	0	NA		0			0			71	6	81	13	85	4
Totals			1512	1268	102	23	152	22	36	92	65	28						

[a] The % of analyte degraded after 120 hr.
[b] The amount of analyte (or interference) observed in 5g sample as a percentage of the medium spike amount.
[c] Analyte results that passed criteria for accuracy study.
[d] Analyte results that failed mixing criteria for accuracy study.
[e] Analyte results that failed quality control criteria for accuracy study.
[f] Analyte results for evaluation of spiking soil after adding water with average equilibration times of 3.5 and 20 hr.
[g] Analyte results for when analytes added to soil prior to adding water, mixing, then allowing overnight equilibration for 19.6hr.

[h] No data used due to background concentration or interference or degradation.

Table S6 Georgia clay soil results by analyte

		Conc[b]		meetcriteria[c]			fail mix criteria[d]			fail QC[e]			sealed mixing[f]		wet spike[g]	
	degrade	% of	run	% recovery			% recovery			% recovery			% recovery		% recovery	
Compound	120 hr[a]	spike	#	#	avg	dev	#	avg	dev	#	avg	dev	avg	dev	avg	dev
dichlorodifluoromethane		0.3	36	0	NA[h]		0			36	43	26	88	31	NA	
chloromethane		1.3	36	0	NA		0			36	35	14	74	8	NA	
vinylchloride		0.1	36	0	NA		0			36	69	22	93	3	94	5
bromomethane		0.1	36	0	NA		0			36	59	13	84	5	96	13
chloroethane		0.8	36	0	NA		0			36	116	44	66	6	89	22
trichlorofluoromethane		0.1	36	0	NA		0			36	84	14	93	8	101	7
diethyl ether		7.5	0	0	NA		0			0			NA		NA	
1,1,2-trichloro-1,2,2-trifluoroethane		0.2	36	36	103	17	0			0			92	3	101	7
acetone		1736.1	0	0	NA		0			0			NA		NA	
1,1-dichloroethene		0.3	36	0	NA		0			36	83	14	85	22	93	14

iodomethane	0.0	36	0	NA		0	36	83	10	99	16	96	14
allylchloride	1.0	0	0	NA		0	0			98	22	113	16
acetonitrile	89.8	0	0	NA		0	0			142	50	119	20
methyl acetate	1.7	36	30	96	16	0	6	80	37	85	34	127	23
carbon disulfide	0.0	36	0	NA		0	36	85	15	75	23	117	9
methylene chloride	1.4	36	36	77	5	0	0			80	15	107	11
MTBE	0.1	36	36	104	4	0	0			97	7	100	12
acrylonitrile	0.1	36	36	91	6	0	0			97	11	103	7
trans-1,2-dichloroethene	0.0	36	36	94	6	0	0			89	3	98	2
1,1-dichloroethane	0.0	36	36	91	3	0	0			89	2	100	3
2,2-dichloropropane	0.2	36	36	101	6	0	0			98	3	97	2
propionitrile	0.1	36	36	113	7	0	0			112	11	106	4
2-butanone	121.4	0	0	NA		0	0			NA		NA	
cis-1,2-dichloroethene	0.0	36	36	94	3	0	0			98	2	100	2
methacrylonitrile	0.1	36	36	105	7	0	0			101	7	104	4
chloroform	0.3	36	36	84	4	0	0			98	2	99	3
bromochloromethane	0.0	36	36	91	3	0	0			96	2	99	4
cyclohexane	0.0	36	36	117	13	0	0			98	2	102	2
1,1,1-trichloroethane	0.0	36	36	109	7	0	0			102	3	101	2
1,1-dichloropropene	0.1	36	36	111	12	0	0			96	1	106	3
Carbon tetrachloride	0.0	36	36	110	11	0	0			102	2	101	2
1,2-dichloroethane	0.0	36	36	92	5	0	0			96	2	97	2
benzene	0.2	36	36	85	3	0	0			98	2	83	37
trichloroethene	0.0	36	36	94	4	0	0			96	1	104	2
methyl cyclohexane	0.1	36	36	129	15	0	0			99	1	107	3
1,2-dichloropropane	0.1	36	36	90	4	0	0			92	1	99	2
methylmethacrylate	0.3	36	36	105	5	0	0			106	4	106	5
dibromomethane	0.0	36	36	104	5	0	0			99	2	102	5

bromodichloromethane		0.0	36	36	97	5	0			0	98	2	105	3
1,4-dioxane		3.2	0	0	NA		0			0	106	5	101	4
4-methyl-2-pentanone		18.5	0	0	NA		0			0	124	8	124	8
trans-1,3-dichloropropene		0.0	36	36	94	3	0			0	95	1	100	3
toluene		2.2	0	0	NA		0			0	103	1	105	2
cis-1,3-dichloropropene		0.0	36	36	95	5	0			0	97	2	101	3
2-hexanone		84.9	0	0	NA		0			0	161	10	172	9
1,1,2-trichloroethane		0.0	36	36	94	5	0			0	97	2	103	5
1,3-dichloropropane		0.0	36	36	97	5	0			0	100	2	103	4
tetrachloroethene		0.0	36	36	111	8	0			0	98	1	101	1
dibromochloromethane		0.0	36	36	106	5	0			0	99	2	103	4
1,2-dibromoethane		0.0	36	36	104	5	0			0	101	2	103	5
chlorobenzene		0.0	36	36	93	3	0			0	99	1	102	1
1,1,1,2-tetrachloroethane		0.0	36	36	94	4	0			0	96	2	99	3
ethylbenzene		0.8	0	0	NA		0			0	99	2	100	1
m,p-xylenes		4.3	0	0	NA		0			0	101	1	101	1
o-xylene		1.4	0	0	NA		0			0	98	2	102	3
styrene	-35%	0.4	36	36	88	6	0			0	95	2	101	2
isopropylbenzene		0.1	36	36	99	4	0			0	98	2	99	1
bromoform		0.0	36	36	110	7	0			0	106	2	109	6
cis-1,4-dichloro-2-butene		0.0	36	36	116	8	0			0	106	3	105	6
1,1,2,2-tetrachloroethane		0.0	36	36	103	6	0			0	104	1	104	6
1,2,3-trichloropropane		0.2	0	0	NA		0			0	NA		NA	
propylbenzene		0.5	0	0	NA		0			0	101	1	104	2
bromobenzene		0.0	36	36	98	4	0			0	99	2	106	3
trans-1,4-dichloro-2-butene		0.0	36	36	117	8	0			0	109	1	107	4
1,3,5-trimethylbenzene		1.2	0	0	NA		0			0	100	1	105	2
2-chlorotoluene		0.1	36	34	95	4	2	100	12	0	102	2	105	2

4-chlorotoluene	0.0	36	34	94	3	2	99	8	0			101	1	103	2
tert-butylbenzene	0.0	36	34	96	5	2	93	5	0			102	2	100	2
sec-butylbenzene	0.2	36	34	101	5	2	100	11	0			101	2	104	2
pentachloroethane	0.0	36	34	91	5	2	92	5	0			96	2	101	3
1,2,4-trimethylbenzene	4.8	0	0	NA		0			0			102	2	106	2
p-isopropyltoluene	2.1	0	0	NA		0			0			104	1	107	3
1,3-dichlorobenzene	0.0	36	34	88	4	2	96	15	0			98	1	103	2
1,4-dichlorobenzene	0.1	36	34	90	4	2	99	16	0			98	1	104	2
n-butylbenzene	0.7	0	0	NA		0			0			99	1	105	2
1,2-dichlorobenzene	0.1	36	34	88	4	2	99	20	0			98	2	103	2
acetophenone	179.4	0	0	NA		0			0			NA		NA	
1,2-dibromo-3-chloropropane	0.0	36	32	139	11	2	151	22	2	150	20	127	3	121	10
nitrobenzene	0.9	36	34	131	11	2	148	16	0			143	10	134	21
1,2,4-trichlorobenzene	0.3	36	34	75	7	2	93	40	0			89	1	95	4
hexachlorobutadiene	0.0	36	34	64	11	2	54	11	0			78	5	96	6
naphthalene	3.0	0	0	NA		0			0			111	3	109	3
1,2,3-trichlorobenzene	0.5	36	34	70	6	2	97	54	0			86	2	93	3
2-methylnaphthalene	4.7	0	0	NA		0			0			97	2	100	4
1-methylnaphthalene	1.9	0	0	NA		0			0			100	3	97	3
Totals		2268	1910	99	14	26	102	25	332	81	32				

[a] The % of analyte degraded after 120 hr.
[b] The amount of analyte (or interference) observed in 5g sample as a percentage of the medium spike amount.
[c] Analyte results that passed criteria for accuracy study.
[d] Analyte results that failed mixing criteria for accuracy study.
[e] Analyte results that failed quality control criteria for accuracy study.
[f] Analyte results for evaluation of spiking and mixing in closed vessel attached to distiller.
[g] Analyte results for when analytes added to soil prior to adding water, mixing, then allowing average of 3 hr equilibration.

[h] No data used due to background concentration or interference or degradation.

Table S7 Oregon farm soil results by analyte

		conc[b]		pass criteria[c]			fail mix criteria[d]			fail QC criteria[e]			wet spike[f]				dry spike[g]			
	degrade	(as % of											3.5 hr		19.7		19.6			
Compound	120 hr[a]	spike)	#	#	avg	dev	#	avg	dev	#	avg	dev	avg	dev	avg	dev	avg	dev		
dichlorodifluoromethane		0.5	36	6	57	7	0			30	55	31	109	51	NA		94	10		
chloromethane		1.5	36	12	68	11	0			24	54	19	94	17	122	10	113	8		
vinylchloride		0.1	36	24	92	15	0			12	65	4	102	13	85	4	99	6		
bromomethane	-62	0.1	36	24	83	12	0			12	62	4	89	9	79	4	83	6		
chloroethane		0.6	36	12	85	28	0			24	83	27	73	14	69	8	97	6		
trichlorofluoromethane		0.1	36	24	119	10	0			12	106	11	107	10	94	6	115	9		
diethyl ether		7.0	0	0	NA[h]		0			0			NA		NA		NA			
1,1,2-trichloro-1,2,2-trifluoroethane		0.2	36	36	130	13	0			0			106	8	92	5	130	3		
acetone		1305.1	0	0	NA		0			0			NA		NA		NA			
1,1-dichloroethene		0.5	36	36	103	9	0			0			95	10	86	4	103	17		

iodomethane	-84	0.1	36	36	92	10	0	0			89	6	88	4	77	4
allylchloride		8.3	0	0	NA		0	0			88	9	90	11	NA	
acetonitrile	-99	2.4	0	0	NA		0	0			84	2	85	2	60	6
methyl acetate		20.9	0	0	NA		0	0			79	13	113	13	NA	
carbon disulfide	-82	0.0	36	36	83	11	0	0			83	11	76	10	83	12
methylene chloride		1.7	36	36	86	9	0	0			88	5	93	6	85	7
MTBE		2.5	0	0	NA		0	0			102	6	111	5	101	3
acrylonitrile	-100	0.1	36	36	85	4	0	0			90	2	92	2	94	7
trans-1,2-dichloroethene		0.0	36	36	86	4	0	0			88	4	83	2	87	6
1,1-dichloroethane		0.0	36	36	101	8	0	0			91	4	95	5	95	3
2,2-dichloropropane		0.0	36	36	126	10	0	0			103	6	101	5	116	5
propionitrile	-100	0.3	36	36	103	6	0	0			105	3	112	3	122	11
2-butanone		83.9	0	0	NA		0	0			171	3	182	4	NA	
cis-1,2-dichloroethene		0.0	36	36	84	3	0	0			86	3	89	3	89	2
methacrylonitrile	-100	0.1	0	0	NA		0	0			87	2	88	2	NA	
chloroform		0.6	36	36	93	6	0	0			92	1	97	2	98	4
bromochloromethane		0.0	36	36	78	5	0	0			86	3	92	5	86	3
cyclohexane		0.1	36	36	119	5	0	0			104	10	103	4	122	3
1,1,1-trichloroethane		0.3	36	36	123	9	0	0			103	5	107	3	121	2
1,1-dichloropropene		0.0	36	36	120	5	0	0			105	9	108	5	127	4
Carbon tetrachloride		0.1	36	36	125	7	0	0			108	10	103	3	128	4
1,2-dichloroethane		0.1	36	36	75	5	0	0			84	2	88	3	84	2
benzene	-84	2.4	0	0	NA		0	0			91	2	96	1	99	2
trichloroethene		4.7	0	0	NA		0	0			88	5	89	1	95	1
Methyl cyclohexane		0.3	36	36	122	6	0	0			106	12	106	6	117	2
1,2-dichloropropane		0.1	36	36	90	4	0	0			90	2	94	2	99	1
methylmethacrylate	-95	1.5	36	33	84	4	0	3	79	2	94	2	85	1	90	3
dibromomethane		0.0	36	0	NA		0	36	68	5	81	3	78	3	80	2

bromodichloromethane	-52	0.2	36	36	74	4	0	0	83	1	80	1	84	2
1,4-dioxane		0.6	36	36	101	4	0	0	101	2	101	2	103	5
4-methyl-2-pentanone		19.5	0	0	NA		0	0	134	2	149	1	NA	
trans-1,3-dichloropropene	-85	0.0	0	0	NA		0	0	88	3	80	2	NA	
toluene		30.2	0	0	NA		0	0	122	6	146	4	NA	
cis-1,3-dichloropropene	-81	0.0	0	0	NA		0	0	74	5	62	2	NA	
2-hexanone	-78	76.4	0	0	NA		0	0	165	5	205	5	NA	
1,1,2-trichloroethane		0.1	36	36	87	6	0	0	96	2	98	4	99	3
1,3-dichloropropane		0.0	36	36	86	6	0	0	99	3	101	1	101	3
tetrachloroethene		2.3	0	0	NA		0	0	112	13	115	3	114	2
dibromochloromethane	-52	0.3	36	36	82	5	0	0	93	3	88	4	91	2
1,2-dibromoethane		0.0	36	36	87	7	0	0	101	5	98	5	97	3
chlorobenzene		0.4	36	36	75	3	0	0	90	8	85	2	82	1
1,1,1,2-tetrachloroethane		0.0	36	36	97	7	0	0	105	4	113	2	116	4
ethylbenzene		10.8	0	0	NA		0	0	120	9	135	4	NA	
m,p-xylenes		42.6	0	0	NA		0	0	134	10	169	6	NA	
o-xylene		19.9	0	0	NA		0	0	126	9	149	6	NA	
styrene	-97	2.0	0	0	NA		0	0	85	9	66	3	NA	
isopropylbenzene		1.7	0	0	NA		0	0	129	13	158	8	135	4
bromoform		0.4	36	36	109	7	0	0	103	7	107	4	116	4
cis-1,4-dichloro-2-butene	-99	0.1	36	36	99	12	0	0	103	5	102	4	100	2
1,1,2,2-tetrachloroethane		0.2	36	36	106	10	0	0	101	6	118	5	116	6
1,2,3-trichloropropane		0.2	0	0	NA		0	0	NA		NA		NA	
propylbenzene		7.2	0	0	NA		0	0	137	18	154	7	NA	
bromobenzene		0.0	36	36	71	4	0	0	89	11	78	2	75	3
trans-1,4-dichloro-2-butene	-100	0.1	36	36	86	8	0	0	96	12	87	3	90	3
1,3,5-trimethylbenzene		18.3	0	0	NA		0	0	141	17	167	6	NA	
2-chlorotoluene		1.4	0	0	NA		0	0	108	12	114	4	93	1

4-chlorotoluene		0.1	36	30	74	5	6	78	8	0			100	13	92	4	78	2
tert-butylbenzene		0.2	36	30	143	13	6	140	21	0			154	20	193	9	151	2
sec-butylbenzene		1.5	0	0	NA		0			0			162	26	202	9	142	4
pentachloroethane		0.0	36	30	94	6	6	91	11	0			106	9	118	3	110	4
1,2,4-trimethylbenzene		77.4	0	0	NA		0			0			175	23	230	9	NA	
p-isopropyltoluene		57.1	0	0	NA		0			0			218	35	309	22	NA	
1,3-dichlorobenzene		0.2	36	30	74	4	6	79	11	0			95	14	89	4	77	2
1,4-dichlorobenzene		2.6	0	0	NA		0			0			92	12	88	4	78	1
n-butylbenzene		8.0	0	0	NA		0			0			167	30	190	7	NA	
1,2-dichlorobenzene		0.2	36	30	99	10	6	113	23	0			97	14	101	4	97	1
acetophenone	-88	33.1	0	0	NA		0			0			147	36	113	18	NA	
1,2-dibromo-3-chloropropane		0.1	36	30	205	18	6	211	36	0			166	25	185	15	207	17
nitrobenzene	-82	3.3	0	0	NA		0			0			121	28	83	13	120	13
1,2,4-trichlorobenzene		0.7	36	19	68	7	6	108	54	11	71	11	94	14	85	7	70	2
hexachlorobutadiene		0.1	36	21	117	23	6	119	21	9	116	18	213	55	263	27	110	3
naphthalene		37.3	0	0	NA		0			0			114	20	121	12	NA	
1,2,3-trichlorobenzene		1.1	36	19	65	7	6	110	67	11	67	8	85	12	86	7	79	4
2-methylnaphthalene		11.6	0	0	NA		0			0			70	17	61	7	NA	
1-methylnaphthalene		6.1	0	0	NA		0			0			75	16	67	6	NA	
			1800	1562	96	25	54	117	41	184	75	20						

[a] The % of analyte degraded after 120 hr.
[b] The amount of analyte (or interference) observed in 5g sample as a percentage of the medium spike amount.
[c] Analyte results that passed criteria for accuracy study.
[d] Analyte results that failed mixing criteria for accuracy study.
[e] Analyte results that failed quality control criteria for accuracy study.
[f] Analyte results for evaluation of spiking soil after adding water with average equilibration times of 3.5 and 20 hr.
[g] Analyte results for when analytes added to soil prior to adding water, mixing, then allowing overnight equilibration for 19.6hr.

[h] No data used due to background concentration or interference or degradation.

Table S8 Greenwich Bay sediment results by analyte

		conc[b]		pass criteria[c]			fail mix criteria[d]			fail QC criteria[e]			wet spike closed mix[f]			
	degrade	(as % of											3.8 hr		18.6 hr	
Compound	110 hr[a]	spike)	run	#	avg	dev	#	avg	dev	#	avg	dev	avg	dev	avg	dev
dichlorodifluoromethane		1.1	36	9	77	11	6	41	46	21	41	12	NA		74	16
chloromethane		7.4	0	0	NA[g]		0			0			NA		68	5
vinylchloride		0.4	36	9	90	14	6	95	74	21	72	7	101	5	73	5
bromomethane	-100	0.6	0	0	NA		0			0			NA		NA	
chloroethane		4.1	36	0	NA		6	105	83	30	97	26	59	4	59	9
trichlorofluoromethane		0.6	36	9	99	12	6	134	91	21	97	6	87	2	82	2
diethyl ether		29.8	0	0	NA		0			0			NA		NA	
1,1,2-trichloro-1,2,2-trifluoroethane		0.2	36	34	102	6	2	211	195	0			95	5	93	3
acetone		325.2	0	0	NA		0			0			NA		NA	
1,1-dichloroethene		0.8	36	34	102	9	2	207	182	0			72	17	96	6

iodomethane	-100	0.0	0	0	NA		0			0			NA		NA	
allylchloride	-99	25.9	0	0	NA		0			0			NA		NA	
acetonitrile		1.6	36	18	61	9	0			18	67	NA	NA		NA	
methyl acetate	-100	54.1	0	0	NA		0			0			NA		NA	
carbon disulfide	-79	30.3	0	0	NA		0			0			NA		NA	
methylene chloride		9.0	0	0	NA		0			0			86	17	119	5
MTBE		0.4	36	34	109	4	2	134	84	0			74	24	119	3
acrylonitrile	-98	0.3	0	0	NA		0			0			NA		NA	
trans-1,2-dichloroethene	-33	0.0	36	34	92	7	2	126	107	0			81	5	73	3
1,1-dichloroethane		0.1	36	34	95	6	2	135	82	0			83	2	87	3
2,2-dichloropropane	-35	0.7	36	34	103	5	2	172	149	0			95	3	94	4
propionitrile	-43	0.7	36	24	133	26	0			12	147	8	128	10	124	7
2-butanone	-32	49.8	0	0	NA		0			0			NA		NA	
cis-1,2-dichloroethene		0.0	36	34	101	4	2	137	103	0			94	2	88	3
methacrylonitrile		0.1	36	36	121	17	0			0			103	6	105	4
chloroform		1.6	36	34	102	4	2	141	101	0			95	1	100	2
bromochloromethane	-70	0.0	36	34	142	6	2	171	118	0			119	2	139	4
cyclohexane		0.2	36	34	98	8	2	176	197	0			96	4	95	2
1,1,1-trichloroethane	-39	0.3	36	34	104	6	2	167	154	0			101	3	96	2
1,1-dichloropropene		0.3	36	34	107	6	2	209	220	0			102	3	103	1
Carbon tetrachloride	-88	0.0	0	0	NA		0			0			NA		NA	
1,2-dichloroethane		0.7	36	34	99	3	2	115	74	0			92	1	109	3
benzene	-51	0.4	36	34	97	3	2	133	94	0			100	2	96	2
trichloroethene		0.3	36	34	112	5	2	161	140	0			112	3	108	3
methyl cyclohexane		0.5	36	0	NA		2	182	209	34	100	16	92	7	96	3
1,2-dichloropropane		0.1	36	34	102	3	2	140	99	0			93	2	110	2
methylmethacrylate	-100	0.1	36	34	92	11	2	101	73	0			99	5	79	4
dibromomethane	-89	0.1	36	34	155	9	2	175	143	0			131	5	146	4

bromodichloromethane	-100	0.1	0	0	NA		0			0			NA		NA	
1,4-dioxane		1.8	36	36	110	14	0			0			107	5	110	6
4-methyl-2-pentanone		0.3	36	36	158	32	0			0			144	6	156	5
trans-1,3-dichloropropene	-100	0.0	0	0	NA		0			0			NA		NA	
toluene		4.1	36	34	98	4	2	150	132	0			101	2	103	2
cis-1,3-dichloropropene	-100	0.0	0	0	NA		0			0			NA		NA	
2-hexanone		1.4	36	36	113	29	0			0			125	5	147	5
1,1,2-trichloroethane		0.7	36	34	130	10	2	165	131	0			110	3	146	6
1,3-dichloropropane		0.1	36	34	117	5	2	161	134	0			109	2	130	2
tetrachloroethene		0.2	36	34	117	9	2	217	257	0			107	1	119	4
dibromochloromethane	-100	0.1	0	0	NA		0			0			NA		NA	
1,2-dibromoethane	-95	0.1	36	34	98	5	2	145	142	0			97	5	75	3
chlorobenzene		0.0	36	34	104	4	2	178	187	0			100	3	99	2
1,1,1,2-tetrachloroethane	-100	0.1	0	0	NA		0			0			NA		NA	
ethylbenzene		0.6	36	34	102	5	2	174	191	0			102	1	103	1
m,p-xylenes		1.9	36	34	102	5	2	180	197	0			99	3	103	2
o-xylene		0.9	36	34	100	4	2	191	214	0			99	2	104	2
styrene	-82	0.3	0	0	NA		0			0			NA		NA	
isopropylbenzene		0.2	36	34	97	8	2	200	247	0			96	4	97	3
bromoform	-100	0.1	0	0	NA		0			0			NA		NA	
cis-1,4-dichloro-2-butene	-100	0.4	0	0	NA		0			0			NA		NA	
1,1,2,2-tetrachloroethane		0.0	36	34	132	10	2	211	221	0			112	3	149	9
1,2,3-trichloropropane		0.3	36	34	153	12	2	276	275	0			138	5	189	10
propylbenzene		0.5	36	27	104	9	6	129	128	3	100	2	103	5	99	4
bromobenzene		0.1	36	34	107	5	2	203	233	0			105	4	108	2
trans-1,4-dichloro-2-butene	-100	0.0	0	0	NA		0			0			NA		NA	
1,3,5-trimethylbenzene		0.8	36	27	98	7	6	125	118	3	93	4	100	5	99	3
2-chlorotoluene		0.3	36	27	102	6	6	127	113	3	98	1	101	4	101	3

4-chlorotoluene		0.2	36	27	102	7	6	129	117	3	98	3	101	5	98	3
tert-butylbenzene		0.0	36	27	102	9	6	128	139	3	98	4	102	8	98	5
sec-butylbenzene		0.4	36	27	100	13	6	128	153	3	93	5	101	10	96	5
pentachloroethane	-100	0.2	0	0	NA		0			0			NA		NA	
1,2,4-trimethylbenzene		2.2	36	27	99	7	6	133	125	3	94	4	98	5	101	3
p-isopropyltoluene		0.6	36	27	103	12	6	135	156	3	97	8	112	11	101	7
1,3-dichlorobenzene		0.3	36	27	91	6	6	125	120	3	86	6	91	4	91	2
1,4-dichlorobenzene	-32	0.5	36	27	91	6	6	130	129	3	88	5	92	4	90	2
n-butylbenzene	-57	0.5	36	27	95	14	6	120	146	3	85	4	103	12	92	7
1,2-dichlorobenzene		0.7	36	27	93	5	6	138	144	3	91	4	89	3	98	3
acetophenone		7.6	36	27	140	25	6	172	137	3	146	17	199	15	NA	
1,2-dibromo-3-chloropropane	-99	0.4	36	27	132	14	6	191	195	3	146	3	162	7	131	6
nitrobenzene	-97	2.1	0	0	NA		0			0			NA		NA	
1,2,4-trichlorobenzene		1.6	36	27	76	9	6	114	96	3	66	4	74	5	75	4
hexachlorobutadiene		0.0	36	27	88	21	6	104	129	3	65	8	107	23	81	14
naphthalene		2.4	36	27	117	10	6	180	153	3	122	2	115	3	138	5
1,2,3-trichlorobenzene		4.2	36	21	74	9	6	108	88	9	67	5	64	4	73	3
2-methylnaphthalene		1.1	36	27	91	11	6	138	106	3	104	5	93	3	117	4
1-methylnaphthalene		1.2	36	27	104	14	6	161	129	3	118	8	95	6	121	6
			2232	1801	106	19	208	152	39	223	95	25				

[a] The % of analyte degraded after 110 hr.
[b] The amount of analyte (or interference) observed in 5g sample as a percentage of the medium spike amount.
[c] Analyte results that passed criteria for accuracy study.
[d] Analyte results that failed mixing criteria for accuracy study.
[e] Analyte results that failed quality control criteria for accuracy study.
[f] Analyte results for evaluation of spiking ssedimet after adding water with average equilibration times of 3.8 and 18.6 hr.
[g] No data used due to background concentration or interference or degradation.

Table S9 Table Internal Standards relative response for the matrices vs. time

			Equilibration Time					
			3 hr		20 hr		120 hours	
	Internal standards	spike[a]	avg	dev	avg	dev	avg	dev
Sand								
	1,4-difluorobenzene-d_4	wet	1.11	0.29	1.05	0.01		
	1,4-difluorobenzene-d_4	dry	0.95	0.05	0.89	0.01		
	chlorobenzene-d_5	wet	1.14	0.33	1.08	0.01		
	chlorobenzene-d_5	dry	0.96	0.06	0.94	0.01		
	1,2-dichlorobenzene-d_4	wet	1.11	0.37	1.08	0.02		
	1,2-dichlorobenzene-d_4	dry	0.95	0.10	0.96	0.02		
	naphthalene-d_8	wet	1.49	0.53	1.04	0.06		
	naphthalene-d_8	dry	0.84	0.24	0.98	0.05		
Georgia clay								
	1,4-difluorobenzene-d_4	wet	1.23	0.02			1.09	0.01
	1,4-difluorobenzene-d_4	dry	1.20	0.02				

chlorobenzene-d_5	wet	1.29	0.03			1.17	0.00
chlorobenzene-d_5	dry	1.31	0.03				
1,2-dichlorobenzene-d_4	wet	1.30	0.03			1.26	0.04
1,2-dichlorobenzene-d_4	dry	1.24	0.03				
naphthalene-d_8	wet	1.52	0.13			1.52	0.01
naphthalene-d_8	dry	1.21	0.09				
Nevada mountain soil							
1,4-difluorobenzene-d_4	wet	1.07	0.26	0.92	0.07	0.69	0.02
1,4-difluorobenzene-d_4	dry	0.68	0.04	0.86	0.03		
chlorobenzene-d_5	wet	0.88	0.23	0.69	0.06	0.46	0.02
chlorobenzene-d_5	dry	0.44	0.04	0.69	0.04		
1,2-dichlorobenzene-d_4	wet	0.45	0.14	0.30	0.04	0.13	0.01
1,2-dichlorobenzene-d_4	dry	0.13	0.03	0.28	0.02		
naphthalene-d_8	wet	0.27	0.09	0.16	0.03	0.02	0.00
naphthalene-d_8	dry	0.07	0.03	0.14	0.02		
Oregon farm soil							
1,4-difluorobenzene-d_4	wet	0.86	0.03	0.77	0.04	0.44	0.03
1,4-difluorobenzene-d_4	dry	0.71	0.02	0.73	0.02		
chlorobenzene-d_5	wet	0.66	0.03	0.55	0.03	0.22	0.03
chlorobenzene-d_5	dry	0.45	0.01	0.49	0.02		
1,2-dichlorobenzene-d_4	wet	0.34	0.03	0.28	0.02	0.09	0.00
1,2-dichlorobenzene-d_4	dry	0.19	0.02	0.23	0.02		
naphthalene-d_8	wet	0.15	0.02	0.14	0.02	0.03	0.00
naphthalene-d_8	dry	0.10	0.02	0.13	0.02		
Sediment							
1,4-difluorobenzene-d_4	wet	1.27	0.05	1.10	0.06	0.53	
chlorobenzene-d_5	wet	1.30	0.08	1.08	0.11	0.46	
1,2-dichlorobenzene-d_4	wet	0.93	0.09	0.86	0.13	0.37	
naphthalene-d_8	wet	0.81	0.13	0.97	0.21	0.29	

[a] Dry indicates adding the internal standard prior to diluting soil with water. Wet indicates internal standards were added to soil after the water was added.