

Validation Report  
Volatile Organic Analyses using the  
*Voyager* Field Portable Gas Chromatograph

Via

Method 6549.0  
"Analysis of Volatile Organic Compounds in Field  
Samples by Manual Headspace Using a Field  
Portable Gas Chromatograph"

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October, 2001

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## I. Preface

In July 2001 the FEMP Soils Project transferred ownership of and responsibility for the Field Portable GC (the PerkinElmer *Voyager*) to the onsite Analytical Laboratory. The unit was physically located in the Laboratory Building already. Several steps were undertaken to obtain training on the *Voyager*:

1. Training manuals were ordered from PerkinElmer (PE) and distributed to potential users of the instrument.
2. A training session by a PE representative was set up for August 15, and several field, Industrial Hygiene (IH) and Laboratory staff were present for the 8-hour training.
3. Following this official training session, the Laboratory staff member spent approximately one month practicing with the instrument and the software until proficiency was obtained.

Actual method validation commenced on September 27, 2001 and was completed October 11, 2001. This validation consisted of analyzing 22 soil samples spiked with the five analytes of concern at environmental levels:

- Trichlorethene (TCE),
- Tetrachloroethene (PCE),
- 1,1-Dichloroethene (1,1-DCE),
- trans-1,2-Dichloroethene, (t-1,2-DCE), and
- cis-1,2-Dichloroethene (c-1,2-DCE)

These samples were analyzed on the *Voyager* GC to determine efficiency of analyte recovery from the matrix.

This report presents all the data, results, and discussion relating to these analyses, as required by the Analytical Laboratory Services Quality Assurance Project Plan, ALS SOP 7516.

## II. Experimental Methodology

### A. Summary of method

When analyzing samples with a field portable Gas Chromatograph rapid turnaround, simplicity of operation and data reproducibility are very important. Therefore the method of injection and sample preparation are very important. Static headspace methods allow soil and water samples to be analyzed in the field with speed and accuracy without using expensive or time-consuming sample preparation equipment.



Static headspace involves a partitioning of volatile components between the aqueous and vapor phases (per Henry's Law) enclosed in a gas tight vial. An appropriate volume of a liquid sample or mass of a solid sample (with water added) is placed in a 40 mL VOA vial and heated. The volatile components (VOCs) move from the aqueous phase to the air space ("headspace") above the aqueous phase until an equilibrium is established between the liquid and gaseous phases inside the vial.. A portion of the headspace gases are withdrawn with a gas tight syringe and injected into the field GC. The GC separates the components and generates qualitative and semi-quantitative results for each component for which the GC was calibrated (the "library").

Compound identification is determined by a match between the retention times of the compound in the calibration standard and of the sample analysis. Compound quantification is determined via a ratio of the peak area of the compound in the calibration standard to the peak area of the compound in the sample (external standard method).

Each sample (headspace gases) must be injected twice to analyze for all five target compounds. The *Voyager* field GC contains three chromatographic columns; columns **B** and **C** are necessary to identify and quantify each of the five target compounds. Sample headspace injection onto Column **B** will be used to identify Trichloroethene and Tetrachloroethene; headspace injection onto Column **C** will be used to identify the three dichloroethene compounds (1,1-DCEe, cis- and trans-1,2-DCEe). One sample preparation may be used for both injections, as long as the time between injections does not exceed 30 minutes.

Interferences can occur with this method and must be minimized. Sources of interferences include operating internal combustion engines, ambient smoke, nearby solvents (including gasoline, kerosene, mineral spirits, and alcohol), and other materials containing volatile components. Interferences can also occur within the sample matrix itself, both positive and negative. Carryover from a contaminated sample is a common cause of interference, as is the use of contaminated dilution water.

#### B. Instrumentation

The instrument utilized for this study was the PerkinElmer *Voyager* Field Portable Gas Chromatograph and the associated *SiteChart*® Software (a Windows based chromatography package developed by PE). The *Voyager* instrument can be operated without the *SiteChart* software out in the field. To obtain hard copies of the data, the GC must be connected to a PC loaded with *SiteChart* and the data downloaded to the PC with the software running.



The Voyager GC is a chromatographic unit containing three chromatographic columns and two detectors. Each column was developed for separating specific volatile organic compounds. Column A retains and separates heavy compounds (having high boiling points); column B retains and separates mid-range compounds (with moderate boiling points) and column C retains and separates light compounds (with low boiling points). For this validation study and for use at the FEMP, Columns B and C are utilized. TCE and PCE are analyzed for using Column B, and the three dichloroethene compounds (1,1-DCE, t-1,2-DCE, and c-1,2-DCE) are analyzed for using column C. Column A is not used for any FEMP Contaminants of Concern (COCs).

Briefly, gas chromatography works in this manner: Carrier gas (nitrogen) flowing through each column sweeps VOCs into the columns and the stationary phases of each column retain the compounds for a specific period of time as they come in contact with the compounds. As the compounds pass through the column (elute) they are passed through the Photoionization detector (PID) which produces ultraviolet (UV) light at 10.6 electron volts (eV). As the VOCs absorb this energy they are ionized (electrons are emitted). These electrons are passed through an electrical field and create a current based on their concentration, which is enhanced by a multiplier and then fed into the microprocessor. This means of detection produces chromatographic peaks, where analyte concentration is proportional to the area of the peak. The PID is sensitive to unsaturated organic compounds, that is, compounds containing carbon-carbon double or triple bonds. The five FEMP volatile organic COCs are all unsaturated compounds, and the PID is sensitive to each of them. (There is also an Electron Capture Detector (ECD) installed, but it is not sensitive to the majority of the FEMP Volatile COCs, and therefore is not used.)

Sample introduction occurs via manual injection of the headspace gases directly onto the pre-column using a gas-tight syringe, which then releases the gases onto the actual chromatographic column. Normally, 200 microliters of headspace gas are injected.

The instrument is first calibrated with one standard, and the samples are analyzed after a successful calibration has been achieved (as evidenced by acceptable lab control standard recoveries). If the Voyager is being used on the field without being connected to a PC, the results are displayed on the screen. Once analyses are completed, the *Voyager* can be connected to a PC and printer, the data files downloaded to the software, and hard copies of all data can then be obtained. If a mobile lab unit is available, with a generator, the *Voyager*, a PC with *SiteChart*, and a printer can be operated simultaneously. Hard copies of data are then available on a near real-time basis. Analysis time per sample is approximately 25 – 30 minutes.



The Voyager has a large working (linear dynamic) range of approximately three to four orders of magnitude (approximately 50 ppb to 100 ppm), and the calibration is stable for at least an eight -hour time period. This allows the analysis of samples with a large range of analyte concentrations to be analyzed without many dilutions and reanalysis.

### C. Standards

This study was conducted using a custom mix of the five target compounds, which were obtained from SPEX. A second mix was obtained from Fisher Scientific. Both standards were purchased at a concentration of 2000 ug/mL, or 2000 ppm for each compound. These solutions were used for preparing Initial calibration standards (ICAL), Lab Control Standards (LCS), Continuing Calibration Verification (CCV) standards, and for spiking the test soil samples. All samples and standards were analyzed using the headspace gases that are generated by the analytical method.

### D. Samples

Twenty-Two (22) certified VOC - free soils were analyzed for this study. For each soil sample, five microliters of the SPEX 2000 ug/mL stock solution were spiked directly into approximately five grams of the soil. 20 mL of VOC-free water were immediately added to the soil/VOC mixture, and the sample vial was quickly capped with a Teflon septum inserted into a plastic screw-top cap. The soil, VOC stock aliquot, and the water were then shaken for two minutes to disperse the VOCs throughout the mixture. The vial was then inverted and inserted into the heating block for approximately 10 minutes in order for the VOCs to establish equilibrium between the aqueous phase and the gas phase (the headspace). Following this period of time, 200 uL of the headspace gases were injected into the *Voyager*. The first injection occurred on Column B, then another 200 uL aliquot of headspace gas was injected into column C. The effective concentration of each VOC was approximately 2 mg/kg, depending on the actual soil weight that was spiked.

Each day that these validation samples were prepared the instrument was first calibrated with a 2.00 ppm standard, then a method blank and LCS were analyzed to verify calibration. (On some days, the LCS was analyzed after some samples were analyzed. In all cases, the LCS values were acceptable.) A final CCV was analyzed each day to check system stability. (The CCV and the LCS were at the same concentration.) All data from these analyses are included in the Attachments to this report, including a summary spreadsheet.



Each of the test soil samples initially was dry- meaning that they contained less than 1.0 percent water by weight. Actual field samples may well contain various amounts of entrained water, but the addition of 20 mL of VOC-free water to all samples should mitigate any differences in moisture content. Other aliquots of the field samples can be dried and the final results corrected for the percent moisture to give dry-weight equivalent results.

### III. Results and Discussion

Twenty-two (22) soil samples were spiked with 5.0 microliters (uL) of the certified 2000 ug/mL custom mix solution containing the five analytes of interest.

Approximately 5 grams of each soil was massed into a 40-mL VOA vial, and the certified solution was added directly to the soil, immediately followed by the careful addition (no air bubbles) of 20 mL of VOC-free distilled water down the side of the vial. The vial was quickly capped and shaken for two minutes, then placed in the heating block. After 10 minutes, 200 uL of headspace gases were directly injected into the GC.

Each sample was prepared (spiked) and analyzed separately, in successive order as just described above. All results were entered onto the "Analysis/ Calculation Record for Voyager Field GC" spreadsheet. Attachments 1 and 2 contain the results of the 22 samples. It is interesting to note that 11 of the soil samples were more sandy in composition, and the other 11 samples were more "organic" in composition, that is more humic, than the 11 sandy samples. Both groups were evaluated separately, and as one population, because many field samples will likely consist of a mixture of sand and humic components. The results are presented below:

#### 1. Summary of Recoveries and Standard deviations, in percents

	Trichloroethene	Tetrachloroethene	1,1-Dichloroethene	Trans-1,2-dichloroethene	Cis-1,2-dichloroethene
Sand	Mean = 78.3 SD = 18.89	Mean = 75.95 SD = 16.22	Mean = 127.59 SD = 18.85	Mean = 149.19 SD = 15.81	Mean = 65.88 SD = 11.41
Soil	Mean = 98.71 SD = 18.84	Mean = 85.65 SD = 13.58	Mean = 139.22 SD = 16.99	Mean = 148.79 SD = 8.38	Mean = 66.17 SD = 6.17
All samples	Mean = 88.5 SD = 21.16	Mean = 80.80 SD = 15.42	Mean = 133.41 SD = 18.50	Mean = 148.49 SD = 12.35	Mean = 66.02 SD = 8.95

It is evident from the above data that the recoveries for TCE, PCE, and 1,1-DCE are higher in soil matrices than in sand matrices, although there is only one standard deviation or less between the means of the two solid matrices. Recoveries for the trans and cis isomers of 1,2- DCE are virtually the same, although precision is substantially better for the more humic soil matrix.



The recoveries for TCE and PCE, as well as cis-1,2-DCE are less than 100 % for the sample population as a whole (although the TCE recoveries in soil are very close to 100%), whereas the mean recoveries for 1,1-DCE and t-1,2-DCE are well above 100 %, for both types of solid samples. There may be some conversion of TCE, PCE, and/or cis-1,2-DCE to 1,1-DCE and trans-1,2- DCE during the extraction process, which involves heating at 50 degrees C. Heating at lower temperatures reduces equilibrium efficiency, and prolongs the equilibrium process, which then lengthens the analysis time per sample. In addition, the equilibrium process and mechanisms may be different between aqueous sample and solid samples, due to interactions with the solid particles. It may be advantageous to use a calibration standard prepared in the solid matrix instead of the water matrix. However, given the limitations and inherent imprecision of this rapid, semi-quantitative method, the improvement in accuracy from standardization in a solid matrix may not yield much improvement in overall sample analysis accuracy. Standard EPA and SW-846 methods do not require standardization with a solid matrix, due to the wide variety of soil types encountered over even a small surface area and with changes in sample depth.

## 2. LCS Recoveries

Aqueous recoveries for the Laboratory Control Sample exhibit good accuracy (True value is 2.00 ppm):

	Trichloroethene	Tetrachloroethene	1,1-Dichloroethene	Trans-1,2-dichloroethene	Cis-1,2-dichloroethene
Mean	1.97 ppm	2.22 ppm	2.08 ppm	1.93 ppm	1.91 ppm
Standard Deviation	0.24 ppm	.035 ppm	0.25 ppm	0.34 ppm	0.40 ppm
Mean + 2 SD	2.46 ppm	2.91 ppm	2.58 ppm	2.61 ppm	2.71 ppm
Mean - 2 SD	1.48 ppm	1.52 ppm	1.58 ppm	1.25 ppm	1.12 ppm

The  $\pm 2$  Standard deviations represents the 95 % Confidence limits for each compound. These values will be used for the acceptance limits for aqueous LCS and CCV QC samples.



### 3. Method Detection Limits

As specified in 40 CFR, a Method Detection Limit (MDL) Study was performed. This was accomplished by analyzing a low-level standard (approximately two times the expected detection limit) seven times on different days. A standard that was at 100 ppb was analyzed for this purpose. Following analysis of this standard seven times, the following MDLs were obtained:

Trichloroethene:	23.16 ppb
Tetrachloroethene:	22.98 ppb
1,1-Dichloroethene:	33.06 ppb
trans-1,2-Dichloroethene:	41.53 ppb
cis-1,2-Dichloroethene:	24.37 ppb

Based on this data, the Soils Projects requirement of an MDL of 50 ppb has been met for each compound. Soil detection limits will vary based on the amount of soil analyzed, but are generally expected to be around 0.20 mg/kg. Using more soil or injecting more than 200 uL will lower the detection limits, but may create analytical problems resulting from added interferent levels.

### IV. Summary and Conclusions

This study has confirmed the manufacturer's claims about instrument utility, stability, and detection limit capability. The software is user-friendly, and sample analyses can be achieved in less than 30 minutes per sample. The analysis of over 20 spiked soil samples plus ten analyst certification samples has demonstrated good recoveries for all analytes, given the limitations of the unit and inherent with the headspace methodology. The method is capable of detecting FEMP COCs at or below the required project detection limits. Sample analysis is straightforward and calculations have been automated via the use of a validated Excel spreadsheet.

This field GC method should greatly assist soil excavation and characterization activities at the FEMP. Rapid generation of results will result in real-time decision-making capability for project personnel, and result in substantial cost-savings over submitting all samples for fixed-base lab analysis.



## V. Attachments

1. Analysis/ Calculation Record for Voyager Field GC- results for 22 spiked soil samples
2. "Analysis/ Calculation Record for Voyager Field GC" Spreadsheet validation
3. Summary of Spiked sample Recoveries
4. Method Detection Limit data and calculations
5. Laboratory Control Samples summary and Acceptance limits calculations



1. Analysis/ Calculation Record for  
Voyager Field GC- results for 22 spiked  
soil samples



Analyst/ Calculation Record for Voyager Field GC

#	sample ID	Date	Time analyzed	Column	mass of soil or volume of water to sample in vial (use 20" for soil and water/)	volume of soil or volume of water added	injected	injected readout	ppm from	addt dilution factors, if any (use "1" for none)	ANALYTE	ppm in sample (soils = mg/kg)	analyst initials	comments	true value ppm	% Rec.	QC	#	QC calculations		
																			QC	QC	
1	1CAL	10/05/2001	0802		20	20	200	2.000	2.000	1	TCE	2.000									
2	1CAL	10/05/2001	0802		20	20	200	2.000	2.000	1	PCE	2.000									
3	1CAL	10/05/2001	0813		20	20	200	2.000	2.000	1	11DCE	2.000									
4	1CAL	10/05/2001	0813		20	20	200	2.000	2.000	1	11DCE	2.000									
5	1CAL	10/05/2001	0813		20	20	200	2.000	2.000	1	c12DCE	2.000									
6	1CAL	10/05/2001	0738		20	20	200	0.000	0.000	1	TCE	0.000									
7	1CAL	10/05/2001	0738		20	20	200	0.000	0.000	1	PCE	0.000									
8	1CAL	10/05/2001	0750		20	20	200	0.000	0.000	1	11DCE	0.000									
9	1CAL	10/05/2001	0750		20	20	200	0.000	0.000	1	11DCE	0.000									
10	1CAL	10/05/2001	0750		20	20	200	0.000	0.000	1	c12DCE	0.000									
11	1CAL	10/05/2001	0824		20	20	200	1.852	1.852	1	TCE	1.852									
12	1CAL	10/05/2001	0824		20	20	200	2.088	2.088	1	PCE	2.088									
13	1CAL	10/05/2001	0825		20	20	200	1.905	1.905	1	11DCE	1.905									
14	1CAL	10/05/2001	0825		20	20	200	1.807	1.807	1	11DCE	1.807									
15	1CAL	10/05/2001	0825		20	20	200	1.882	1.882	1	c12DCE	1.882									
16	1CAL	10/05/2001	1240		20	20	200	0.000	0.000	1	TCE	0.000									
17	1CAL	10/05/2001	1240		20	20	200	0.000	0.000	1	PCE	0.000									
18	1CAL	10/05/2001	1253		20	20	200	0.000	0.000	1	11DCE	0.000									
19	1CAL	10/05/2001	1253		20	20	200	0.000	0.000	1	11DCE	0.000									
20	1CAL	10/05/2001	1253		20	20	200	0.000	0.000	1	c12DCE	0.000									
21	1CAL	10/05/2001	1317		20	20	200	0.541	0.541	1	TCE	2.217									
22	1CAL	10/05/2001	1317		20	20	200	0.448	0.448	1	PCE	1.816									
23	1CAL	10/05/2001	1331		20	20	200	0.548	0.548	1	11DCE	2.246									
24	1CAL	10/05/2001	1331		20	20	200	0.689	0.689	1	11DCE	2.824									
25	1CAL	10/05/2001	1331		20	20	200	0.330	0.330	1	c12DCE	1.332									
26	1CAL	10/05/2001	1213		20	20	200	2.000	2.000	1	TCE	2.000									
27	1CAL	10/05/2001	1213		20	20	200	2.000	2.000	1	PCE	2.000									
28	1CAL	10/05/2001	1227		20	20	200	2.000	2.000	1	11DCE	2.000									
29	1CAL	10/05/2001	1227		20	20	200	2.000	2.000	1	11DCE	2.000									
30	1CAL	10/05/2001	1227		20	20	200	2.000	2.000	1	c12DCE	2.000									
31	1CAL	10/05/2001	1240		20	20	200	0.000	0.000	1	TCE	0.000									
32	1CAL	10/05/2001	1240		20	20	200	0.000	0.000	1	PCE	0.000									
33	1CAL	10/05/2001	1252		20	20	200	0.000	0.000	1	11DCE	0.000									
34	1CAL	10/05/2001	1252		20	20	200	0.000	0.000	1	11DCE	0.000									
35	1CAL	10/05/2001	1252		20	20	200	0.000	0.000	1	c12DCE	0.000									
36	1CAL	10/05/2001	1307		20	20	200	0.202	0.202	1	TCE	1.184									
37	1CAL	10/05/2001	1307		20	20	200	0.312	0.312	1	PCE	1.224									
38	1CAL	10/05/2001	1318		20	20	200	0.530	0.530	1	11DCE	2.078									
39	1CAL	10/05/2001	1318		20	20	200	0.207	0.207	1	11DCE	2.773									
40	1CAL	10/05/2001	1318		20	20	200	0.720	0.720	1	c12DCE	1.255									
41	1CAL	10/05/2001	1329		20	20	200	0.633	0.633	1	TCE	1.424									
42	1CAL	10/05/2001	1329		20	20	200	0.933	0.933	1	PCE	1.541									
43	1CAL	10/05/2001	1343		20	20	200	0.918	0.918	1	11DCE	2.424									
44	1CAL	10/05/2001	1343		20	20	200	0.788	0.788	1	11DCE	3.082									
45	1CAL	10/05/2001	1343		20	20	200	0.358	0.358	1	TCE	1.398									
46	1CAL	10/05/2001	1355		20	20	200	0.401	0.401	1	TCE	1.573									
47	1CAL	10/05/2001	1355		20	20	200	0.464	0.464	1	PCE	1.820									
48	1CAL	10/05/2001	1407		20	20	200	0.988	0.988	1	11DCE	2.337									
49	1CAL	10/05/2001	1407		20	20	200	0.781	0.781	1	11DCE	3.083									
50	1CAL	10/05/2001	1407		20	20	200	0.347	0.347	1	c12DCE	1.361									
51	1CAL	10/05/2001	1421		20	20	200	0.282	0.282	1	TCE	1.082									
52	1CAL	10/05/2001	1421		20	20	200	0.915	0.915	1	PCE	1.188									
53	1CAL	10/05/2001	1431		20	20	200	0.653	0.653	1	11DCE	2.121									
54	1CAL	10/05/2001	1431		20	20	200	0.744	0.744	1	11DCE	2.802									
55	1CAL	10/05/2001	1431		20	20	200	0.341	0.341	1	c12DCE	1.284									
56	1CAL	10/05/2001	1449		20	20	200	0.388	0.388	1	TCE	1.481									
57	1CAL	10/05/2001	1449		20	20	200	0.421	0.421	1	PCE	1.588									
58	1CAL	10/05/2001	1501		20	20	200	0.658	0.658	1	11DCE	2.471									
59	1CAL	10/05/2001	1501		20	20	200	0.822	0.822	1	11DCE	3.098									
60	1CAL	10/05/2001	1501		20	20	200	0.383	0.383	1	c12DCE	1.443									
61	1CAL	10/05/2001	1512		20	20	200	0.408	0.408	1	TCE	1.537									
62	1CAL	10/05/2001	1512		20	20	200	0.458	0.458	1	PCE	1.729									
63	1CAL	10/05/2001	1525		20	20	200	0.658	0.658	1	11DCE	2.482									
64	1CAL	10/05/2001	1525		20	20	200	0.635	0.635	1	11DCE	3.145									
65	1CAL	10/05/2001	1525		20	20	200	0.382	0.382	1	c12DCE	1.439									

Voyager calc. sheets

66	LCS	B	10/08/2001	1537	20.00	20	200	0.983	1	TCE	0.983	RC	old LCS	2.00	49.15	66
67	LCS	B	10/08/2001	1537	20.00	20	200	1.157	1	PCE	1.157	RC		2.00	57.85	67
68	LCS	C	10/08/2001	1548	20.00	20	200	2.333	1	11DCE	2.333	RC		2.00	118.65	68
69	LCS	C	10/08/2001	1548	20.00	20	200	2.400	1	112DCE	2.400	RC		2.00	120.00	69
70	LCS	C	10/08/2001	1548	20.00	20	200	1.739	1	c12DCE	1.739	RC		2.00	86.95	70
71	ICAL	B	10/09/2001	0908	20.00	20	200	2.000	1	TCE	2.000	RC				71
72	ICAL	B	10/09/2001	0908	20.00	20	200	2.000	1	PCE	2.000	RC				72
73	ICAL	C	10/09/2001	0920	20.00	20	200	2.000	1	11DCE	2.000	RC				73
74	ICAL	C	10/09/2001	0920	20.00	20	200	2.000	1	112DCE	2.000	RC				74
75	ICAL	C	10/09/2001	0920	20.00	20	200	2.000	1	c12DCE	2.000	RC				75
76	MB	B	10/09/2001	0930	20.00	20	200	0.039	1	TCE	0.039	RC	<50 ppb- OK			76
77	MB	B	10/09/2001	0930	20.00	20	200	0.043	1	PCE	0.043	RC	<50 ppb- OK			77
78	MB	C	10/09/2001	0942	20.00	20	200	0.000	1	11DCE	0.000	RC	<50 ppb- OK			78
79	MB	C	10/09/2001	0942	20.00	20	200	0.007	1	112DCE	0.007	RC	<50 ppb- OK			79
80	MB	C	10/09/2001	0942	20.00	20	200	0.000	1	c12DCE	0.000	RC	<50 ppb- OK			80
81	soil test 8	B	10/09/2001	0956	5.06	20	200	0.396	1	TCE	1.565	RC	TV= 1.98 mg/kg	1.98	79.05	81
82	soil test 8	B	10/09/2001	0956	5.06	20	200	0.352	1	PCE	1.391	RC	TV= 1.98 mg/kg	1.98	70.27	82
83	soil test 8	C	10/09/2001	1008	5.06	20	200	0.715	1	11DCE	2.826	RC	TV= 1.98 mg/kg	1.98	142.73	83
84	soil test 8	C	10/09/2001	1008	5.06	20	200	0.875	1	112DCE	2.668	RC	TV= 1.98 mg/kg	1.98	134.75	84
85	soil test 8	C	10/09/2001	1008	5.06	20	200	0.299	1	c12DCE	1.182	RC	TV= 1.98 mg/kg	1.98	59.69	85
86	soil test 9	B	10/09/2001	1019	5.06	20	200	0.570	1	TCE	2.253	RC	TV= 1.98 mg/kg	1.98	113.79	86
87	soil test 9	B	10/09/2001	1019	5.06	20	200	0.475	1	PCE	1.877	RC	TV= 1.98 mg/kg	1.98	94.82	87
88	soil test 9	C	10/09/2001	1030	5.06	20	200	0.800	1	11DCE	3.162	RC	TV= 1.98 mg/kg	1.98	159.70	88
89	soil test 9	C	10/09/2001	1030	5.06	20	200	0.770	1	112DCE	3.043	RC	TV= 1.98 mg/kg	1.98	153.71	89
90	soil test 9	C	10/09/2001	1030	5.06	20	200	0.395	1	c12DCE	1.581	RC	TV= 1.98 mg/kg	1.98	78.85	90
91	CCV	B	10/09/2001	1230	20.00	20	200	1.660	1	TCE	1.660	RC	TV= 2.00 ppm	2.00	83.00	91
92	CCV	B	10/09/2001	1230	20.00	20	200	1.607	1	PCE	1.607	RC	TV= 2.00 ppm	2.00	80.35	92
93	CCV	C	10/09/2001	1241	20.00	20	200	1.912	1	11DCE	1.912	RC	TV= 2.00 ppm	2.00	95.60	93
94	CCV	C	10/09/2001	1241	20.00	20	200	1.408	1	112DCE	1.408	RC	TV= 2.00 ppm	2.00	70.40	94
95	CCV	C	10/09/2001	1241	20.00	20	200	1.576	1	c12DCE	1.576	RC	TV= 2.00 ppm	2.00	78.80	95
96	soil test 10	B	10/09/2001	1257	4.97	20	200	0.273	1	TCE	1.099	RC	TV= 2.01 mg/kg	2.01	54.66	96
97	soil test 10	B	10/09/2001	1257	4.97	20	200	0.228	1	PCE	0.918	RC	TV= 2.01 mg/kg	2.01	45.85	97
98	soil test 10	C	10/09/2001	1307	4.97	20	200	0.547	1	11DCE	2.201	RC	TV= 2.01 mg/kg	2.01	109.51	98
99	soil test 10	C	10/09/2001	1307	4.97	20	200	0.561	1	112DCE	2.258	RC	TV= 2.01 mg/kg	2.01	112.32	99
100	soil test 10	C	10/09/2001	1307	4.97	20	200	0.270	1	c12DCE	1.087	RC	TV= 2.01 mg/kg	2.01	54.06	100
101	soil test 11	B	10/09/2001	1320	4.97	20	200	0.382	1	TCE	1.537	RC	TV= 2.01 mg/kg	2.01	76.48	101
102	soil test 11	B	10/09/2001	1320	4.97	20	200	0.311	1	PCE	1.252	RC	TV= 2.01 mg/kg	2.01	62.28	102
103	soil test 11	C	10/09/2001	1332	4.97	20	200	0.782	1	11DCE	3.147	RC	TV= 2.01 mg/kg	2.01	158.56	103
104	soil test 11	C	10/09/2001	1332	4.97	20	200	0.777	1	112DCE	3.127	RC	TV= 2.01 mg/kg	2.01	155.56	104
105	soil test 11	C	10/09/2001	1332	4.97	20	200	0.200	1	c12DCE	0.805	RC	TV= 2.01 mg/kg	2.01	40.04	105
106	soil test 12	B	10/09/2001	1345	5.18	20	200	0.422	1	TCE	1.636	RC	TV= 1.94 mg/kg	1.94	84.31	106
107	soil test 12	B	10/09/2001	1345	5.18	20	200	0.435	1	PCE	1.686	RC	TV= 1.94 mg/kg	1.94	88.91	107
108	soil test 12	C	10/09/2001	1357	5.18	20	200	0.740	1	11DCE	2.888	RC	TV= 1.94 mg/kg	1.94	147.85	108
109	soil test 12	C	10/09/2001	1357	5.18	20	200	0.721	1	112DCE	2.795	RC	TV= 1.94 mg/kg	1.94	144.05	109
110	soil test 12	C	10/09/2001	1357	5.18	20	200	0.322	1	c12DCE	1.248	RC	TV= 1.94 mg/kg	1.94	64.33	110
111	soil test 13	B	10/09/2001	1408	5.18	20	200	0.810	1	TCE	2.364	RC	TV= 1.94 mg/kg	1.94	121.87	111
112	soil test 13	B	10/09/2001	1408	5.18	20	200	0.554	1	PCE	2.147	RC	TV= 1.94 mg/kg	1.94	110.68	112
113	soil test 13	C	10/09/2001	1419	5.18	20	200	0.811	1	11DCE	3.143	RC	TV= 1.94 mg/kg	1.94	162.03	113
114	soil test 13	C	10/09/2001	1419	5.18	20	200	0.747	1	112DCE	2.895	RC	TV= 1.94 mg/kg	1.94	149.24	114
115	soil test 13	C	10/09/2001	1419	5.18	20	200	0.355	1	c12DCE	1.376	RC	TV= 1.94 mg/kg	1.94	70.93	115
116	soil test 14	B	10/09/2001	1434	4.93	20	200	0.515	1	TCE	2.089	RC	TV= 2.03 mg/kg	2.03	102.92	116
117	soil test 14	B	10/09/2001	1434	4.93	20	200	0.407	1	PCE	1.651	RC	TV= 2.03 mg/kg	2.03	81.34	117
118	soil test 14	C	10/09/2001	1448	4.93	20	200	0.759	1	11DCE	3.079	RC	TV= 2.03 mg/kg	2.03	151.68	118
119	soil test 14	C	10/09/2001	1448	4.93	20	200	0.698	1	112DCE	2.832	RC	TV= 2.03 mg/kg	2.03	139.49	119
120	soil test 14	C	10/09/2001	1448	4.93	20	200	0.334	1	c12DCE	1.355	RC	TV= 2.03 mg/kg	2.03	66.75	120
121	soil test 15	B	10/09/2001	1457	4.93	20	200	0.691	1	TCE	2.803	RC	TV= 2.03 mg/kg	2.03	138.09	121
122	soil test 15	B	10/09/2001	1457	4.93	20	200	0.551	1	PCE	2.235	RC	TV= 2.03 mg/kg	2.03	110.11	122
123	soil test 15	C	10/09/2001	1508	4.93	20	200	0.815	1	11DCE	3.306	RC	TV= 2.03 mg/kg	2.03	162.87	123
124	soil test 15	C	10/09/2001	1508	4.93	20	200	0.751	1	112DCE	3.047	RC	TV= 2.03 mg/kg	2.03	150.08	124
125	soil test 15	C	10/09/2001	1508	4.93	20	200	0.375	1	c12DCE	1.521	RC	TV= 2.03 mg/kg	2.03	74.94	125

126	final CCV	B	10/09/2001	1523	20.00	20	200	1.688	1	TCE	1.688	RC		2.00	84.40	126
127	final CCV	B	10/09/2001	1523	20.00	20	200	1.695	1	PCE	1.695	RC		2.00	84.75	127
128	final CCV	C	10/09/2001	1534	20.00	20	200	1.817	1	11DCE	1.817	RC		2.00	90.85	128
129	final CCV	C	10/09/2001	1534	20.00	20	200	1.465	1	112DCE	1.465	RC		2.00	73.25	129
130	final CCV	C	10/09/2001	1534	20.00	20	200	1.537	1	c12DCE	1.537	RC		2.00	76.65	130
131	ICAL	B	10/10/2001	0937	20.00	20	200	2.000	1	TCE	2.000	RC				131
132	ICAL	B	10/10/2001	0937	20.00	20	200	2.000	1	PCE	2.000	RC				132
133	ICAL	C	10/10/2001	0948	20.00	20	200	2.000	1	11DCE	2.000	RC				133
134	ICAL	C	10/10/2001	0948	20.00	20	200	2.000	1	112DCE	2.000	RC				134
135	ICAL	C	10/10/2001	0948	20.00	20	200	2.000	1	c12DCE	2.000	RC				135
136	MB	B	10/10/2001	1000	20.00	20	200	0.017	1	TCE	0.017	RC	<50 ppb - OK			136
137	MB	B	10/10/2001	1000	20.00	20	200	0.010	1	PCE	0.010	RC	<50 ppb - OK			137
138	MB	C	10/10/2001	1012	20.00	20	200	0.000	1	11DCE	0.000	RC	<50 ppb - OK			138
139	MB	C	10/10/2001	1012	20.00	20	200	0.010	1	112DCE	0.010	RC	<50 ppb - OK			139
140	MB	C	10/10/2001	1012	20.00	20	200	0.000	1	c12DCE	0.000	RC	<50 ppb - OK			140
141	soil test 16	B	10/10/2001	1024	4.92	20	200	0.502	1	TCE	2.041	RC	TV = 2.03 mg/kg	2.03	100.52	141
142	soil test 16	B	10/10/2001	1024	4.92	20	200	0.419	1	PCE	1.703	RC	TV = 2.03 mg/kg	2.03	83.90	142
143	soil test 16	C	10/10/2001	1035	4.92	20	200	0.685	1	11DCE	2.785	RC	TV = 2.03 mg/kg	2.03	137.17	143
144	soil test 16	C	10/10/2001	1035	4.92	20	200	0.788	1	112DCE	3.203	RC	TV = 2.03 mg/kg	2.03	157.80	144
145	soil test 16	C	10/10/2001	1035	4.92	20	200	0.309	1	c12DCE	1.256	RC	TV = 2.03 mg/kg	2.03	61.88	145
146	soil test 17	B	10/10/2001	1422	4.92	20	200	0.397	1	TCE	1.614	RC	TV = 2.03 mg/kg	2.03	79.50	146
147	soil test 17	B	10/10/2001	1422	4.92	20	200	0.377	1	PCE	1.533	RC	TV = 2.03 mg/kg	2.03	75.49	147
148	soil test 17	C	10/10/2001	1434	4.92	20	200	0.520	1	11DCE	2.114	RC	TV = 2.03 mg/kg	2.03	104.13	148
149	soil test 17	C	10/10/2001	1434	4.92	20	200	0.667	1	112DCE	2.711	RC	TV = 2.03 mg/kg	2.03	133.57	149
150	soil test 17	C	10/10/2001	1434	4.92	20	200	0.262	1	c12DCE	1.065	RC	TV = 2.03 mg/kg	2.03	52.47	150
151	soil test 18	B	10/10/2001	1448	4.92	20	200	0.505	1	TCE	2.053	RC	TV = 2.03 mg/kg	2.03	101.13	151
152	soil test 18	B	10/10/2001	1448	4.92	20	200	0.413	1	PCE	1.679	RC	TV = 2.03 mg/kg	2.03	82.70	152
153	soil test 18	C	10/10/2001	1457	4.92	20	200	0.625	1	11DCE	2.541	RC	TV = 2.03 mg/kg	2.03	125.16	153
154	soil test 18	C	10/10/2001	1457	4.92	20	200	0.812	1	112DCE	3.301	RC	TV = 2.03 mg/kg	2.03	162.60	154
155	soil test 18	C	10/10/2001	1457	4.92	20	200	0.352	1	c12DCE	1.431	RC	TV = 2.03 mg/kg	2.03	70.49	155
156	CCV	B	10/10/2001	1517	20.00	20	200	2.145	1	TCE	2.145	RC	OK	2.00	107.25	156
157	CCV	B	10/10/2001	1517	20.00	20	200	2.326	1	PCE	2.326	RC	OK	2.00	116.30	157
158	CCV	C	10/10/2001	1528	20.00	20	200	1.983	1	11DCE	1.983	RC	OK	2.00	99.15	158
159	CCV	C	10/10/2001	1528	20.00	20	200	2.021	1	112DCE	2.021	RC	OK	2.00	101.05	159
160	CCV	C	10/10/2001	1528	20.00	20	200	1.887	1	c12DCE	1.887	RC	OK	2.00	94.35	160
161	ICAL	B	10/11/2001	0945	20.00	20	200	2.000	1	TCE	2.000	RC				161
162	ICAL	B	10/11/2001	0945	20.00	20	200	2.000	1	PCE	2.000	RC				162
163	ICAL	C	10/11/2001	0957	20.00	20	200	2.000	1	11DCE	2.000	RC				163
164	ICAL	C	10/11/2001	0957	20.00	20	200	2.000	1	112DCE	2.000	RC				164
165	ICAL	C	10/11/2001	0957	20.00	20	200	2.000	1	c12DCE	2.000	RC				165
166	MB	B	10/11/2001	1009	20.00	20	200	0.017	1	TCE	0.017	RC	< 50 ppb - ok			166
167	MB	B	10/11/2001	1009	20.00	20	200	0.014	1	PCE	0.014	RC	< 50 ppb - ok			167
168	MB	C	10/11/2001	1019	20.00	20	200	0.000	1	11DCE	0.000	RC	< 50 ppb - ok			168
169	MB	C	10/11/2001	1019	20.00	20	200	0.000	1	112DCE	0.000	RC	< 50 ppb - ok			169
170	MB	C	10/11/2001	1019	20.00	20	200	0.000	1	c12DCE	0.000	RC	< 50 ppb - ok			170
171	soil test 19	B	10/11/2001	1037	5.08	20	200	0.381	1	TCE	1.500	RC	TV = 1.97 mg/kg	1.97	76.14	171
172	soil test 19	B	10/11/2001	1037	5.08	20	200	0.346	1	PCE	1.362	RC	TV = 1.97 mg/kg	1.97	69.15	172
173	soil test 19	C	10/11/2001	1047	5.08	20	200	0.682	1	11DCE	2.685	RC	TV = 1.97 mg/kg	1.97	136.30	173
174	soil test 19	C	10/11/2001	1047	5.08	20	200	0.731	1	112DCE	2.878	RC	TV = 1.97 mg/kg	1.97	146.09	174
175	soil test 19	C	10/11/2001	1047	5.08	20	200	0.309	1	c12DCE	1.217	RC	TV = 1.97 mg/kg	1.97	61.75	175
176	soil test 20	B	10/11/2001	1100	5.08	20	200	0.510	1	TCE	2.008	RC	TV = 1.97 mg/kg	1.97	101.92	176
177	soil test 20	B	10/11/2001	1100	5.08	20	200	0.439	1	PCE	1.728	RC	TV = 1.97 mg/kg	1.97	87.73	177
178	soil test 20	C	10/11/2001	1112	5.08	20	200	0.712	1	11DCE	2.803	RC	TV = 1.97 mg/kg	1.97	142.29	178
179	soil test 20	C	10/11/2001	1112	5.08	20	200	0.768	1	112DCE	3.024	RC	TV = 1.97 mg/kg	1.97	153.48	179
180	soil test 20	C	10/11/2001	1112	5.08	20	200	0.341	1	c12DCE	1.343	RC	TV = 1.97 mg/kg	1.97	68.15	180
181	soil test 21	B	10/11/2001	1124	5.18	20	200	0.398	1	TCE	1.537	RC	TV = 1.93 mg/kg	1.93	79.62	181
182	soil test 21	B	10/11/2001	1124	5.18	20	200	0.364	1	PCE	1.405	RC	TV = 1.93 mg/kg	1.93	72.82	182
183	soil test 21	C	10/11/2001	1136	5.18	20	200	0.645	1	11DCE	2.490	RC	TV = 1.93 mg/kg	1.93	129.03	183
184	soil test 21	C	10/11/2001	1136	5.18	20	200	0.722	1	112DCE	2.788	RC	TV = 1.93 mg/kg	1.93	144.44	184
185	soil test 21	C	10/11/2001	1136	5.18	20	200	0.323	1	c12DCE	1.247	RC	TV = 1.93 mg/kg	1.93	64.62	185
186	soil test 22	B	10/11/2001	1150	5.18	20	200	0.498	1	TCE	1.923	RC	TV = 1.93 mg/kg	1.93	99.63	186
187	soil test 22	B	10/11/2001	1150	5.18	20	200	0.404	1	PCE	1.560	RC	TV = 1.93 mg/kg	1.93	80.82	187
188	soil test 22	C	10/11/2001	1200	5.18	20	200	0.672	1	11DCE	2.595	RC	TV = 1.93 mg/kg	1.93	134.43	188
189	soil test 22	C	10/11/2001	1200	5.18	20	200	0.777	1	112DCE	3.000	RC	TV = 1.93 mg/kg	1.93	155.44	189
190	soil test 22	C	10/11/2001	1200	5.18	20	200	0.355	1	c12DCE	1.371	RC	TV = 1.93 mg/kg	1.93	71.02	190
191	CCV	B	10/11/2001	1235	20.00	20	200	2.110	1	TCE	2.110	RC		2.00	105.50	191
192	CCV	B	10/11/2001	1235	20.00	20	200	2.350	1	PCE	2.350	RC		2.00	117.50	192
193	CCV	C	10/11/2001	1223	20.00	20	200	1.758	1	11DCE	1.758	RC		2.00	87.90	193
194	CCV	C	10/11/2001	1223	20.00	20	200	1.692	1	112DCE	1.692	RC		2.00	84.60	194
195	CCV	C	10/11/2001	1223	20.00	20	200	1.285	1	c12DCE	1.285	RC		2.00	64.25	195

000014

2. "Analysis/ Calculation Record for  
Voyager Field GC" Spreadsheet  
validation

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4125

# Validation of Voyager Calculation Spreadsheet (Excel)

## Cell Formula

$$\text{final result (ppm in sample)} = \frac{\text{ppm from instr.} \times 200 \text{ ul} \times \text{Volume of water added to sample}}{\text{ul injected} \times \text{mass or volume}}$$

X dilution factors

Examples: line 26 - ICAL for TCE :

$$\text{final result} = \left( \frac{(2.00 \text{ ppm}) \times (200 \text{ ul}) \times (20 \text{ ml})}{200 \text{ ul} \times 20 \text{ ml}} \right) \times 1 = \boxed{2.00 \text{ ppm}}$$

line 65: soil test 7, for c-1,2-DCE :

$$\text{final result} = \left( \frac{(0.382 \text{ ppm}) \times (200 \text{ ul}) \times (20 \text{ ml})}{200 \text{ ul} \times 5.31 \text{ g}} \right) \times 1 = \boxed{1.439 \text{ ppm}}$$

## QC calculations    QC % Recovery

cell formulas:

$$\% R = \frac{\text{final result (ppm)}}{\text{True Value (ppm)}} \times 100\%$$

000016

Zg 2

4125

examples = line 22, soil test 1, ACE

$$\% R = \frac{2.217 \text{ ppm}}{2.05 \text{ ppm}} \times 100\% = \boxed{108.15\%}$$

(rounding in software yields 108.16% on spreadsheet)

line 86<sup>91</sup> - CCV for TCE

$$\% R = \frac{1.660}{2.00} \times 100 = \boxed{83.00\%}$$

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### 3. Summary of Spiked sample Recoveries

run #	True Value	TCE		PCE		1,1- DCE		t-1,2-DCE		c-1,2-DCE	
		result	%R	result	%R	result	%R	result	%R	result	%R
1	2.05	2.22	108.29	1.82	88.78	2.25	109.76	2.82	137.56	1.35	65.85
2	1.96	1.18	60.20	1.22	62.24	2.08	106.12	2.77	141.33	1.26	64.29
3	1.96	1.42	72.45	1.54	78.57	2.42	123.47	3.08	157.14	1.40	71.43
4	1.96	1.57	80.10	1.82	92.86	2.34	119.39	3.06	156.12	1.36	69.39
5	1.88	1.06	56.38	1.19	63.30	2.12	112.77	2.80	148.94	1.28	68.09
6	1.88	1.46	77.66	1.59	84.57	2.47	131.38	3.10	164.89	1.44	76.60
7	1.88	1.54	81.91	1.73	92.02	2.48	131.91	3.15	167.55	1.44	76.60
8	1.98	1.57	79.29	1.39	70.20	2.83	142.93	2.67	134.85	1.18	59.60
9	1.98	2.25	113.64	1.88	94.95	3.16	159.60	3.04	153.54	1.56	78.79
10	2.01	1.10	54.73	0.92	45.77	2.20	109.45	2.26	112.44	1.09	54.23
11	2.01	1.54	76.62	1.25	62.19	3.15	156.72	3.13	155.72	0.80	39.80
12	1.94	1.64	84.54	1.69	87.11	2.87	147.94	2.80	144.33	1.25	64.43
13	1.94	2.36	121.65	2.15	110.82	3.14	161.86	2.90	149.48	1.38	71.13
14	2.03	2.09	102.96	1.65	81.28	3.08	151.72	2.83	139.41	1.36	67.00
15	2.03	2.80	137.93	2.24	110.34	3.30	162.56	3.05	150.25	1.52	74.88
16	2.03	2.04	100.49	1.70	83.74	2.78	136.95	3.20	157.64	1.26	62.07
17	2.03	1.61	79.31	1.53	75.37	2.11	103.94	2.71	133.50	1.06	52.22
18	2.03	2.05	100.99	1.68	82.76	2.54	125.12	3.30	162.56	1.43	70.44
19	1.97	1.50	76.14	1.36	69.04	2.68	136.04	2.88	146.19	1.22	61.93
20	1.97	2.01	102.03	1.73	87.82	2.80	142.13	3.02	153.30	1.34	68.02
21	1.93	1.54	79.79	1.41	73.06	2.49	129.02	2.79	144.56	1.25	64.77
22	1.93	1.93	100.00	1.56	80.83	2.59	134.20	3.00	155.44	1.37	70.98

results are in ppm, dry weight (mg/kg)

means:		88.50	80.80	133.41	148.49	66.02
std. Dev.		21.16	15.42	18.50	12.35	8.95
95% conf. Intervals	upper	130.83	111.65	170.40	173.19	83.93
	lower	46.18	49.96	96.42	123.79	48.12
sand means (#1-11)		78.30	75.95	127.59	148.19	65.88
soils means (#12-22)		98.71	85.65	139.22	148.79	66.17
sand Std.dev.		18.89	16.22	18.85	15.81	11.41
soils std. Dev		18.84	13.58	16.99	8.38	6.17

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4. Method Detection Limit data and calculations

## FIELD GC METHOD DETECTION LIMITS CALCULATION

10/16/2001 15:52 nominal value = 100 ppb

trial	TCE	PCE	1,1-DCE	t-1,2-DCE	c-1,2-DCE
1	102	108	90	131	78
2	83	107	94	139	72
3	95	101	107	120	85
4	88	110	113	150	92
5	84	105	93	141	70
6	83	89	82	159	82
7	84	109	100	151	75

std. Dev.            7.37        7.31        10.52        13.21        7.76  
 MDL (water)        23.16        22.98        33.06        41.53        24.37 in ppb  
 MDL (soil- 5 g)    92.63        91.94        132.26        166.13        97.50 in ppb

MDL (soil- 5 g)	0.09	0.09	0.13	0.17	0.10	in mg/kg
FRL (mg/kg)	25	3.6	0.41	0.16	0.16	

5. Laboratory Control Samples summary  
and Acceptance limits calculations

2 ppm LCS data: Sept. 27- Oct. 11, 2001

results are in mg/L

TCE	PCE	11DCE	t-1,2DCE	c-1,2-DCE
2.24	2.49	1.91	1.81	1.98
1.87	2.2	2.44	2.34	2.6
1.88	2	2.44	1.91	1.95
1.85	2.09	2.07	2.21	2.34
2.04	2.9	2.15	2.04	2.21
1.86	2.19	2.33	2.4	1.74
1.63	2.05	1.91	1.41	1.58
2.22	2.3	1.82	1.47	1.54
2.4	2.6	1.98	2.02	1.89
1.66	1.61	1.76	1.69	1.29
1.69	1.7			
2.15	2.33			
2.11	2.35			

95 % C.I.

mean	1.97	2.22	2.08	1.93	1.91
std. Dev	0.24	0.35	0.25	0.34	0.40
mean + 2 SD	2.46	2.91	2.58	2.61	2.71
mean- 2 SD	1.48	1.52	1.58	1.25	1.12
95% conf. Limits (in percent recov.)	74 - 123	76-146	79-129	62-131	56-136