

Observations and Practical Experience in Soil Gas Analysis from a Laboratory Perspective

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Overview of Discussion Points

- Canister sample holding time
 - Stability study results
- SW846 8260B vs. EPA TO-15
 - Observations & Recommendations

Canister Sample Holding Time

- March 2007: VOC Stability Study
 - Goal: Evaluate stability of VOCs in real world ambient air
 - Data presented at May 2007 AWMA Symposium on Air Quality Measurement Methods and Technology
- Collected 360L ambient air sample, spiked with 76 VOCs
- Analyzed 32 spiked canisters
 - Day 1, 3, 7, 10, 14, 19, 24, 30, 64, 91, 339
 - Day 1 = 10 replicates. All others = 5 replicates (3 replicates for Day 339)



Canister VOC Holding Time Study

- For all 76 VOCs:
 - Day 30 Mean %Recovery =100.1%
 - Day 1-30 Mean Relative Standard Deviation (RSD, n=8) = 7.0%
- Looking at a select list for an extended period:

Compound	Day 1 (AVG 10)	Day 30	Day 30 %R	Day 339 (AVG 3)	Day 339 %R
Vinyl Chloride	0.4642	0.3780	81.4%	0.5037	108.5%
1,1-Dichloroethene	0.5147	0.4986	96.9%	0.5547	107.8%
Benzene	1.1255	1.1296	100.4%	1.3277	118.0%
Trichloroethene	0.6022	0.6316	104.9%	0.6650	110.4%
Toluene	2.8826	2.8692	99.5%	3.3020	114.5%
Tetrachloroethene	0.6404	0.6708	104.7%	0.7913	123.6%

Naphthalene	0.5030	0.5184	103.1%
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NOTE: Units = $\mu\text{g}/\text{m}^3$

- These results show that the majority of VOCs, including naphthalene, should be stable in a canister for at least 30 days

Canister VOC Holding Time Study

Compound	CAS Stability Study (2007)			Oliver <i>et al.</i> (1986)	Brymer <i>et al.</i> (1996)	Bontempo <i>et al.</i> (2004)
	Day 1, µg/m ³	Day 30, µg/m ³	% Change	% Change (days)	% Change (days)	% Change (days)
Vinyl Chloride	0.464	0.378	18.6%	11% (7)	7.4% (30)	2.8% (33)
1,1-Dichloroethene	0.515	0.500	3.1%	6.3% (7)	5.0% (30)	5.7% (33)
Benzene	1.126	1.130	0.36%	1.9% (7)	6.5% (30)	63.5% (33)
Trichloroethene	0.602	0.632	4.9%	4.9% (7)	3.2% (30)	7.3% (33)
Toluene	2.883	2.869	0.46%	2.3% (7)	9.8% (30)	65.8% (33)
Tetrachloroethene	0.640	0.671	4.8%	10% (7)	6.3% (30)	7.3% (33)
Naphthalene	0.503	0.518	3.1%	N/A	32% (30)	N/A

Oliver *et al.*: ambient air used, 5.1-11.1 µg/m³ spike level, Summa canisters, GC/ECD/FID analysis (data from 6L new canisters)

Brymer *et al.*: humidified N₂ used, 6.8-20.0 µg/m³ spike level, Summa canisters, GC/PID/FID/ELCD analysis

Bontempo *et al.*: humidified N₂, 0.026-0.056 µg/m³ spike level, Summa/Silonite/Silcosteel canisters, GC/MS

Soil Gas Analysis

- Observation: Many practitioners have informed us that their confirmatory TO-15 canister results do not agree with 8260B onsite results
- General trend shows TO-15 results higher than 8260B
- Observation: CA has no lab certification program for soil gas (or any vapor phase) samples (8260B or TO-15)

Use of SW846 8260B

- Method allows analysis of vapor phase samples
 - Section 1.2 “For air samples, Method 5041 provides methodology for desorbing volatile organics from trapping media (Methods 0010, 0030, and 0031). In addition, direct analysis utilizing a sample loop is used for sub-sampling from Tedlar® bags (Method 0040).”
- The method does not have a precisely defined procedure for vapor phase sample introduction with purge and trap analytical equipment—therefore...
- Analytical procedures must be (significantly) modified to accommodate vapor phase samples

Use of SW846 8260B

- In 2004, CAS performed an informal confidential poll of 8 mobile/fixed labs performing 8260B analysis of soil gas samples
 - 3 different syringe types were used for sample transfer
 - 5 different volumes of analysis were used (1ml- 100ml)
 - 2 different means of instrument calibration
 - 4 different means of sample introduction
- These results show that there is no unified approach for analyzing soil gas via 8260B.

Use of SW846 8260B

- In general, procedures described to us in the survey did not include any quality control check to *specifically* measure potential losses of vapor phase samples at the point of introduction in the analytical system
- QC checks being performed (e.g. MS/MSD) were not relevant to the actual sample matrix (soil gas)

Recommendations

- Increase canister holding time from 72 hours to at least 30 days
- Establish state laboratory certification for soil gas analysis by SW846-8260B and EPA TO-15
- Clarify procedure for 8260B analysis (sample introduction)
- Require lab to perform some type of vapor phase check sample in every batch
- Revise Soil Gas Advisory document to more easily allow soil gas sampling with canisters/EPA TO-15
- Regulators should *demand* high quality and legally defensible data for risk assessment purposes

Questions/Discussion

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