

A Passive Canister Sampling Challenge Encountered During the EPA SAT Program

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Challenge Encountered

- At select SAT sites a grouped pattern of four VOCs identified.
- Acrylonitrile, Dichloromethane, Toluene, and Styrene
- Grouped VOCs not typically measured in ambient air samples.
- Measured concentrations not typical of ambient air samples.

Possible Causes -- Laboratory

- As Dichloromethane (Methylene Chloride) and Toluene are regularly used in laboratories, the potential for a laboratory contamination was investigated.

Air Toxics Laboratory Air Sample

- Grouped compounds either not identified or identified at low concentrations in Air Toxics lab air sample

AirTox Lab
Air

Sampler Name
Timer#
Sample Date
Analysis Date
Canister #
Sample Description

10/14/2009
10/15/2009
A21026

Lab Air

ANALYTE	RESULT	UNITS
1,1,2-Trichloroethane	ND	ppbv
1,2,4-Trimethylbenzene	ND	ppbv
1,2-Dichloroethane	ND	ppbv
1,2-Dichloropropane	ND	ppbv
1,3,5-Trimethylbenzene	ND	ppbv
1,3-Butadiene	ND	ppbv
Acetonitrile	ND	ppbv
Acetylene	0.43	ppbv
Acrolein	ND	ppbv
Acrylonitrile	ND	ppbv
Benzene	0.11	ppbv
Bromomethane	ND	ppbv
Carbon Disulfide	ND	ppbv
Carbon Tetrachloride	0.10	ppbv
Chlorobenzene	ND	ppbv
Chloroethane	ND	ppbv
Chloroform	ND	ppbv
Chloromethane	0.66	ppbv
Dichlorodifluoromethane	0.67	ppbv
Dichloromethane	0.10	ppbv
Dichlorotetrafluoroethane	ND	ppbv
Ethylbenzene	ND	ppbv
m,p-Xylene	ND	ppbv
Methyl Ethyl Ketone	ND	ppbv
Methyl Isobutyl Ketone	ND	ppbv
n-Octane	ND	ppbv
o-Xylene	ND	ppbv
Propylene	0.16	ppbv
Styrene	ND	ppbv
Tetrachloroethylene	ND	ppbv
Toluene	0.25	ppbv
Trichloroethylene	ND	ppbv
Trichlorofluoromethane	0.29	ppbv
Trichlorotrifluoroethane	0.10	ppbv

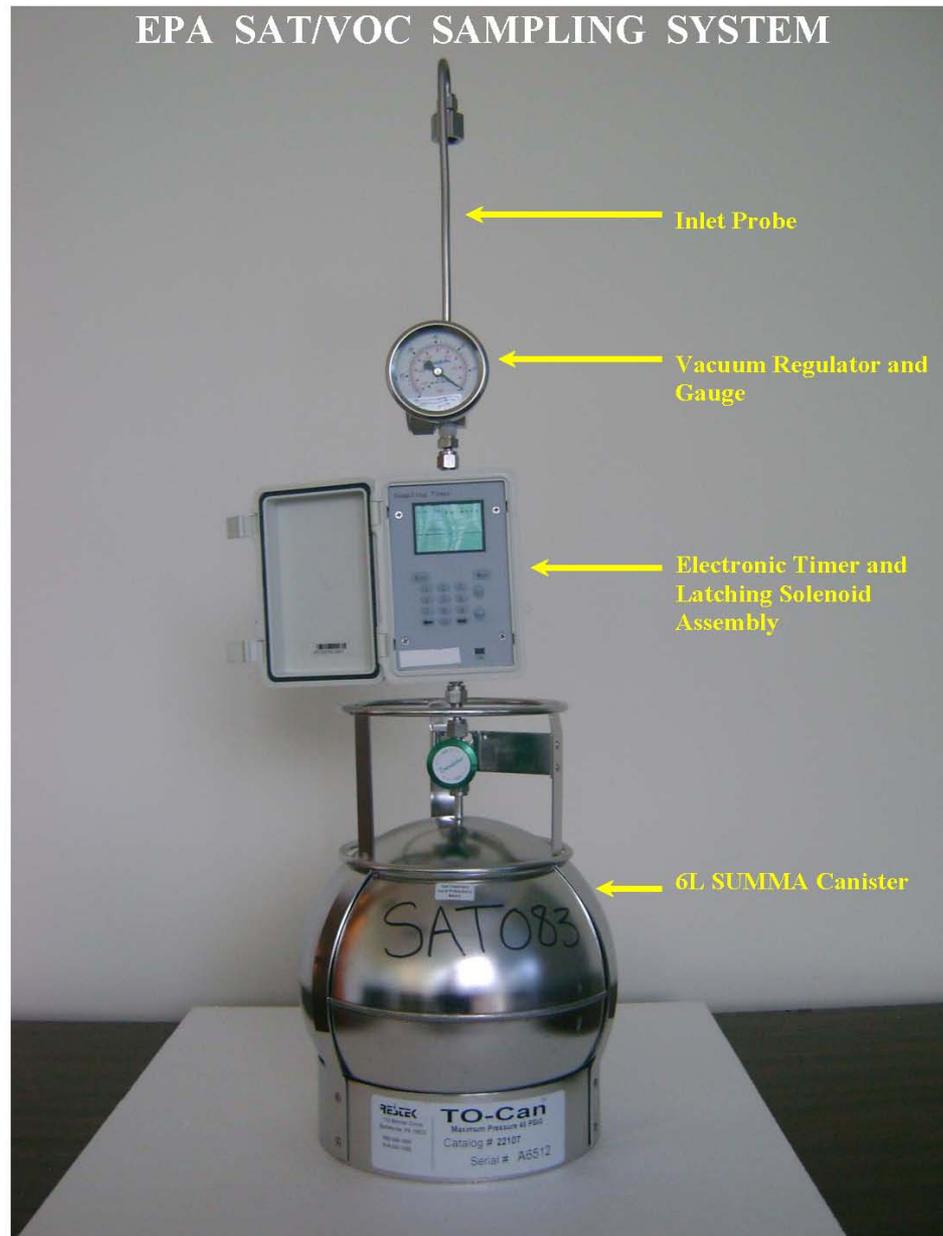
Other EPA National Monitoring Program (NMP) Samples

- Samples from other NMP efforts (UATMP, NATTS, and CSATAM) and State and local agency programs were assessed.
- Problem isolated to EPA SAT program samples only.

Common Element

- What field element was common to all sites performing VOC monitoring for the SAT program?
- VOC collection system determined to be the one common field element.

EPA SAT VOC Sampling System



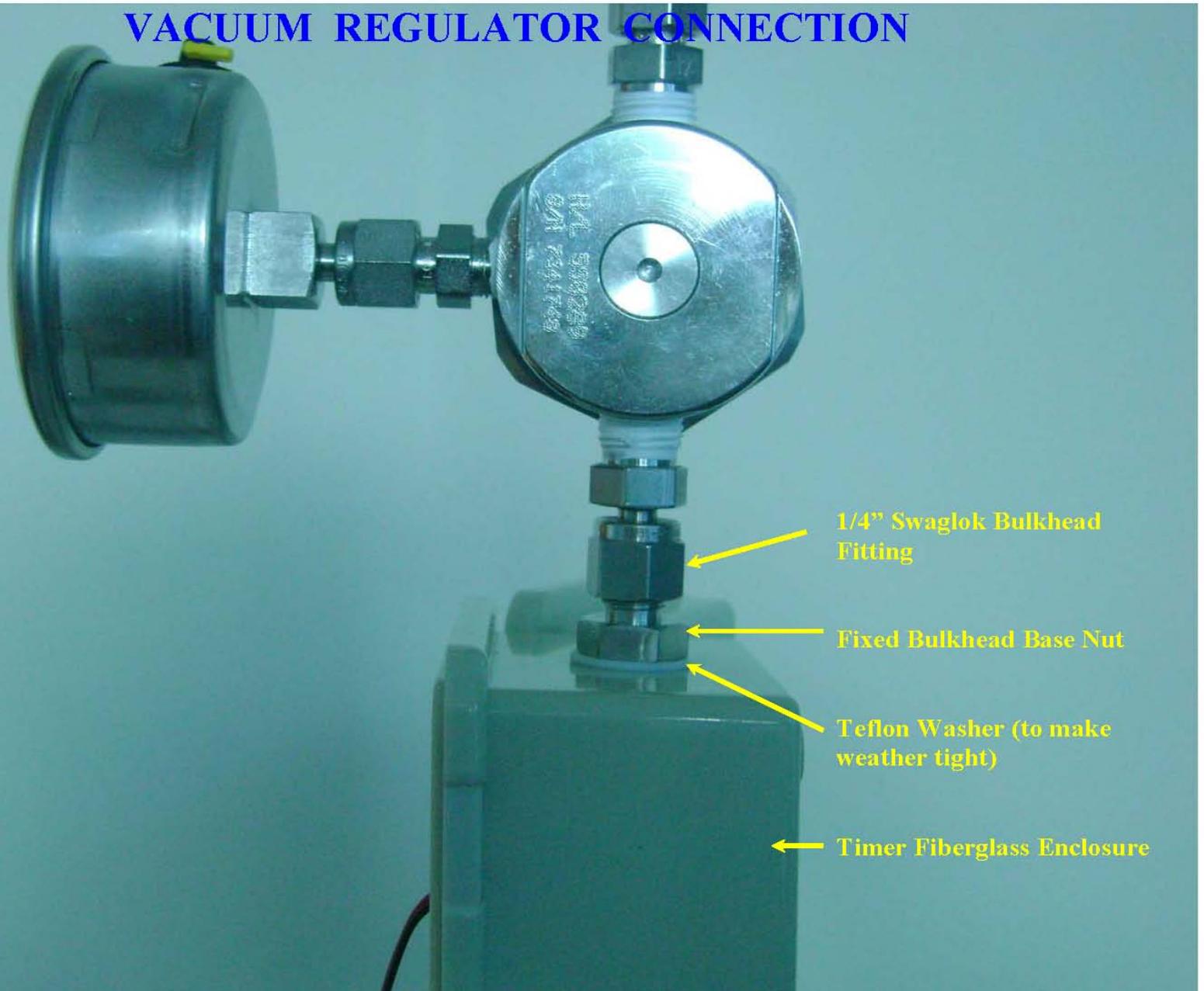
Problem Identified

- Sampler from site exhibiting grouped pattern problem returned to ERG.
- System inlet leak discovered.

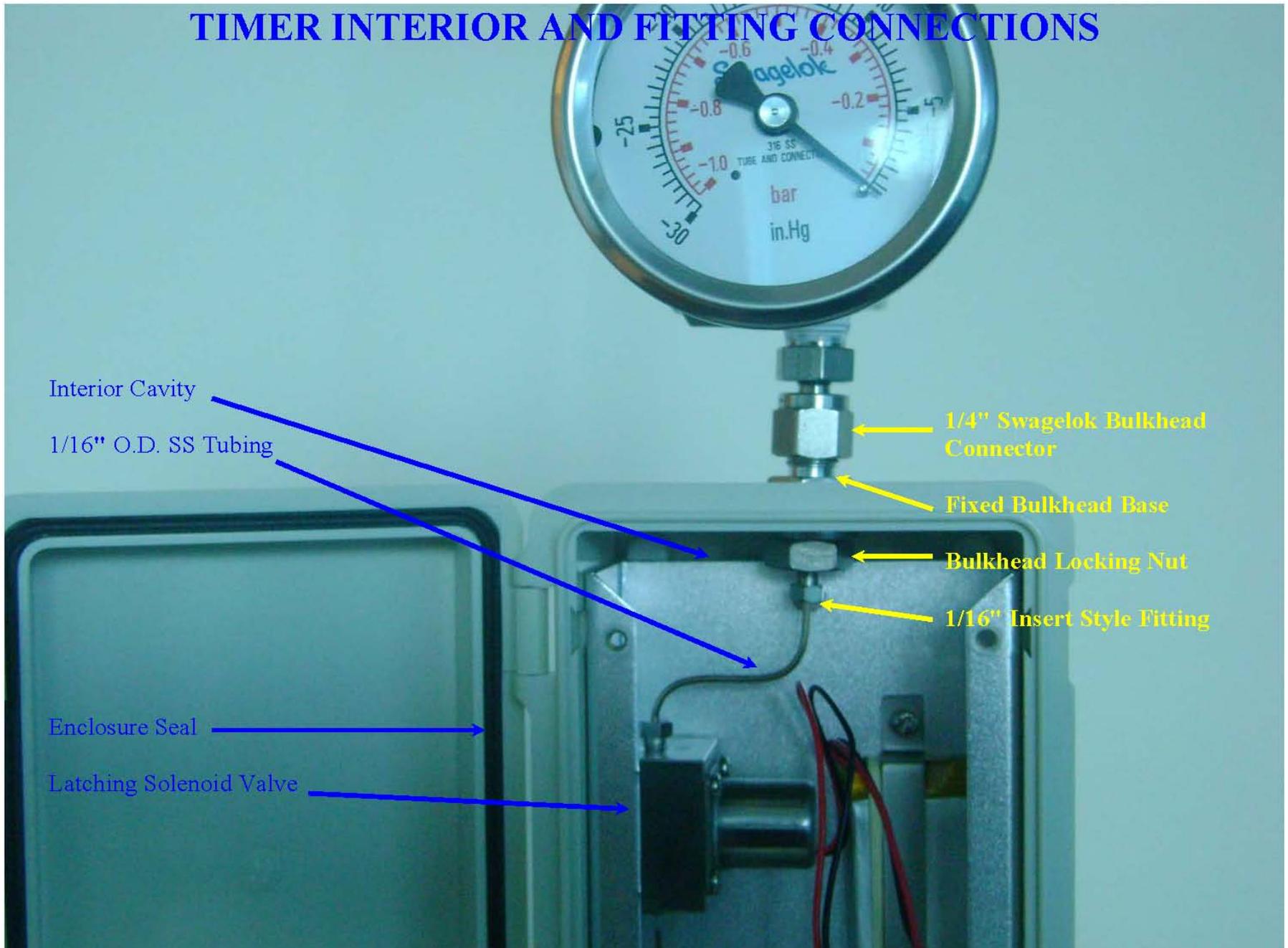
SAT VOC System Inlet Leak

- Inlet bulkhead fitting returned from site displaying grouped pattern found to be exceedingly loose.
- Leak at point where 1/6" SS tubing connects to single ferrule insert style fitting at inlet bulkhead.
- Leak located inside of the fiberglass timer enclosure.

VACUUM REGULATOR CONNECTION



TIMER INTERIOR AND FITTING CONNECTIONS



Interior Cavity

1/16" O.D. SS Tubing

Enclosure Seal

Latching Solenoid Valve

1/4" Swagelok Bulkhead Connector

Fixed Bulkhead Base

Bulkhead Locking Nut

1/16" Insert Style Fitting

Sampler Challenged “As Is”

- Humidified zero air collected through sampler for 24 hours (temp. = ~76 °F).
- Grouped compounds identified in elevated concentrations.

Sampler Name	SACA SCH
Timer#	SAT/VOC-31
Sample Date	10/11/2009
Analysis Date	10/12/2009
Canister #	SAT113
Sample Description	"As Is" Sample

ANALYTE	RESULT	UNITS
1,1,2-Trichloroethane	ND	ppbv
1,2,4-Trimethylbenzene	0.03	ppbv
1,2-Dichloroethane	ND	ppbv
1,2-Dichloropropane	ND	ppbv
1,3,5-Trimethylbenzene	ND	ppbv
1,3-Butadiene	ND	ppbv
Acetonitrile	0.13	ppbv
Acetylene	0.09	ppbv
Acrolein	0.12	ppbv
Acrylonitrile	1.63	ppbv
Benzene	0.06	ppbv
Bromomethane	ND	ppbv
Carbon Disulfide	ND	ppbv
Carbon Tetrachloride	0.02	ppbv
Chlorobenzene	ND	ppbv
Chloroethane	ND	ppbv
Chloroform	ND	ppbv
Chloromethane	0.26	ppbv
Dichlorodifluoromethane	0.08	ppbv
Dichloromethane	8.51	ppbv
Dichlorotetrafluoroethane	ND	ppbv
Ethylbenzene	0.34	ppbv
m,p-Xylene	0.26	ppbv
Methyl Ethyl Ketone	0.13	ppbv
Methyl Isobutyl Ketone	ND	ppbv
n-Octane	ND	ppbv
o-Xylene	0.12	ppbv
Propylene	0.11	ppbv
Styrene	0.68	ppbv
Tetrachloroethylene	ND	ppbv
Toluene	3.81	ppbv
Trichloroethylene	ND	ppbv
Trichlorofluoromethane	0.04	ppbv
Trichlorotrifluoroethane	ND	ppbv

Repaired Sampler Challenged

- Humidified zero air collected through sampler for 24 hours (temp. = ~76 °F).
- Grouped compounds concentrations consistent with typical zero air sample results.

Sampler Name	SACA SCH
Timer#	SAT/VOC-31
Sample Date	10/13/2009
Analysis Date	10/14/2009
Canister #	5077
Sample Description	Repaired Sample

ANALYTE	RESULT	UNITS
1,1,2-Trichloroethane	ND	ppbv
1,2,4-Trimethylbenzene	ND	ppbv
1,2-Dichloroethane	ND	ppbv
1,2-Dichloropropane	ND	ppbv
1,3,5-Trimethylbenzene	ND	ppbv
1,3-Butadiene	ND	ppbv
Acetonitrile	ND	ppbv
Acetylene	0.33	ppbv
Acrolein	ND	ppbv
Acrylonitrile	ND	ppbv
Benzene	0.12	ppbv
Bromomethane	ND	ppbv
Carbon Disulfide	ND	ppbv
Carbon Tetrachloride	0.11	ppbv
Chlorobenzene	ND	ppbv
Chloroethane	ND	ppbv
Chloroform	0.02	ppbv
Chloromethane	0.59	ppbv
Dichlorodifluoromethane	ND	ppbv
Dichloromethane	0.05	ppbv
Dichlorotetrafluoroethane	0.59	ppbv
Ethylbenzene	0.02	ppbv
m,p-Xylene	0.05	ppbv
Methyl Ethyl Ketone	0.12	ppbv
Methyl Isobutyl Ketone	ND	ppbv
n-Octane	ND	ppbv
o-Xylene	0.02	ppbv
Propylene	0.13	ppbv
Styrene	ND	ppbv
Tetrachloroethylene	ND	ppbv
Toluene	0.13	ppbv
Trichloroethylene	ND	ppbv
Trichlorofluoromethane	0.27	ppbv
Trichlorotrifluoroethane	0.09	ppbv

Simulated Leak Challenge

- Bulkhead fitting manually rotated to cause leak at the insert style fitting.
- Humidified zero air collected through sampler for 24 hours (temp. = ~76 °F).

Sampler Name SAPA SCH
 Timer# SAT/VOC-20
 Sample Date 10/14/2009
 Analysis Date 10/15/2009
 Canister # SAT030
 Simulated Leak through
 Timer

ANALYTE	RESULT	UNITS
1,1,2-Trichloroethane	ND	ppbv
1,2,4-Trimethylbenzene	0.20	ppbv
1,2-Dichloroethane	ND	ppbv
1,2-Dichloropropane	ND	ppbv
1,3,5-Trimethylbenzene	0.08	ppbv
1,3-Butadiene	ND	ppbv
Acetonitrile	1.14	ppbv
Acetylene	0.38	ppbv
Acrolein	0.48	ppbv
Acrylonitrile	21.15	ppbv
Benzene	0.19	ppbv
Bromomethane	ND	ppbv
Carbon Disulfide	ND	ppbv
Carbon Tetrachloride	0.04	ppbv
Chlorobenzene	0.06	ppbv
Chloroethane	ND	ppbv
Chloroform	0.04	ppbv
Chloromethane	0.69	ppbv
Dichlorodifluoromethane	0.24	ppbv
Dichloromethane	45.79	ppbv
Dichlorotetrafluoroethane	ND	ppbv
Ethylbenzene	3.56	ppbv
m,p-Xylene	2.49	ppbv
Methyl Ethyl Ketone	0.41	ppbv
Methyl Isobutyl Ketone	ND	ppbv
n-Octane	0.57	ppbv
o-Xylene	1.09	ppbv
Propylene	0.23	ppbv
Styrene	3.76	ppbv
Tetrachloroethylene	0.04	ppbv
Toluene	56.45	ppbv
Trichloroethylene	0.12	ppbv
Trichlorofluoromethane	0.12	ppbv
Trichlorotrifluoroethane	0.04	ppbv

Challenge Simulating Ambient Conditions

- Three timers (2 new, 1 refurbished) had bulkhead fitting rotated to facilitate leaks at the individual insert style fittings.
- Timer enclosures skin temperature elevated to 100 – 105 °F to simulate exposure to the summer sun.
- Humidified zero air simultaneously collected through each sampler for 24 hours.

Elevated Temperature Results

- Heat significantly elevated the observed concentrations for the grouped pollutants in 2 of 3 cases.

Sampler Name	SIM Leak #3	SIM Leak #4	SIM Leak #5	
Timer#	11841	10037	11100	
Sample Date	10/15/2009	10/15/2009	10/15/2009	
Analysis Date	10/16/2009	10/16/2009	10/16/2009	
Canister #	5077	SAT097	SAT030	
Sample Description				
		Simulated Leak through Timer		
		Shell Temp ~100 - 105°F		
ANALYTE	RESULT	RESULT	RESULT	UNITS
1,1,2-Trichloroethane	0.04	ND	ND	ppbv
1,2,4-Trimethylbenzene	0.34	ND	0.16	ppbv
1,2-Dichloroethane	2.46	ND	0.79	ppbv
1,2-Dichloropropane	ND	0.02	ND	ppbv
1,3,5-Trimethylbenzene	0.08	ND	ND	ppbv
1,3-Butadiene	0.13	ND	ND	ppbv
Acetonitrile	7.22	0.66	4.45	ppbv
Acetylene	16.02	0.52	0.21	ppbv
Acrolein	4.31	0.25	3.11	ppbv
Acrylonitrile	148.80	4.17	105.12	ppbv
Benzene	1.26	0.14	0.98	ppbv
Bromomethane	0.11	ND	0.16	ppbv
Carbon Disulfide	0.14	ND	0.15	ppbv
Carbon Tetrachloride	0.09	0.11	0.09	ppbv
Chlorobenzene	0.21	ND	ND	ppbv
Chloroethane	0.15	ND	0.20	ppbv
Chloroform	0.28	0.07	0.25	ppbv
Chloromethane	2.45	0.85	2.57	ppbv
Dichlorodifluoromethane	0.21	0.70	ND	ppbv
Dichloromethane	448.78	19.98	351.50	ppbv
Dichlorotetrafluoroethane	ND	ND	ND	ppbv
Ethylbenzene	43.15	0.57	2.46	ppbv
m,p-Xylene	33.49	0.49	1.20	ppbv
Methyl Ethyl Ketone	1.43	0.33	0.85	ppbv
Methyl Isobutyl Ketone	0.14	ND	ND	ppbv
n-Octane	ND	ND	ND	ppbv
o-Xylene	10.13	0.16	0.61	ppbv
Propylene	0.55	0.51	0.75	ppbv
Styrene	77.35	0.62	28.51	ppbv
Tetrachloroethylene	0.17	ND	0.29	ppbv
Toluene	91.81	1.18	24.62	ppbv
Trichloroethylene	1.54	0.21	1.56	ppbv
Trichlorofluoromethane	0.11	0.27	0.12	ppbv
Trichlorotrifluoroethane	0.04	0.12	0.08	ppbv

Conclusions

- Leak at point where 1/6" SS tubing connects to single ferrule insert style fitting at inlet bulkhead located inside fiberglass enclosure.
- Potential cause of leak is rotation of the inlet bulkhead fitting.
- Grouped pattern consistent with formulation associated with fiberglass.
- It is probable that temperature and the application of high vacuum exacerbate the contamination.

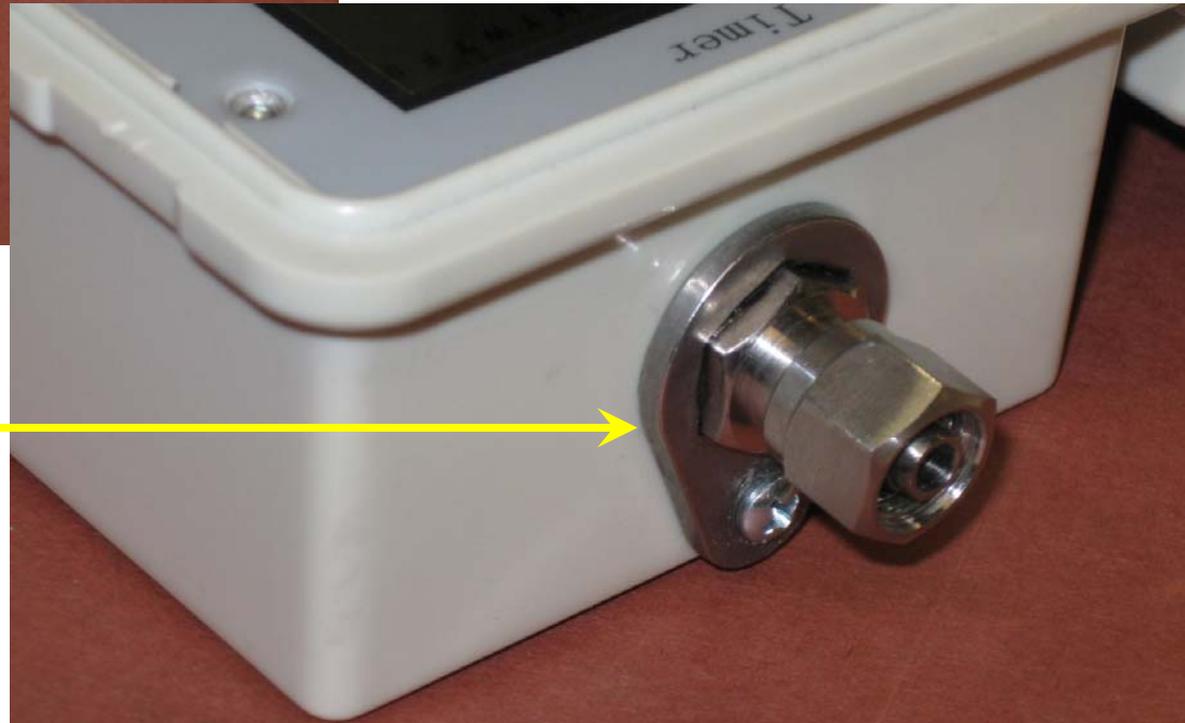
Possible Resolutions 1

- Instruct that a 5/8" wrench be used to secure the fixed nut of the bulkhead fitting in place so that it cannot rotate when attaching the vacuum regulator to the inlet or canister to the outlet.
- Add bulkhead fixed nut stabilizer.



Without
bulkhead nut
stabilizer

With
bulkhead nut
stabilizer



Possible Resolutions 2

- Change bulkhead fittings to a style that utilizes a 1/16" double ferrule (Swagelok[®]) fitting to connect to the 1/16" SS tubing, in place of the insert style fitting.
- Change to a timer enclosure made of metal instead of fiberglass.

Status

- EPA guidance to SAT sites still monitoring:
 - To eliminate contamination potential, remove timer from the sampling train and manually open and close the valve
 - For those sites where not feasible (e.g., long distance between duty station and monitoring site):
 - Take precaution to ensure that neither of the bulkhead fittings become loose
 - *Note: data resulting from samples collected with the timer may be invalidated if suspected of being contaminated*
- EPA working to determine what data invalidation protocol / criteria may be appropriate