

A Micro-GC Based Chemical Analysis System

DEFIANT TECHNOLOGIES BOOTH #2518
PATRICK LEWIS, PRLEWIS@DEFIANT-TECH.COM, 505-307-3576
DOUG ADKINS, PHD, GEORGE DULLECK, GARY FUEHRER, ROBERT
SANCHEZ, LEE TU, JACY GANSZ



www.defiant-tech.com

FROG-4000™

A Microsystem for VOCs in Air, Water, and Soil



- 2.2 kgs (with battery)
- Battery Life approximately 6 to 8 hrs.
- Air carrier gas– no specialty gases are required
- Programmable GC temperature profile
- Upload your own calibration and operating parameters
- Quantitative results display on screen or watch chromatography real-time with a computer
- Software enables user to evaluate results
- On-board data storage of chromatograms

What Type of Sample May be Measured on the FROG-4000?



- ✓ Vapor Intrusion
- ✓ Soil Gas
- ✓ Industrial Hygiene
- ✓ Indoor Air Quality
- ✓ Acetaldehyde and BTEX in Carbonation



- ✓ Groundwater Monitoring
- ✓ Fuel Dilution
- ✓ Site Investigation
- ✓ Remediation processes
- ✓ Oil Spill in Sea water

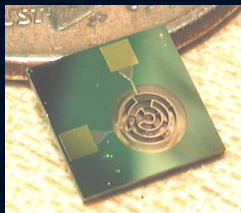


- ✓ Minimize Dig and Haul Operations
- ✓ Land-farming
- ✓ Solvents in Pharmaceutical Products
- ✓ Crude Oil through Methanol Extraction

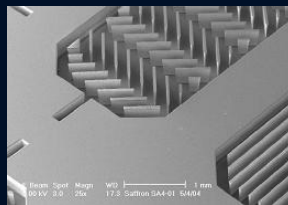
The Tools Defiant Uses to Make High Confidence Chemical Analysis Systems

www.defiant-tech.com

Collection



3d
Preconcentrator:
high capacity
chemical
collection.

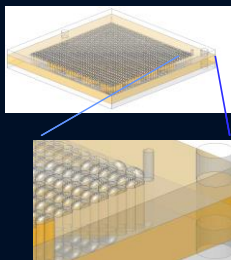


Tortuous Path
Preconcentrator:
Collect and retain
volatile chemicals



Smart
Preconcentrator:
redefining dynamic
range. The first real
step to intelligent
systems

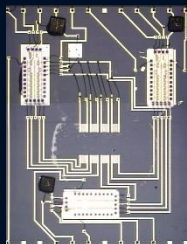
Separation



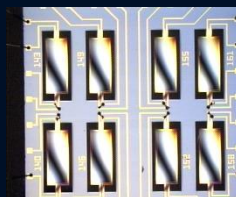
Circular x-section
column. High
resolution
chemical
separations

By combining the components on this page, Defiant can design chemical analysis systems for a variety of applications. Of course, each component has a number of selective films that can be applied to the surface. A proprietary manifold allows us to connect these devices like Legos™.

Detection



SAW Array with
integrated
electronics for DC-
in;DC-out
operation.



Pivot
Plate
resonator

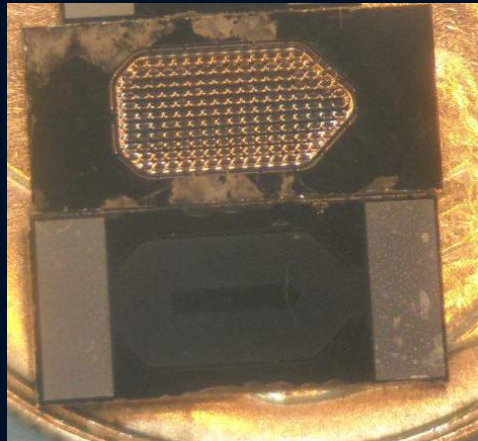


Micro Nitrogen
Phosphorus detector
– element specific
chemical detection

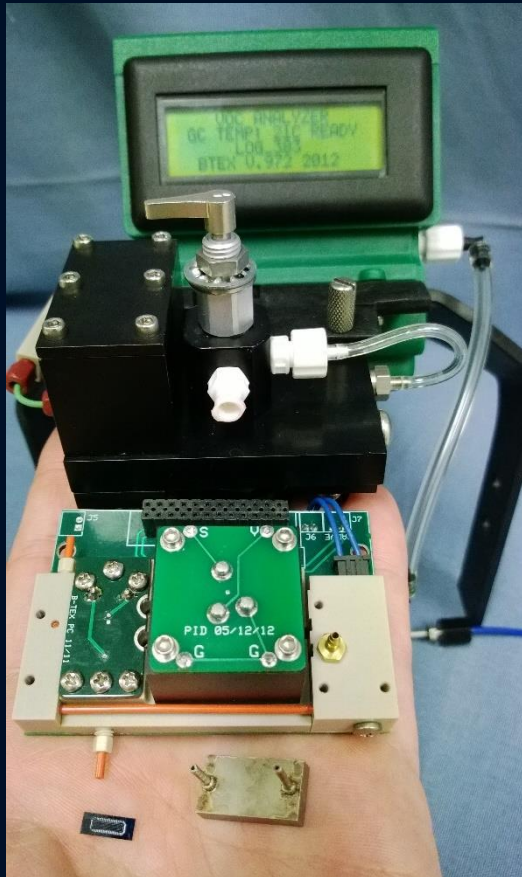
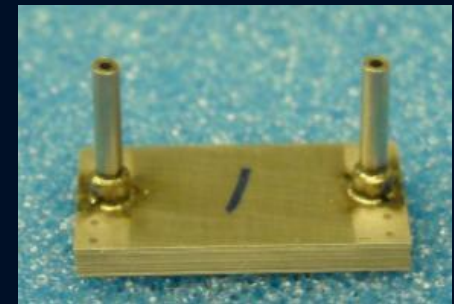
How Defiant Makes it All Possible

MEMS (Micro Electro Mechanical Systems) Processing enables batch fabrication of micro components in silicon, steel, and other materials. The result is very compact, low power devices that when married with the right materials can perform the same function as their bench top analogs.

**MEMS
Preconcentrator**

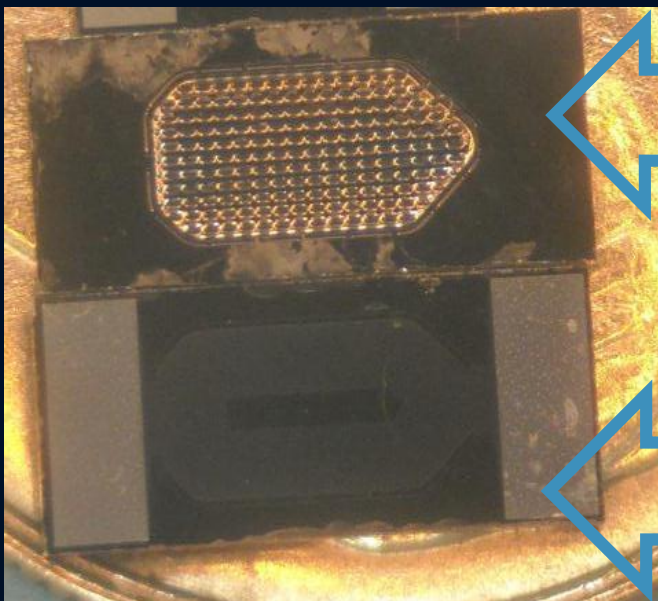


Micro GC Column



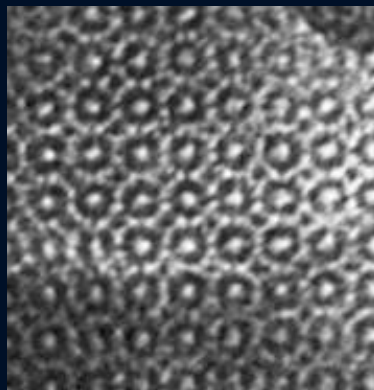
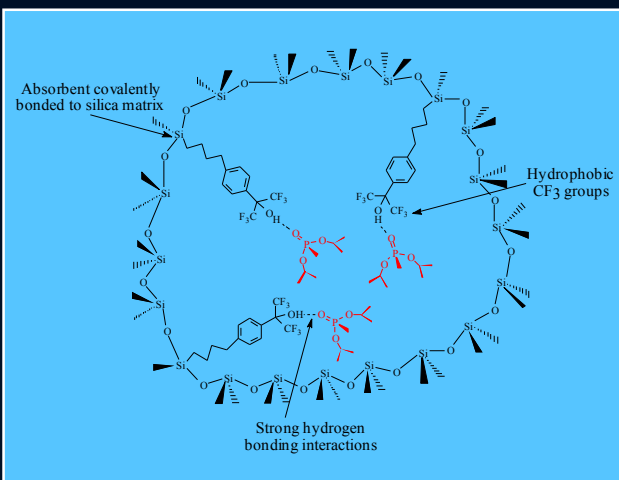
**Photoionization
Detector**



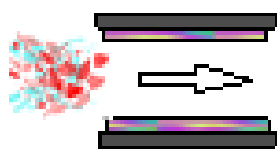


The features on this side of the die are coated with a sol gel material. The plurality of fins and mass of sol gel applied help to collect and retain volatile organic compounds.

This side of the die shows two silvery pads and an oval shaped resistive heater. A small DC voltage is applied to this heater to thermally desorb the chemicals collected by the opposite side.



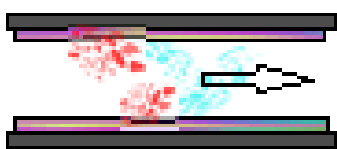
Defiant's Micro GC Column



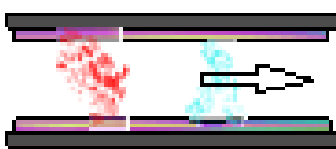
Analytes
Injected



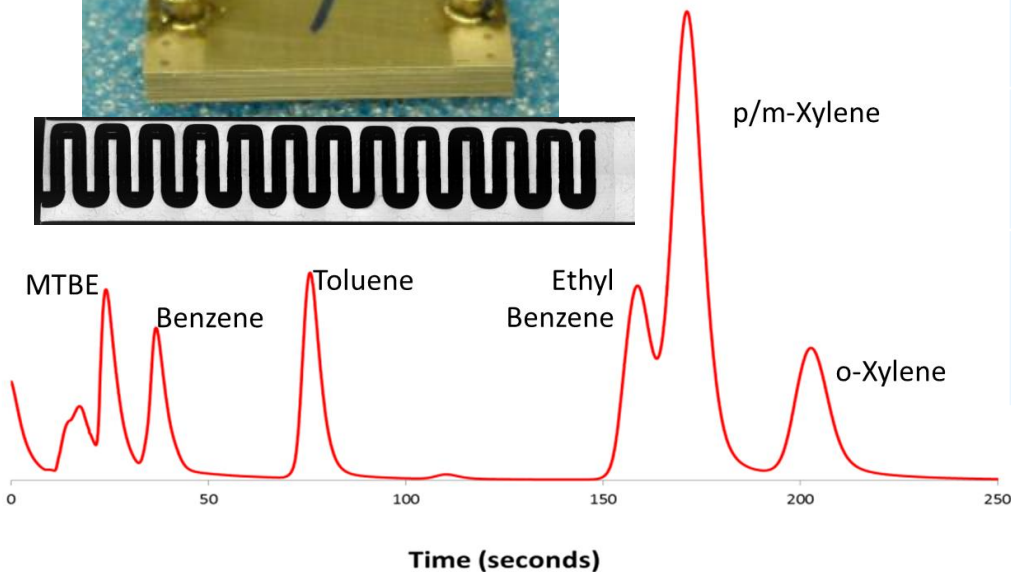
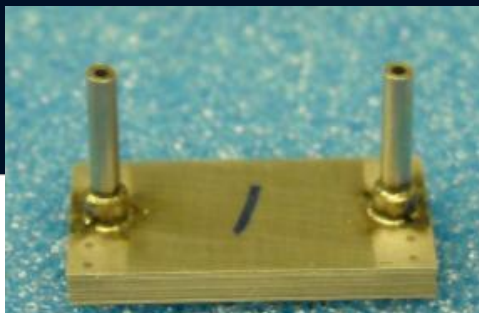
Solubility in Phase
Determines
Retention



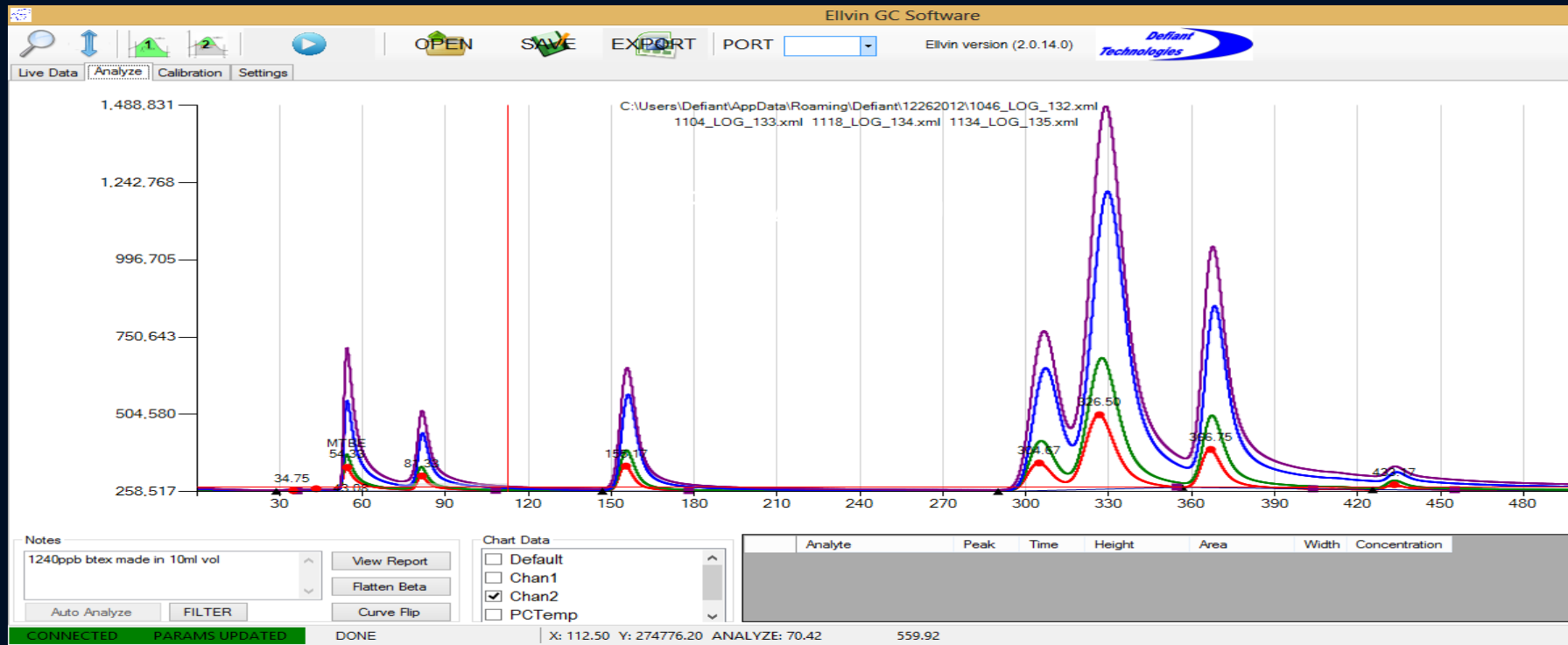
Analytes Separate based
on Boiling Point



Analytes Exit
Column



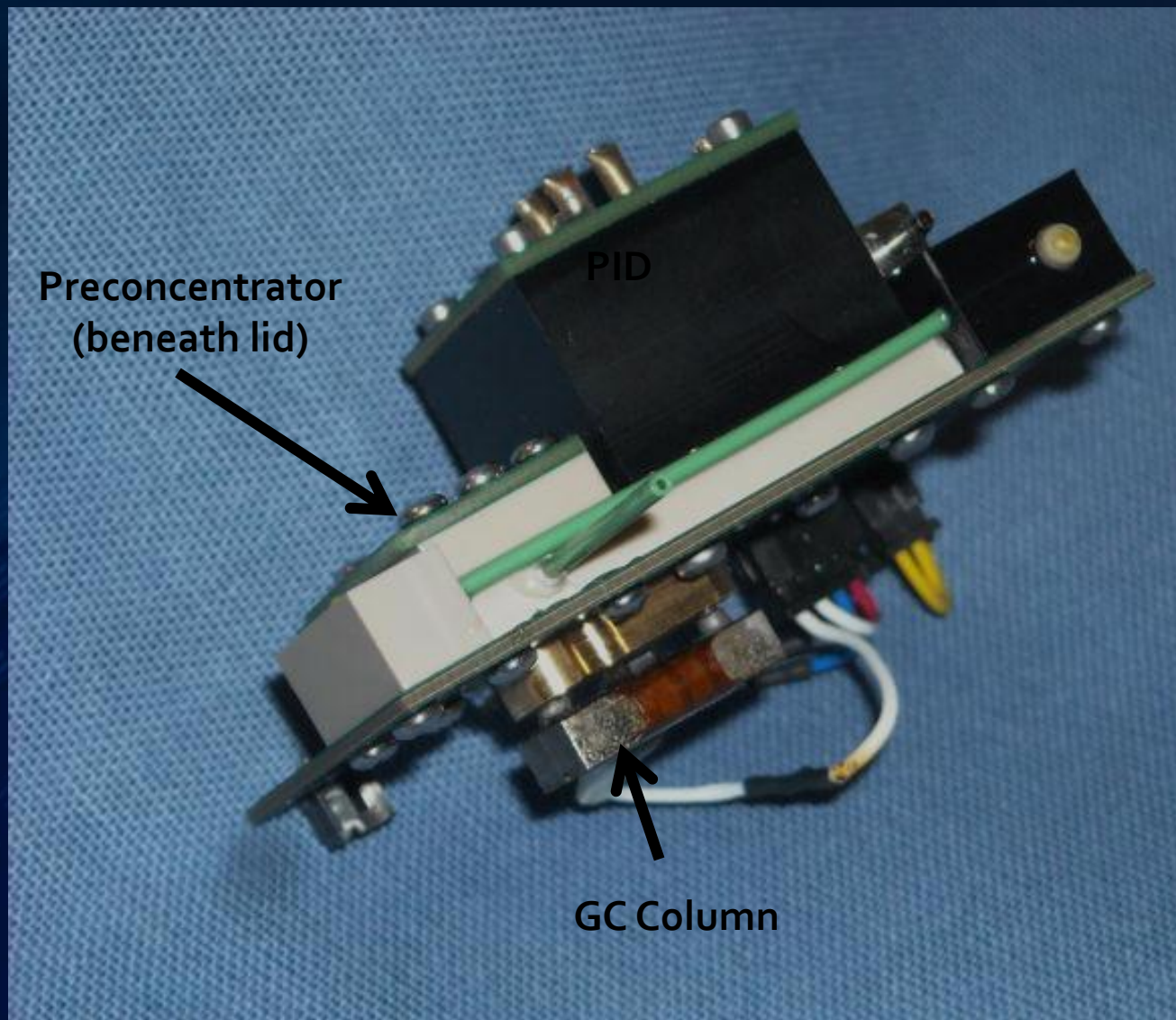
Chemical Detection Range	Vinyl Chloride (MW 62.5)	2-Methyl Naphthalene (MW 142.2)
Boiling Point at 1 atm	-13.3 °C	244.7 °C
Vapor Pressure at 25 °C	2980 mmHg	0.067 mmHg



Retention Time Statistics

	Benzene	Toluene	Ethylbenzene	p/m-Xylene	o-Xylene
Average (sec)	81.67	156.03	307.33	328.21	368.32
Standard Deviation (sec)	0.20	0.36	1.50	1.41	0.84
% RSD	0.25%	0.23%	0.49%	0.43%	0.23%

FROG-4000™ Gas Module



The Gas Module, as we call it, is the electronic and fluidic manifold that integrates all of the MEMS components that power the FROG. A micro-fabricated preconcentrator and micro-fabricated GC column complement a miniature photoionization detector (PID).

Water Analysis: Results from Well SP-1 Tacoma Washington

Sample ID: SP-1	FROG-4000™ (µg/L)	GC/MS (µg/L)	% Rec	RPD
VC	11	9.1	121%	18.9%
t-1,2-DCE	56	57	98%	1.8%
c-1,2-DCE	93	100	93%	7.3%
TCE	160	170	94%	6.1%
PCE	6.0	5.6	107%	6.9%



Real World Applications & Solutions

Elk River in West Virginia

Freedom Industries



Chemical spill affects much of Charleston On Thursday a chemical spill caused much of Charleston to shut down. The chemical, called 4-methylcyclohexane methanol, is a foaming agent used in the coal preparation process. It is harmful if swallowed and could be harmful if inhaled.

A 40,000 gallon tank leaks and overruns a containment area.

An estimated 5,000 gallons escapes. Some of which flows into the Elk River.

Afterwards a licorice-like smell envelops parts of the city.

Freedom Industries

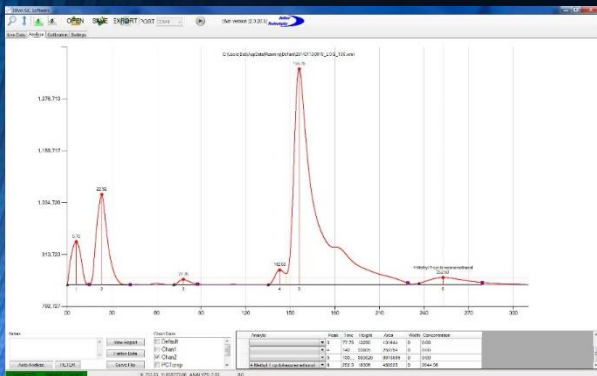
Leaking tank

Ohio Pa. W. Va. Va. Spill

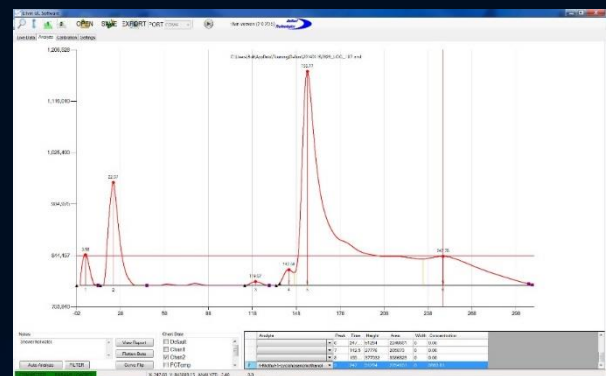
Counties affected

SOURCES: ESRI, Fisher Scientific, American Association of Poison Control Centers AP

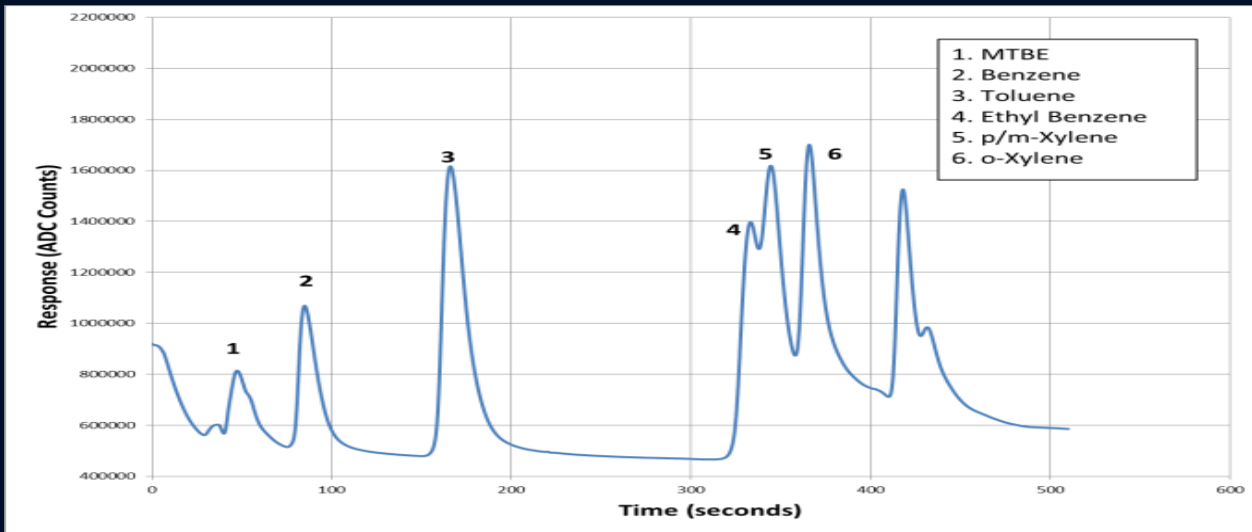
American Water Company



Analysis in < 8 minutes on site
vs.
24 hours by 5 independent offsite labs

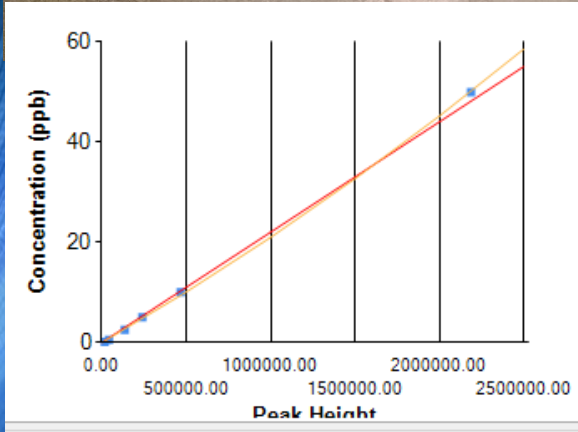
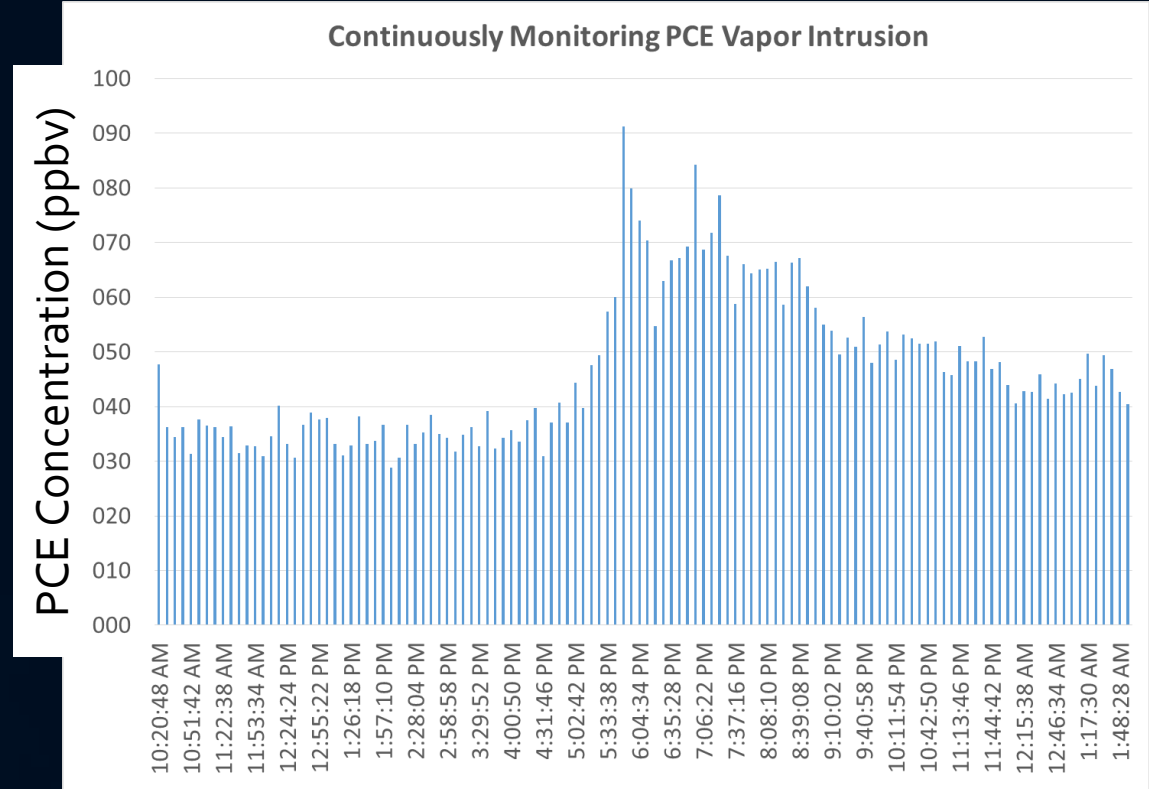
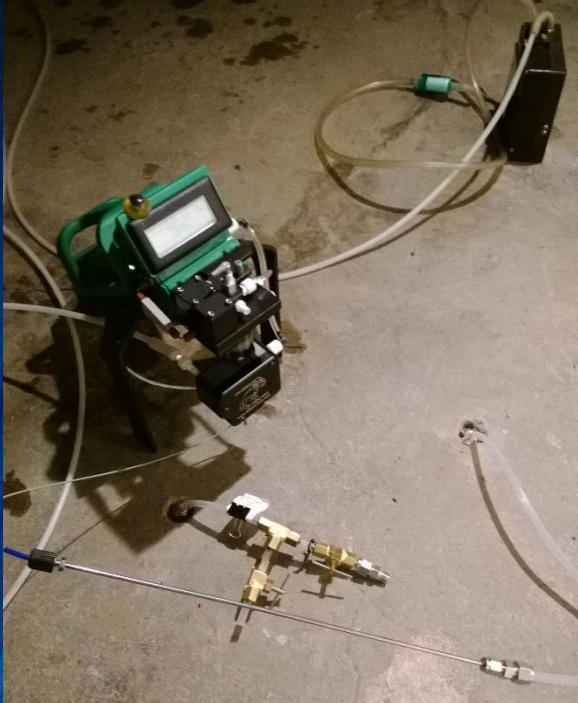


Analysis of Soils: Certified Reference Material by Methanol Extraction



Analyte	FROG-4000™ (µg/kg)	Certified Concentration (µg/kg)	% Rec
MTBE	8900	9450	94%
Benzene	20400	20200	101%
Toluene	49300	51400	96%
Ethylbenzene	43700	40100	109%
p/m-Xylene	28900	30400	95%
o-Xylene	36300	33600	108%

Air Sampling with the FROG-4000 Vapor Intrusion



Concentration	Fit
0.1	C
0.5	C
2.5	C
5	C
10	C
50	C

Time of Day

Height	Equation
EQ	$Y=1.6417E-012X^2+ 1.9330E-005X + 0.0000E+000$
QUAD R ² :	0.99965
EQ	$Y=2.2029E-005X + 0.0000E+000$
LINE R ² :	0.99786

Ellvin GC Software

Ellvin version (2.1.9.234)

Defiant Technologies

Live Data Analyze Calibration Settings

PC FIRE MTBE Benzene Ethylbenzene o-Xylene ANALYZE

Identify Analyte

[VP: 95.7128.1]

Chemical Name	CAS	VP
Benzene	71-43-2	94.8
Ethyl Acetate	141-78-6	93.2
n-Butylamine	109-73-9	92.9
2-Butanone (MEK)	78-93-3	90.6
Vinyl Acetate	108-05-4	90.16
Bromoacetone	598-31-2	90
Cyclohexene	110-83-8	89

Name: Methyl Acrylate
 CAS: 96-33-3
 Vapor Pressure: 86.56

OK Cancel

Notes
 Ta=300,Tb=60,Tc=30,Ct=40,Ht=100,COLL ECT=10,CLEAN=4,PRESETTLE=4,SETTL E=2,FIRE=6

View Report
 Flatten Beta
 Curve Flip

Auto Analyze FILTER

Chart Data
 Hide Start of Run
 Chan1
 Chan2
 PCTemp
 GCTemp
 GCProfile

Integration Results

Analyte	Peak	Time	Height	Area	Concentration
	3	46.08	341134	2893523	
	4	67.08	2046757	16603320	
	5	81.17	1167337	11960018	
	6	117.25	2639591	30916410	

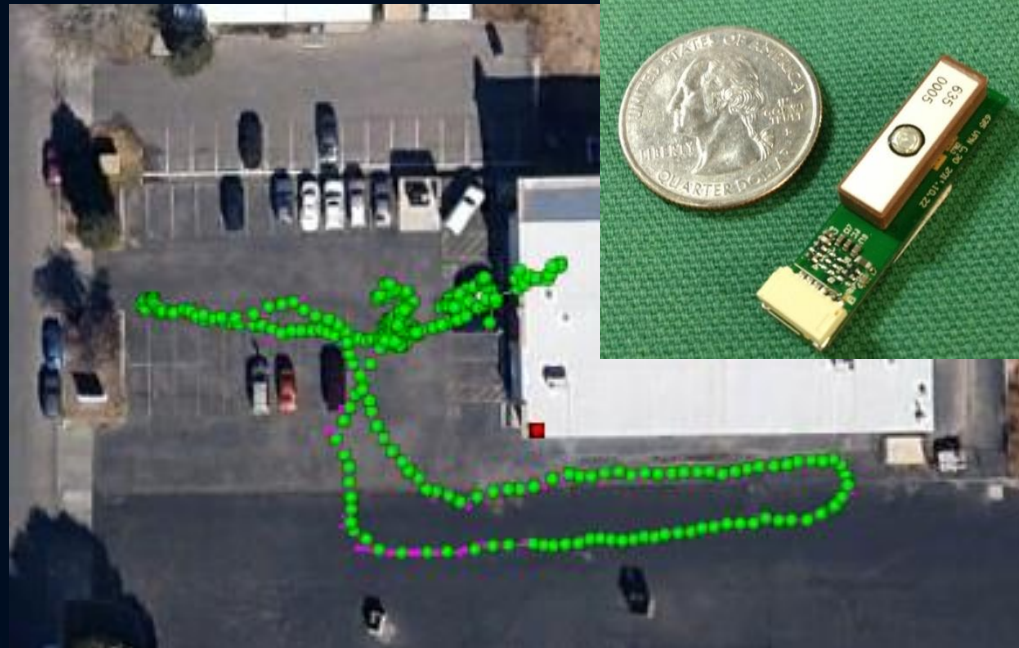
DISCONNECTED NO PARAMS ... X:0.0 Y:0.0 ANALYZE:0.0 0.0

4:43 PM 2/27/2014

Optional Features for the FROG-4000™

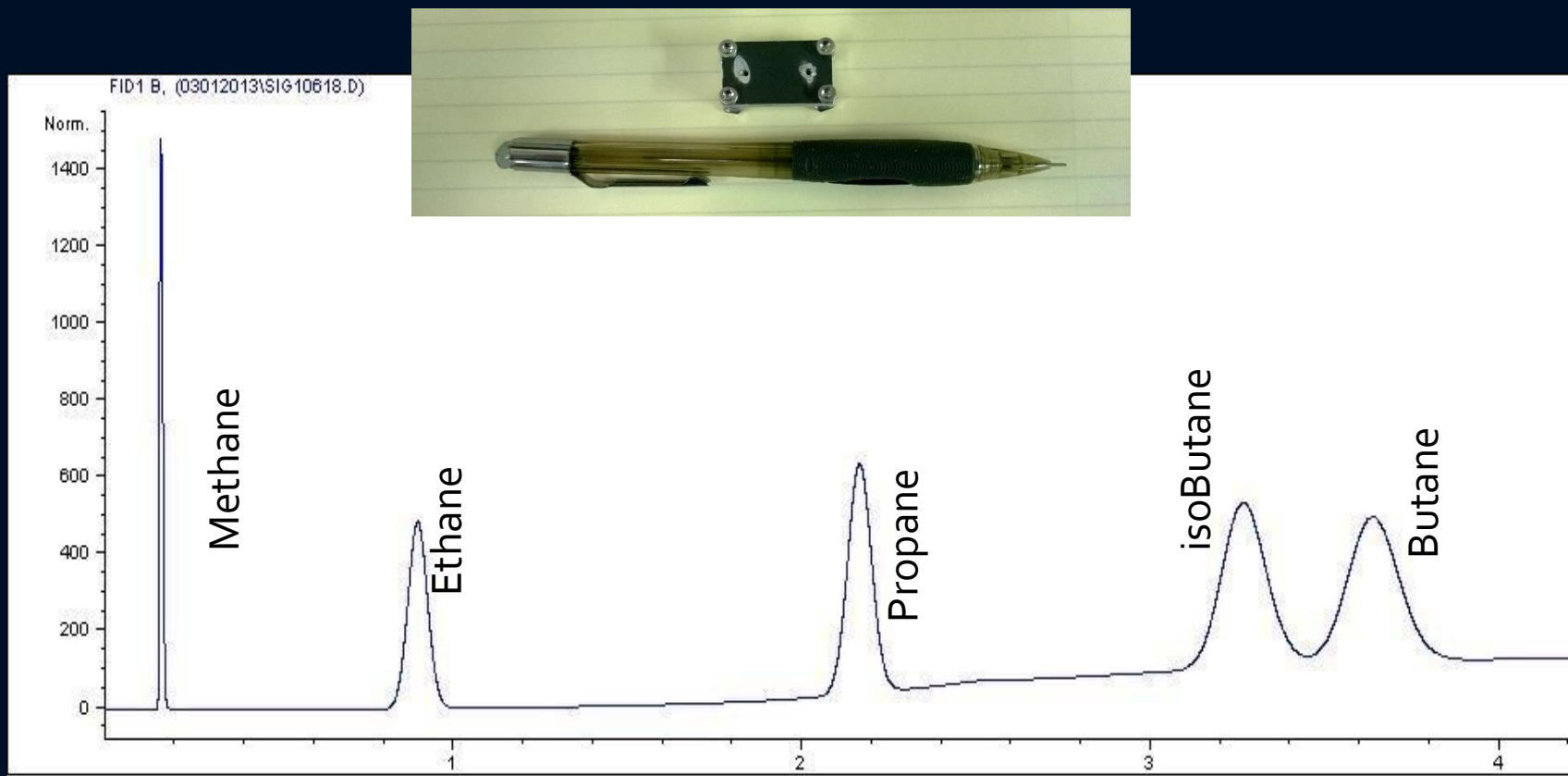


Bottle Heater for water and
soil analysis



Log GPS Coordinates for Every Analysis

Below we show a device that has the same amount of packing and the same length of channel but in a smaller footprint. This design enables easier integration because it is compact and simplified heating since a Kapton heater may be applied to the surface for direct heating.



Questions



VISIT DEFIANT TECHNOLOGIES AT BOOTH 2518