



Homeland Security: Working Together

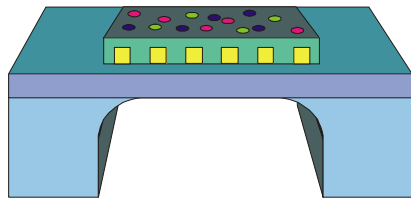
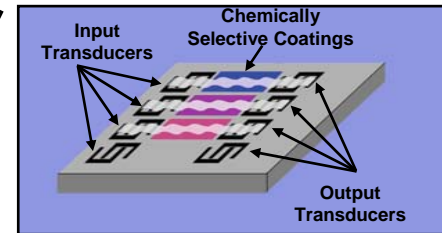
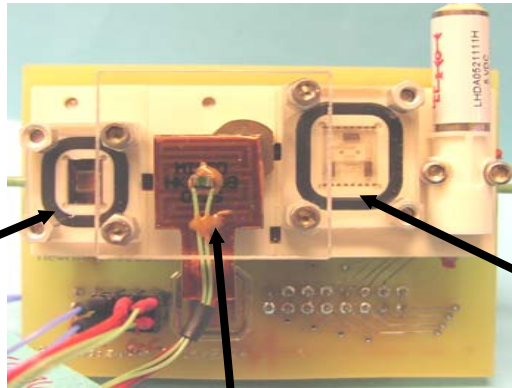
Research & Development Partnerships in Homeland Security

April 27-28, 2005

Boston, MA

Field Portable Analyzer™

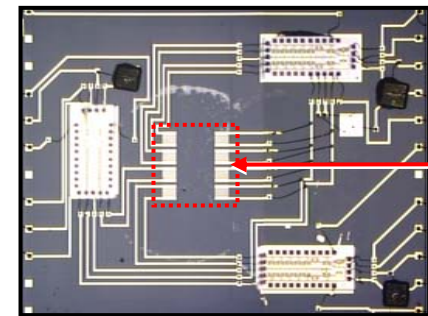
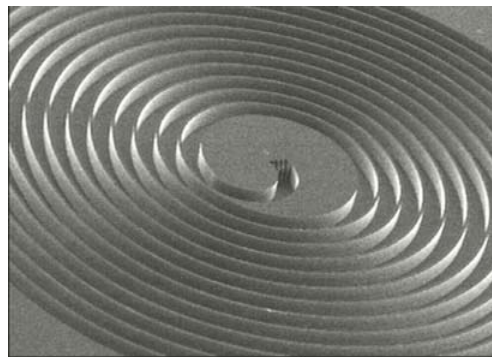
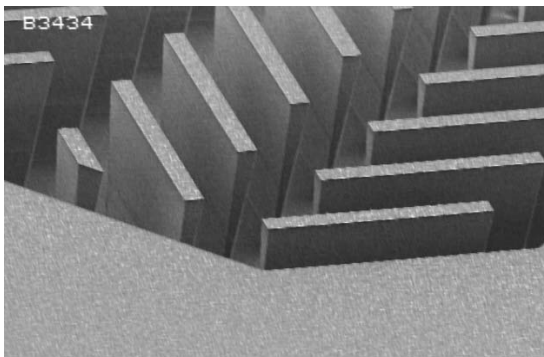
A hand-held chemical analysis system that uses three integrated modular components



Preconcentrator accumulates analytes of interest

Gas Chromatograph separates analytes in time

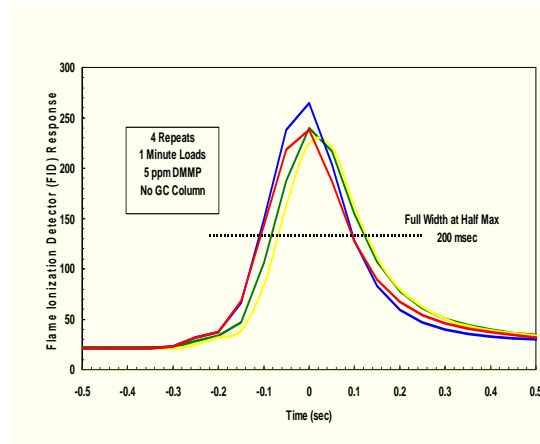
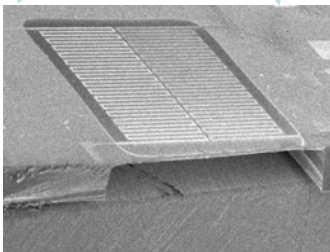
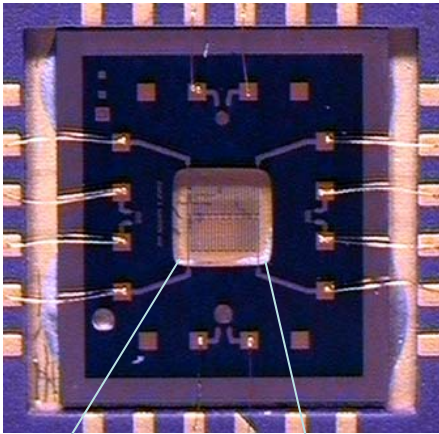
Acoustic Sensors provide sensitive detection



SAW Array

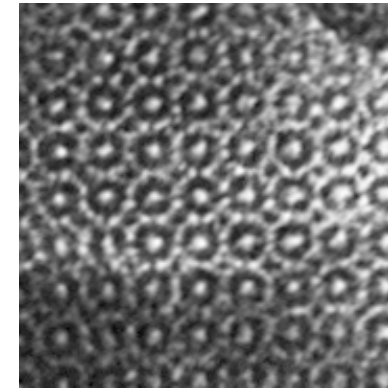
Preconcentrator

- Accumulates analytes from low concn. inlet
- Thermally desorbs a narrow, higher concentration pulse
- Serves as injector to GC column (no valve requ.)

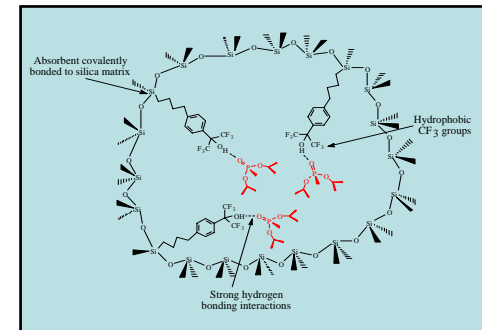


Rapid Thermal Desorption from Micromachined Preconcentrator

Sol-gels provide thin film adsorbents with high uptake and chemical selectivity

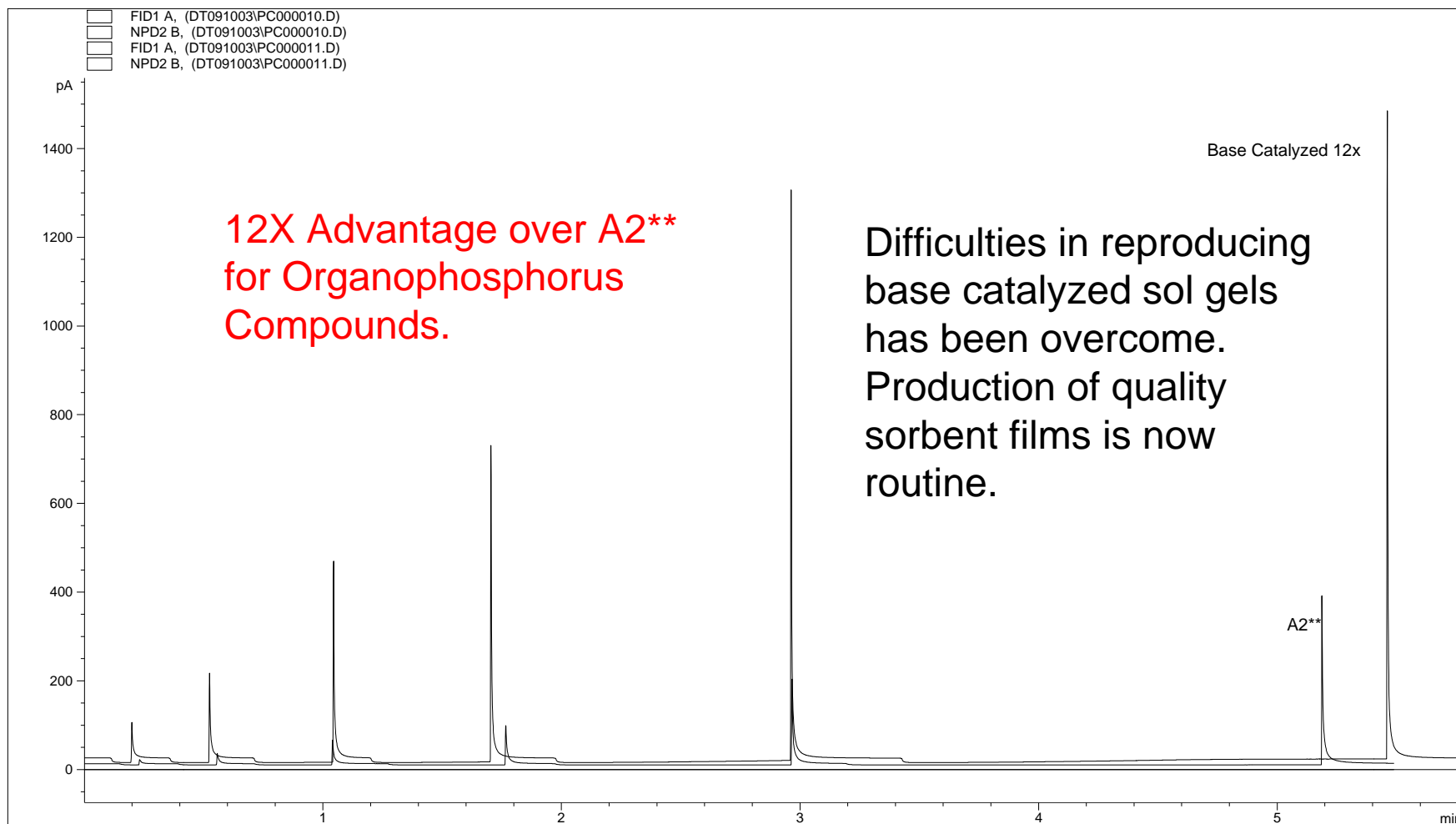


Tailored Porosity

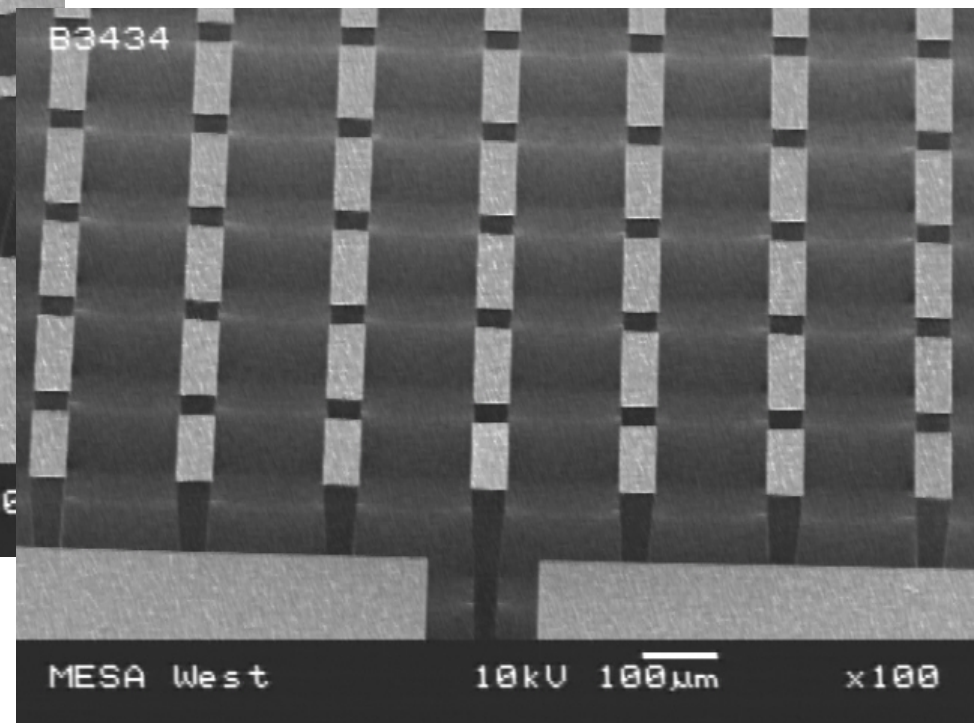
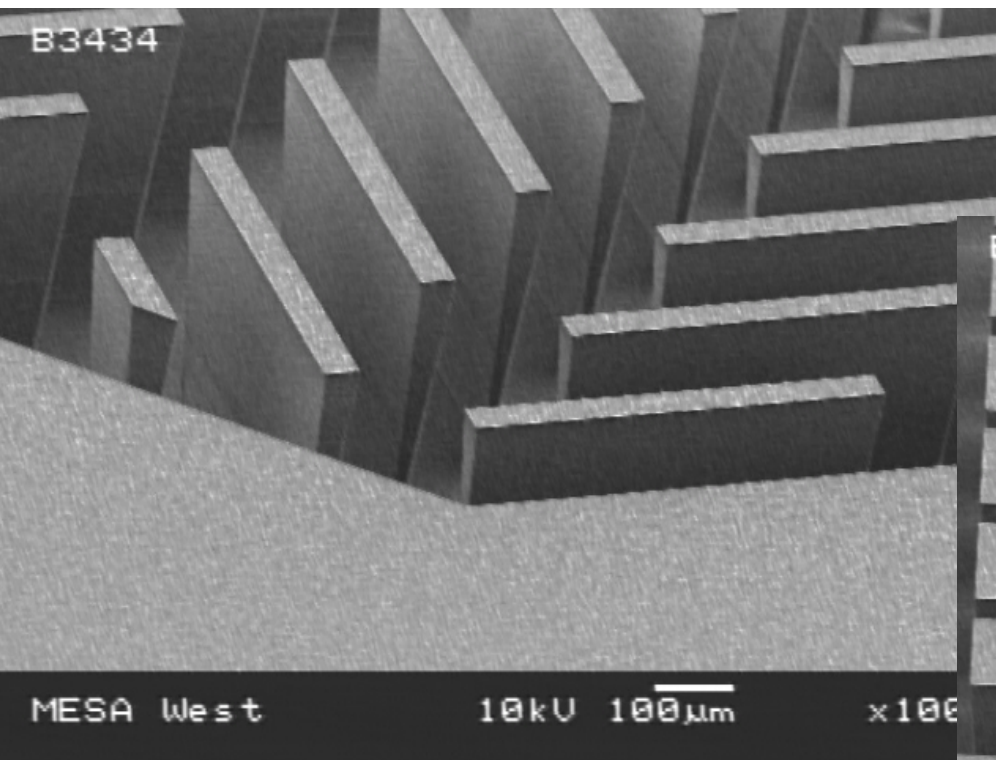


Tailored Surface Chemistry

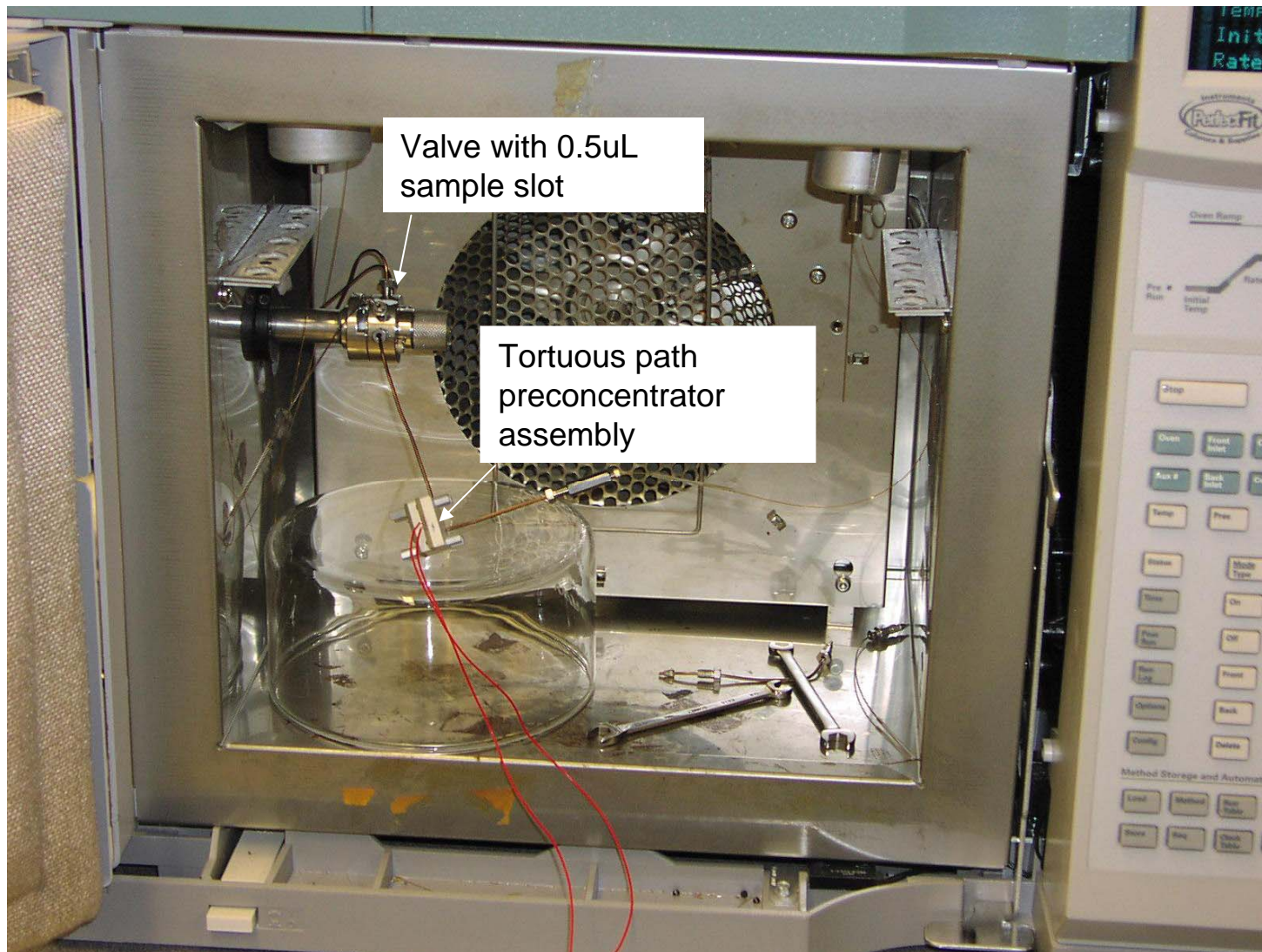
Comparison: Base Catalyzed vs A2** Increasing Collection Times



Highly Tortuous Path Preconcentrator for Enhanced Collection of Volatile TICs



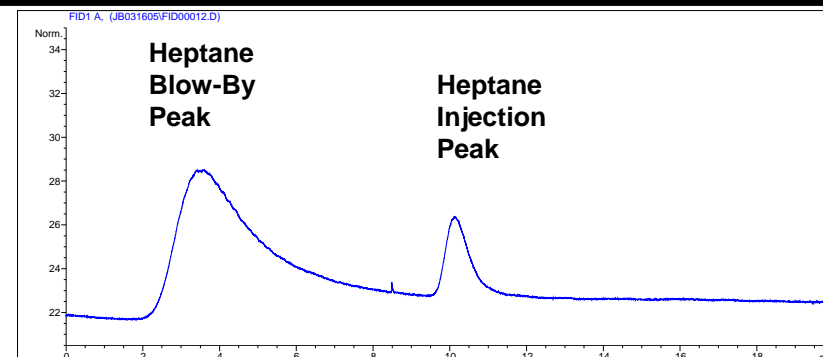
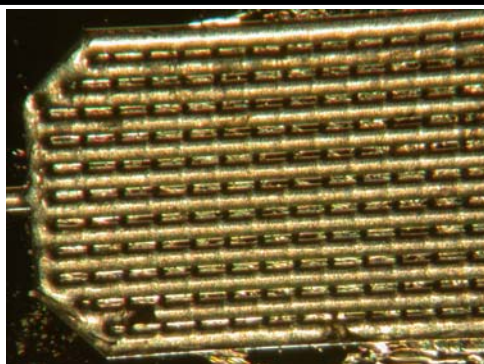
Experimental Setup to Test Preconcentrator Efficiency



Tortuous Path Base-Catalyzed Sol-Gel Coated Preconcentrator Collection Efficiency for Various Analytes

Analyte	Mass Injected (ng)	Area of Blow-by Peak	Area of Injection Peak	*% efficiency	Vapor Pressure (mmHg @ 25 C)
Methylene Chloride	987	1576	113	6.7	435
Chloroform	619	432	81	16	197
Toluene	64	0	890	100	28.4
Hexane	613	3703	128	3.3	151
Heptane	236	1470	137	8.5	46
Octane	75	450	325	42	14.1

One of the
Lower
Tortuosity
Designs

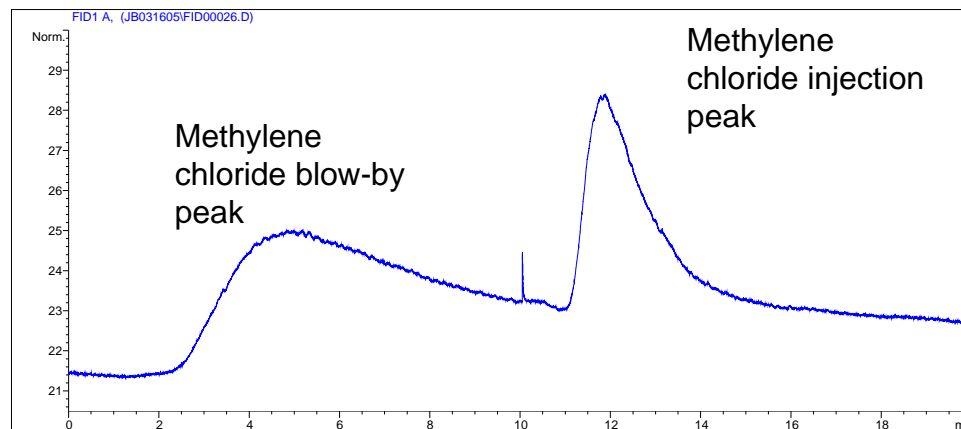
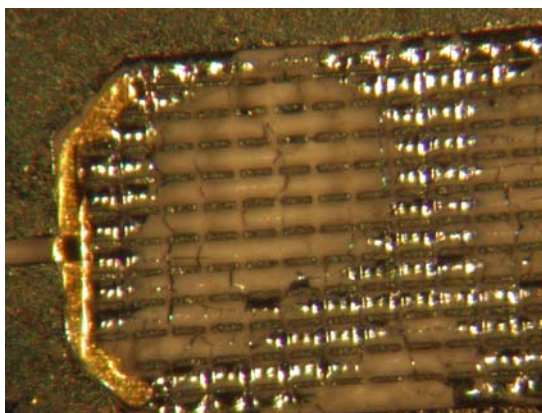


* % efficiency = $\text{Area injected} / (\text{Area blow-by} + \text{Area injected}) \times 100$

Tortuous Path Base-Catalyzed Sol-Gel Excessively Coated Preconcentrator Collection Efficiency for Various Analytes

Analyte	Mass Injected (ng)	Area of Blow-by Peak	Area of Injection Peak	*% efficiency	Vapor Pressure (mmHg @ 25 C)
Methylene Chloride	1657	2092	523	20	435
Chloroform	910	334	538	62	197
Hexane	613	926	3584	79	151
Heptane	236	11	1799	99	46

One of the Lower Tortuosity Designs



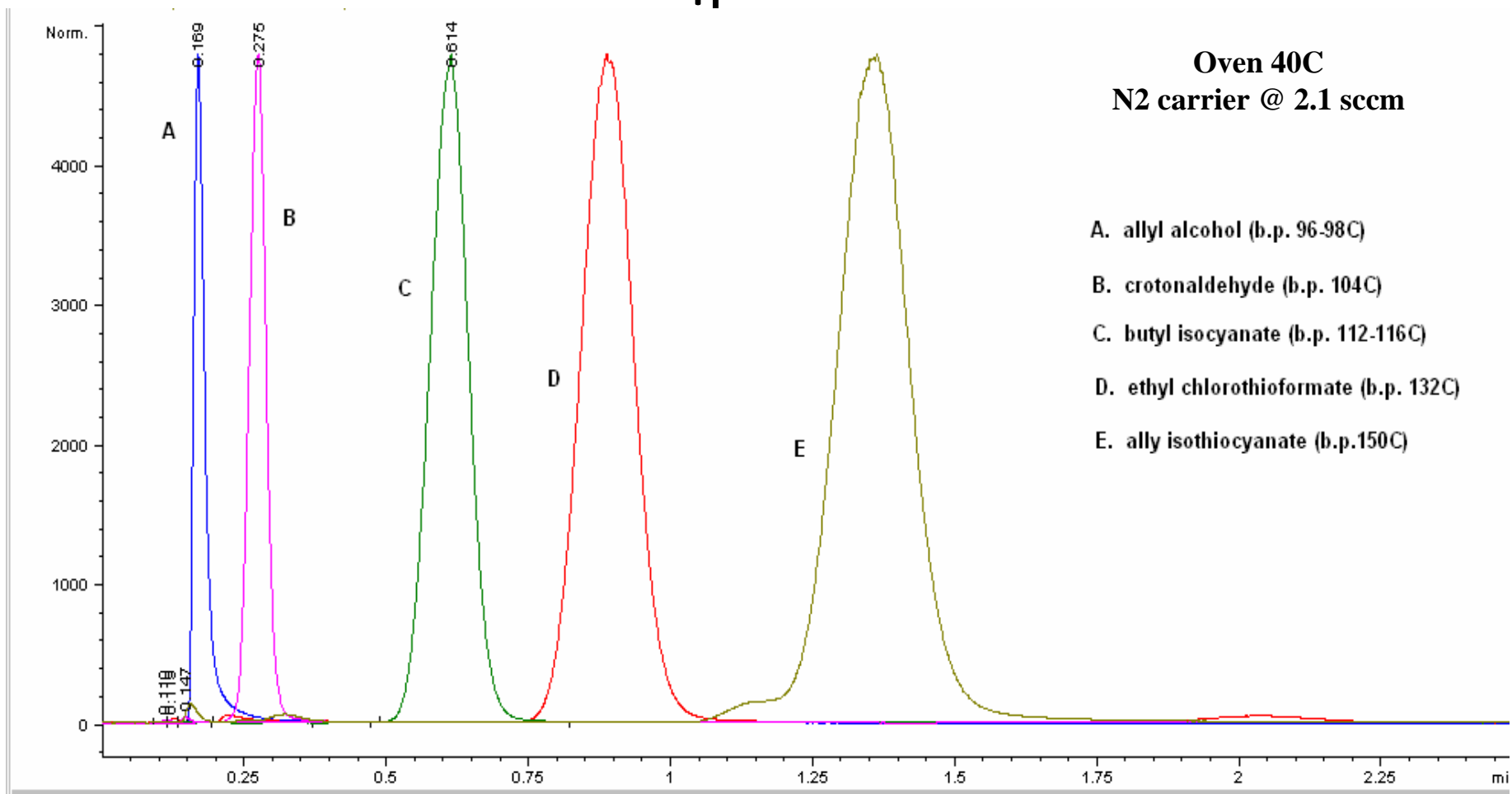
* % efficiency = $\text{Area injected} / (\text{Area blow-by} + \text{Area injected}) \times 100$

Volatile TICS Chromatography

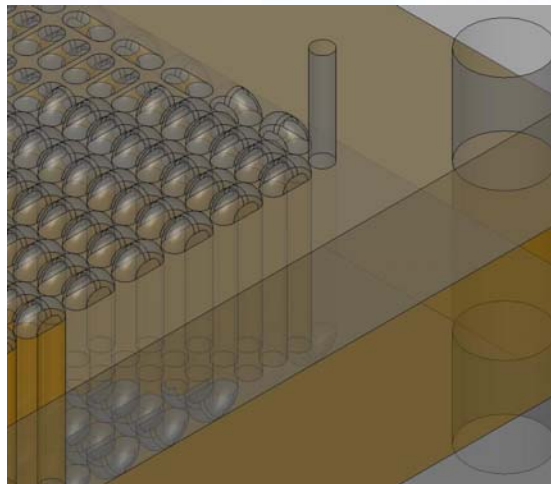
2 meter silicon based column

phase = polydimethylsiloxane

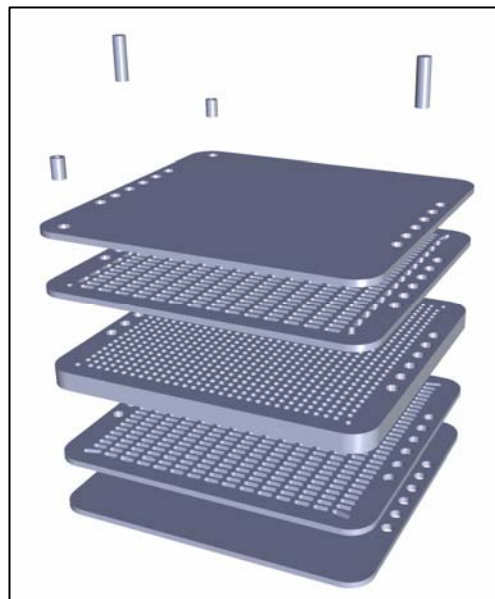
precursor - dimethyl (dimethylamino) vinyl



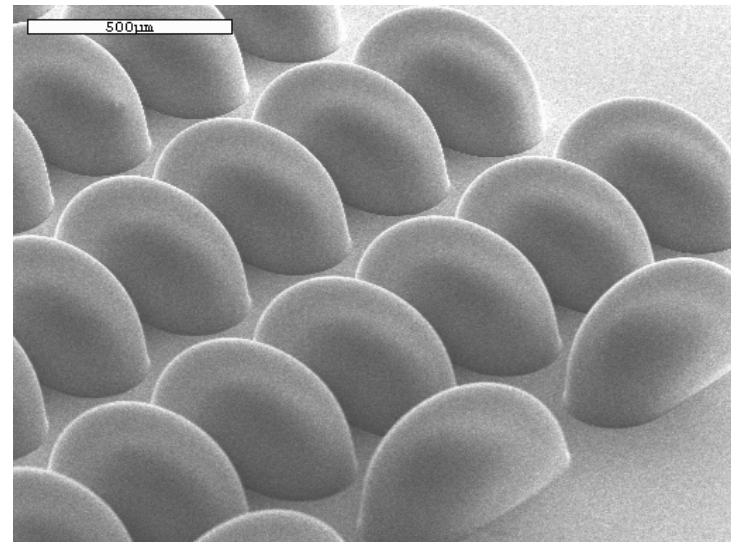
Nickel LIGA RTA Columns



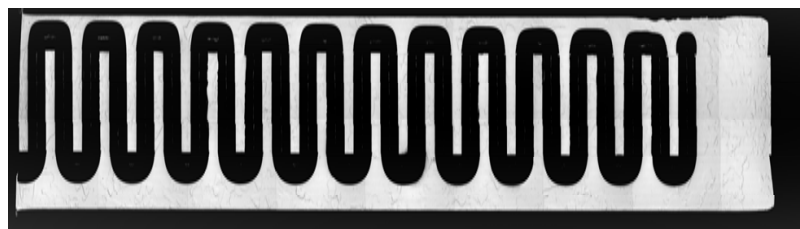
Exploded View of LIGA fabricated GC



New Circular X-Section GC Column to minimize band broadening. Longer columns can be made by stacking center sections

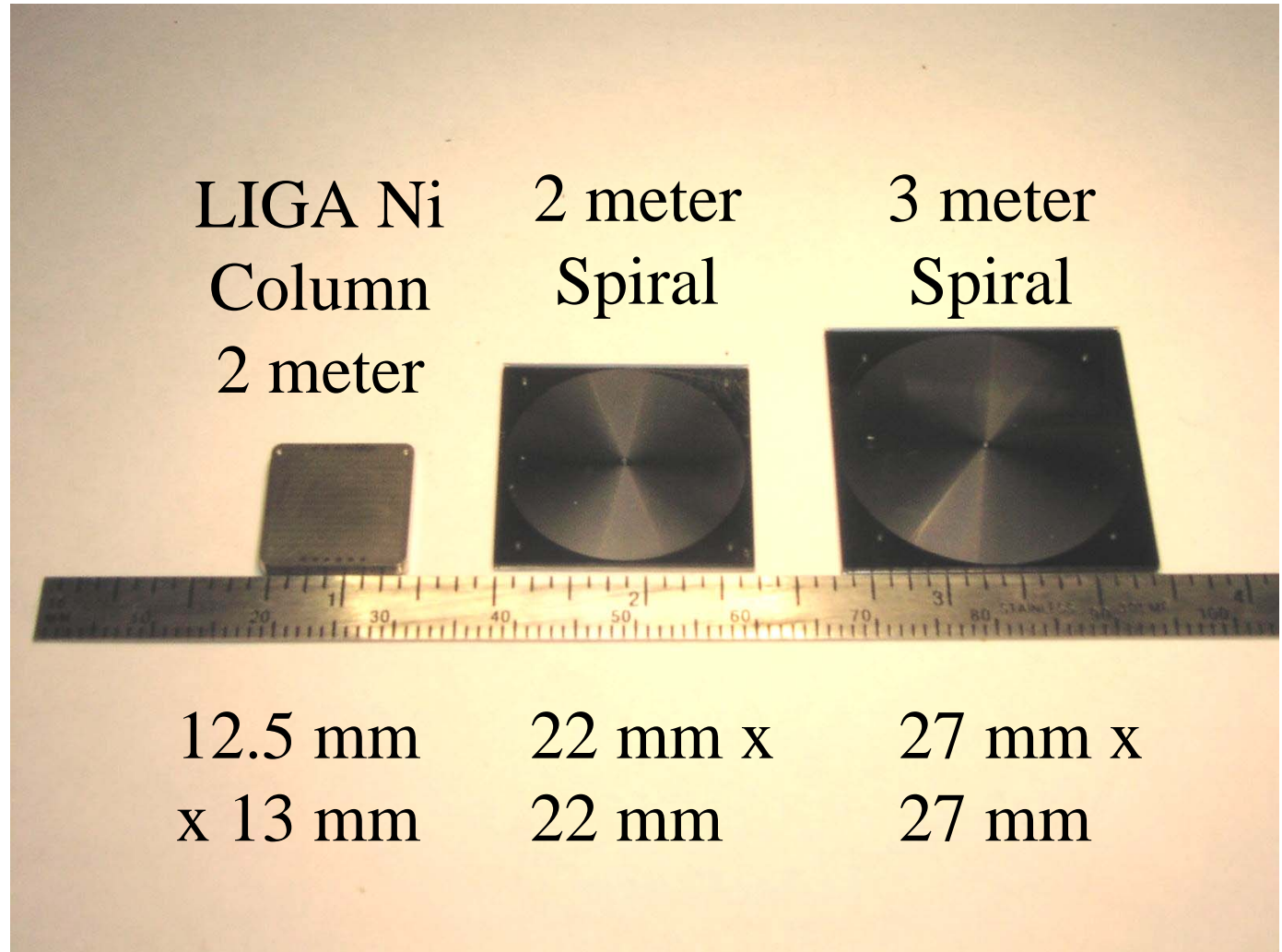


LIGA Molds for Rounded Turn Arouds.



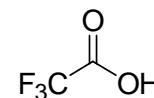
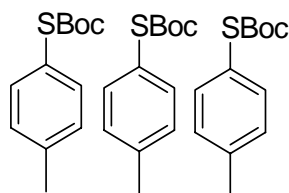
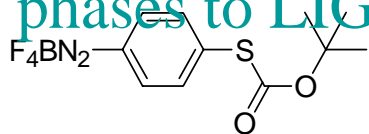
New GC Column Designs

- Length increase but footprint doesn't
- Minimizes corners where phase builds up



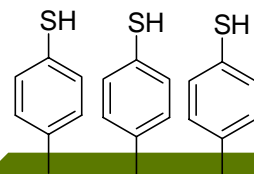
Diazonium Assembly Chemistry

Provides a “materials based” solution to attaching various stationary phases to LIGA generated GC columns



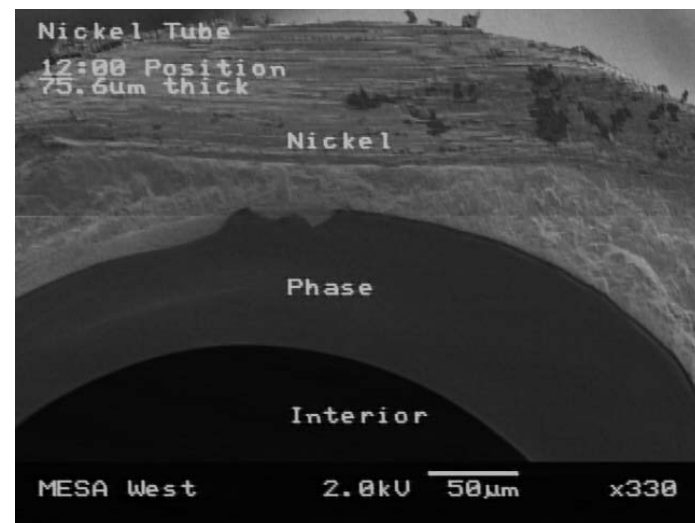
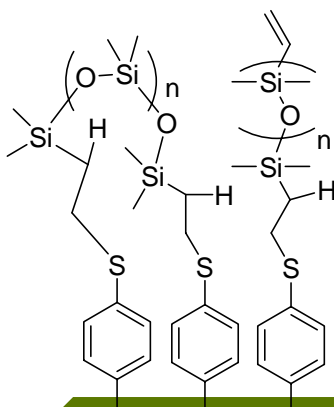
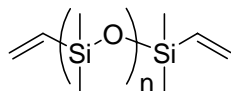
Ni Surface

Ni Surface

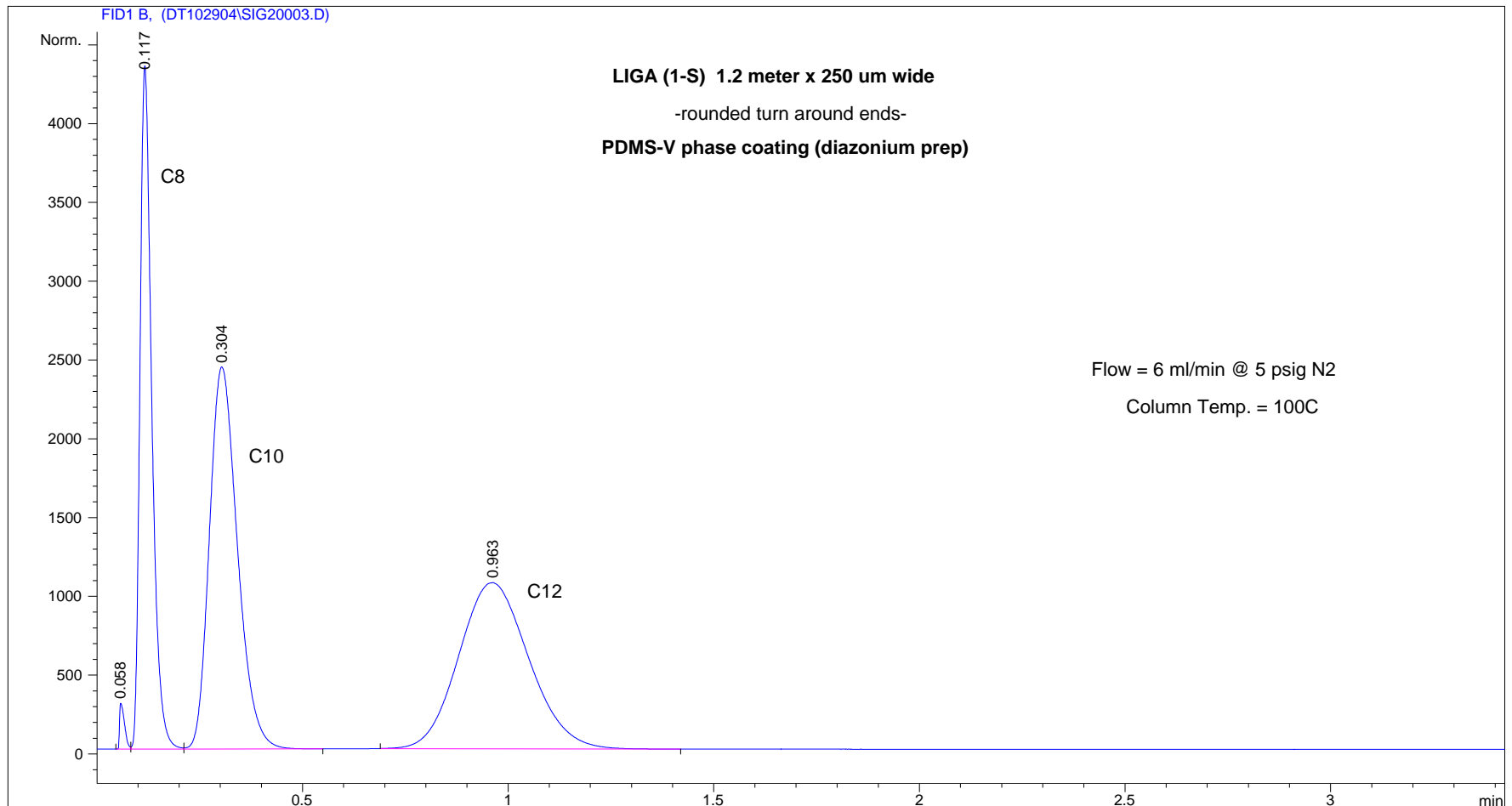


Ni Surface


Ni Surface



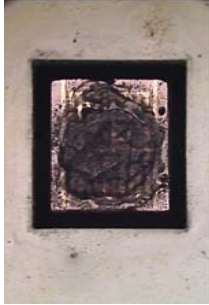
New Column with Homologous Series



Micro-Nitrogen Phosphorus Detector



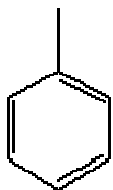
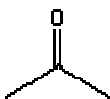
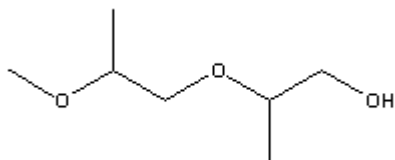
An example of one of the thermally isolated low power heated surfaces used as a basis for element specific detection



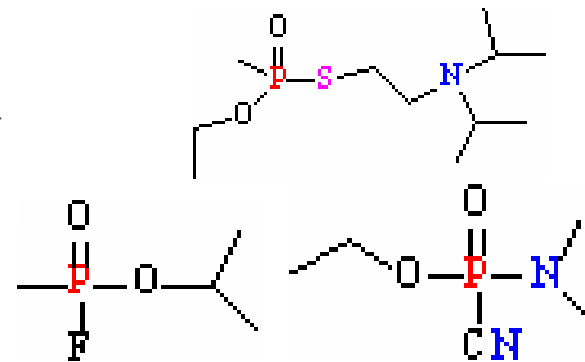
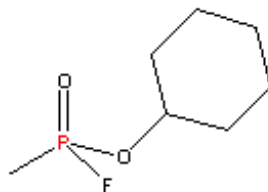
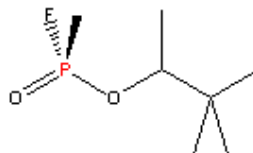
An alkali metal hydroxide catalyzed sol gel is used as a low work function surface. Thermochemically formed electronegative radicals abstract an electron from the hot sol gel surface.

This detector is currently working in phosphorus and nitrogen specific mode without the use of hydrogen gas. Ideal for handheld fielded microsystems.

This detector gives only a minimal signal for fuels and industrial solvents.



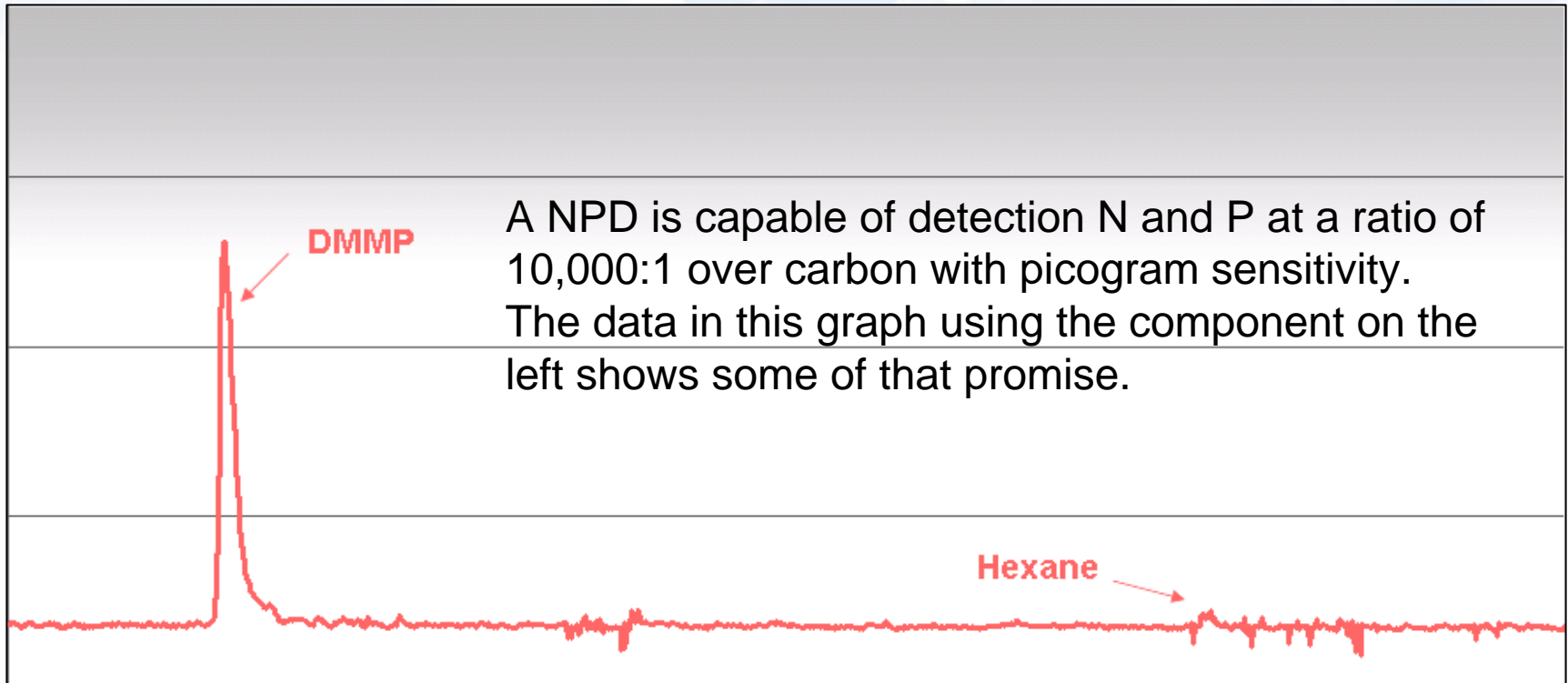
The signal is maximized for phosphorus or nitrogen containing compounds.



microNPD Detector -- TICs

	N	P	S	X
Acrylonitrile	x			
Allyl amine	x			
Allyl chlorocarbonate				x
Carbonyl sulfide			x	
Chloroacetone				x
Chloroacetonitrile	x			x
Chlorosulfonic acid			x	x
1,2 Dimethylhydrazine	x			
Ethylene dibromide				x
Methanesulfonyl chloride			x	x
Methyl bromide				x
Methyl chloroformate				x
Methyl chlorosilane				x
Methyl hydrazine	x			
Methyl mercaptan			x	
Phosphine		x		
Phosphorous oxychloride		x		x
Phosphorous pentafluoride		x		x
tert-Octyl mercaptan			x	
Trichloroacetyl chloride				x
Trifluoroacetyl chloride				x
Allyl isothiocyanate	x		x	
Carbonyl fluoride				x
Chloroacetaldehyde				x
Chloroacetyl chloride				x
Dimethyl sulfate			x	
Diphenylmethane-4'-diisocyanate	x			
Ethyl chloroformate				x
Ethyl chlorothioformate		x	x	x
Ethyl phosphonothioicdichloride		x		x
Ethyl phosphorous dichloride		x		x
Isobutyl chloroformate				x
Isopropyl chloroformate				x
Isopropyl isocyanate	x			
n-Butyl chloroformate				x
n-Butyl isocyanate	x			
n-Propyl chloroformate				x
Parathion	x	x	x	
Perchloromethyl mercaptan			x	x
sec-Butyl chloroformate				x
tert-Butyl isocyanate	x			
Tetraethyl pyrophosphate		x		
Toluene 2,4-diisocyanate	x			
Toluene 2,6-diisocyanate	x			
Carbon disulfide			x	

Sandia's Micro-NPD



A NPD is capable of detection N and P at a ratio of 10,000:1 over carbon with picogram sensitivity. The data in this graph using the component on the left shows some of that promise.

Data from these experiments will be used to design ion collection schemes on the same die to further miniaturize the NPD.

Acetonitrile was used to verify N detection but not pictured.

Micro NPD as the Detector for a Micro Analytical System

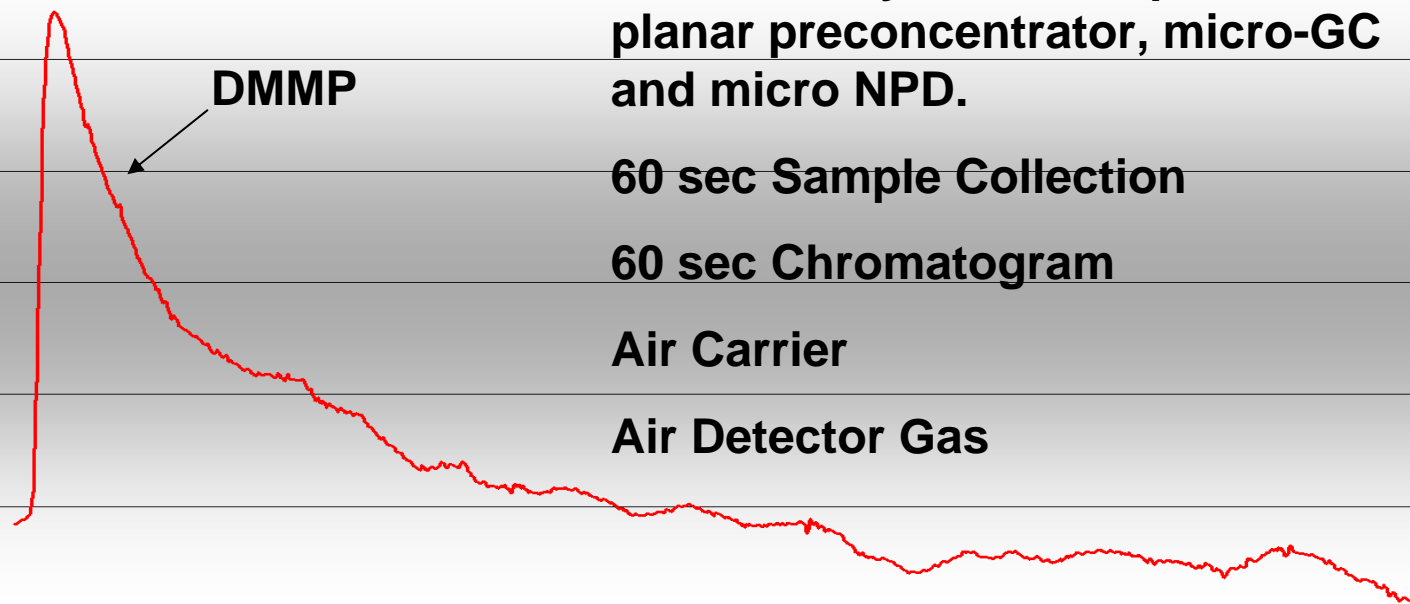
Preconcentrator selectively accumulates species of interest



Gas chromatograph separates species in time



Element specific detection for high confidence detection



This microsystem incorporated a planar preconcentrator, micro-GC and micro NPD.

60 sec Sample Collection

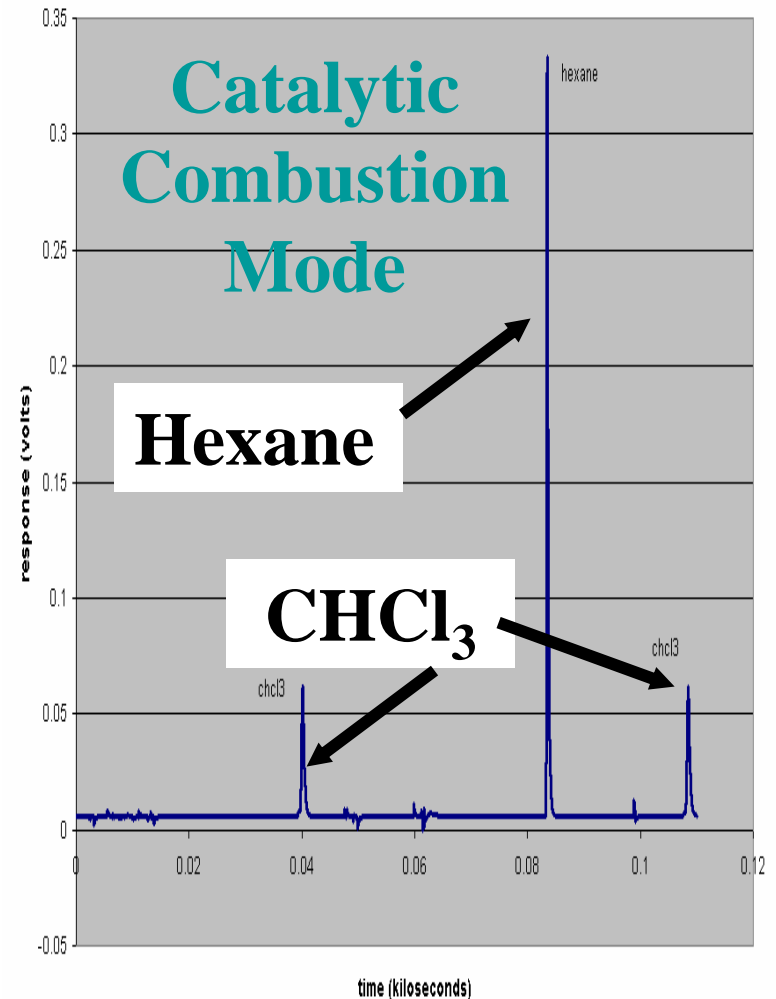
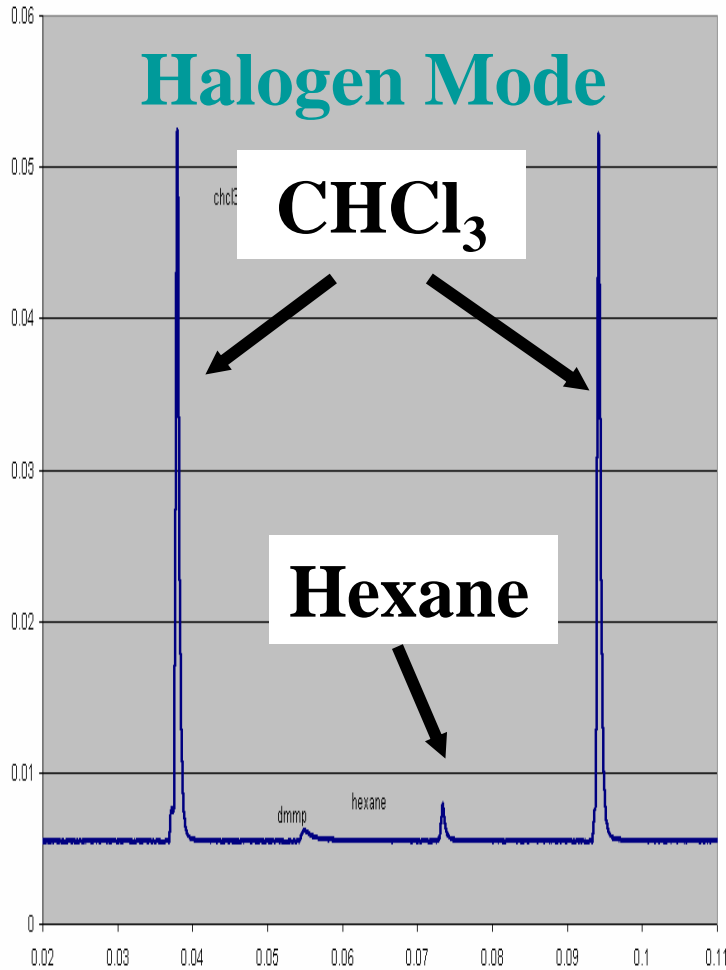
60 sec Chromatogram

Air Carrier

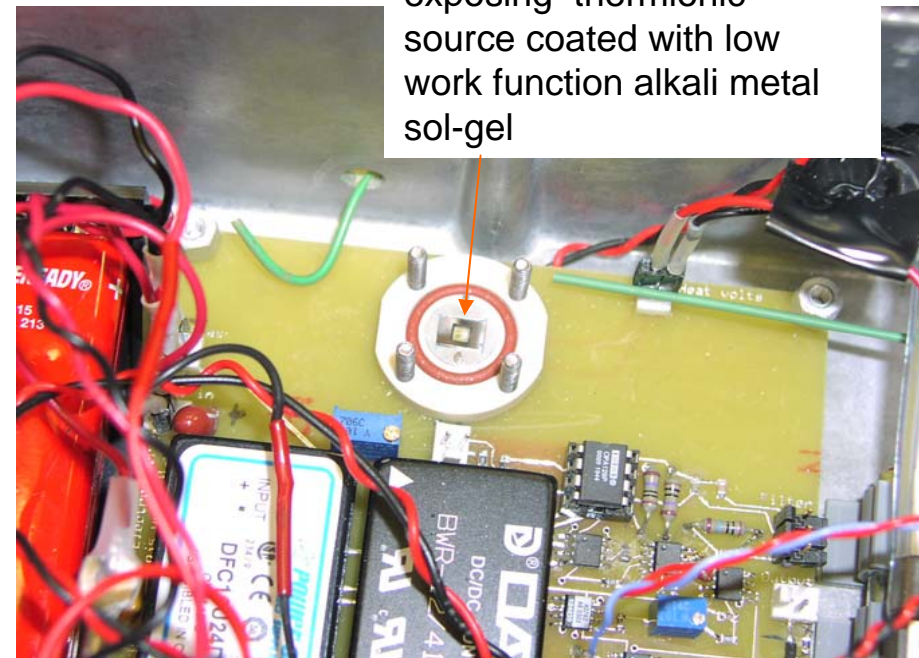
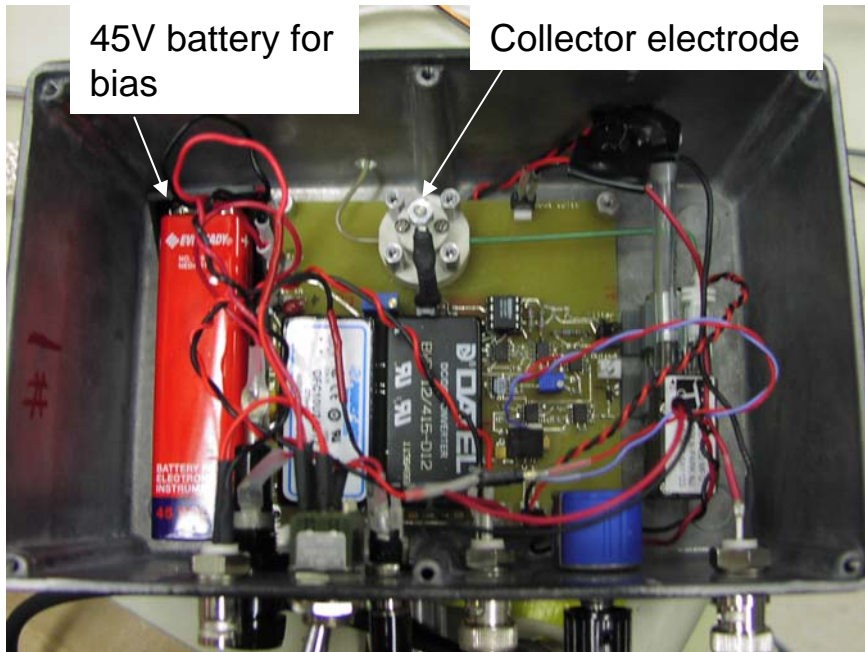
Air Detector Gas

Surface Ionization Detector from TID

Research inspired by our Success with Micro-TID



New Miniaturized Electrometer Circuit and Components for the Micro TID Detector

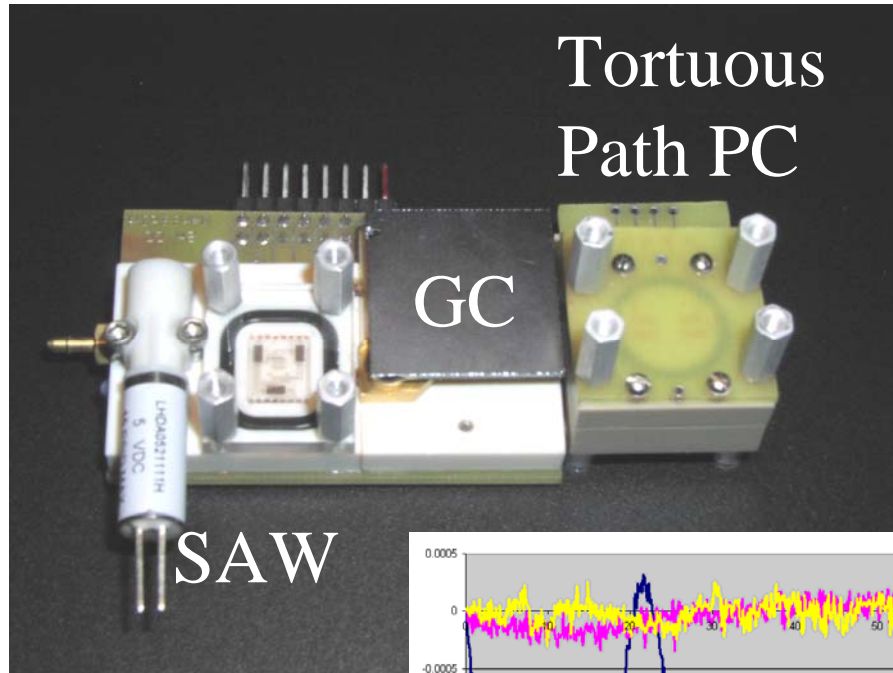


New Core Microsystem for TIC Analysis

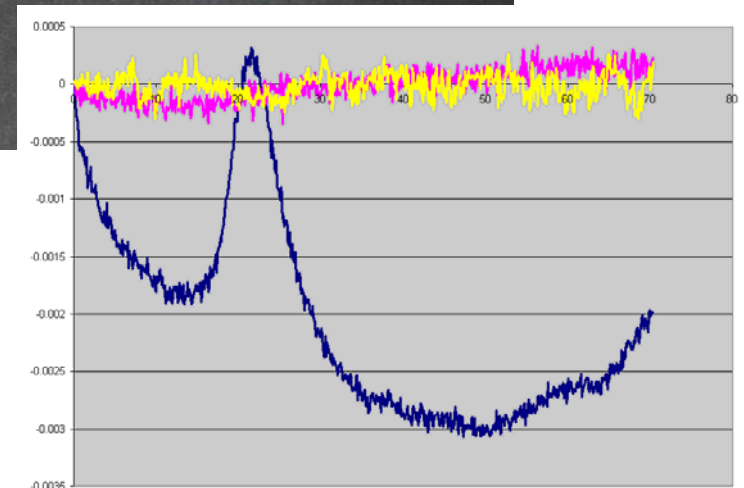
GOVERNMENT
C&E News Nov 22, 2004 CONCEN

Runaway reaction leads to toxic release, chemical board says

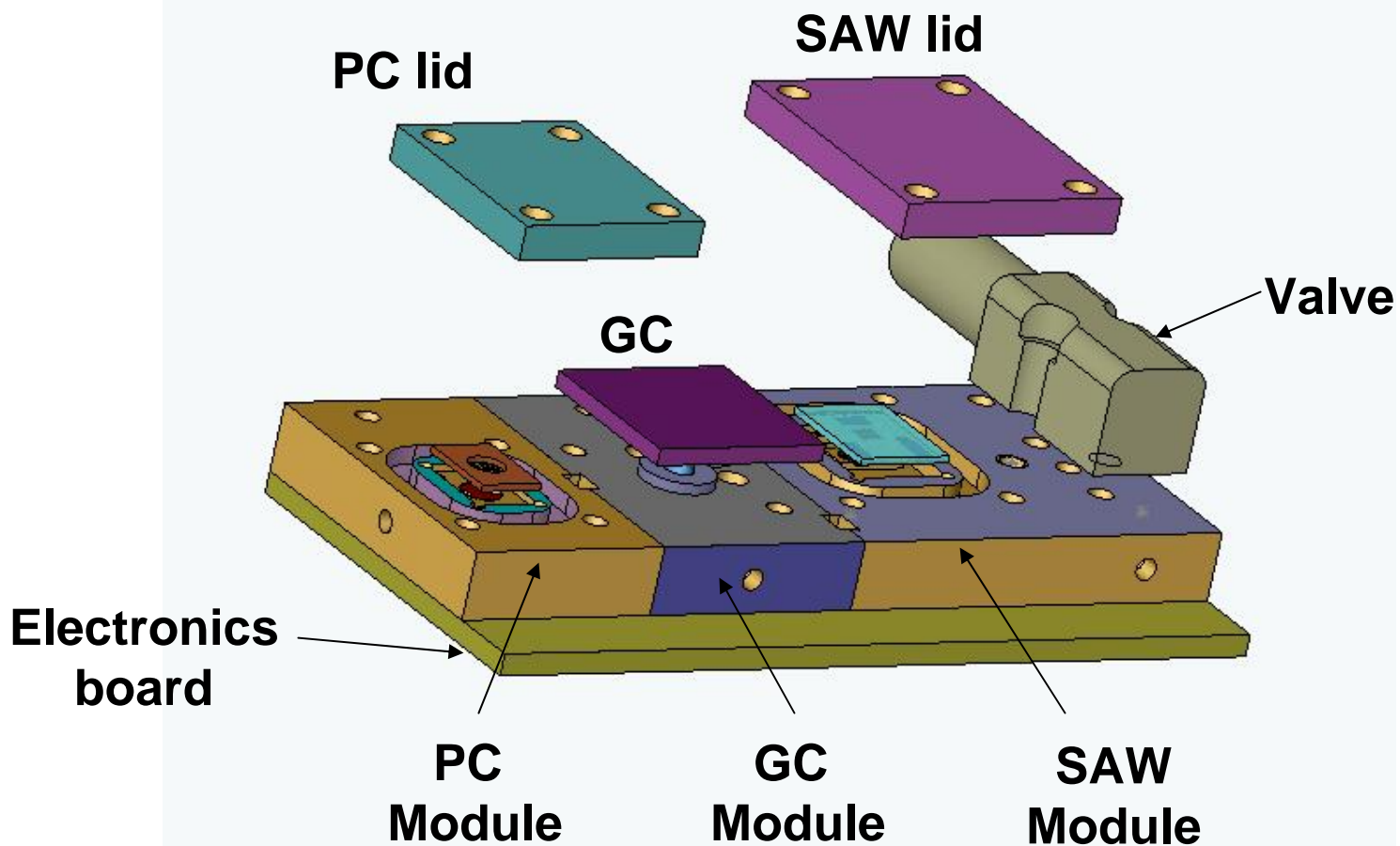
A chemical company's inability to control a chemical reaction and an erroneous accident emergency plan led to injuries of 17 police and ambulance personnel and the evacuation of more than 100 homes and businesses, says the Chemical Safety & Hazard Investigation Board in a draft accident report concerning MFG Chemical Inc., Dalton, Ga. Released last week, the report says a cloud of toxic allyl alcohol and possibly hydrogen chloride was released in April when a safety device on a 2,000-gal chemical reactor burst while the company's first production-scale batch of triallyl cyanurate was being processed. Board investigators say MFG was unable to control heat generated in the process, and that caused the runaway reaction. However, the board also says the company's emergency plan focused only on allyl alcohol's flammability and not its toxicity. As a result, MFG was not equipped to handle the toxic release and failed to communicate the release's toxicity to emergency responders and the community. Responders, the report says, lacked protective equipment and had no monitoring devices to detect airborne toxicity. They were forced to withdraw, as were ambulance crews who were also unaware of the toxicity and were sickened and unable to treat or remove victims. The accident is the latest in a string of incidents caused by uncontrolled chemical reactions, says the board, which is currently investigating seven similar accidents. A final report on the MFG incident is expected in the spring.



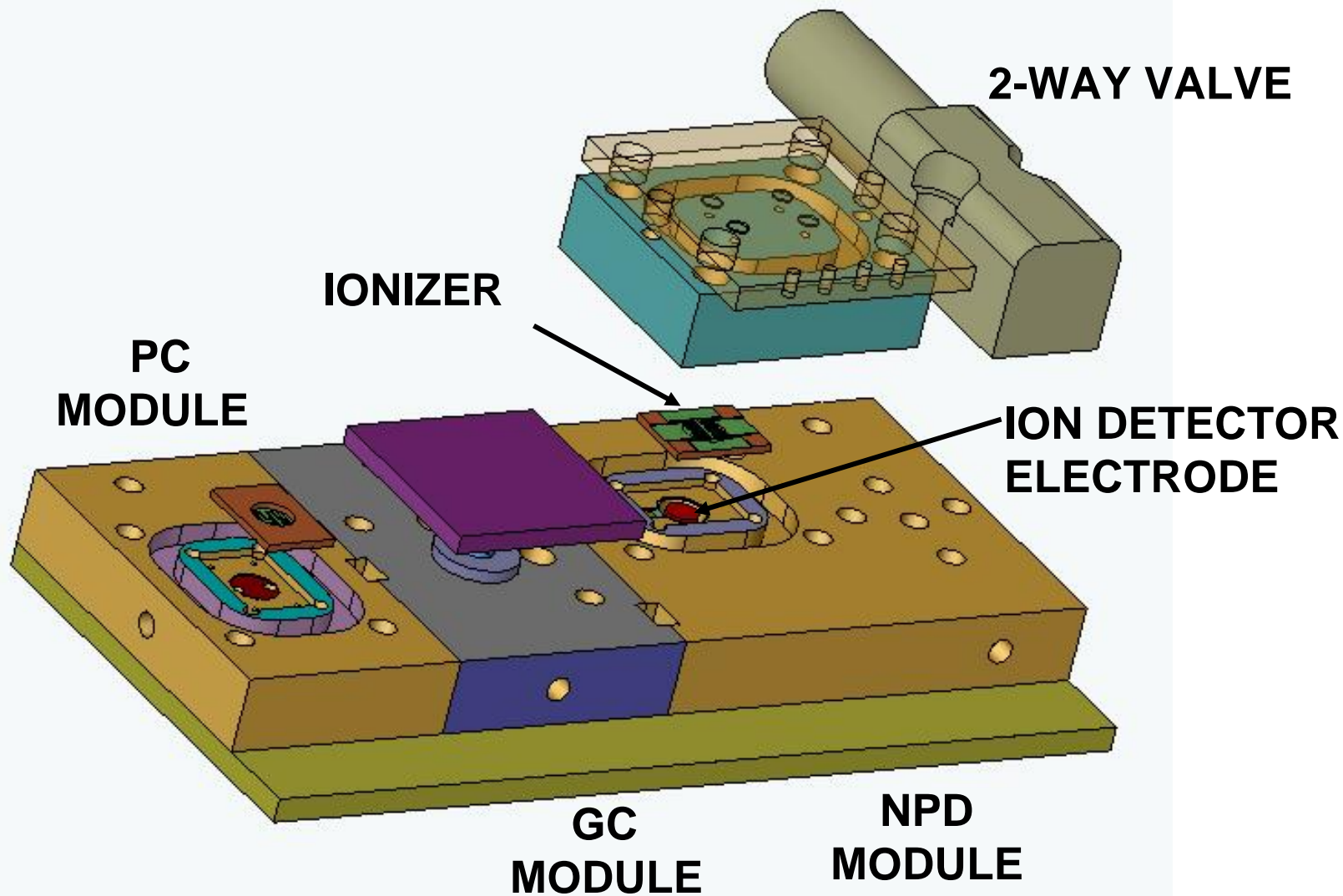
60 sec collection
of 22ppm Allyl
Alcohol, 60 sec
chromatography



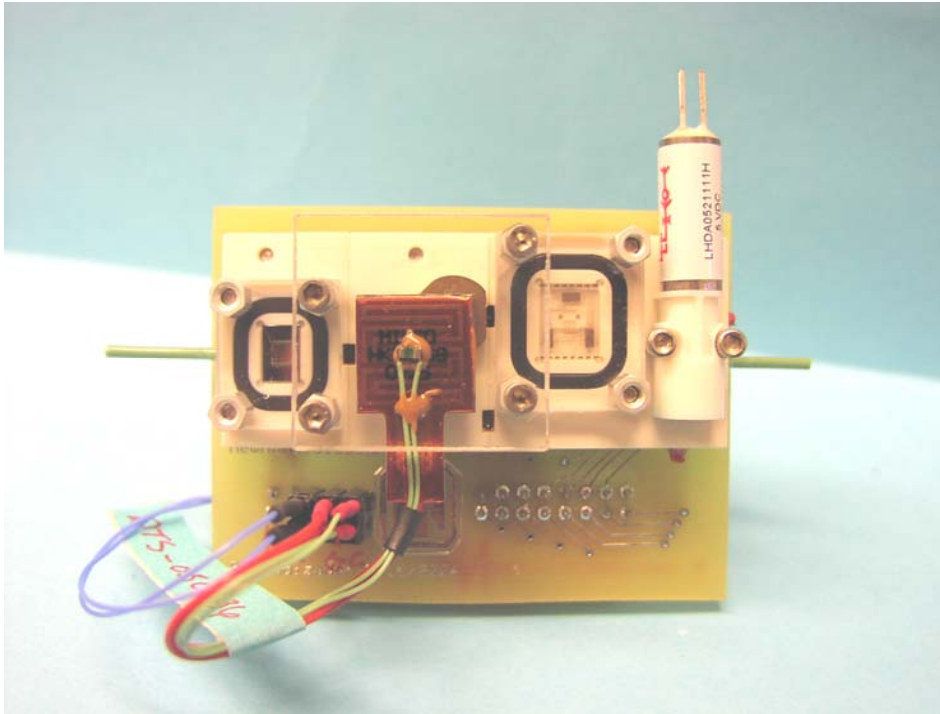
Modular MicroChemLab™ Architecture (With SAW Detector)



Modular MicroChemLab™ Architecture (with NPD Detector)



Modular Design Manifold



- Simple component change
- Easy to test new components
- Shortens design time

PDA Controlled Dual Channel Field Analyzer

