

Micro Total Analytical Systems Department Overview

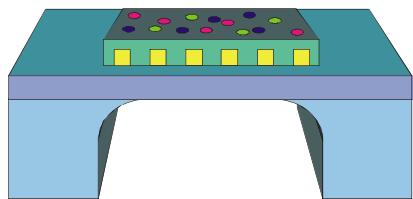
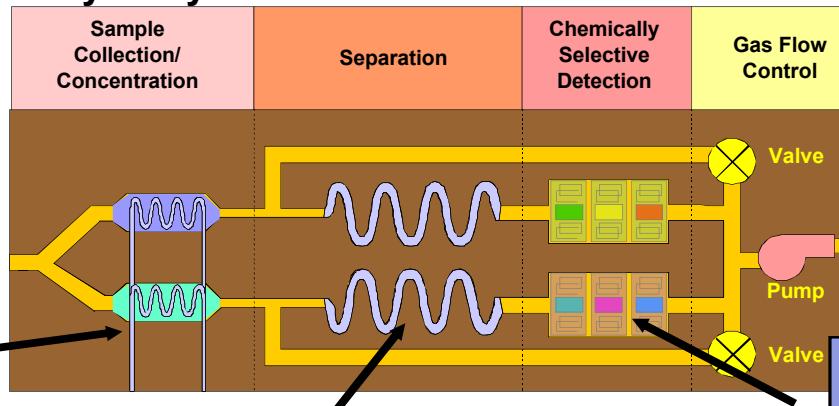
Patrick Lewis, Kamyar Rahimian, Richard
Kottenstette, Joy Byrnes, David Wheeler,
Larry Stotts

prlewis@sandia.gov 505-284-3315

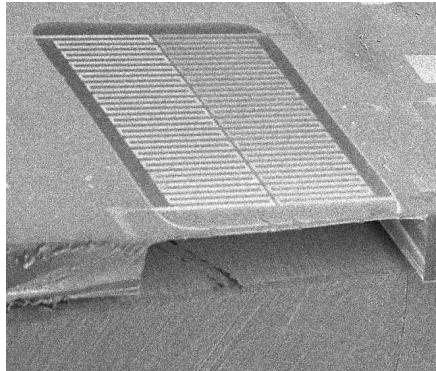
Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company,
for the United States Department of Energy's National Nuclear Security Administration
under contract DE-AC04-94AL85000.

Field Portable Chemical Analysis System

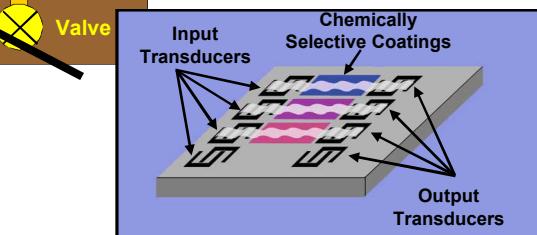
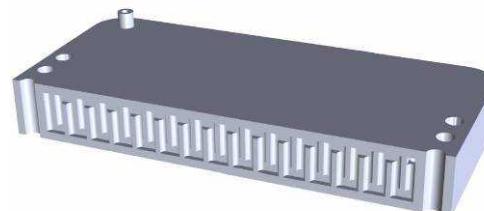
A hand-held chemical analysis system that uses three microfabricated analysis stages



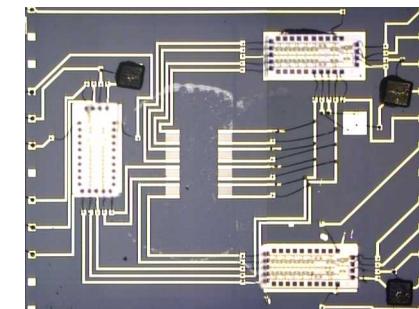
Preconcentrator accumulates species of interest



Gas Chromatograph separates species in time

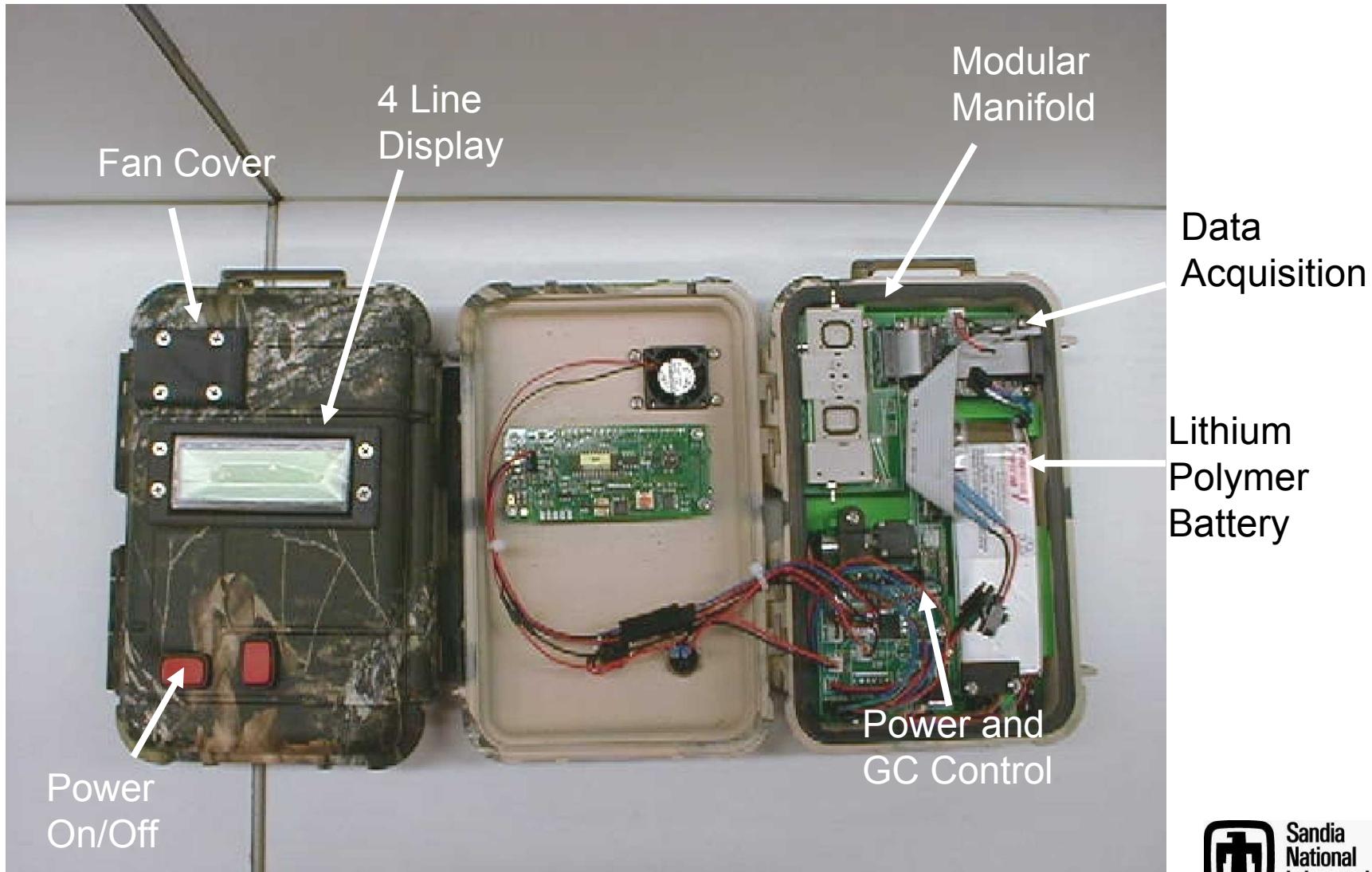


Acoustic Sensors provide sensitive detection

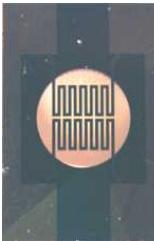
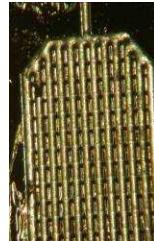
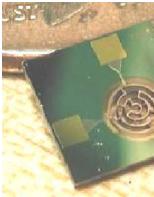
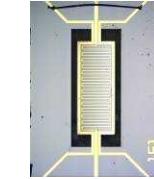
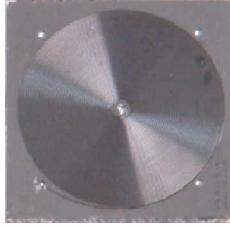
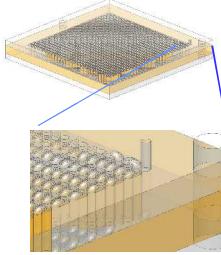
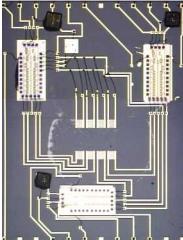
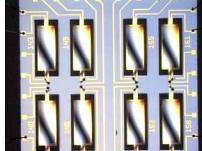
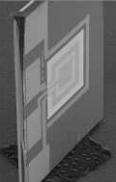
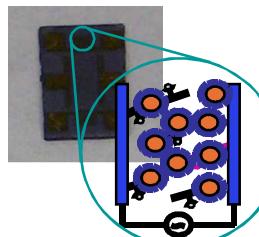


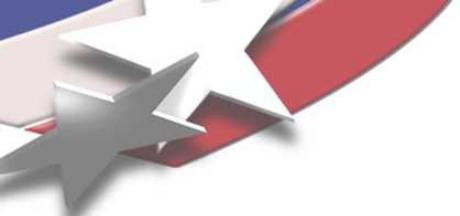


Handheld Chemical Analysis System



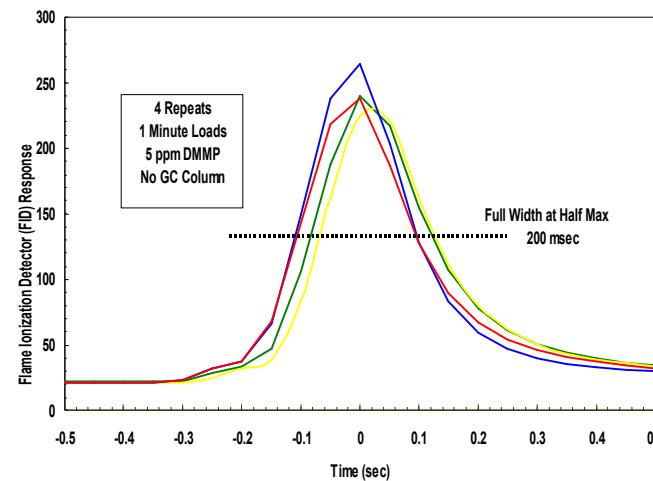
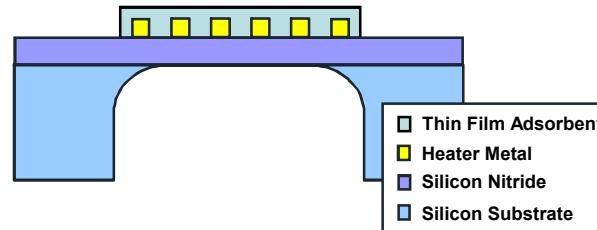
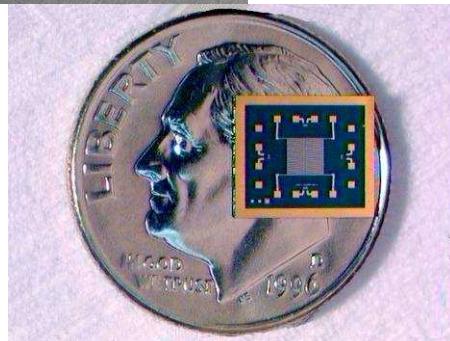
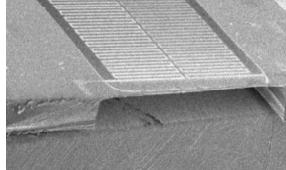
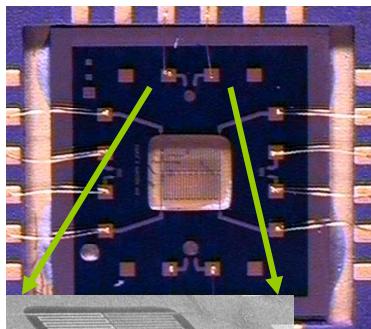
Micro Analytical System Components

Field Tested	Technology Maturity Level	Requires More Development
Sample Collection	 Planar PC Sharp sample inject – most mature 	 Tortuous Path PC better retention even for VOCs  3d PC Increased surface area and improved flow contact Smart PC – enables intelligent operation and greatest dynamic range (miosis to IDLH)
Separation	 High aspect ratio column in silicon 	Circular x-section column made in Ni using LIGA. Highest resolution separation
Detection	 SAW Array with integrated electronics for DC-in;DC-out operation. 	 Micro Flame Ionization Detector → “universal” GC detector.  Pivot Plate resonator for high temp applications.  Micro Nitrogen Phosphorus detector the most sensitivity with the greatest rejection of interferences.



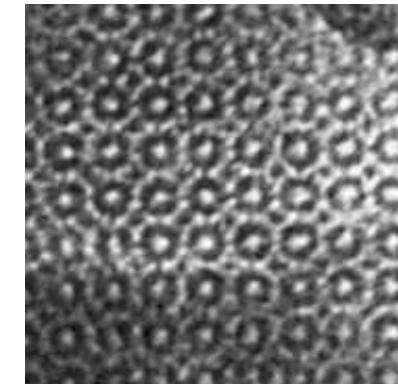
Preconcentrator

Thermally Isolated Heater Provides Rapid and Low Power Thermal Desorption of Analyte Collected into Thin Film Adsorbent

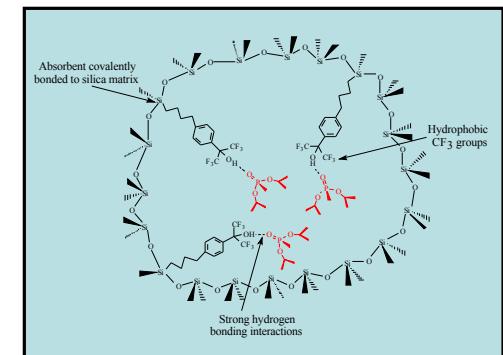


Rapid Thermal Desorption from Micromachined Preconcentrator

Novel Sol-Gel Techniques Provide Thin Film Adsorbents with High Uptake and Chemical Selectivity



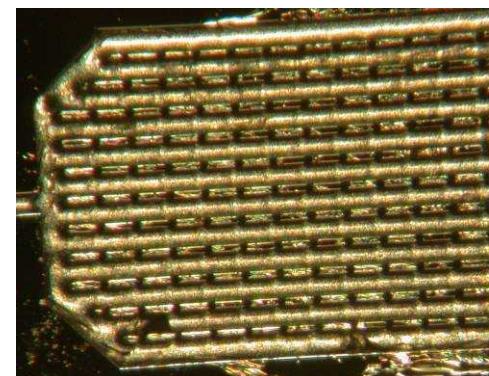
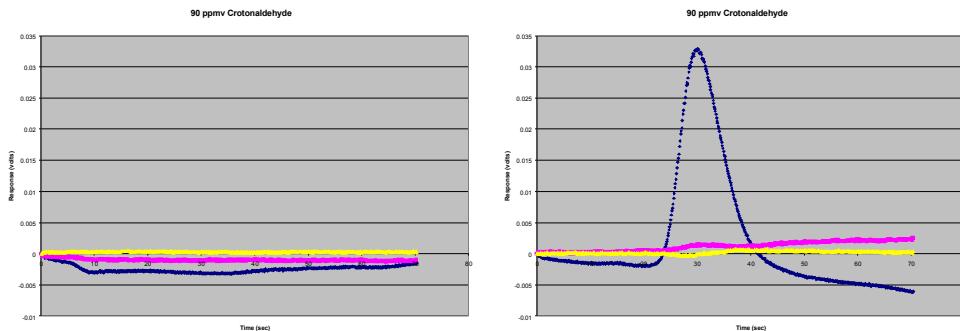
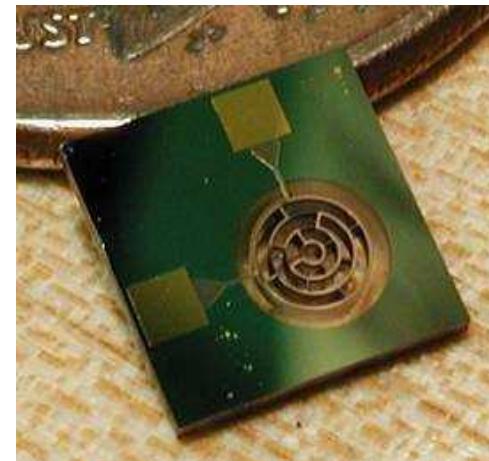
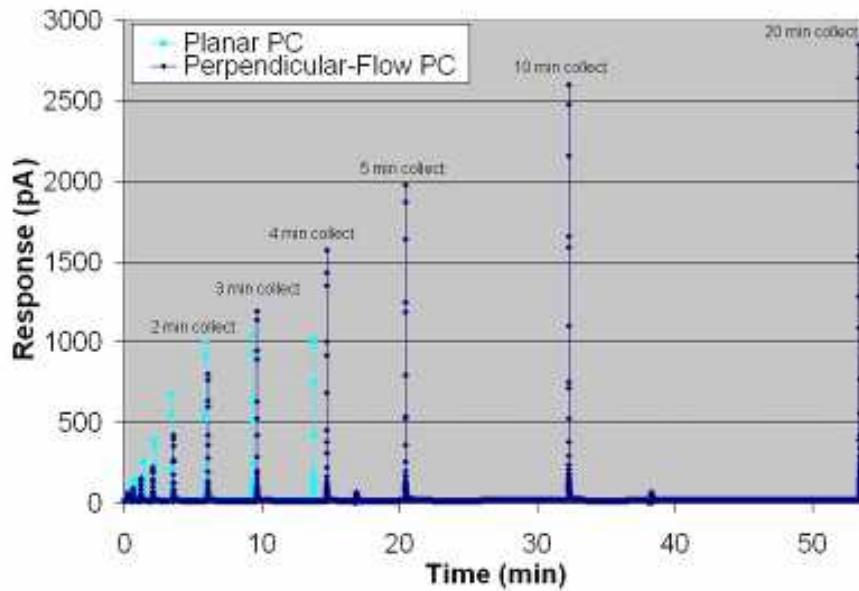
Tailored Porosity



Tailored Surface Chemistry

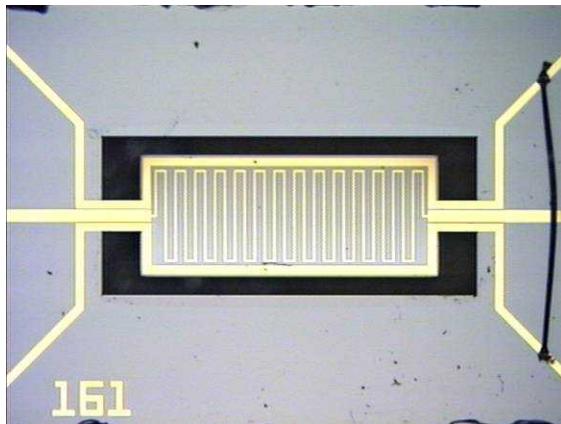


High Capacity micro-Preconcentrators





SMART PC Tests

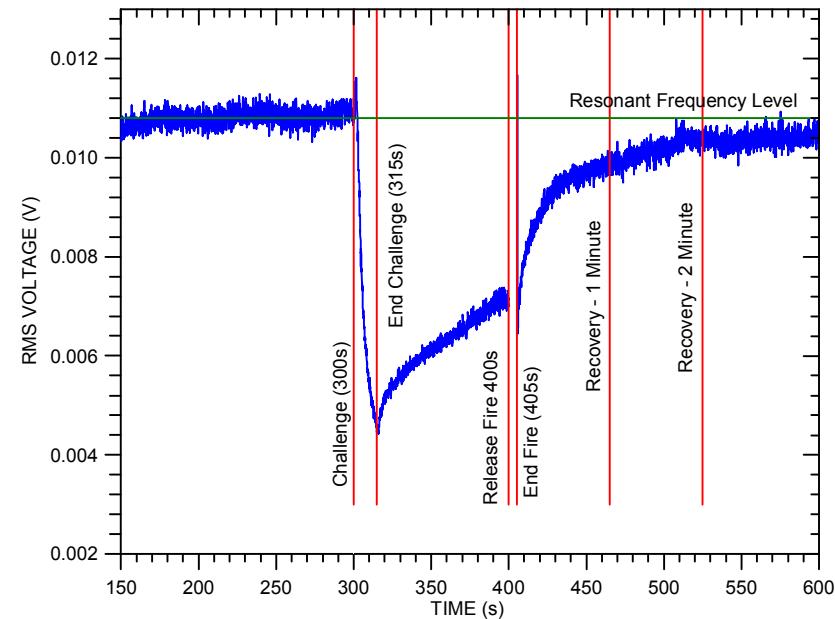


SMART PC tests

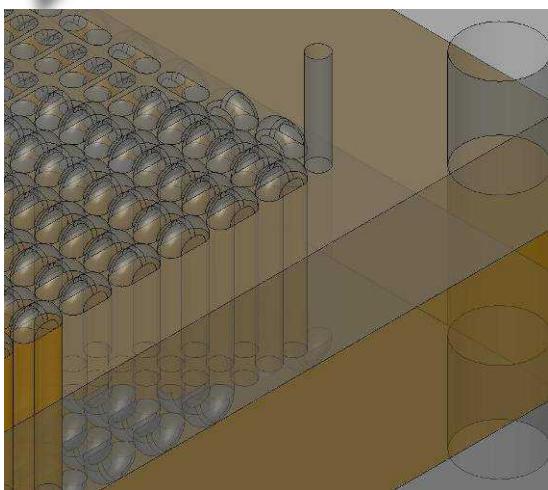
- Paddle at resonance, 11584 Hz
- DMMP challenge
- Challenge removed – 85s hold
- 5s fire on heater
- Recovery in ~120s

Tests on device TTH3-06

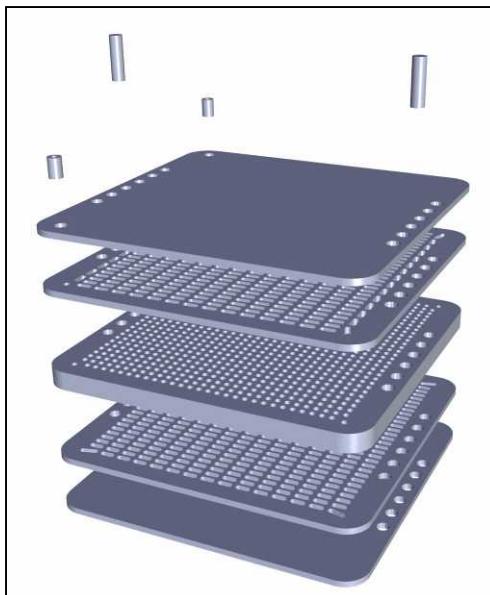
- Coated with 2 μ g of Solgel
- Resonance at 11584 Hz
- 74 ohm heater



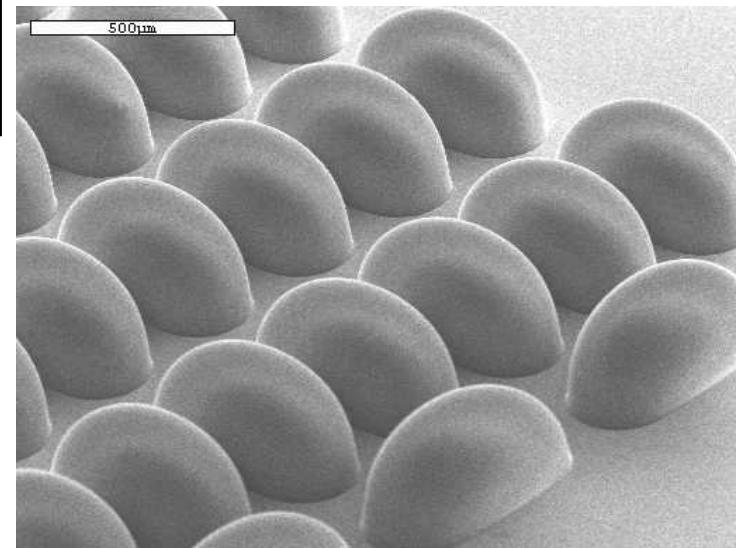
Nickel LIGA RTA Columns



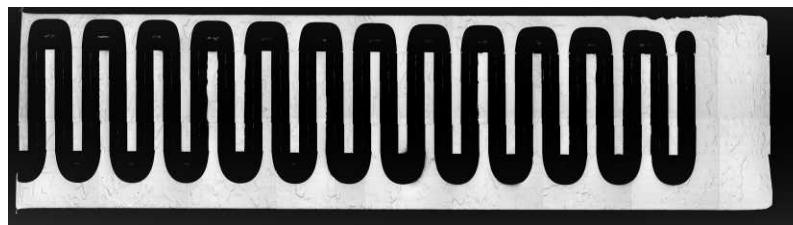
Exploded View of
LIGA fabricated GC



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

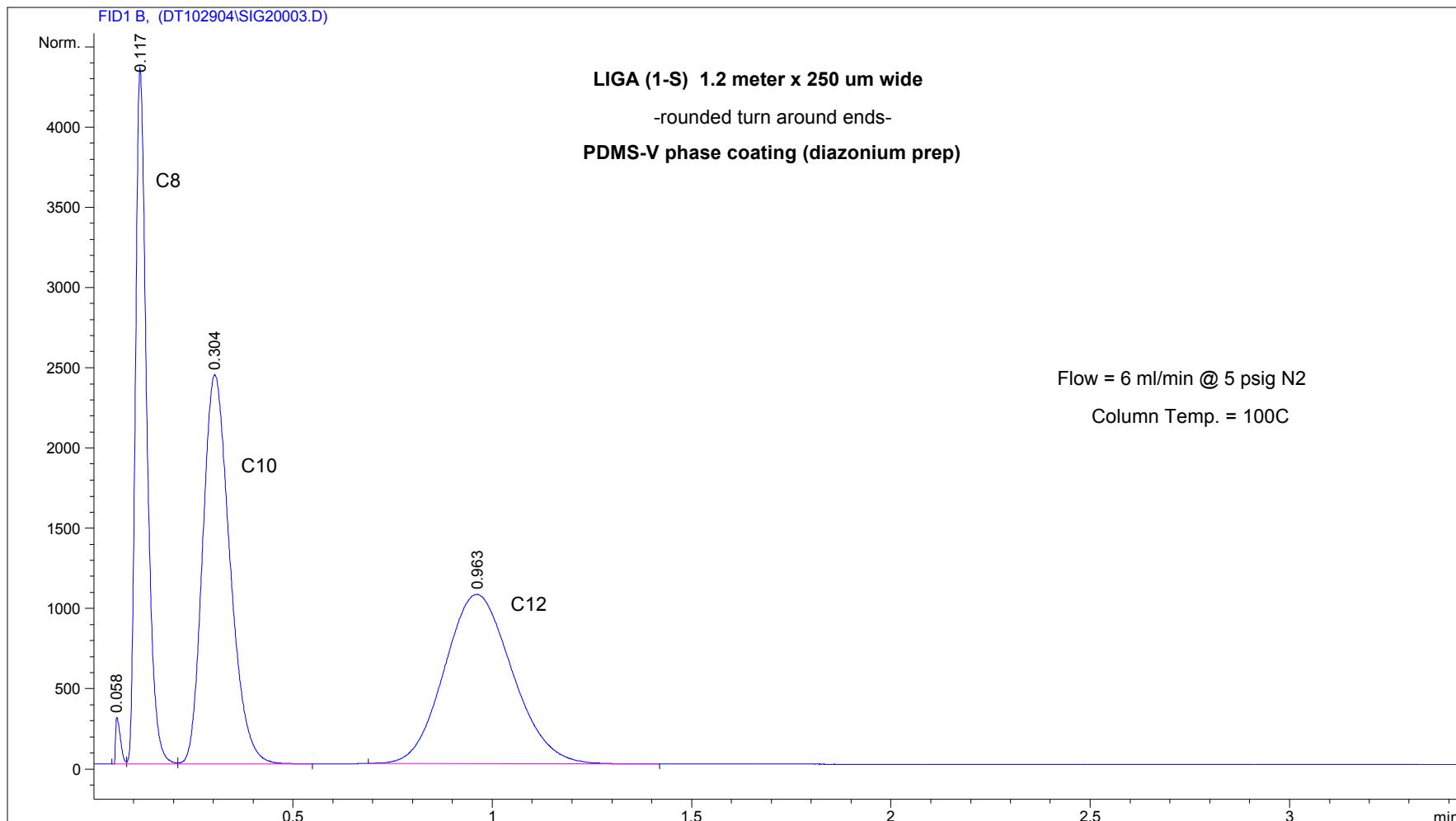


LIGA Molds for Rounded Turn Arounds.



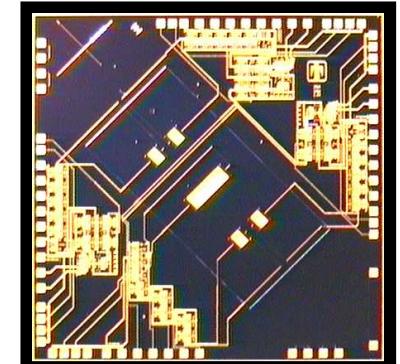
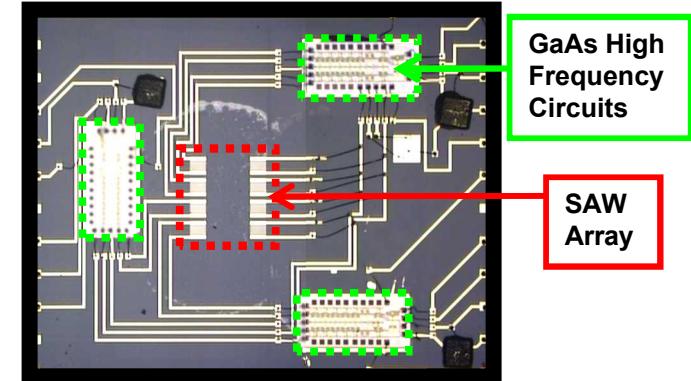
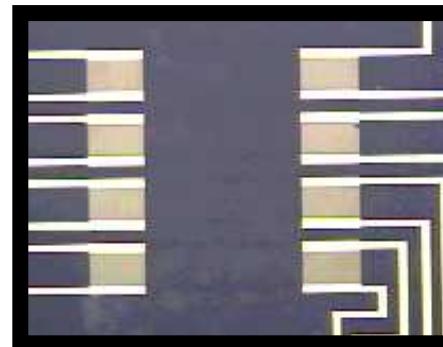
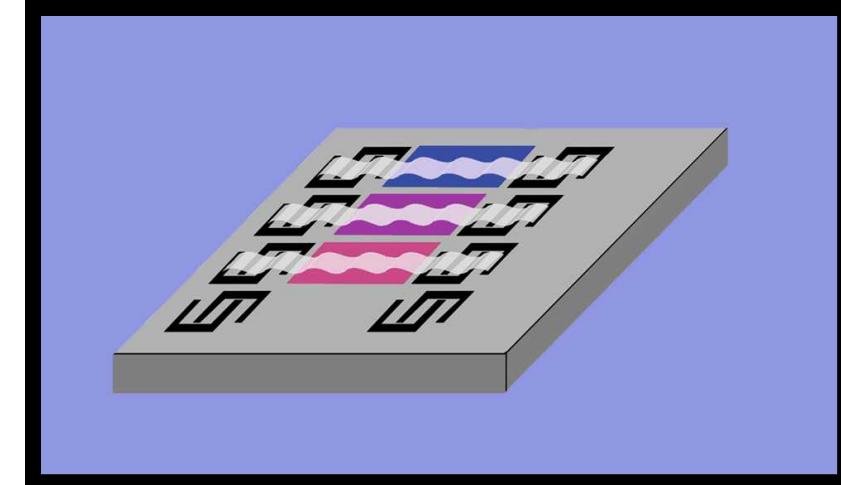


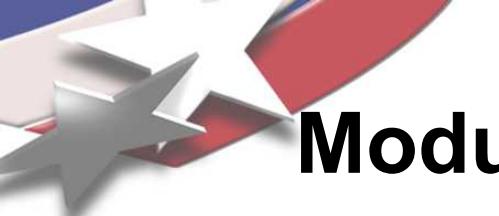
Homologous Series



Surface Acoustic Wave (SAW) Detector

- Surface acoustic wave is excited/detected using interdigital transducers on a piezoelectric substrate
- Sensor coating momentarily absorbs analytes eluted from GC column, changing SAW velocity (phase shift).
- Pattern of responses from array augments discrimination of GC separation

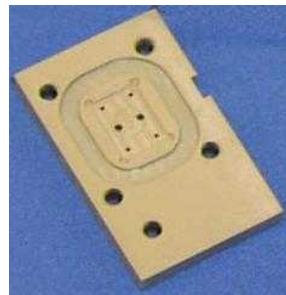




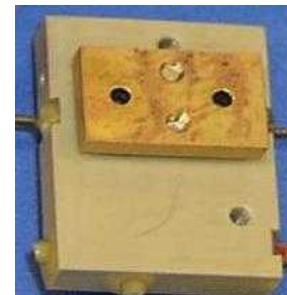
Modular Manifold for Gas Components

- Modules can operate in parallel to maximize analytes and minimize time
- Different modules can be added to change detector or preconcentrator
- Easy to replace components to facilitate testing different films
- Each piece makes electrical and fluidic connection

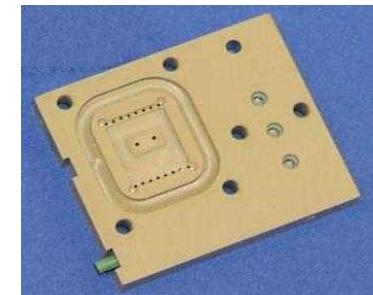
Preconcentrator manifold



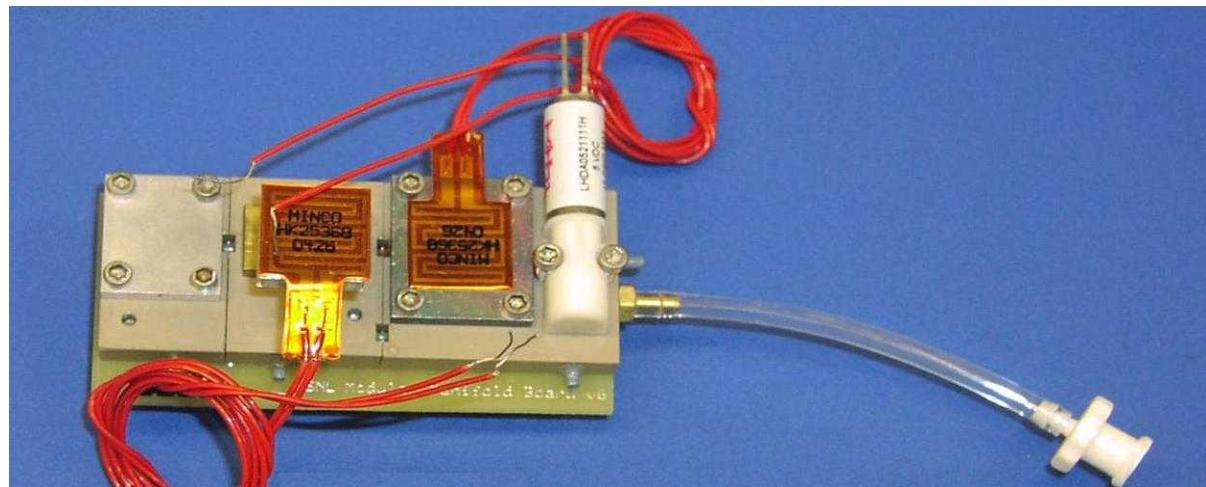
GC Column manifold



SAW Array Manifold

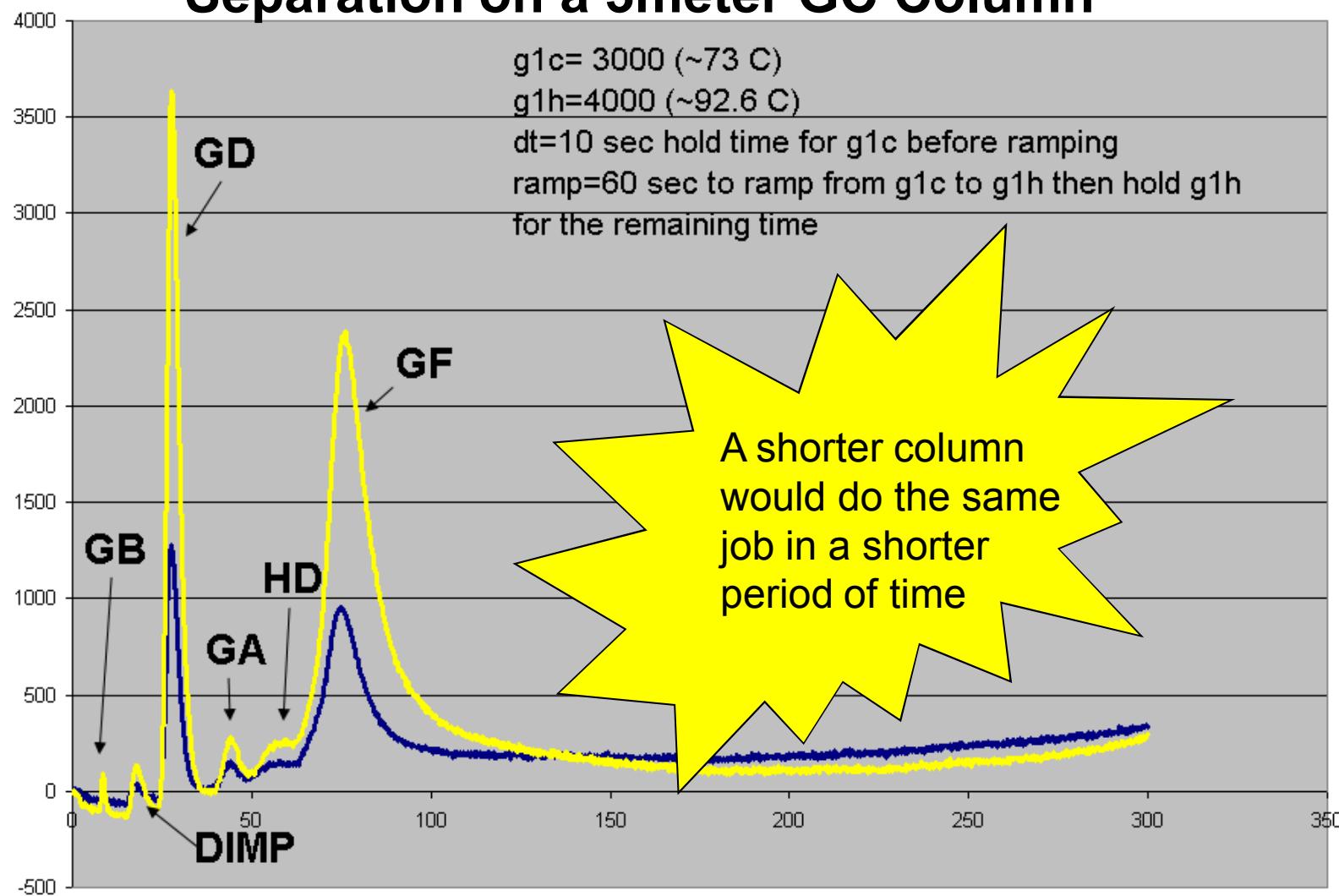


Adding modules together results in an easy to use manifold



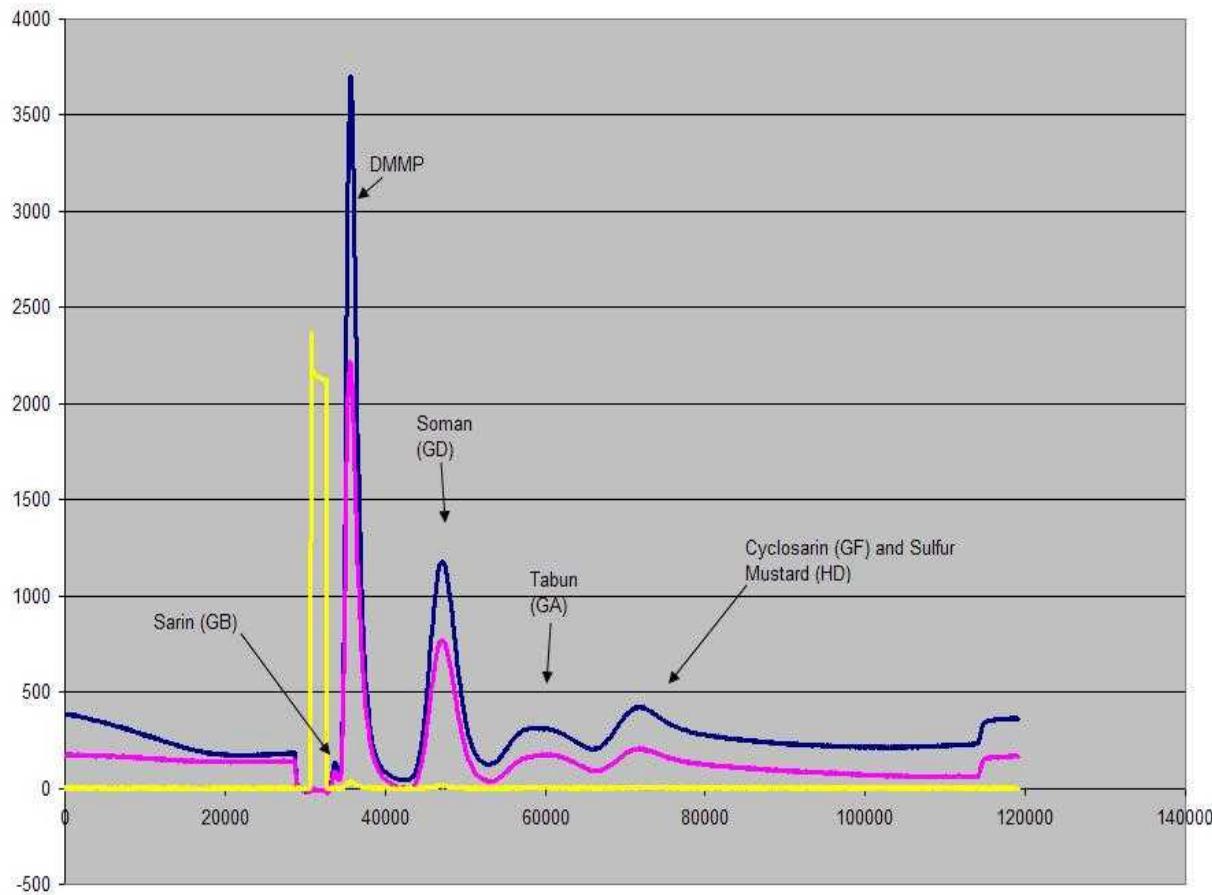
CW Agent, Byproduct, and Blister Agent Separation on a 3meter GC Column

Some detectors will merely tell you "Nerve" or "Blister". Our GC column enables this system to report which nerve or which blister.



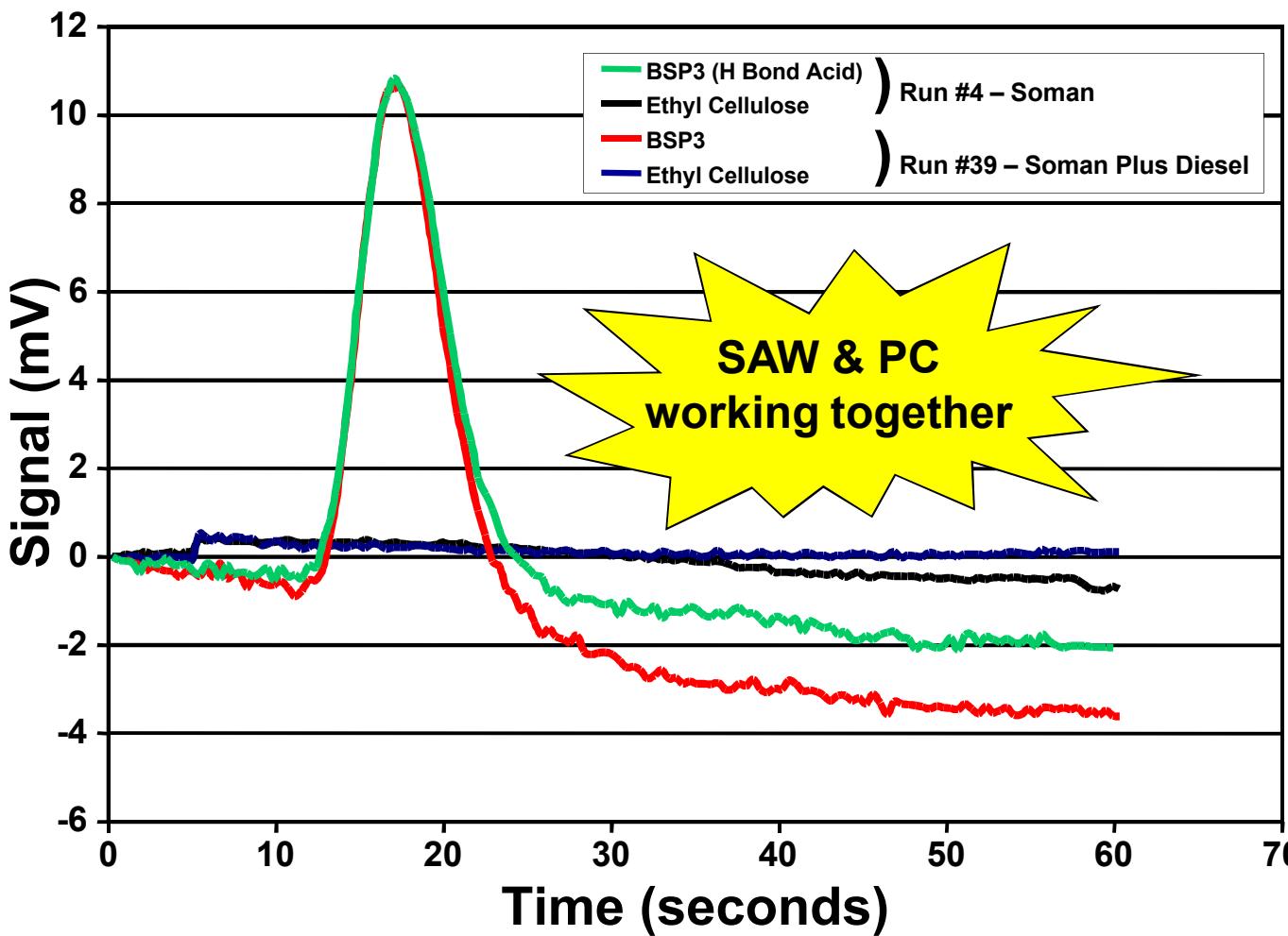


Handheld System Separating 5 Target Chemicals



Onboard
Calibration and
peak
searching
provide
Chemical
name and
concentration
to a 4 line
display

Selective Preconcentration of GD in High Background of Diesel



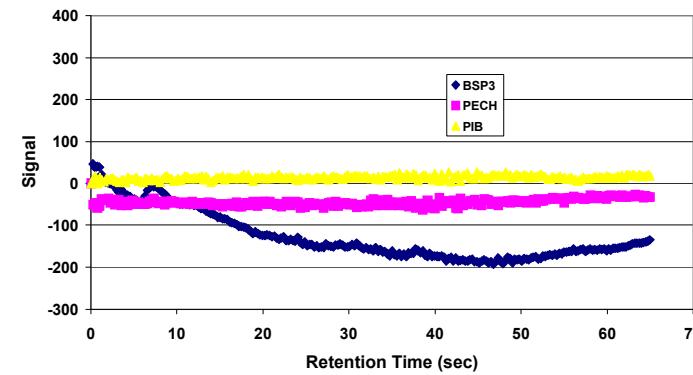
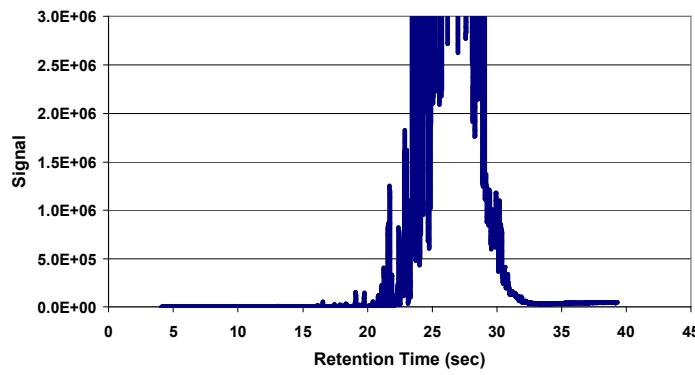
Two analyses overlaid. In one only GD is present, in the second the same concentration of GD is in a high background of diesel.

There is no change in response or retention time, the diesel is ignored by the front end preconcentrator material.

A special acknowledgement for Kwok Ong and the Applied Chemistry Team at ECBC for providing the test environment for this evaluation.



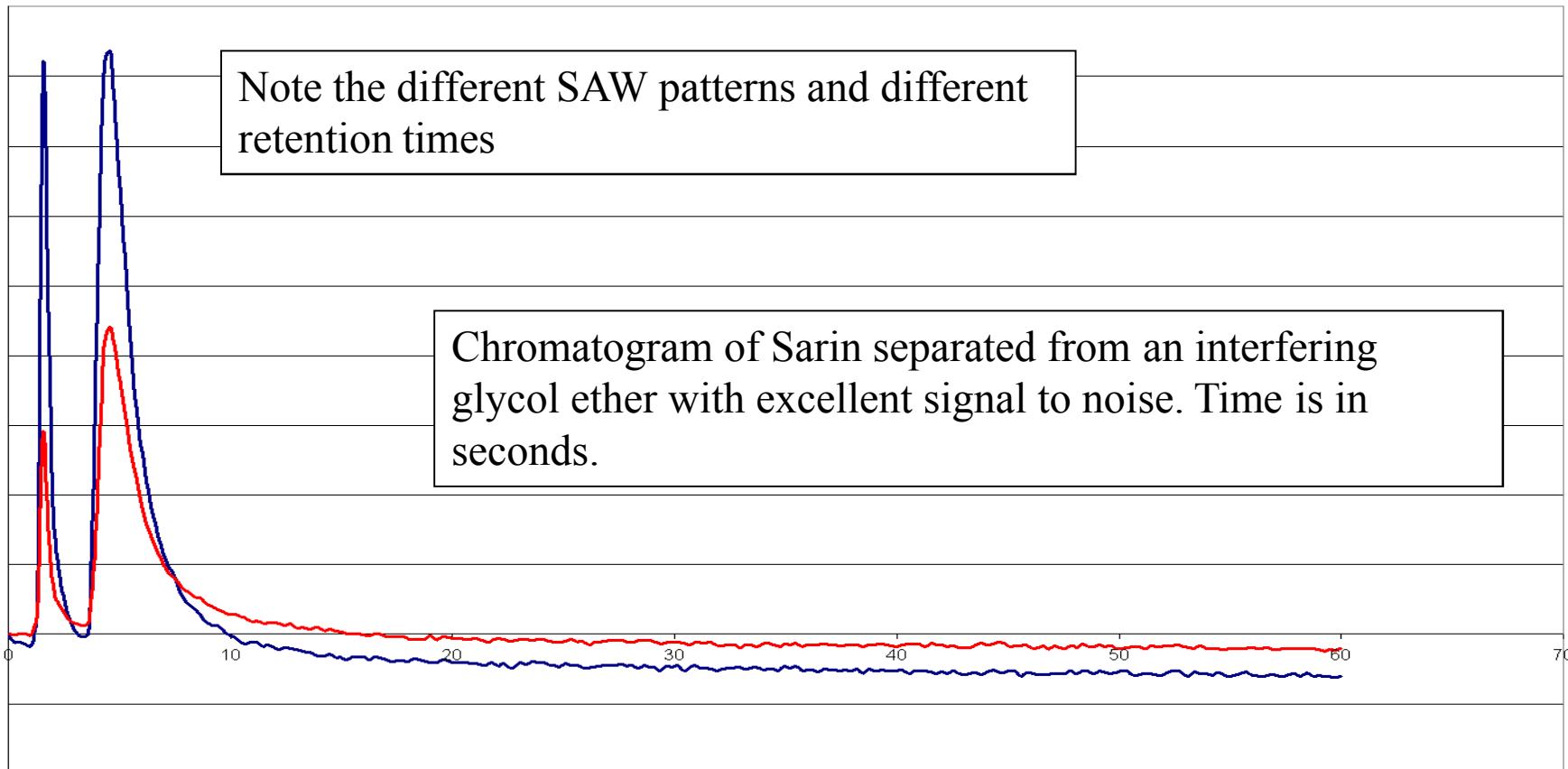
The μ ChemLab is insensitive to petrochemical (JP8) interferent



Thanks to SBCCOM and KAFB Refueling Shop

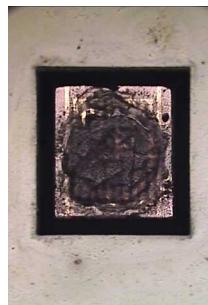


Separating Interferences Resolving Sarin and Diethylene Glycol Ethyl Ether



Micro Nitrogen-Phosphorus Detector Operating Principles

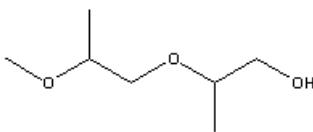
An example of one of the thermally isolated low power heated surfaces used as a basis for element specific detection



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.

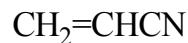
Unlike COTS NPDs, the Sandia μ NPD operates in both phosphorus and nitrogen specific modes without hydrogen gas – ideal for fieldable portable detection systems

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

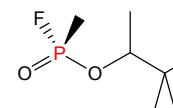


The signal is maximized for phosphorus or nitrogen containing compounds (e.g., nerve agents, many TICs).

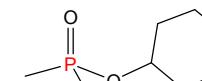
Typical TIC's



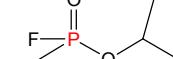
CWA's



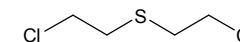
Soman-GD



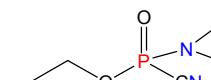
Cyclosarin-GF



Sarin-GB



Sulfur mustard-HD



Tabun-GA



Sandia μ NPD Preliminary Lab Prototype Results

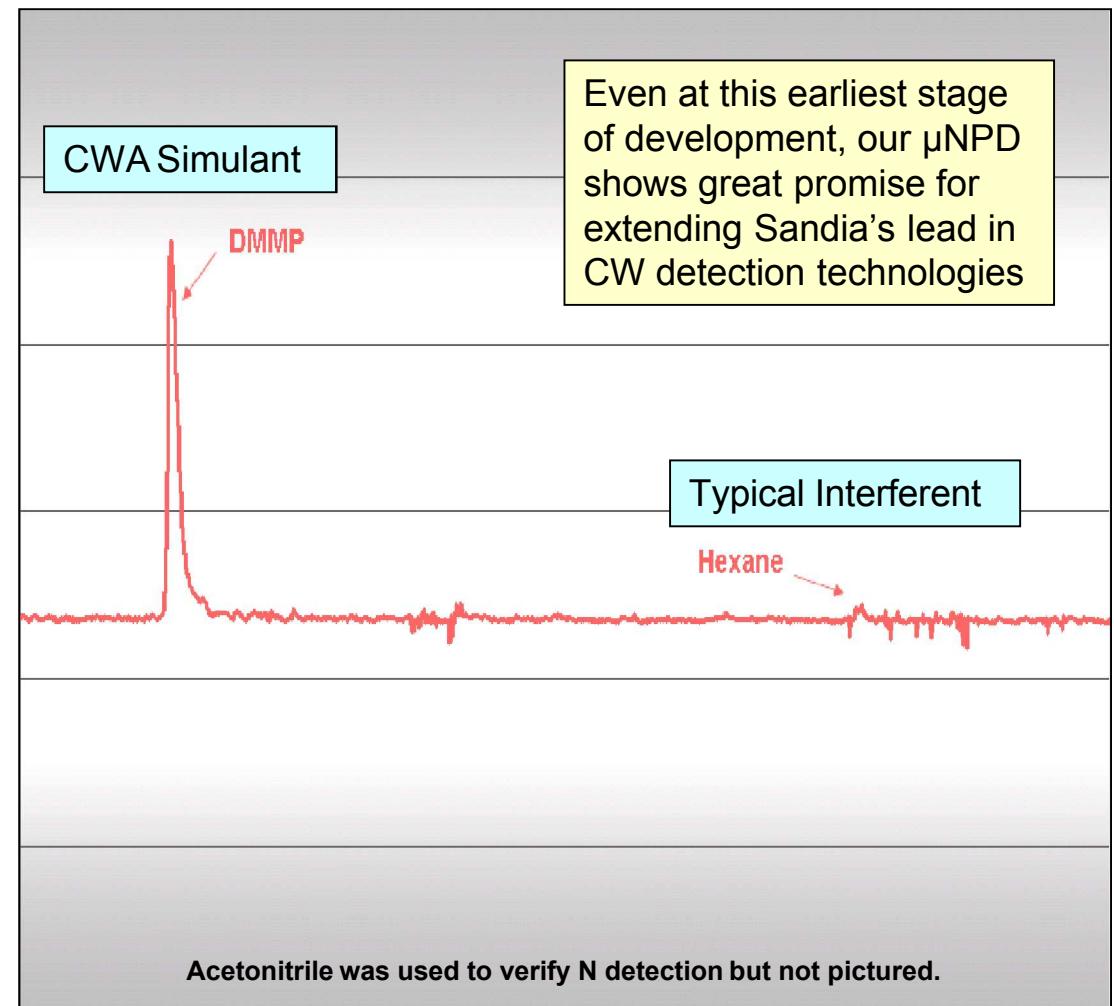
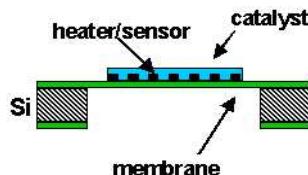
NPDs are capable of detecting N and P at a ratio of up to 100,000:1 over carbon with picogram sensitivity.

By comparison, our Sandia SAW detectors (RL6) have at best 100:1 relative sensitivity.

Uncoated



Coated





Conclusion

Sandia's Micro Chemical Analysis System

- Very sensitive and selective
- Low false alarm rate
- Can be designed for many different analytes
- Field Proven technology