

Micro Total Analytical Systems

Department Overview

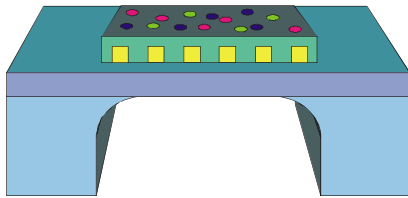
Patrick Lewis, Kamyar Rahimian, Richard
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Larry Stotts

prlewis@sandia.gov 505-284-3315

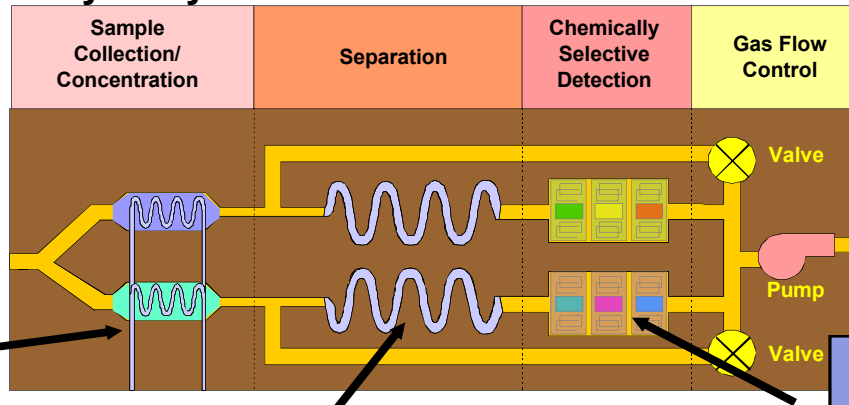
Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company,
for the United States Department of Energy's National Nuclear Security Administration
under contract DE-AC04-94AL85000.

Field Portable Chemical Analysis System

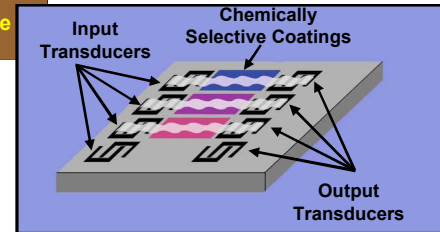
A hand-held chemical analysis system that uses three microfabricated analysis stages



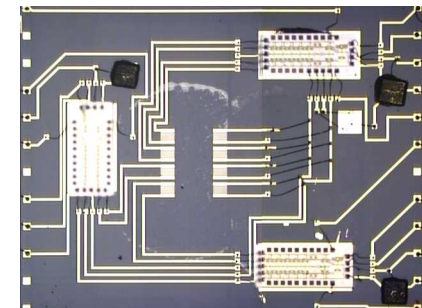
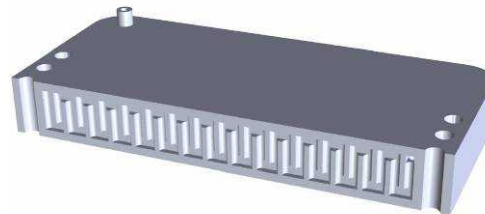
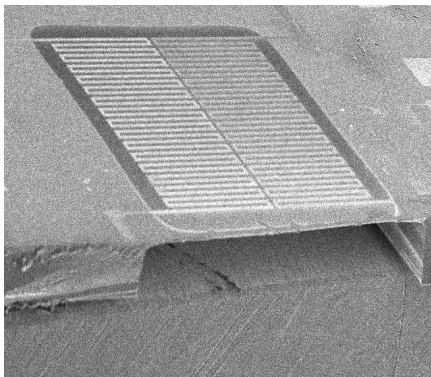
Preconcentrator accumulates species of interest



Gas Chromatograph separates species in time

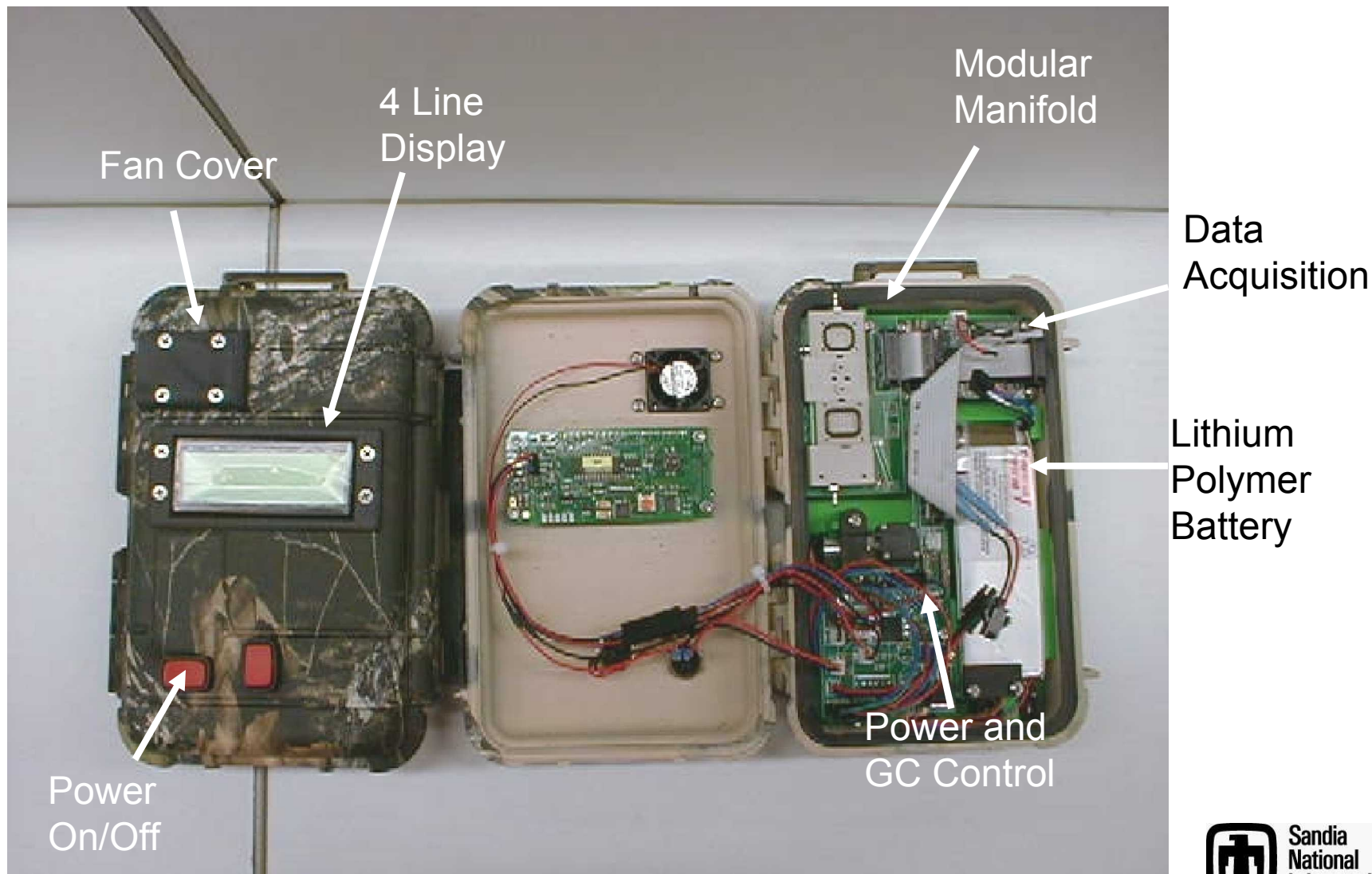


Acoustic Sensors provide sensitive detection



SAW Array

Handheld Chemical Analysis System



Micro Analytical System Components

Field
Tested

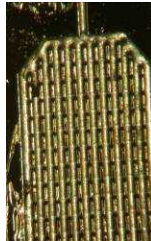
Technology Maturity Level

Requires
More
Development

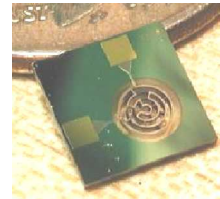
Sample Collection



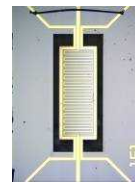
Planar PC
Sharp
sample
inject – most
mature



Tortuous
Path PC
better
retention
even for
VOCs

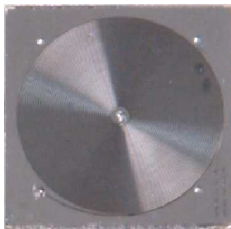


3d PC
Increased
surface
area and
improved
flow contact

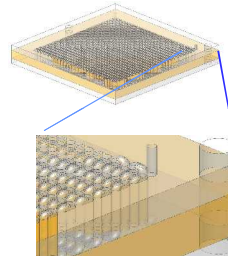


Smart PC –
enables intelligent
operation and
greatest dynamic
range (miosis to
IDLH)

Separation

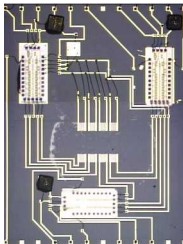


High aspect
ratio column
in silicon



Circular x-section
column made in Ni
using LIGA. Highest
resolution separation

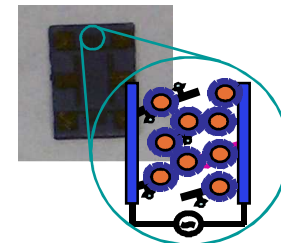
Detection



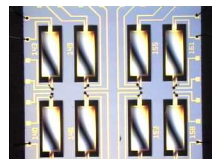
SAW Array
with integrated
electronics for
DC-in;DC-out
operation.



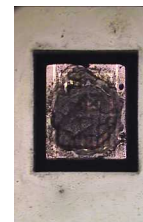
Micro Flame
Ionization
Detector →
“universal” GC
detector.



Organic / Nanoparticle
based detector for low
power high sensitivity
CW detection.



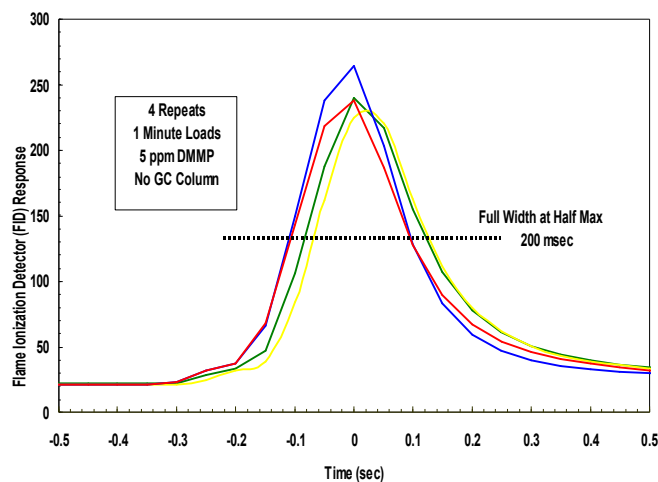
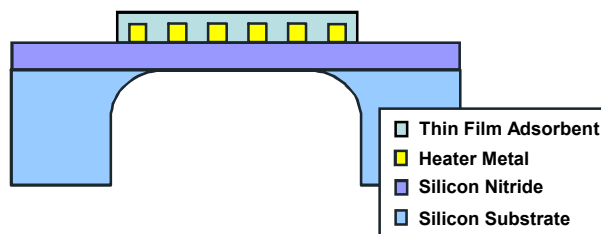
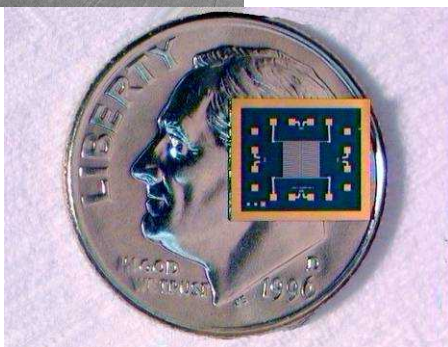
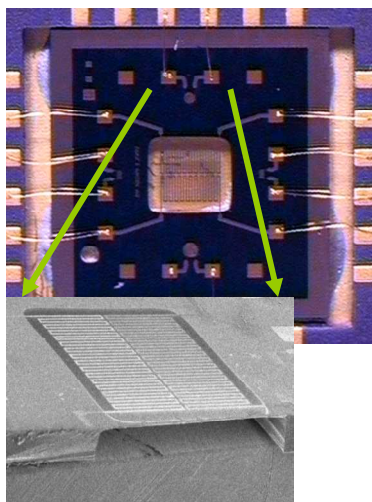
Pivot Plate
resonator for
high temp
applications.



Micro Nitrogen Phosphorus
detector the most sensitivity
with the greatest rejection of
interferences.

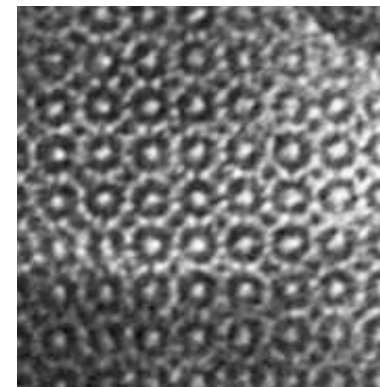
Preconcentrator

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

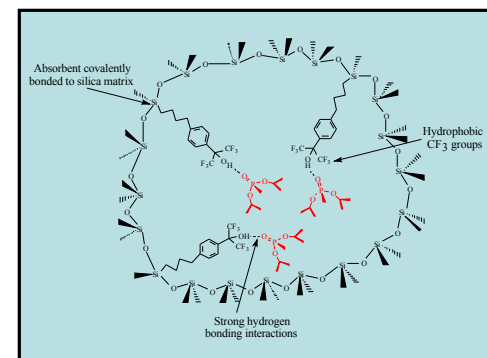


Rapid Thermal Desorption from Micromachined Preconcentrator

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

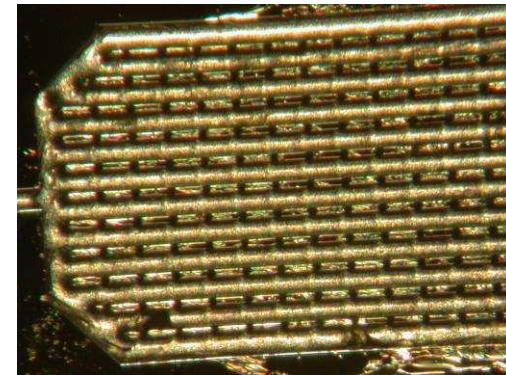
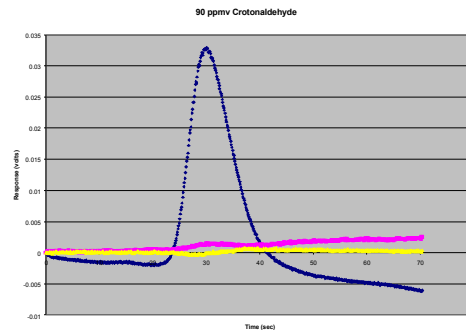
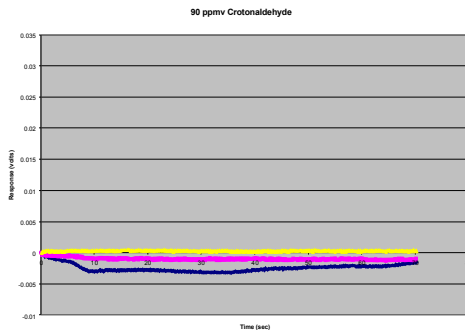
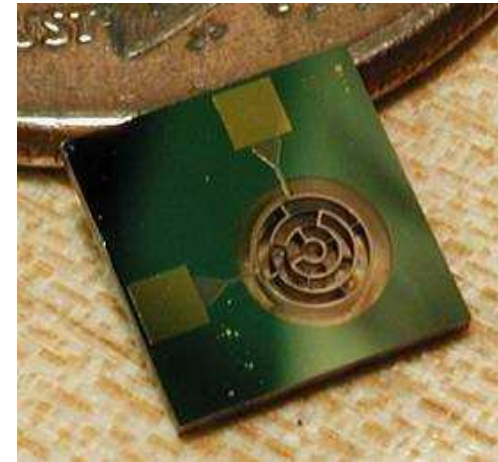
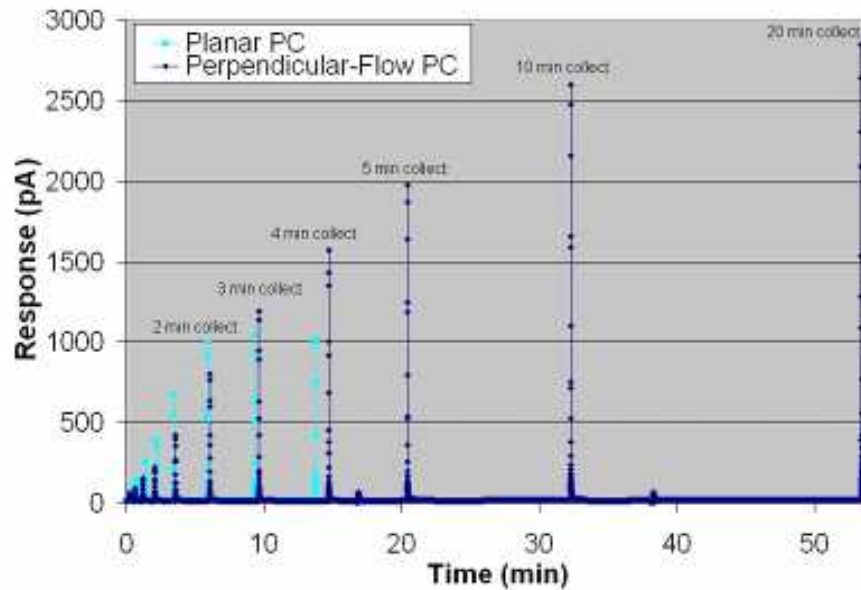


Tailored Porosity

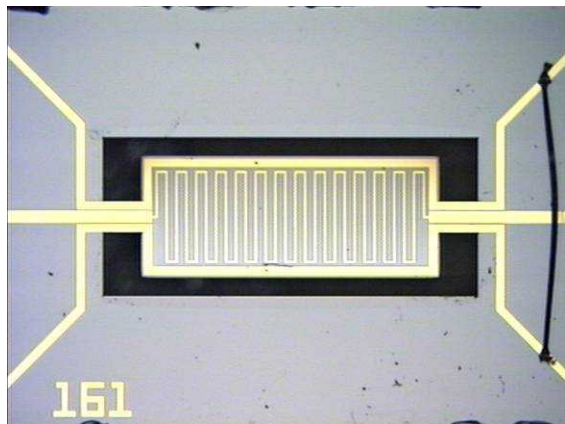


Tailored Surface Chemistry

High Capacity micro-Preconcentrators



SMART PC Tests

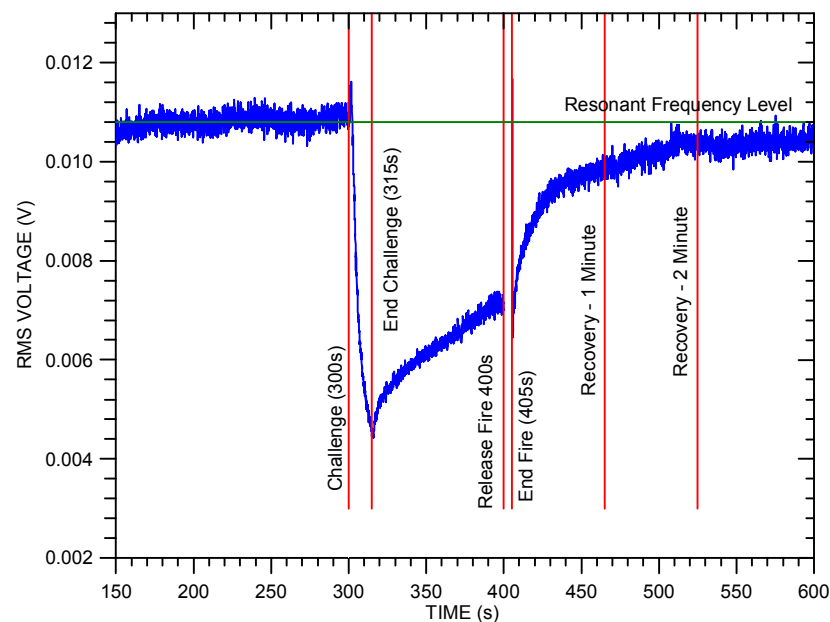


SMART PC tests

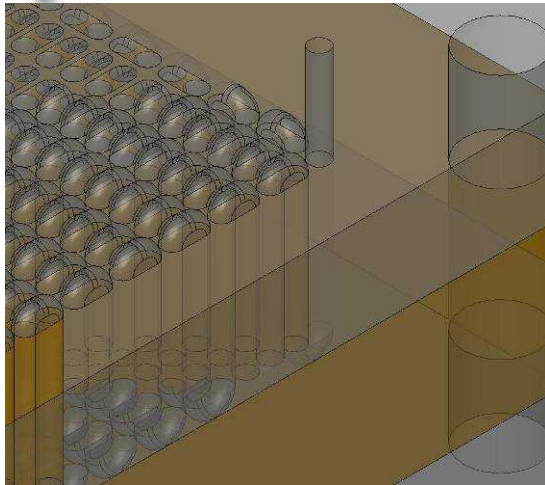
- Paddle at resonance, 11584 Hz
- DMMP challenge
- Challenge removed – 85s hold
- 5s fire on heater
- Recovery in ~120s

Tests on device TTH3-06

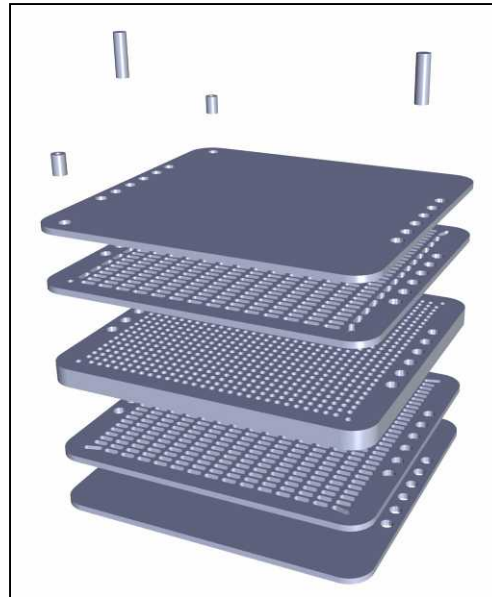
- Coated with 2 μg of Solgel
- Resonance at 11584 Hz
- 74 ohm heater



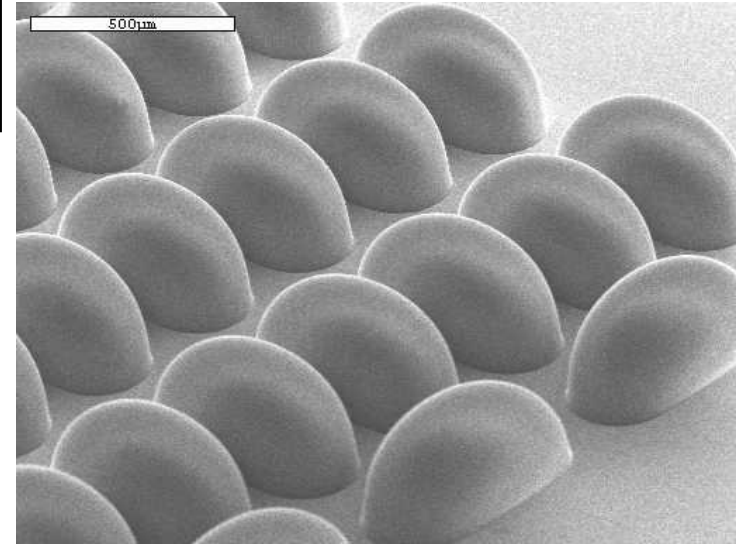
Nickel LIGA RTA Columns



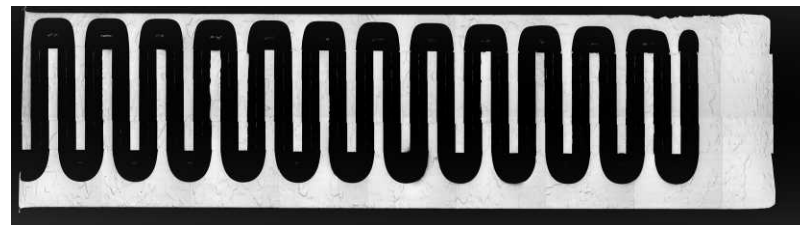
Exploded View of
LIGA fabricated GC



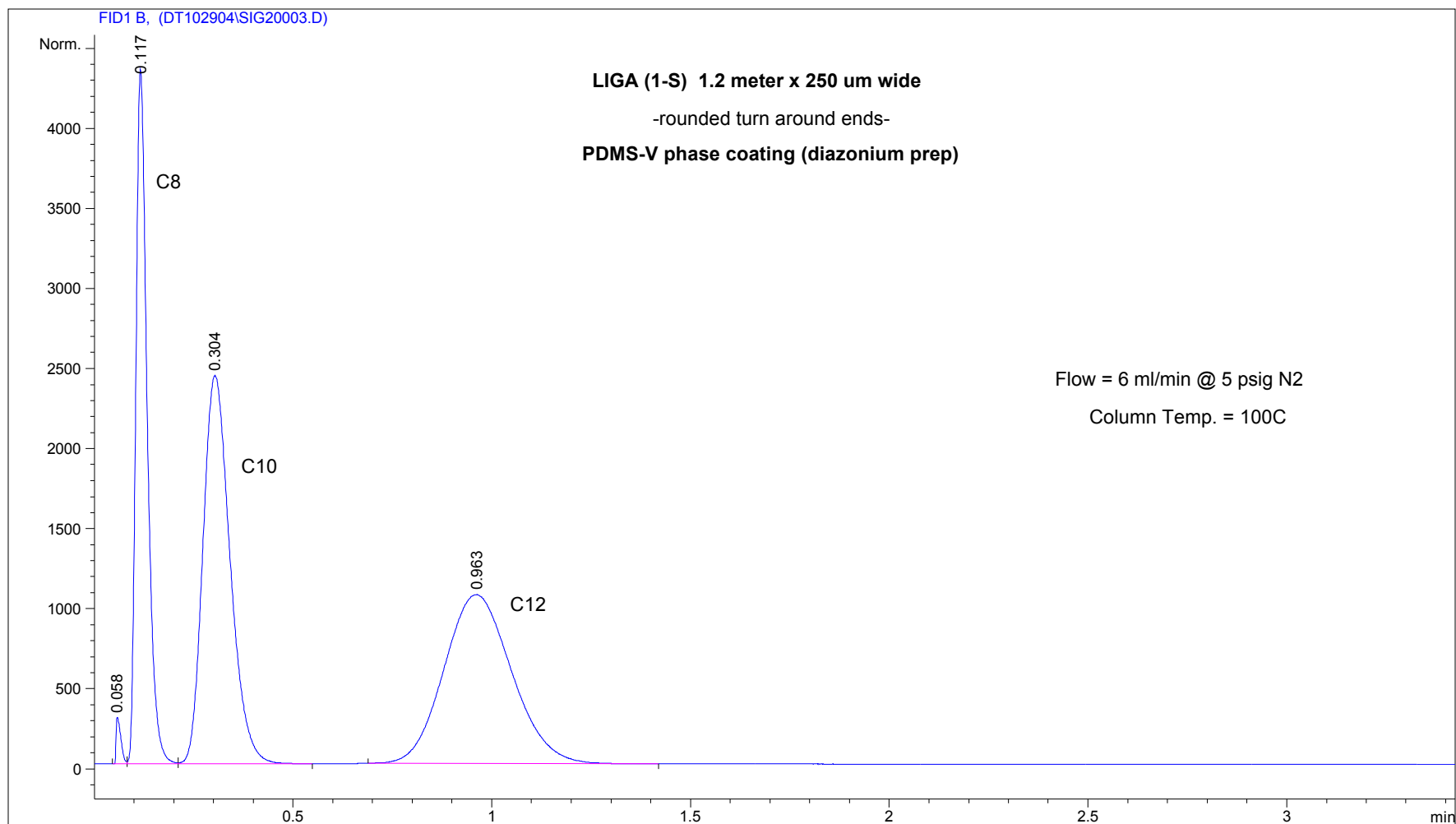
New Circular X-
Section GC Column
to minimize band
broadening. Longer
columns can be
made by stacking
center sections



LIGA Molds for Rounded
Turn Arounds.

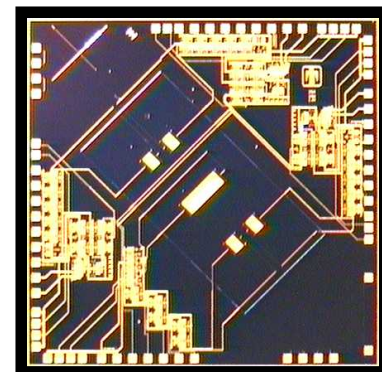
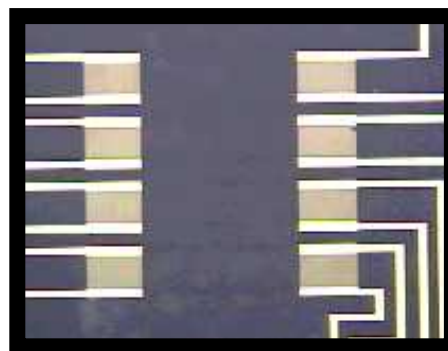
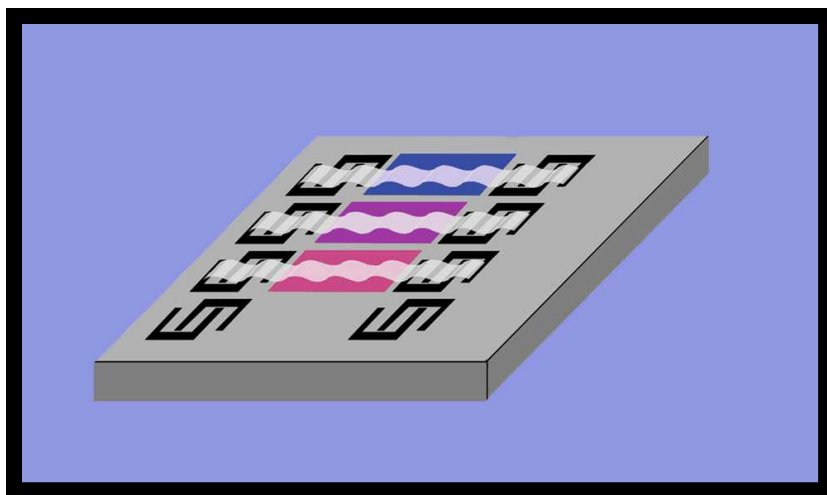
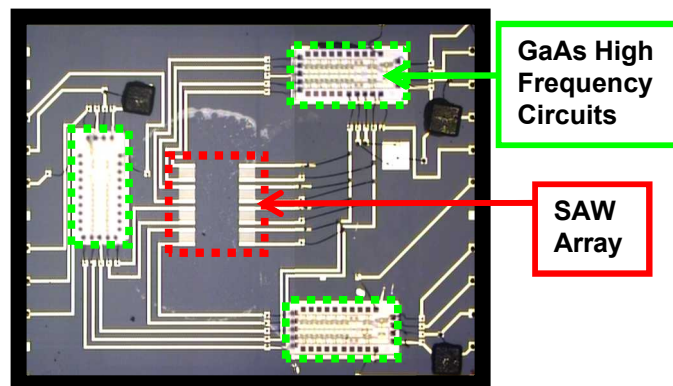


Homologous Series



Surface Acoustic Wave (SAW) Detector

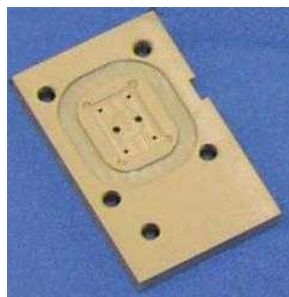
- Surface acoustic wave is excited/detected using interdigital transducers on a piezoelectric substrate
- Sensor coating momentarily absorbs analytes eluted from GC column, changing SAW velocity (phase shift).
- Pattern of responses from array augments discrimination of GC separation



Modular Manifold for Gas Components

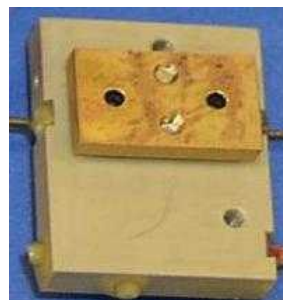
- Modules can operate in parallel to maximize analytes and minimize time
- Different modules can be added to change detector or preconcentrator
- Easy to replace components to facilitate testing different films
- Each piece makes electrical and fluidic connection

Preconcentrator manifold



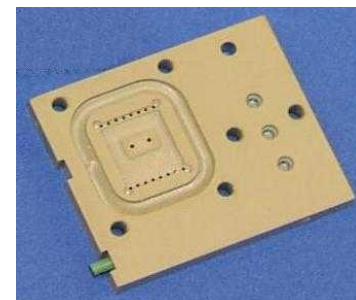
+

GC Column manifold

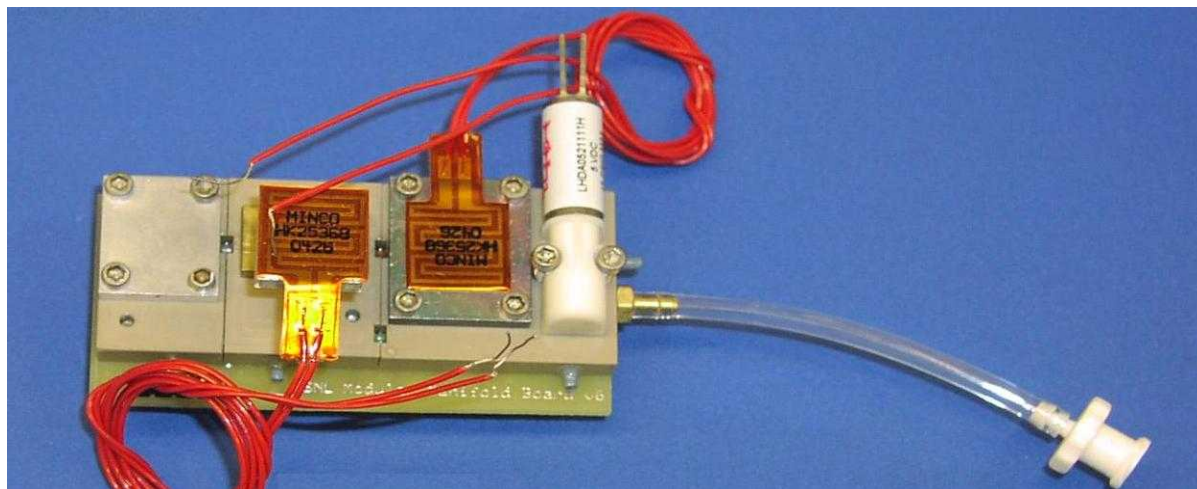


+

SAW Array Manifold

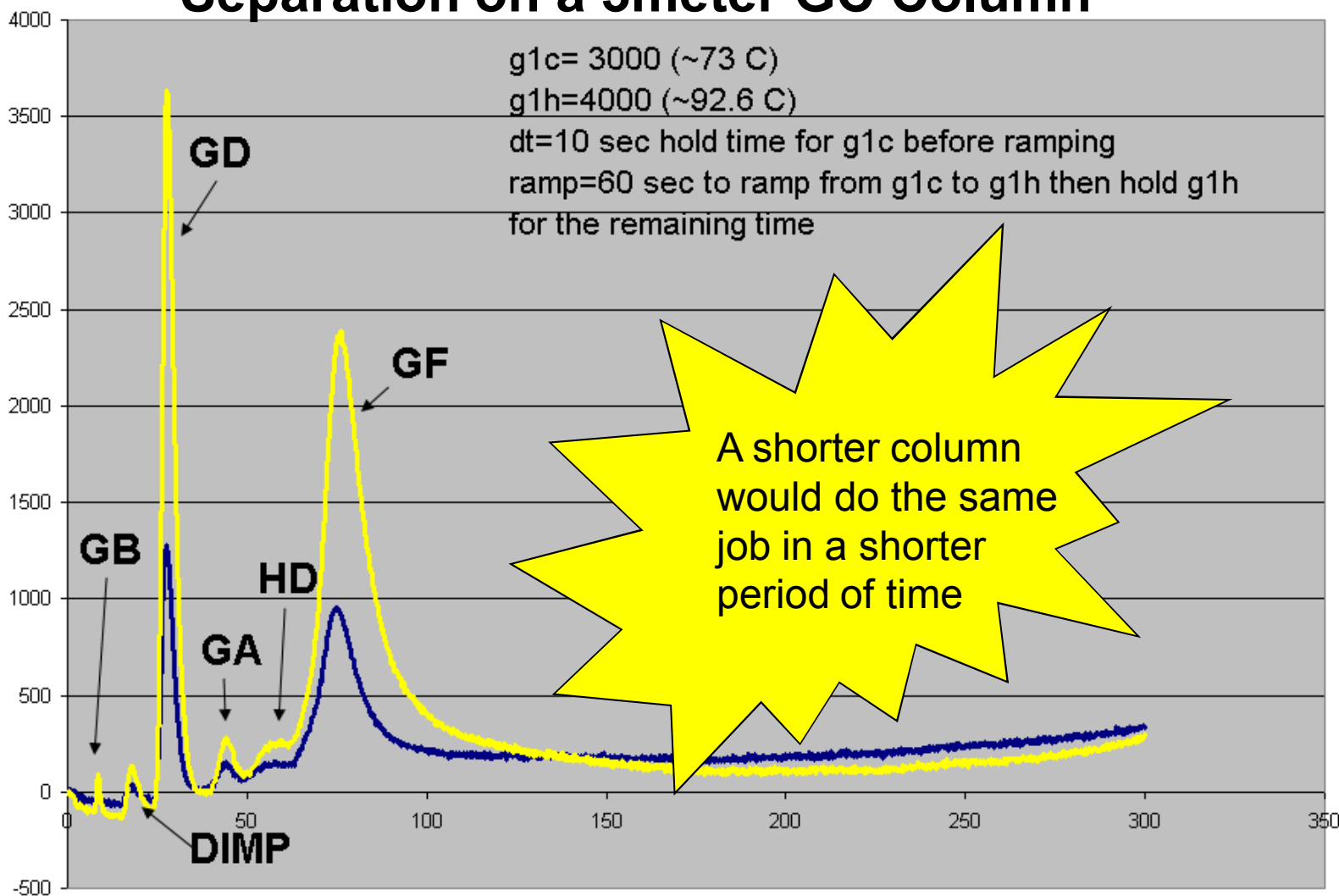


Adding modules together results in an easy to use manifold

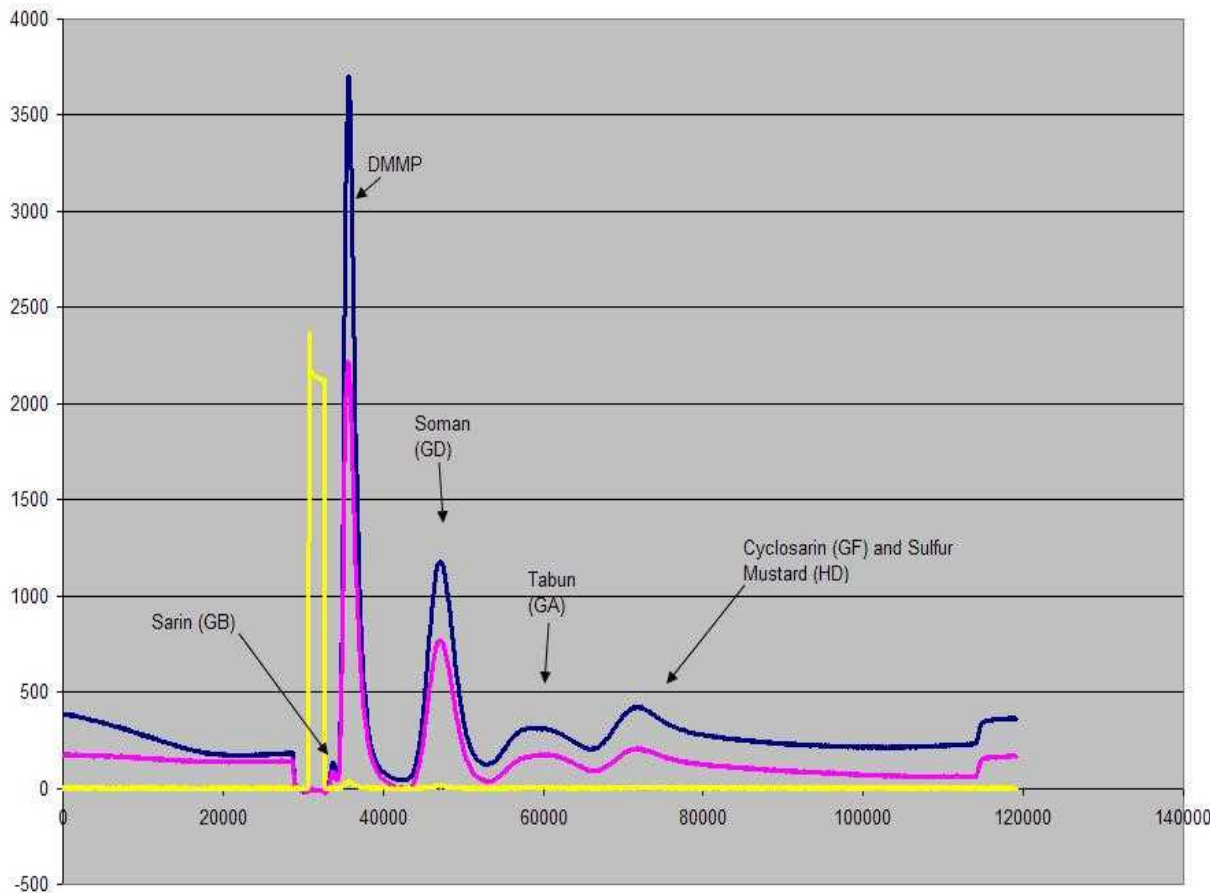


CW Agent, Byproduct, and Blister Agent Separation on a 3meter GC Column

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

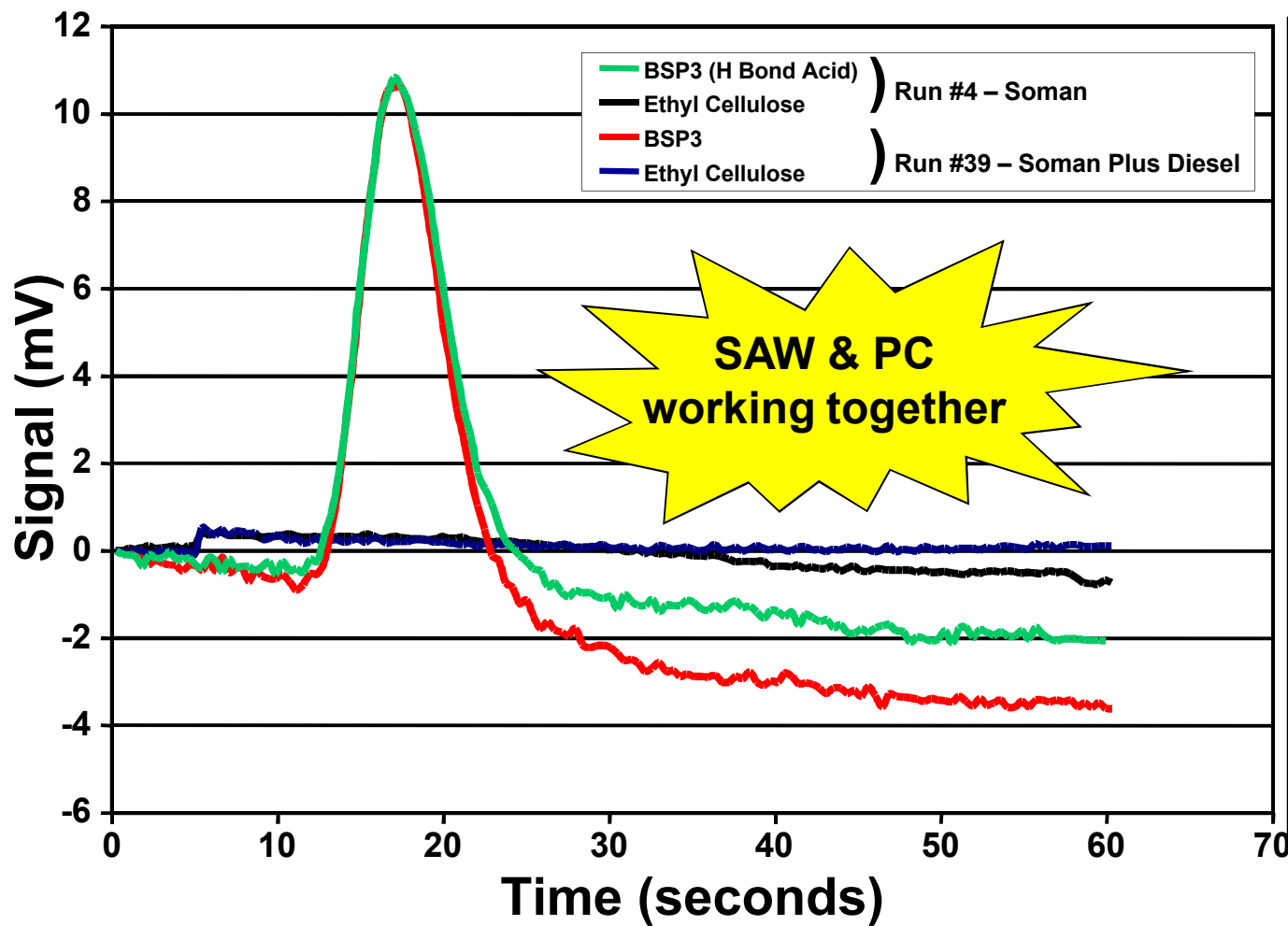


Handheld System Separating 5 Target Chemicals



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

Selective Preconcentration of GD in High Background of Diesel

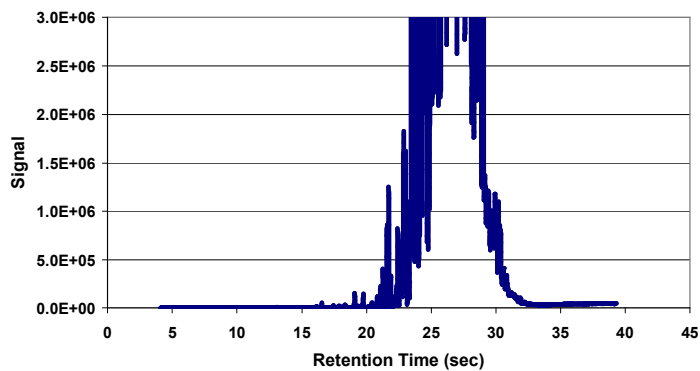


Two analyses overlaid. In one only GD is present, in the second the same concentration of GD is in a high background of diesel.

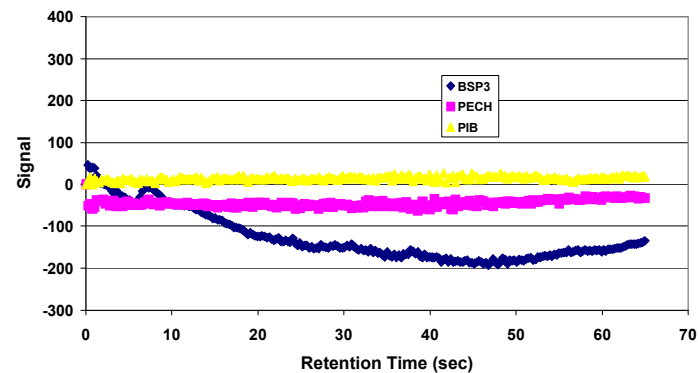
There is no change in response or retention time, the diesel is ignored by the front end preconcentrator material.

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



TD/GC/MS from Shop Floor

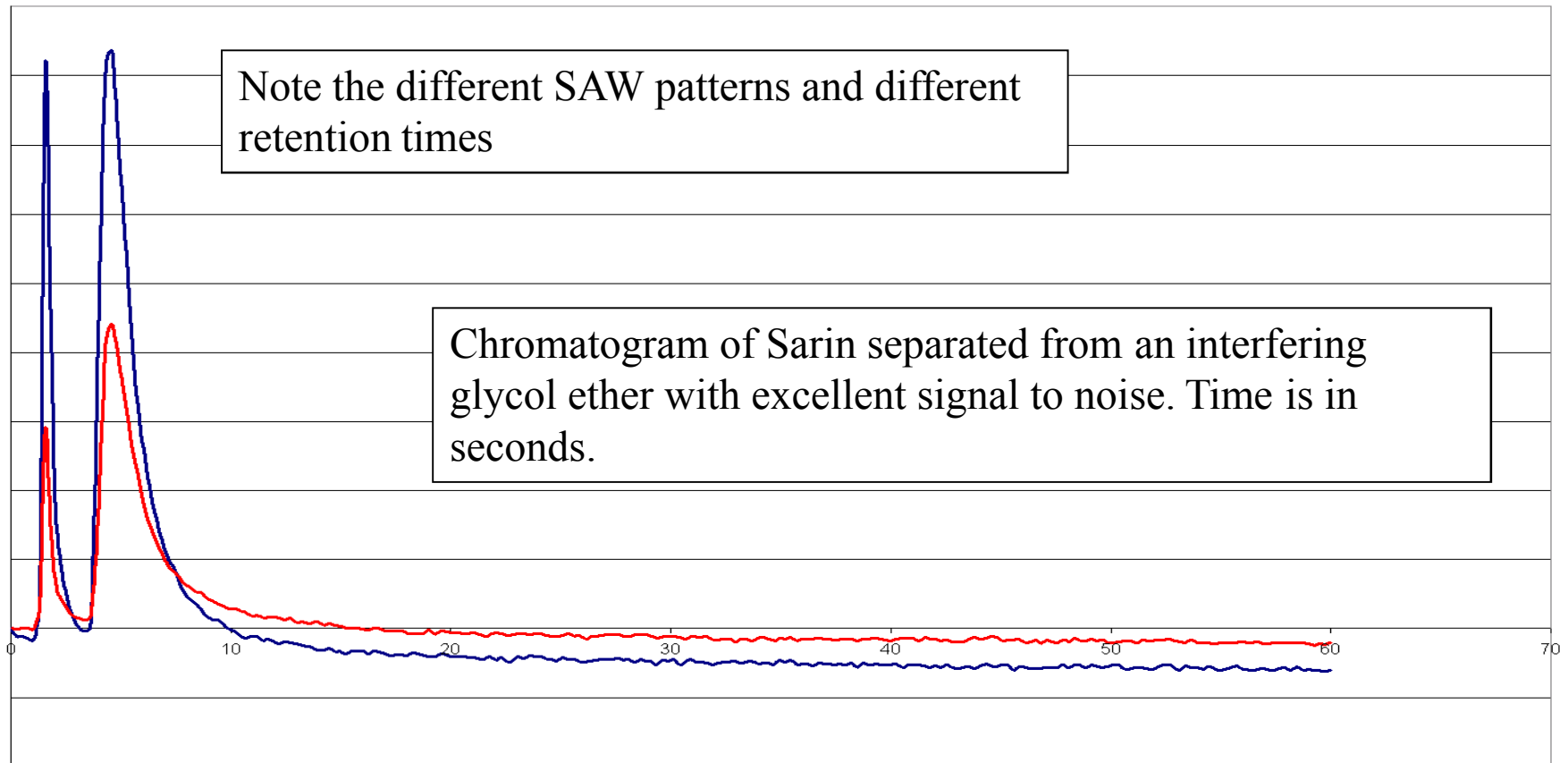


μ ChemLab from Shop Floor

Thanks to SBCCOM and KAFB Refueling Shop

Separating Interferences

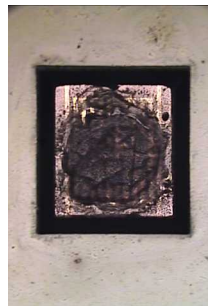
Resolving Sarin and Diethylene Glycol Ethyl Ether



Micro Nitrogen-Phosphorus Detector Operating Principles



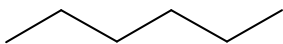
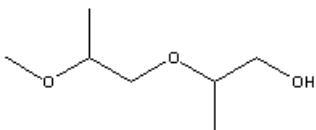
An example of one of the thermally isolated low power heated surfaces used as a basis for element specific detection



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.

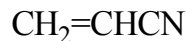
Unlike COTS NPDs, the Sandia μ NPD operates in both phosphorus and nitrogen specific modes without hydrogen gas – ideal for fieldable portable detection systems

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

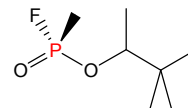


The signal is maximized for phosphorus or nitrogen containing compounds (e.g., nerve agents, many TICs).

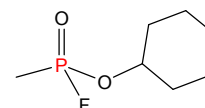
Typical TIC's



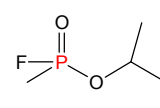
CWA's



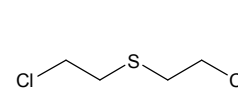
Soman-GD



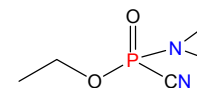
Cyclosarin-GF



Sarin-GB



Sulfur mustard-HD



Tabun-GA

Sandia μ NPD Preliminary Lab Prototype Results

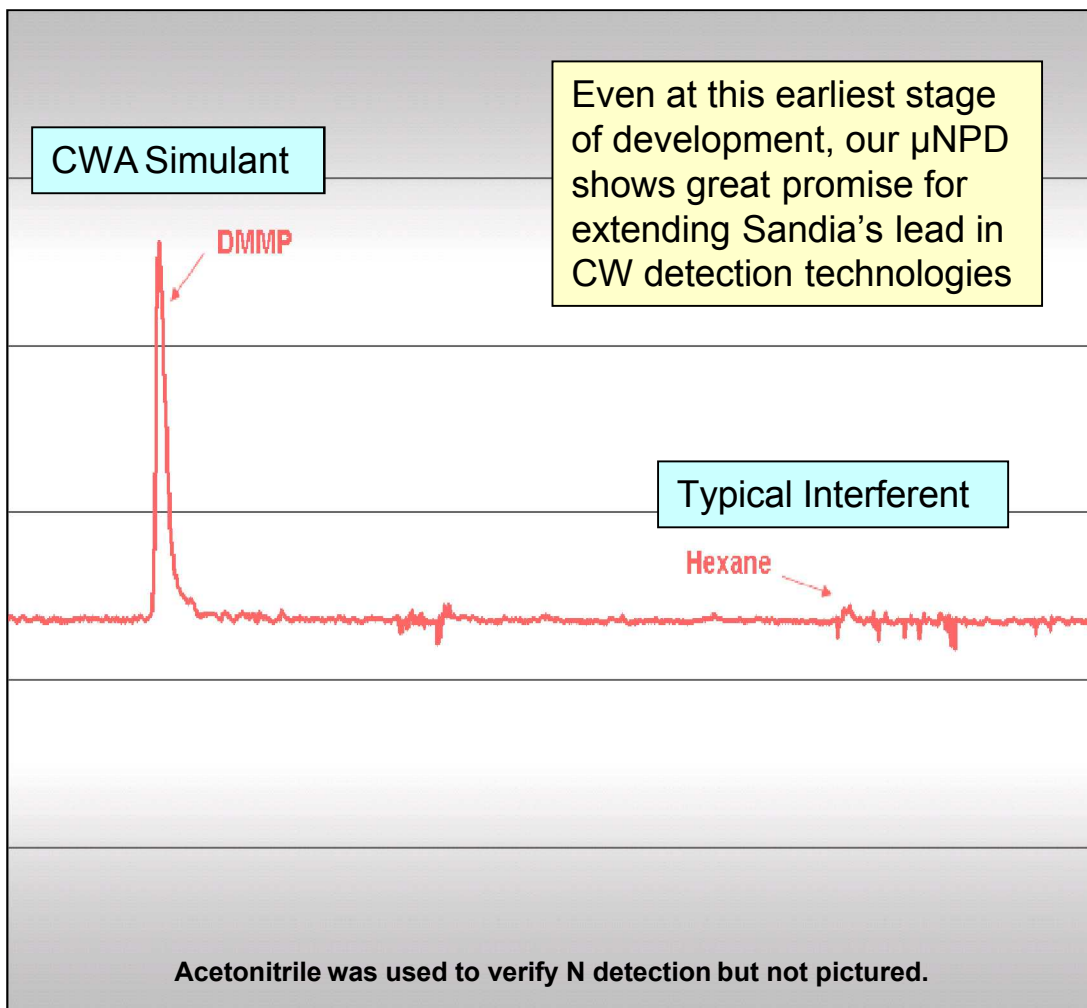
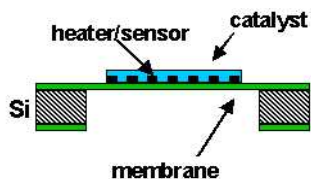
NPDs are capable of detecting N and P at a ratio of up to 100,000:1 over carbon with picogram sensitivity.


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