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4th Microsensors Workshop
Albuquerque, NM, 10/17/06

Microfabricated Preconcentrators for Portable Chemical Analysis Systems

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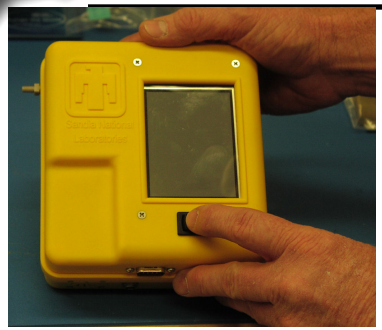
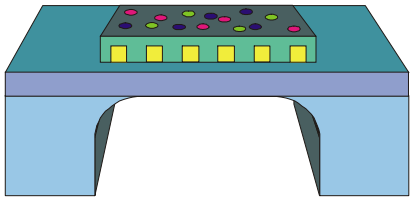
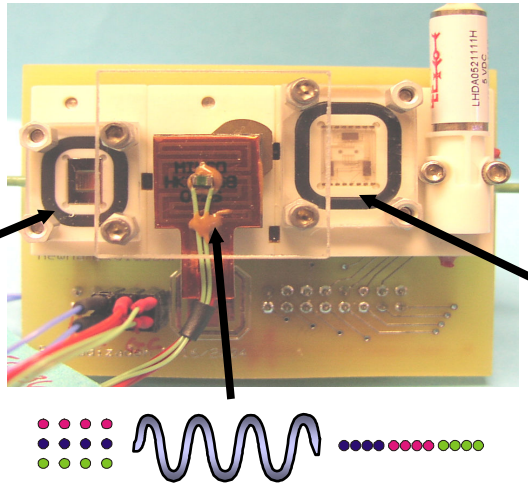
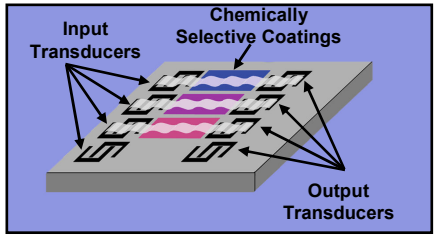
Sandia's MicroChemLab™

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





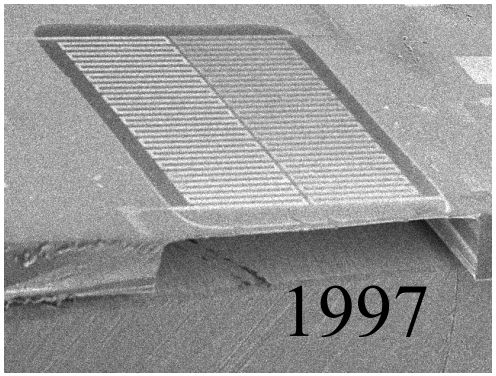


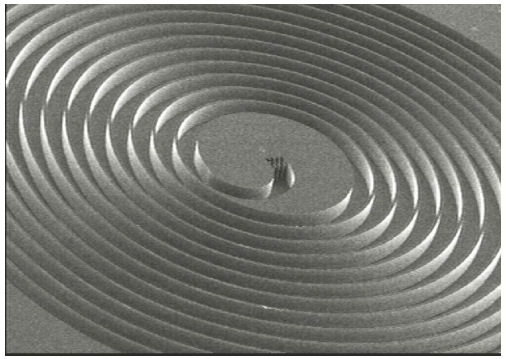


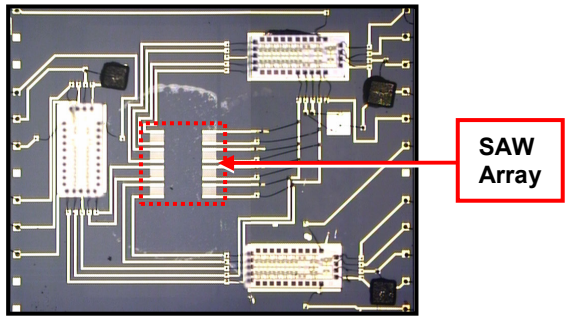
Preconcentrator accumulates analytes of interest

Gas Chromatograph separates analytes in time

Acoustic Sensors provide sensitive detection







US Patents: 6,171,378, 6,527,835

IEEE Sensors Journal, 6 (3) 784-795, 2006.

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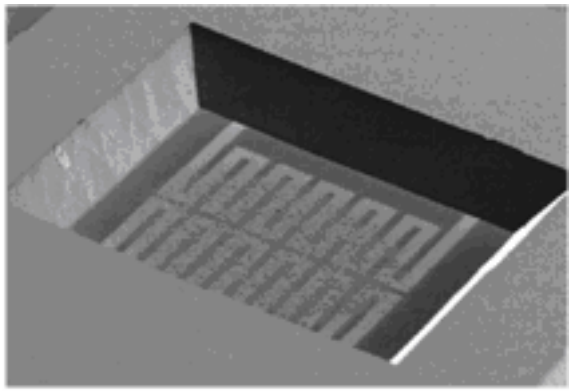
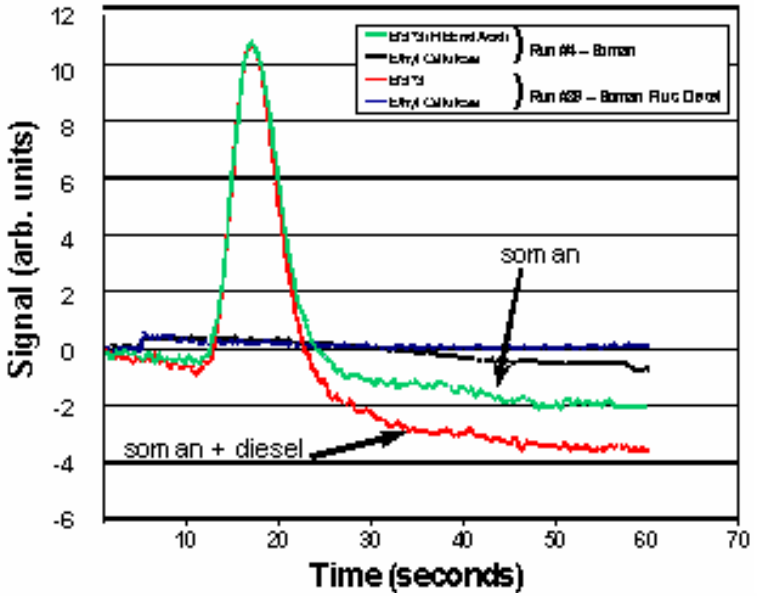
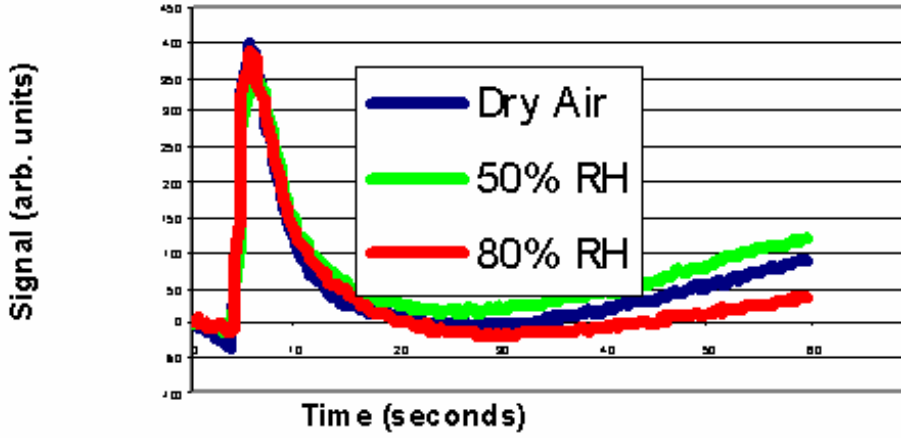
Planar MicroFabricated Preconcentrators

- Low C, high efficiency adsorbent platform
  - 2000°C/W; 10msec ramp
- Minimal flow restriction
  - 5 psig, 200 mL/min
- Concentrate targets
- Reject interferants
- Rapid release - a non-mechanical GC injector
- Bosch or KOH etched to SiN







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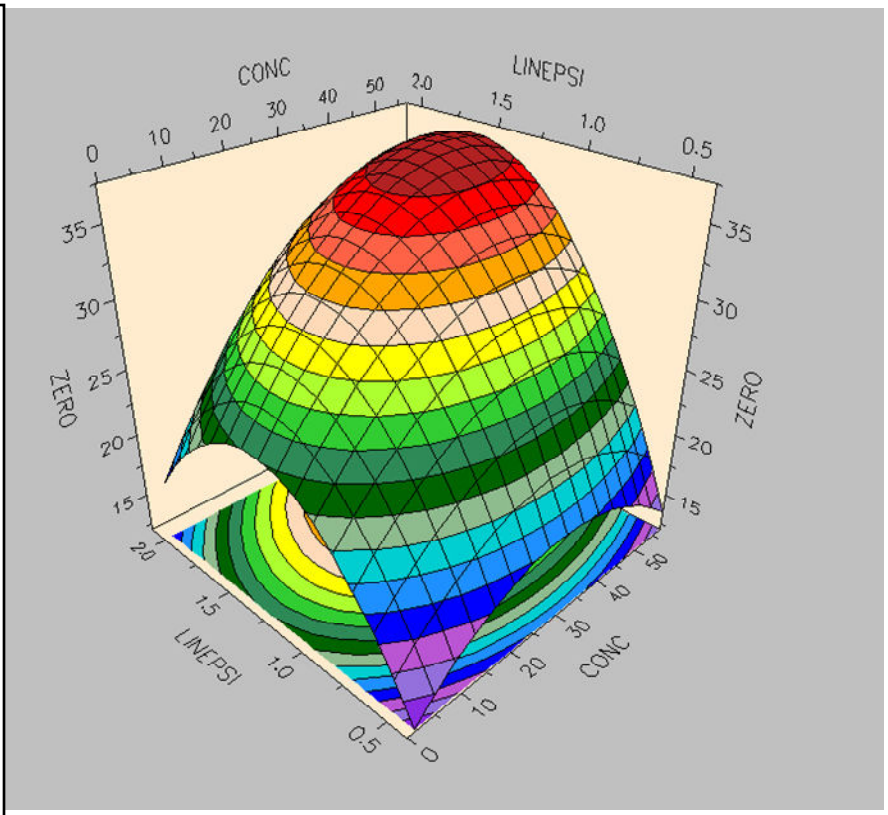
CFD Modeling and DOE

DOE

- Carboxen 1000 and light hydrocarbon
- Varied [C], collection time (tc), desorption flow (f), temperature (T) and desorption time (td)
- GC/FID Agilent 6890
- Statistica - full quadratic

Conclusions

- Peak area, Ap, increases with [C]
- Maximum in Ap with f
- Peak width, W, is not influenced by [C]
- W decreases with f
- Max in W with T
  - heated area increase, degradation
- Increased tailing with T
- Pe ~ convection/diffusion increases with f, [C] and decreases with T



R. J. Simonson, et. al., "Optimization of a Microfabricated Planar Preconcentrator," Proceedings of the 2nd Joint Conference on Point Detection for Chemical and Biological Defense, Williamsburg, VA 3/1-5/2004, Manuscript K1.

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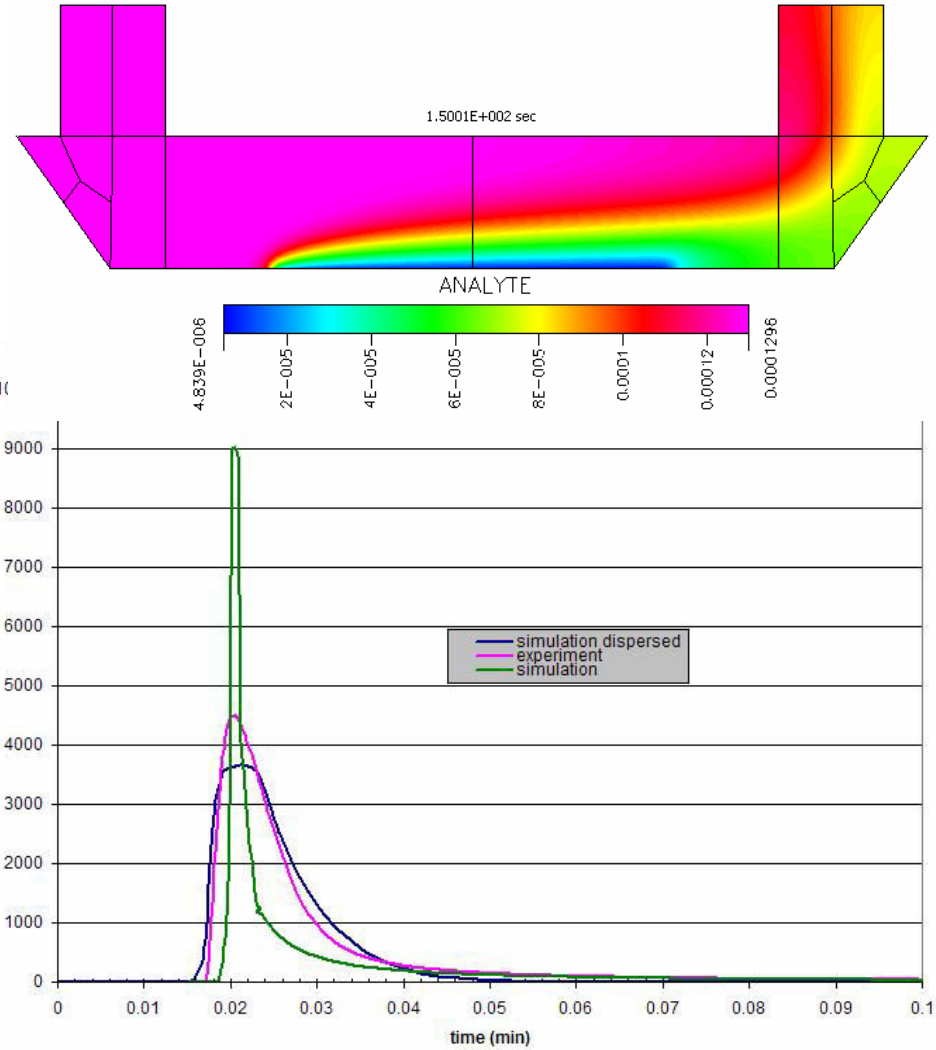
CFD Modeling (ESI Group) & DOE

- Computational Fluid Dynamics
- 2D – flow and adsorbent scaled
- Simplified thermal model
- Unity sticking
- Calibration on DOE
- Adsorption: A + s → A(s)
  - k = 36,500 s-1; 25,300 s-1 from Modified-Wheeler
- Desorption: A(s) → A + s
  - first-order Arhennius 30.1 kJ/mol
- Can predict other DOE runs
- Aris-Taylor Diffusion

- Diffusion is a dominant effect

- Did not predict fall off in Ap with f
  - Quadratic or simplicity of model; turbulence not an issue



R.P. Manginell, Sekhar Radharishnan, et. al., "Two-dimensional modeling & simulation of mass transport in microfabricated preconcentrators", submitted IEEE Sensors Journal.

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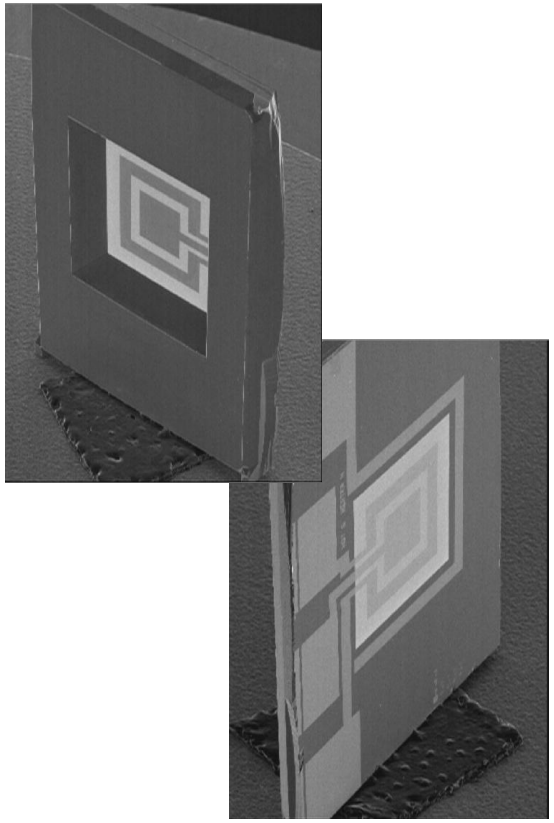
3DPCs as a supplement or replacement for the planar PC

Planar PC

1. Low C, high thermal efficiency
2. Fast response, low power
3. Collection limitations

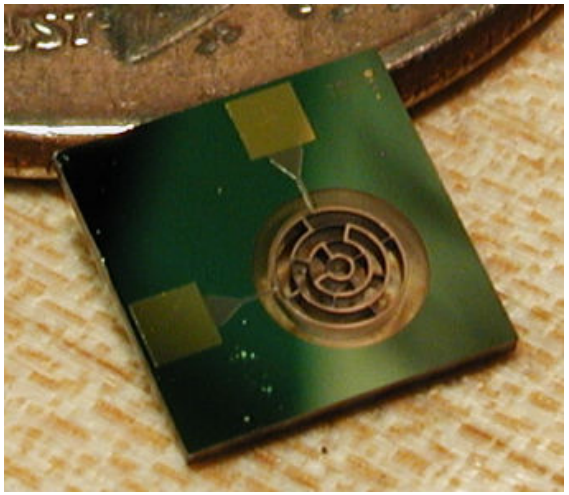
3DPCs

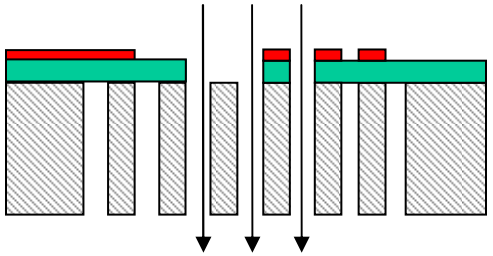
1. Planar PC items 1 & 2 retained
2. Smaller diffusion length, higher area, flow through
3. Pressure balance possible



Improved collection performance

More analytes like volatile organic compounds (VOC)





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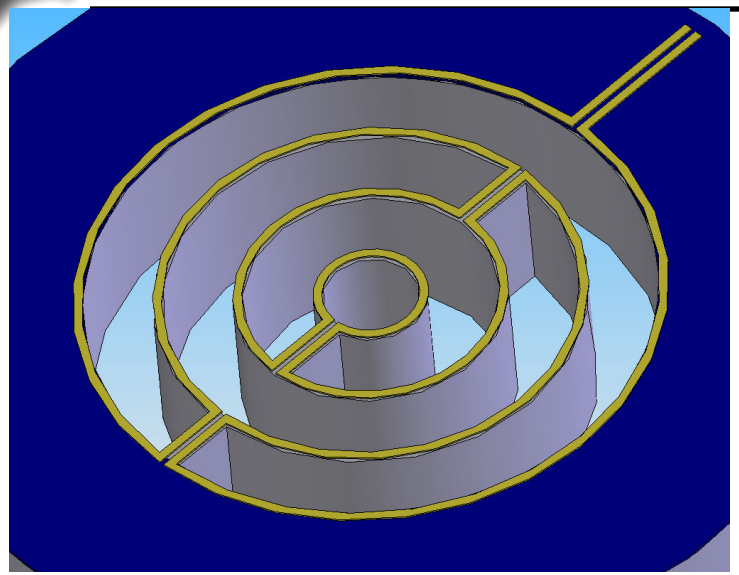
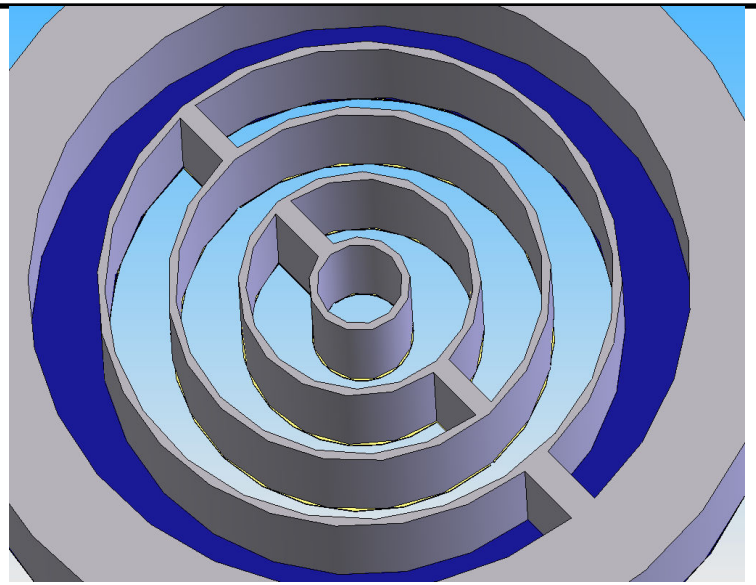
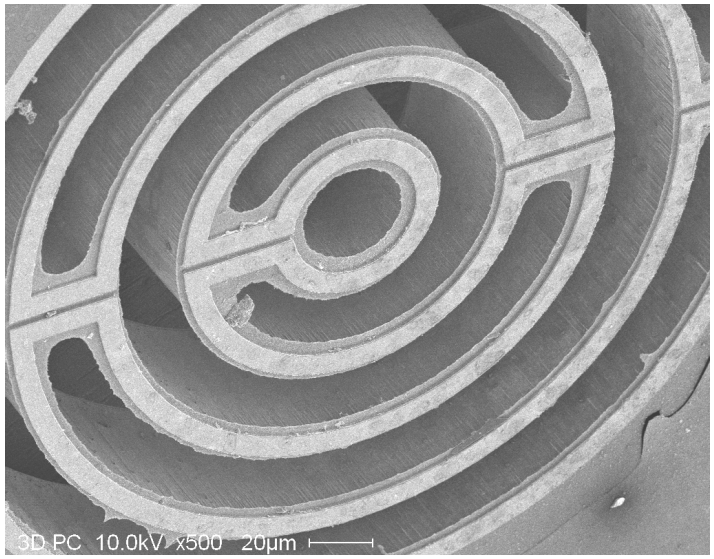
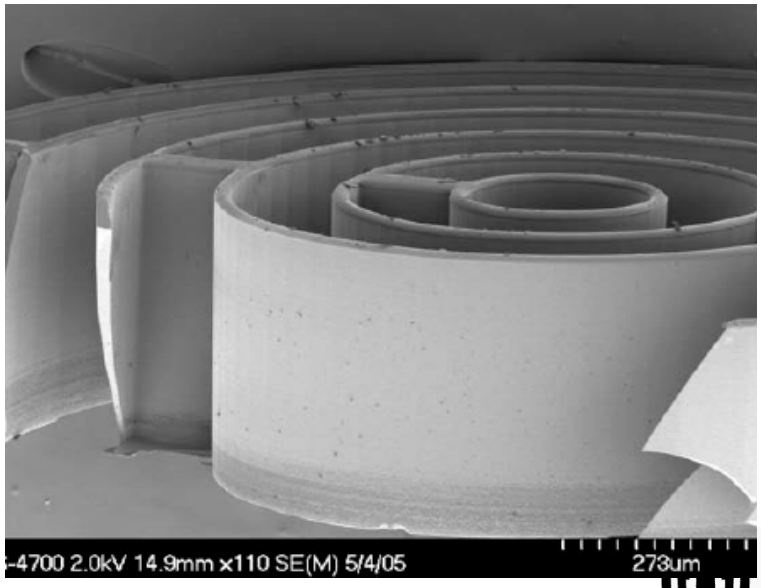
Types of 3DPCs: perpendicular flow. Etching thanks to ITC.









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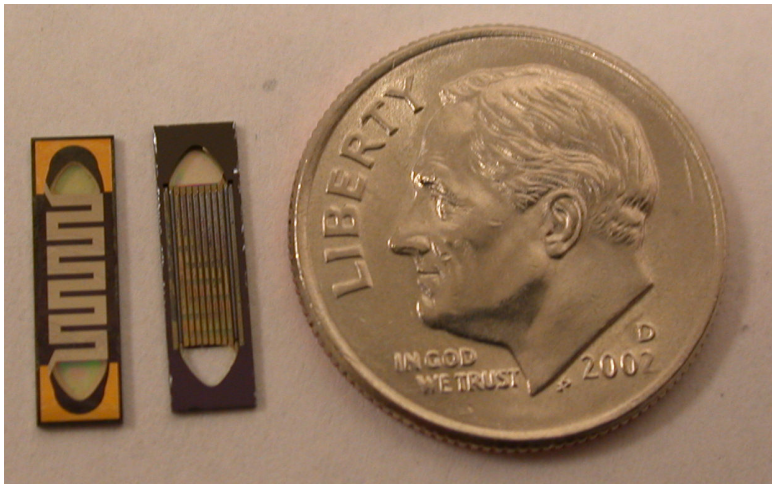
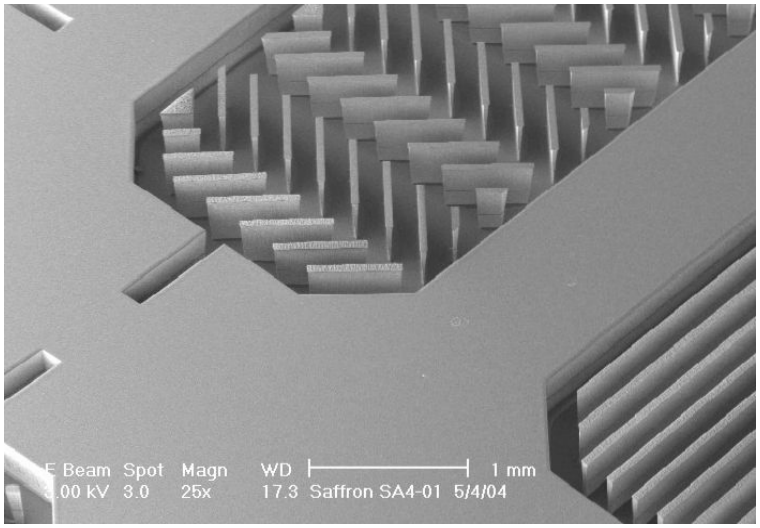
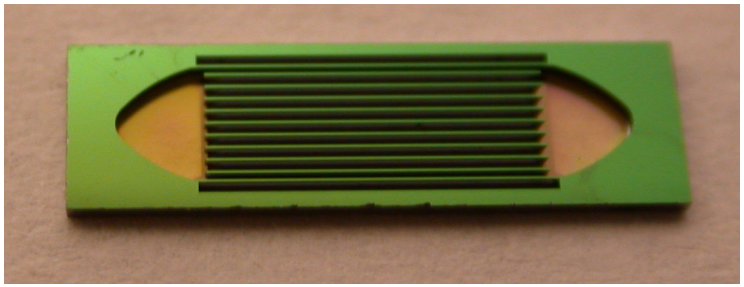
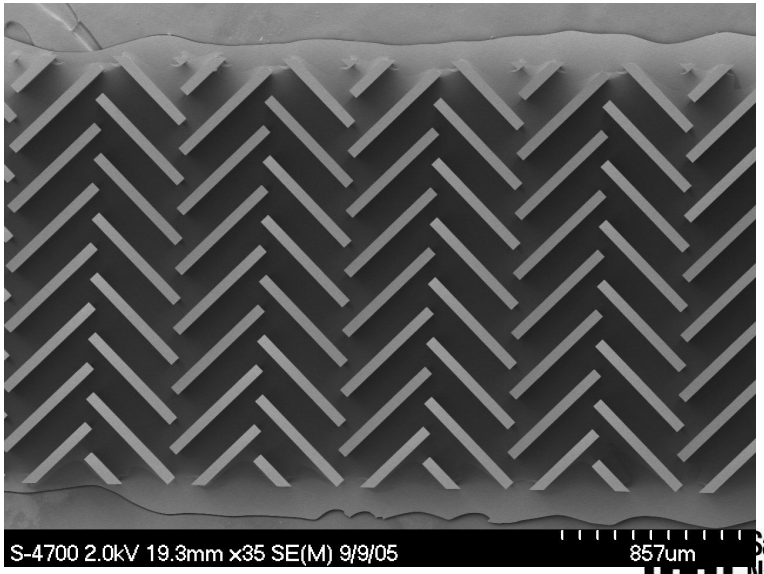
3DPC: parallel flow and tortuous path









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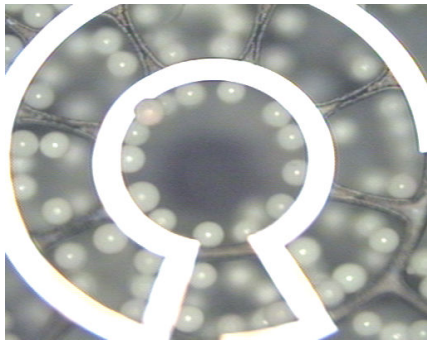
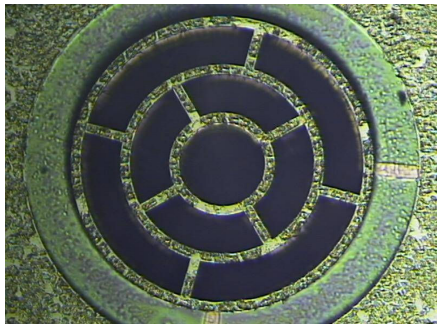
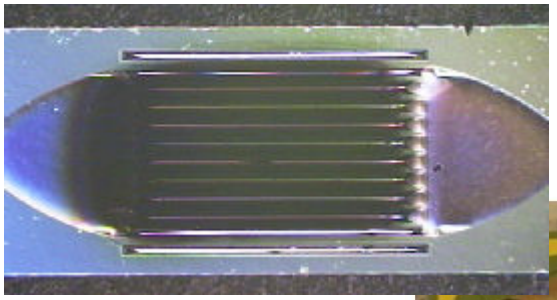
3DPCs, coatings and target analytes: enhanced collection

- **Spray and drop coating of sols**
  - CW agents, TICs
  - Explosives – usually need tortuous 3DPC
  - Automated spray with tilt
- **Commercial packing in PDMS binder OR using packing stops**
  - Toxic Industrial Chemicals (TICs) and Tri-Halomethanes (THMs)
  - PoropakQ, HayesepA, Carboxen
- **Laser ablation of nanoporous carbon**
  - Conformal coating; TICs

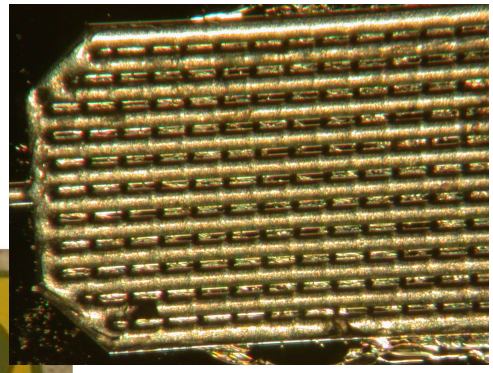




"Perpendicular flow"



"Parallel flow"



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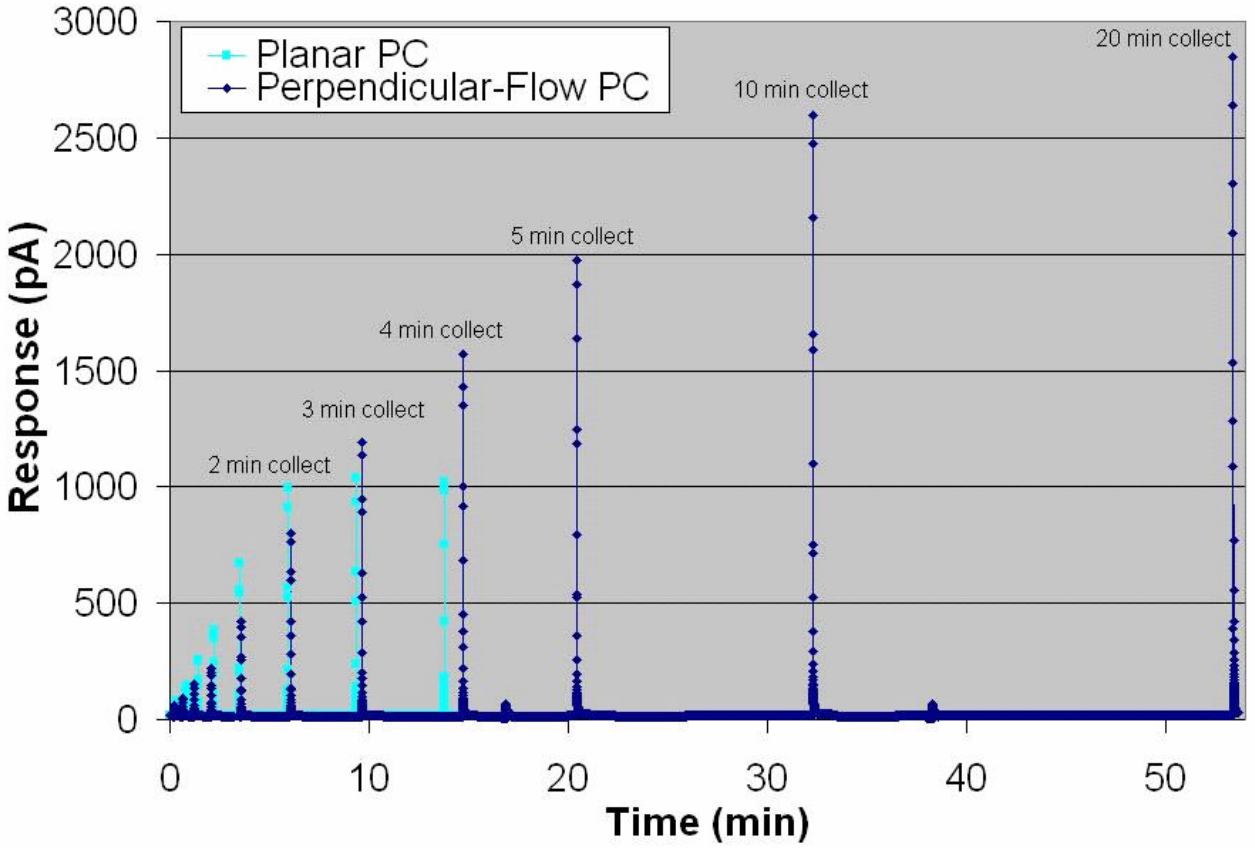
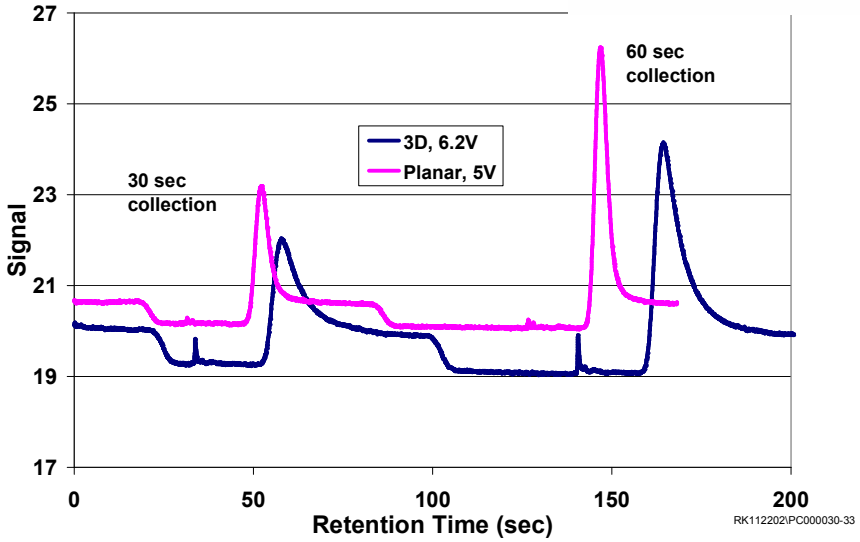
3D design aids DMMP collection and release





Thermal uniformity, improved mass transfer

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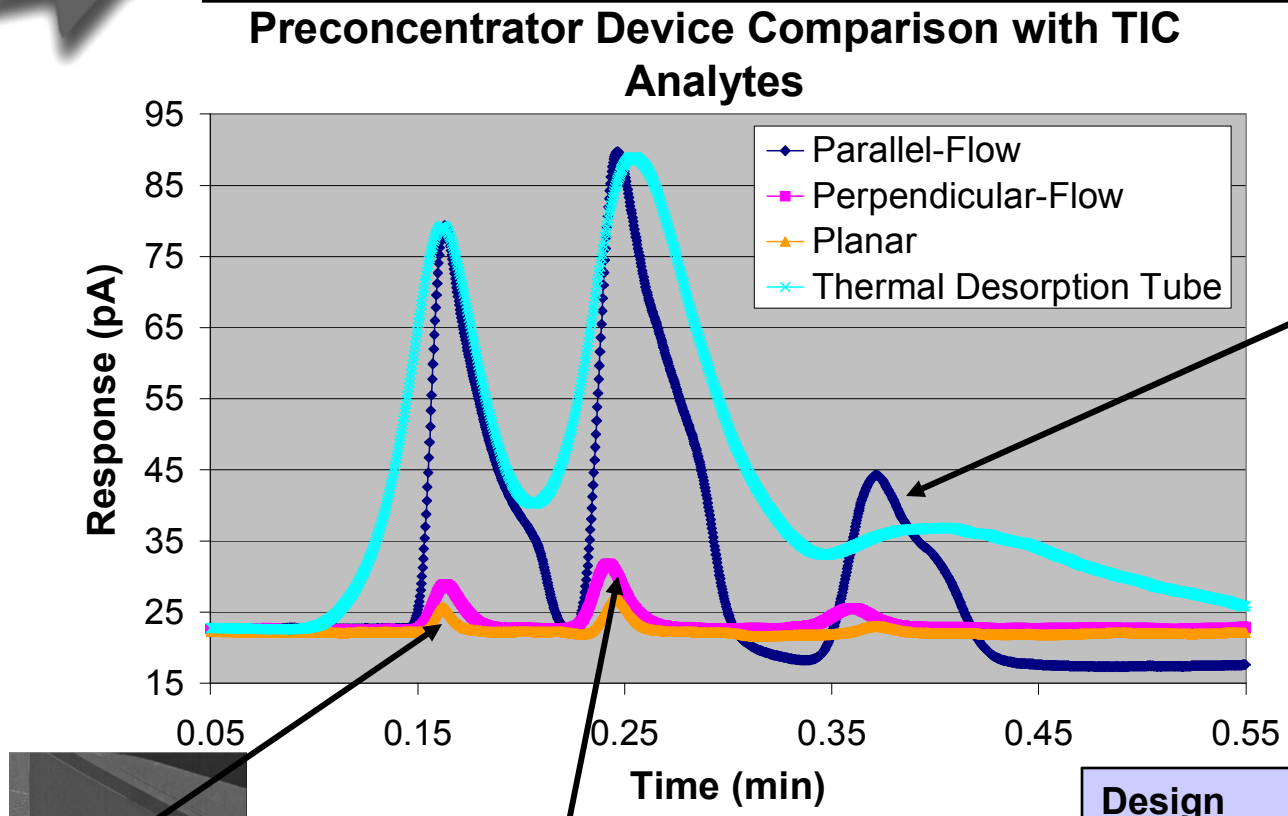
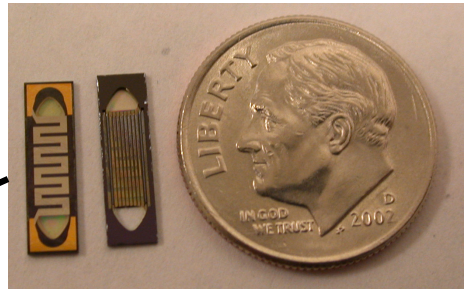
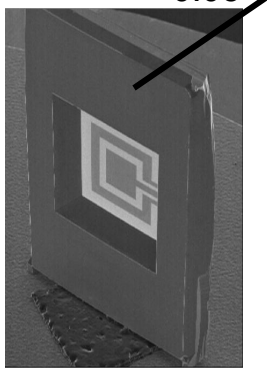
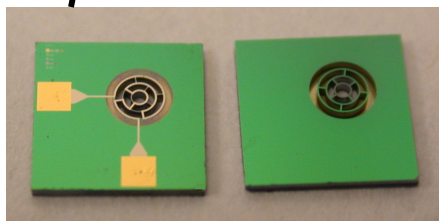
Comparison of Collectors





0.6 W vs 3 W

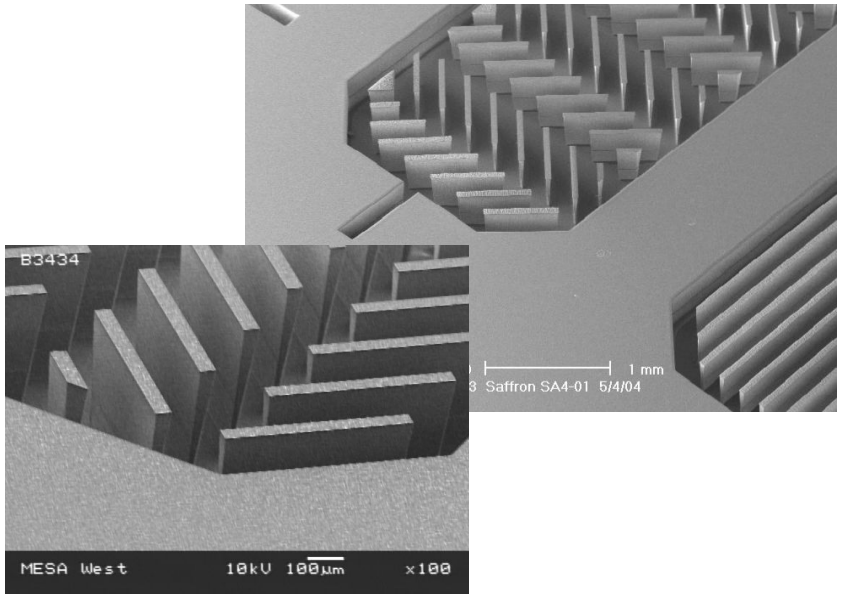


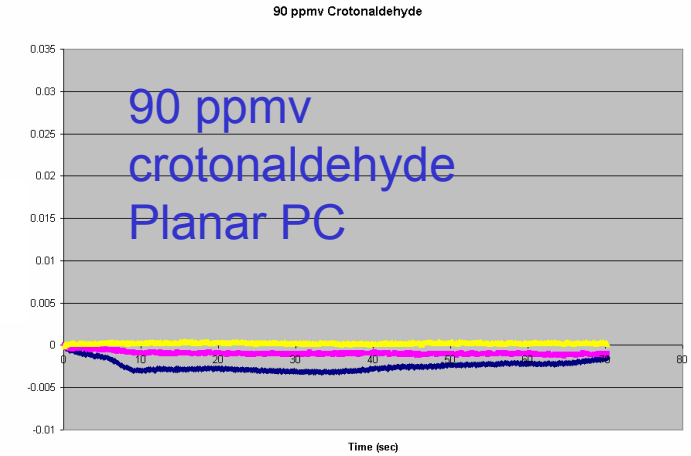


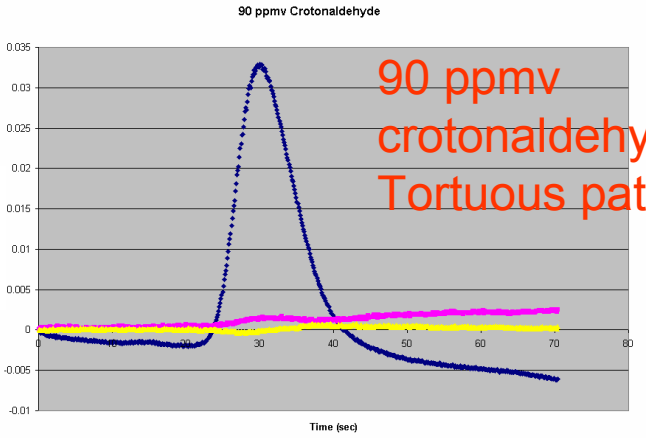
Design	t90 to 200C	Power
Planar	10 msec	100 mW
3D	0.6-1 sec	200-600 mW
Tubular	~minutes	~watts

TIC collection

- Improved collection/desorption – higher surface area, better contact, lower dead volume
- Lower level detection, new analytes
- Ease of assembly
- Water, and VOCs can now be addressed
- TIC, THM, CW
- Explosives







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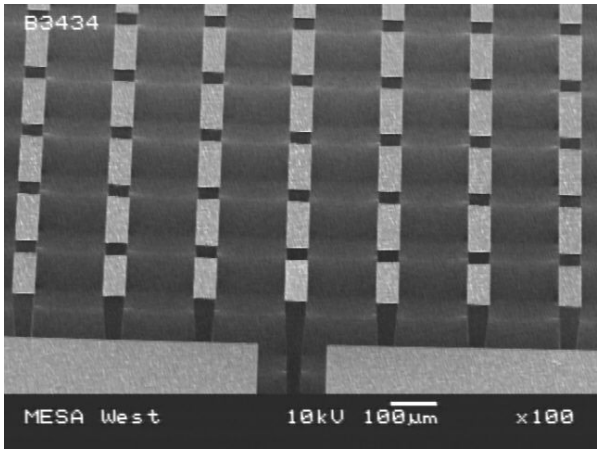
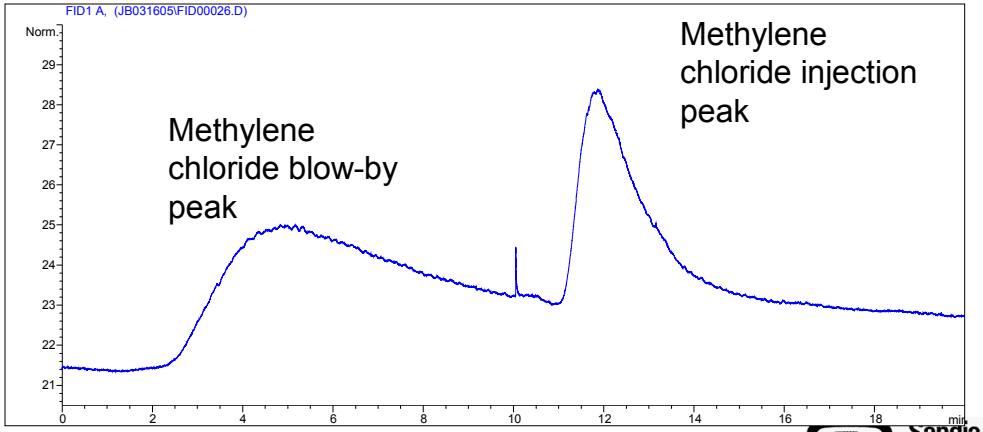
Tortuous Path Base-Catalyzed Sol-Gel Excessively Coated Preconcentrator Collection Efficiency for Various Analytes

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 = Area injected/ (Area blow-by+ Area injected) x100**

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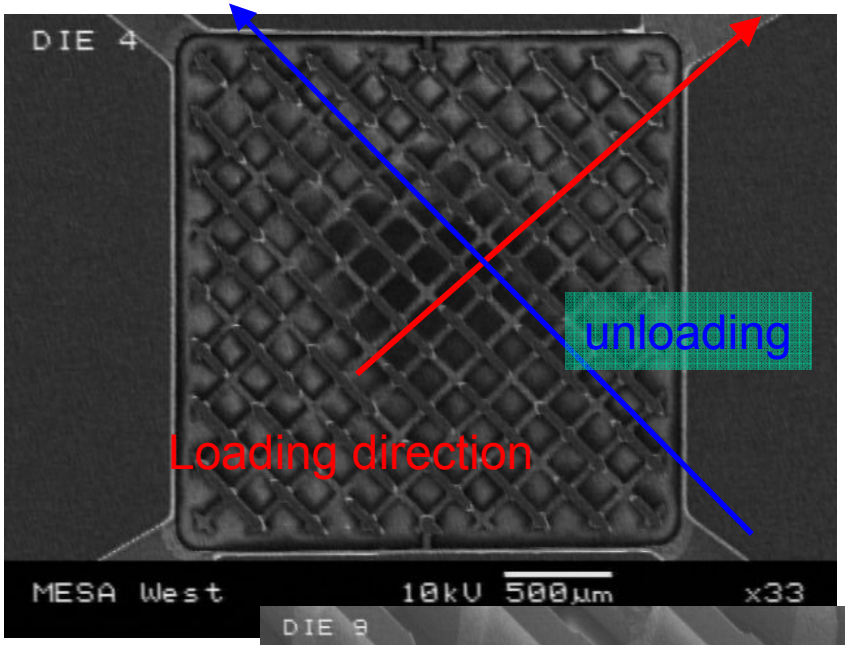
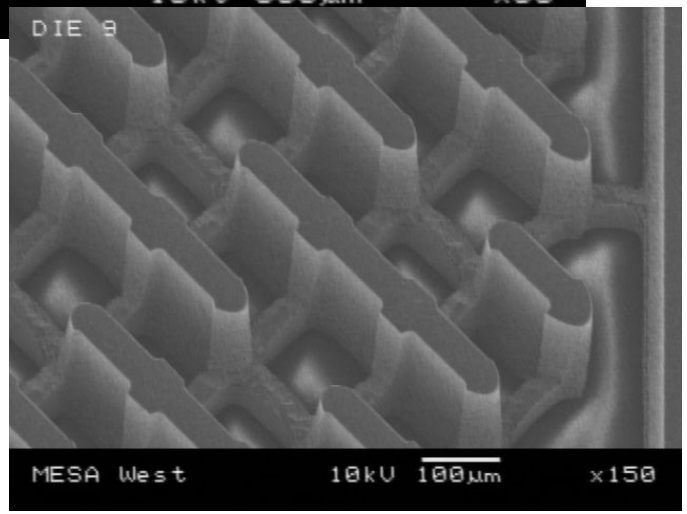
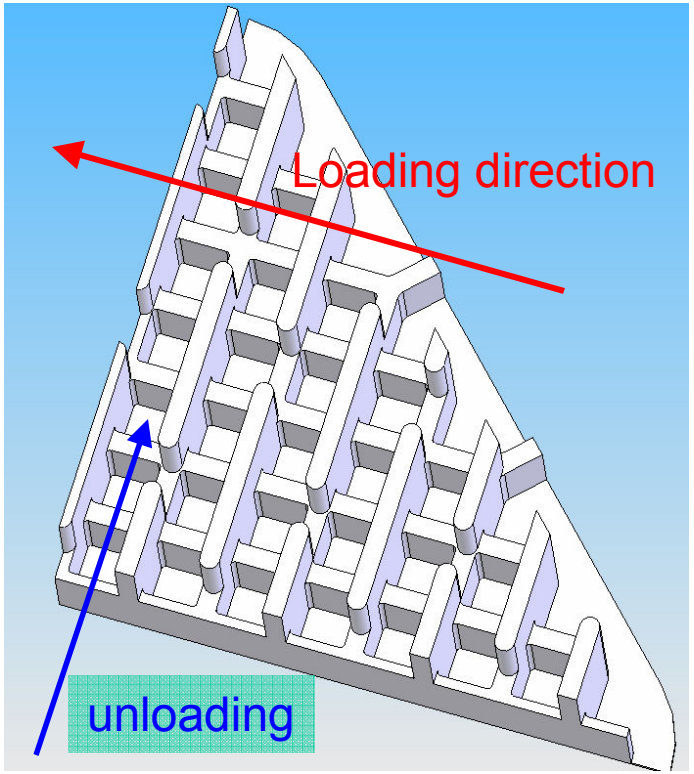
2 layer photolithography is used to create partial height struts/support and full height adsorbent fins







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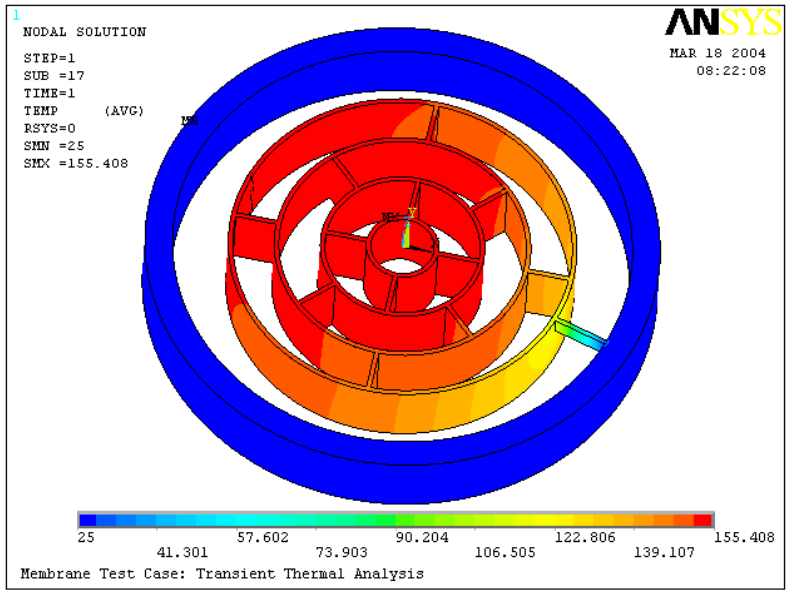
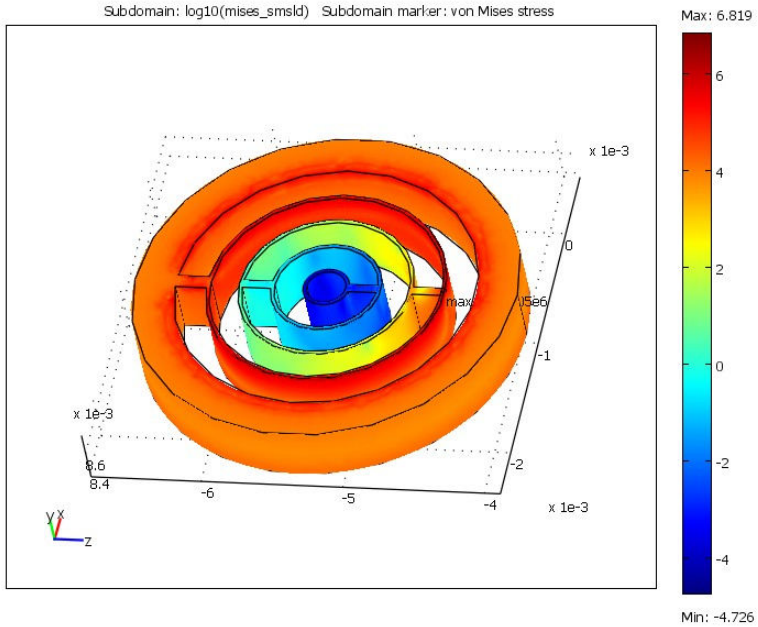
Modeling Efforts



Thermal: Power and desorption speed can be further reduced:
1.3 sec to 0.3 sec to 200ºC

Half wall thickness
Even with a support strut



Mechanical: struts are important

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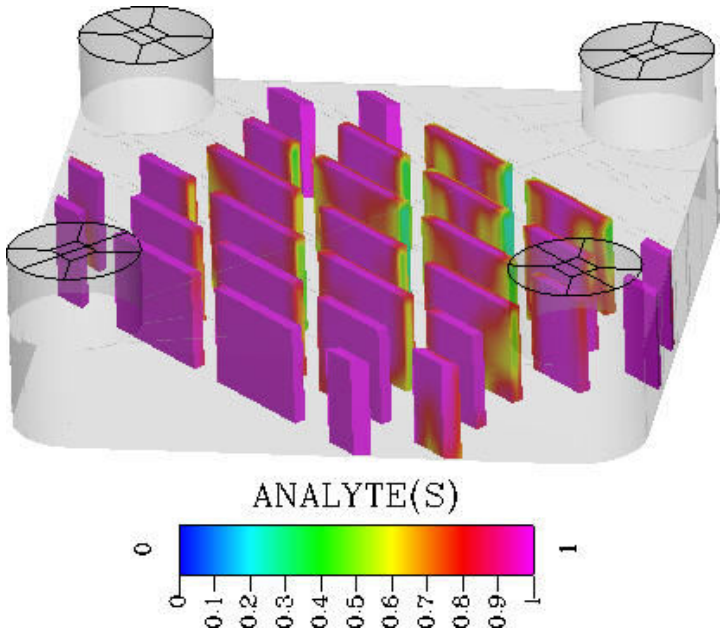
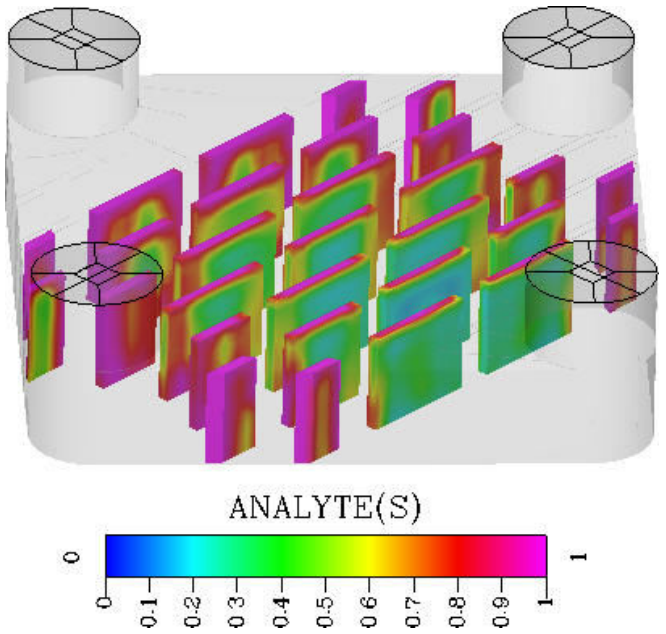
Computational Fluid Dynamics





Analyte surface fraction on the surface of the pillars for 300mL/min flow rate (Left) Front side of the pillars (Right) Backside of the pillars

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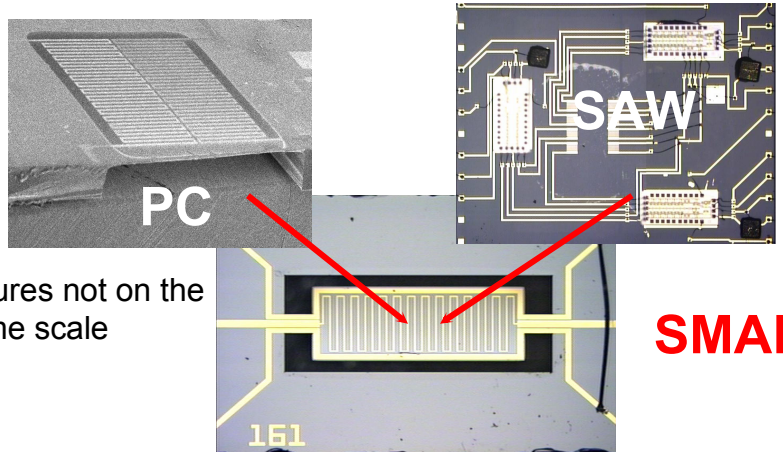
Smart PC™ combines preconcentration and detection to accelerate and automate detection

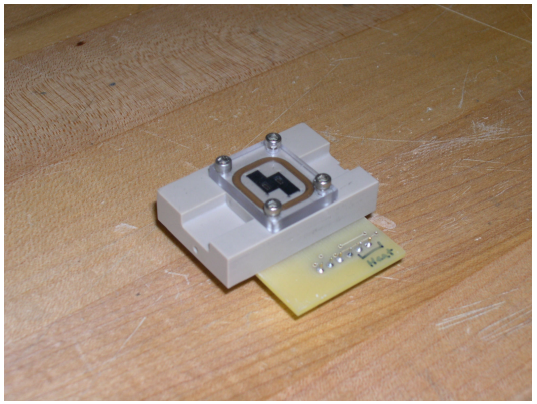
- DOD: reduced analysis times with increasing target concentration
- MEMS resonator with a heater/adsorbent weighs the sample & decides when it has collected enough
- Modular fixtures
- Circuit autotunes, autozeros
- Software subtract reference and smooth

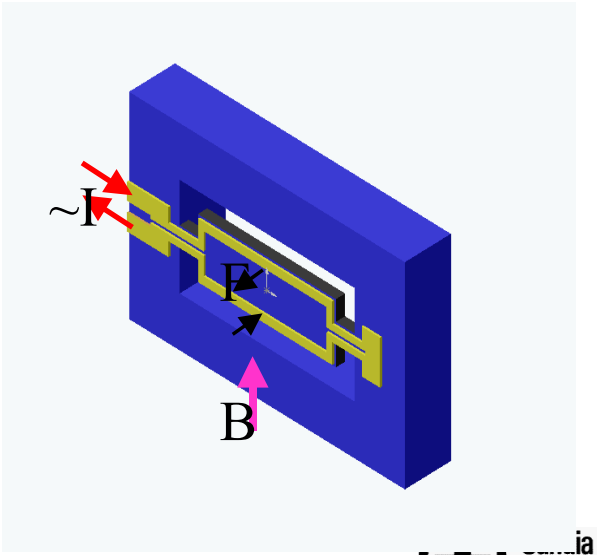


Figures not on the same scale

SMART PC™

Reduced need for trained operators





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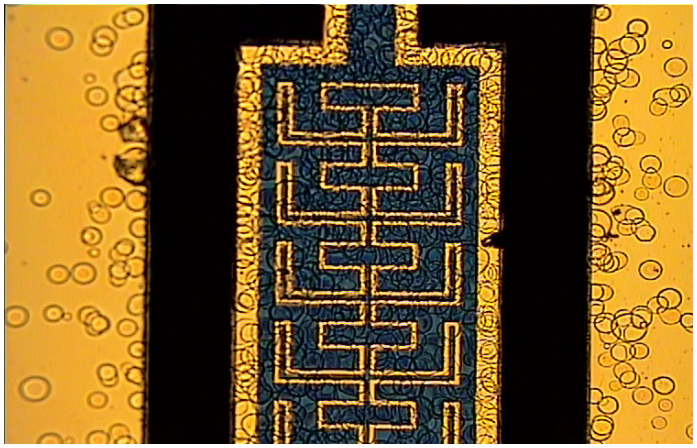
Coating results to date

Sol Gel

- DMNB
  - Good collection - even short times
- DNT
  - Low response
- DMMP
  - Very good response

NPC

- DMNB
  - Gradual collection
- DNT
  - Gradual collection
- DMMP
  - Very distinct response



DKAP – similar to BSP3, stronger acid functionality

- ~10x response of sol gel
- Mostly useful for detection

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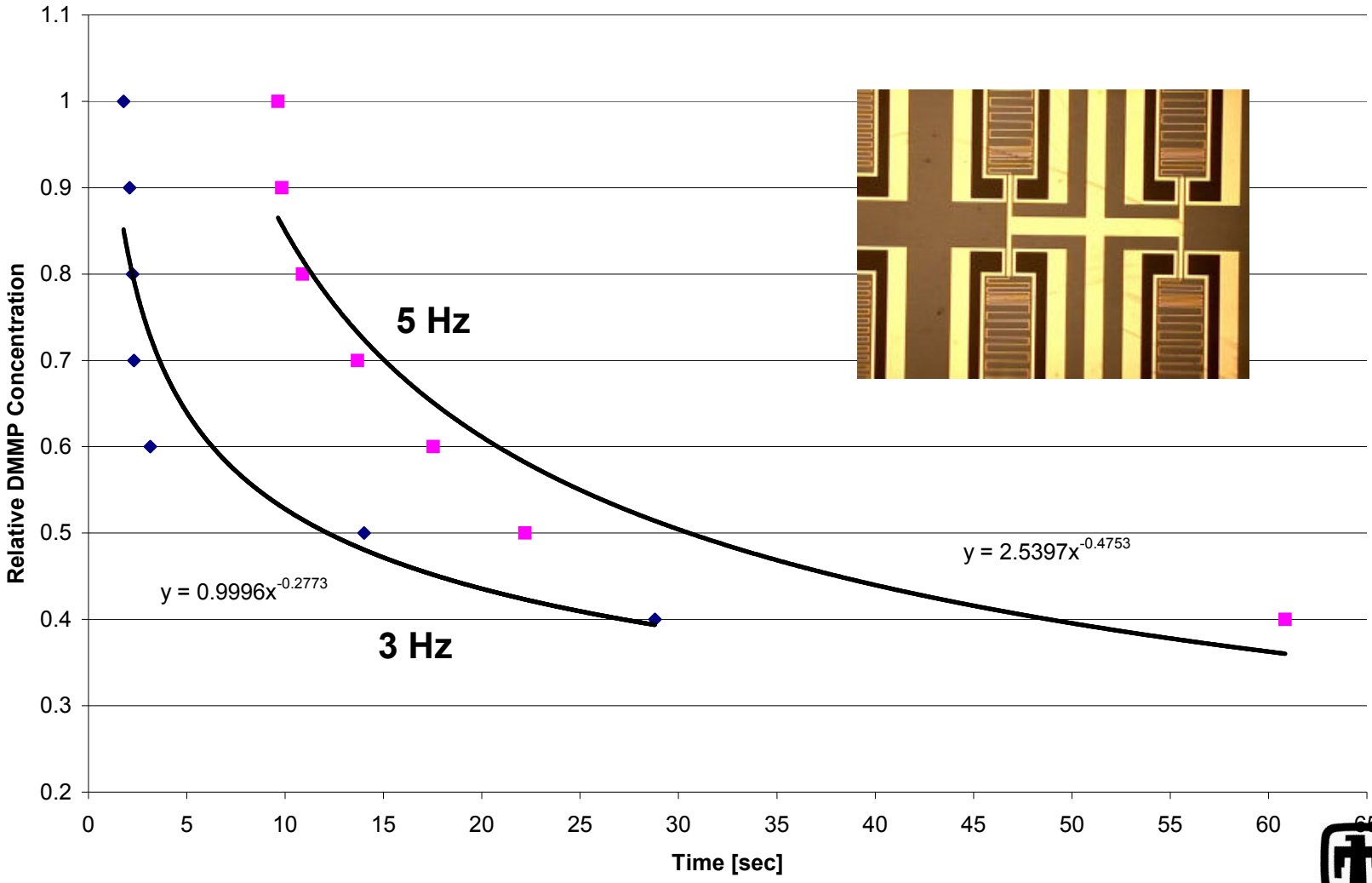
Decreasing collection time with concentration: accelerating analysis relative to typical PCs

Collection time required for a ~3Hz and ~5Hz frequency shift at a relative DMMP concentration Using a SolGel device with flow of 60 SCCM



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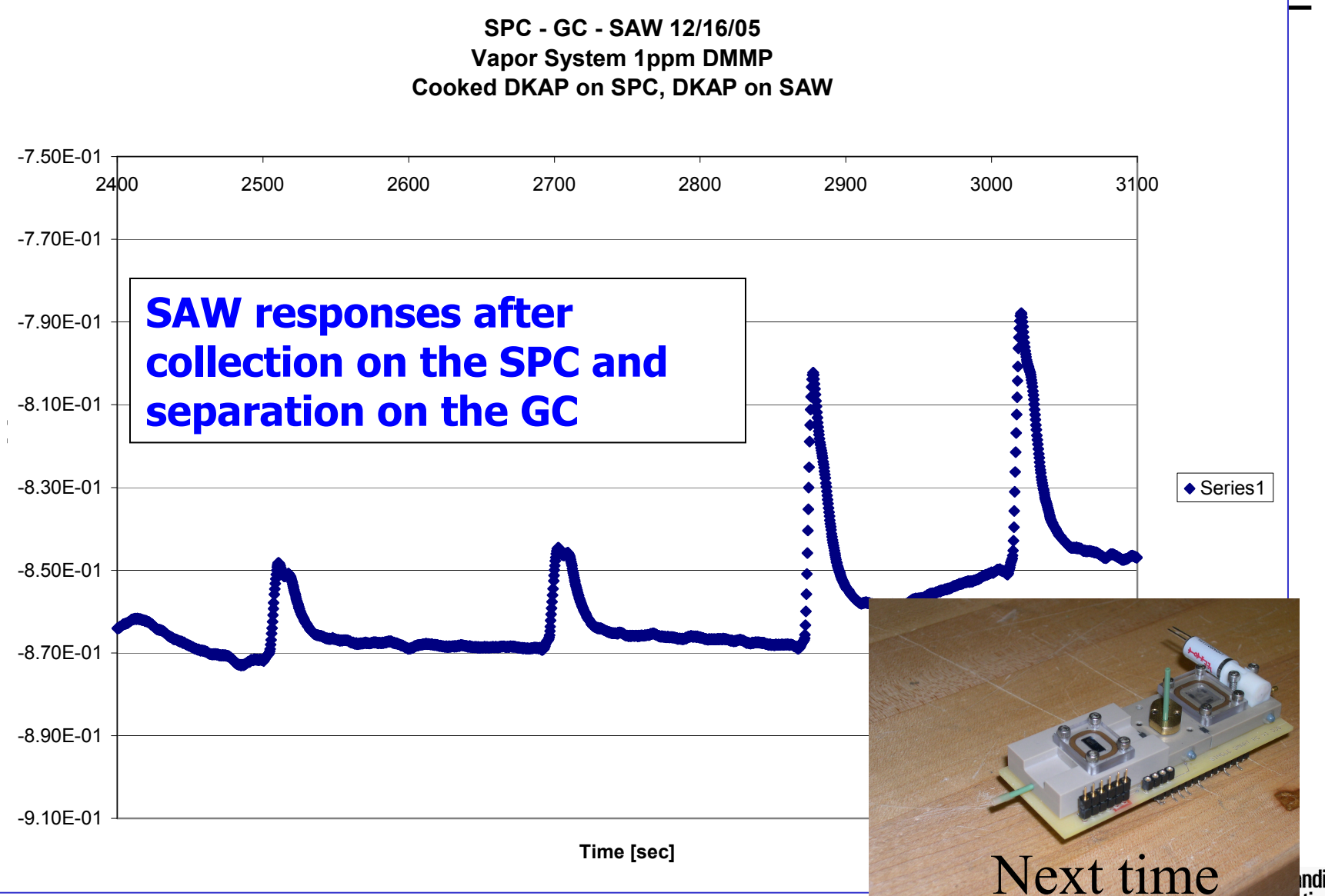
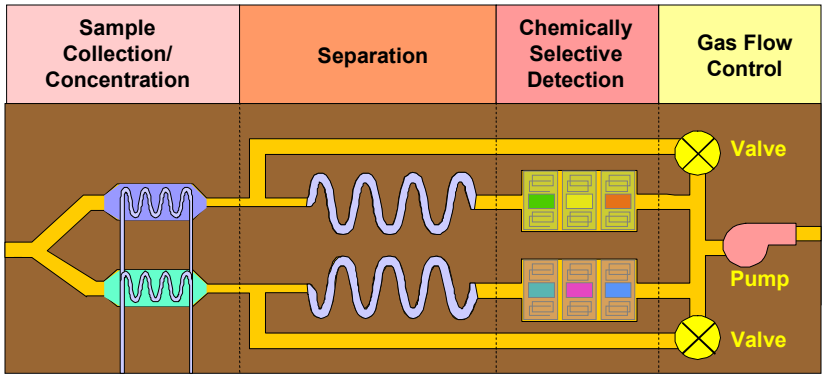
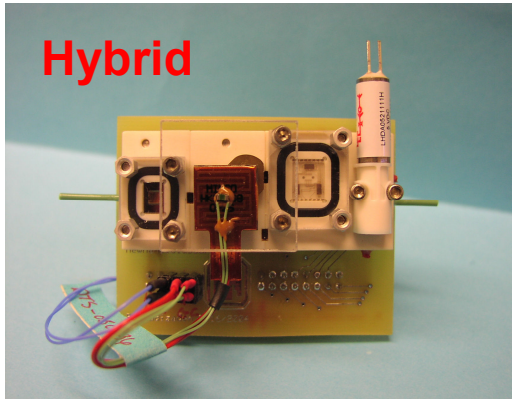
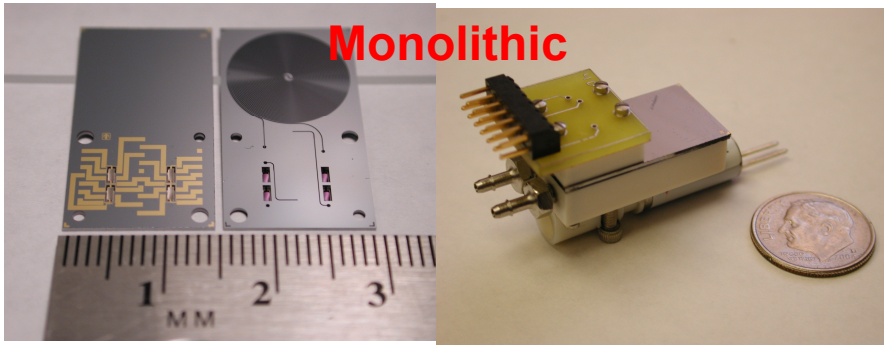
Detection with selectivity: 7 times faster at LC50 of Sarin



The microchemlab uses preconcentration, separation and selective detection to perform real-world analysis: hybrid or monolithic packaging plays an important role







+ Modular adaptability
- Unheated transfer
- Long transfer
- Relatively larger
+ Reduced thermal isolation concerns

+ Lowest dead volume – best performance
+ Heated, short transfer
+ Long transfer
+ Relatively smaller
- Thermal isolation issues – solved by ramping
+GCs tested with CW simulants

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Acknowledgments

- Kevin Linker
- DARPA MGA, Dennis Polla
- Sandia Labs LDRD Office
- Dr. Elizabeth George and Dr. Randy Long, DHS

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Extras follow

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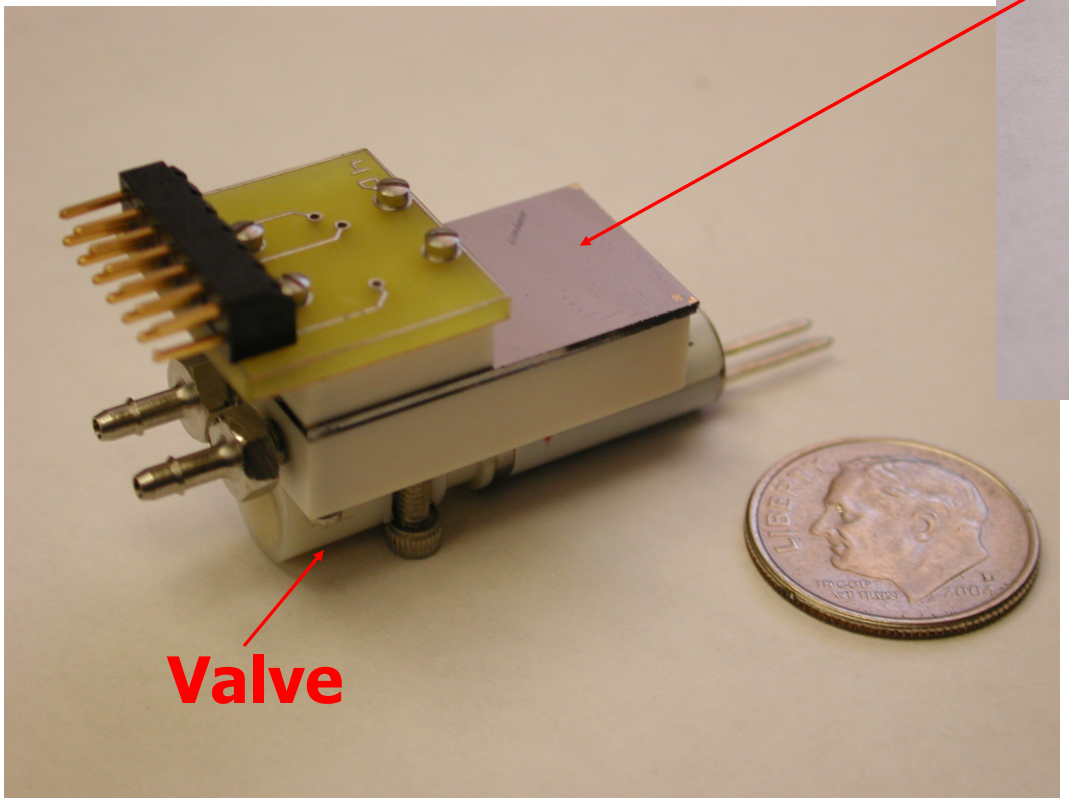
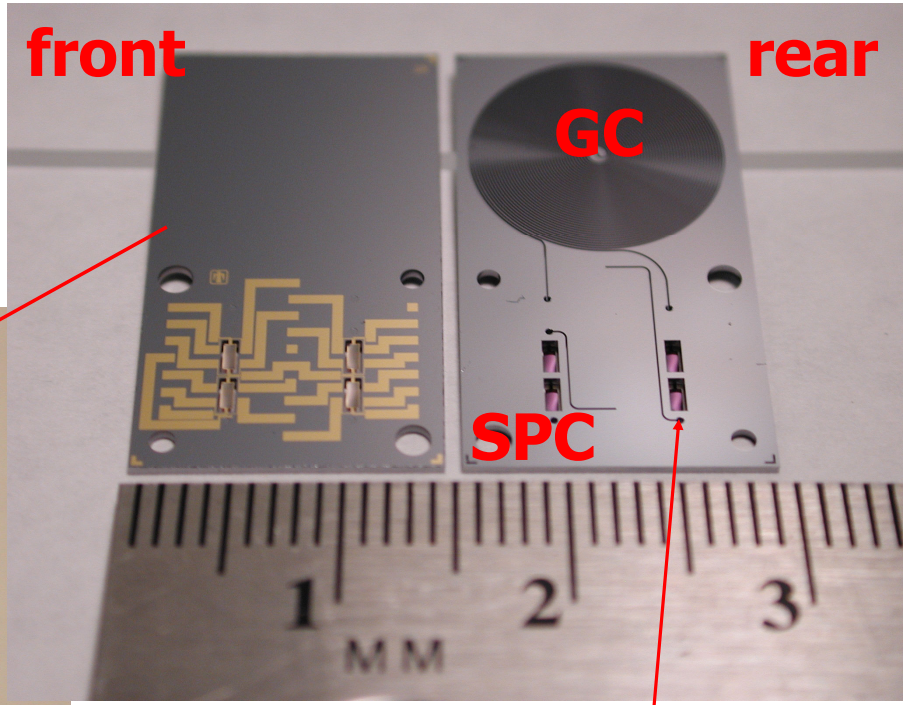
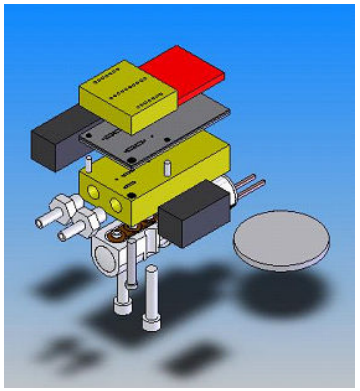
Integrated System for reduced dead volume, size, cost, etc.

- **Smallest MicroChemlab Yet**
- **Coatings, GCs demonstrated**







SPC Detectors

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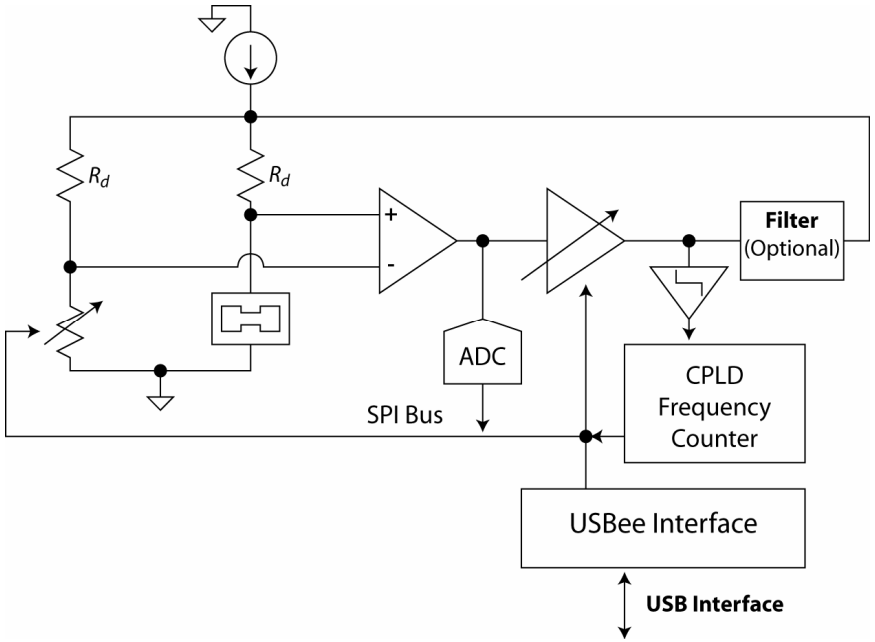
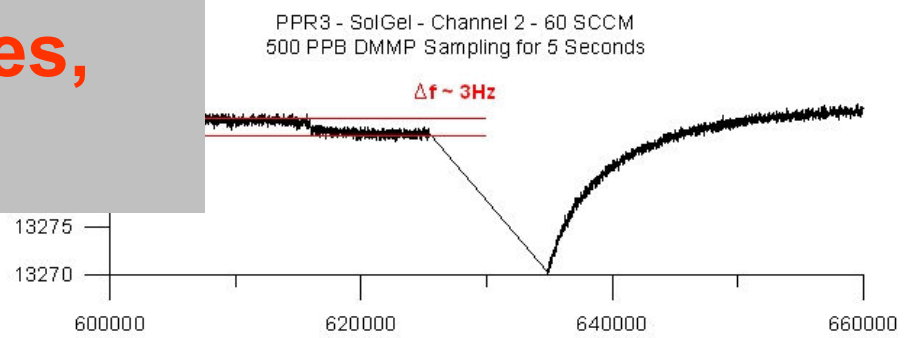
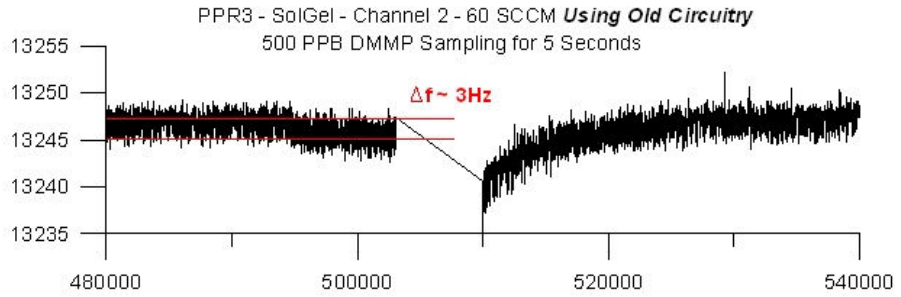
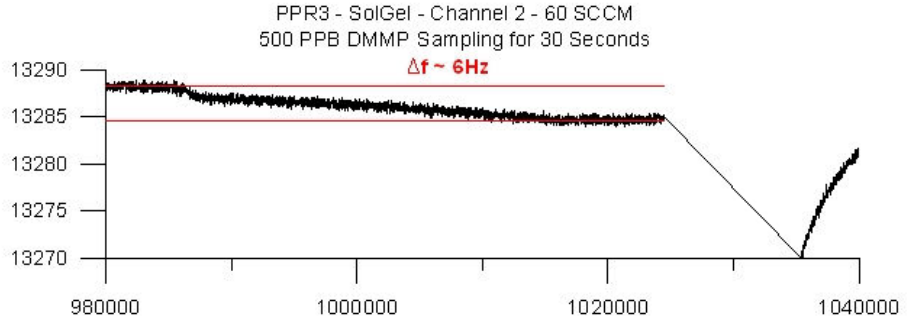
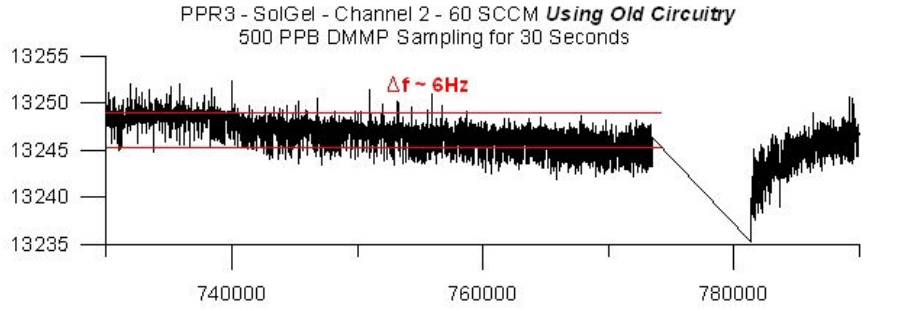
Circuit autotunes, autozeros











- **Improved filtering, power regulation and potentiomete resolution**
- **Software subtraction of reference and smoothing**