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A Review of Microfabricated Preconcentrators for Portable Chemical Analysis Systems

Ronald P. Manginell, Matthew Moorman, D. Adkins†, P. Lewis, D. Porter, R. Hadizadeh, D. Copic, R. J. Simonson, R. Kottenstette, J. Whiting, S. Radhakrishnan*, R. Cernosek

Sandia National Labs, Albuquerque, NM
rpmangi@sandia.gov

†Defiant Technologies, Inc., Albuquerque, NM
www.defiant-tech.com

***ESI-Group, Hunstville, Alabama**

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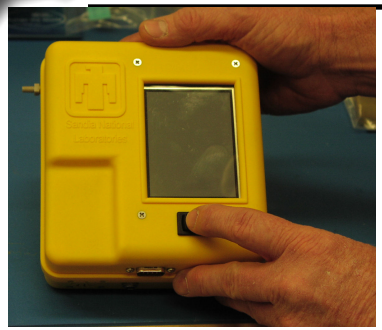
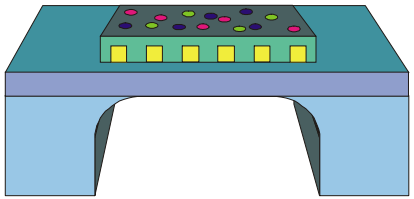
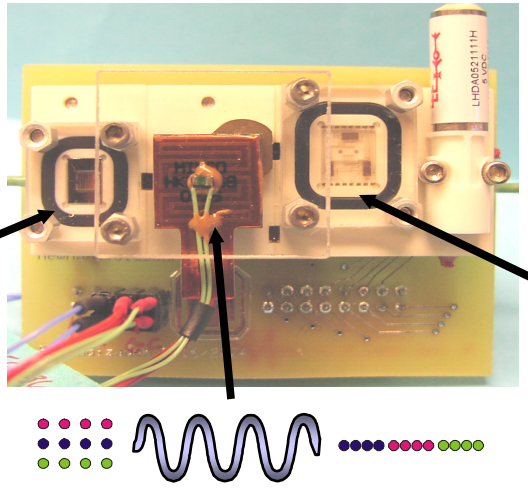
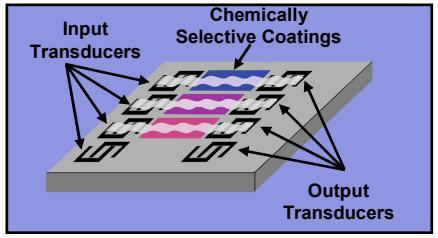
Sandia's MicroChemLab™

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





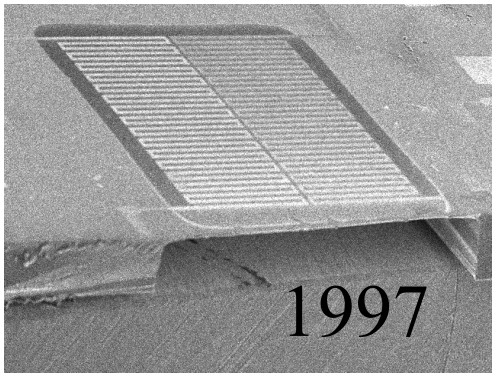


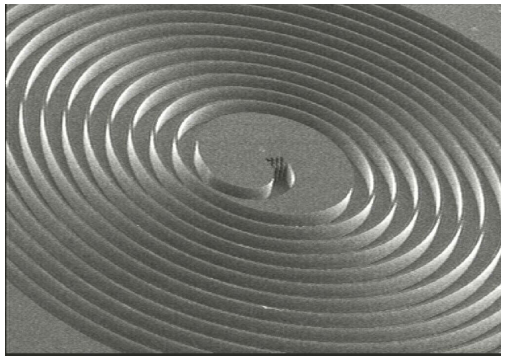


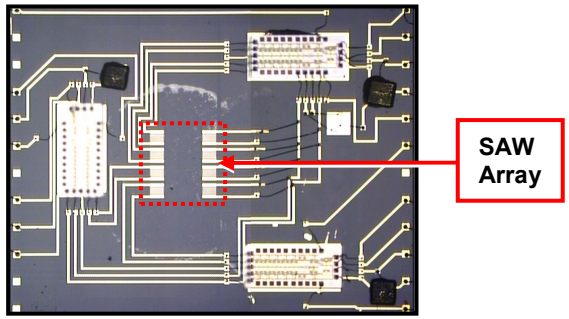
Preconcentrator accumulates analytes of interest

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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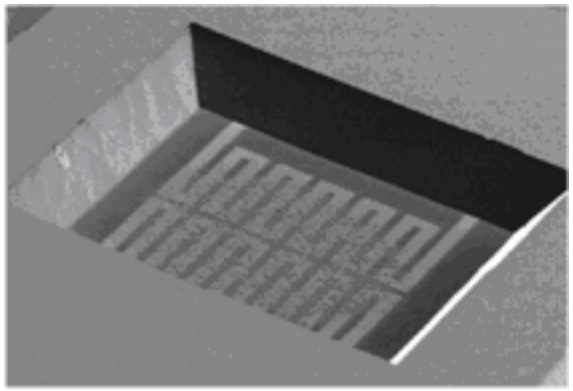
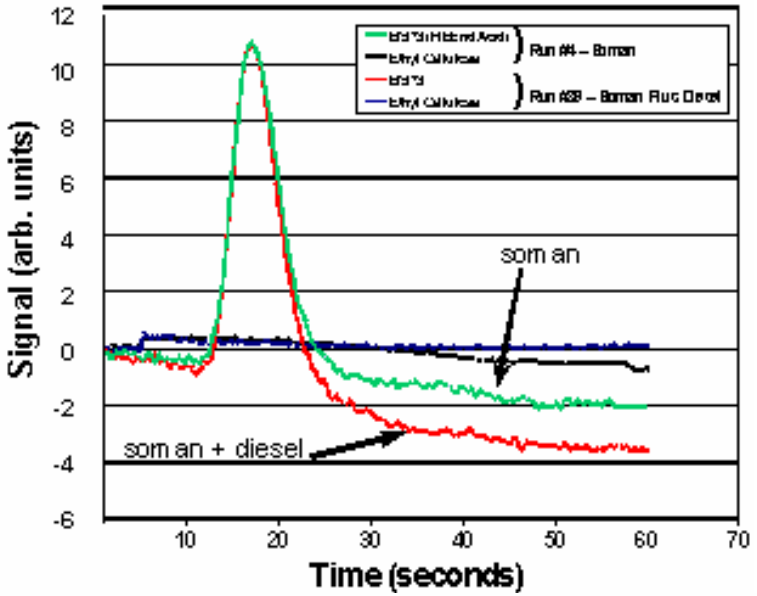
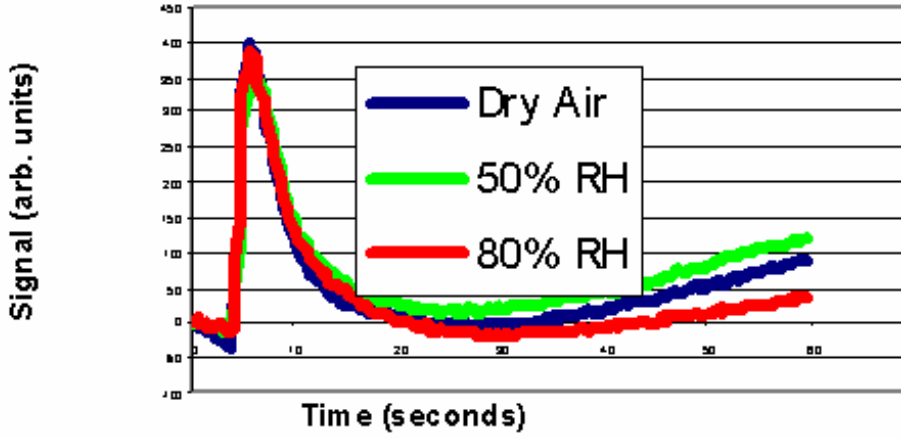
Planar MicroFabricated Preconcentrators

- 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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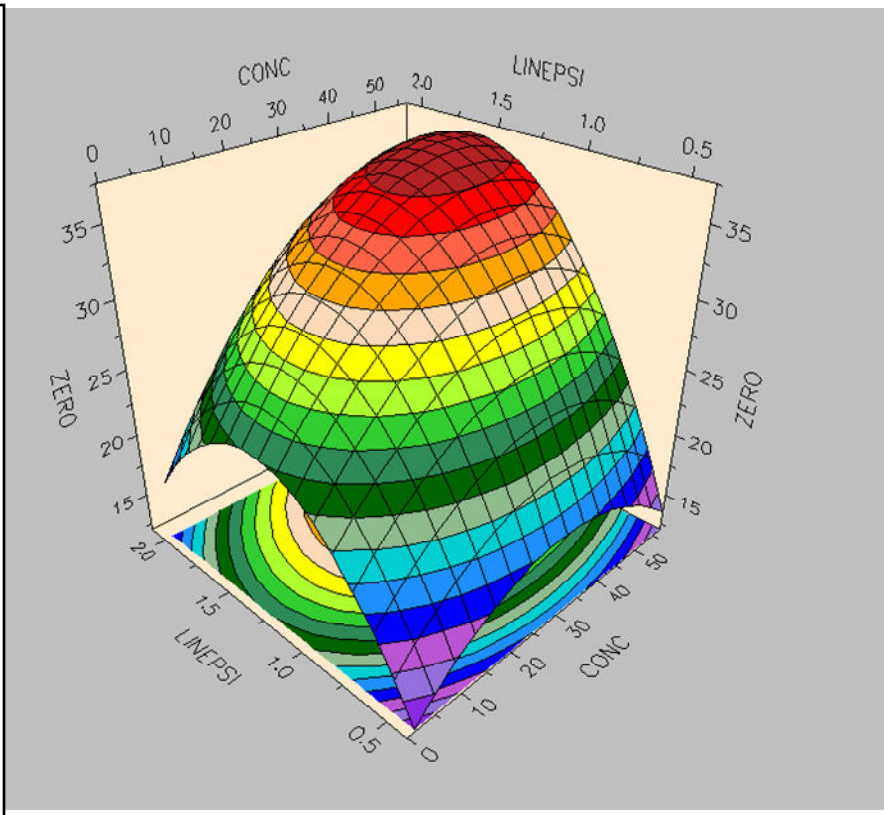
CFD Modeling and DOE

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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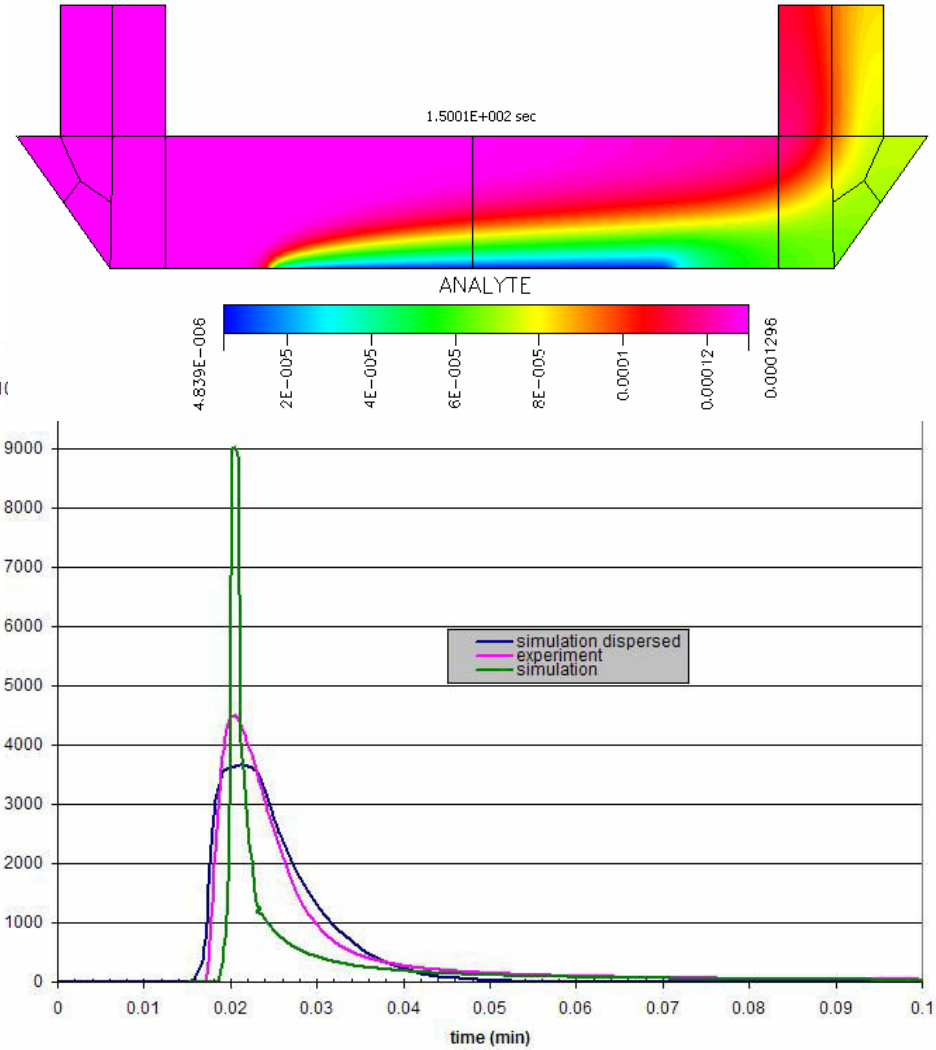
CFD Modeling (ESI Group) & DOE

- 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", accepted IEEE Sensors Journal.

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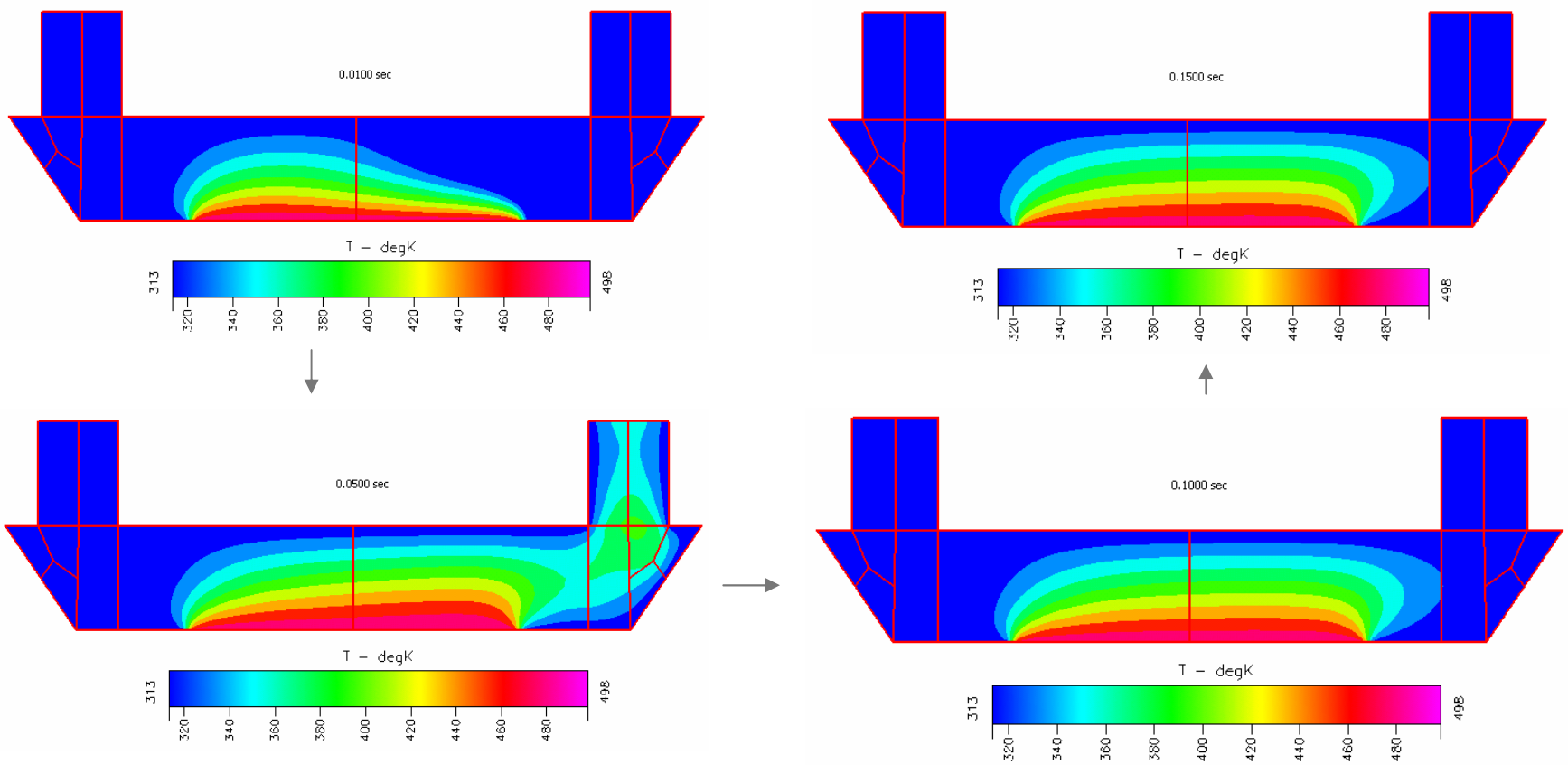
Eye Candy: Thermal pulse can be seen on a downstream detector



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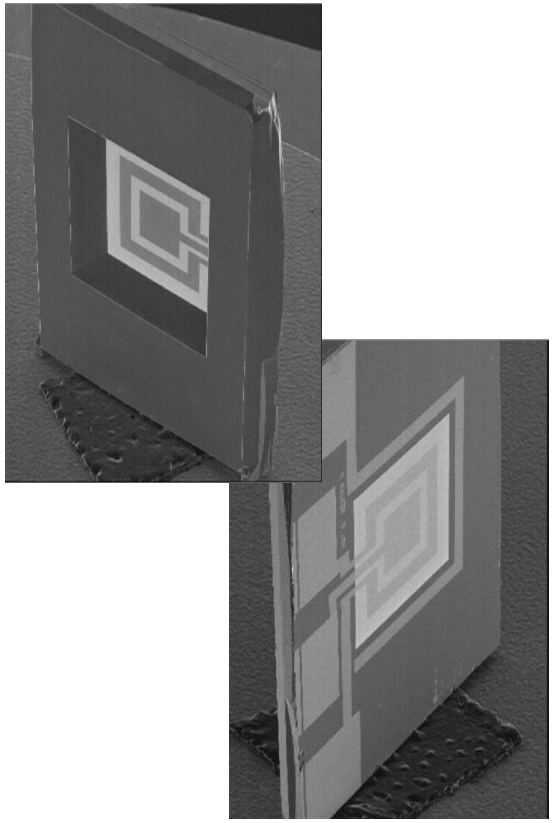
3DPCs as a supplement or replacement for the planar PC

Planar PC

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

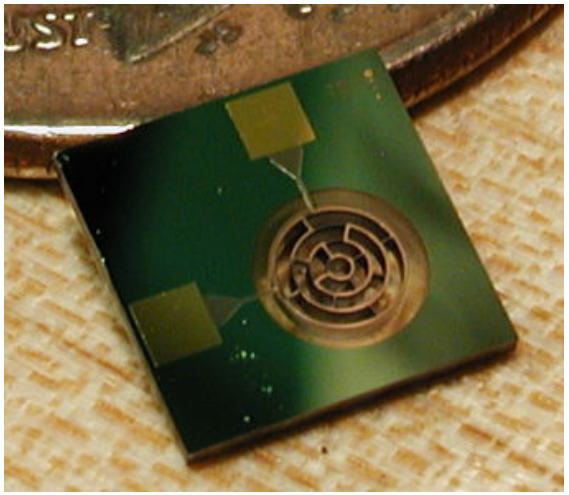
3DPCs

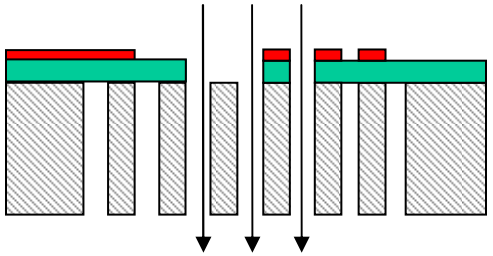
1. Planar PC items 1 & 2 retained
2. Smaller diffusion length, higher area, flow through
3. Pressure balance possible



Improved collection performance

More analytes like volatile organic compounds (VOC)





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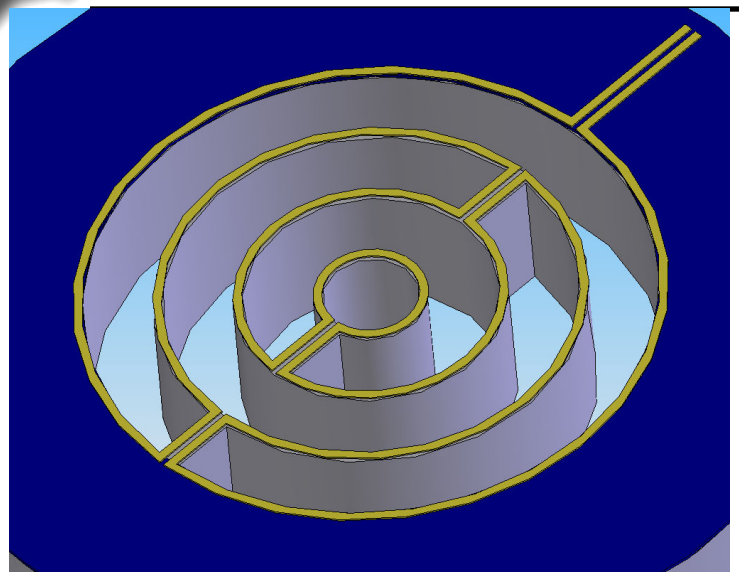
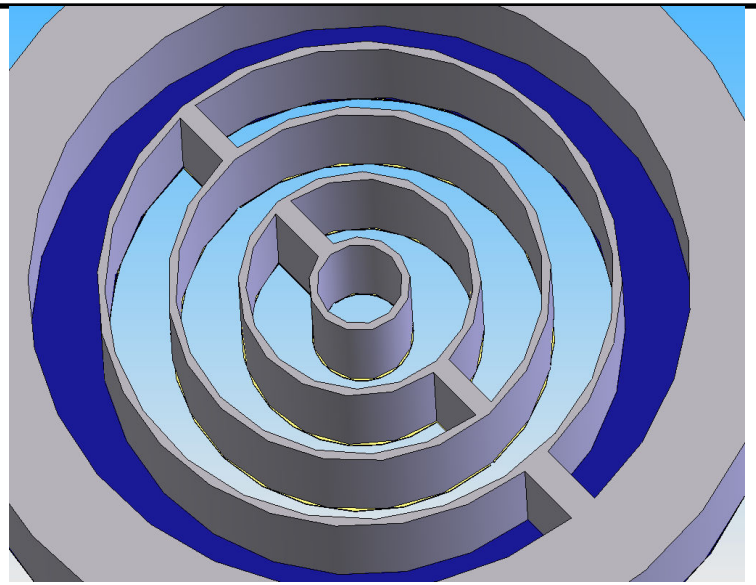
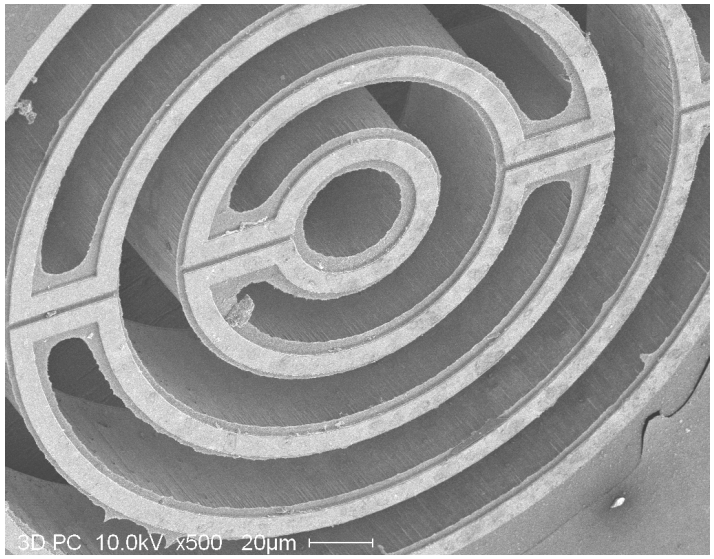
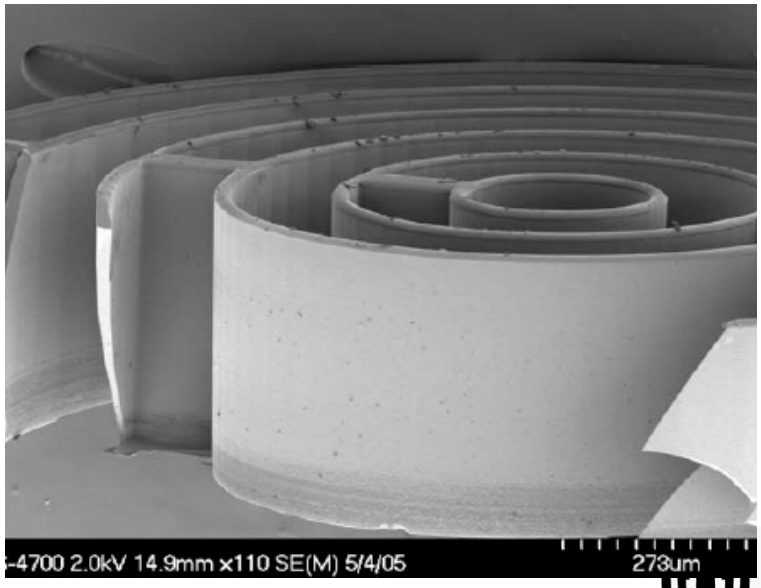
Types of 3DPCs: perpendicular flow. Etching thanks to ITC.









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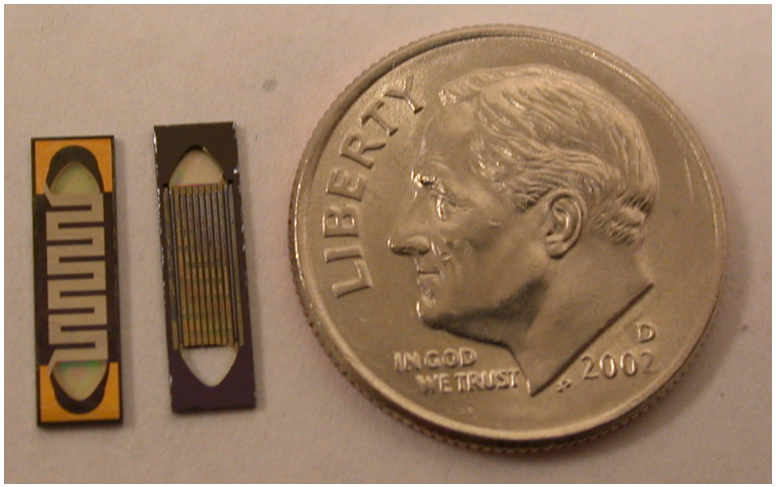
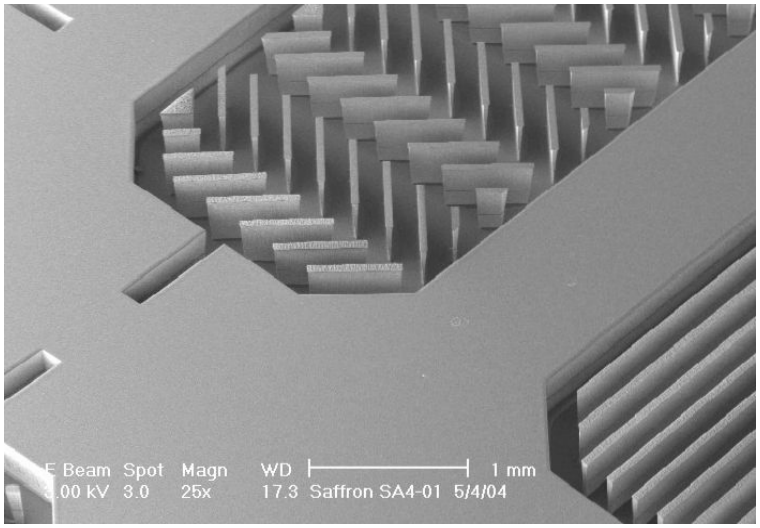
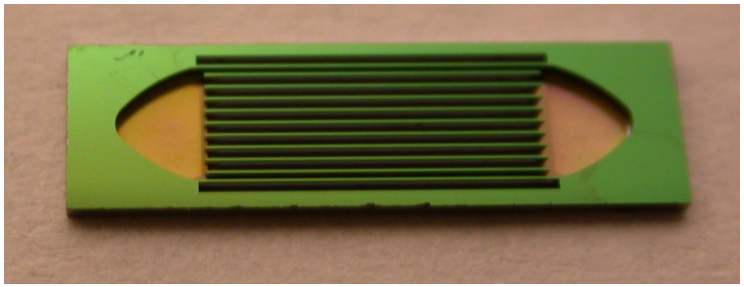
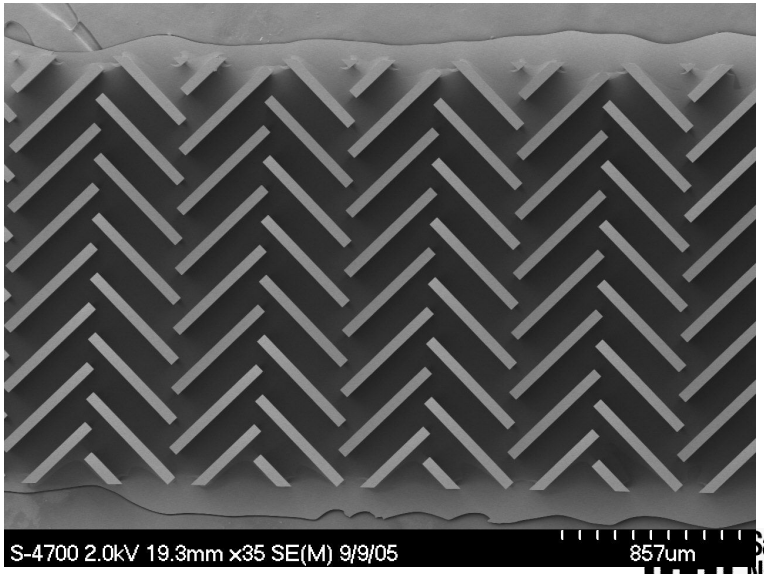
3DPC: parallel flow and tortuous path









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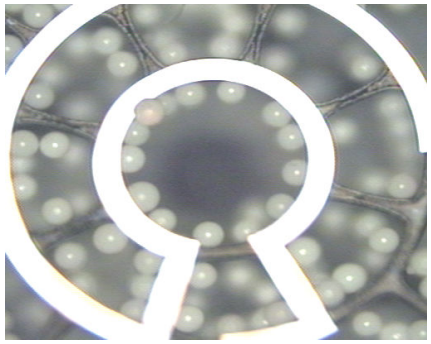
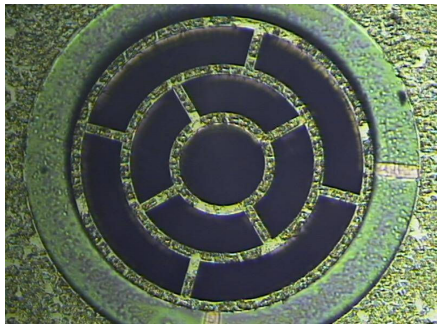
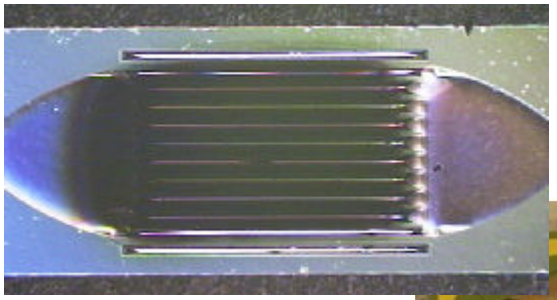
3DPCs, coatings and target analytes: enhanced collection

- **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

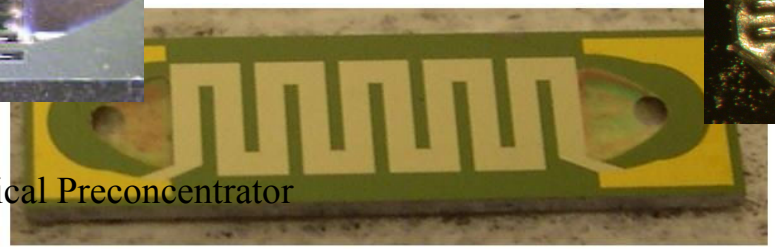


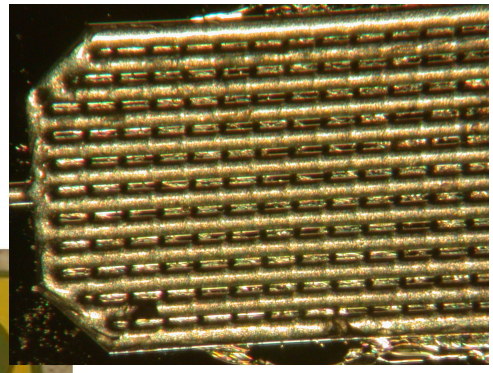


"Perpendicular flow"



"Parallel flow"





US 7,118,712 Non-Planar Chemical Preconcentrator

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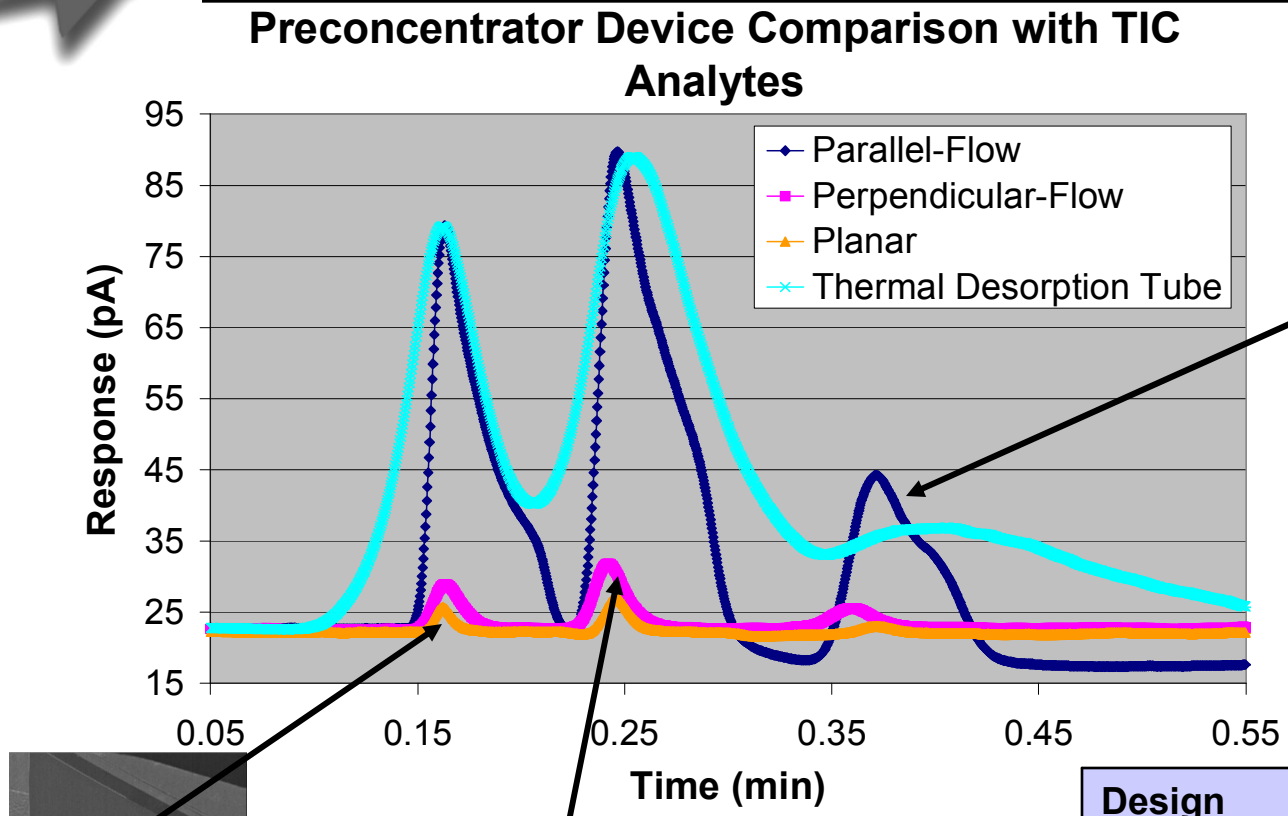
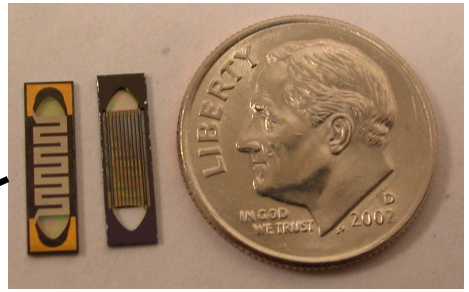
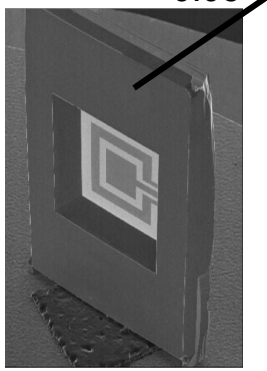
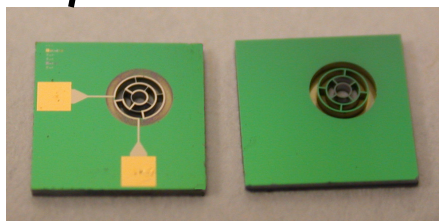
Comparison of Collectors





0.6 W vs 3 W

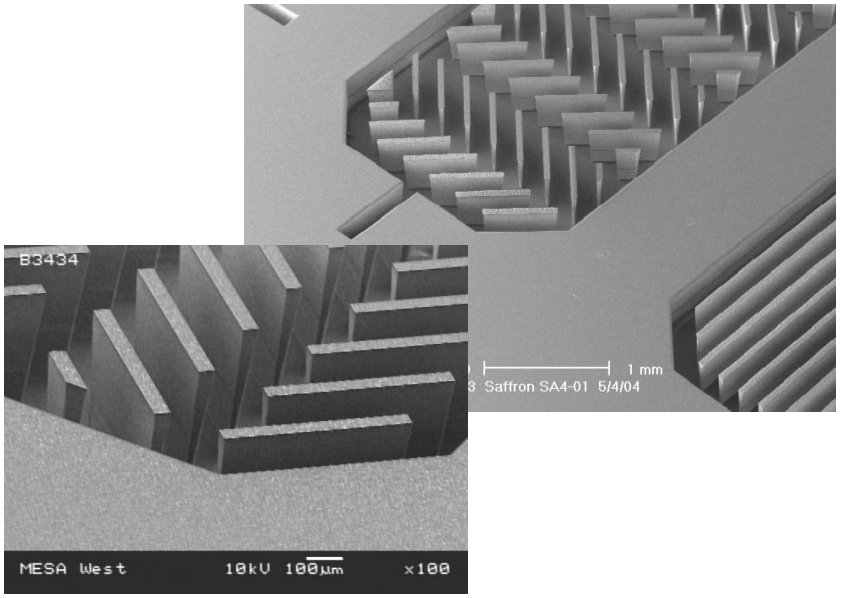


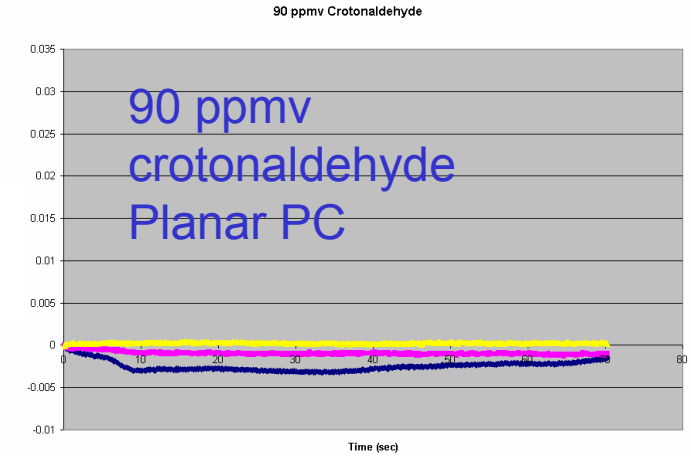


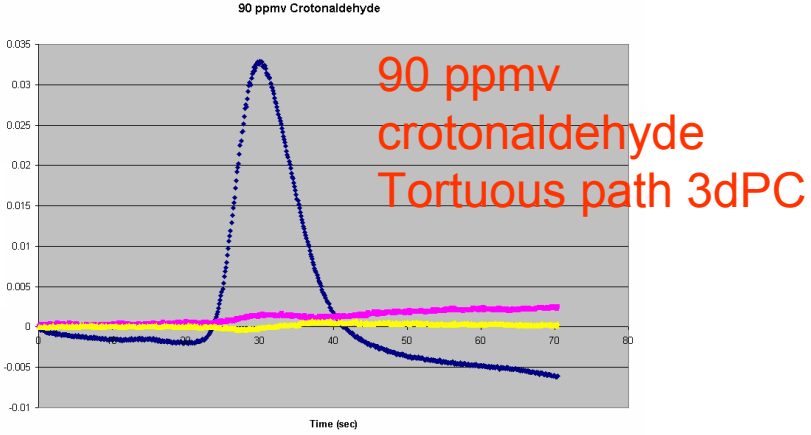
Design	t90 to 200C	Power
Planar	10 msec	100 mW
3D	0.6-1 sec	200-600 mW
Tubular	~minutes	~watts

Benefits of 3D Design

- 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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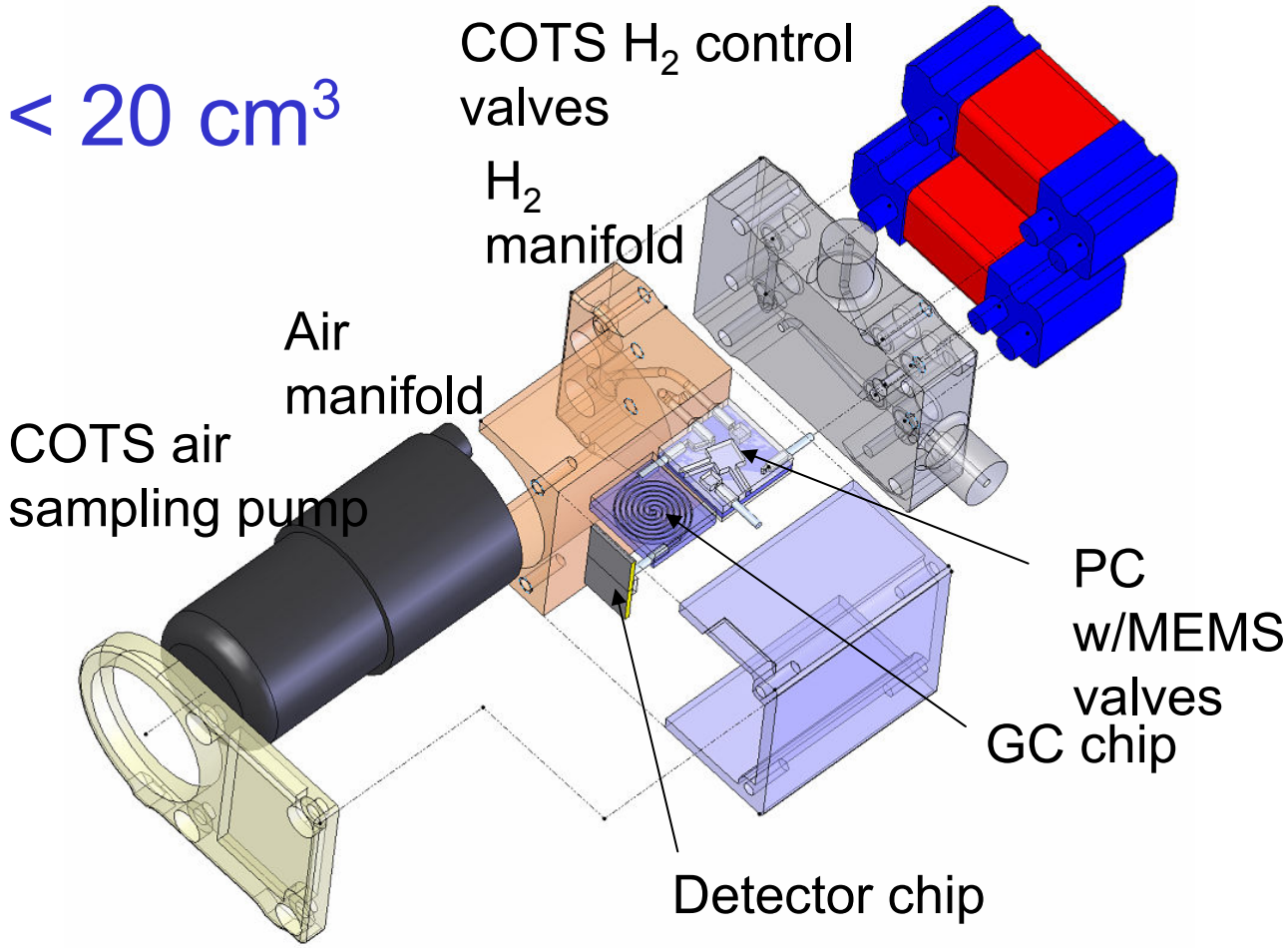
DARPA MGA: Performance requirements drive system architecture enhancements

Maintain: Low false alarm rate, <1/200,000

Increase: Analysis speed, Analytical channel capacity

Decrease:

- System volume < 20 cc
- Limit of detection
- Energy consumed per analysis



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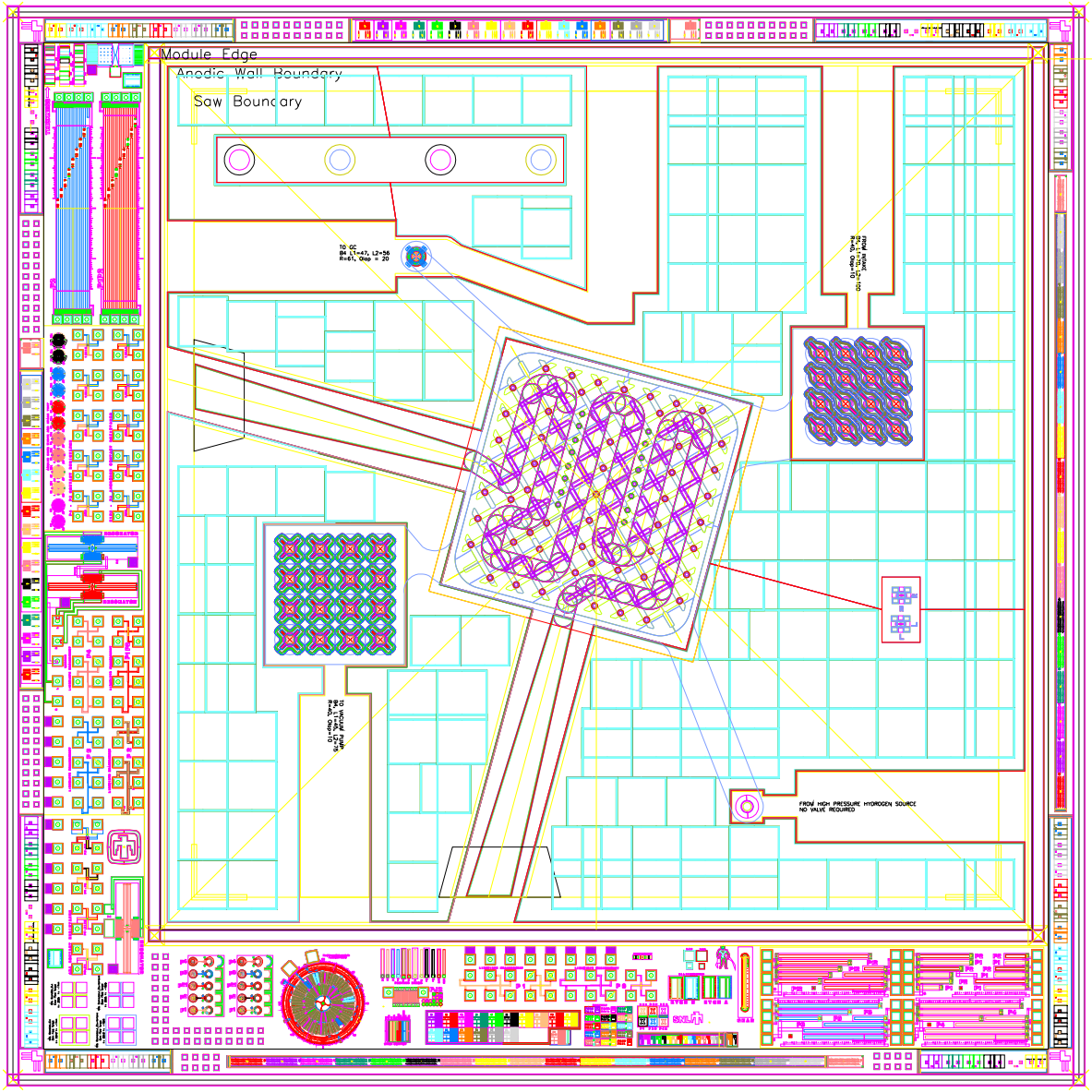
DARPA MGA: MEMS valves on PC chip limit inlet volume



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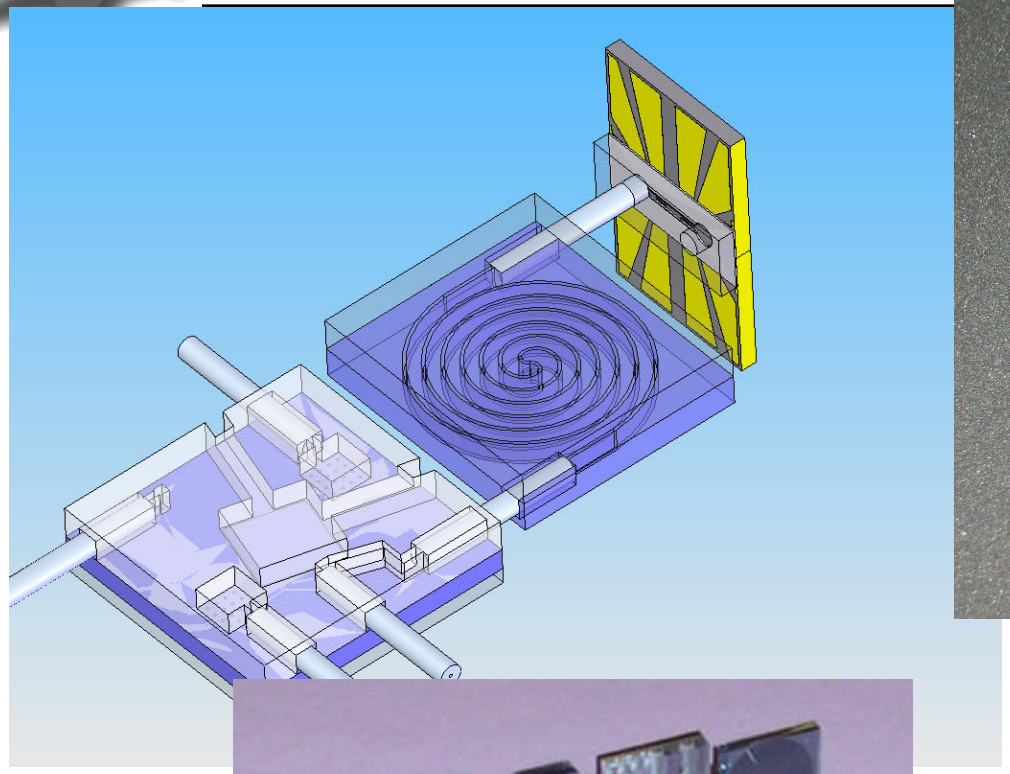
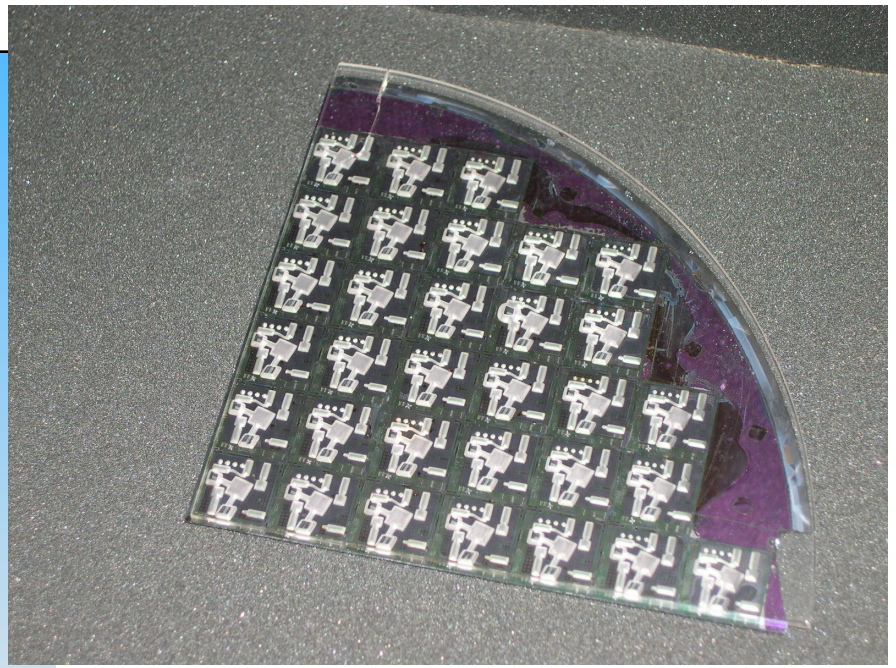
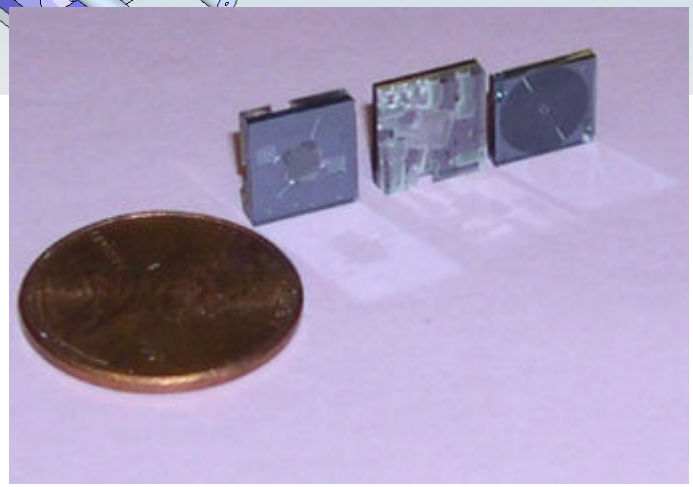
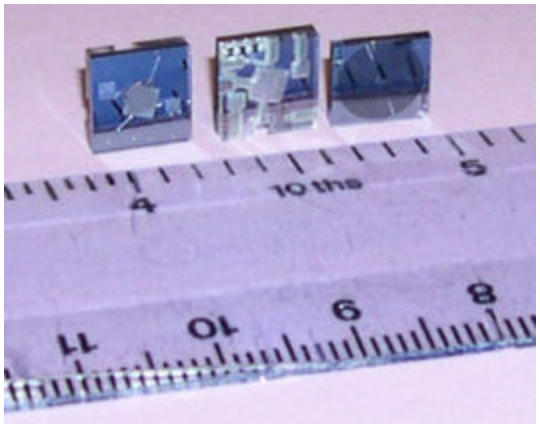
DARPA









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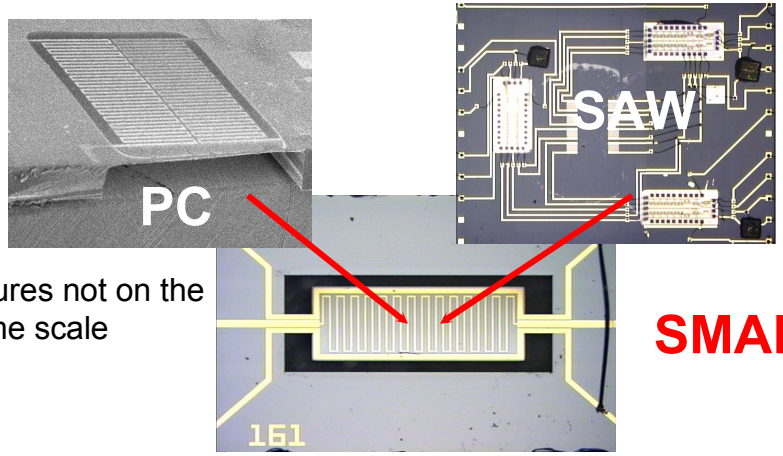
Smart PC™ combines preconcentration and detection to accelerate and automate detection

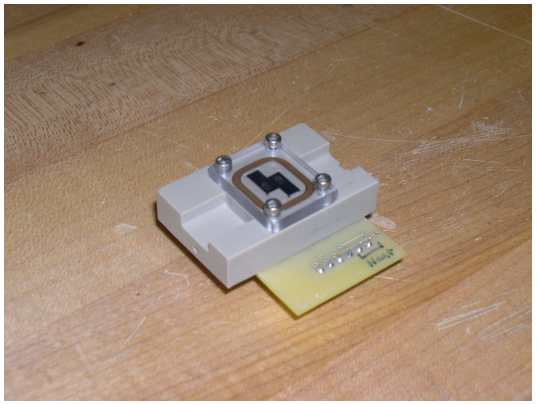
- DOD: reduced analysis times with increasing target concentration
- MEMS resonator with a heater/adsorbent weighs the sample & decides when it has collected enough
- Modular fixtures
- Circuit autotunes, autozeros
- Software subtract reference and smooth

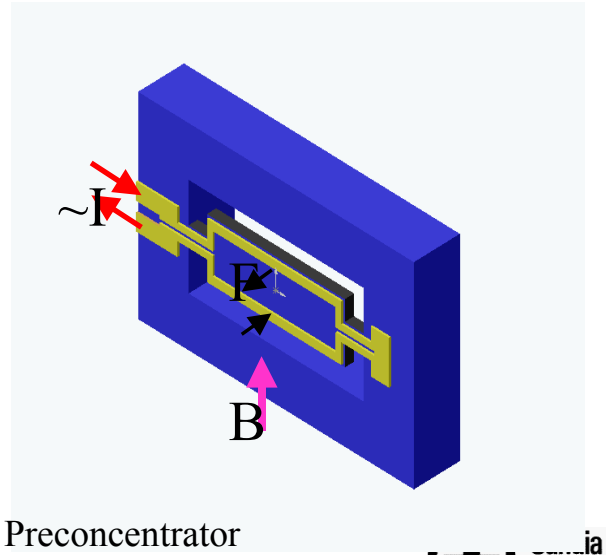


Figures not on the same scale

SMART PC™

Reduced need for trained operators





USPTO 7,168,298 Mass Sensitive Preconcentrator

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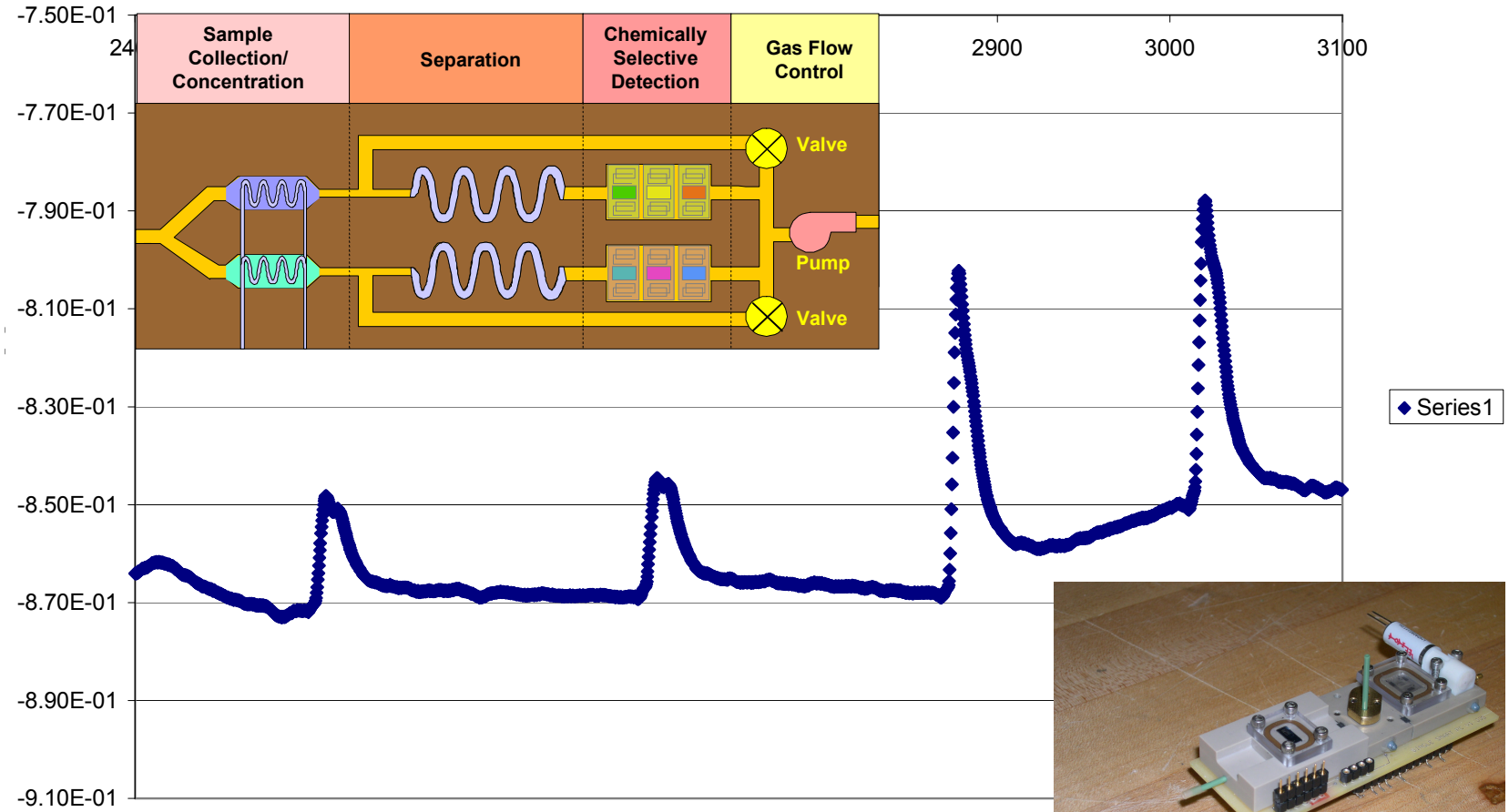
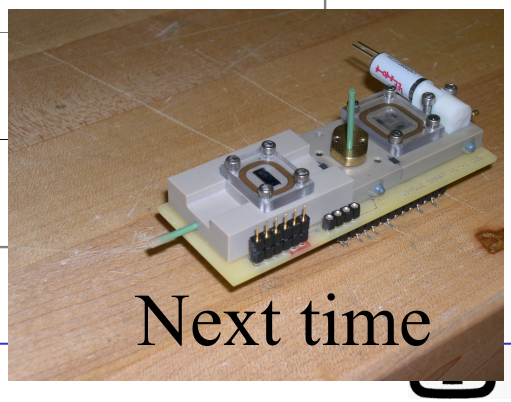
Detection with selectivity: 7 times faster at LC50 of Sarin

SPC - GC - SAW 12/16/05
Vapor System 1ppm DMMP
Cooked DKAP on SPC, DKAP on SAW





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Conclusions

- Microfabrication can be used to make a variety of preconcentrators
- Planar devices can be very fast, non-mechanical GC injectors
- 3D designs have higher capacity and can allow a broader range of analytes and/or lower concentration
- 3D designs with integrated passive valves are in testing for DARPA
- Smart PC allows for fast analysis when concentrations are the highest

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