

Micromachined Gas Chromatography Microsystem For Complex Gas Analysis

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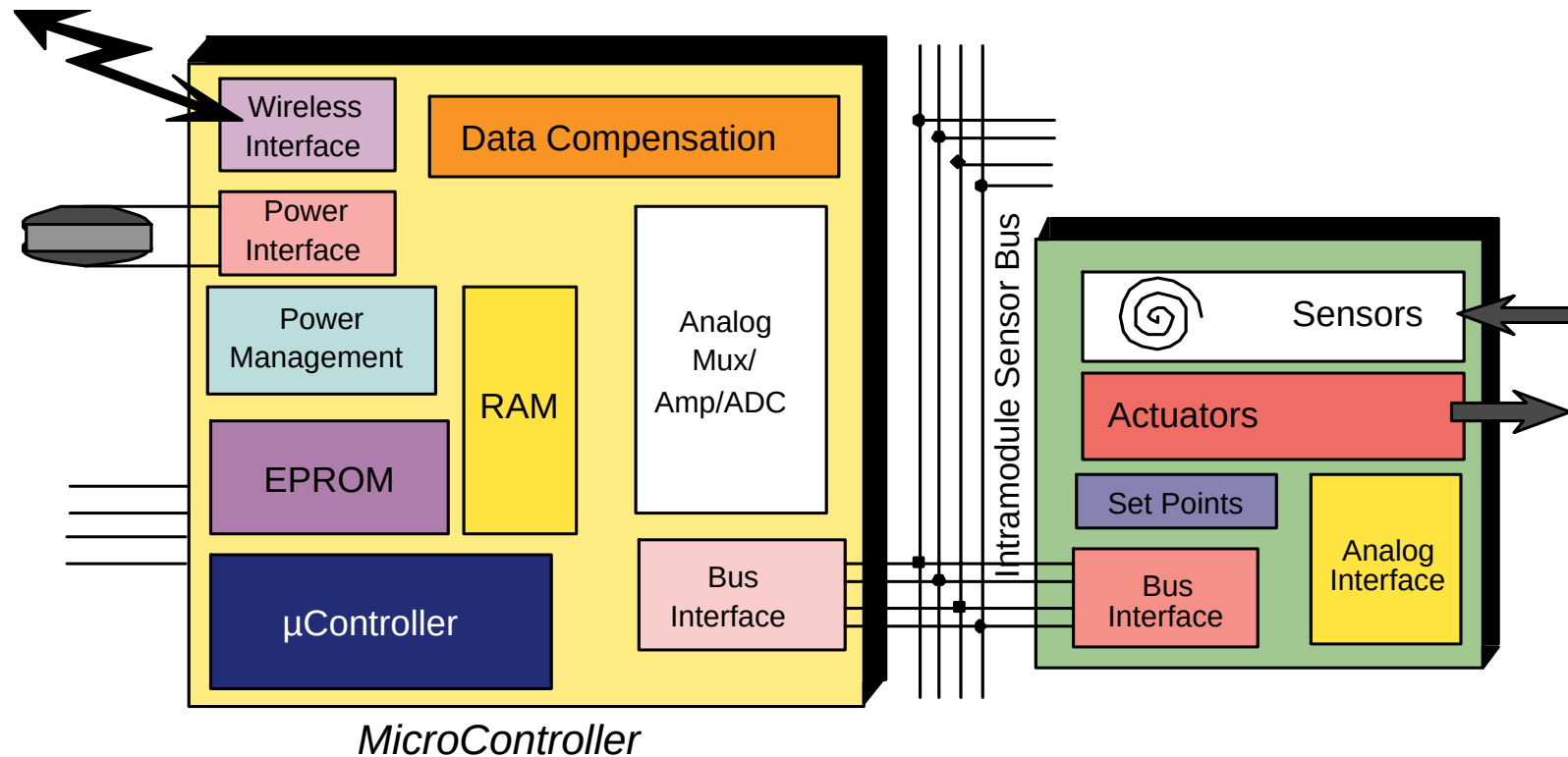
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Outline

- Wireless Integrated Microsystems (WIMS), Applications
- Gas Analysis Using μ GC
- The “**Actuator**”: Integrated Gas Micropump
- Concluding Remarks, Future Trends



Generic Architecture for Wireless Integrated Microsystems (WIMS)



Key Components:

Power Source, Micropower **MicroController** with Power Management and Data Compensation, Software, **Wireless I/O**, Integrated **Programmable Transducers** with a High-Performance Standard Interface, Hermetic **Packaging**



WIRELESS INTEGRATED MICROSYSTEMS (WIMS)

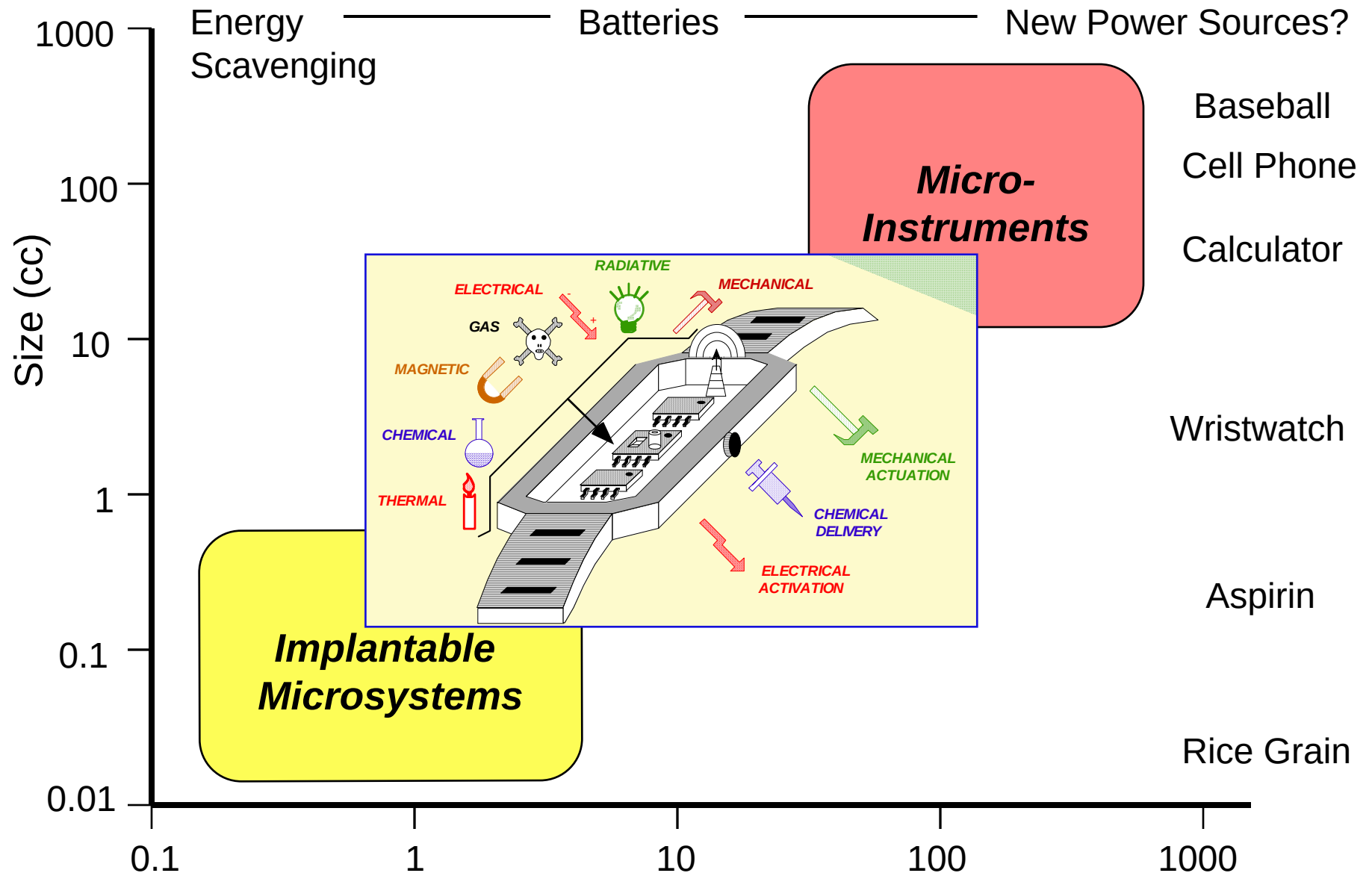
Integrated sensors and microactuators merged with micropower signal processing electronics and wireless communications on a common substrate, sometimes fabricated monolithically.

..... Bringing Together

- **Integrated Sensors and Microactuators (MEMS)**
 - **Micropower Microelectronics**
 - **Wireless Communications**



Microsystem Drivers: Power and Size



MEMS and Integrated Microsystems:

Pervasive Engineered Microsystems

Applications:

- Weather Forecasting and Environmental Monitoring
- Biomedical Systems: Diagnostic and Therapeutic
 - Homeland Security and Defense Applications
 - Communication Systems (RF and Optical)
- Consumer Electronics, Appliances, Entertainment
- Transportation Systems (vehicles, smart highways, infrastructure)
 - Adaptive Automated Manufacturing Tools (including VLSI)
 - Smart Homes and Wide-Ranging Consumer Products
 - Space Probes and Launch/Satellite Instrumentation



Sensors For Environmental Monitoring

- *Physical/Radiative Parameters*

- *Barometric Pressure*
- *Humidity*
- *Temperature*
- *Others: flow, magnetic field, visible, IR*

Capacitive sensors
Polymer-based sensors
Bandgap ckts.

- *Chemical Parameters (not yet developed)*

- *Organic Vapor Air Pollutants (EPA “189”)*
- *Inorganic Gas Air Pollutants (SO₂, NO_x)*
- *Liquid Pollutants (Heavy Metals)*

μGCs
Electrochemical
Potentiometric

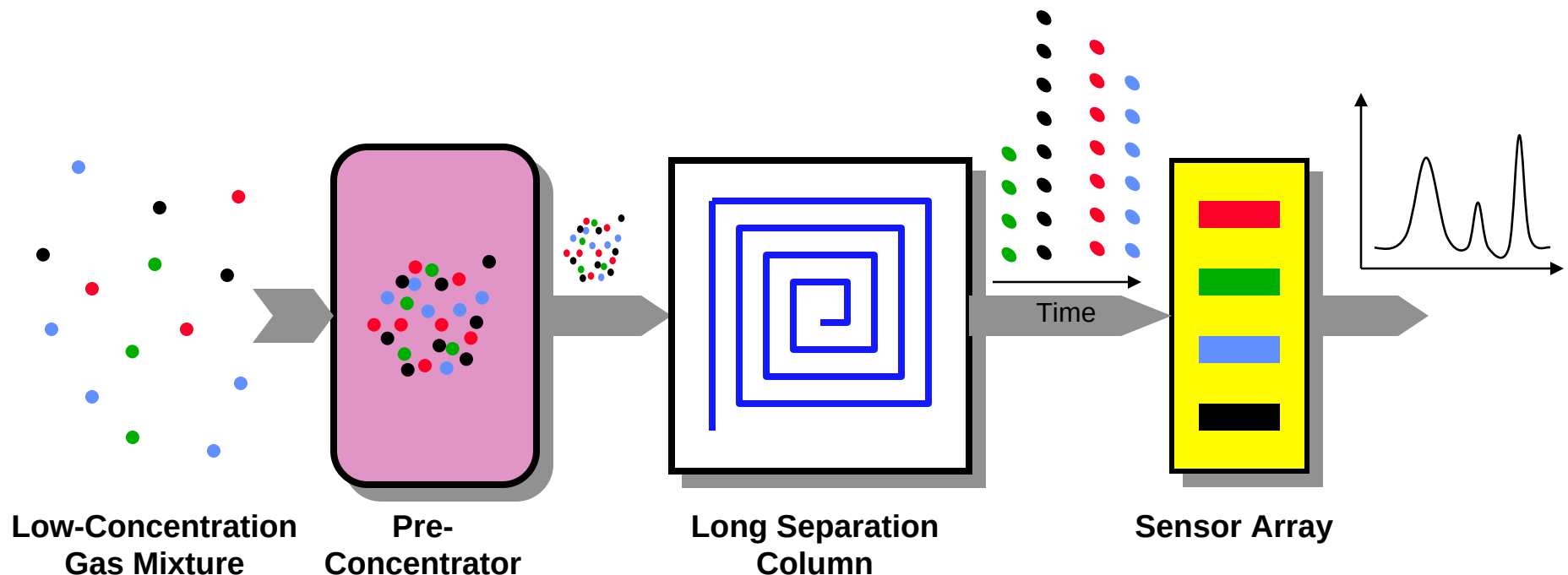
Chemical (Gas) Sensing of Air Quality *A Micro Gas Chromatograph (μGC)*

Targeting the top 45 gases from the EPA “Air Toxics” List



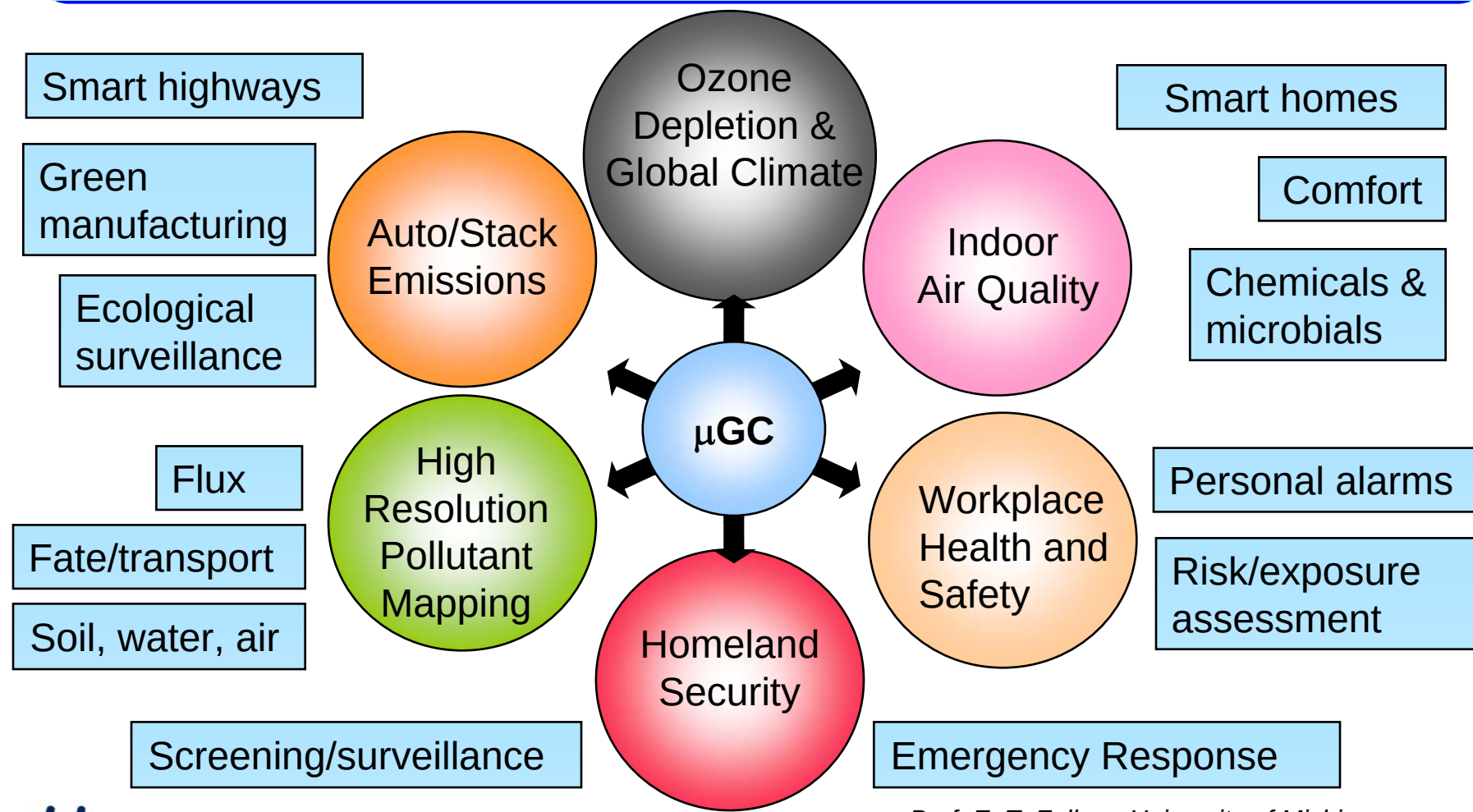
Basic Operation of a Gas Chromatograph (GC)

- Collect sample of a complex mixture of air/gas sample over some time
- Adsorb the sample onto a pre-concentrator (PC) to increase concentration
- Apply a fast heat pulse to release the adsorbed gas from the PC
- Pass concentrated plug of gas through a long tube (column) coated with a polymer
- As the complex mixture passes through the column, different species will take different time to travel through the column, and so they get separated in time
- The separated mixture is passed over a sensor array or a mass spectrometer for identification of individual components and recognition of the complex gas.



Integrated μ GC For Gas Analysis

*Versatile Microanalytical System for Trace Analysis
of Complex Mixtures of Atmospheric Pollutants*



Why Miniaturize the GC?

- **Scaling Laws (+ and –)**
 - + Low mass: rapid, **low power** heating (cooling)
 - + Narrower columns: **higher resolution** with shorter columns
 - + Lower “dead volumes”: **higher resolution** and **sensitivity**
 - + **Reduced sample** size (mass): if proper detector is used
 - + **Reduced size and weight**
 - Larger pressure drop: makes **pumping more difficult**

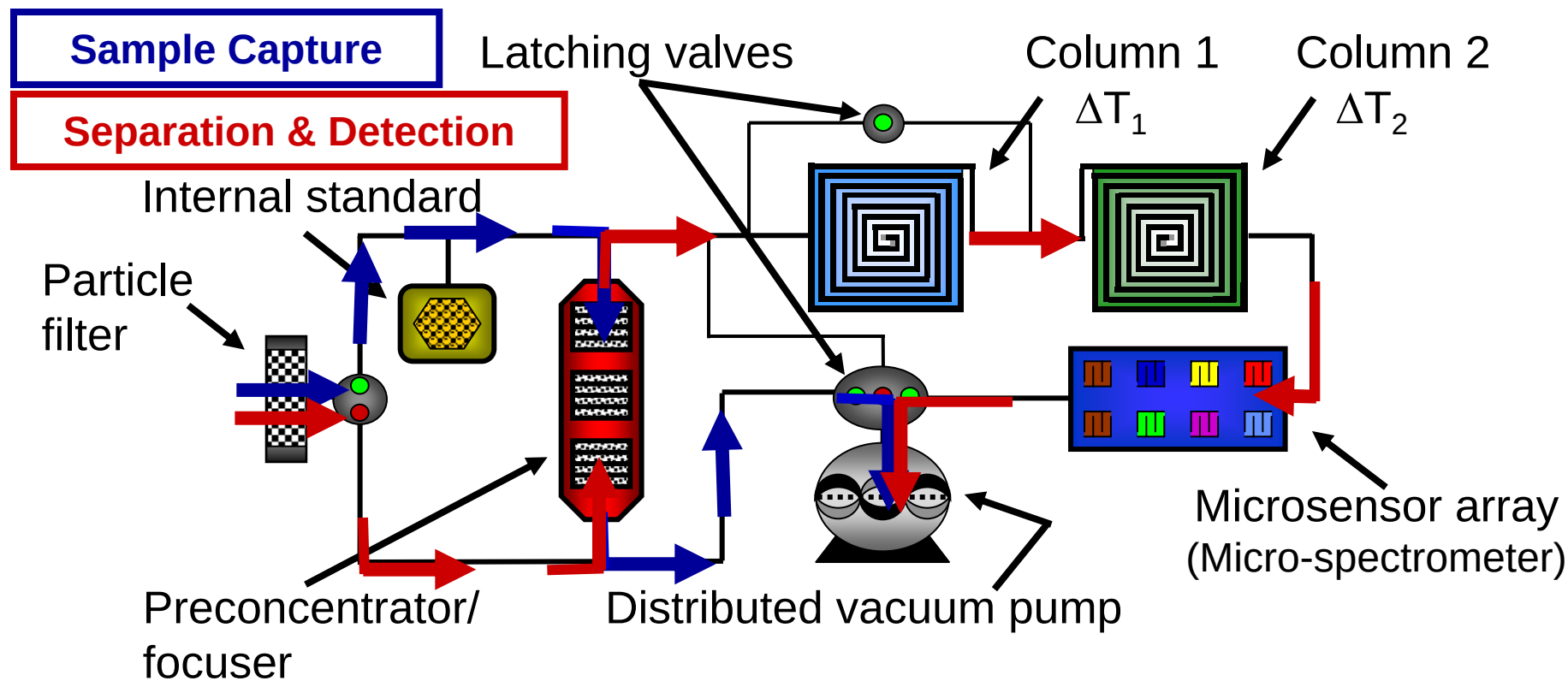


“Micro-GC” Efforts

- **1970s: First “GC-on-a-Chip” (μ GC): 1970-79 (Stanford)**
 - Terry, Jerman, Angell, *IEEE Trans. Elec. Dev.*, 1979
- **1980s: Bruns, Microsensor Technology, Inc. (MTI)**
 - Commercial “mini” GC: Micromachined injector, TCD detector, conventional column
- **1994: Kolesar, et al. (TCU)**
 - Lab prototype: ammonia, nitrogen dioxide
- **1998- : Frye-Mason, et al. (Sandia)**
 - μ ChemLab – 1st MEMS subsystem for CWAs; Lewis et al., *IEEE Sensors*, 2006
- **1998- : Spangler (Technispan)**
 - Modeling of column efficiency
- **1999: Yu, et al. (LLNL)**
 - Lab prototype: 8 lbs, 24 W
- **2000: Hesketh, et al. (GA Tech)**
 - Low-mass Parylene u-columns
- **2000: Müller, et al. (Hamburg)**
 - SLS Microtech.: commercial prototype
- **2000- : Wise, Sacks, Pang, Najafi, Zellers, et al. (U. Mich.)**
 - WIMS Center: 1st all MEMS μ GC for VOC mixtures
- **2004- : DARPA MGA Program**
 - Honeywell, Sandia, U. Illinois; ultra-small,-fast; CWA detection
- **2005: Lorenzelli et al. (U. Ferrara)**
 - Lab prototype; bio applications



Integrated Micro Gas Analyzer Based on Gas Chromatography

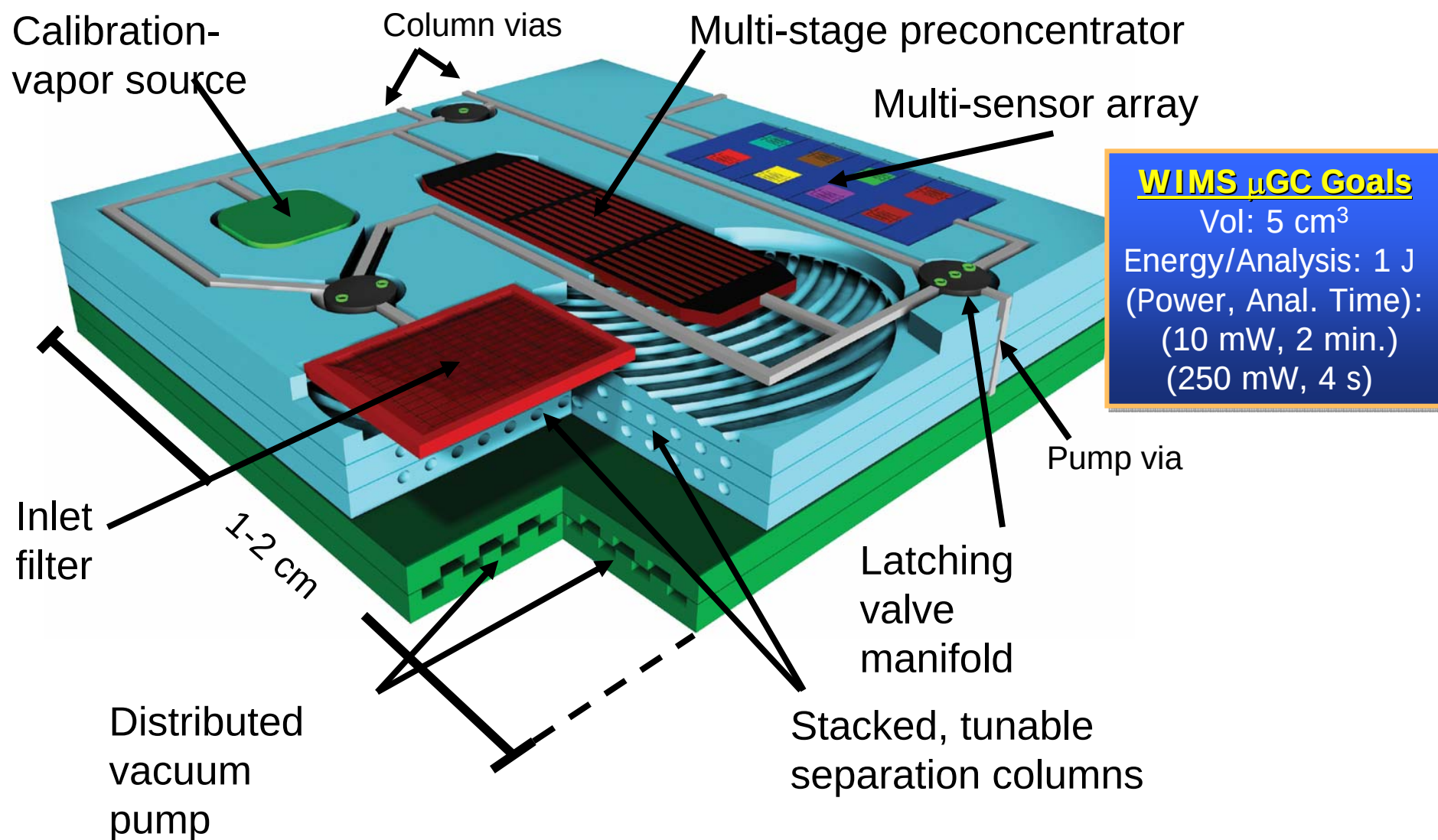


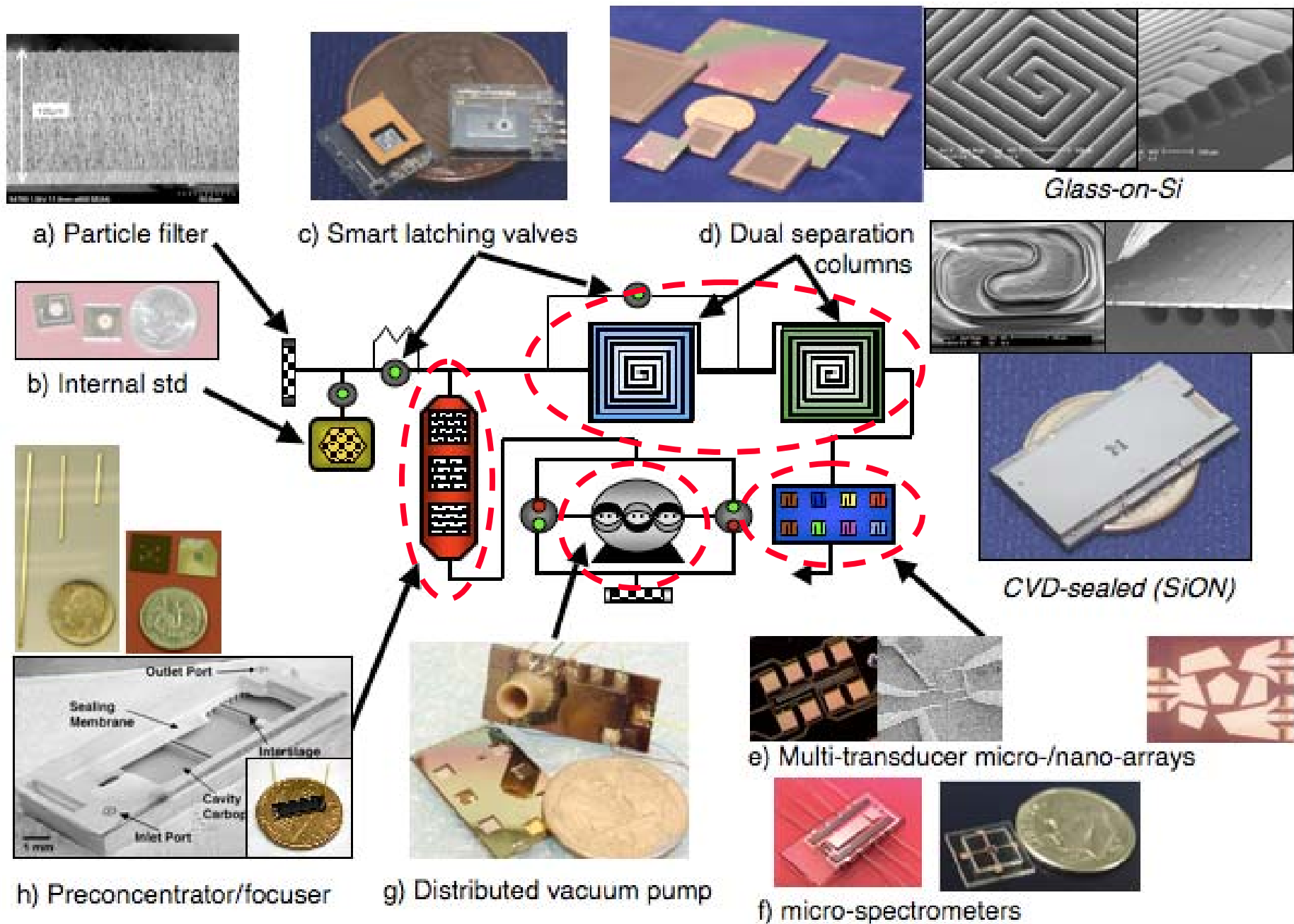
TARGETED PERFORMANCE:

- 30-50 Organic-Vapor Pollutants per Analysis
 - **Detection Levels: < 1ppb per analyte**
- **Analysis times: 1 minute (general); 5 sec (specific)**
 - Realized in 10cc and at <10mW (average)

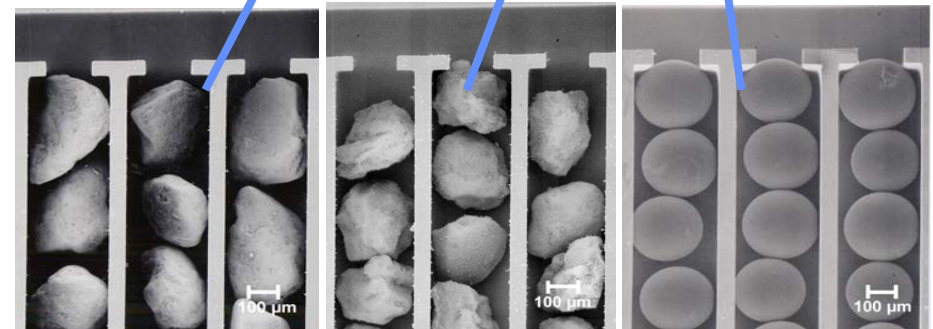
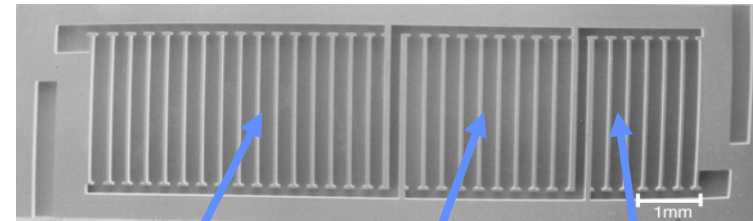
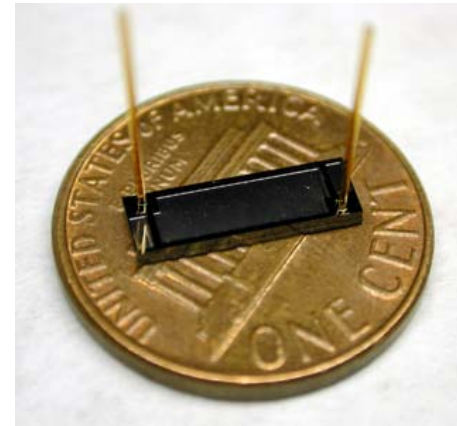
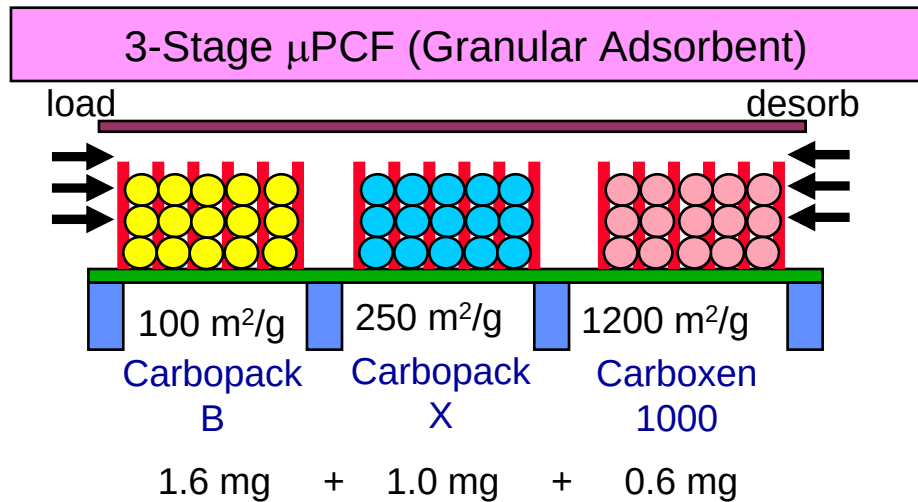


Michigan WIMS μ GC Vision





Multi-stage Preconcentrator Focuser (μ PCF)



Device Features

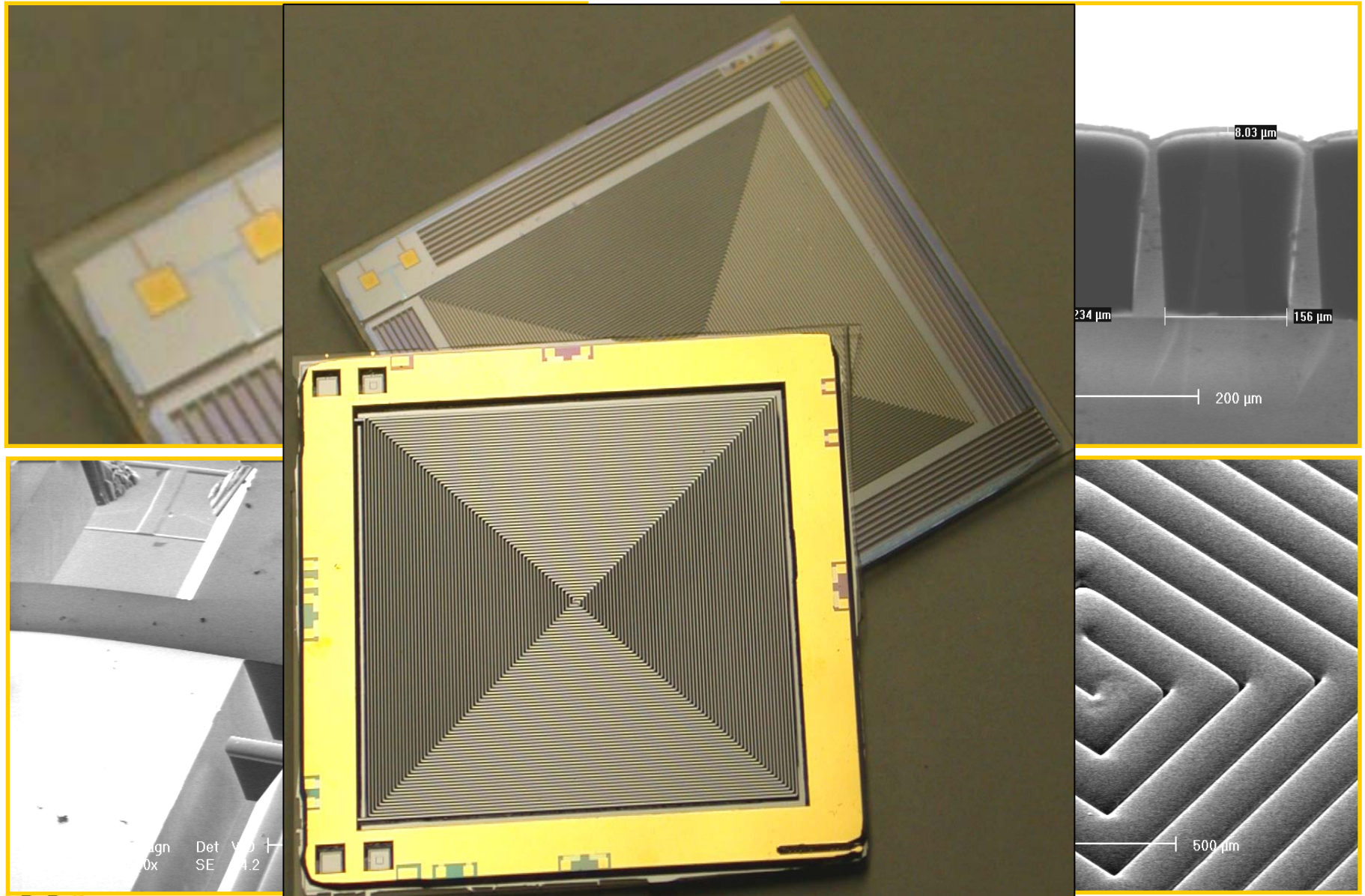
- 3 mm x 9 mm active area
- 50- μ m thick Si “floor”
- 50 x 3000 μ m slats (heat-exchangers)
- 220 μ m gaps for adsorbents
- 385 μ m tall
- **Precon factors >5000-fold**

Tian, Pang, Wise, Zellers, JMEMS, 2005



LOW-MASS SILICON SEPARATION COLUMNS

Wise, Agah, University of Michigan



THE WIMS μ GC — Gen-0.6

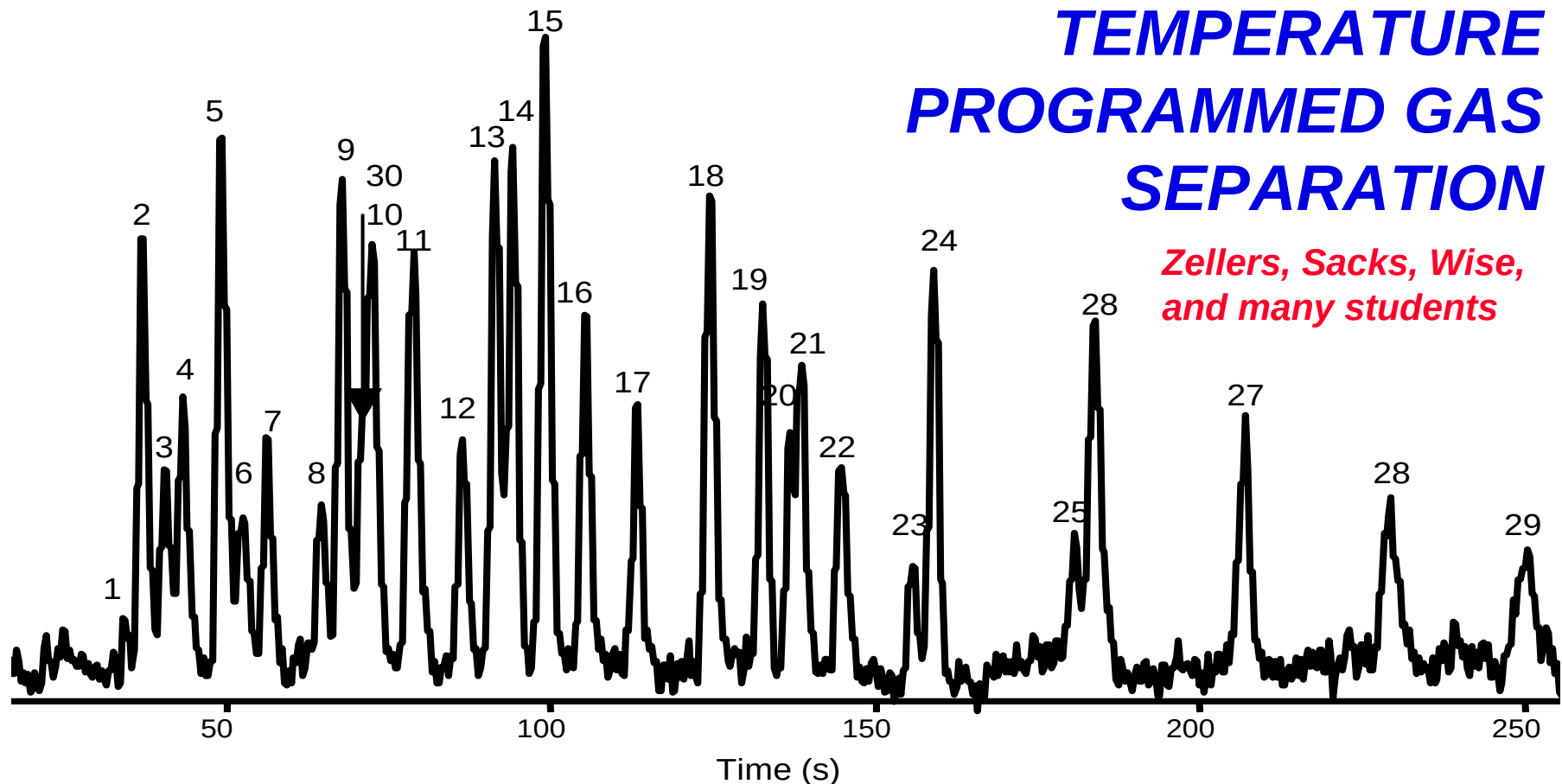
Calculator Size

THE WIMS μ GC — Gen-0.6

Calculator Size

TEMPERATURE PROGRAMMED GAS SEPARATION

*Zellers, Sacks, Wise,
and many students*

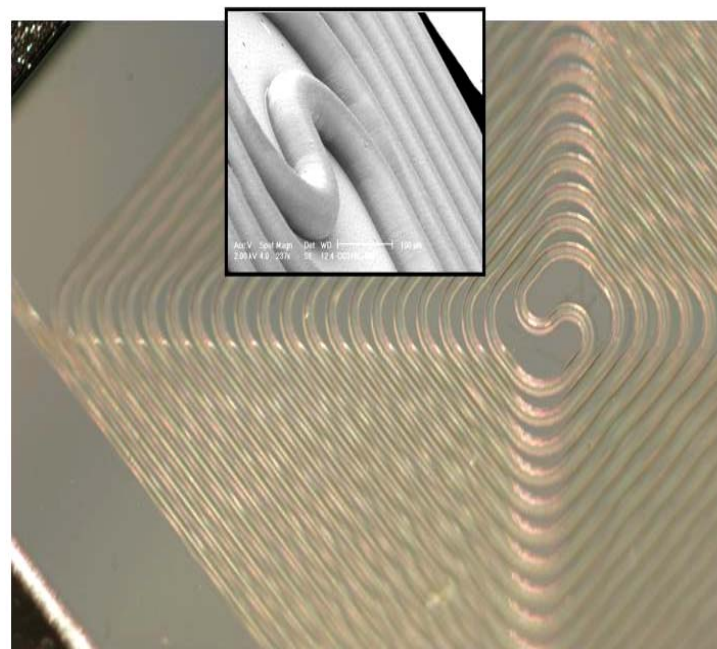
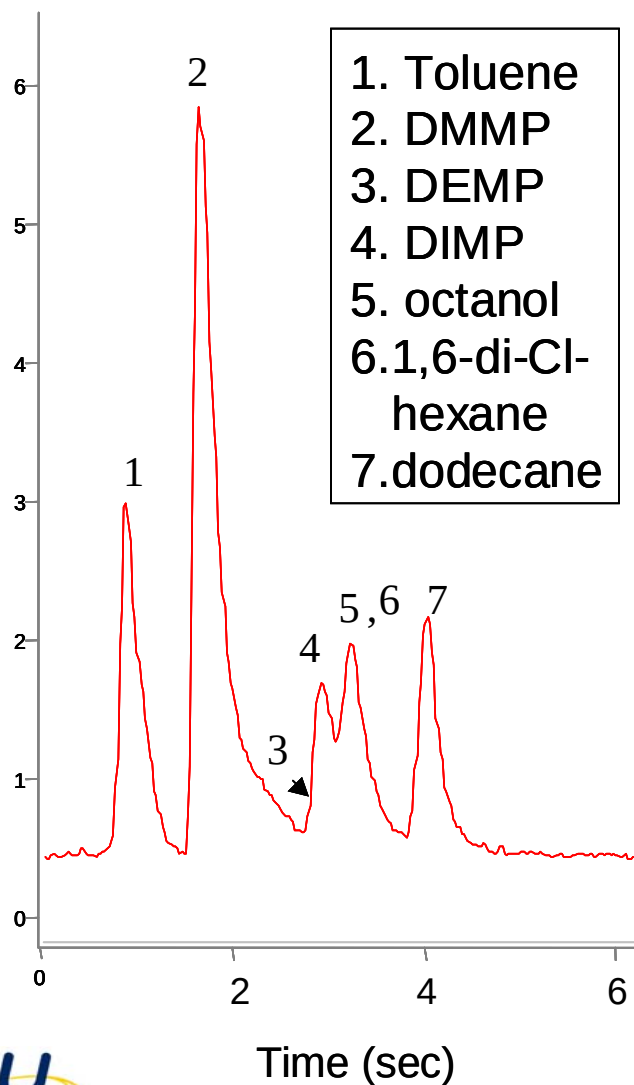


- Thirty air pollutants spanning three orders of magnitude in vapor pressure were separated in **4.2min on a single 3m Si-glass column** coated with polydimethylsiloxane and temperature programmed at 20°C/min.
- Producing **12,000 theoretical plates**, this is the **highest resolution micro-column** ever reported.



Ultra- Small, Low-Power, and Fast Micromachined Separation Columns for Fast Detection of Chemical Warfare Agents

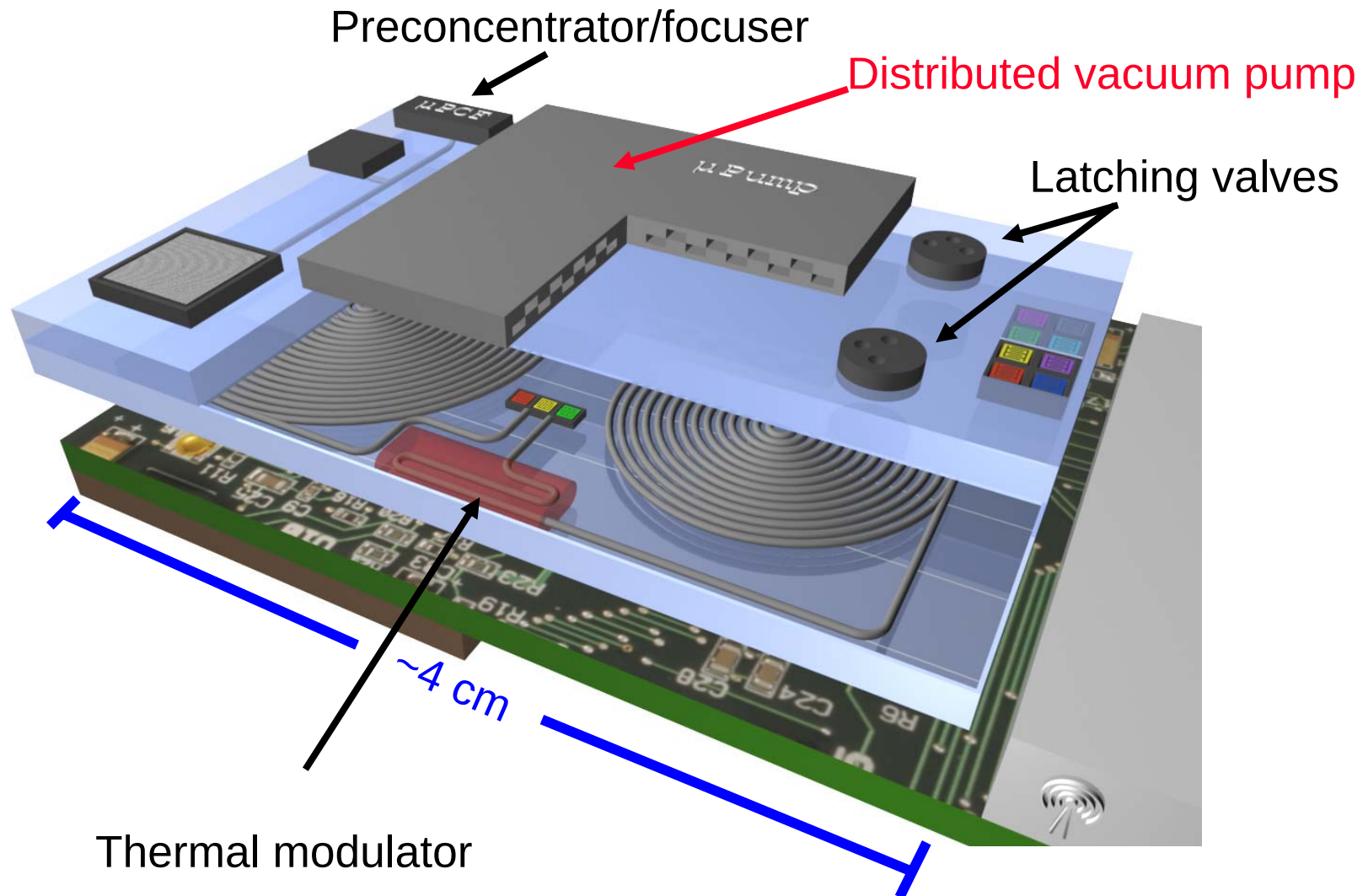
Agah, Potkay, Wise, Zellers, Sacks,...



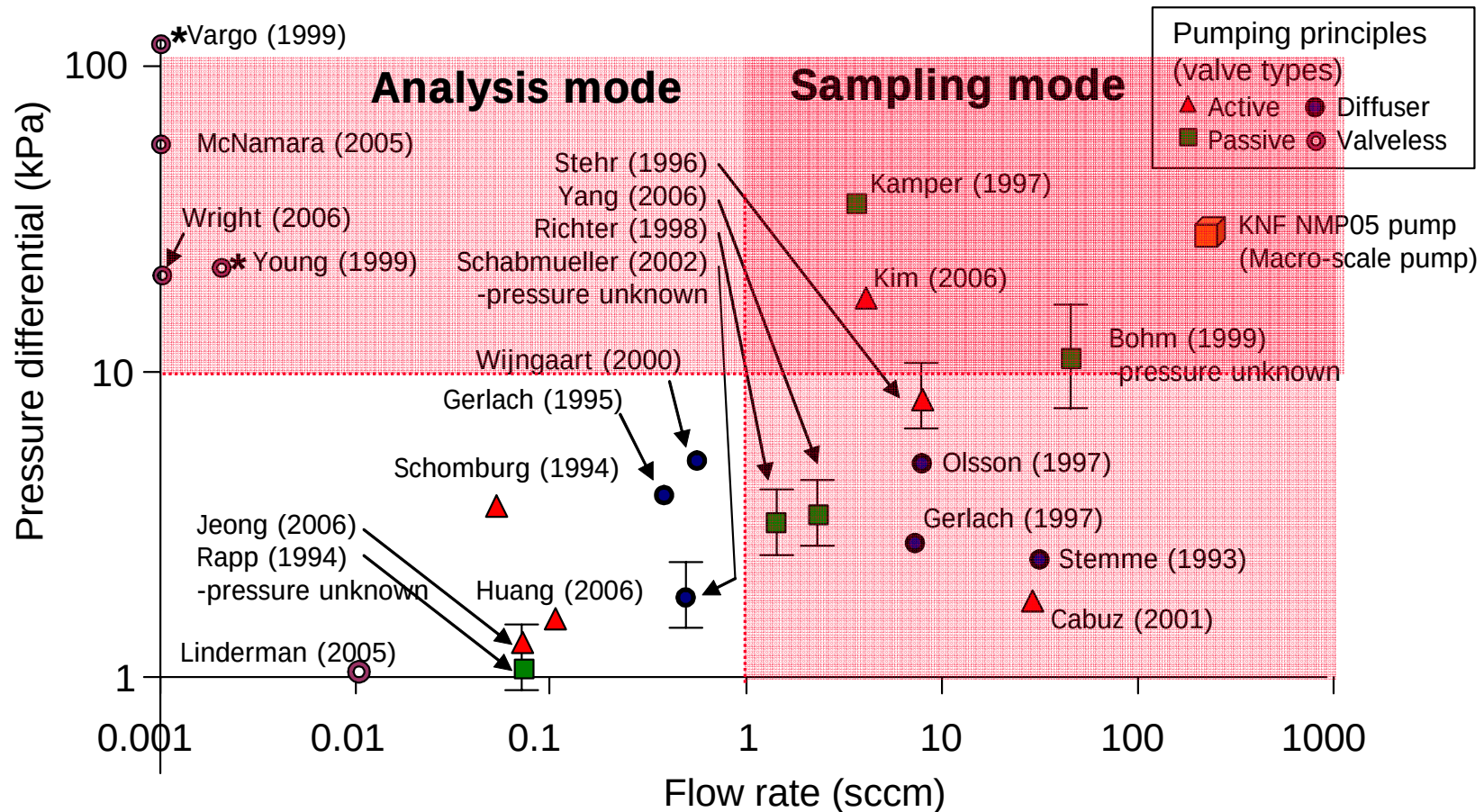
- Using **25cm** silicon-glass columns programmed at 1600°C/min, a seven-component mixture of chemical warfare simulants can be separated in **4 seconds**
- CVD-sealed ultra-low-mass columns (above) promise still faster responses using entirely new column architectures (2D GC)



WIMS μ GC Actuators



Summary of Previous Gas Micropumps



- **No previous gas micropump meets the WIMS μ GC requirements.**
- **Size and power consumption are not included in the graph.**
- Low flow rate $\leftarrow$ small stroke volume, slow op., gas compressibility.
- Low pressure $\leftarrow$ weak force of a membrane, leakage.
- Single mode operation



Integrated Multi-stage Gas Micropump

- **Goal: Develop a miniature micropump for the μ GC:**

- **Flow Rate:** 2-50sccm, For Pressures: 0.2 to 0.5 Atm
- **Power:** <100mW
- **Size:** <1cm x 1cm x 2mm

- **Approach:**

- **Peristaltic multi-stage**

- ↳ High pressure

- **Electrostatic actuation:**

- ↳ Fast, Low-Power

- **Double-sided curved electrodes:**

- ↳ Large displacement

- ↳ High Flow

- **Polymer Membranes**

- ↳ Large displacement

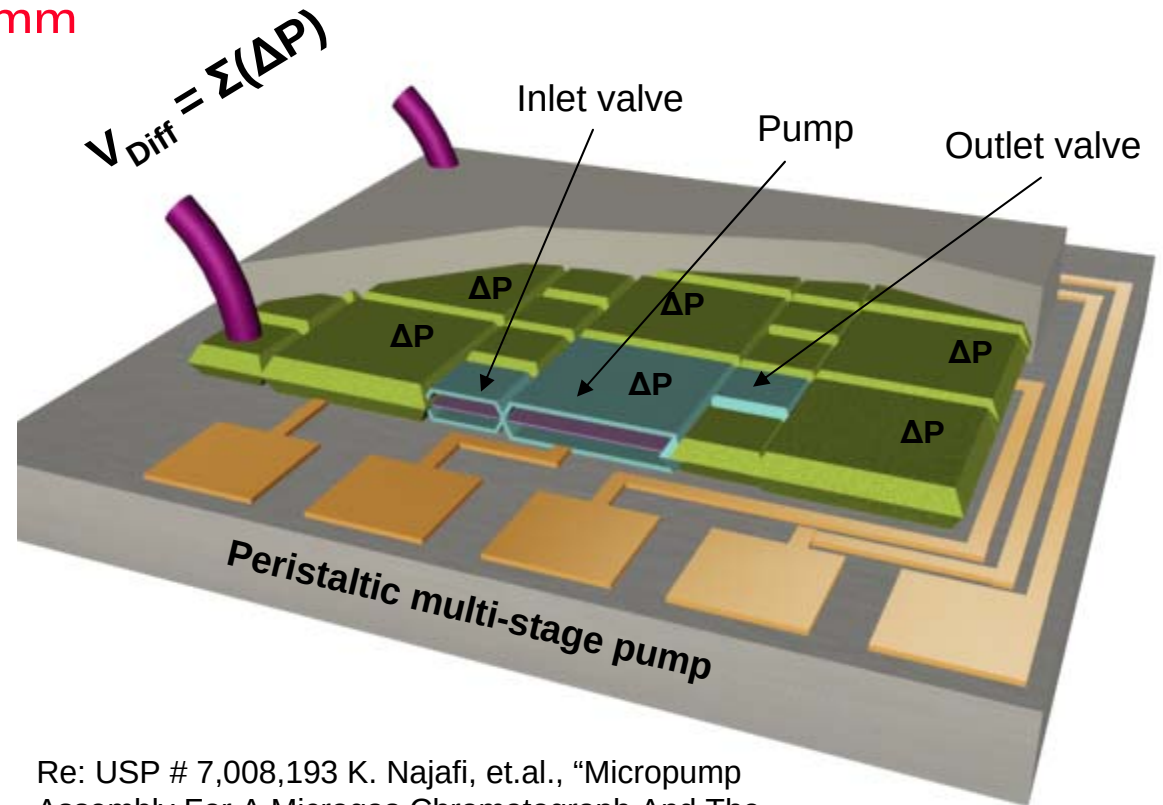
- ↳ Low-Power

- **Resonant Operation**

- ↳ High Flow

- ↳ Low-Power

Fluidic Bucket Brigade: High Flow, Low-Pressure Per Stage



Re: USP # 7,008,193 K. Najafi, et.al., "Micropump Assembly For A Microgas Chromatograph And The Like", March 7, 2006

The Operation of a Single Stage of the Pump

- Two bonded Si wafers, sandwiching pump & valve membranes
- One membrane and two valves for each two pump chambers
- Checker-board active valves, dual electrode pull-pull electrostatic drive

Electrostatic
Actuation

Active Micro
Valves

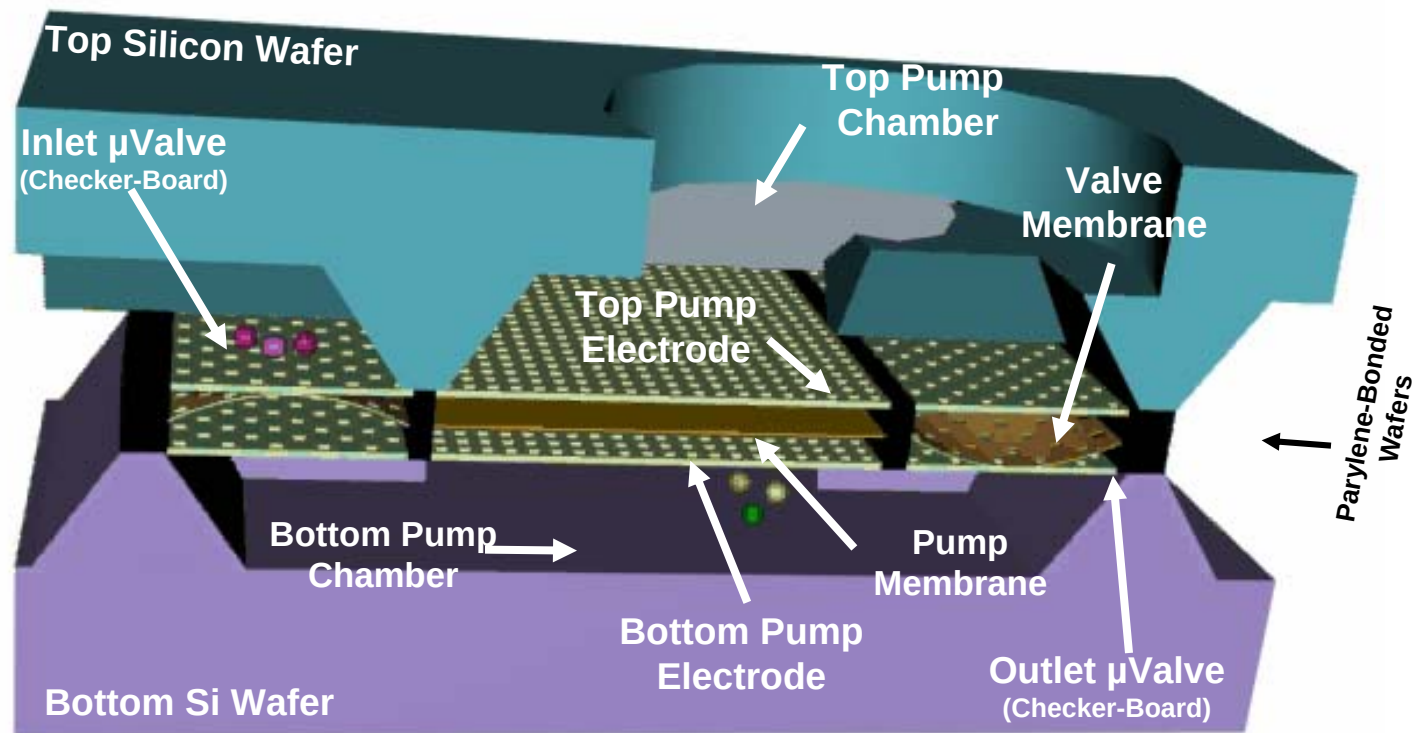
Multi-Stage
Design

Polymer
Membrane

Dual-Electrode
Actuation

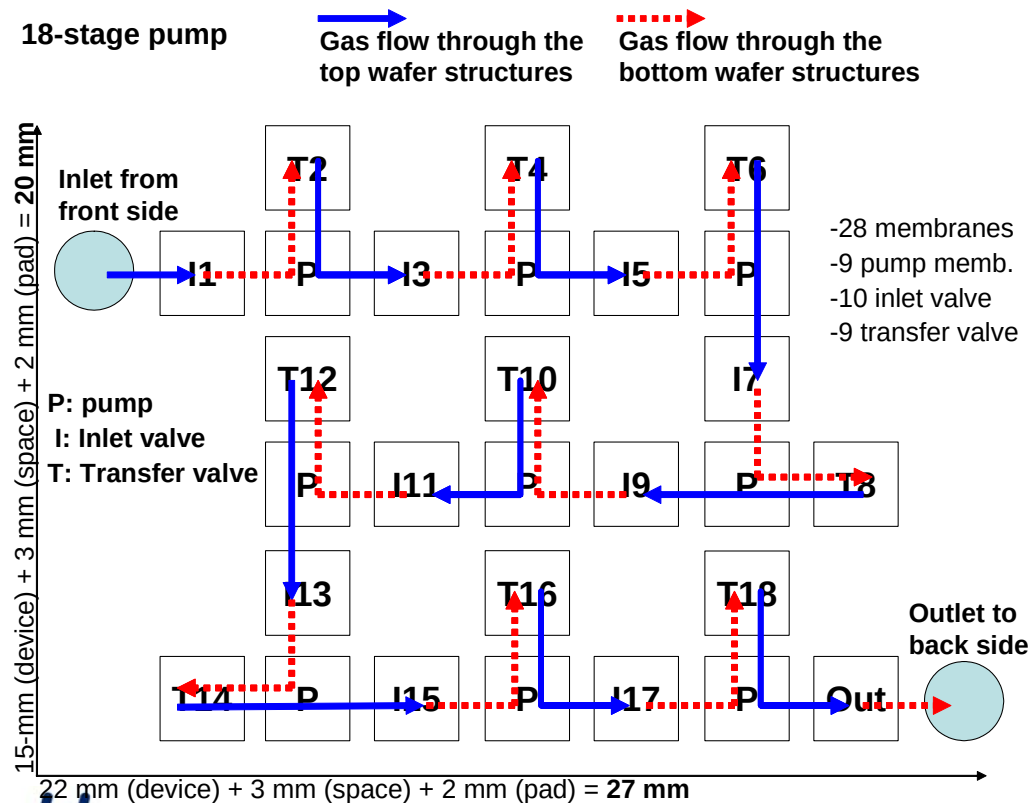
Dual-Chamber
Layout

Curved
Electrode

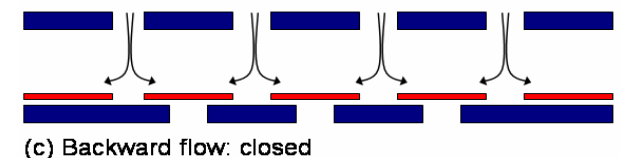
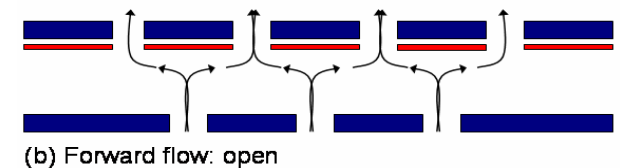
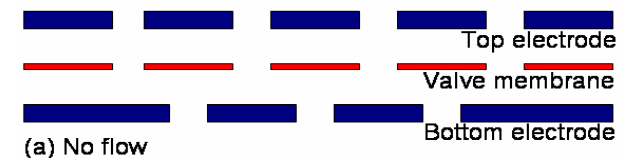
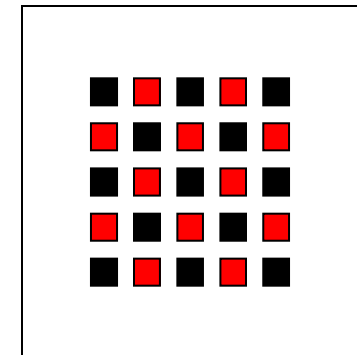


Multi-Stage Layout and the Microvalves

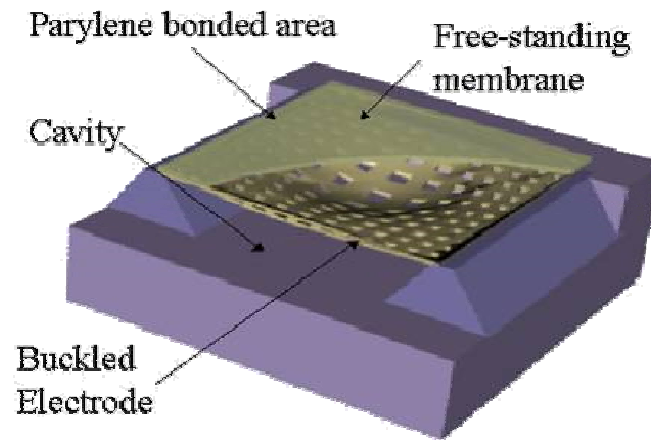
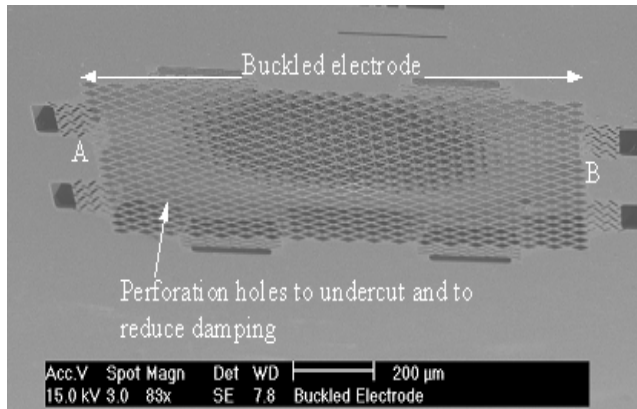
- The multi-stage pump can be layed out to generate any number of stages needed.
- Layout of 18-stage pump shown below. Two-, four-, and 18-stage pumps have been designed and fabricated.
- Gas flow is controlled by the integrated checkerboard microvalves shown on the right.



- Hole on top electrode and valve membrane
- Hole on bottom electrode



Microfabrication and Technologies



Parylene Wafer Bonding

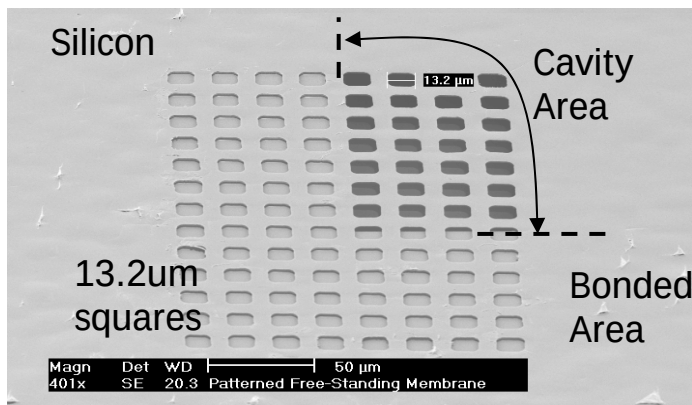
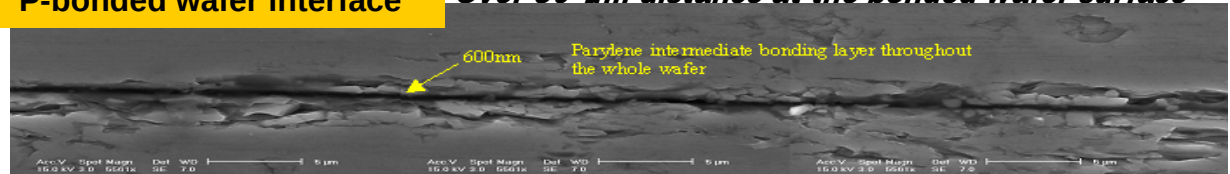
- Low-Temp, <230°C
- Thin Layers (<0.5µm)
- Reliable, No Voids

Curved Electrodes

- Efficient Electrostatic Drive
- High Force, Low-Voltage
- No Need for Special Techn.

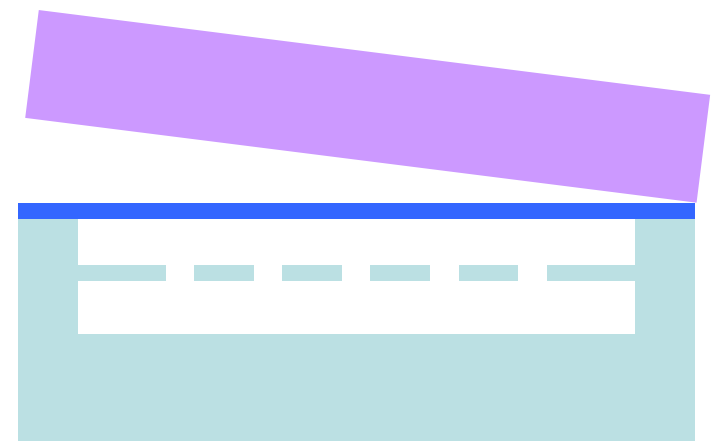
P-bonded wafer interface

Over 50-µm distance at the bonded wafer surface

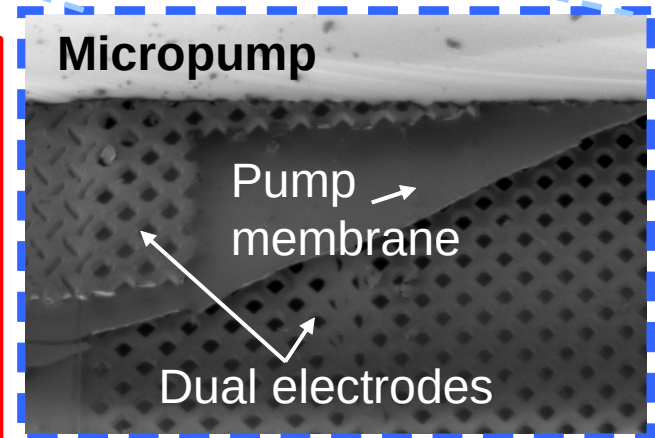
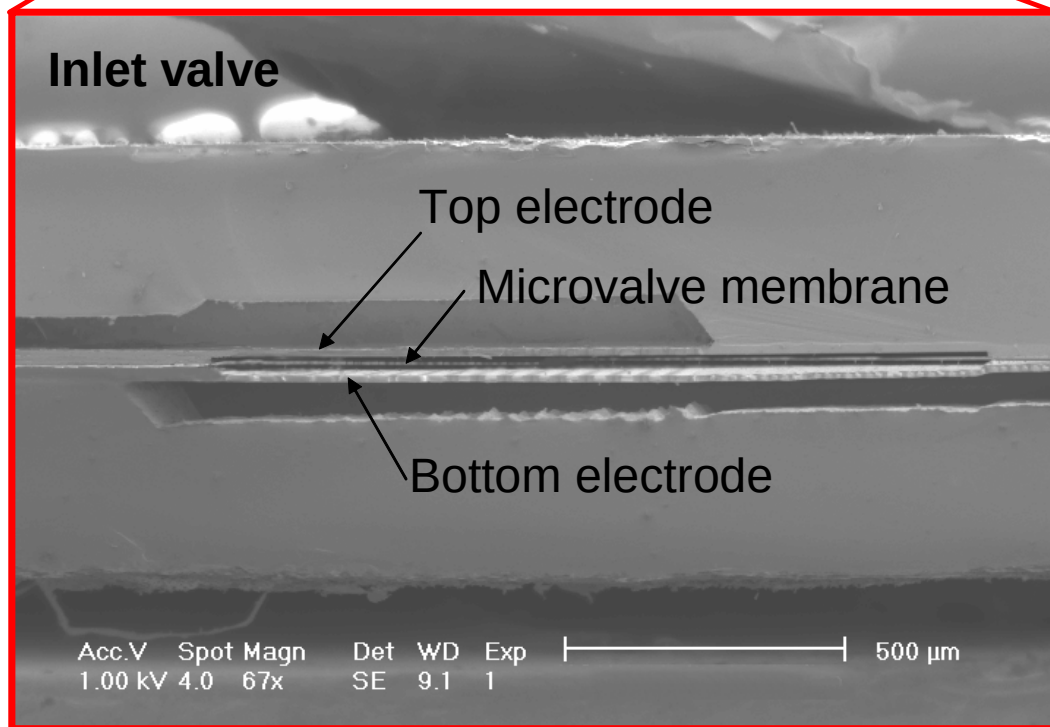
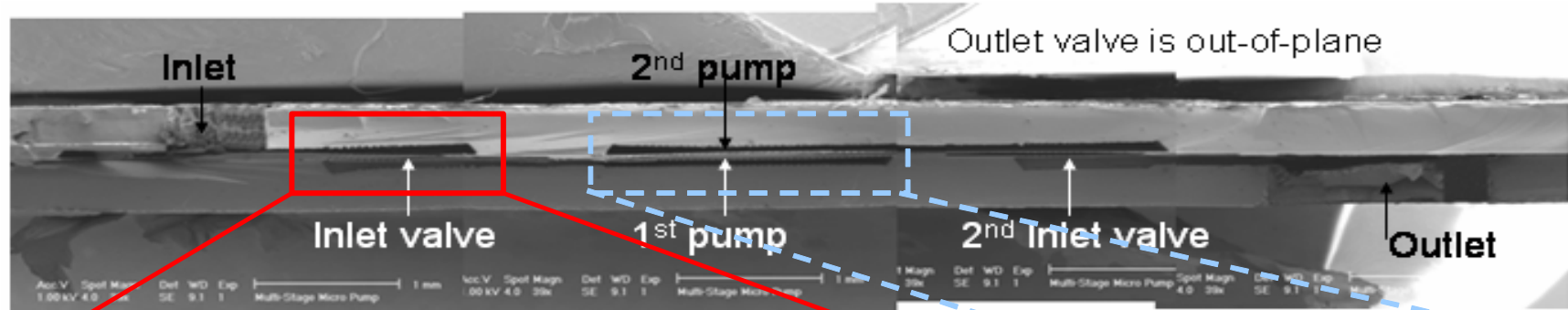


Parylene Membrane Transfer

- Wafer-Level
- High-yield (>90%)
- Thin films, over deep cavities

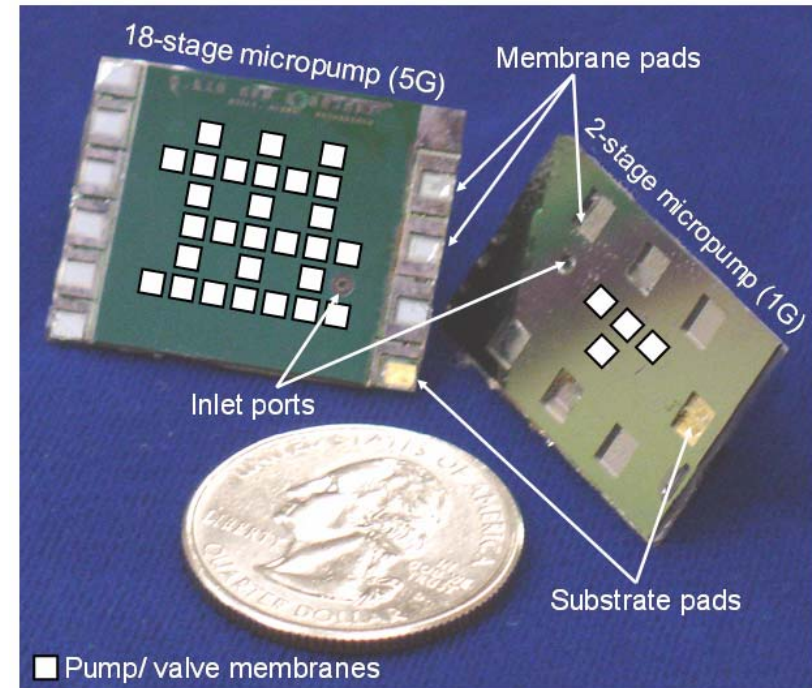
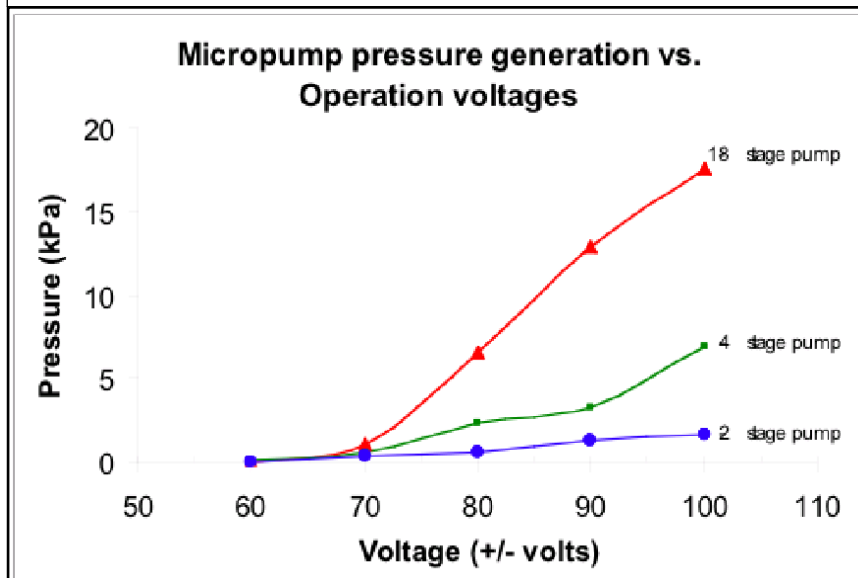
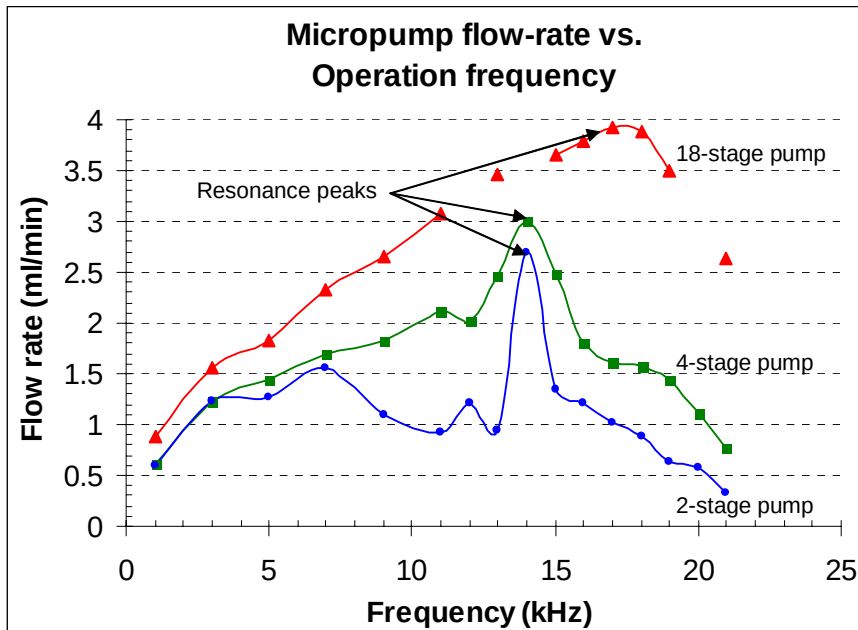


Microfabrication and Technologies



- A complete fluidic path through two wafers
- Dual-electrodes
- Polymer membrane

A Multistage Peristaltic MEMS Pump for a μ GC



18-stage peristaltic micropump

- Volume = 3.8 cm^3
- Active timing control of microvalves
- 17 kHz operating frequency
- Produces air flow rates of $4 \text{ cm}^3/\text{min}$
- Generates pressures up to 18 kPa
- Total power dissipation of $\sim 57 \text{ mW}$
- Highest pressure of any micropump

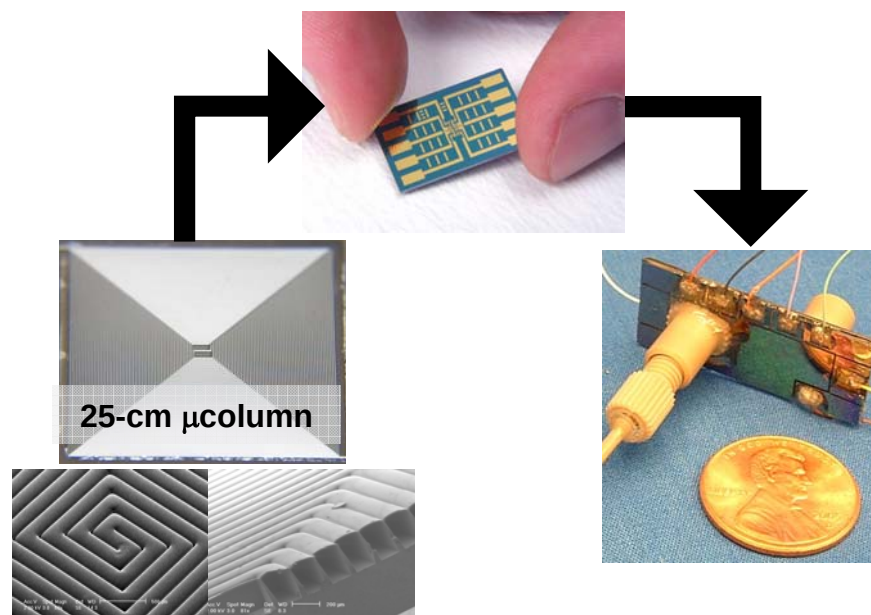
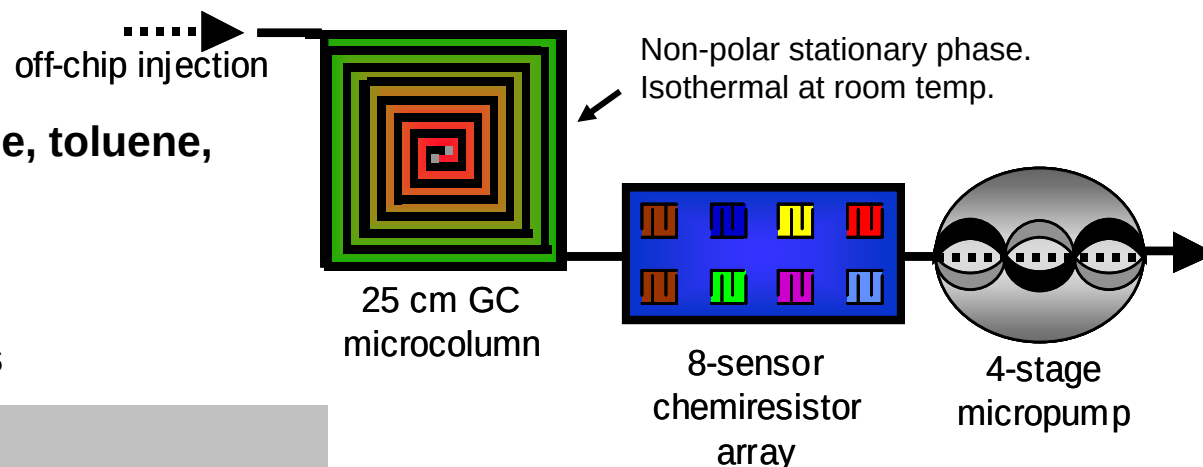
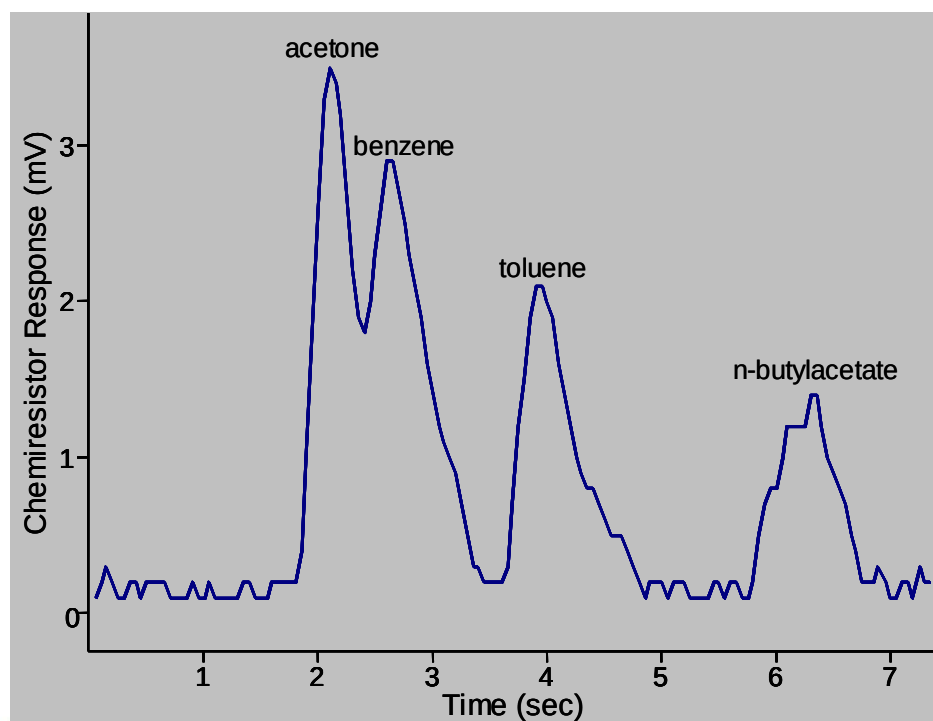
Pumping Air Bubbles



Integration of WIMS μ Column, μ Array, and μ Pump (μ CAP Subsystem)

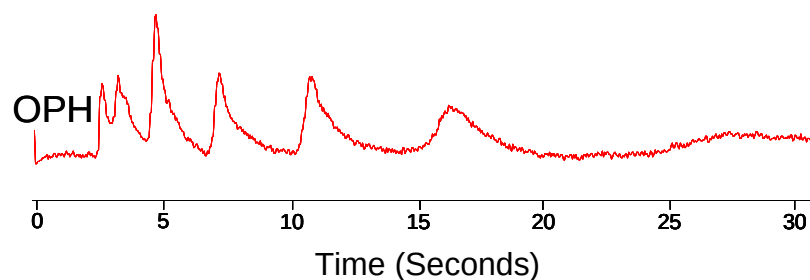
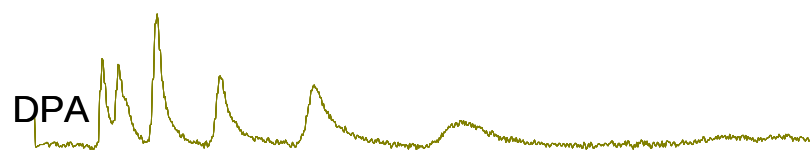
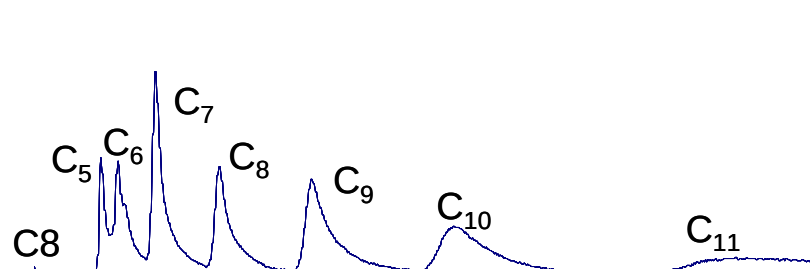
Ultimate Application: First Reported!
Testing of a Gas Micropump In a μ GC

- 4 VOCs: acetone, benzene, toluene, butyl acetate
- flow rate = 0.25 cc/min
- press. diff. = 3.5kPa
- analyt. cycle = 7 seconds

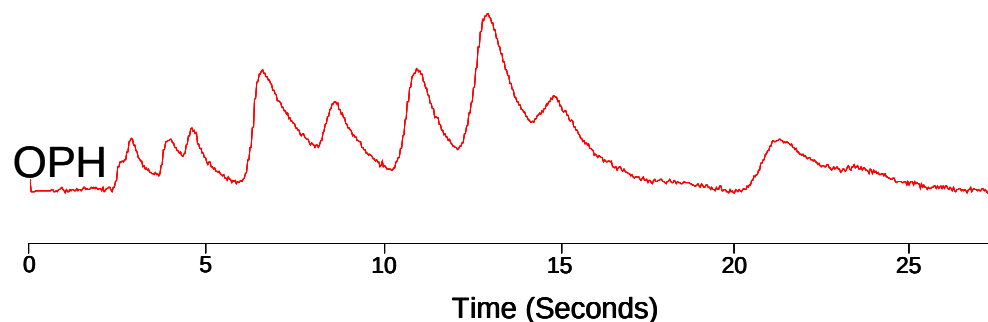
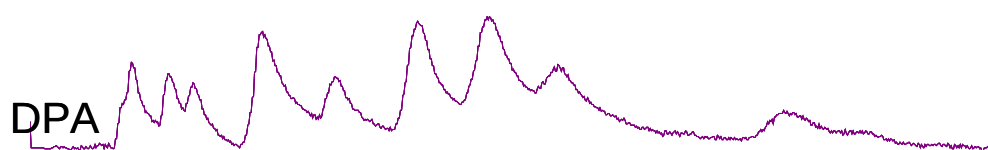
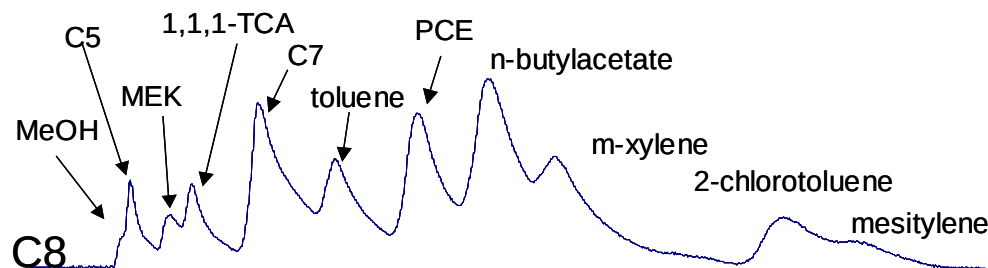


Latest Results from μ CAP Subsystem

High-Speed, Temperature-Programmed Vapor Separations



Separation of 7 alkanes
in < 30 sec



Separation of 11 VOCs
in < 25 sec



From Sensors/Actuators to Instruments

Integrated Sensor/Actuator ***Integrated Instruments***

- | | | |
|--|--|--|
| • Work on one parameter |  | • Monitor multiple parameters |
| • Customized |  | • Generic for broad applications |
| • Limited Selectivity, specificity, sensitivity |  | • High Selectivity, Specificity, Sensitivity |
| • Limited Dynamic Range |  | • Wide Dynamic Range |
| • Only Senses |  | • Measures and Monitors |
| • Robustness hammered out of device, material, process |  | • Robustness Delivered by μ System |
| • One device at a time |  | • Many devices, redundancy, range, |
| • Priced as commodity |  | • Priced as an Instrument, now, but... |



From MEMS ..to.. Micro-Instruments ..to.. Micro-Systems

Chip-Scale Instruments

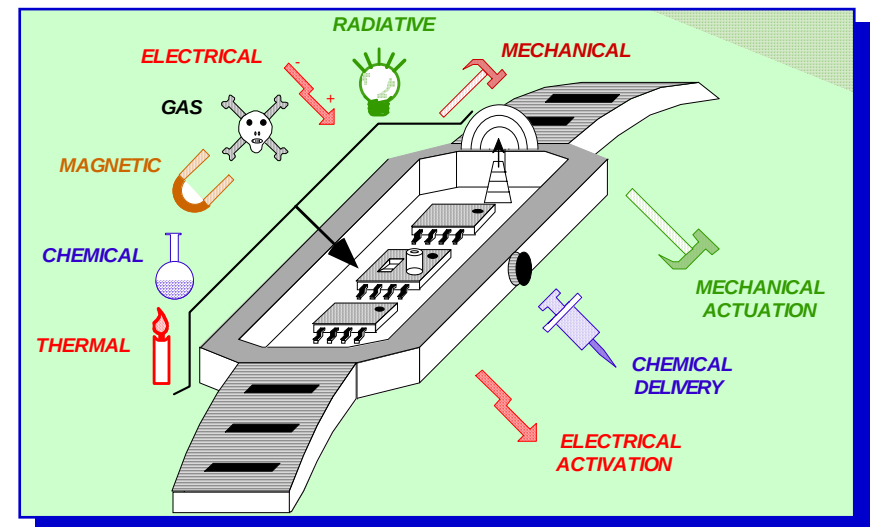
CSI-DARPA

MEM Devices

- Sensor
- Actuator
- Resonator
- Package
- Microstructure
- ...

μ Instruments

- Atomic Clock
- μ Mechanical
- Comm/Processing
- Radiation Detectors
- Gas Analysis
- Chemical/Biological Analysis
- Warfare agents
- ...



- Smaller
- Lighter
- Better
- Cheaper



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 - Joe Potkay, Masoud Agah
 - Khalil Najafi (Deputy Director)
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 - Stella Pang
 - Wei-Cheng Tian, Helena Chan
 - Yogesh Gianchandani
 - Bhaskar Mitra
- Mechanical Engineering
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- Chemistry
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