Micromachined Gas Chromatography Microsystem For Complex Gas Analysis

Khalil Najafi

Center for Wireless Integrated MicroSystems (WIMS)

UNIVERSITY OF MICHIGAN

WIMS
NSF • ERC
Wireless Integrated Microsystems

Ann Arbor, Michigan 48109-2122
Tel: (734) 763-6650, FAX: (734) 763-9324
najafi@umich.edu
www.wimserc.org

MTO DARPA MICROSYSTEMS TECHNOLOGY OFFICE

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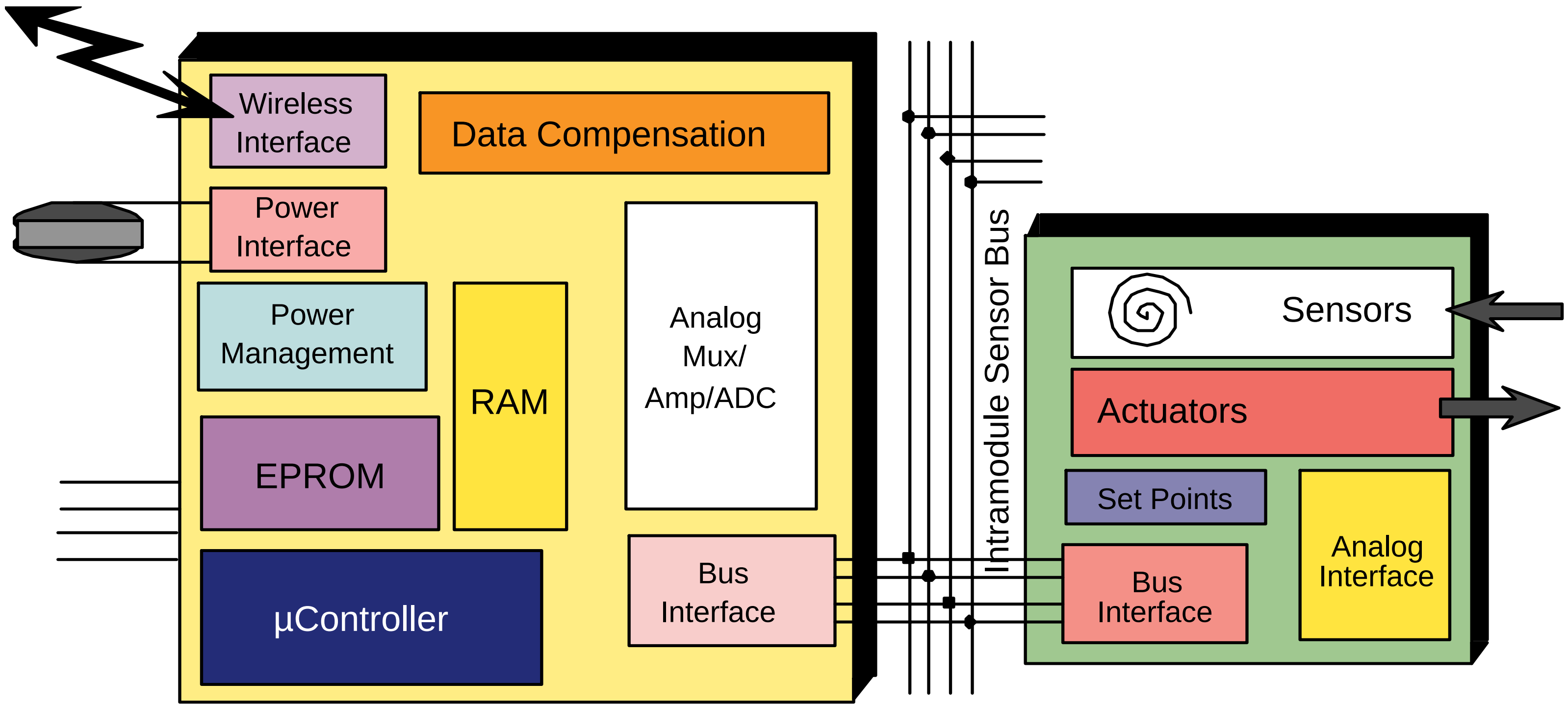
Outline

- Wireless Integrated Microsystems (WIMS), Applications
- Gas Analysis Using µGC
- The "Actuator": Integrated Gas Micropump
- Concluding Remarks, Future Trends

Generic Architecture for Wireless Integrated Microsystems (WIMS)



MicroController

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

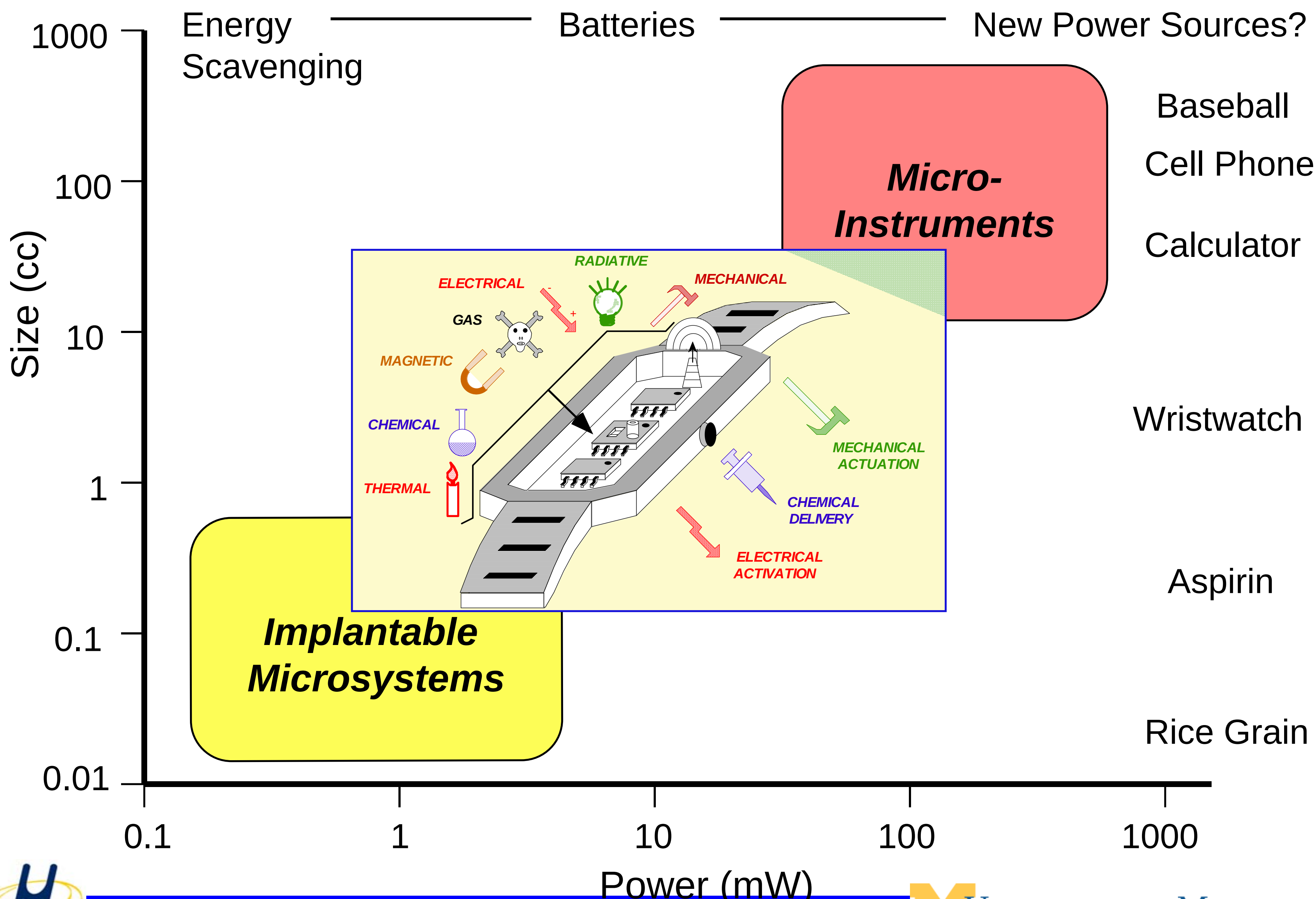
WIRELESS INTEGRATED MICROSYSTEMS (WIMS)

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*** — ***Capacitive sensors***
  - ***Humidity*** — ***Polymer-based sensors***
  - ***Temperature*** — ***Bandgap ckts.***
  - ***Others: flow, magnetic field, visible, IR***

- *Chemical Parameters (not yet developed)*
  - ***Organic Vapor Air Pollutants (EPA "189")*** — ***µGCs***
  - ***Inorganic Gas Air Pollutants (SO_2, NO_x)*** — ***Electrochemical***
  - ***Liquid Pollutants (Heavy Metals)*** — ***Potentiometric***

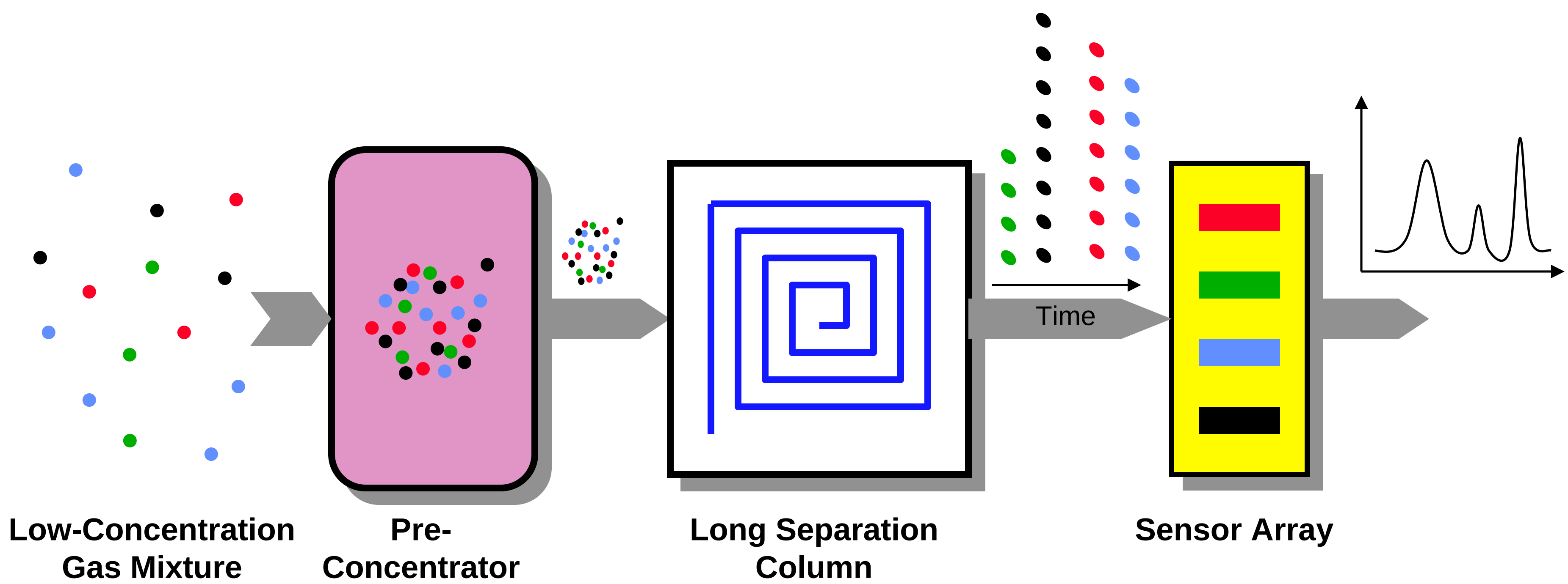
Chemical (Gas) Sensing of Air Quality

A Micro Gas Chromatograph (µGC)

Targeting the top 45 gases from the EPA "Air Toxics" List

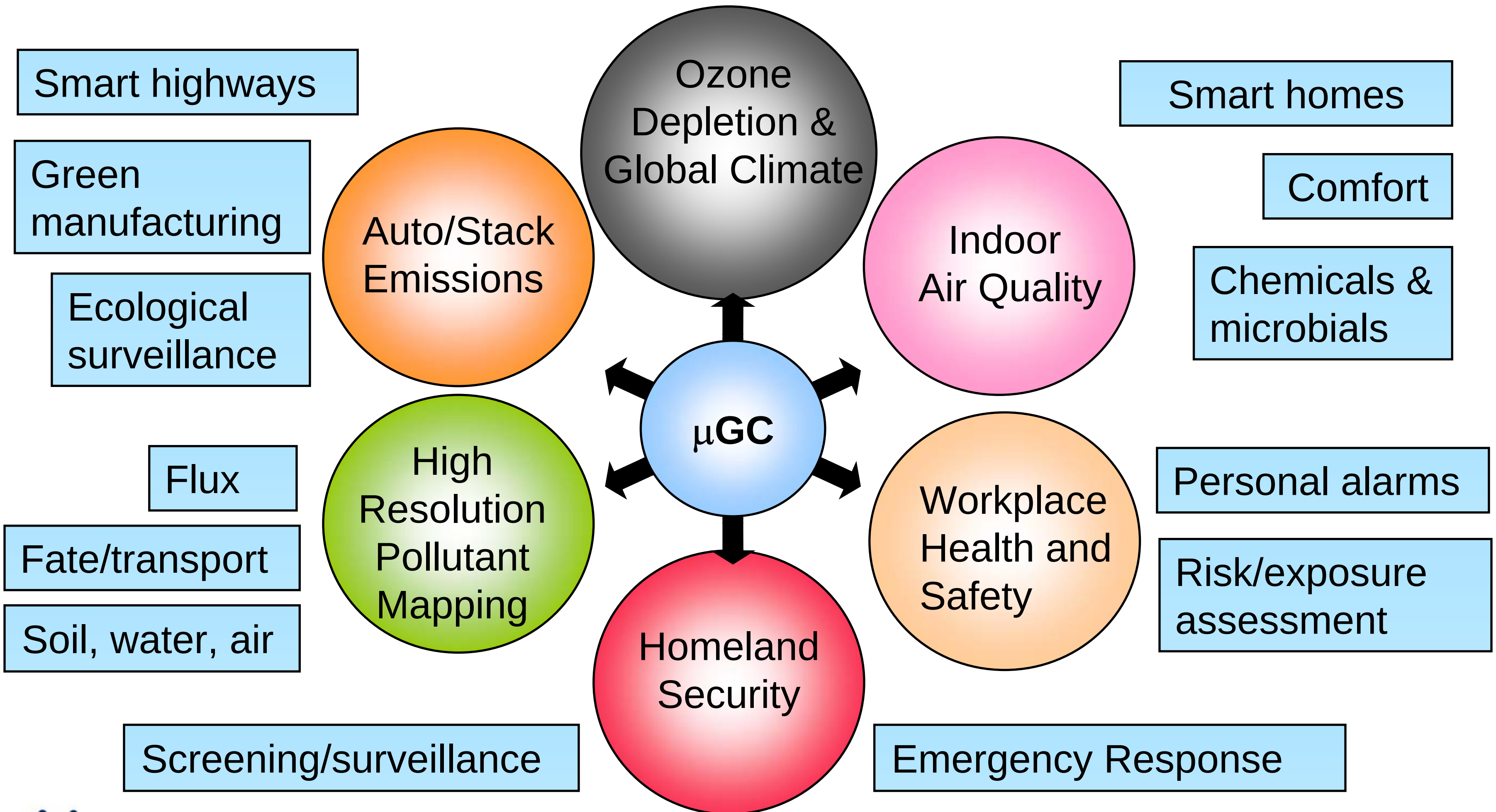
Basic Operation of a Gas Chromatograph (GC)

- 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 μGC For Gas Analysis

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



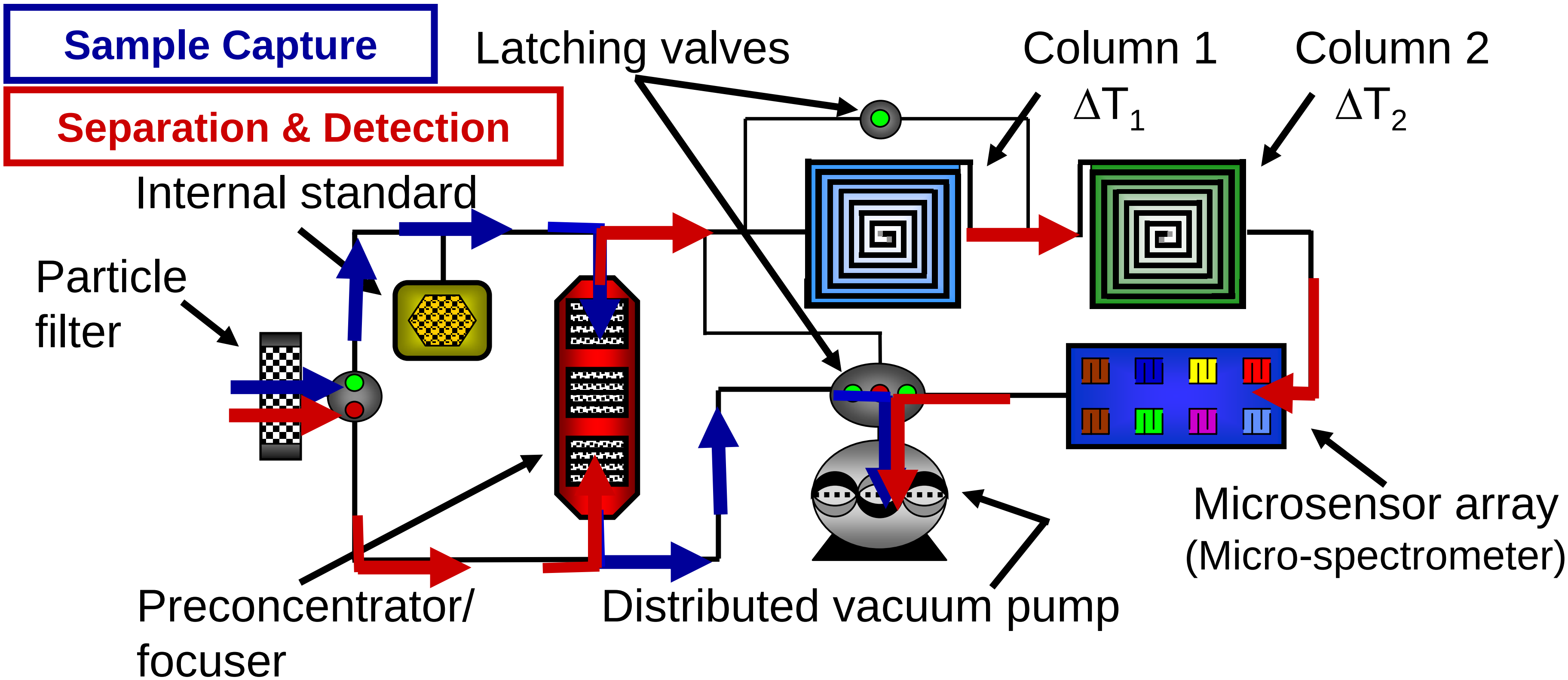
Why Miniaturize the GC?

- **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" (μGC): 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)**
  - μChemLab – 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 μGC 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

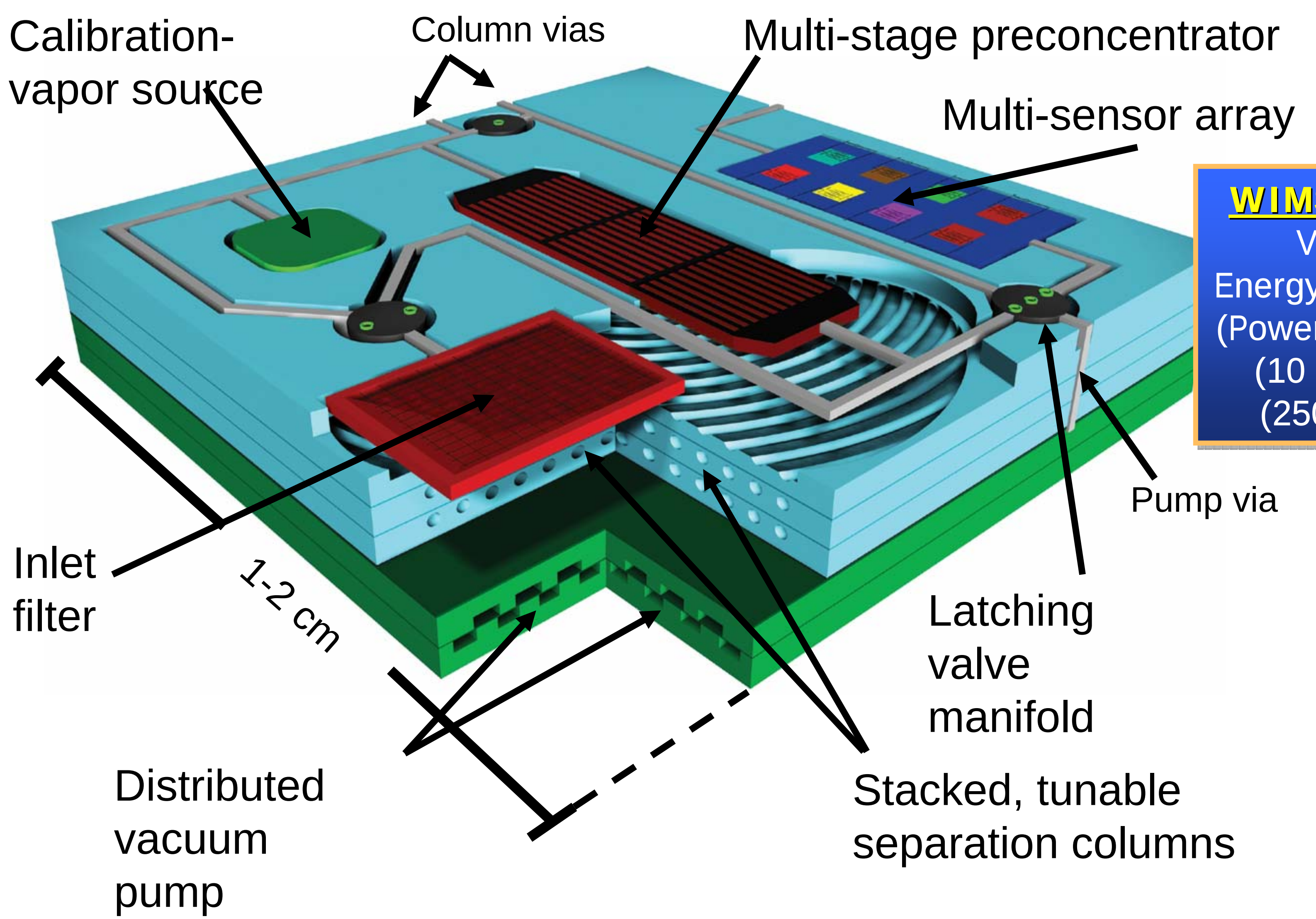
Integrated Micro Gas Analyzer Based on Gas Chromatography



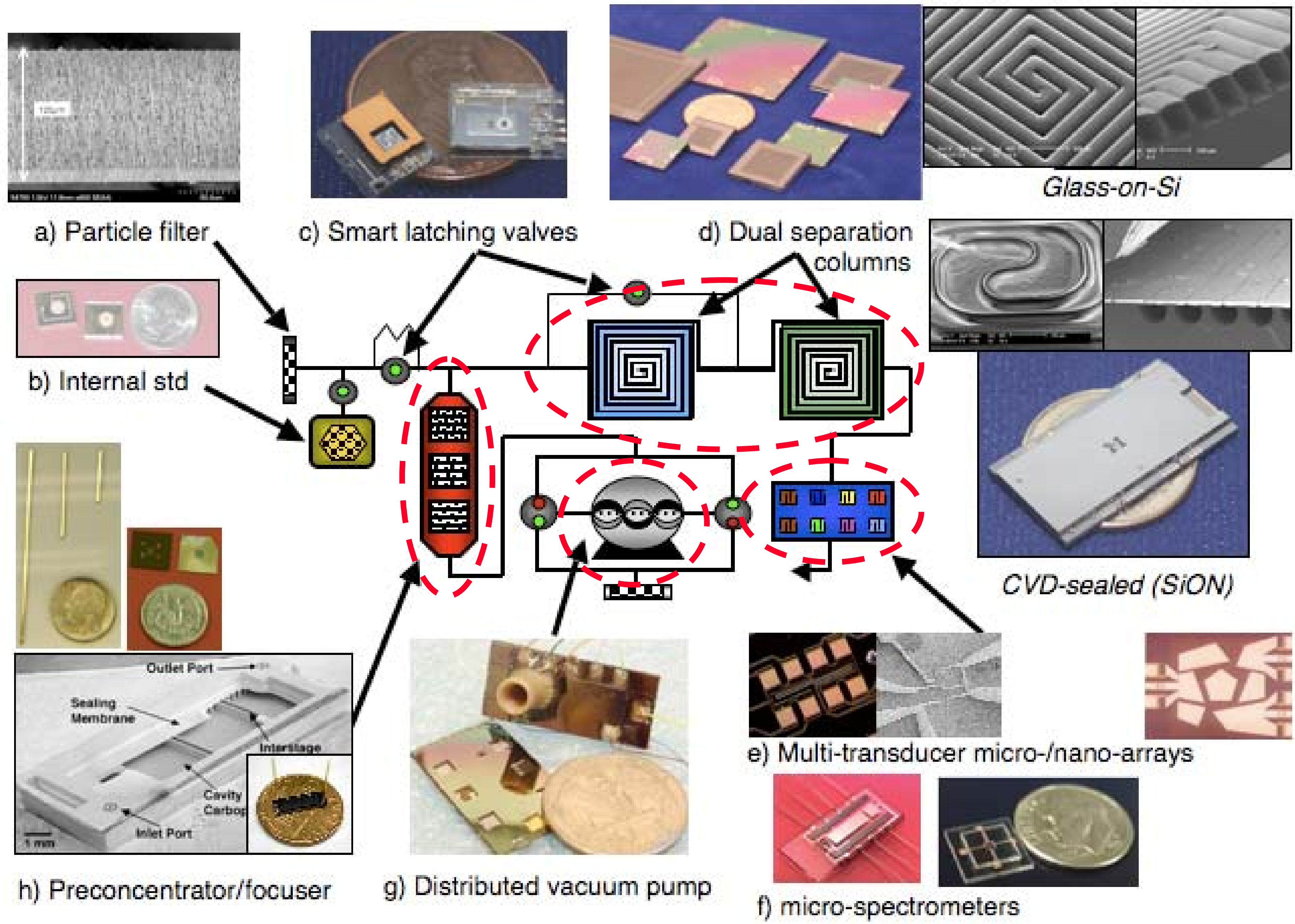
TARGETED PERFORMANCE:

- 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 μGC Vision

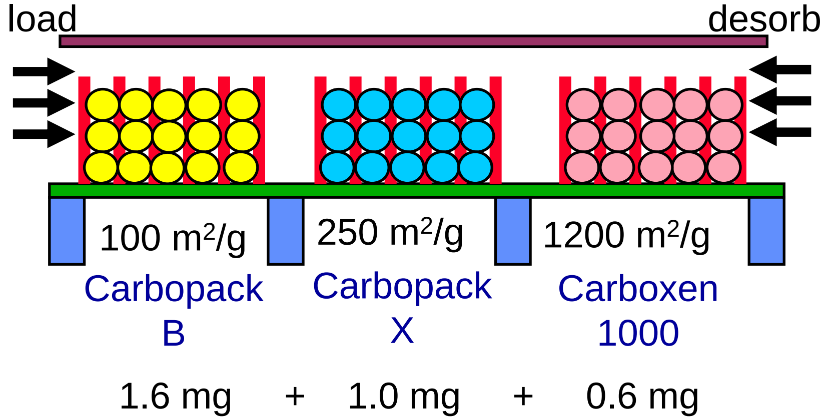


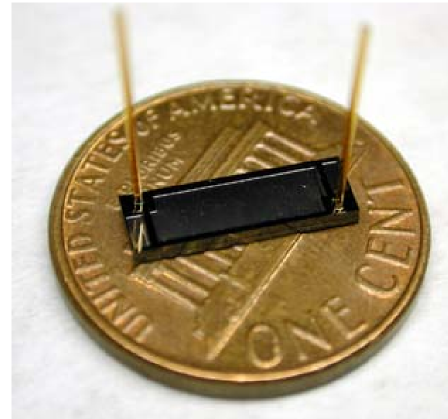
WIMS μGC Goals
Vol: 5 cm^3
Energy/Analysis: 1 J
(Power, Anal. Time):
(10 mW, 2 min.)
(250 mW, 4 s)

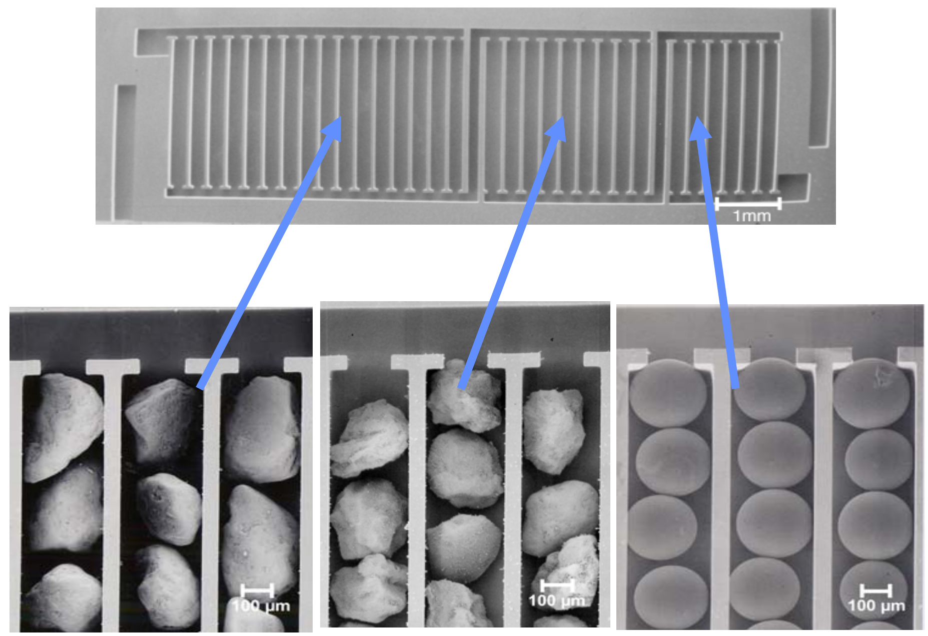

a) Particle filter
c) Smart latching valves
d) Dual separation columns
Glass-on-Si
b) Internal std
CVD-sealed (SiON)
Outlet Port
Sealing Membrane
Interstage
Cavity
Inlet Port
1 mm
e) Multi-transducer micro-/nano-arrays
h) Preconcentrator/focuser
g) Distributed vacuum pump
f) micro-spectrometers

Multi-stage Preconcentrator Focuser (μPCF)

3-Stage μPCF (Granular Adsorbent)







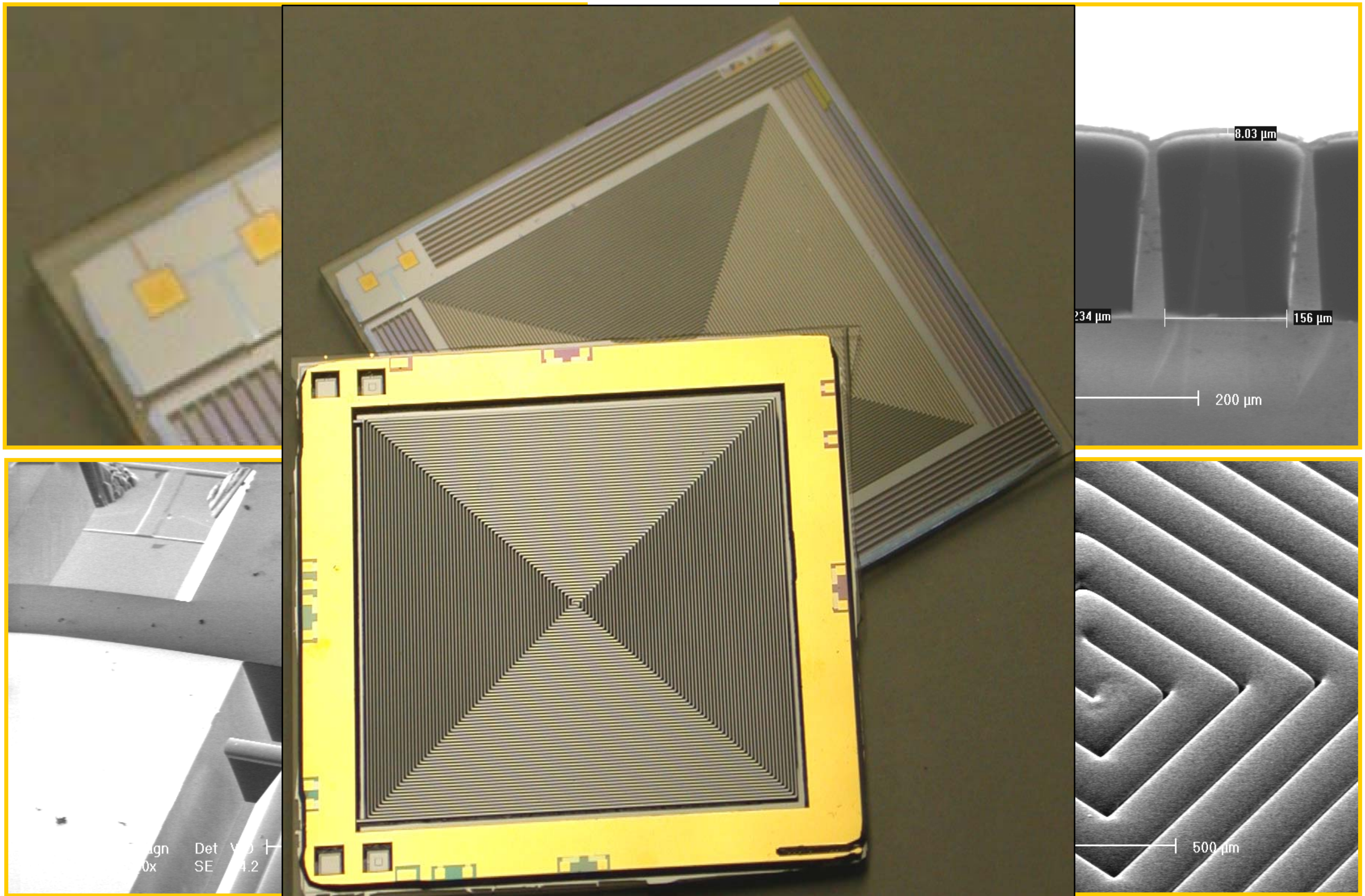
Tian, Pang, Wise, Zellers, JMEMS, 2005

Device Features

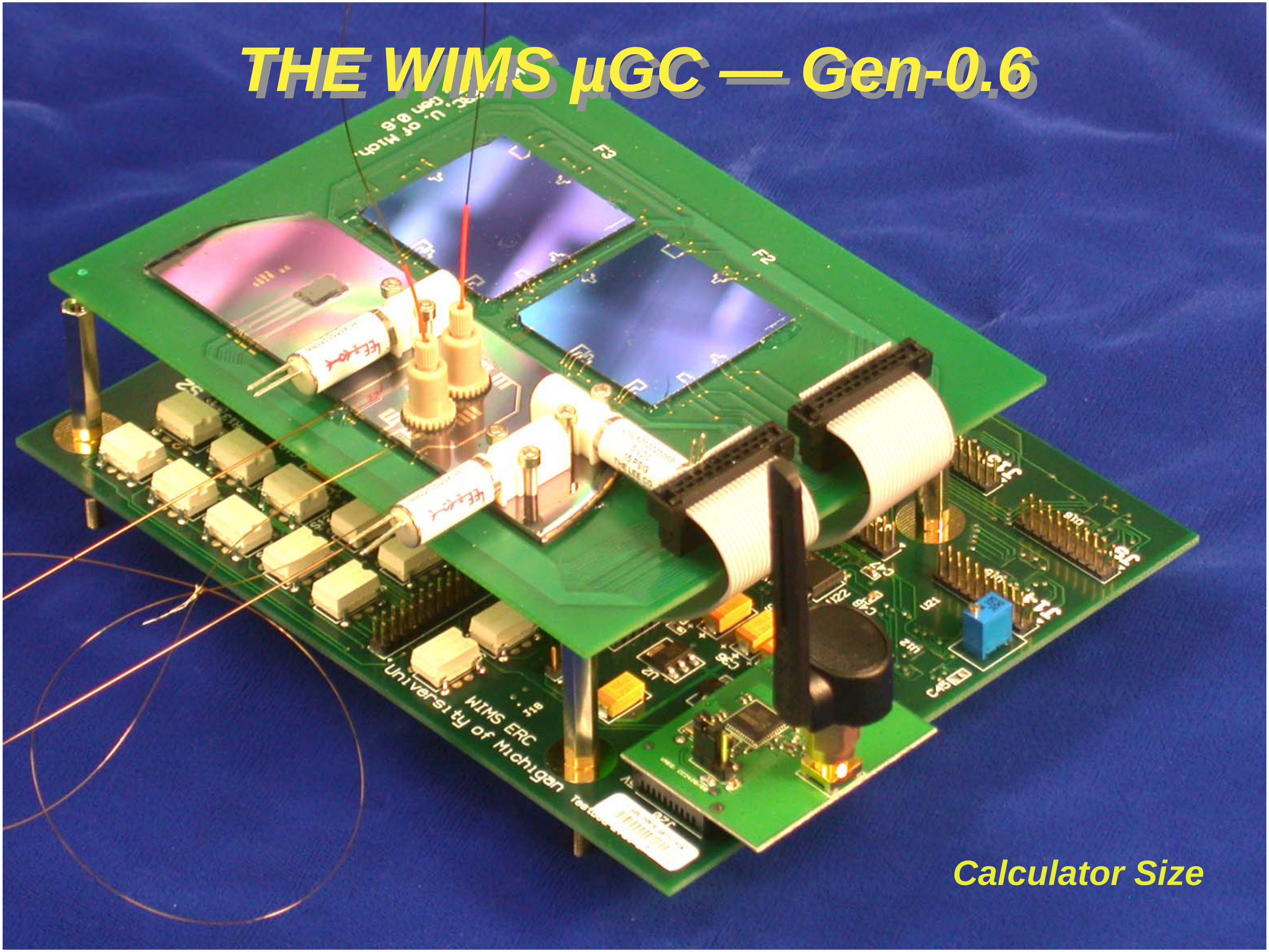
- **3 mm x 9 mm active area**
- **50-μm thick Si “floor”**
- **50 x 3000 μm slats (heat-exchangers)**
- **220 μm gaps for adsorbents**
- **385 μm tall**
- **Precon factors >5000-fold**

LOW-MASS SILICON SEPARATION COLUMNS

Wise, Agah, University of Michigan



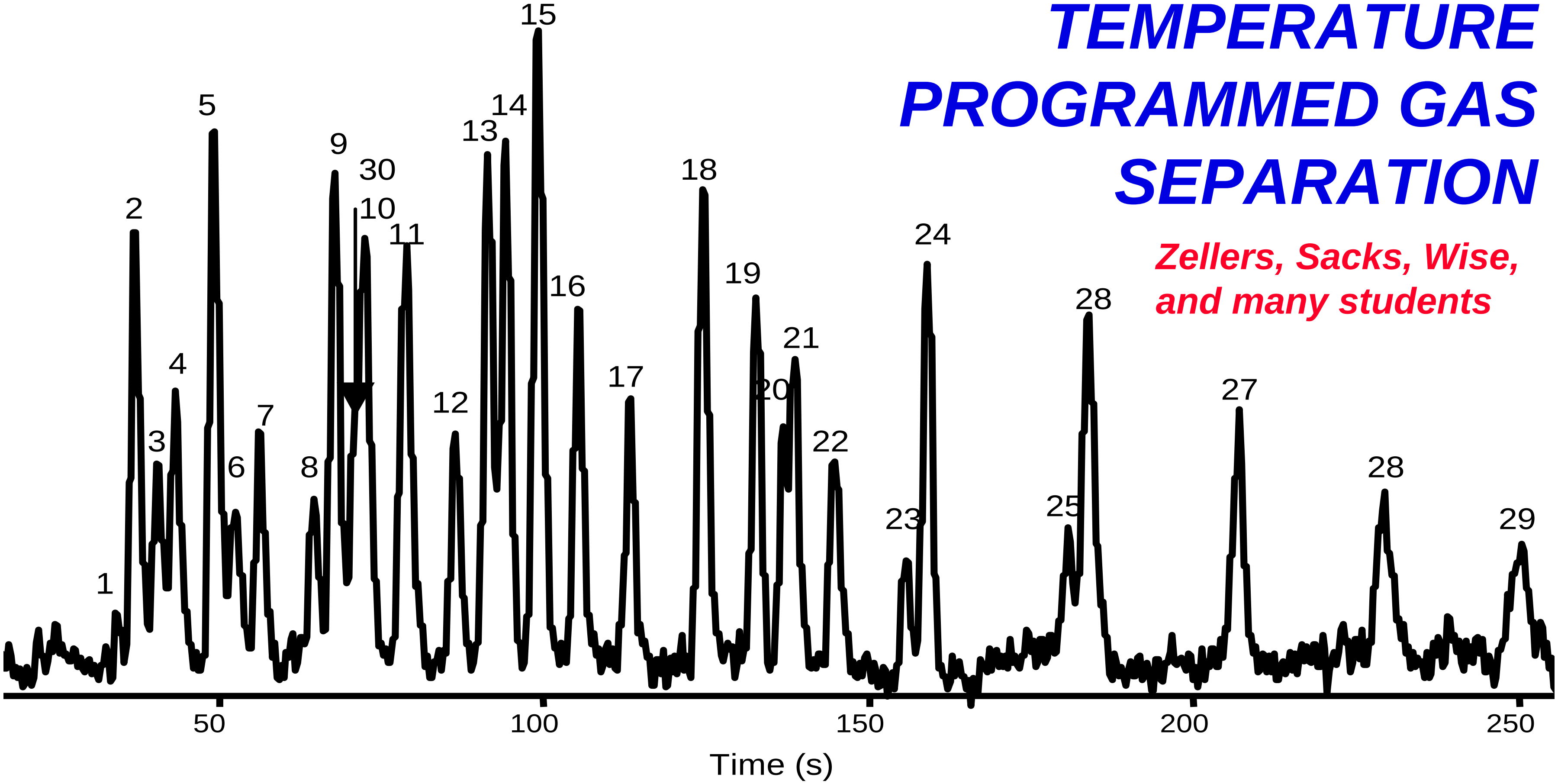
THE WIMS µGC — Gen-0.6



Calculator Size

TEMPERATURE PROGRAMMED GAS SEPARATION

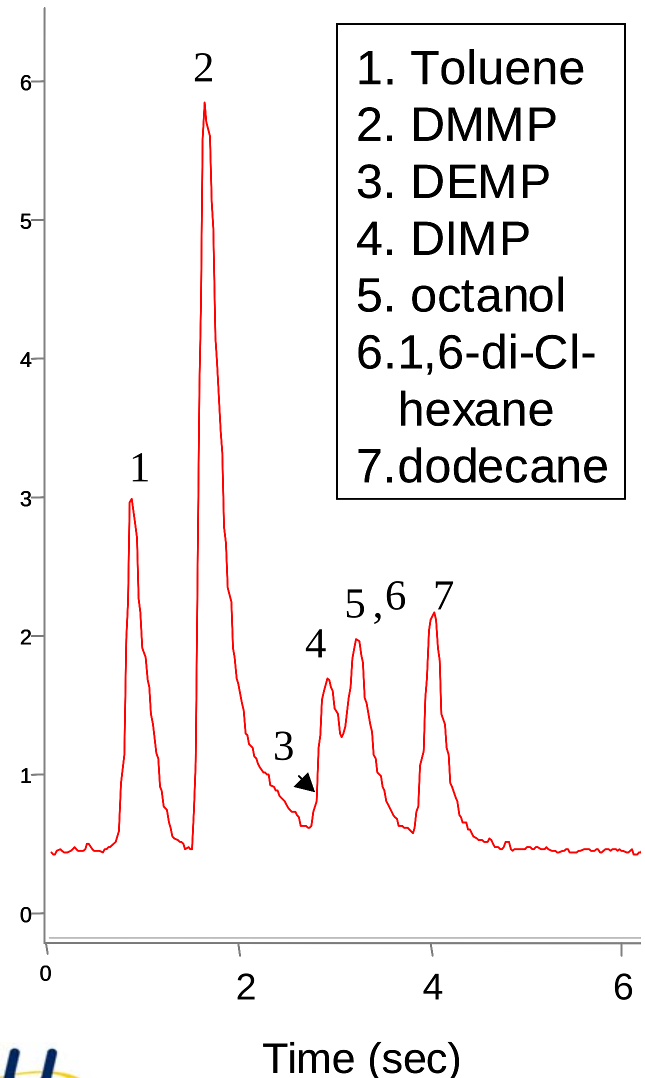
Zellers, Sacks, Wise, and many students

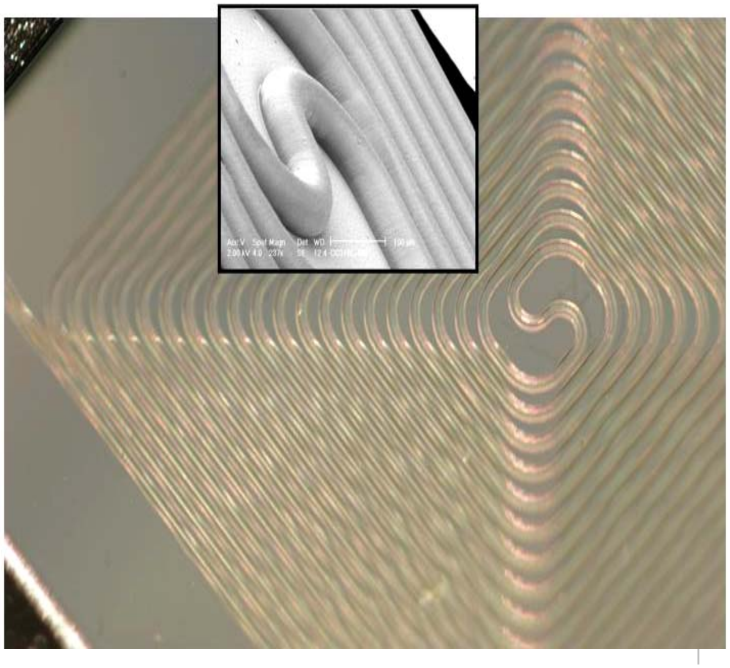


- Thirty air pollutants spanning three orders of magnitude in vapor pressure were separated in ***4.2min on a single 3m Si-glass column*** coated with polydimethylsiloxane and temperature programmed at 20°C/min.
- Producing ***12,000 theoretical plates***, this is the **highest resolution micro-column**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

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

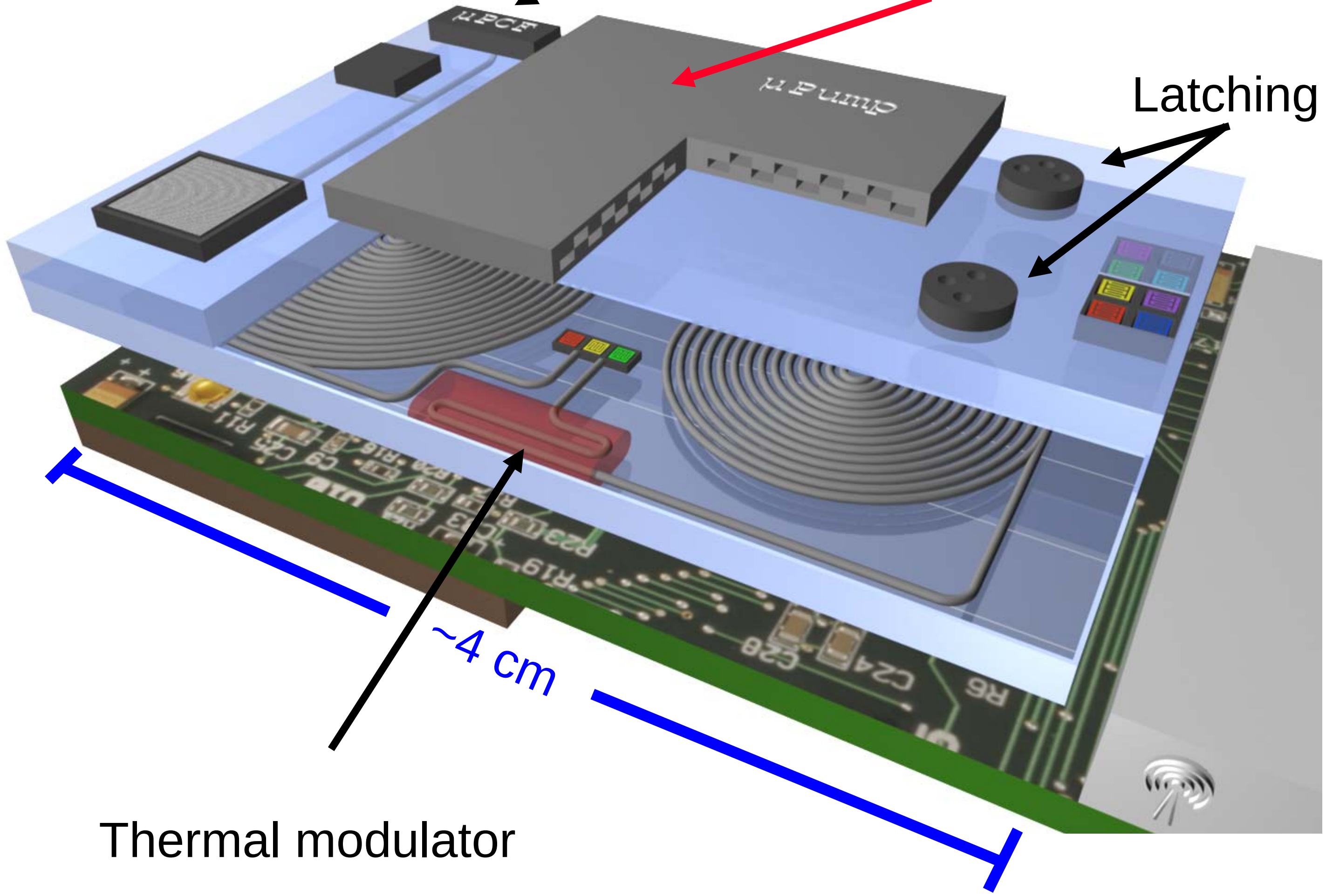
Agah, Potkay, Wise, Zellers, Sacks,…



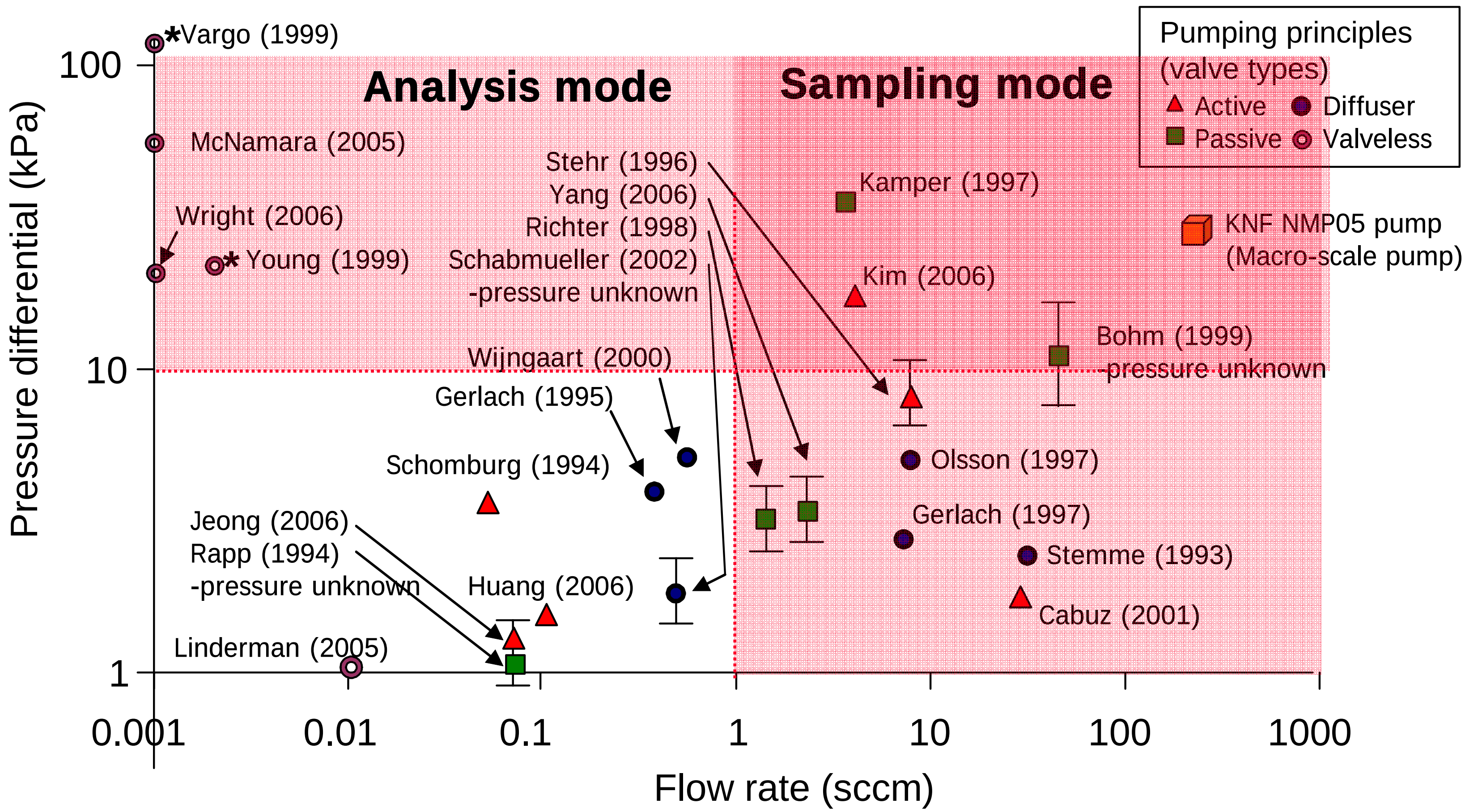


- Using ***25cm*** silicon-glass columns programmed at 1600°C/min, a seven-component mixture of chemical warfare simulants can be separated in ***4 seconds***
- CVD-sealed ultra-low-mass columns (above) promise still faster responses using entirely new column architectures (2D GC)

WIMS μGC Actuators



Summary of Previous Gas Micropumps

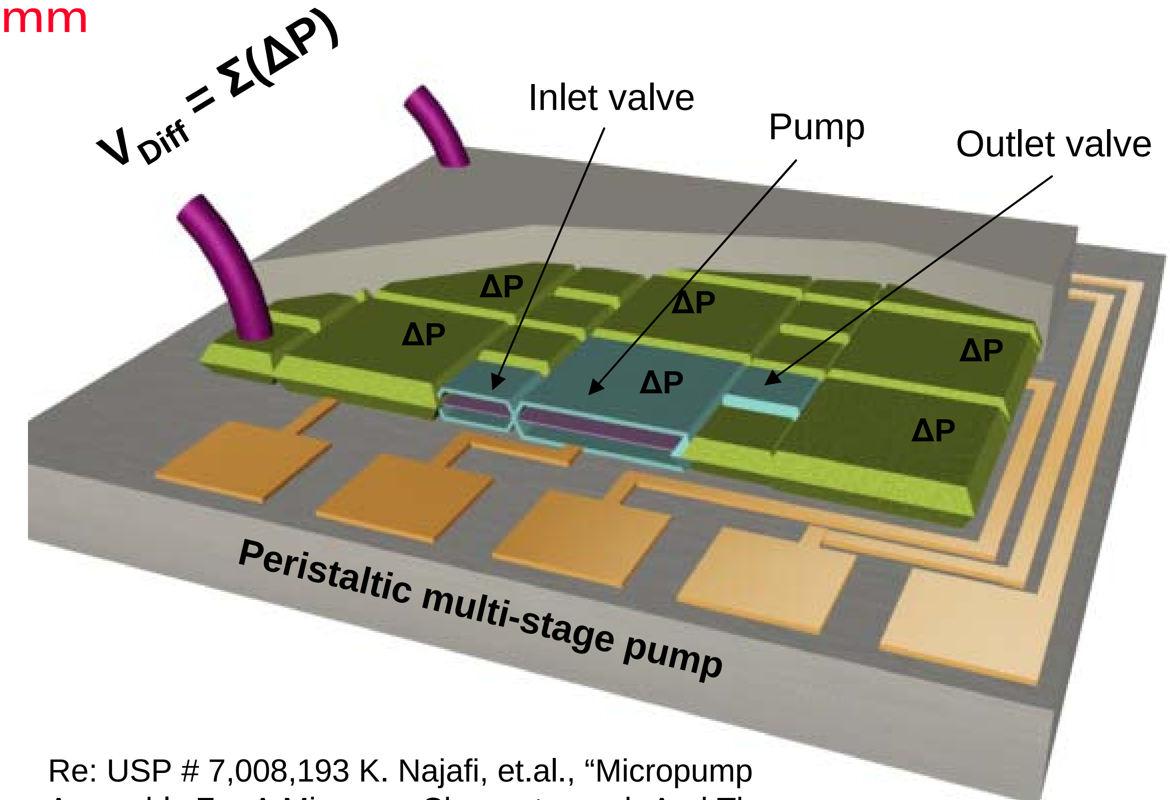


- **No previous gas micropump meets the WIMS μGC requirements.**
- **Size and power consumption are not included in the graph.**
- Low flow rate ← small stroke volume, slow op., gas compressibility.
- Low pressure ← weak force of a membrane, leakage.
- Single mode operation

Integrated Multi-stage Gas Micropump

- *Goal:* **Develop a miniature micropump for the µGC:**
  - **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



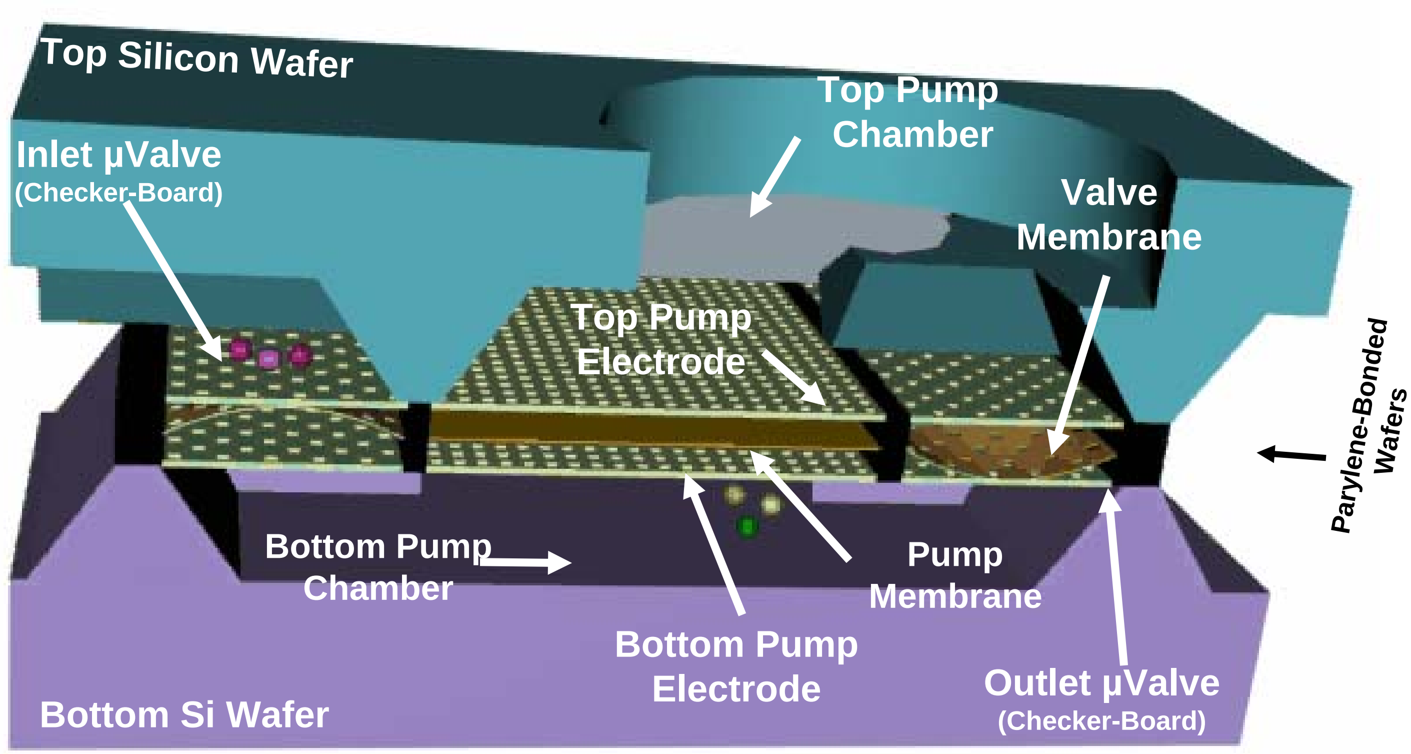
Re: USP # 7,008,193 K. Najafi, et.al., "Micropump Assembly For A Microgas Chromatograph And The Like", March 7, 2006

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

The Operation of a Single Stage of the Pump

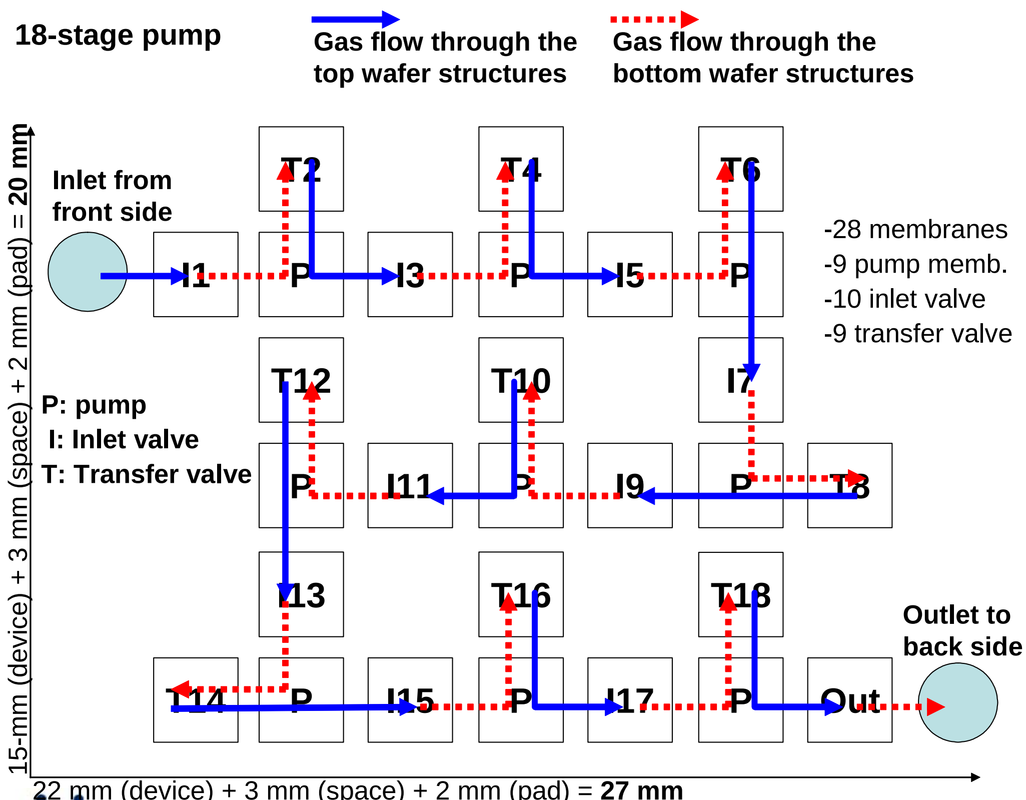
- Electrostatic Actuation
- Active Micro Valves
- Multi-Stage Design
- Polymer Membrane
- Dual-Electrode Actuation
- Dual-Chamber Layout
- Curved Electrode

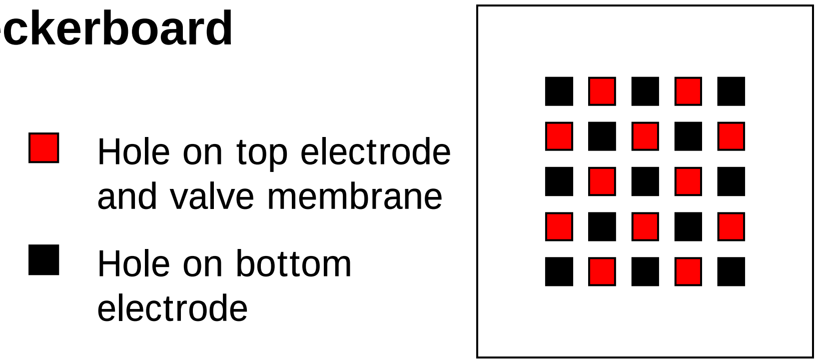
- Two bonded Si wafers, sandwiching pump & valve membranes
- One membrane and two valves for each two pump chambers
- Checker-board active valves, dual electrode pull-pull electrostatic drive

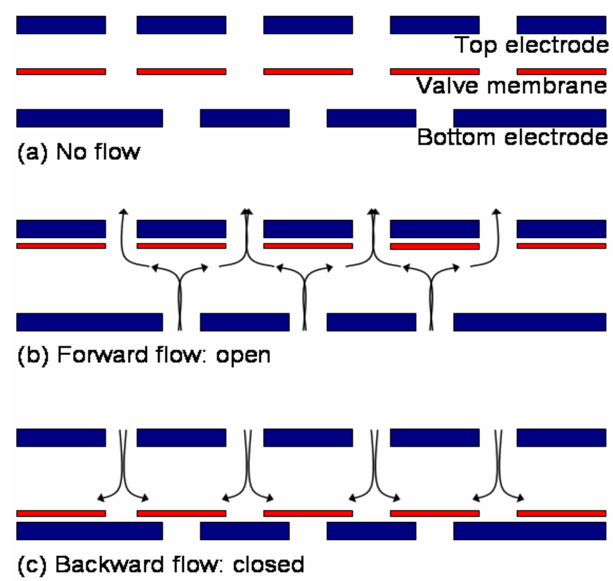


Multi-Stage Layout and the Microvalves

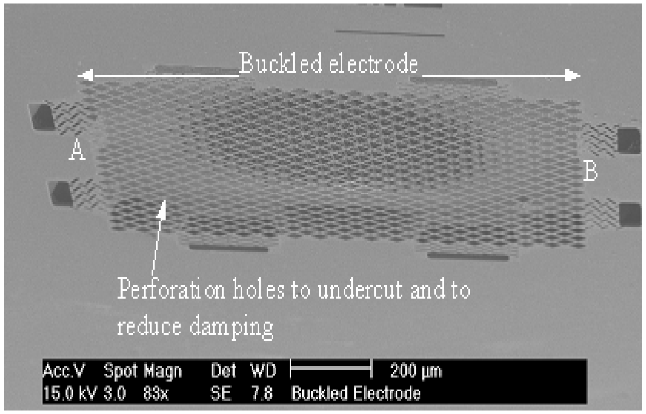
- **The multi-stage pump can be layed out to generate any number of stages needed.**
- **Layout of 18-stage pump shown below. Two-, four-, and 18-stage pumps have been designed and fabricated.**
- **Gas flow is controlled by the integrated checkerboard microvalves shown on the right.**

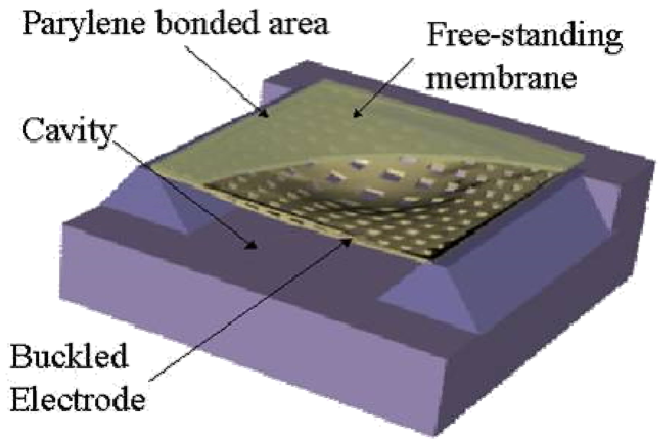






Microfabrication and Technologies





Parylene Wafer Bonding

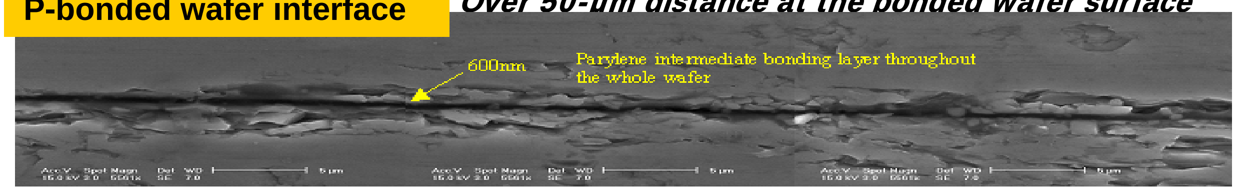
- **Low-Temp, <230°C**
- **Thin Layers (<0.5µm)**
- **Reliable, No Voids**

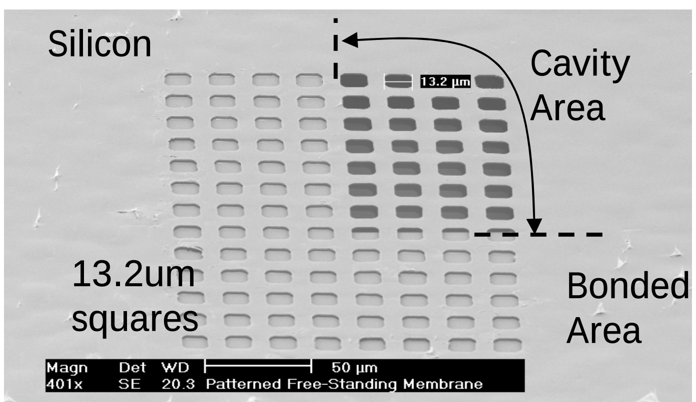
Curved Electrodes

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

P-bonded wafer interface

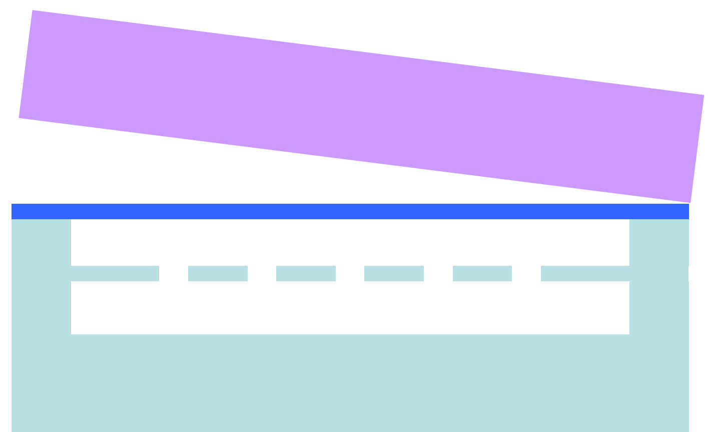
Over 50-um distance at the bonded wafer surface



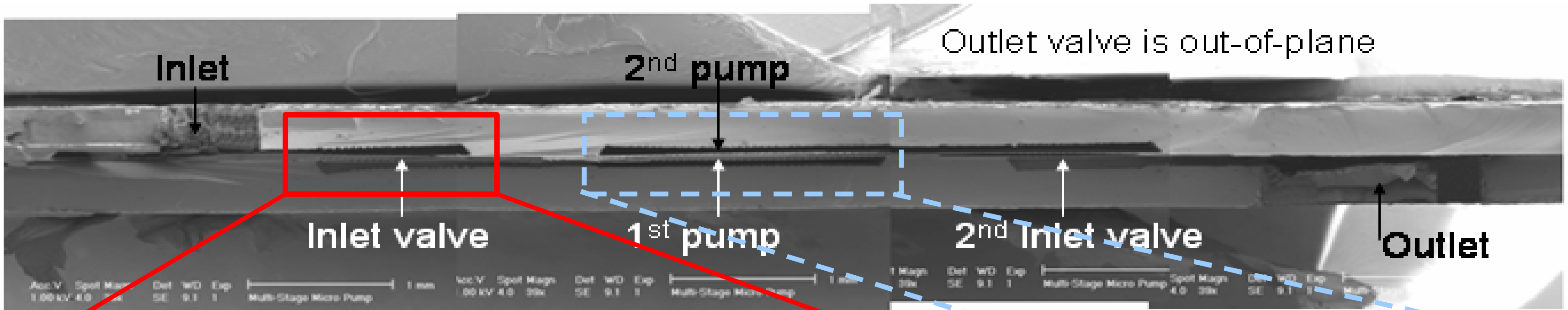


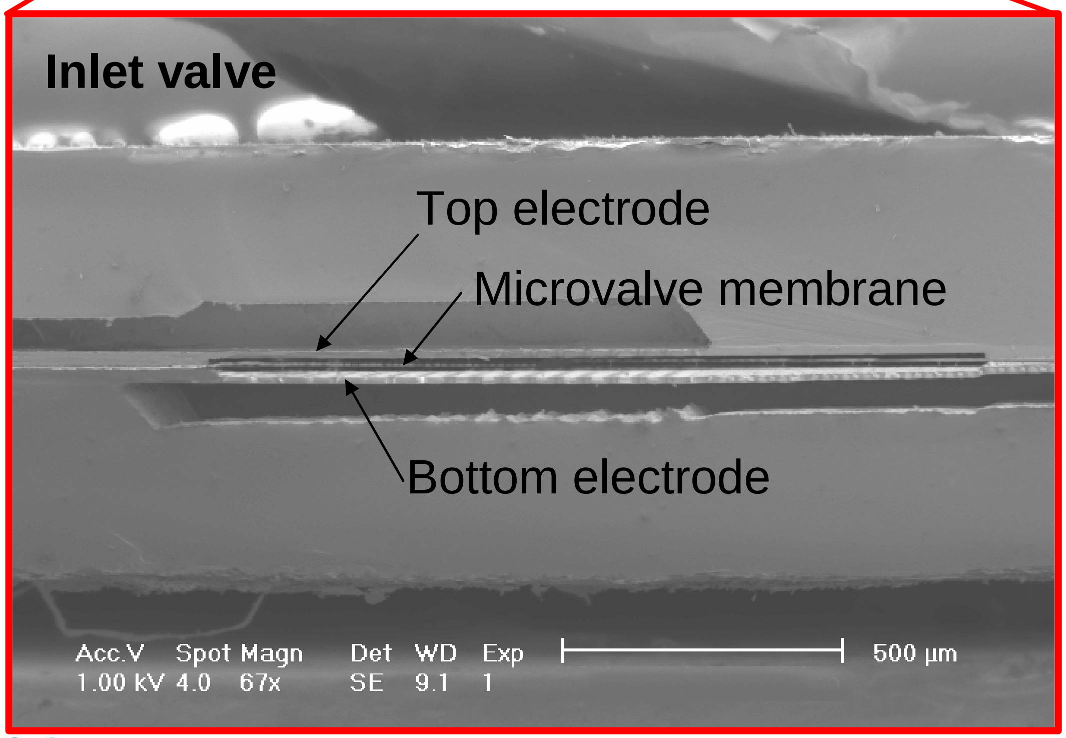
Parylene Membrane Transfer

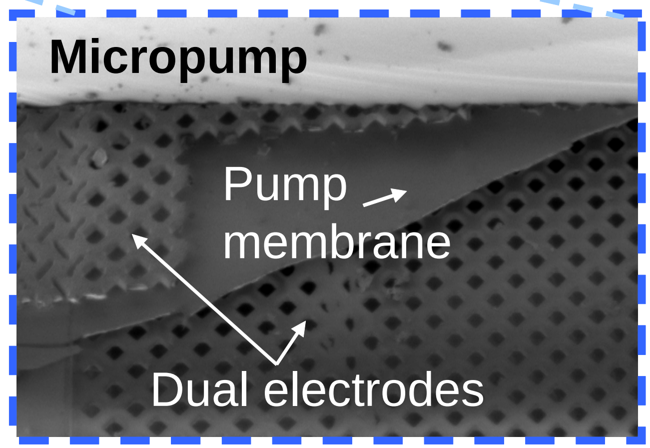
- **Wafer-Level**
- **High-yield (>90%)**
- **Thin films, over deep cavities**



Microfabrication and Technologies

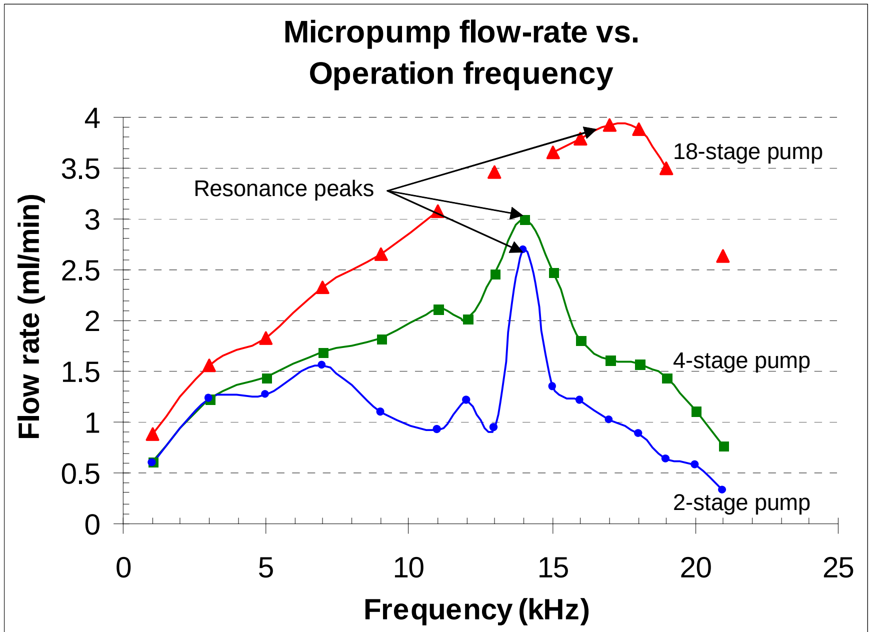


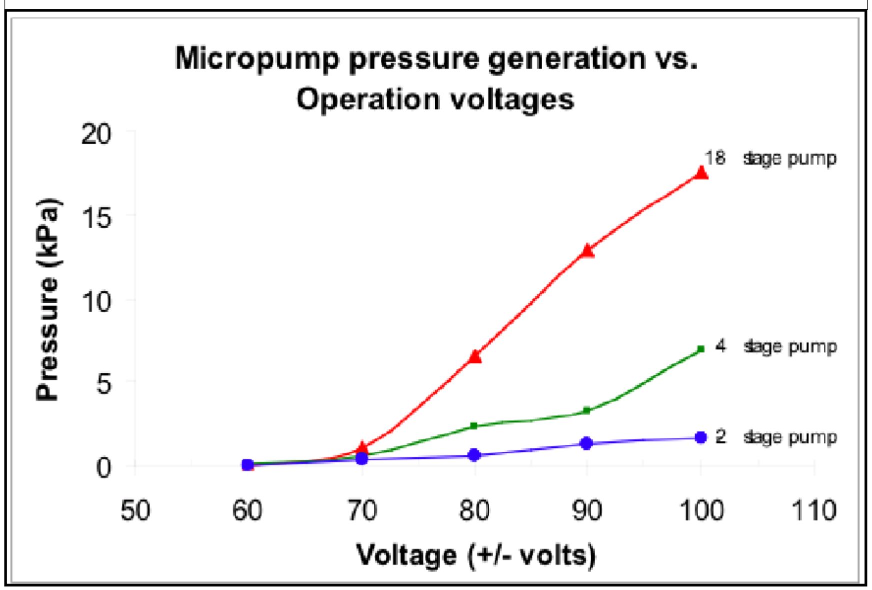


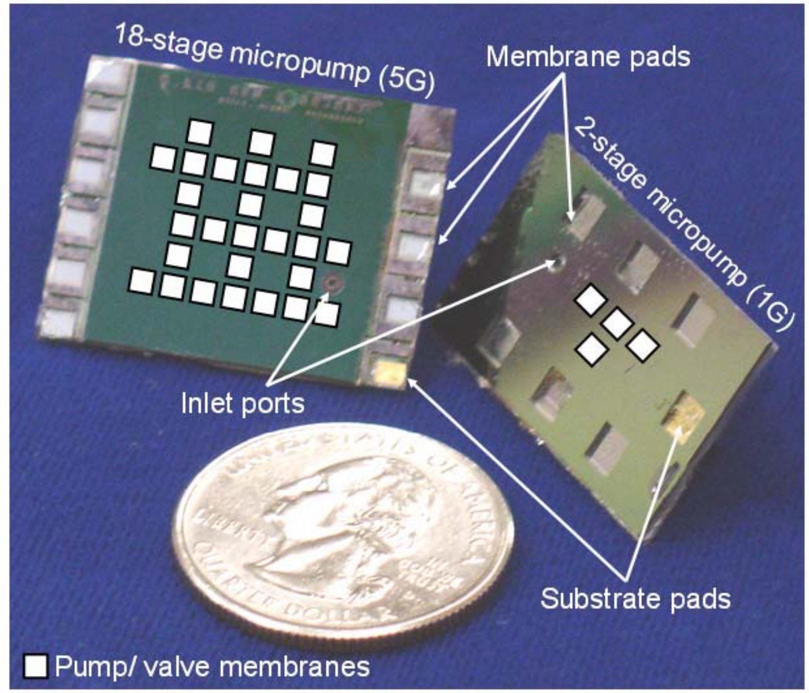


- **A complete fluidic path through two wafers**
- **Dual-electrodes**
- **Polymer membrane**

A Multistage Peristaltic MEMS Pump for a μGC



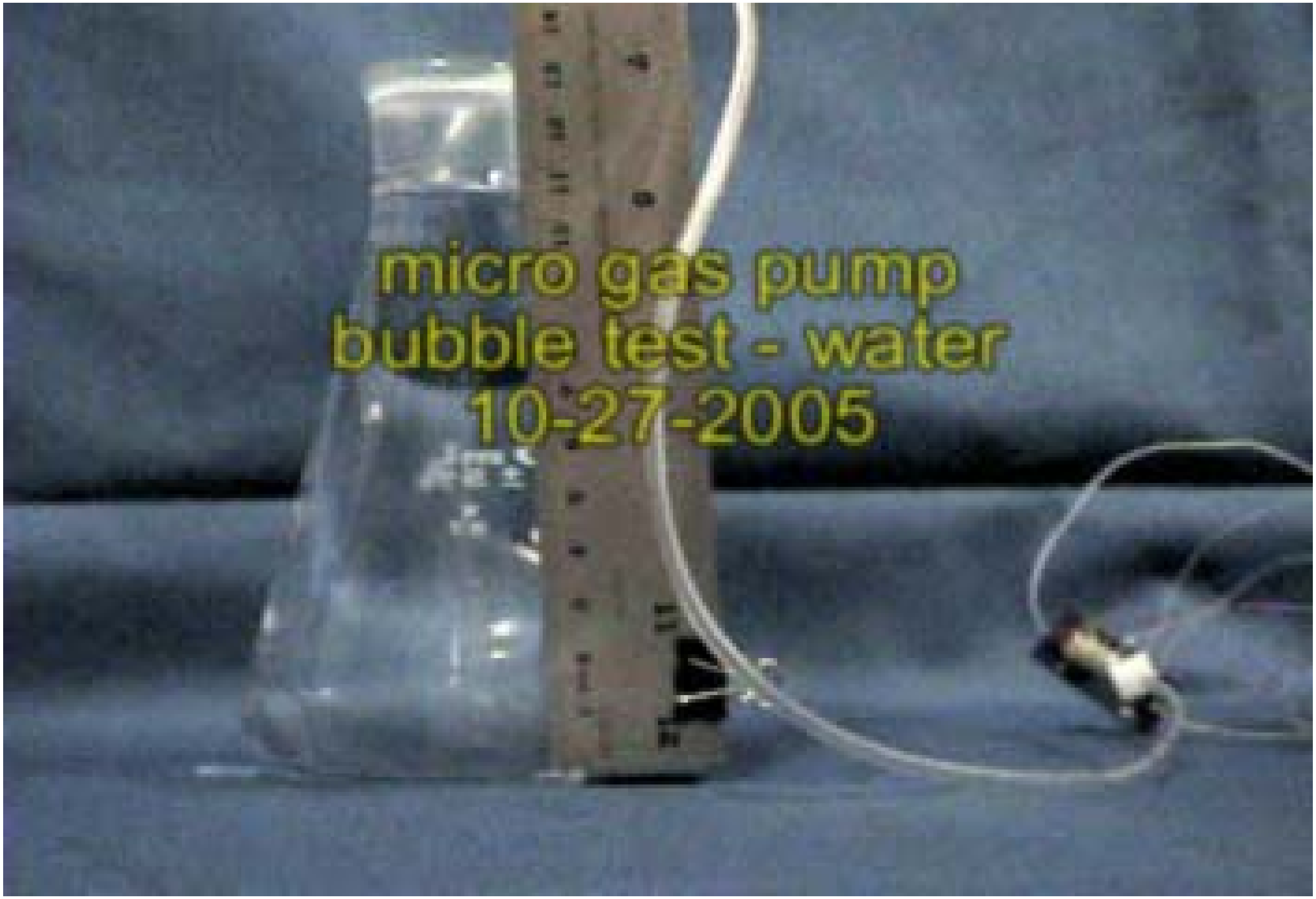




18-stage peristaltic micropump

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

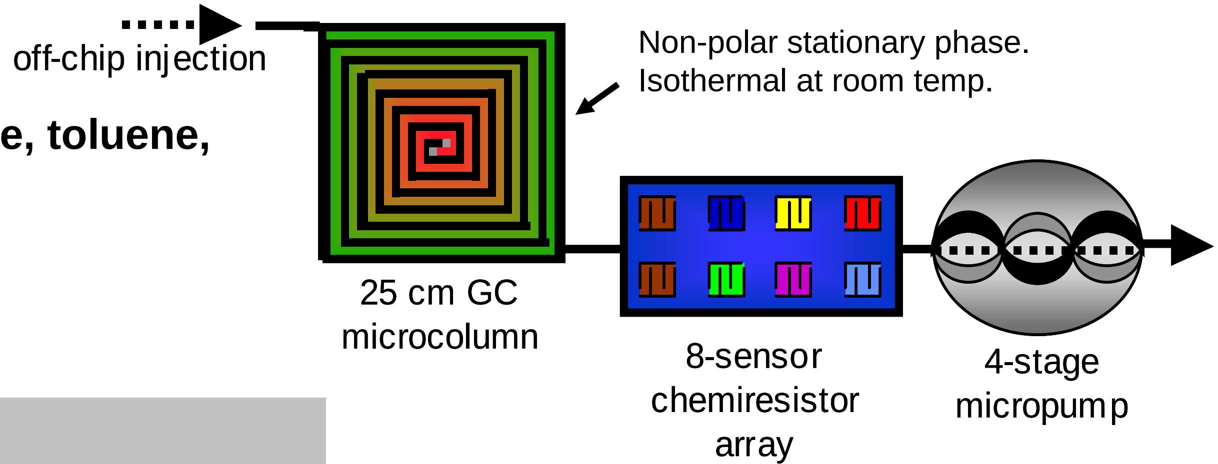
Pumping Air Bubbles

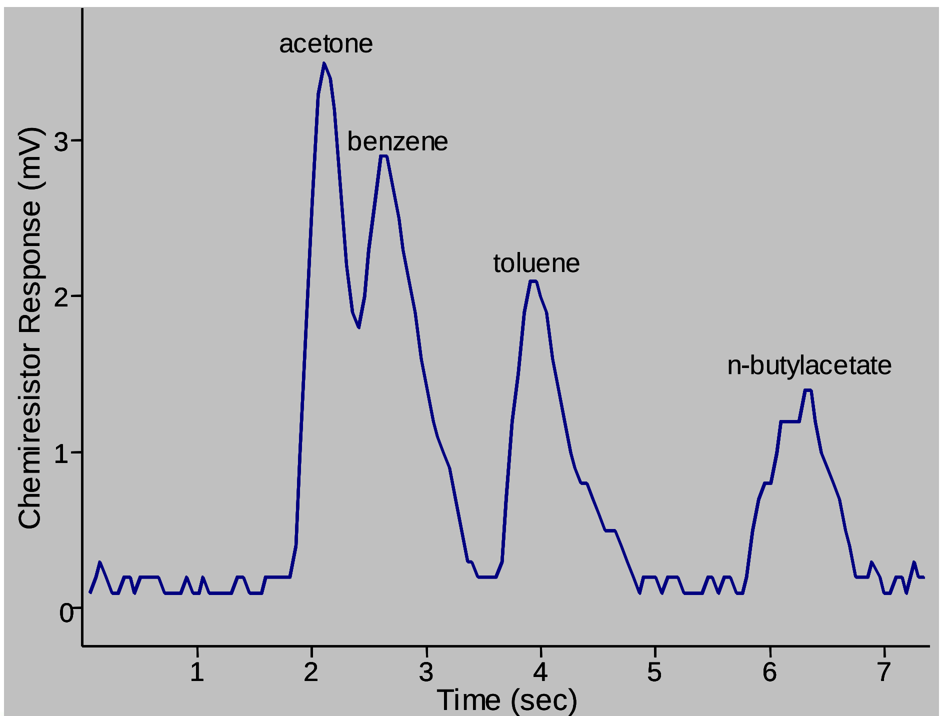


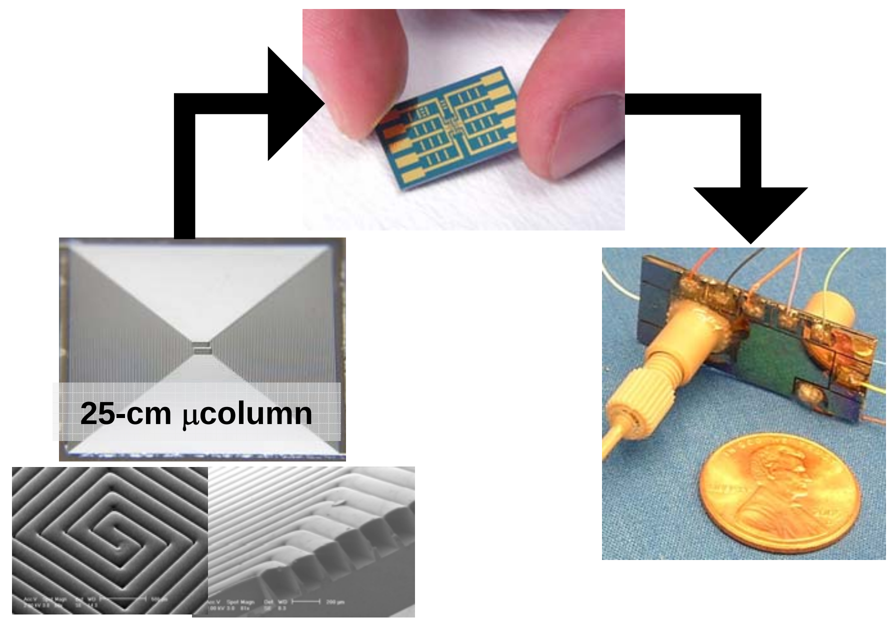
Integration of WIMS μColumn, μArray, and μPump (μCAP Subsystem)

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

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

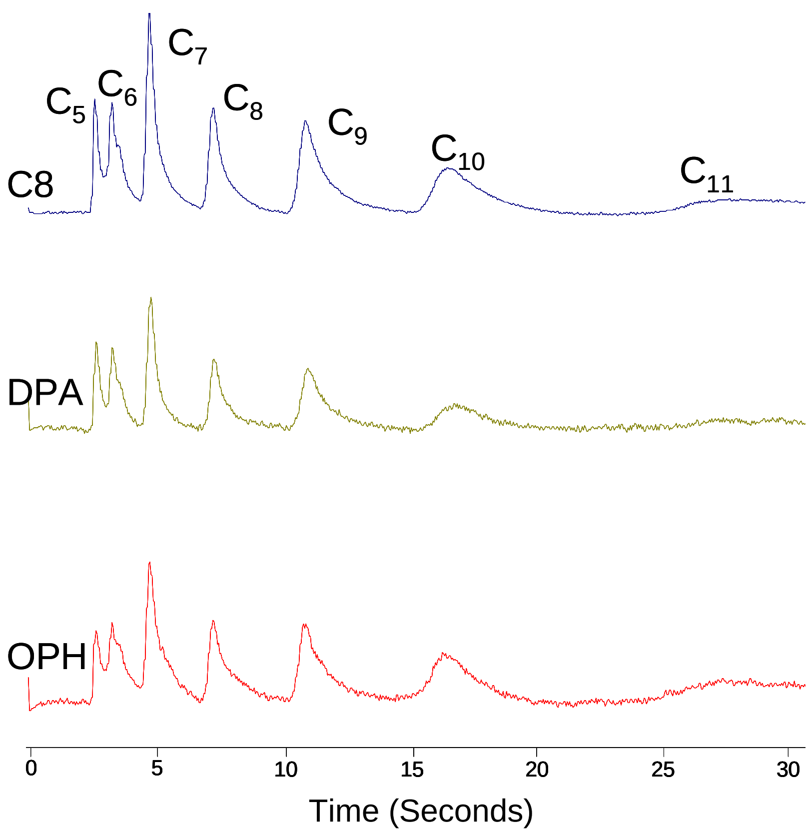


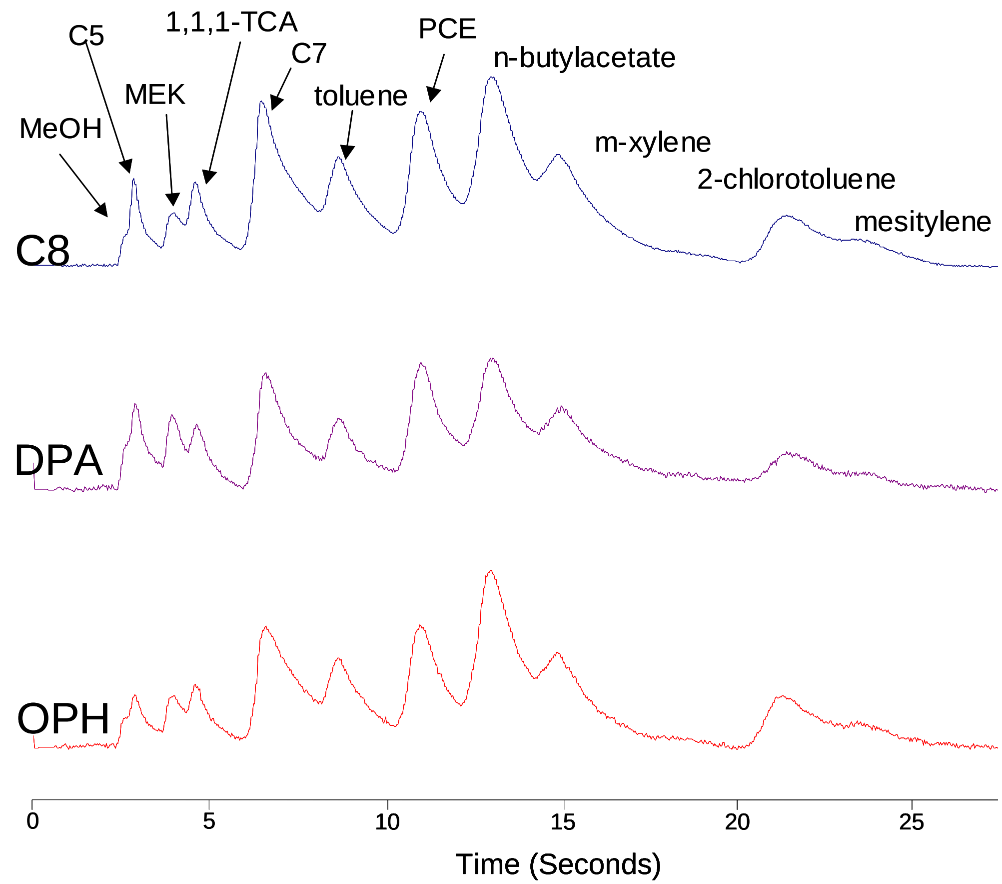




Latest Results from μCAP Subsystem

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

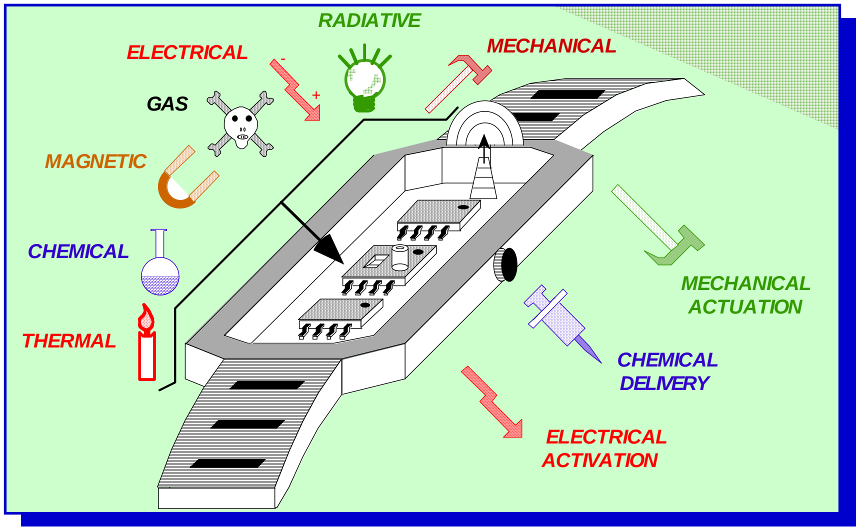
Integrated Sensor/Actuator	→	Integrated Instruments
Work on one parameter	→	Monitor multiple parameters
Customized	→	Generic for broad applications
Limited Selectivity, specificity, sensitivity	→	High Selectivity, Specificity, Sensitivity
Limited Dynamic Range	→	Wide Dynamic Range
Only Senses	→	Measures and Monitors
Robustness hammered out of device, material, process	→	Robustness Delivered by µSystem
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
- …

µInstruments
- Atomic Clock
- µMechanical Comm/Processing
- Radiation Detectors
- Gas Analysis
- Chemical/Biological Analysis
- Warfare agents
- …



- Smaller
- Lighter
- Better
- Cheaper

Acknowledgments: Collaboration Team

- Electrical Engineering
  - Ken Wise (Center Director)
    - 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
  - Massoud Kaviany
    - Luciana Da Silva
- Aerospace Engineering
  - Luis Bernal
    - Aaron Astle

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  - Richard Sacks
    - Randy Lambertus, Shaelah Reidy, Josh Whiting
  - Ted Zellers
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- Environmental Health Sciences
  - Ted Zellers
    - Vincent Lu, Qiongyan Zhong, Chunguang Jin, Gustavo Serrano
- Michigan Tech University
  - Paul Bergstrom (Elec & Comp Eng.)
    - Jin Zheng
- Michigan State University
  - Dean Aslam (Elec & Comp Eng.)
    - Yang Lu
- Fabrication & System Integration:
  - Robert Gordenker, Katharine Beach, Cathy Morgan, Josh Whiting, Kate Plass, Craig Friedrich, Evan Gamble