



# ***Historical Review: Fast & micro Gas Chromatography***

**Dr. Ed Overton, Professor Emeritus**

**Department of Environmental Sciences**

**Louisiana State University School of the Coast & Environment**

**Presenter: John Crandall**





## ***First... about Ed***



- **Analytical-Environmental Chemistry – trace organic analyses using high resolution separation techniques and high resolution gas chromatography-mass spectrometry**
- **Environmental monitoring**
- **Environmental analyses and their associated data interpretation, management and presentation techniques**
- **Application of analytical techniques and chemical principles to emergency spill responses**
- **Chemical hazard evaluations and risk assessments**
- **Analytical instrument development for use with hazardous chemical spill incidents, ambient environmental monitoring, and monitoring in closed (indoor) environments**
- **Development of methods to detect, assess and mitigate environmental chemical hazards**

# More Importantly



- **Because of his environmental measurement needs Ed has been...**
  - Vigorous supporter of Fast, micro and transportable gas chromatography
  - Inventor in pursuit of analytical problem solving
  - Team leader at LSU's School of the Coast & Environment
  - Leading participant in the IFPAC and ONSITE communities
  - Leading voice of reason regarding crude oil spills and their impact on the environment
  - And MOST importantly...

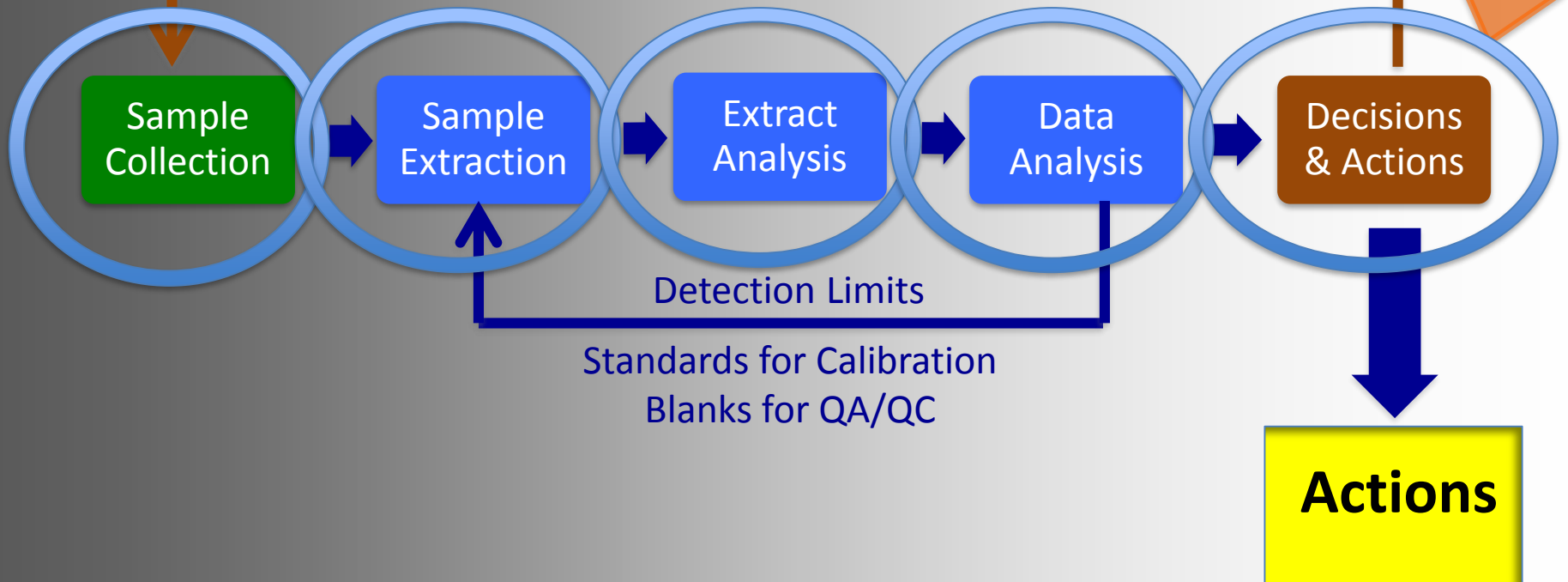


**Educator and mentor inspiring others to join the field**

# Analytical Process

Providing Information Needed for Decision Making

Analytical Rational, Data Quality Objectives, Cost, Time



# Types of Air Pollution



Criteria Pollutants

Acid Rain

Industrial Pollutants

Oxidized organics (TO15)

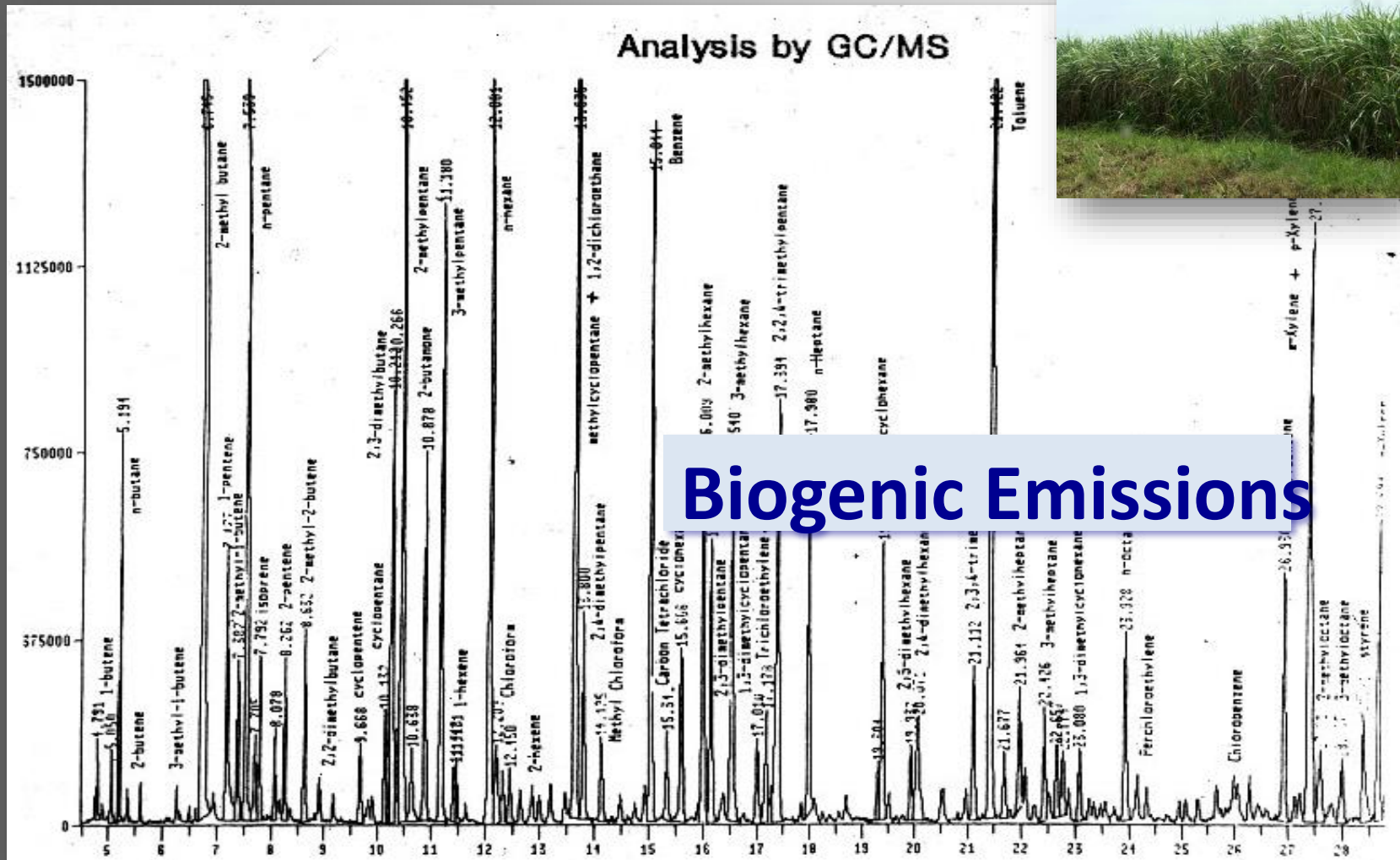
Ozone Precursors

Air Toxics (TO14)

Greenhouse Gases

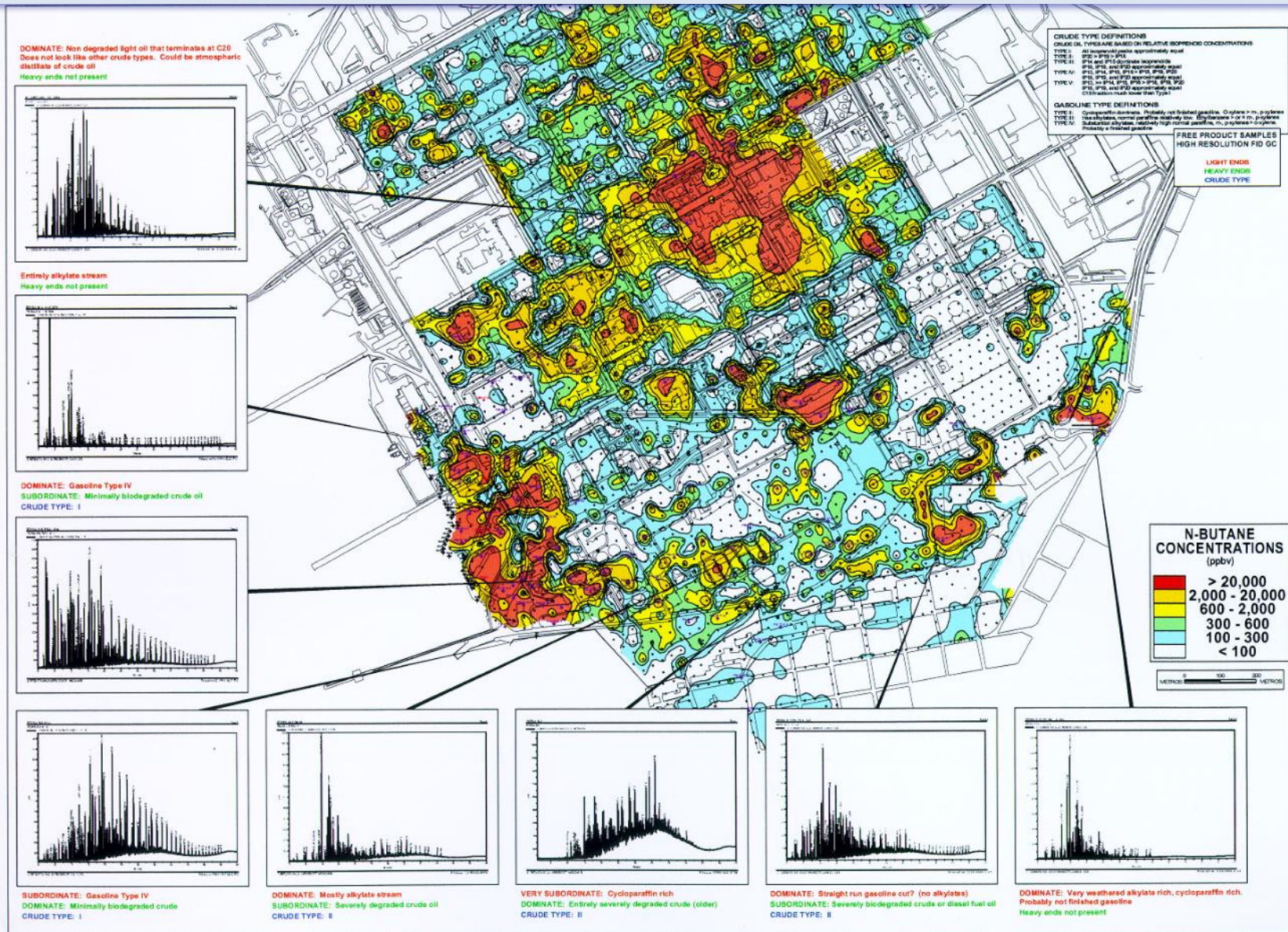
Indoor Air

# GCMS Chromatogram of Rural Air Sample collected in the middle of a field





# Refinery Site: GC Analysis of Selected Free Product Samples and Airborne Butane Conc.





## Vapor trail

Elephants also possess one of the most well developed senses of smell in the animal kingdom. This keen **sense of smell** is used not only to **locate food and water sources but also for communication**. Elephants detect and process many chemical signals in a wide variety of smells throughout their environment. Sources of odors used in chemical **communication between elephants** include urine, feces, saliva, and secretions from the temporal gland.

**TABLE 3. Distinctive volatiles in temporal gland emissions, breath, and urine of Asian elephants in the United States and India**

	Acetone	Isoprene	Butanal	2-Butanone	2-Methyl-3-buten-2-ol	2,3-Butanediol	Dimethyl disulfide	4-Heptanone
PostM-TG	x							
PreM-TG	X	X	X	X				
PreM-TG <sup>a</sup>	X	X	X	X				
Skin-C	X							
PreM-B	x	X	X	X	X			
PreM-B <sup>a</sup>	x	X	X	X	x			
PostM-B	x							
PostM-B <sup>a</sup>	x							
Preg-B	x	X		x		X		
Preg-B <sup>a</sup>	x	X		x		X		
Preg-U	x			x			x	X
Preg-U <sup>a</sup>	x			x			x	X
Mahkna U	x			x			X	

<sup>a</sup>Control samples, U.S. studies [Rasmussen and Perrin, 1999].

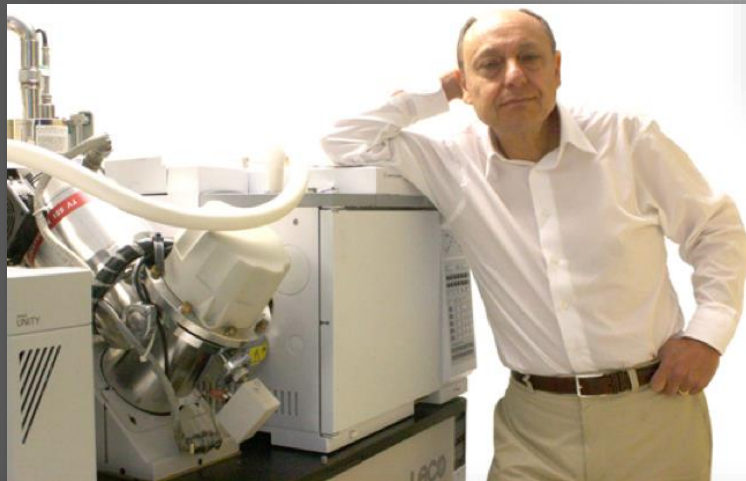
X, high concentration; x, lower concentration; M-TG, musth temporal gland secretions; C, control; M-B, musth breath; Preg, pregnant; B, breath; U, urine.



BreathLink rapid point-of-care breath test for breast cancer

# Effect of influenza vaccination on oxidative stress products in breath

Michael Phillips<sup>1,2,6</sup>, Renee N Cataneo<sup>1</sup>, Anirudh Chaturvedi<sup>1</sup>, Patrick J Danaher<sup>3</sup>, Anantrai Devadiga<sup>1</sup>, David A Legendre<sup>4</sup>, Kim L Nail<sup>4</sup>, Peter Schmitt<sup>5</sup> and James Wai<sup>1</sup>



Breathometer



## DIAGNOSTICS

### Point-of-care breath test for biomarkers of active pulmonary tuberculosis

Michael Phillips<sup>a,b,\*</sup>, Victoria Basa-Dalay<sup>c</sup>, Jaime Blais<sup>a</sup>, Graham Bothamley<sup>d</sup>, Anirudh Chaturvedi<sup>a</sup>, Kinjal D. Modi<sup>h</sup>, Mauli Pandya<sup>a</sup>, Maria Piedad R. Natividad<sup>e</sup>, Urvisch Patel<sup>a</sup>, Nagsen N. Ramraje<sup>f</sup>, Peter Schmitt<sup>g</sup>, Zarir F. Udawadia<sup>h</sup>

<sup>a</sup>Menssana Research Inc., Breath Research Laboratory, EDC III, 211 Warren Street, Newark, NJ 07103, USA  
<sup>b</sup>Department of Medicine, New York Medical College, Valhalla, NY, USA  
<sup>c</sup>Center for Tuberculosis Research, Angelo King Medical Research Center, De La Salle Health Sciences Institute, Cavite, Philippines  
<sup>d</sup>Department of Respiratory Medicine, Homerton University Hospital NHS Foundation Trust, London E9 6SR, UK  
<sup>e</sup>Center for Respiratory Medicine, The University of Santo Tomas Hospital (USTH), Espana Boulevard, Manila 1008, Philippines  
<sup>f</sup>Sir J.J. Group of Hospitals, Byculla, Mumbai 400008, India  
<sup>g</sup>Schmitt & Associates, 211 Warren St, Newark, NJ 07103, USA  
<sup>h</sup>P.D. Hinduja National Hospital and Research Center, Veer Savarkar Marg, Mahim, Mumbai 400016, India



### Prediction of breast cancer using volatile biomarkers in the breath

Michael Phillips<sup>1,2</sup>, Renee N. Cataneo<sup>1</sup>, Beth Ann Ditkoff<sup>3</sup>, Peter Fisher<sup>4</sup>, Joel Greenberg<sup>1</sup>, Ratnasiri Gunawardena<sup>5</sup>, C. Stephan Kwon<sup>6</sup>, Olaf Tietje<sup>7</sup>, and Cynthia Wong<sup>2,5</sup>

<sup>1</sup>Menssana Research Inc., 1 Horizon Road, Suite 1415, Fort Lee, NJ 07024, USA; <sup>2</sup>Department of Medicine, New York Medical College, Valhalla, NY USA; <sup>3</sup>Department of Surgery, Columbia University Medical Center, New York, NY 10032, USA; <sup>4</sup>Department of Pathology, Columbia University Medical Center, New York, NY 10032, USA; <sup>5</sup>Department of Medicine, Saint Vincents Catholic Medical Centers of New York, Staten Island Region, New York USA; <sup>6</sup>Department of Laboratory Medicine, Saint Vincents Catholic Medical Centers of New York, Staten Island Region, New York USA; <sup>7</sup>SystAim GmbH, Pfingstweidstr. 31a, CH 8005, Zürich, Switzerland

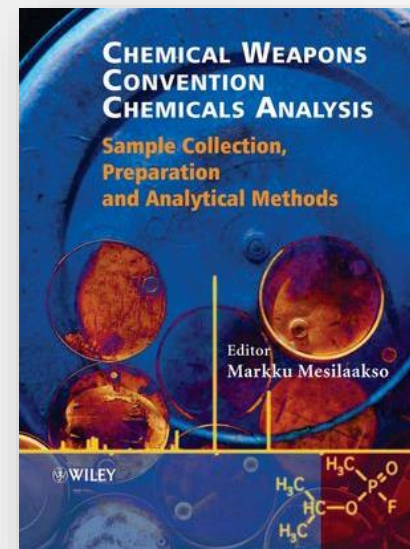
### Diagnostic Accuracy of Canine Scent Detection in Early- and Late-Stage Lung and Breast Cancers

Michael McCulloch, Tadeusz Jezierski, Michael Broffman, Alan Hubbard, Kirk Turner, and Teresa Janecki



**Process & Environmental monitoring**  
**Wellhead monitoring**  
**Emergency response**  
**Chemical Weapons Convention**  
**Chemical Warfare Agent Detection**  
**Infectious Disease Detection**

# Mobile Labs



# Organic Compound Analysis

**Volatile Organic Compounds**

**VOCs**

>3000 mm to ~0.1mm Hg VP

VC to DCB

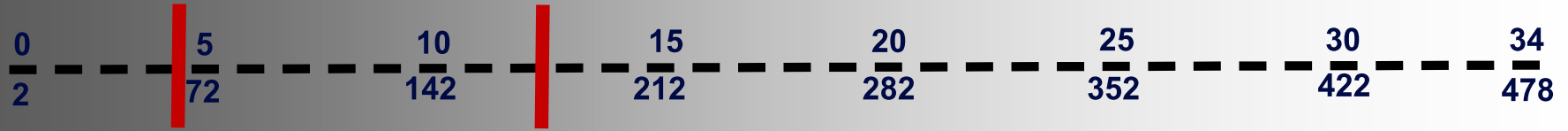
**Semi-volatile Organic Compounds**

**SVOCs**

~0.1mm to 0.000001mm Hg VP



# Carbon Number Range (RI = Carbon # times 100)



## Molecular Weight Range (for Saturate Hydrocarbons)

### Gases



>250mm VP

% to ppm

micropacked columns

loop injectors

VOCs

250mm to 0.1mm VP

ppm to ppb

thick film columns

sorbent traps

Semivolatile Organic Compounds

0.1mm to 0.000001mm VP

ppm to high ppt

thin film capillary columns

sorbent traps or flash evaporators

Pesticides, Industrial Pollutants

Fugitive Emissions

Industrial Emissions

Oil Spill Chemicals, PAHs

Green House Gases

Ozone Precursors

TO-14 Compounds

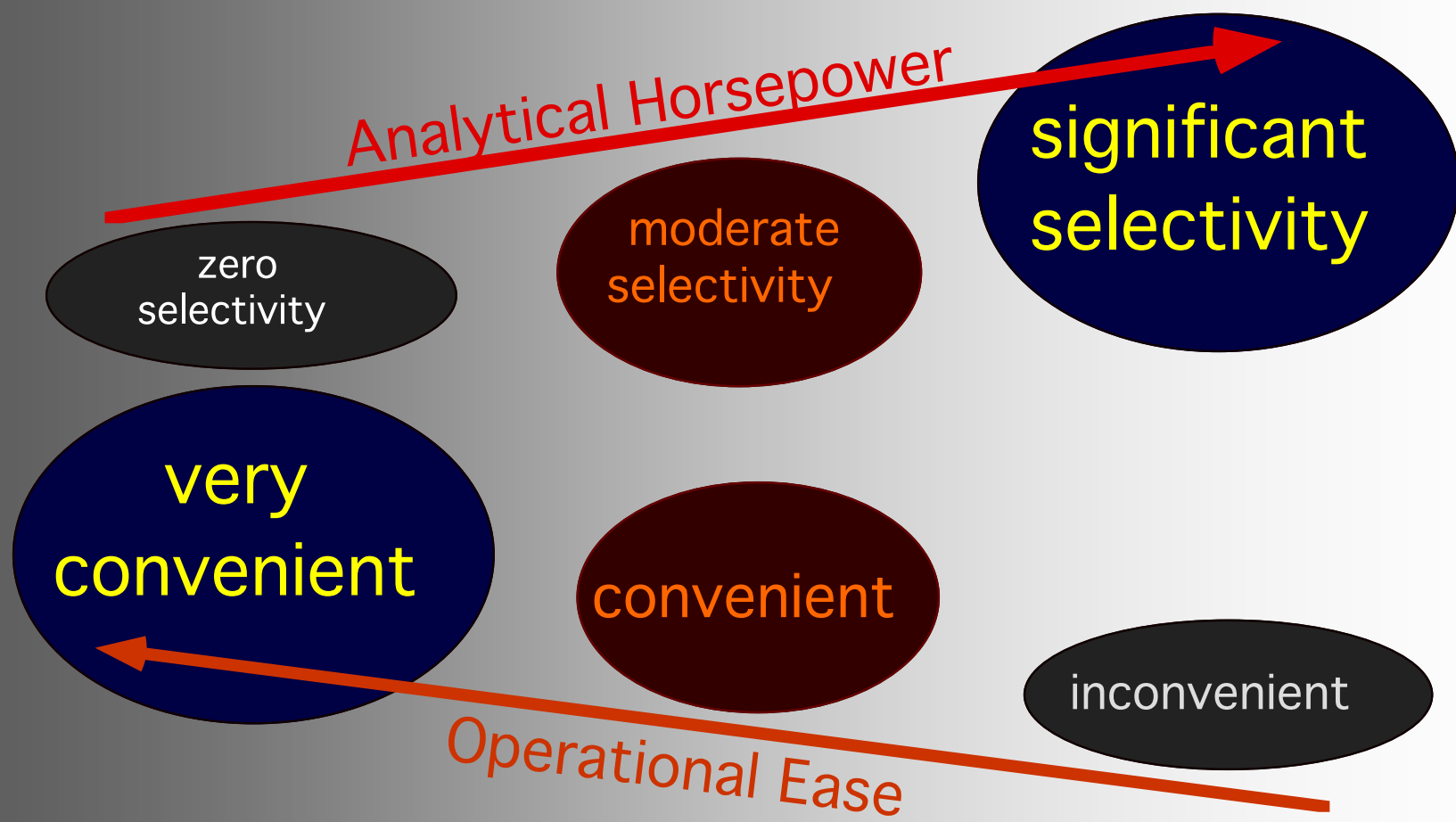
CWA Compounds

Breath Diagnostic Compounds



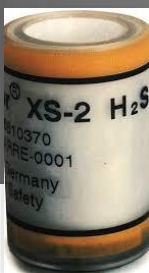
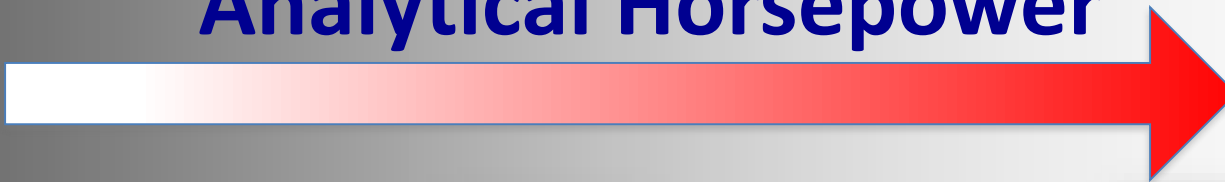
Compound Specific Analysis  
(tunably selective)  
Portability  
(transportable to portable)

= *Analytical Horsepower*  
= *Operational Ease*



Issues: false positives vs false negatives

# Analytical Horsepower



GC, IMS



GCMS



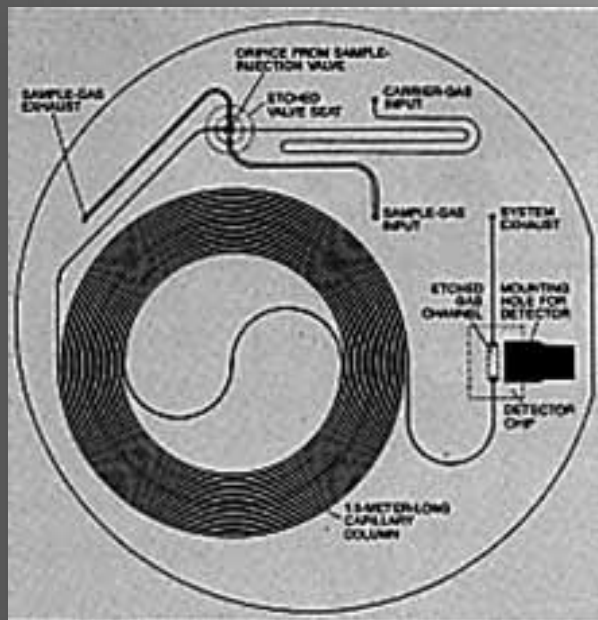
Sensors



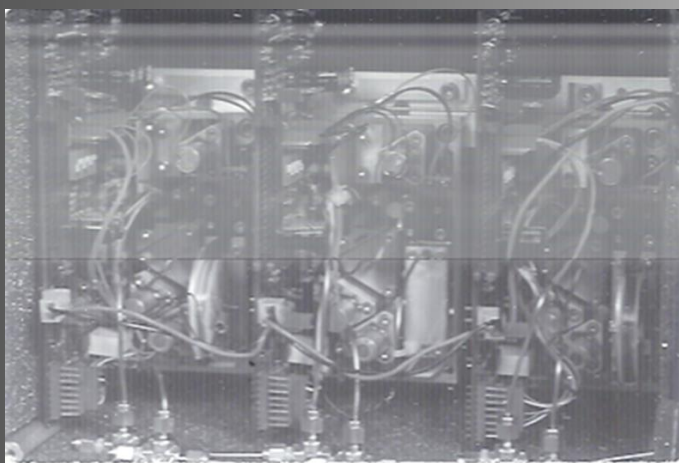
Ease of Use, Cost



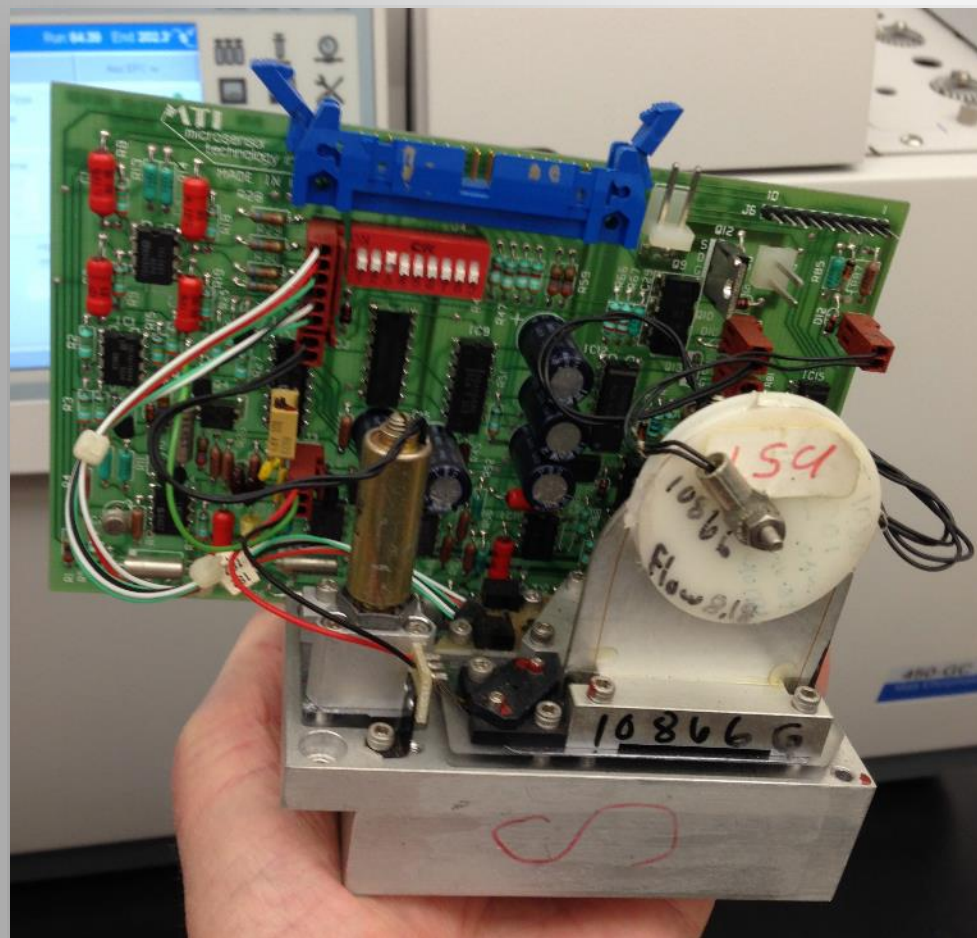
# Stanford "GC on a chip"



**Micromonitor 500**

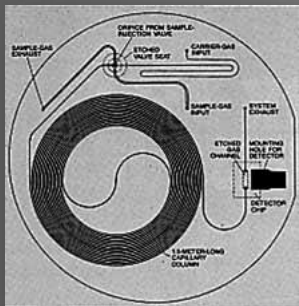


**Microtech NGA**

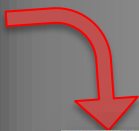




# Silicon micro GC Evolution



Stanford Silicon GC



MTI Micromonitor 500



Inficon Fusion



Agilent 490 PRO



Silicon Injector Chip



Agilent 3000



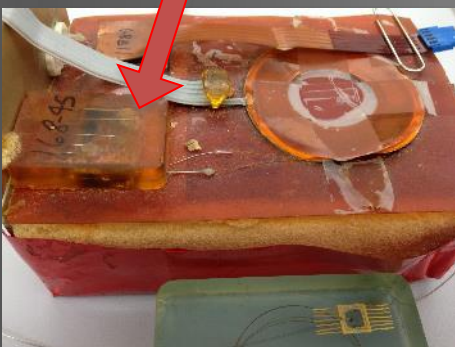
SLS μGC



C2V



CP 3200 Varian



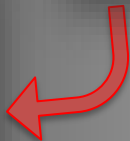
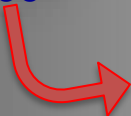
MTI Micromonitor 200



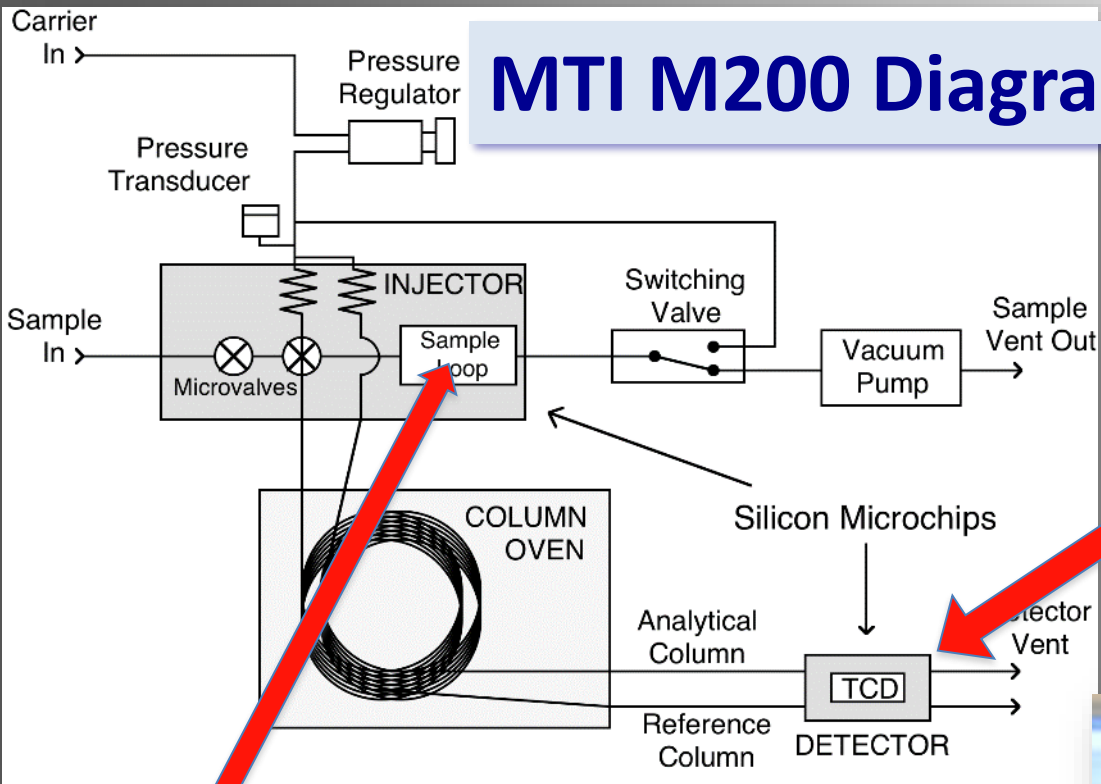
MTI M200



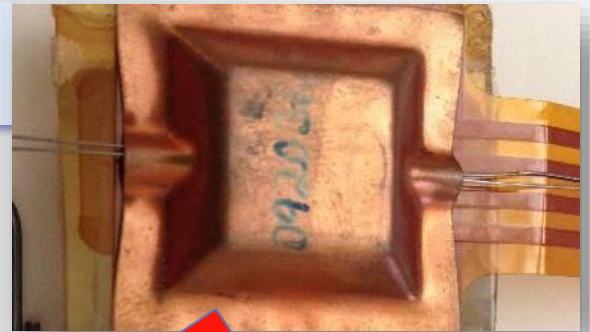
Chrompack CP 2000







# MTI M200 Diagram



Silicon TCD

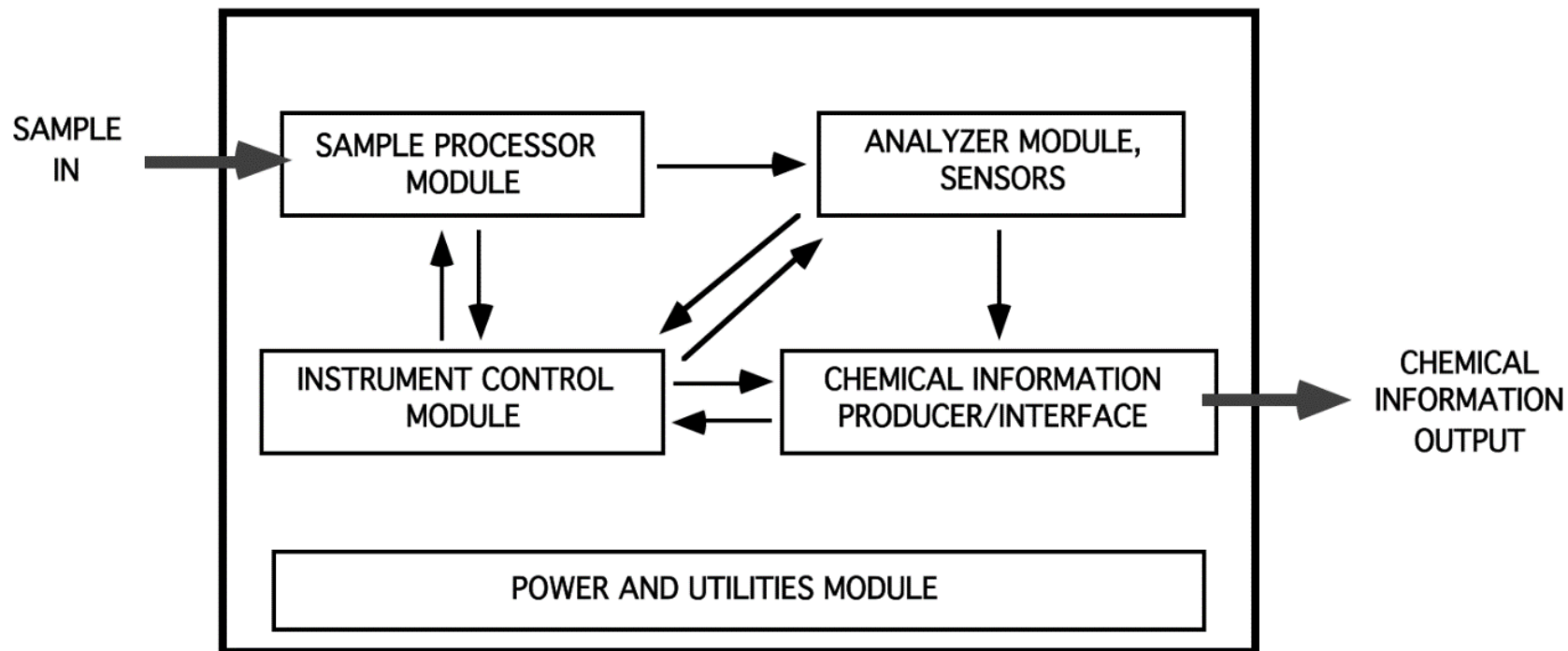
M200 GC module insulated



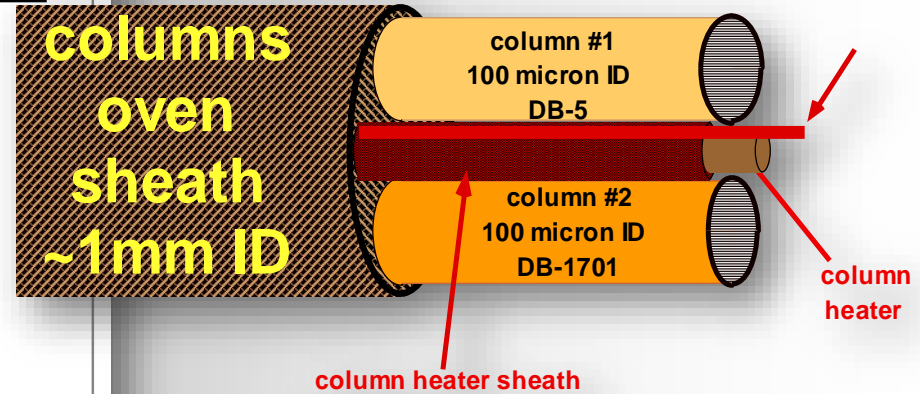
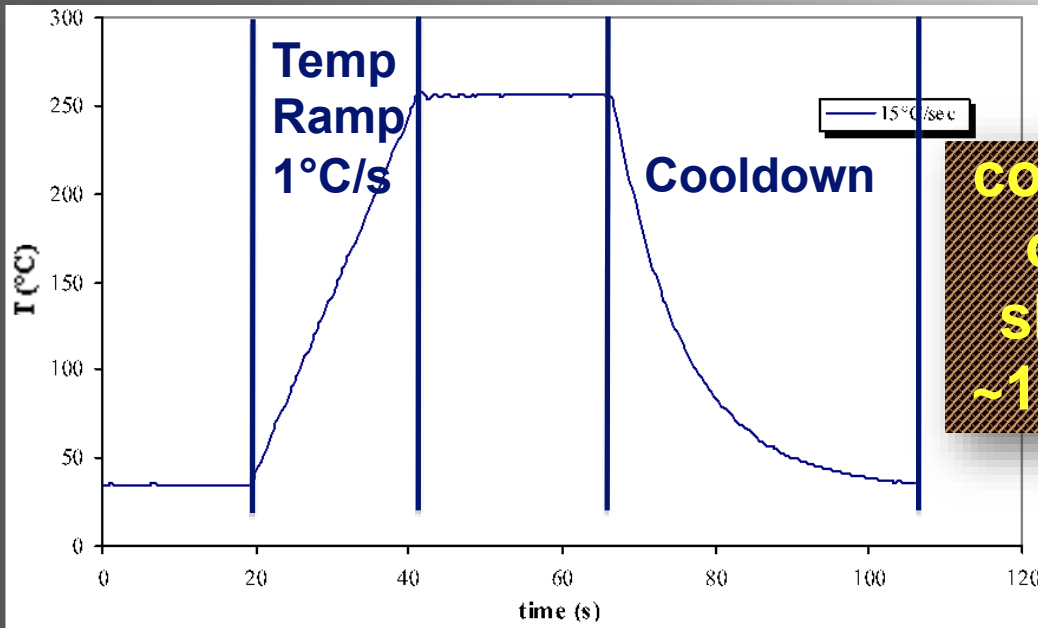
Acrylic encapsulated Silicon injector chip

only gases  
Ppm detection limits

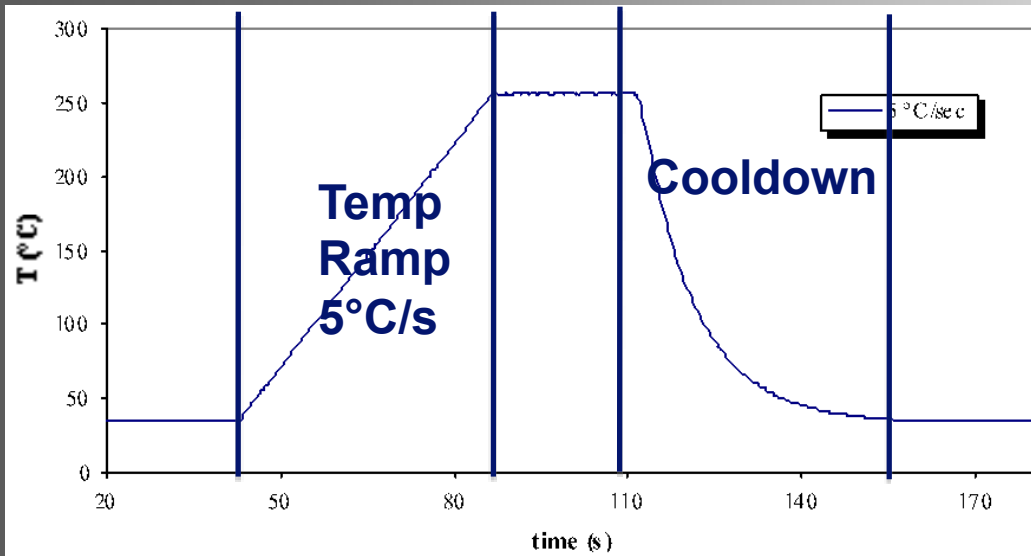
# IDEALIZED FIELDABLE ANALYTICAL INSTRUMENT FUNCTIONAL DIAGRAM



In the early 1900s, the DNA had a need for a portable analyzer for on-site detection of CWA precursors as required by the CWC.



## The microFAST GC's Column Temperature vs. Heating Rates



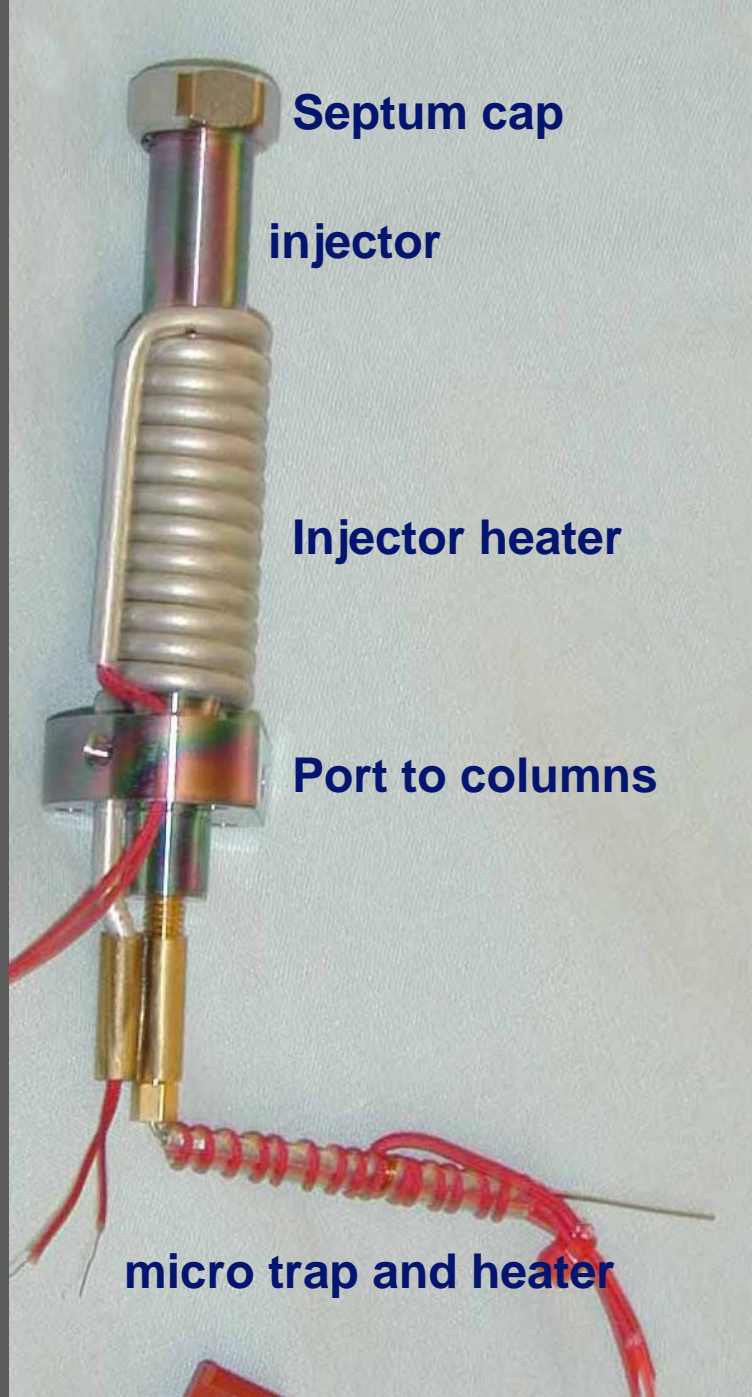
**A GC needs HEAT!  
microFAST GC  
delivered it.**

# What is the microFAST GC?



- A highly sensitive and selective micro gas chromatograph.
- A GC capable of very fast, low level compound measurements in the laboratory or field.
- Key features include *small size, ease of use, speed of analysis, and transportability.*
- Productivity and cost advantages over traditional GC units.

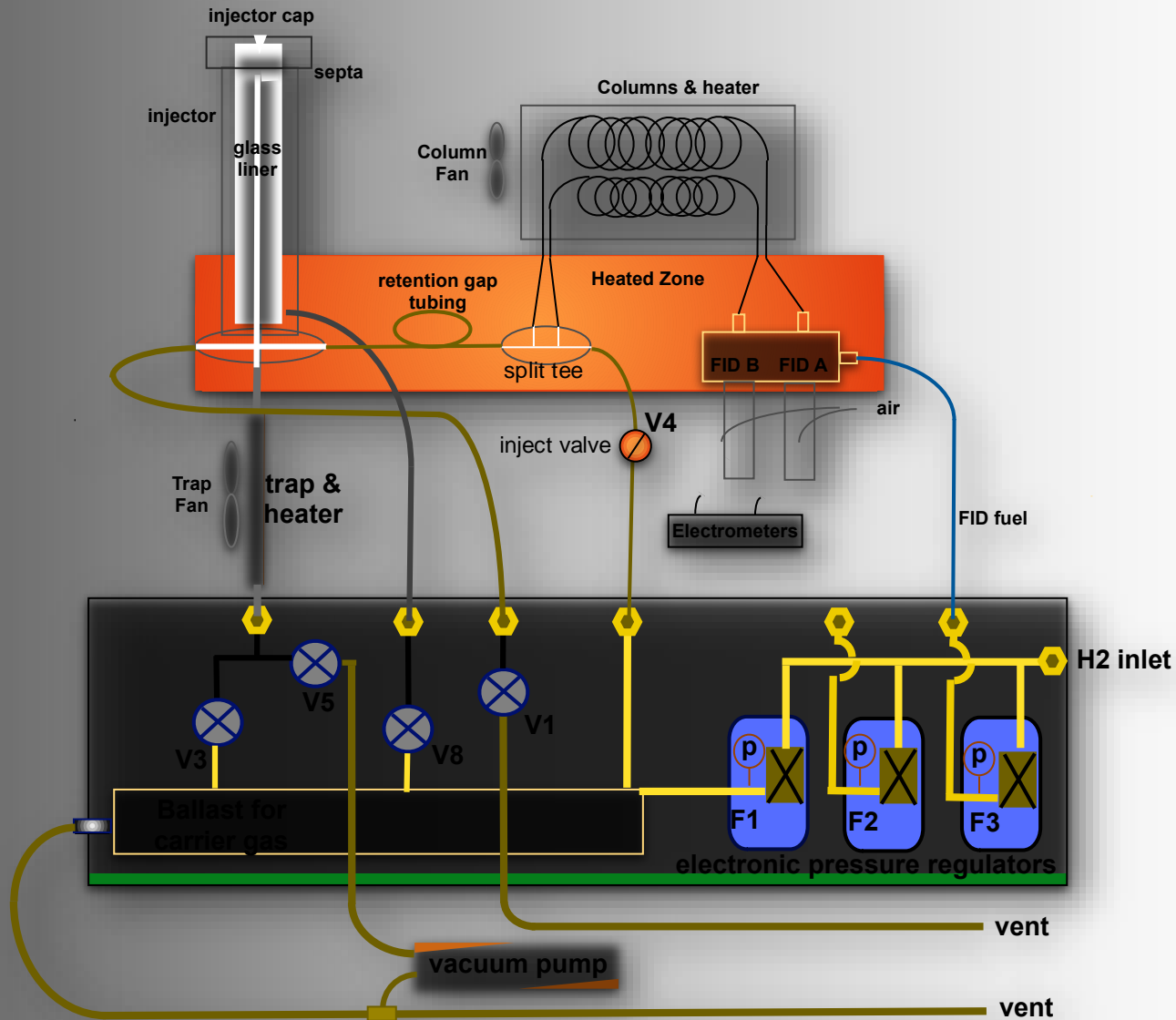




## Concentration Trap Injector Facilitates Multi Sampling Capacity:

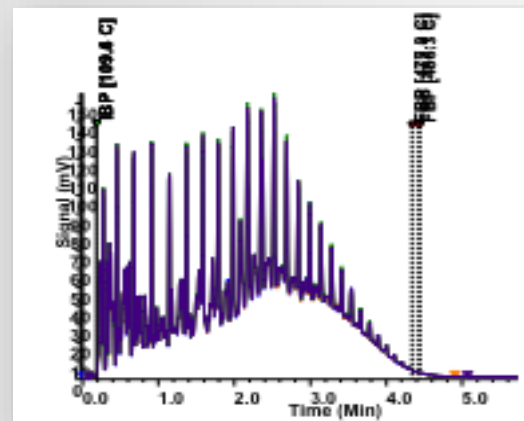
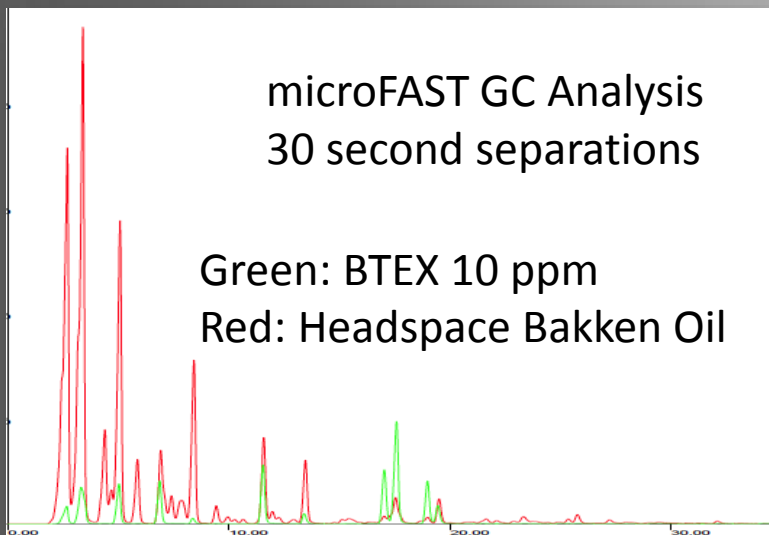
- Normal/large volume injections
- Gases, dilute gases (ppb level)
- Static and dynamic headspace
- SPME
- Membrane & external concentrators
- Purge and trap extracts
- Liquid organic solvent extracts
- Neat organic liquid mixtures
- Aqueous liquids
- Thermal desorption tubes
- Thermal and SCF extracts

# microFAST GC Functional Diagram



microFAST GC Analysis  
30 second separations

Green: BTEX 10 ppm  
Red: Headspace Bakken Oil



Falcon Calidus 101HT  $\mu$ GC

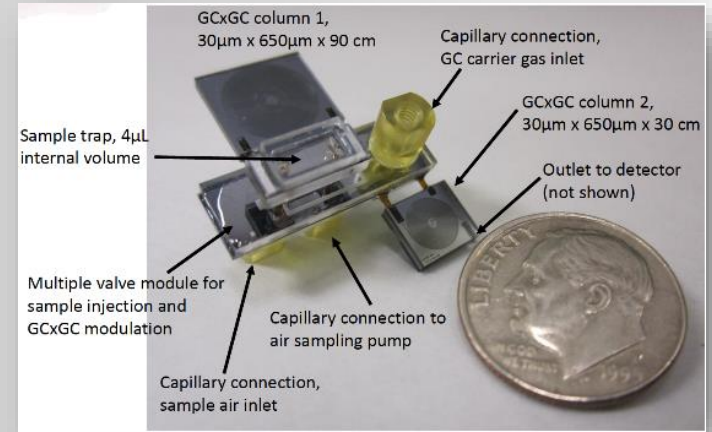
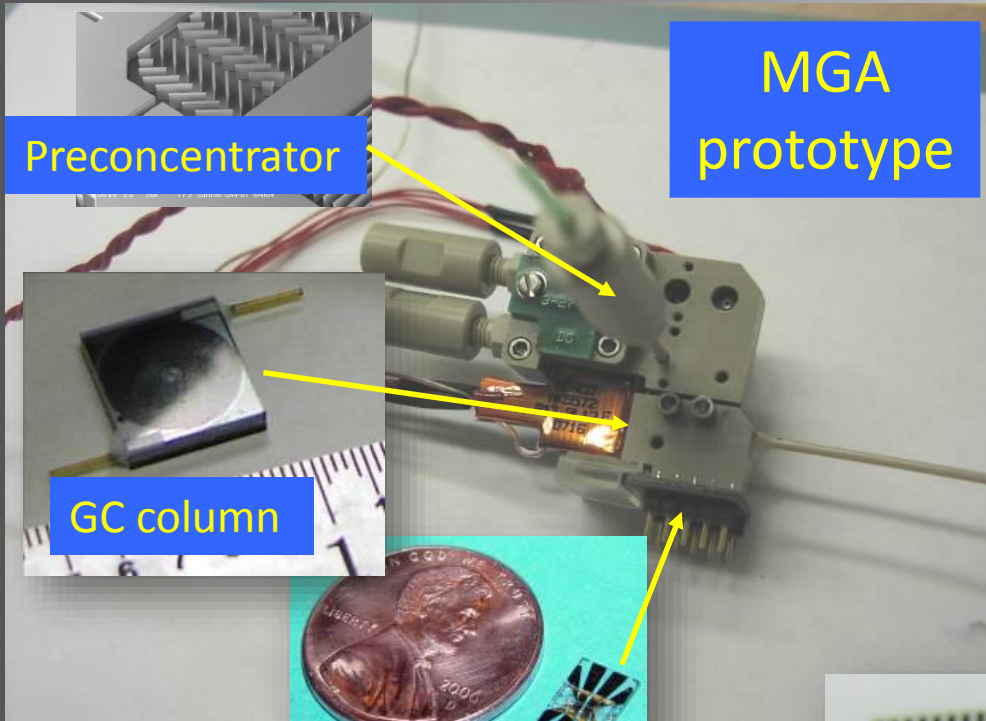


microFAST GC

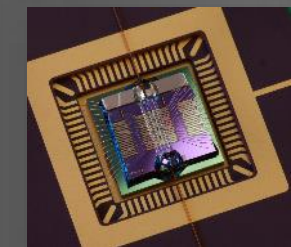




# Micro Fabricated GC Systems

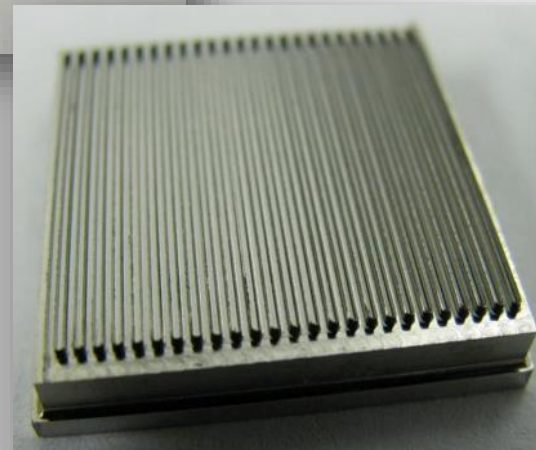
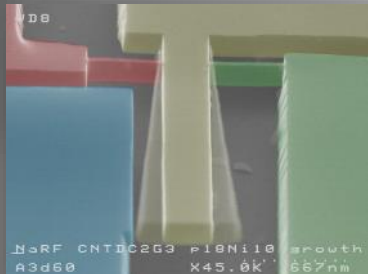


## Aluminum Column

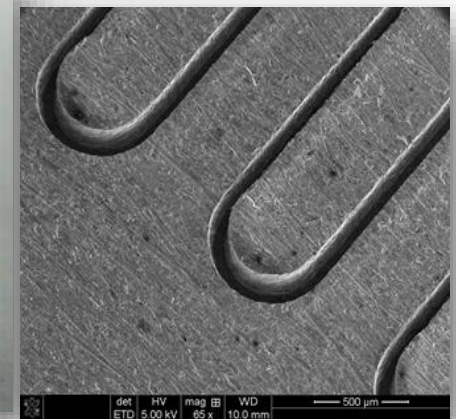


Vibrating  
Cantilever  
Detector

Nanoparticle  
chemiresistor



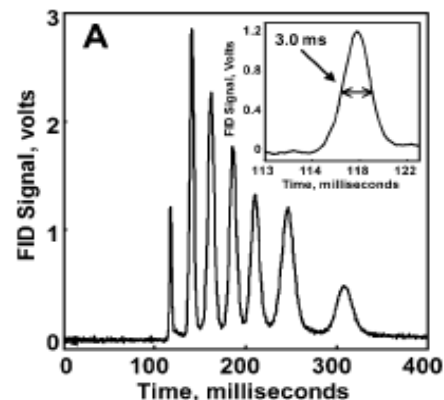
Mold Insert



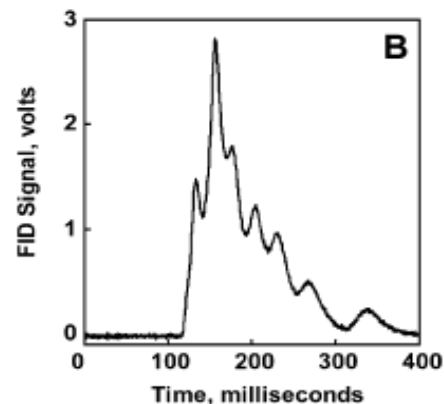


**Fast GC Analysis**  
required extremely fast  
sample band injections  
also fast temperature  
programming, fast detector  
response

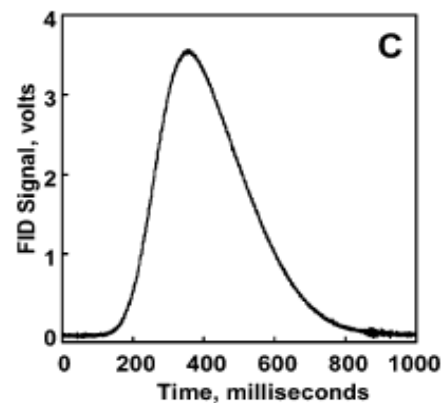
**2.5 ms**



**20 ms**



**>1 sec**

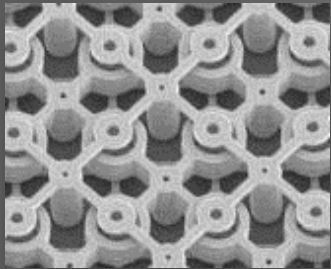


1 meter DB-5 column  
100 $\mu$  ID 0.4 $\mu$  film  
150 $^{\circ}$  C isothermal  
85 psi head pressure



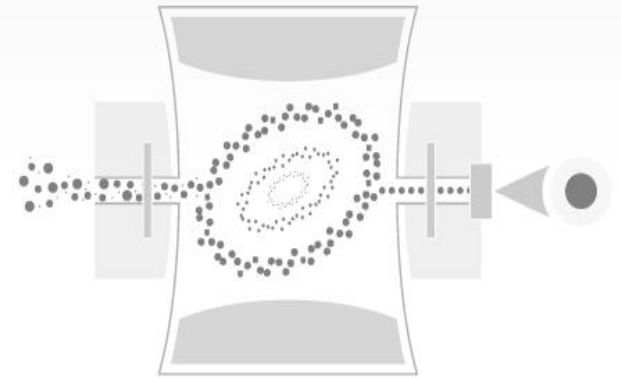
# 908 Devices

27 Drydock Ave., 8th Floor  
Boston, MA 02210



## Purpose-built

Many applications just don't require all the complexity these laboratory platforms incorporate. We are building ridiculously small, and elegantly simple purpose-built products based on remarkable mass spectrometry technology. These systems are designed for specific applications in security, biotechnology,



## near atmospheric pressure Ion Trap Mass Spectrometer



## Secret sauce

At the heart of our systems are molecular traps a thousand times smaller than those in conventional mass spectrometers. These diminutive traps can operate much closer to atmospheric pressures and enable us to use dramatically smaller pumps, ionizers, detectors and electronics than existing laboratory or luggage mass spectrometers. We call this



**Questions?**