Overview of DR R's Research Interests

- **Environmental analysis of heavy metals in sediments, fish, and other matrices**

- **Development of methodologies for the determination of methyl mercury in fish and other matrices**

- **Evaluation of new micro-columns as sample adsorbents and GC columns in lab-on-a-chip designs**

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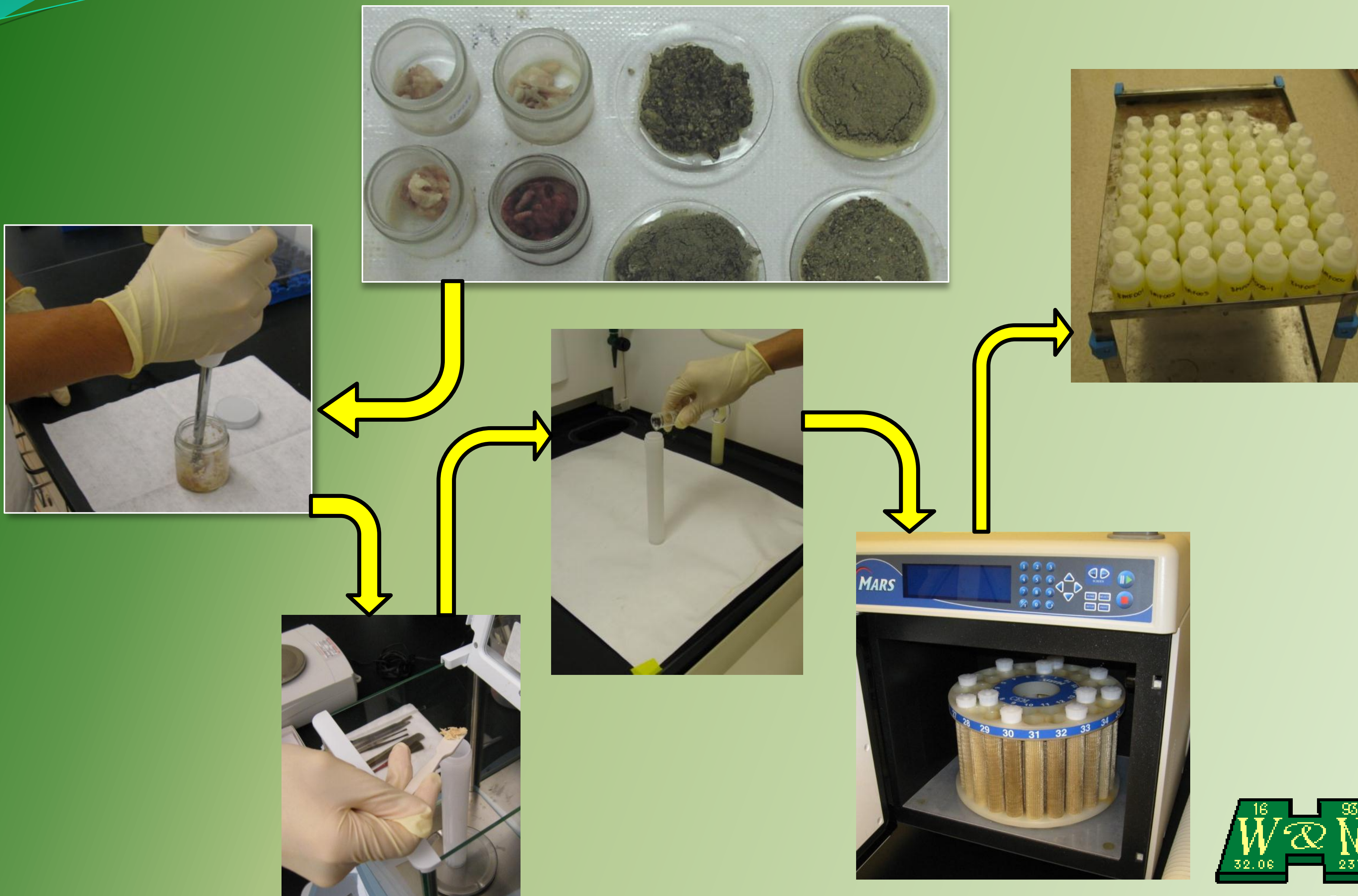
Heavy Metals in Sediments and Fish



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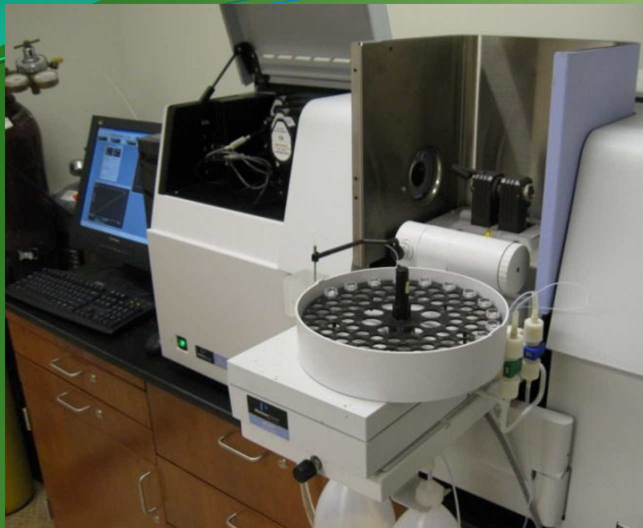
Several instruments are used for elemental determinations:



Graphite furnace atomic absorption spectrometry

Inductively coupled plasma spectroscopy

Automated mercury analyzer – based on atomic absorption from reduction of mercury via $SnCl_2$ (sub-ppb limits of detection)

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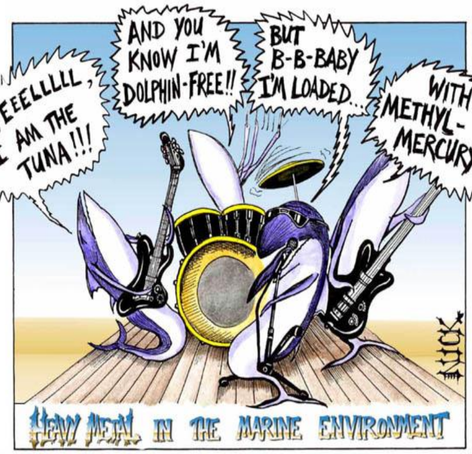
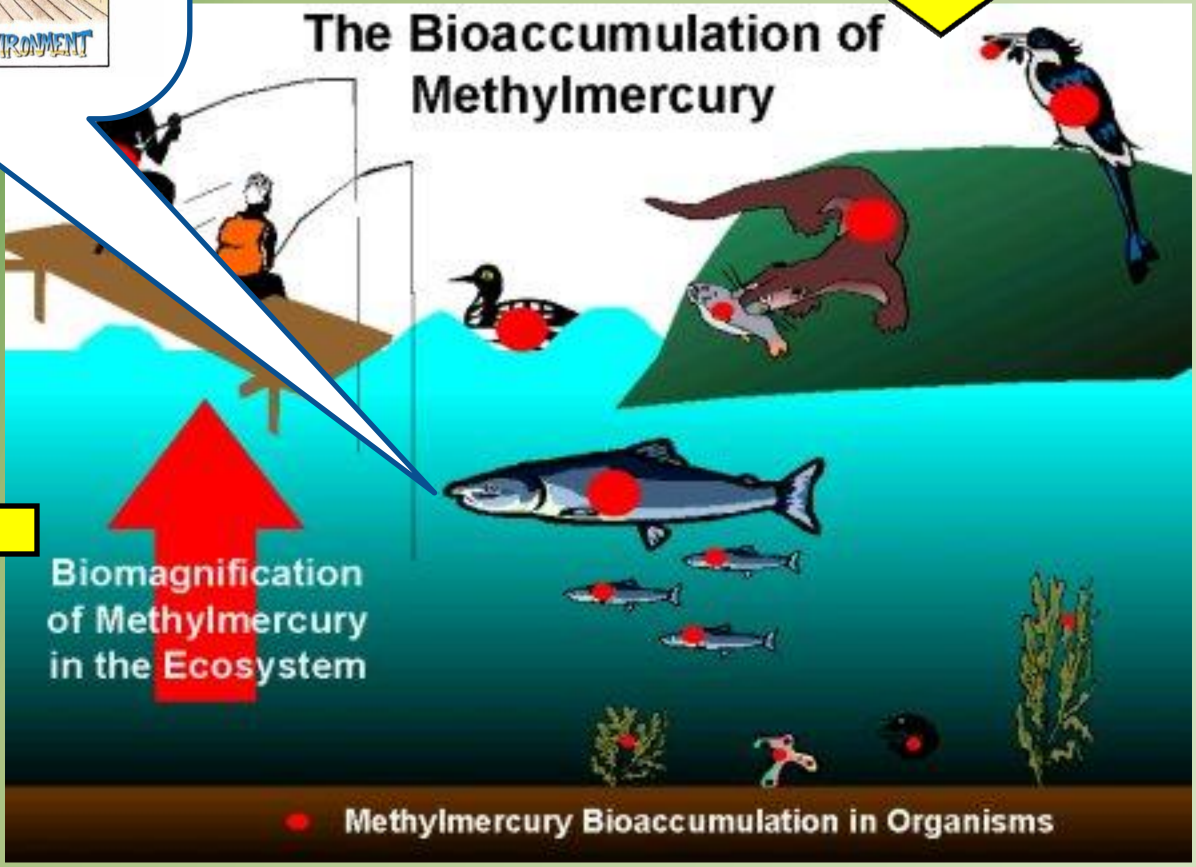
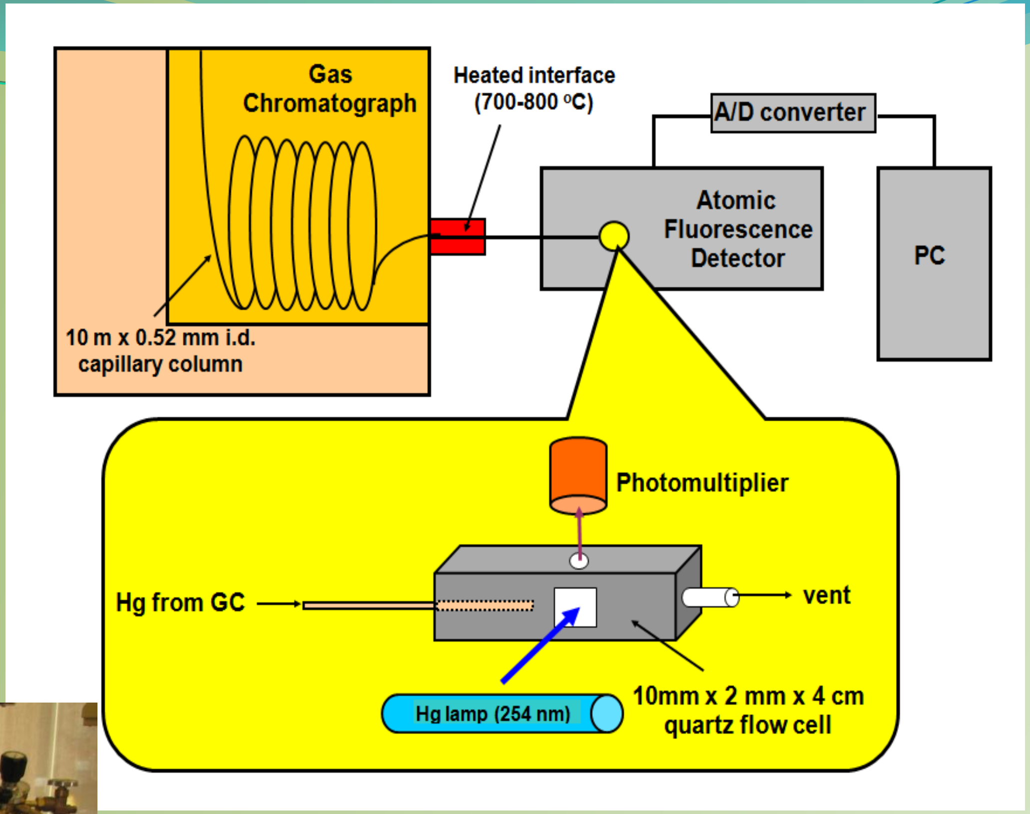
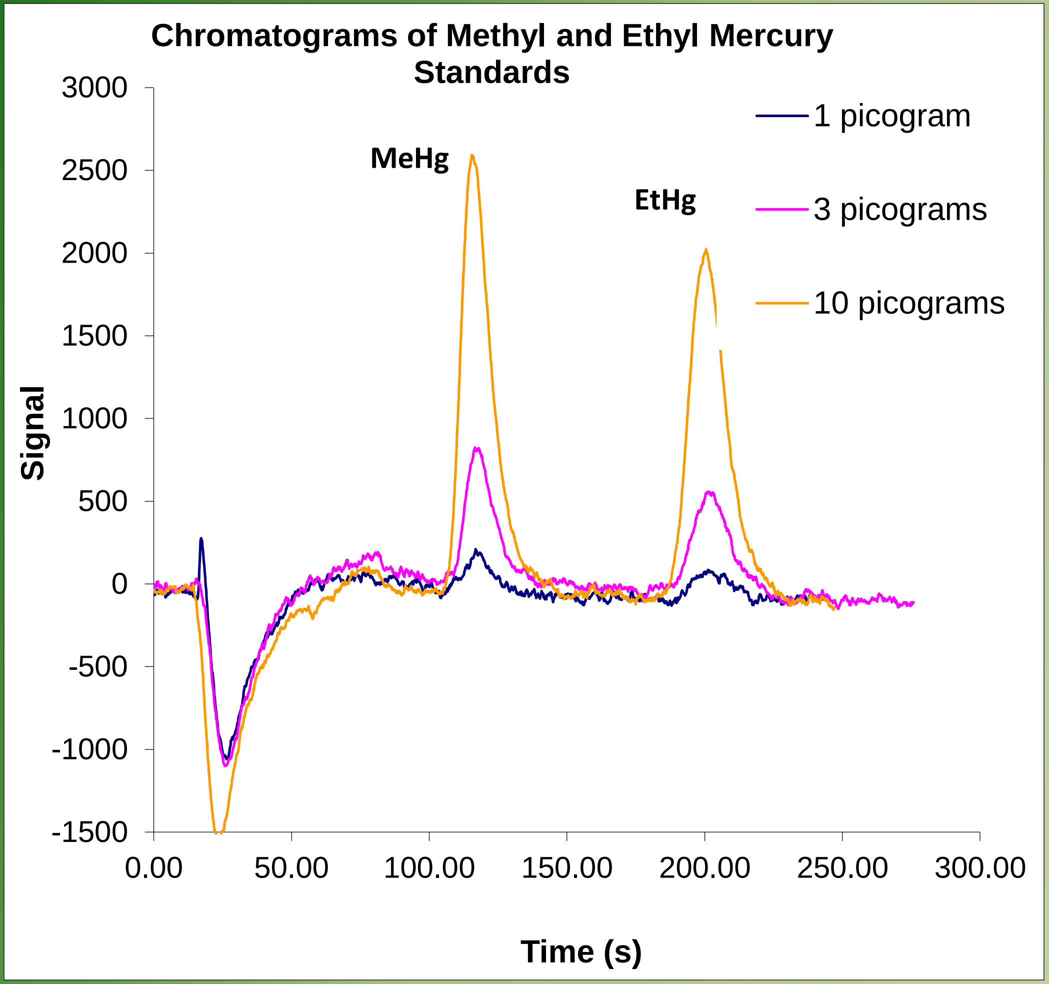

WEEELLLLL, I AM THE TUNA!!!
AND YOU KNOW I'M DOLPHIN-FREE!!
BUT B-B-BABY I'M LOADED...
WITH METHYL-MERCURY!!!
HEAVY METAL IN THE MARINE ENVIRONMENT


The Bioaccumulation of Methylmercury
Biomagnification of Methylmercury in the Ecosystem
Methylmercury Bioaccumulation in Organisms

Gas Chromatography-Atomic Fluorescence Spectroscopy (GC-AFS)



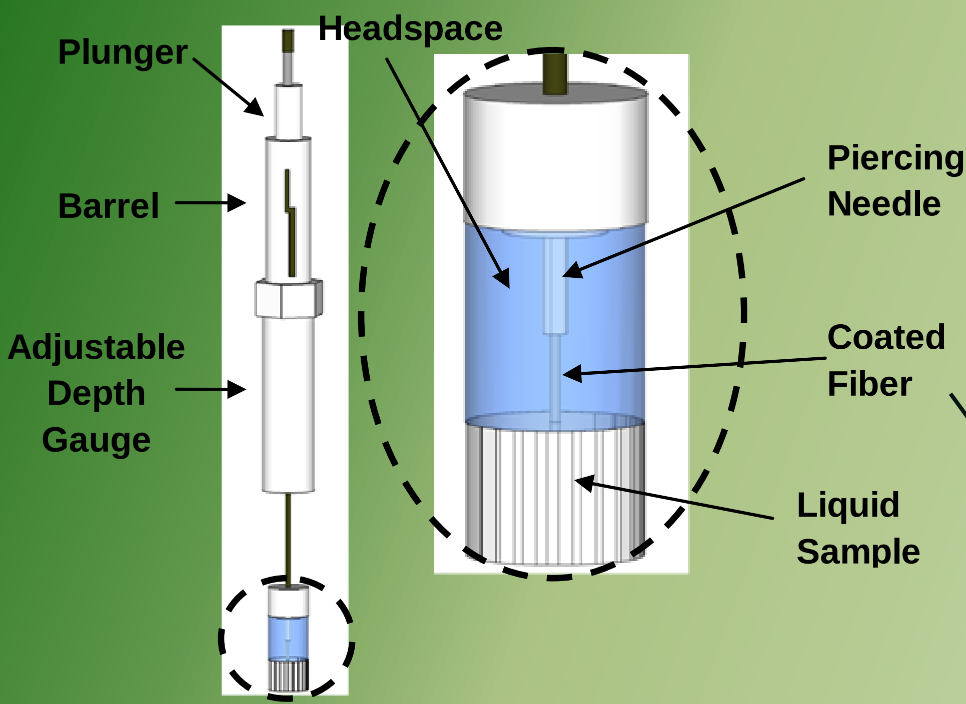


Current research is focused on simply extracting methylmercury as the chloride salt (MeHgCl) and using EtHgCl as an internal standard. Both of these can be separated using GC.

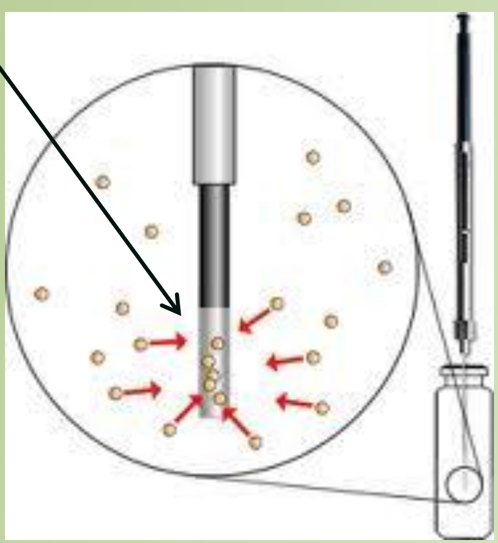
MeHg-R + HCl $\rightarrow$ MeHgCl

An extraction procedure is being developed for removal of MeHg from tissue by using saturated salt solutions and sonication methods.

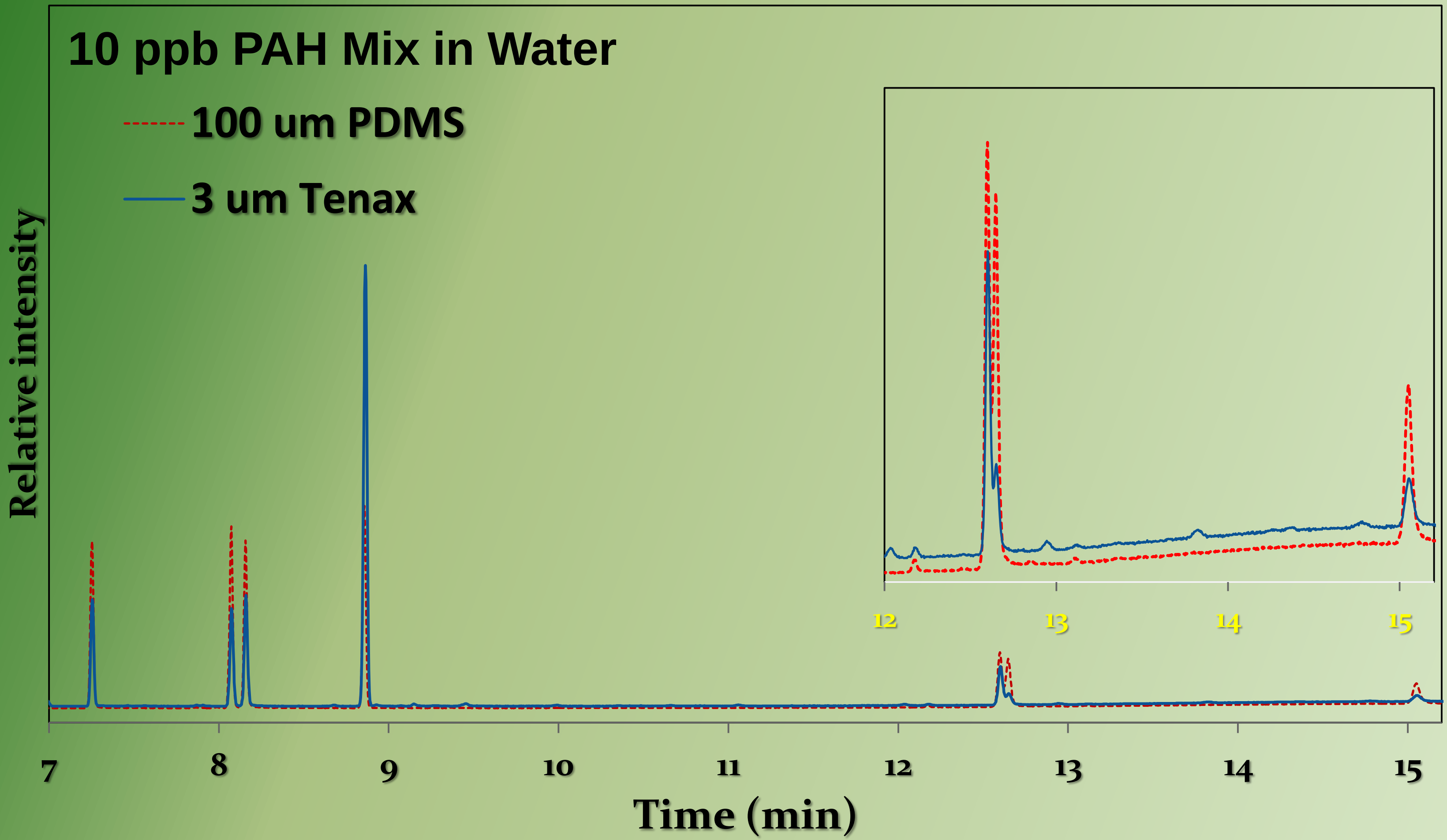
SPME – Solid Phase MicroExtraction



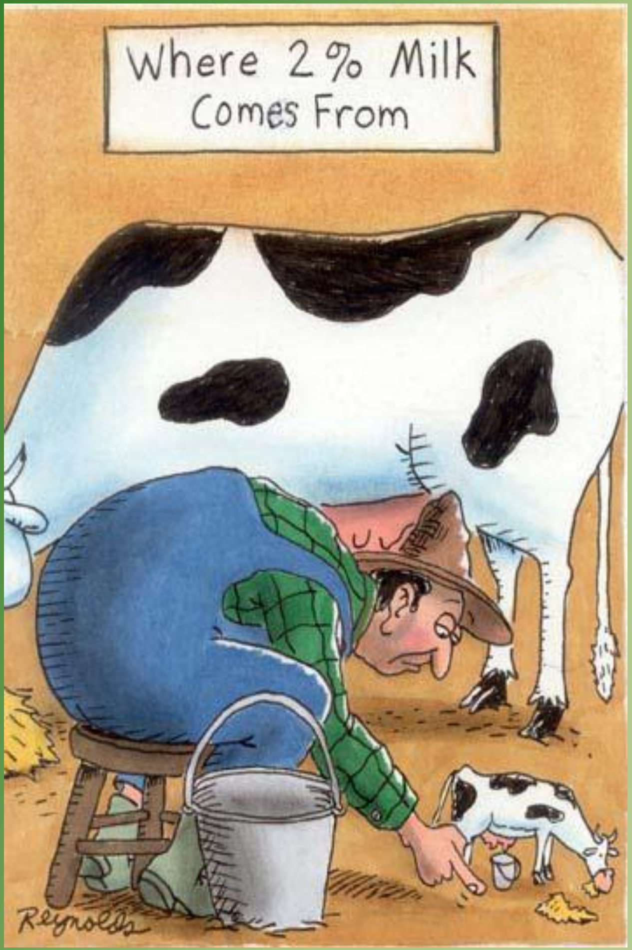
- Methodology for coating fibers with new adsorbent (Tenax) for high temperature applications (>300 C) being developed.
- Currently limited by 3 um coating on the fiber. Needs to be 10-50 um to compete with commercially available fibers.

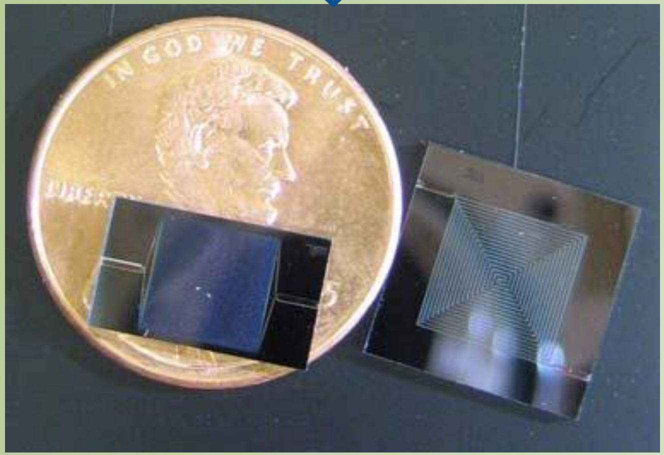


SPME – Solid Phase MicroExtraction



Micro-GC columns and Preconcentrators





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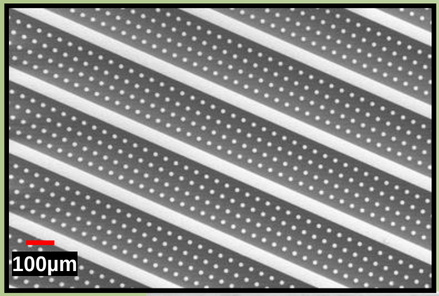
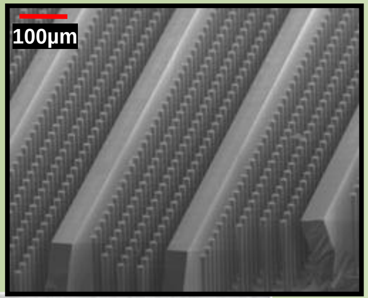
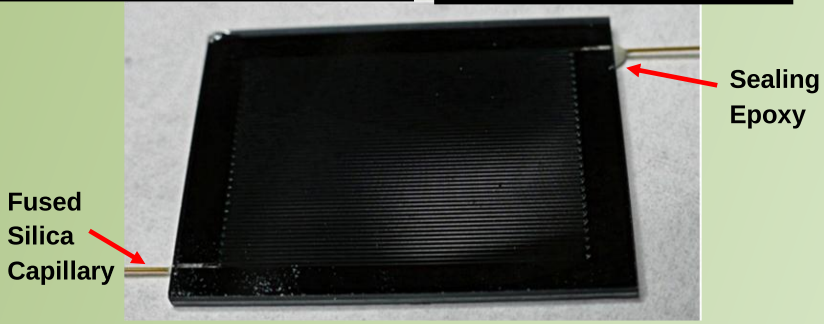
Micro-GC and Preconcentrators

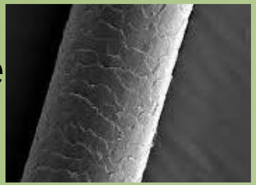
- Significant interest in the development of low power micro-instrumentation
- Major obstacles in micro-GC include low sample capacity (for separations) and low breakthrough volumes (for preconcentration of analytes)
- One possible solution is to use columns consisting of nanofabricated columns throughout the primary column channel

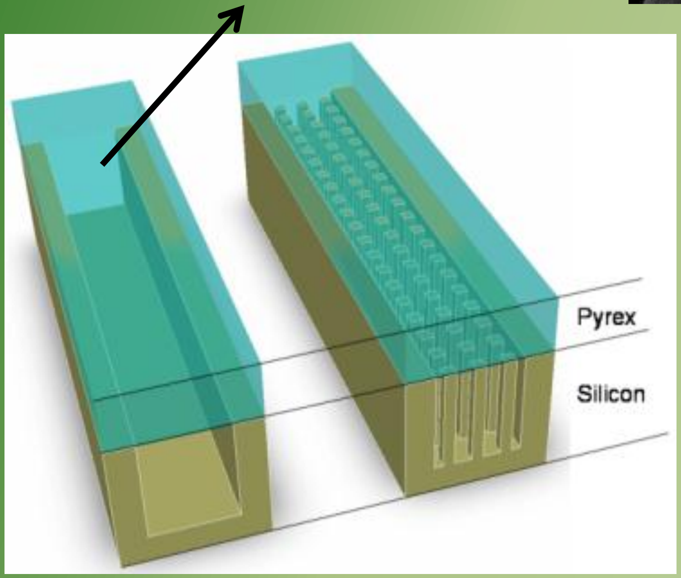






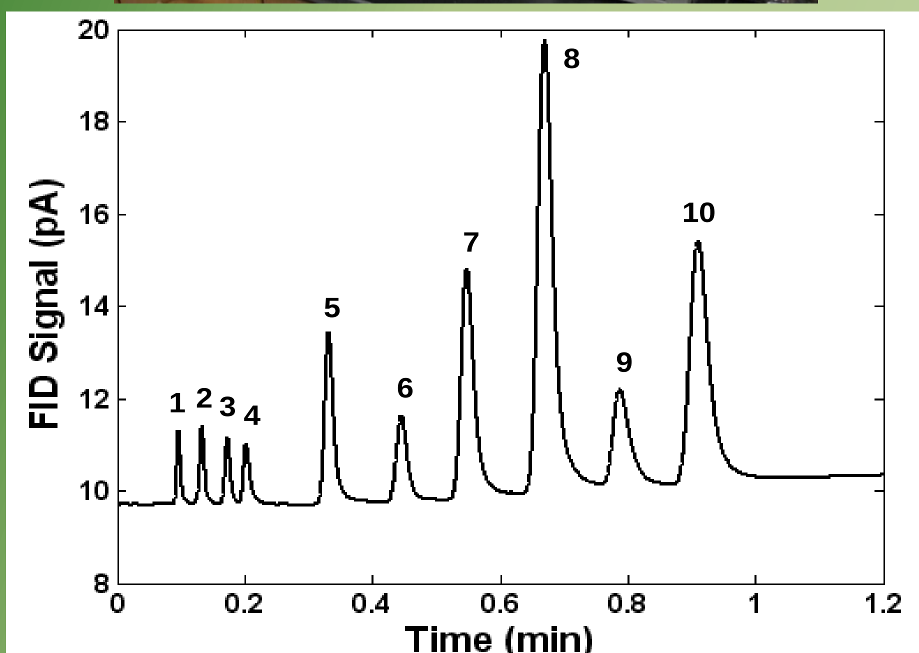
This width is comparable to the diameter of a human hair!!!





- Two NSF collaborations with Virginia Tech for which we are responsible for evaluating:
  - Column performance relative to conventional capillary columns for separations using different stationary phases, column coating techniques, and column architectures.
  - Performance of preconcentration adsorbers with respect to breakthrough volumes (sample capacity) using various stationary phases and column geometries.

MEMS (microelectromechanical systems) Column Evaluation



VOC vapor mix

1) Dichloromethane
2) Chloroform
3) Carbon tetrachloride
4) Dibromomethane
5) Toluene
6) Tetrachloroethylene
7) Chlorobenzene
8) p-xylene
9) 1,1,2,2-tetrachloroethane
10)Bromobenzene

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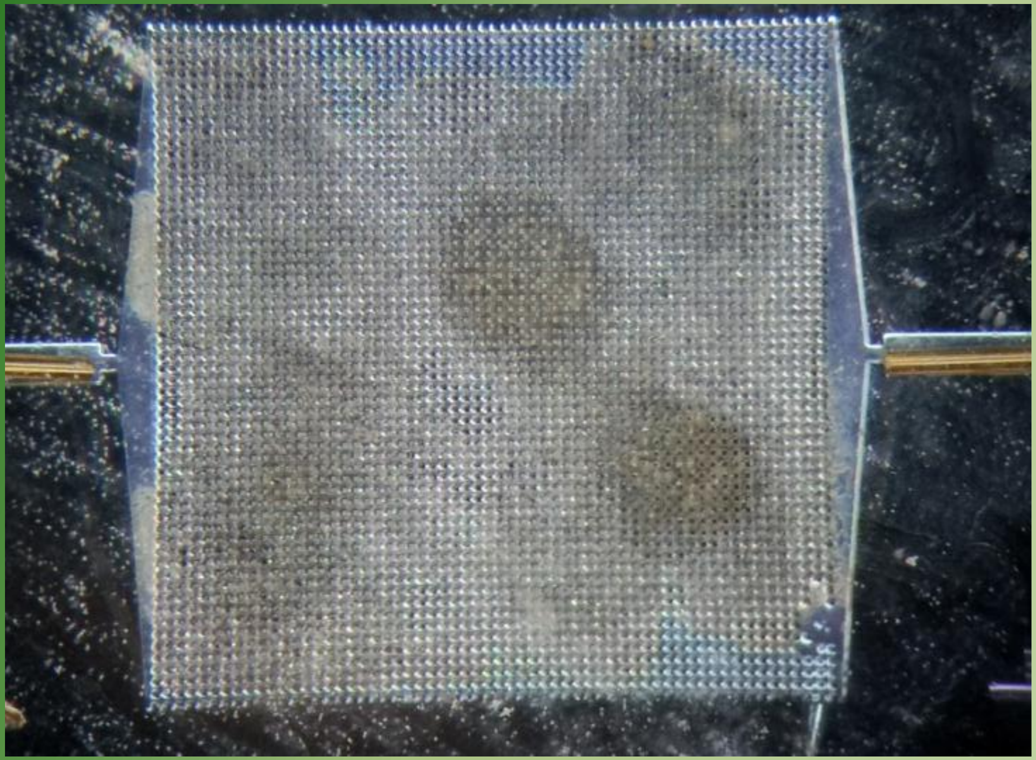
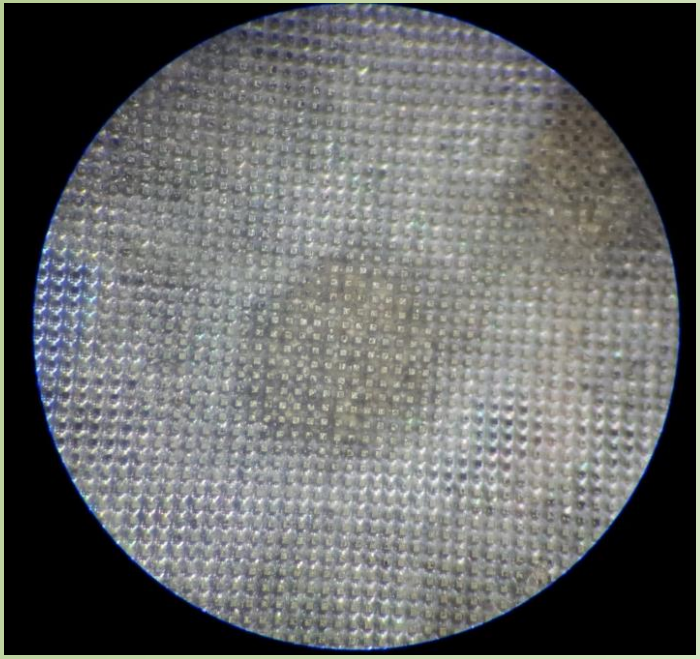
Micro-preconcentrators for micro-GC applications

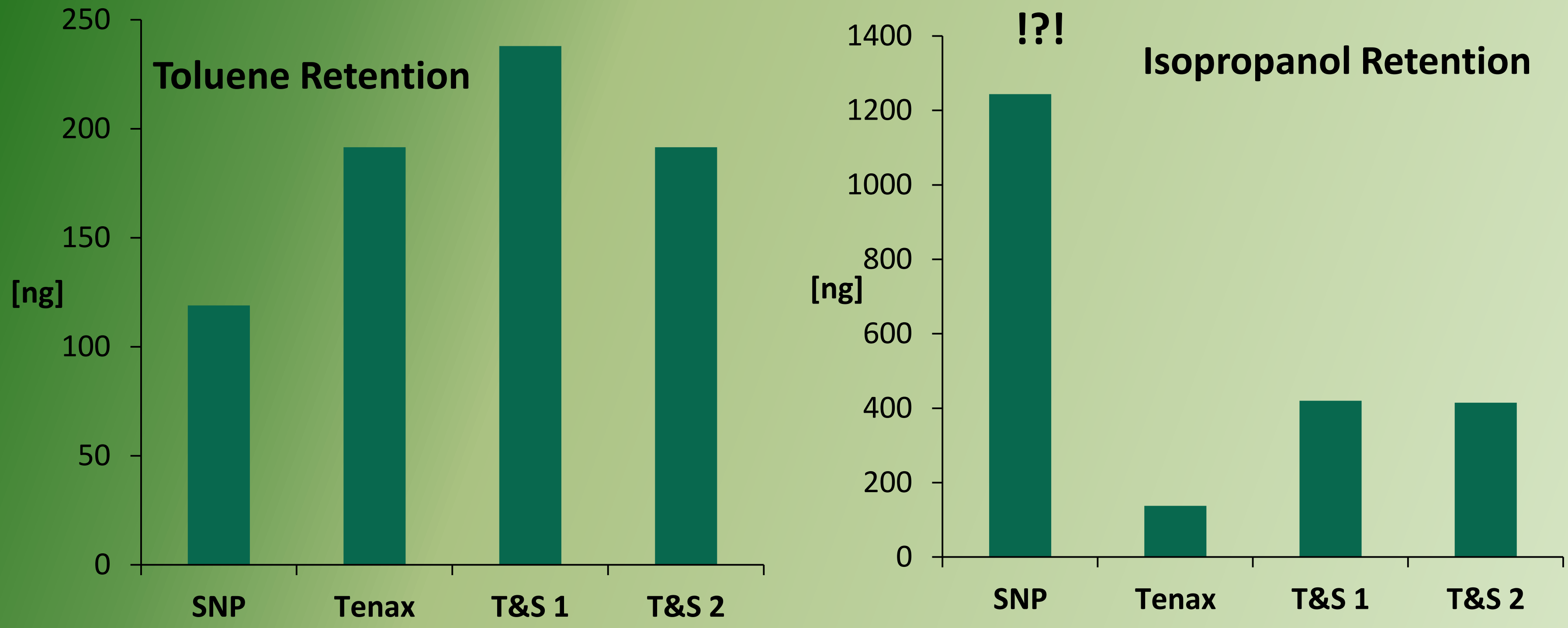
What provides the best medium for maximum adsorption?





~1 cm

Microconcentrator Performance



New column coating technology

In the **sol-gel** coating method, the surface treatment, deactivation, column coating, and immobilization occur simultaneously, not in individual steps.

Advantages:

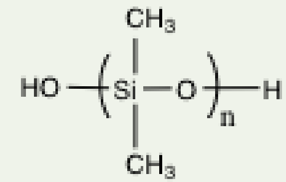
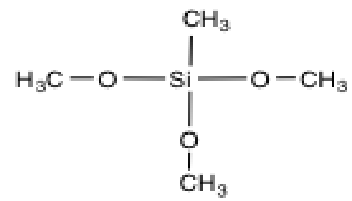
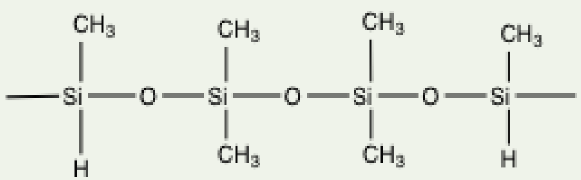
Single step process greatly simplifies procedure

Polymer physically bonds to the column walls at active hydroxide sites

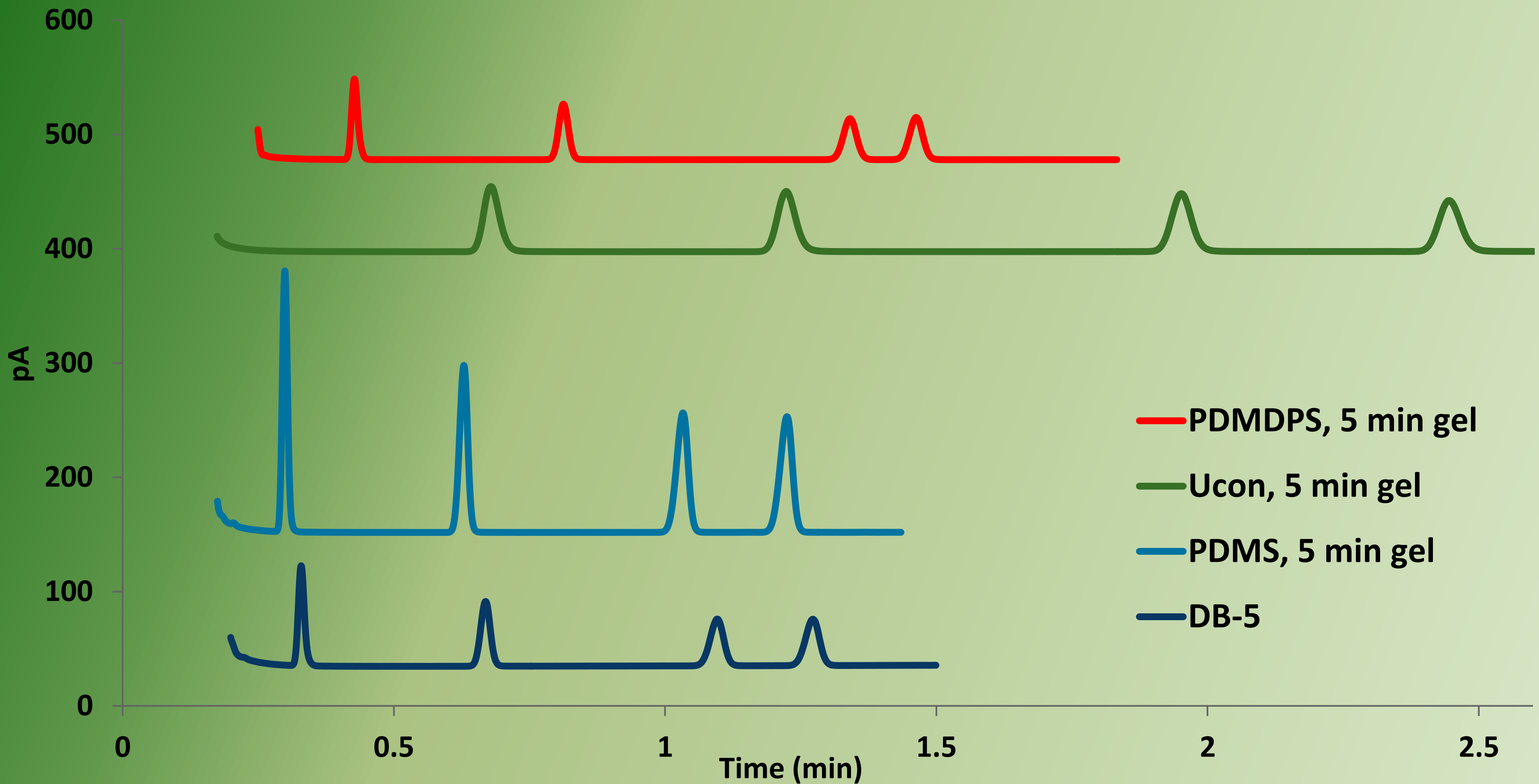
Process already available:

Sol-Gel Column Technology for Single-Step Deactivation, Coating, and Stationary-Phase Immobilization in High-Resolution Capillary Gas Chromatography. *Analytical Chem,* **1997,** 69, 4566-4576.

Malik, Abdul; Wang, Dongxin. Capillary Column and Method of Making. Patent No. WOO2000011463, March 2, 2000.

Materials	Function	Structure
poly(dimethylsiloxane) (PDMS)	stationary phase polymer	
methyltrimethoxysilane (MTMS)	precursor	
poly(methylhydrosiloxane) (PMHS)	deactivation reagent	
trifluoroacetic acid, 95% (TFA)	catalyst	CF_3COOH
dichloromethane	solvent	CH_2Cl_2

Separation of Aromatics



Test mix: benzene, toluene, chlorobenzene, xylene in pentane (1 ppthousand by volume)

Temperature: 30°C - 15°C/min - 80°C; Pressure: 5 psi

Stationary Phase	Sol-Gel Time (min)	Optimum Pressure (psi)	Theoretical Plates (per meter)
DB-5 (standard)	---	7.5	7080
PDMS sol-gel	5	5	5840
Ucon sol-gel	5	2.5	1590
PDMDPS sol-gel	5	5	5670
MEMS-SNP	10	5	2110

? QUESTIONS ?

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