

Soild Phase Microextraction for Detection of Chemical Warfare Agents



Gary Hook, PhD, CIH
CDR, MSC, USN

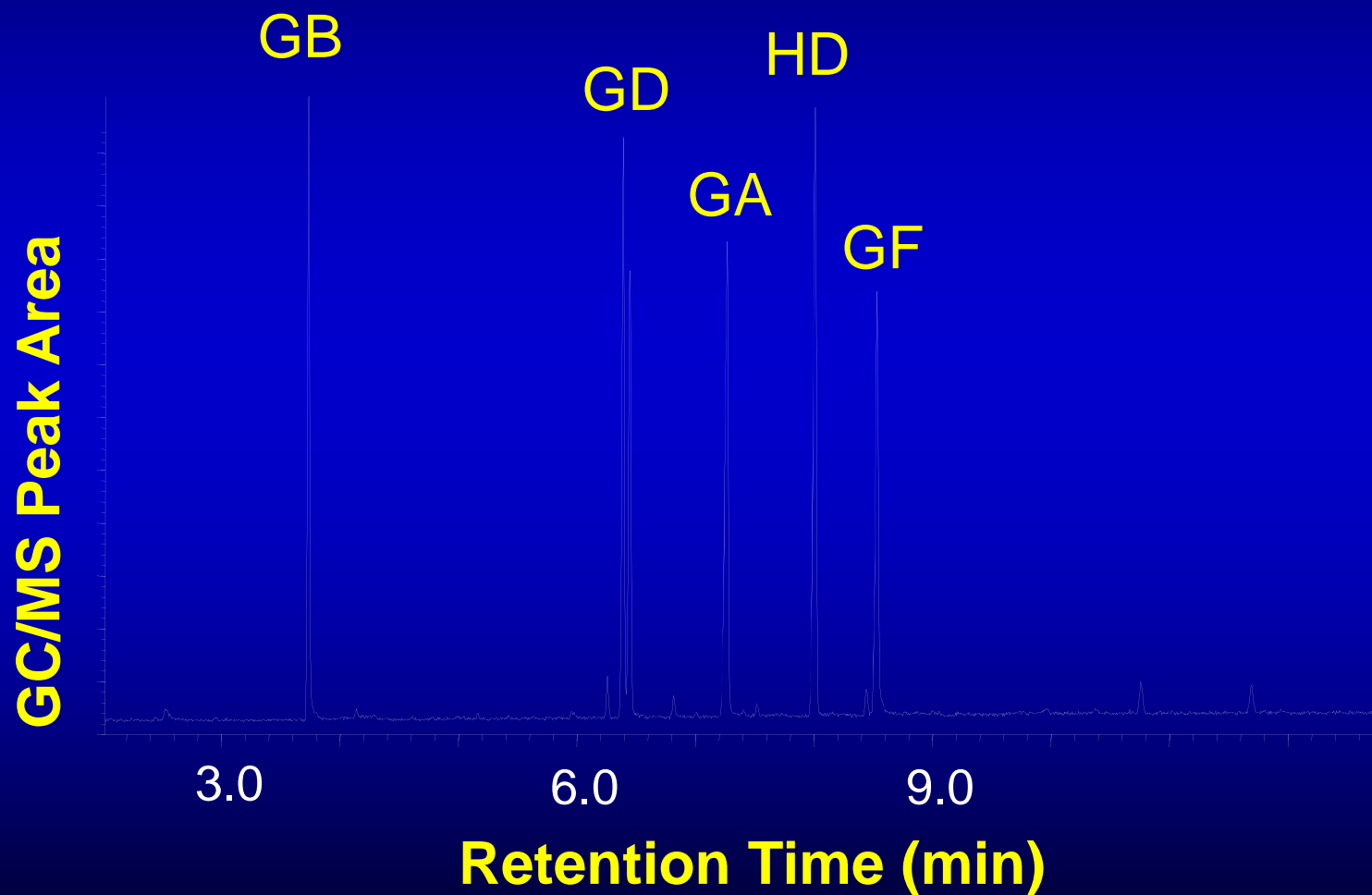
Where are we going?

- Qualitative detection
- Quantitation
- Current Research
- Training for GC/MS Operators
- SPME Equipment

Field Portable GC/MS



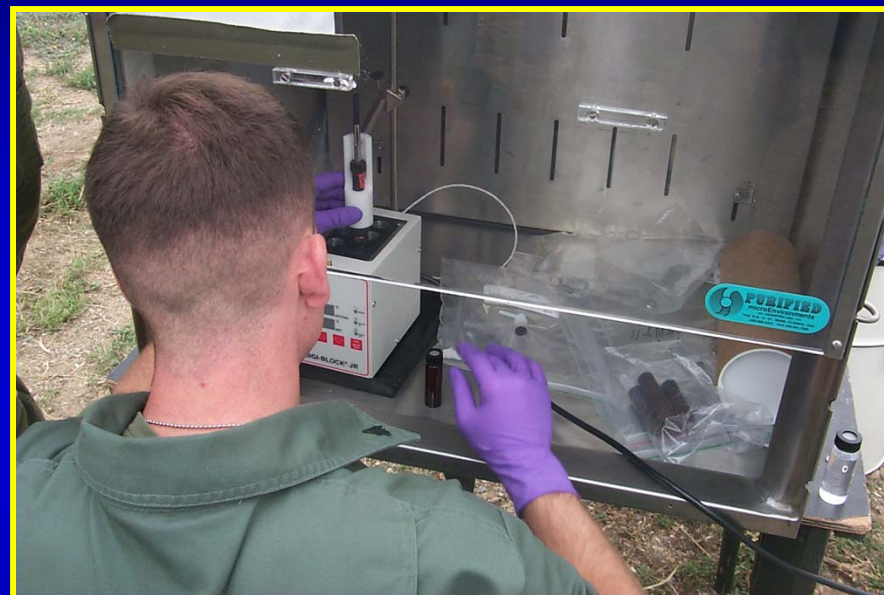
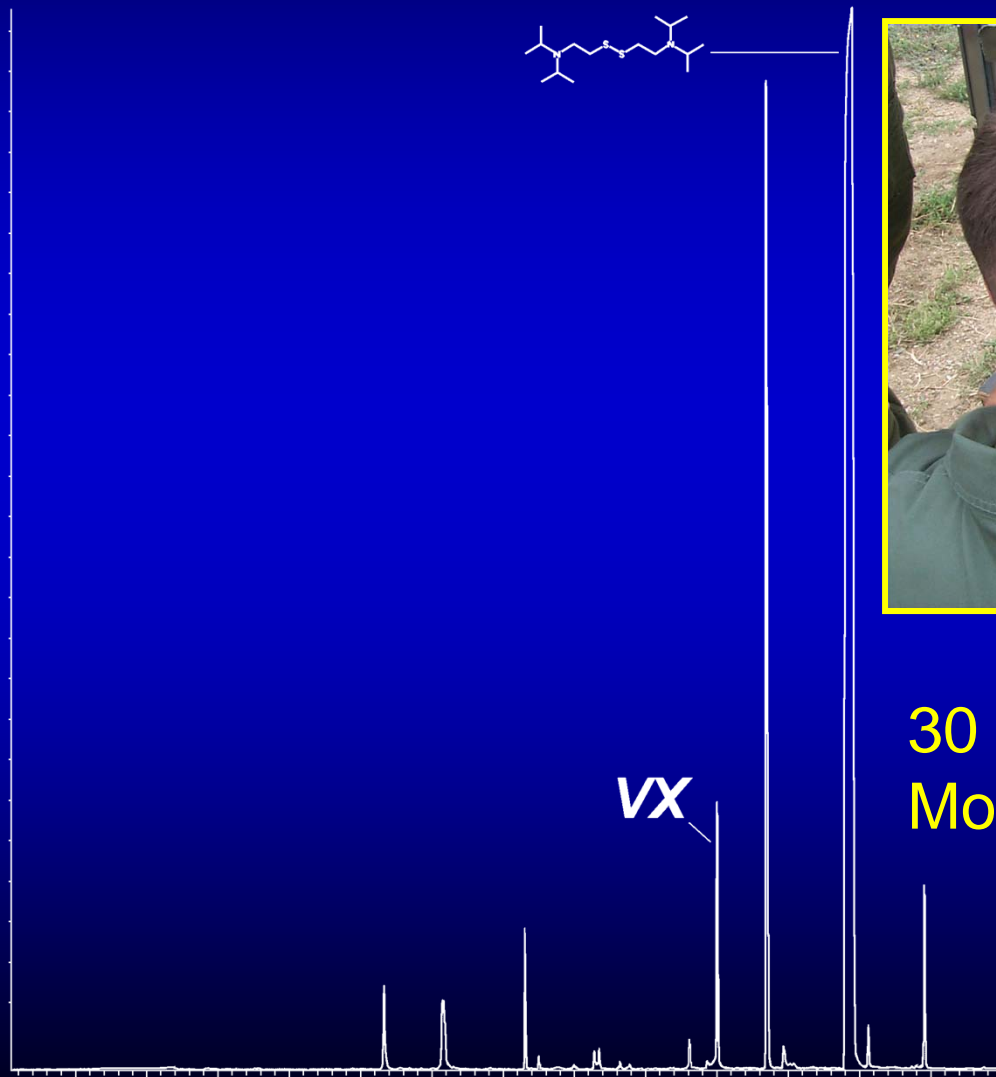
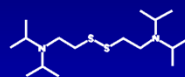
SPME of 5 Agent Mix



Quantitative

- VX
- Sulfur Mustard (HD)
- Sarin (GB)

VX Degradation SPME Method



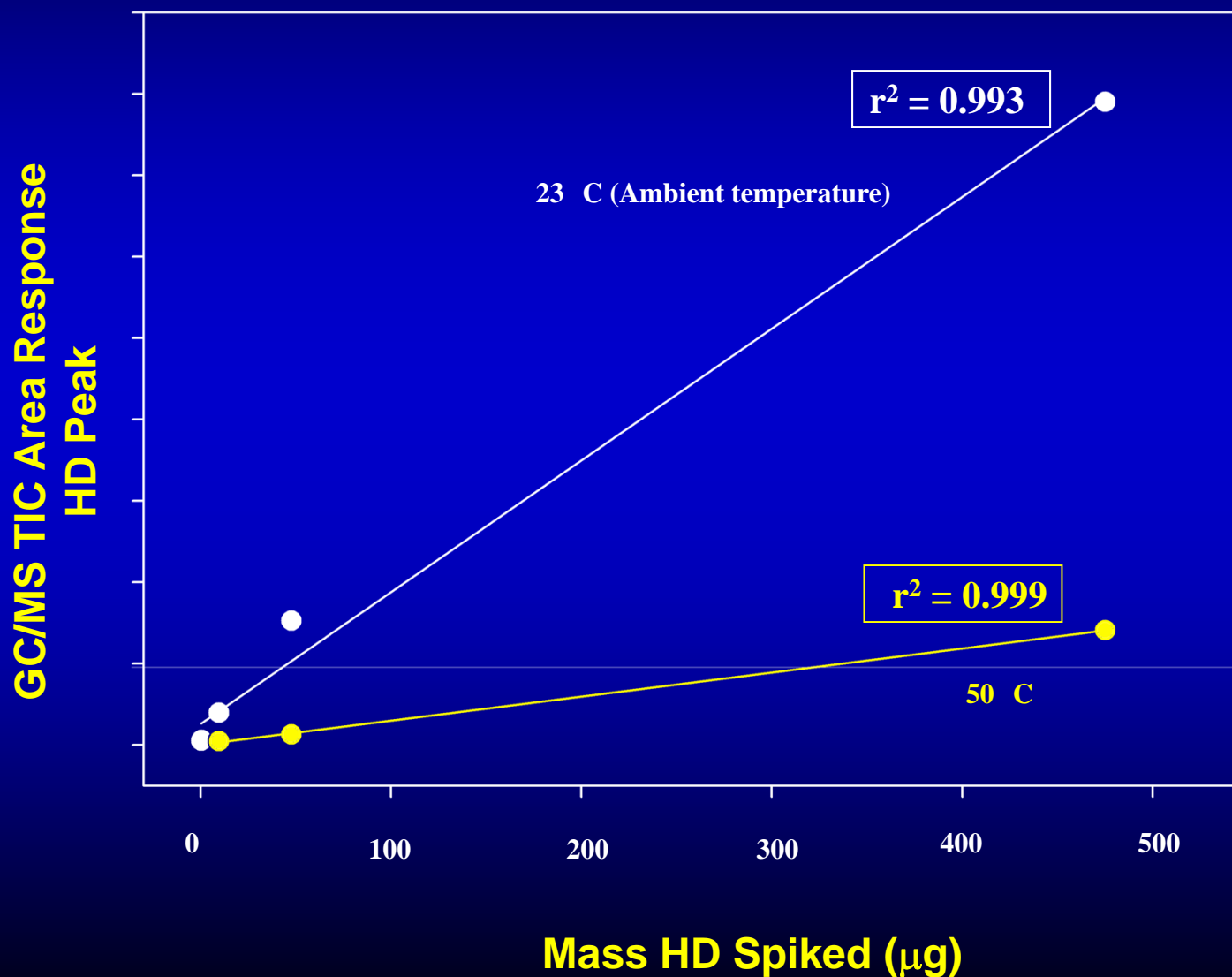
30 minute passive SPME sample
Mobile-laboratory field analysis

HD Soil Concentrations vs Peak Area

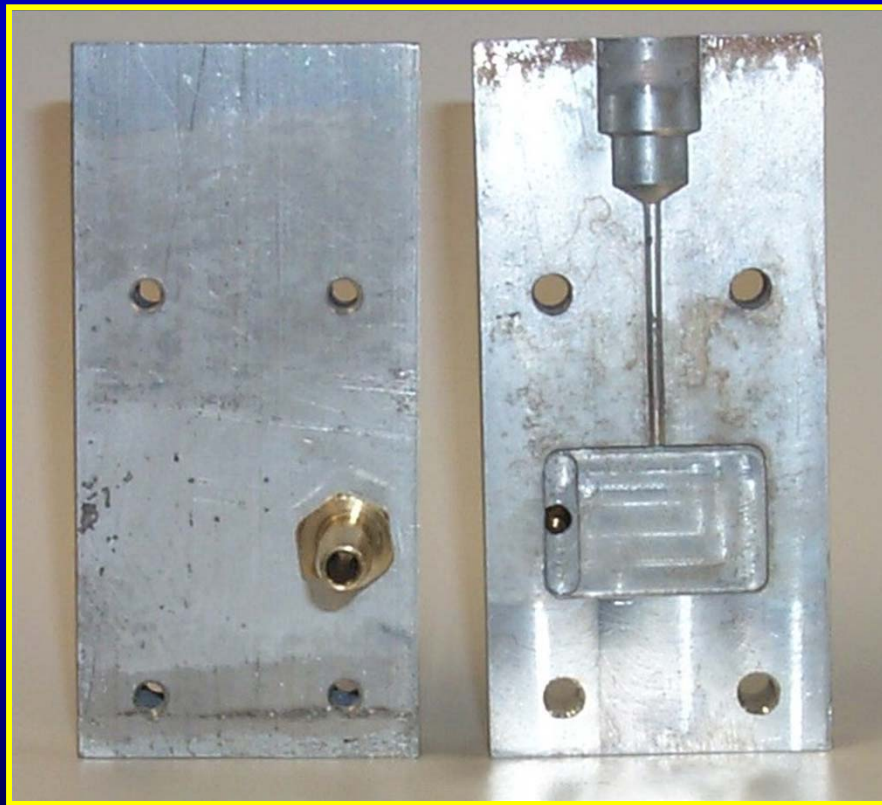
0.237 to 475 ppm_m Spiked to San Joaquin Soil

Headspace SPME

Ambient Temperature and 50 °C, 30 min Extraction



Portable Dynamic Air Sampler



Calculating the PDAS-SPME Airborne Concentration

$$C_g = n \ln [(b + \delta)/b] / 2\pi D_g L t \quad (1)$$

$$\delta = 9.52b / Re^{0.62} Sc^{0.38} \quad (2)$$

$$Re = 2u/bv \quad (3)$$

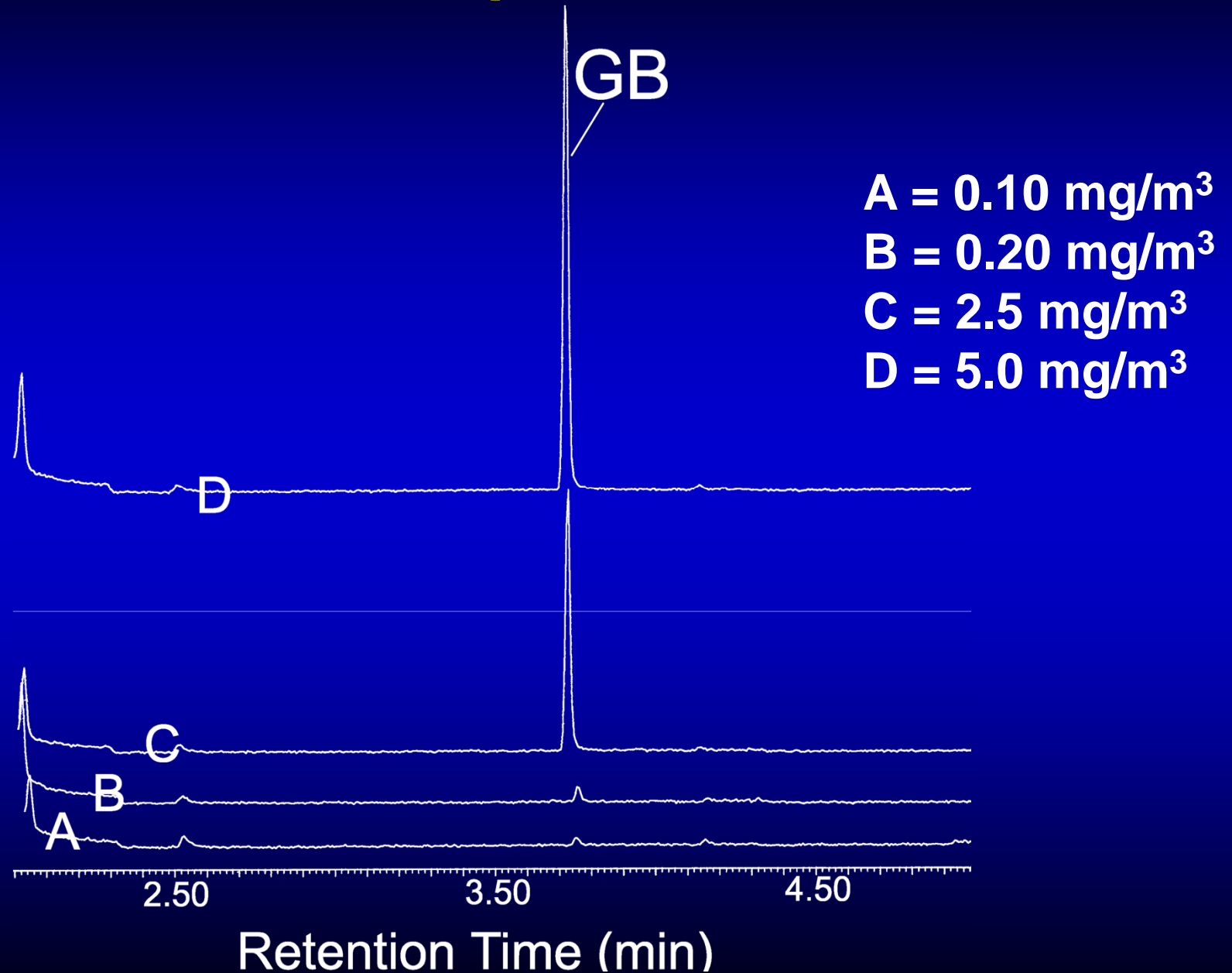
$$Sc = v/D_g \quad (4)$$

Koziel *et al.* Anal. Chem. (2000) 5178

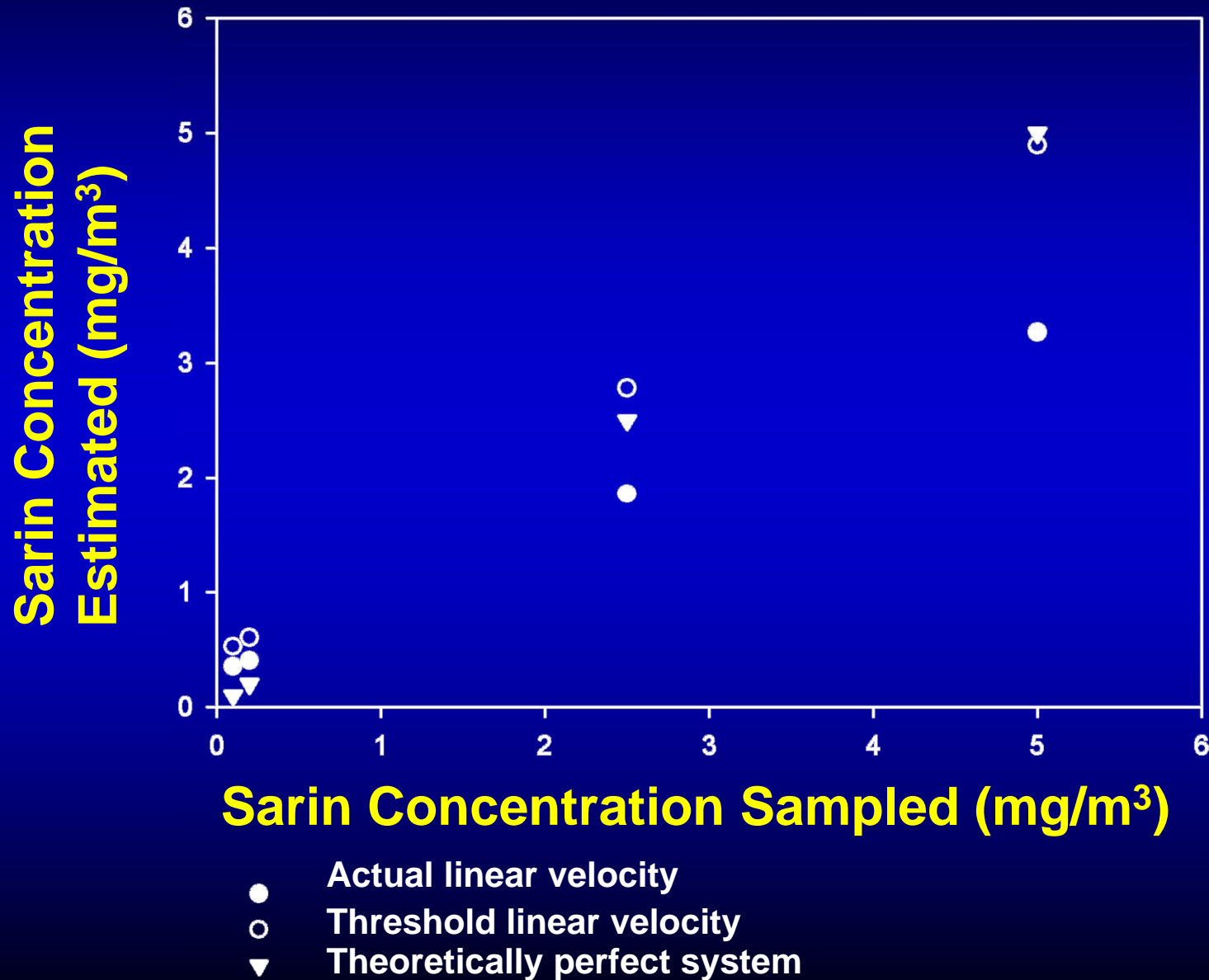
PDAS-SPME System



Dynamic SPME Samples of Airborne Sarin



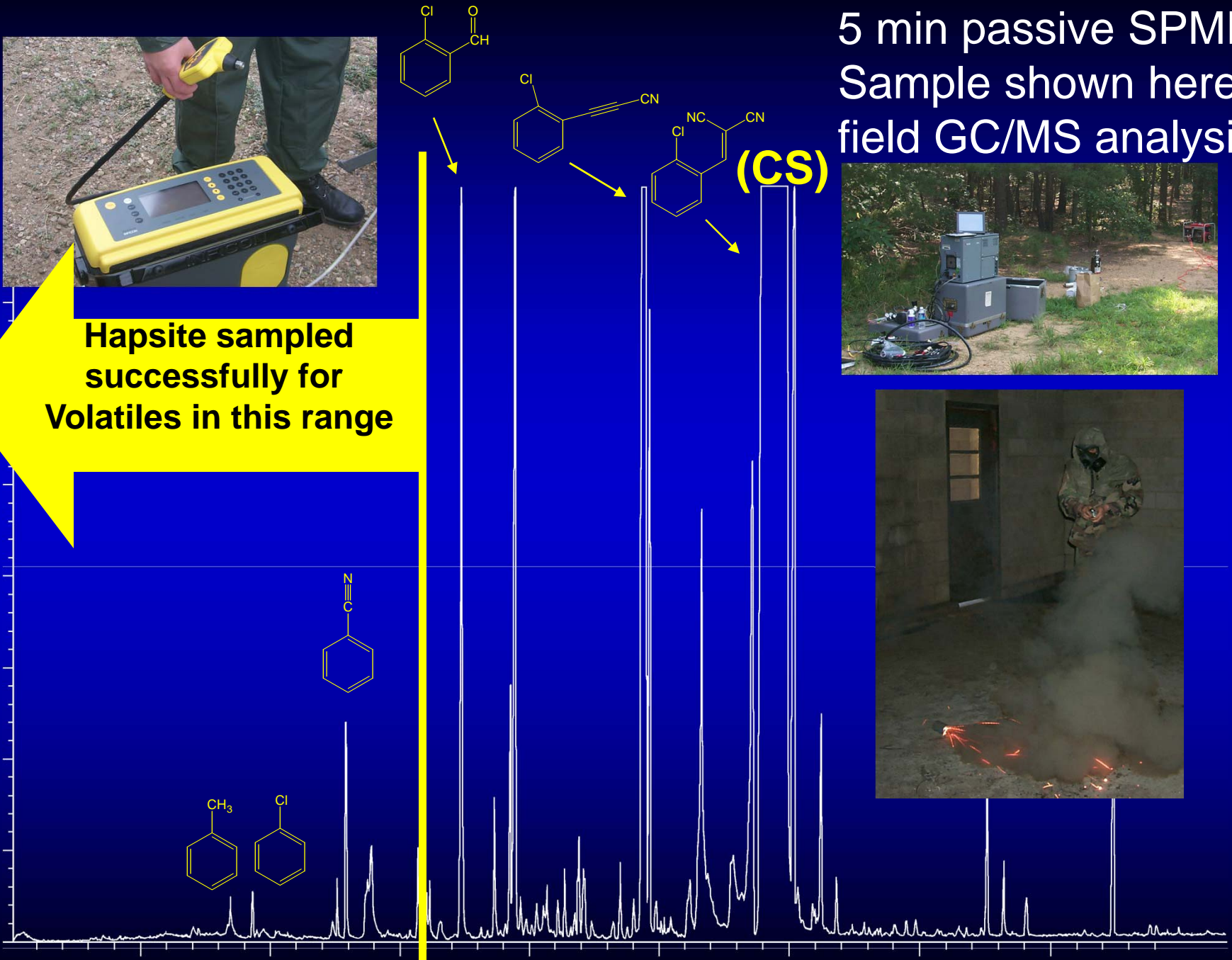
Comparison of Dynamic SPME Sample Concentration Estimates



5 min passive SPME
Sample shown here,
field GC/MS analysis



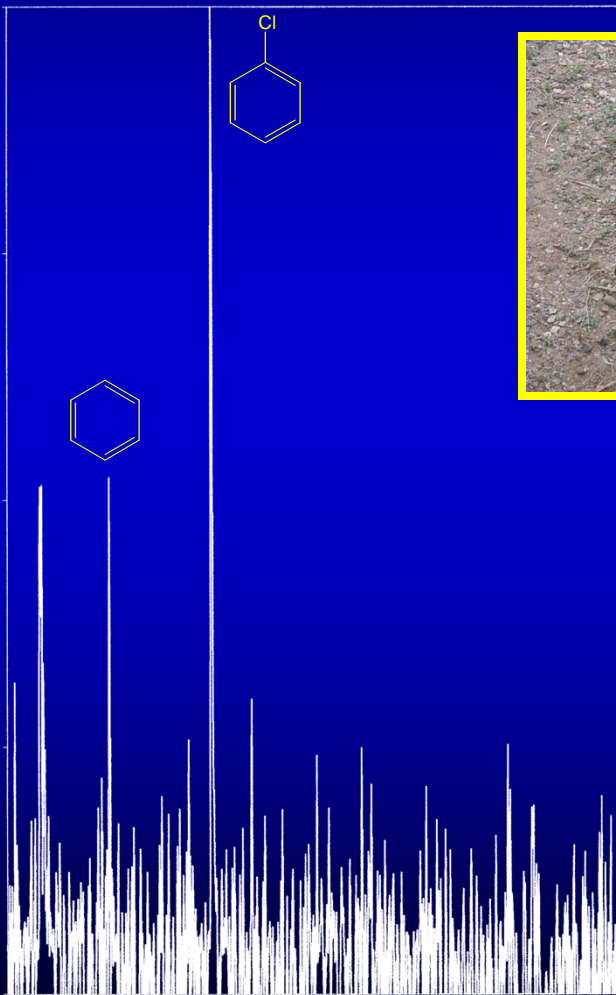
Hapsite sampled
successfully for
Volatiles in this range



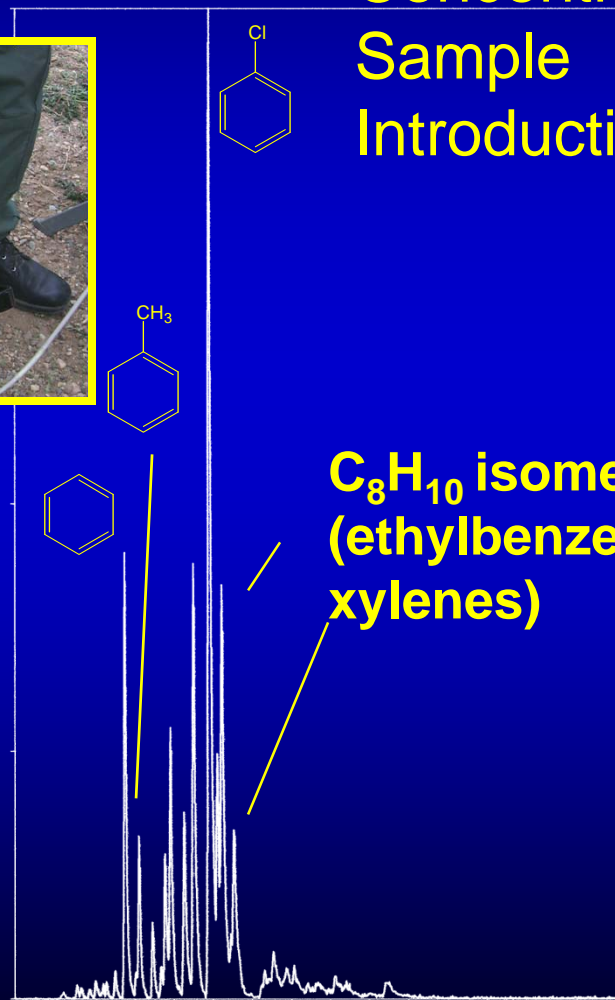
Same atmosphere as previous slide

only light compounds observed

Gas Loop
Sample Introduction



Tenax
Concentrator
Sample
Introduction

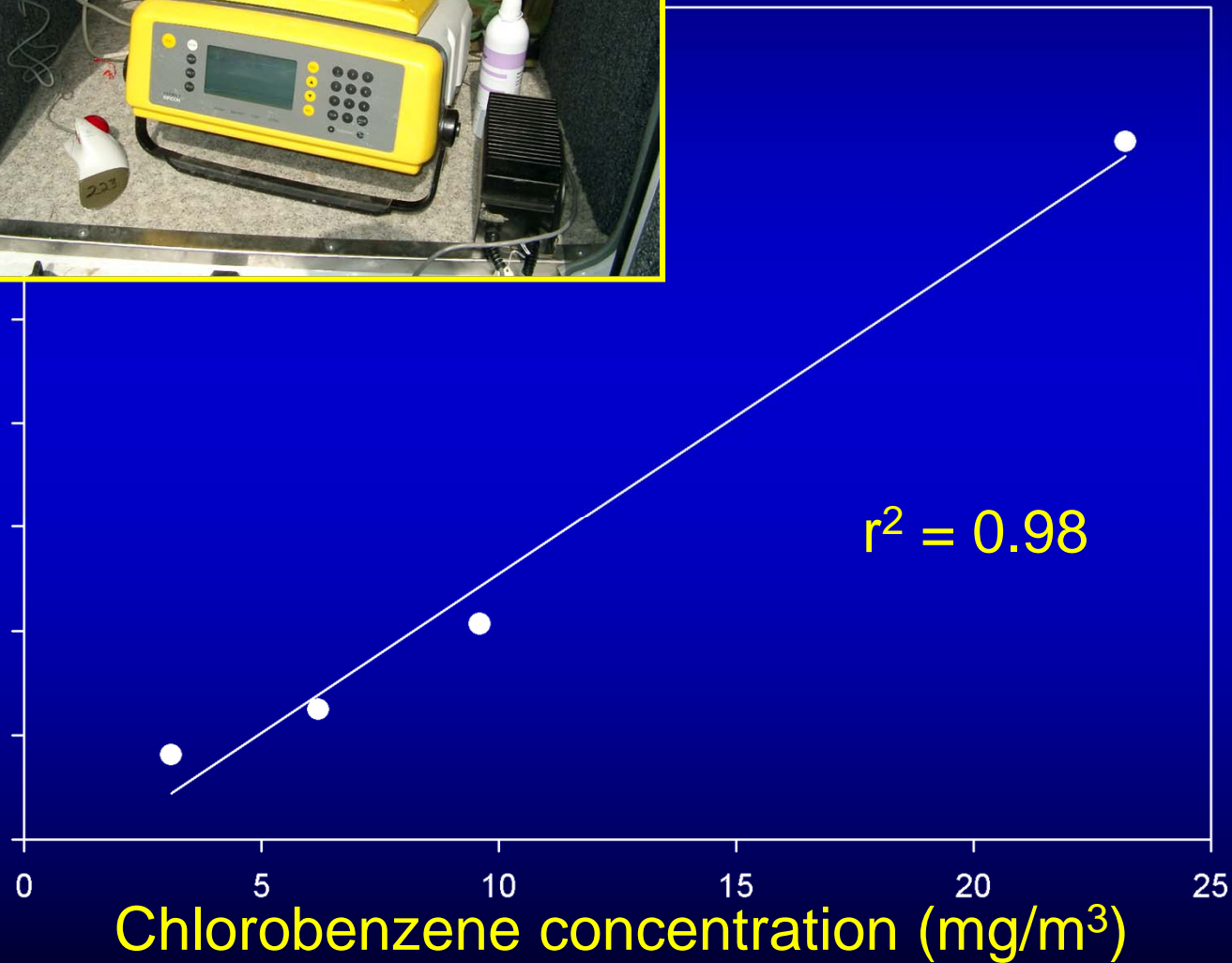


C₈H₁₀ isomers
(ethylbenzene,
xylenes)

Hapsite 112 m/z ion current
GC/MS peak area



Calibration of Hapsite with Dynamically Generated Chlorobenzene Gas Phase Standard



Current and Future Research

- Irradiated Mail: AFRRRI, JHU – APL, NIST
- Detection of CWAs and T₂-Toxin in water: SBCCOM, DSO National Laboratory Singapore
- Detection of TICs in Water: US Army Health Research Center
- Development of Field CWA Detection Methods for SPME-GC/MS and Hapsite: MARCORPSYSCOM, CBRIF, DRDC-Suffield
- Low Thermal Mass Columns: FBI Forensics Research Laboratory
- Hapsite-SPME Injection Port: Inficon

Training for GC/MS Operators



SPME Equipment

- **GC/MS**
- **SPME injection port liner**
 - available from multiple vendors of GC/MS supplies
- **SPME fibers and holders**
 - Supelco
 - Field Forensics

Where have we been?

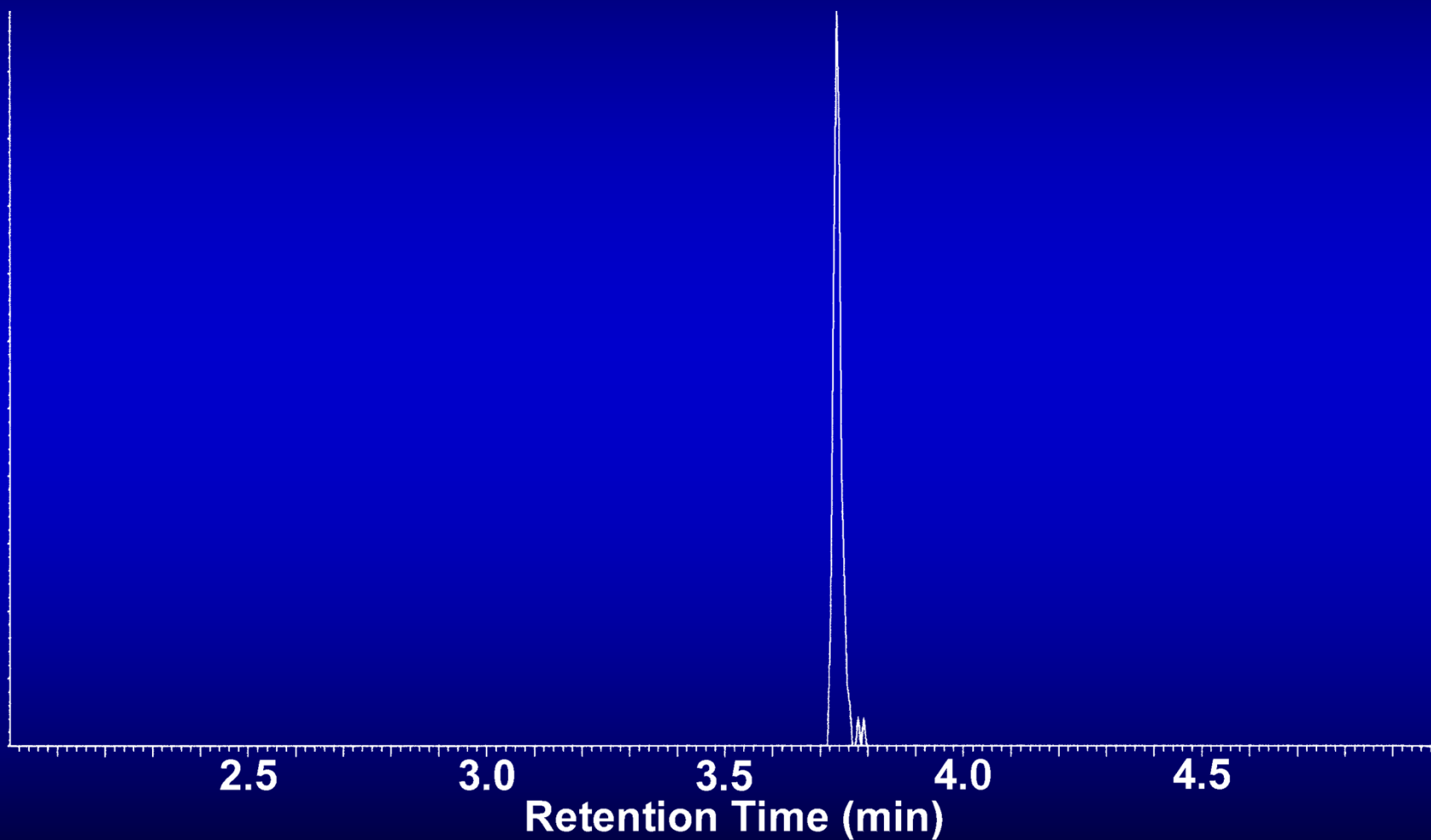
- Qualitative detection
- Quantitation
- Current Research
- Training for GC/MS Operators
- SPME Equipment

Questions?

- ghook@usuhs.mil
- 301-295-2642/9296/0391
- DSN: 295

30 s Dynamic SPME Sample of Sarin (0.10 mg/m³)

GC/MS 99 m/z Peak Area Response



Sulfur Mustard (HD)

