Modular Quantitative Air Sampling for Field Analysis using Person-Portable GC-MS

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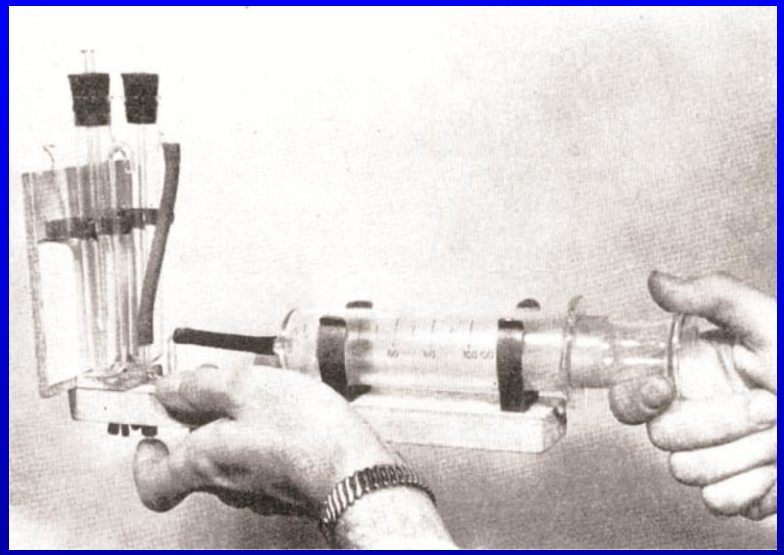
Real-Time Quantitative Detection: 1953



Gisclard *et al.* described the benefits from Systems that provide "on the spot" information:

(1) Can observe and quantify peak exposures (not possible with longer period TWA sampling)

(2) More samples in same amount of time

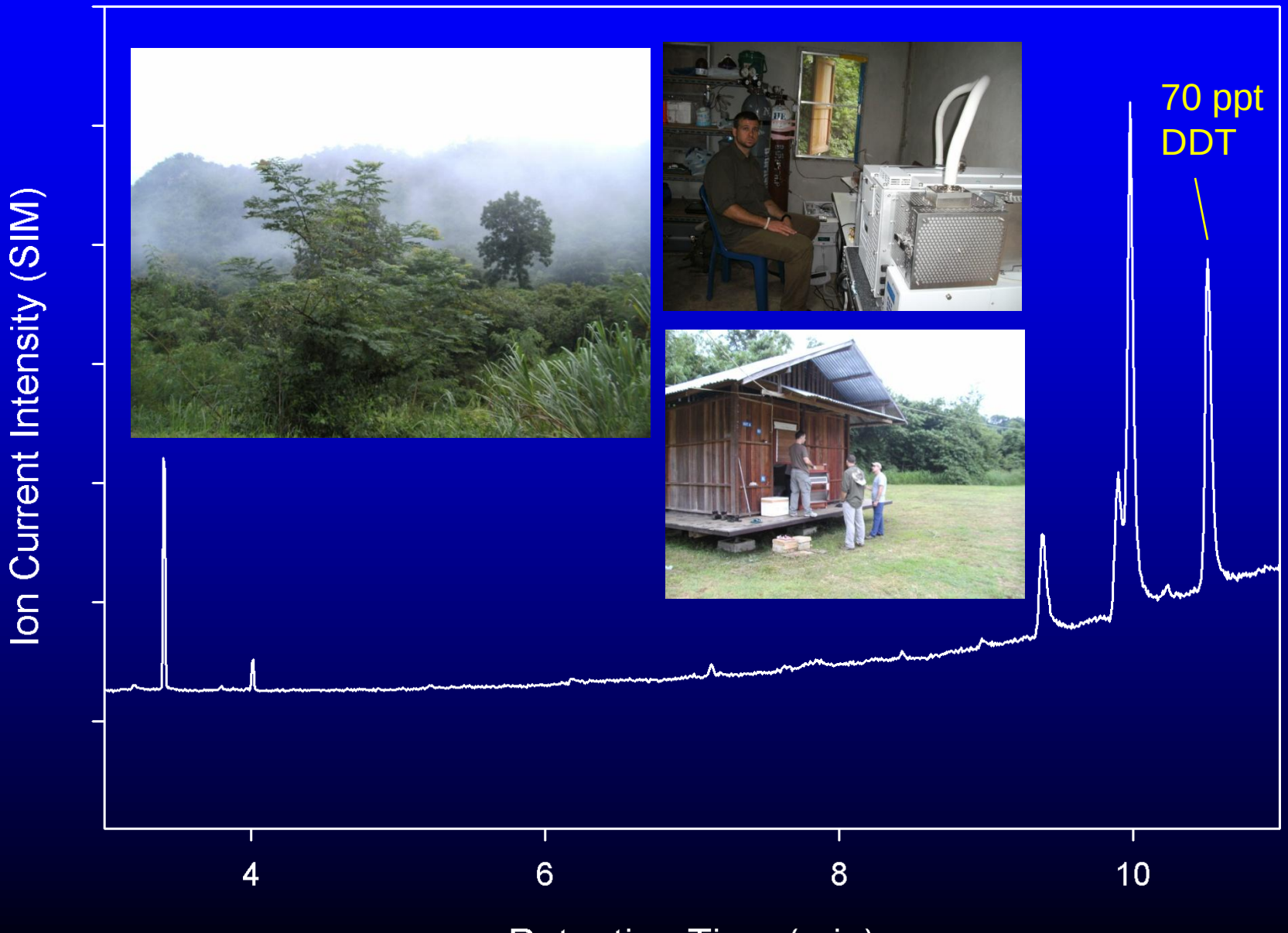
(3) Information is obtained where lab facilities Are not readily accessible

(4) "A minimum of equipment is required"

From Gisclard *et al.*: A Simple Device for Air Analysis. *AIHA Quarterly, 14(1):*23-25 (1953)

Near Real-Time Quantitative Detection: 2010

DDT Vapor in Test Huts (Thailand)



Real-Time Detection Hierarchy

Level 4

Level 3

Level 2

Level 1

Transportable Detection Systems

Man-portable Detection Systems

Small, Handheld Instrumental Detectors

Small, Handheld Simple Detectors

Complexity, Cost, Training Level

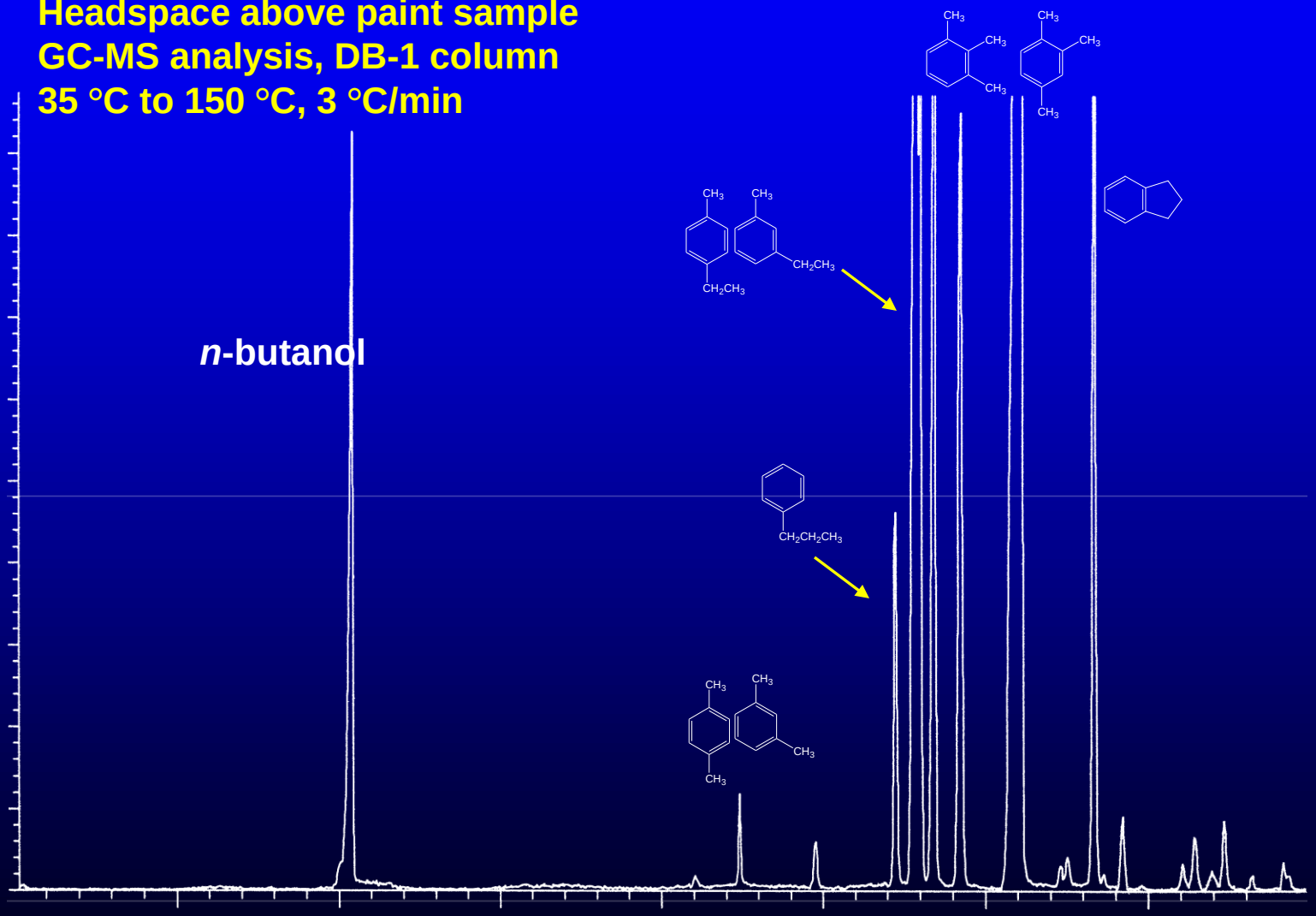
USS NIMITZ (CVN 68)

Hazard Recognition –screening by SPME/GC-MS

Formula 150 paint – incomplete MSDS information

Screening by Headspace SPME for Identification of Solvents in Formula 150 Paint

SPME Sampling
Headspace above paint sample
GC-MS analysis, DB-1 column
35 °C to 150 °C, 3 °C/min

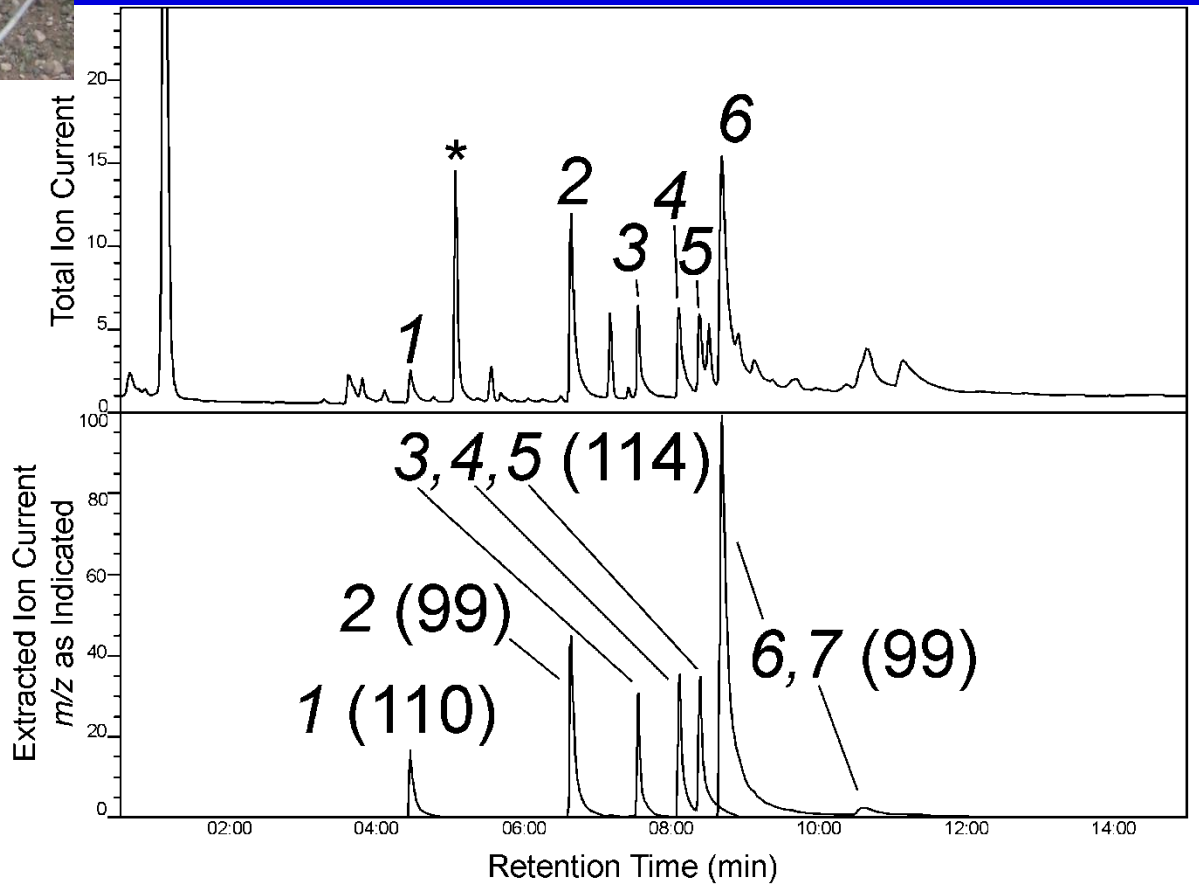


1st Generation Person-Portable GC-MS

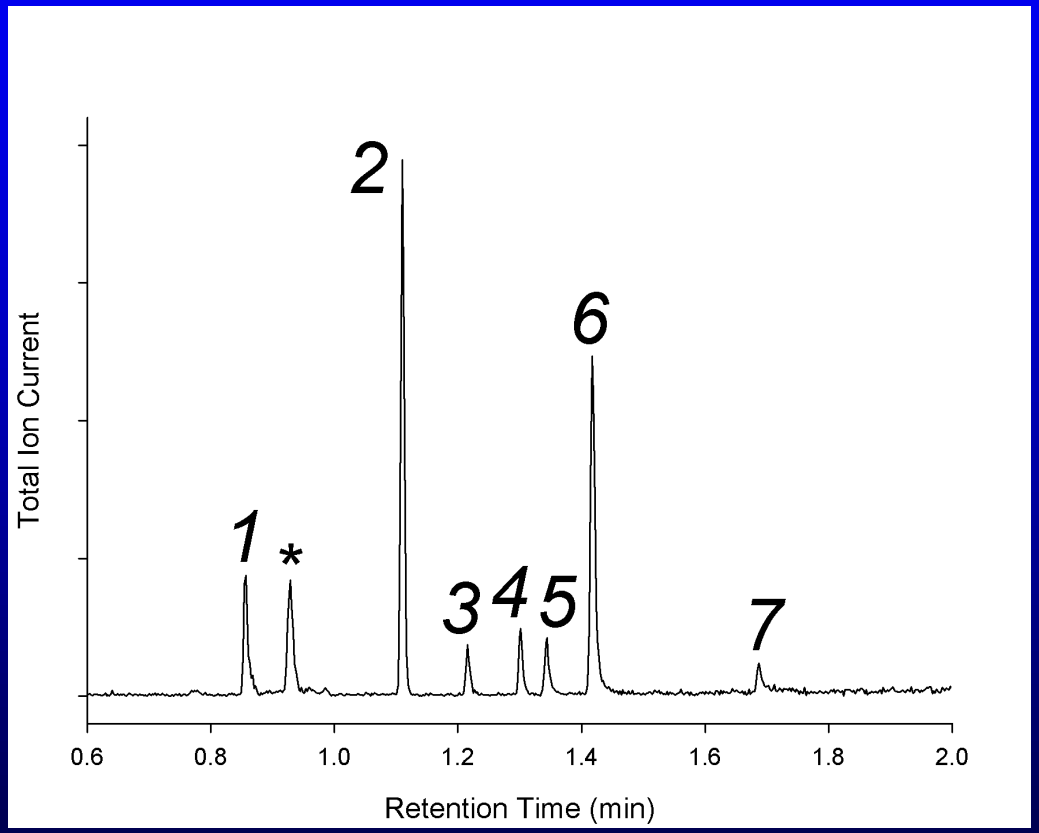
Chemical (NEG) high vacuum pump GC-MS
Membrane interface impairs chromatography

SPME Sample, Warmup on Shore Power

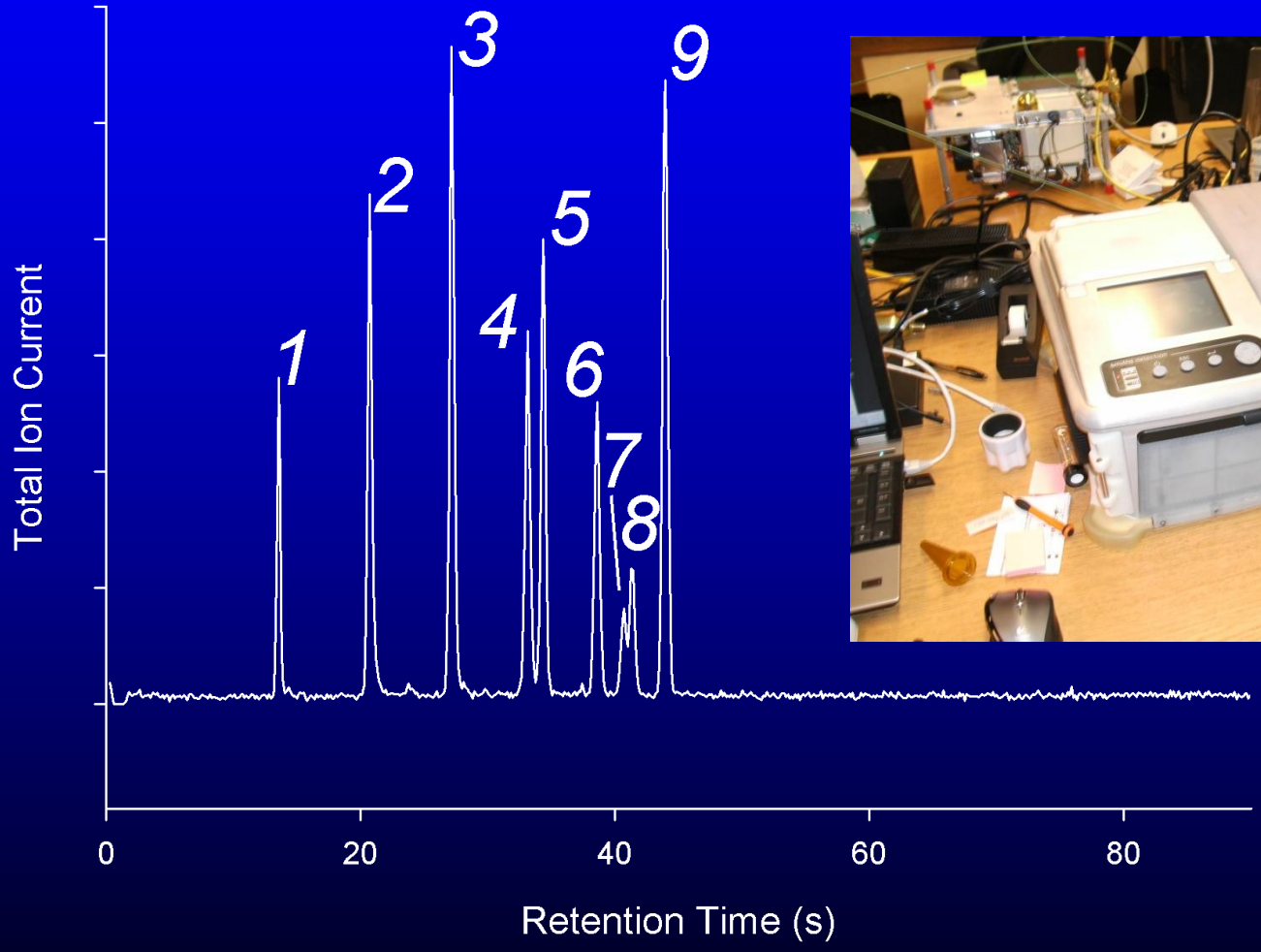
Trimethyl phosphate (1)
Through
Tributyl phosphate (7)



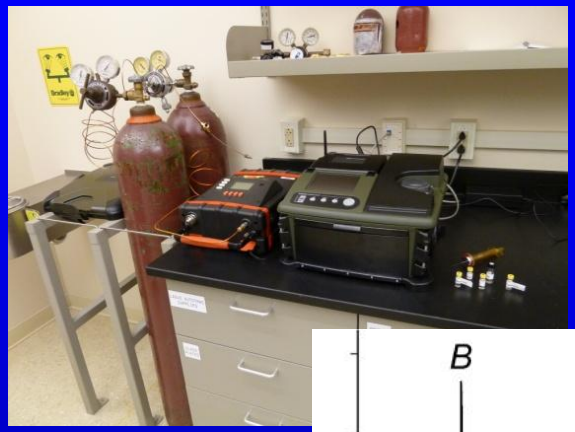
2nd Generation Person-Portable GC-MS

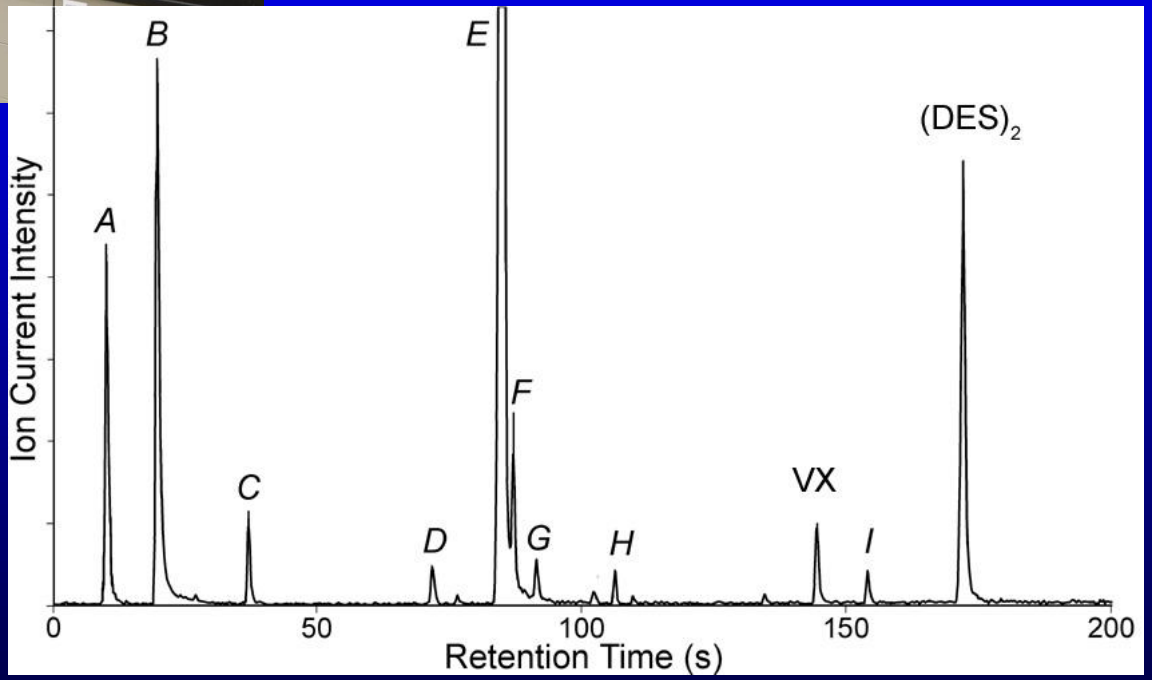


Light Solventrs (Acetone Through Dimethylcyclohexanes) in a Person-Portable Package: SPME Sampling

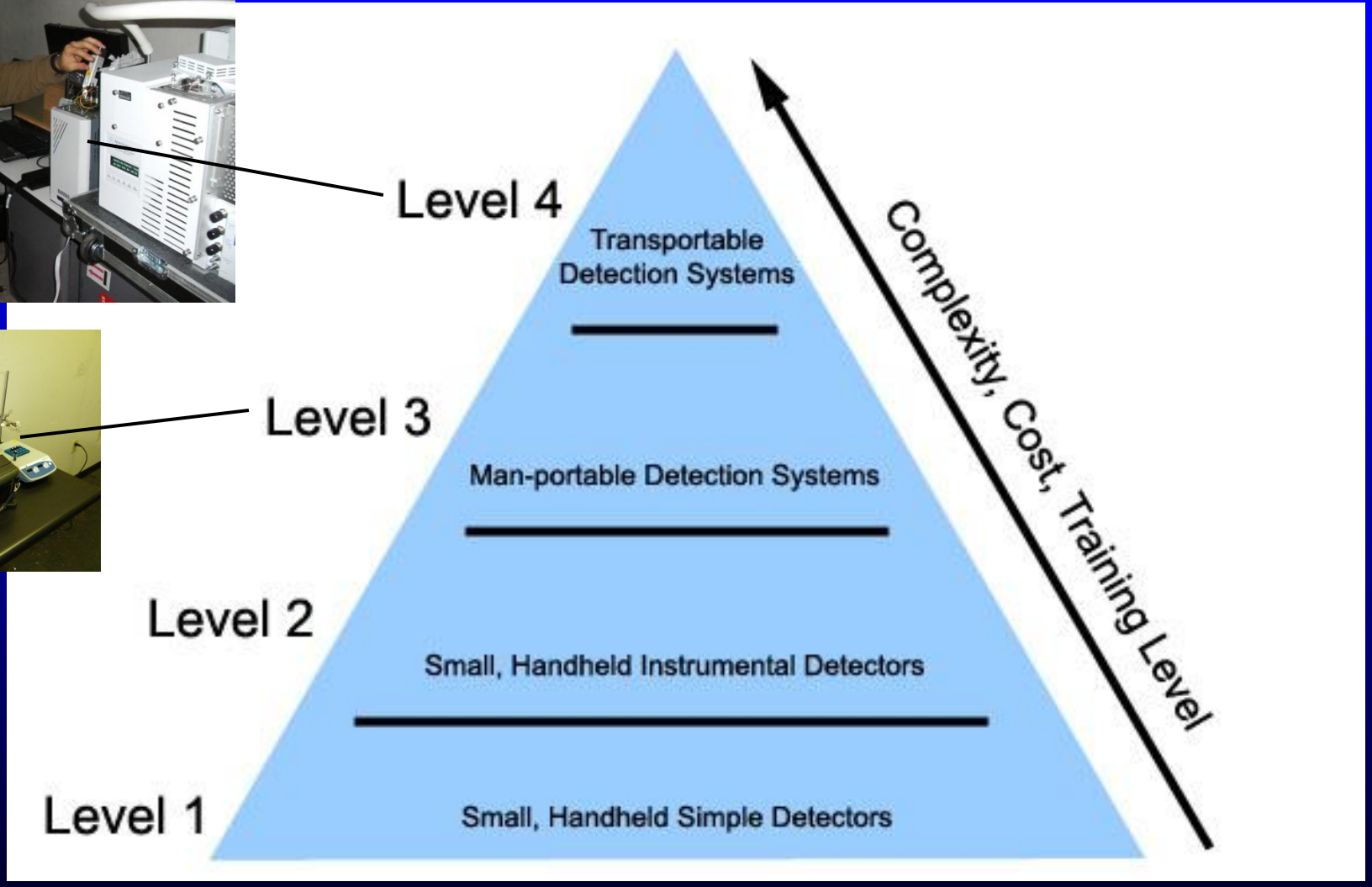


Degraded VX SPME Sample





Quantitative Thermal Desorption in the Field



Quantitative TD Capabilities Needed

Simple modular design desired: **no valves (maintains person-portability)**

Challenge is to match the low GC column flow with thermal desorption stream

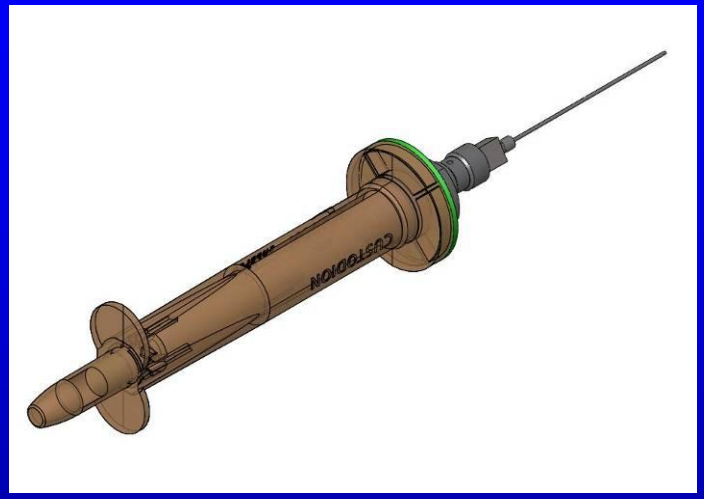
Needle trap (sorbent-packed) proposed as (1) direct sample device, and (2) as a concentrator for larger (89 mm) TD tube when greatest sensitivity is required

Needle Trap

A convenient sample injection method

Approximate mass and dimensions

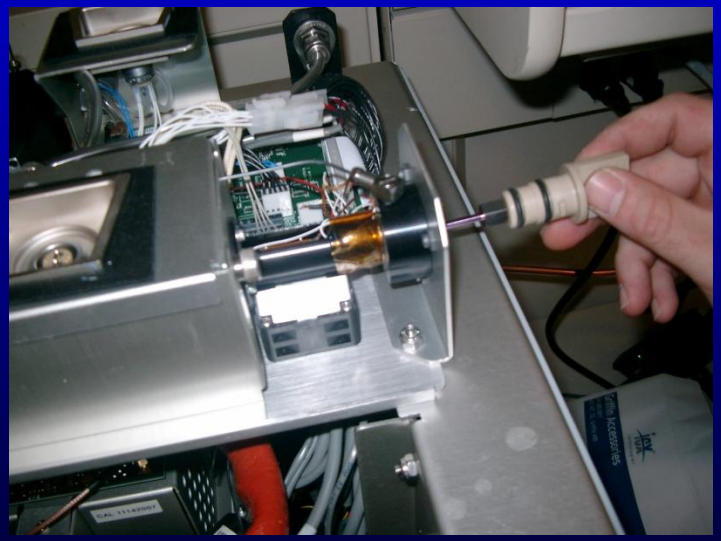
- 50 g
- 1.5” OD x 9” length (tip of needle to end of handle)



No power required for sampling

Very small volume of sorbent material allows exhaustive sampling, or can serve as preconcentrator for desorption of large TD tube

Existing TD Approaches

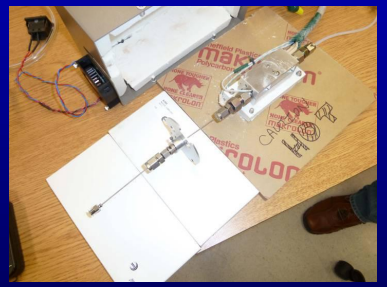


Add-on TD

Integrated TD

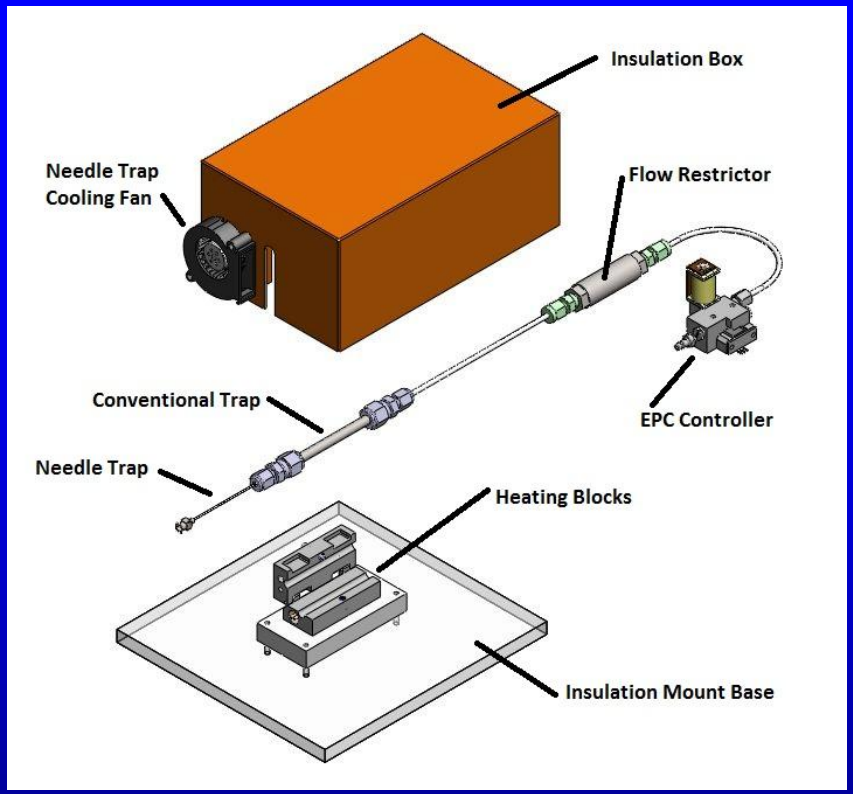
Person-Portable GC-MS

Integrated TD, external power required to warm up for analysis of large TD sorbent tube

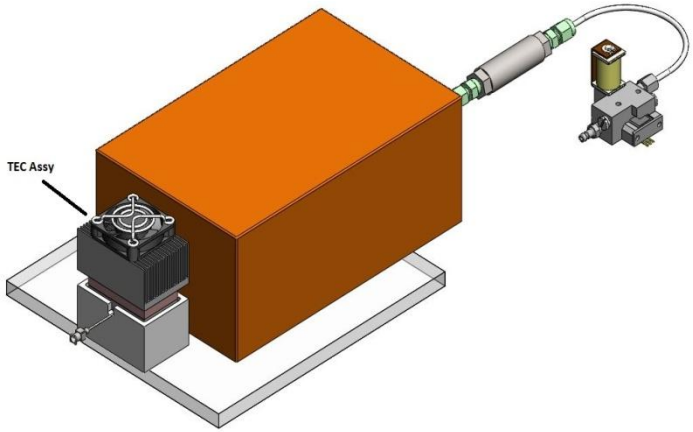


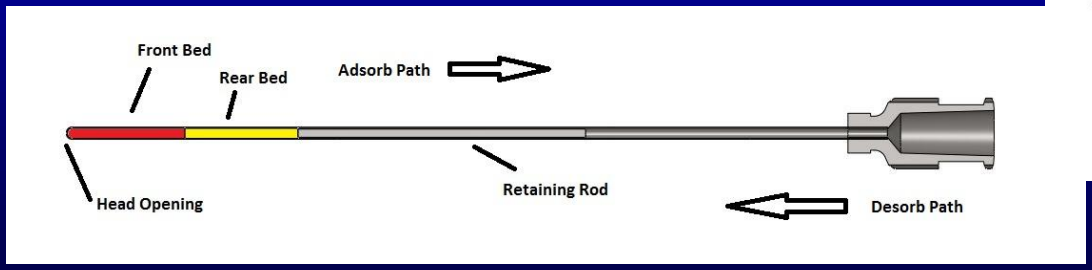
Modular TD desired without the need for external power, even for analysis of large TD sorbent tube

Prototype Needle Trap Platform

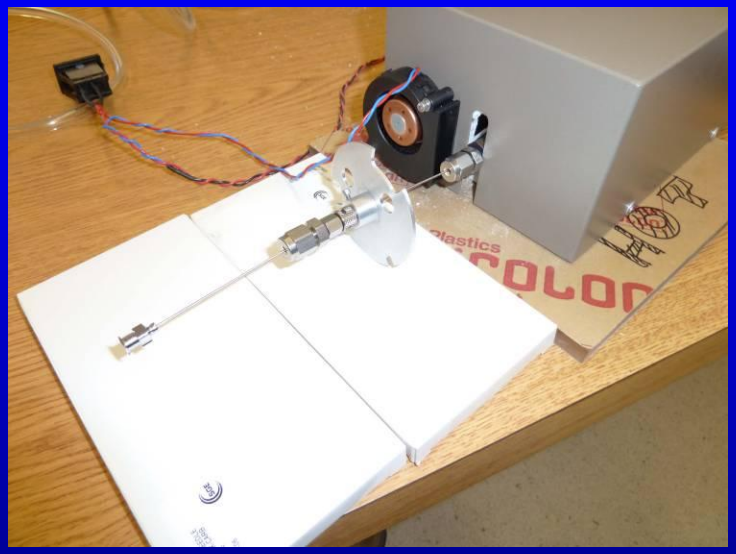


- **Heating Block Specifications**
  - Maintain 300C temperature
  - Provide a flow rate of 1-30ml/min through the 2nd Stage Trap
  - Keep Needle trap relatively cool during desorption phase
  - 1st Stage Trap heating time (from ambient to within 5°C of 300C): 47 secs
  - Temperature of needle trap at front of packing material when inserted into 2nd stage trap: 60C; Midway through": 37C; End: 28C.





Thermal Desorption of 6 Analytes from Tenax (89 mm Tube) to Needle Trap



Small battery-operated module to desorb 89 mm tube onto needle trap (He cartridge)

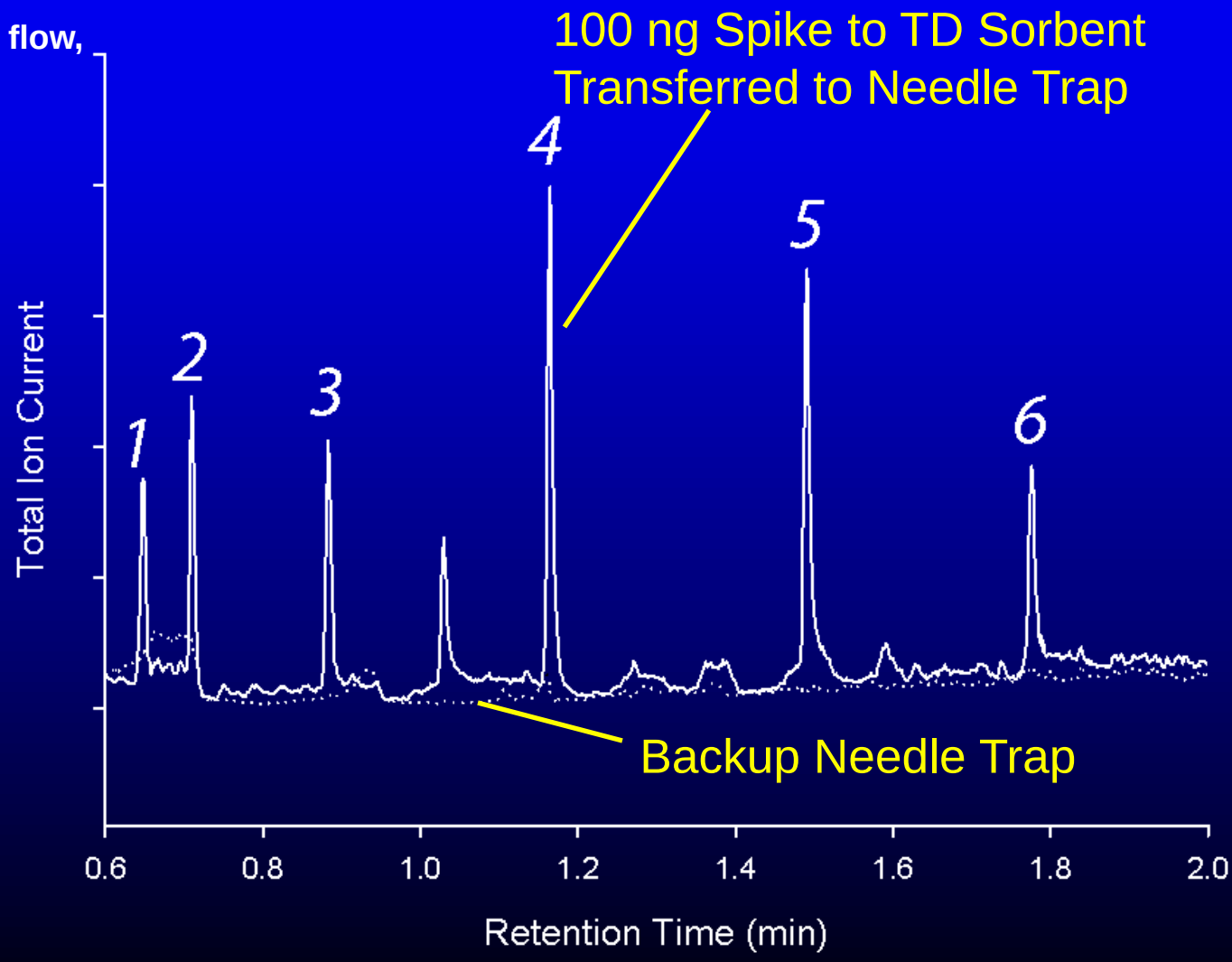
Needle trap directly interfaces into existing GC injector with He purge (supplied by automated column flow diversion into NT) that matches GC column flow

30 mL/min flow from crude desorption system onto needle trap cleared sorbent tube –no needle trap breakthrough (toluene = lightest analyte); NT at room temperature

Spike to TD Tube Transfer to Needle Trap for GC-TMS Analysis

1 = Toluene
2 = Perchloroethylene
3 = Trimethyl phosphate
4 = Triethyl phosphate
5 = Tripropyl phosphate
6 = Tributyl phosphate

35 mL/min TD desorb flow,
5 min 270 C



Extracted Ion Trace
12 ng Spike to TD Tube, Transfer to Needle Trap for GC-TMS Analysis

1 **= Toluene**
2 **= Perchloroethylene**
3 = Trimethyl phosphate
4 **= Triethyl phosphate**
5 **= Tripropyl phosphate**
6 **= Tributyl phosphate**

35 mL/min TD desorb flow,
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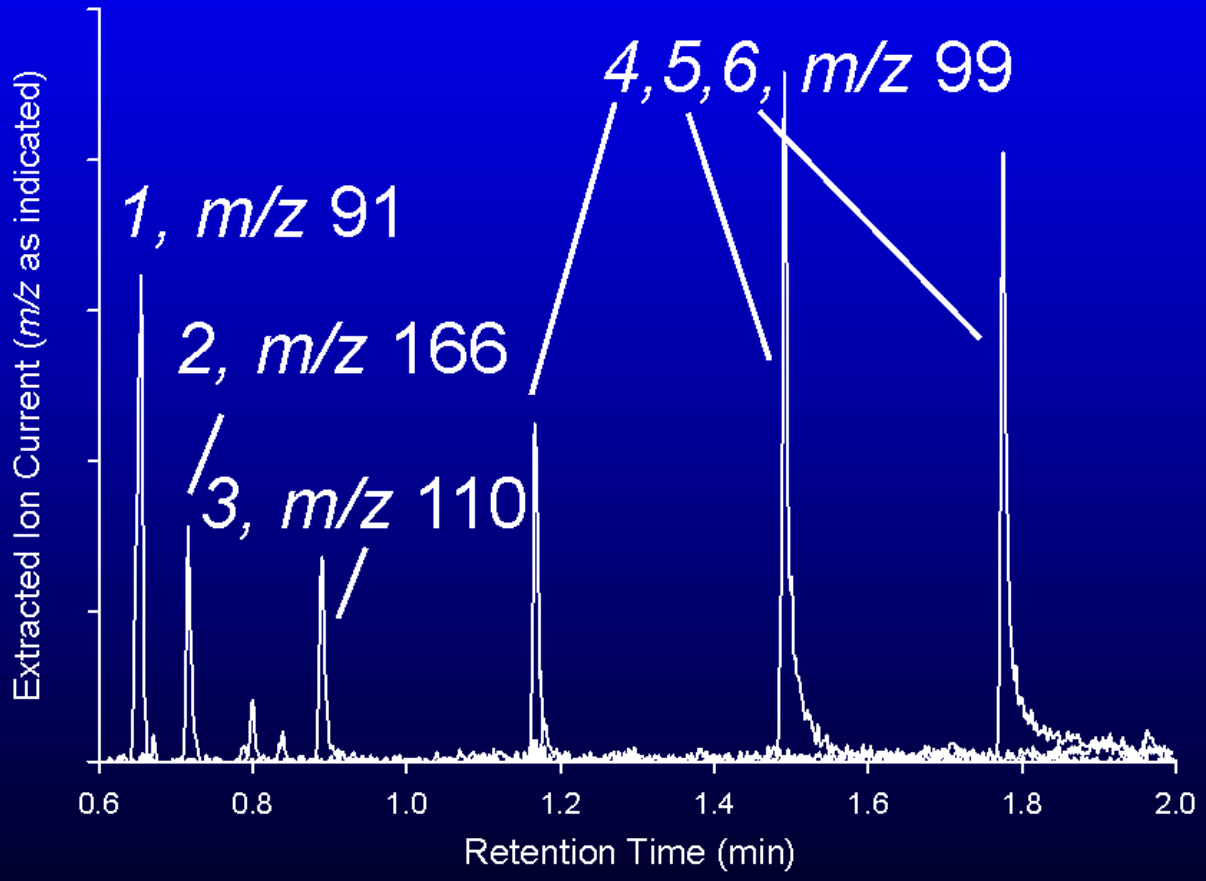
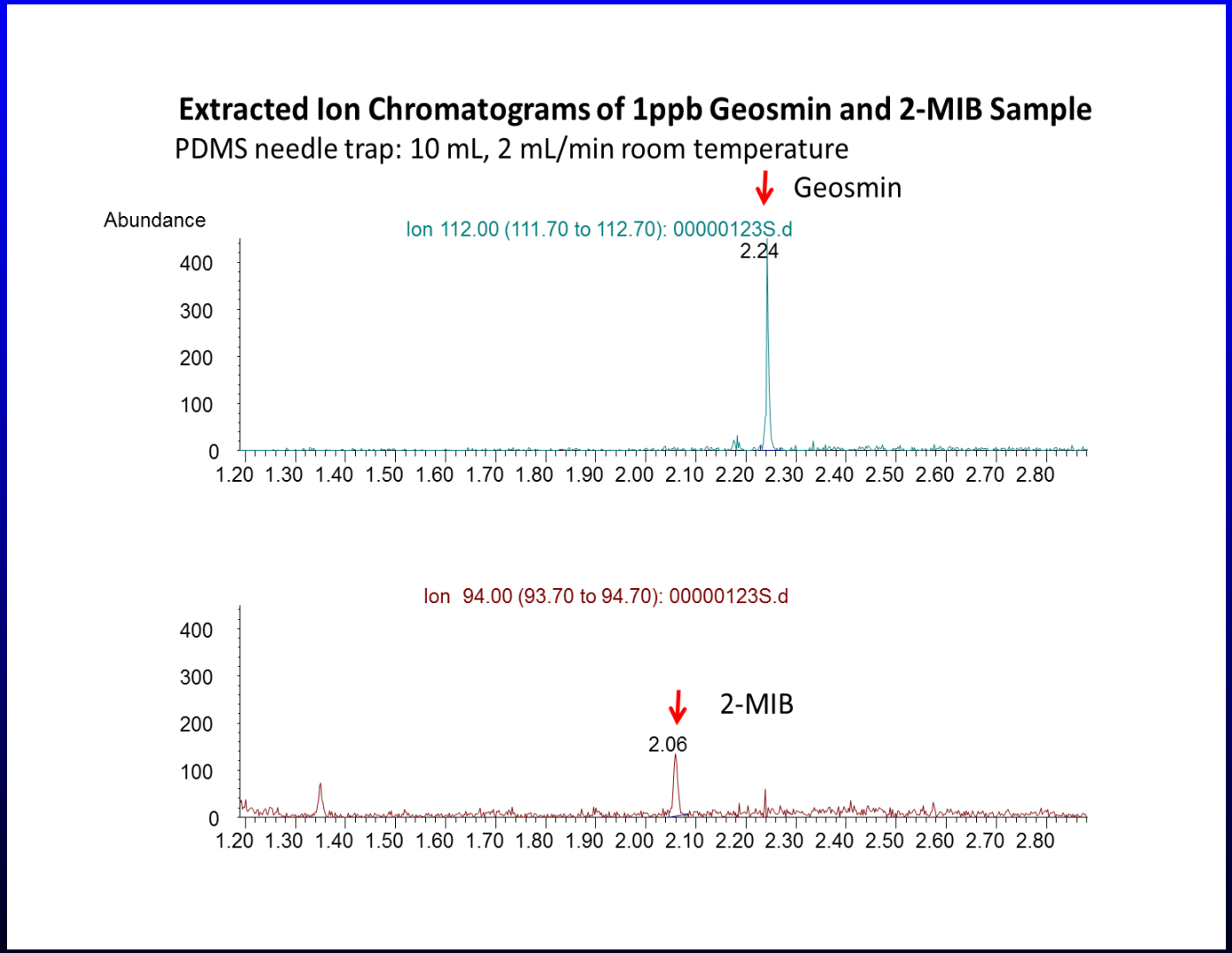


Extracted Ion Trace
1 ppb Geosmin and 2-Methylisoborneol from Water



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