Field-Portable GC/MS Characterization and Testing

Garth E. Patterson, John W. Grossenbacher, J. Mitchell Wells, Anthony J. Cochran, Caryn O'Hearn, Brent D. Rardin, Brent A. Knecht, Jason L. Springston, Mark A. Gregory, Dennis J. Barket, Jr.

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3000 Kent Avenue
West Lafayette, IN 47906

Field Portable Mass Spectrometry

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MIDDLE EAST

The Chicken Defense

U.S. troops will be using poultry to detect chemical attacks in Iraq

Socialstyrelsen

Aktuellt | Ämnen | Hälsa & Sjukvård

The Terrorist Attack with Sarin in Tokyo

Summary, experience and conclusions

Posted on Sat, Mar. 01, 2003

Operation Field Chicken shut down

BY RON HARRIS
St. Louis Post-Dispatch

Federal trial begins over explosive-sniffing dogs who bombed

Mon Jun 9, 2:40 PM ET

Add Offbeat - AFP to My Yahoo!

WASHINGTON (AFP) - A dog trainer went to court, to face federal US charges he sold the US government guard dogs that could not sniff out dynamite or other high explosives.

TOP NEWS | HOME | TOP NEWS > Latest crime and

Tuesday, September 7, 2004

NEWS

Updated September 28, 2001, 11:00 a.m. E

Future terrorist attacks worry people living near ammunition, chemical depots

- Latest News
- Business
- Government & Policy
- Science/Technology
- Career & Employment
- ACS News

Latest News

June 14, 2004
Volume 82, Number 24
p. 15

CHEMICAL WEAPONS

LAB TESTS CONFIRM SARIN IN IRAQI SHELL

Sophisticated analyses uphold tests fingering nerve agent in roadside bomb

Home Page | World | U.S. | Weather | Business at CNNMONEY | Sports at SI.com | Politics

Richard Reid pleads guilty

Faces minimum sentence of 60 years

Wednesday, January 22, 2003 Posted: 10:57 AM EST (1557 GMT)

- Oxford
- Jacksonville
- Calhoun County
- Clay County
- Cleburne County
- Randolph County
- Talladega County
- Legislature
- State

Chemical weapons concerns rising

By Richard Raeke
Star Chemical Weapons Correspondent
10-02-2001

CAMPUS | CITY & STATE | BUSINESS | NATION & WORLD | OPINION | SPORTS | ARTS | FEATURES | WEEKEND | CLASSIFIEDS

Officials may store VX in Newport

Disposal plant considers building tank farm to house byproduct

From AP reports

E-mail this story | Print this story

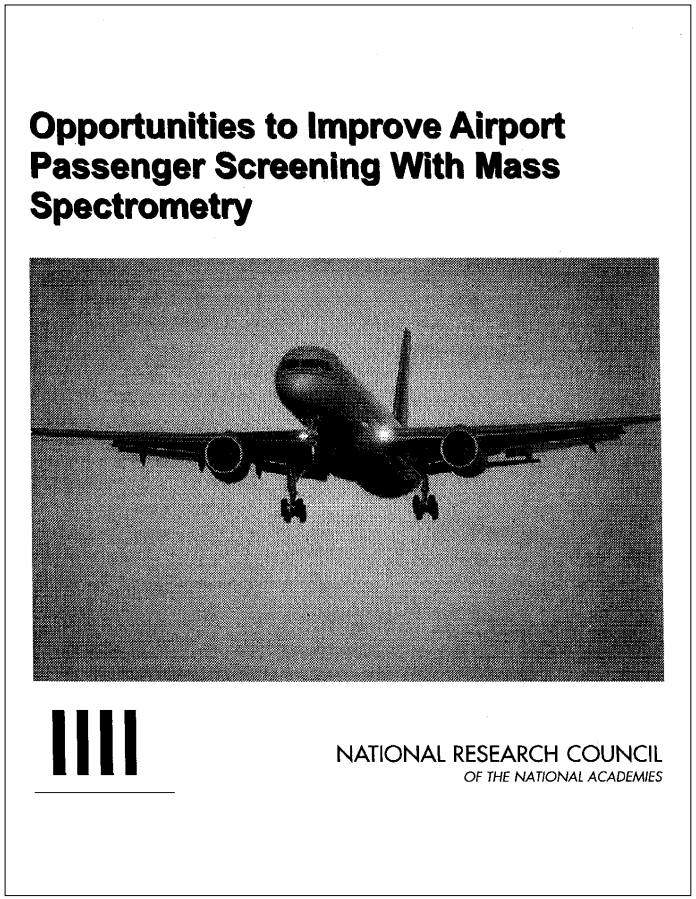
Published Thursday, July 3, 2003

HEADLINES

Tuesday, September 7

Frustration prompts schools to ban cell phones

Field Portable Mass Spectrometry



Committee on Assessment of Security Technologies for Transportation, National Research Council of the National Academies. The National Academies Press: Washington, D.C., 2004.

"To improve upon the trace detection systems currently employed in airports across the US, mass spectrometry (MS) is an obvious candidate to consider. It has become the gold standard for resolving high-consequence analyses ..."

But

"Mass spectrometers have historically been large, complex systems. ...commercially-available chemical analysis systems...are not designed for an environment as harsh as an airport or other transportation arena."

-or a battlefield, a depot perimeter, an office building HVAC system...

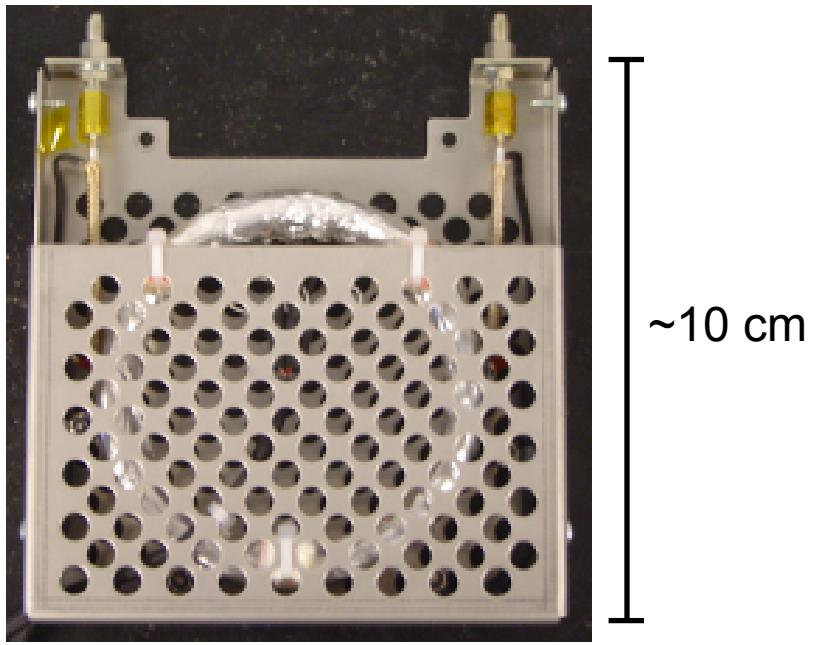
Griffin's Portable GC/MS

Cylindrical Ion Trap Mass Analyzer

- Easily miniaturized trap geometry
- MS/MS capability
- Lower voltage, higher pressure operation
- Internal electron ionization (EI)

Low Thermal Mass-GC

- Ramp rate ≥200˚ C/min
- Maximum Temp 350˚C
- Cooling time ~2 min
- Variety of phases
- 1 m – 30 m standard



Griffin's Portable GC/MS

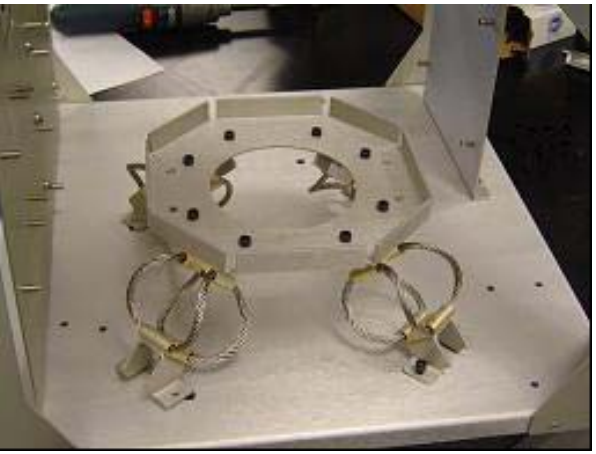
Minotaur 300

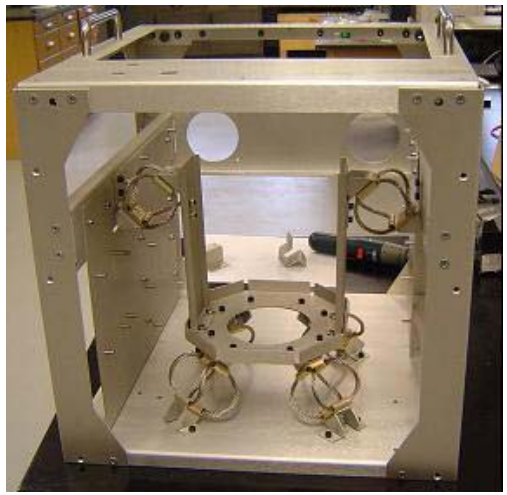
Minotaur 400

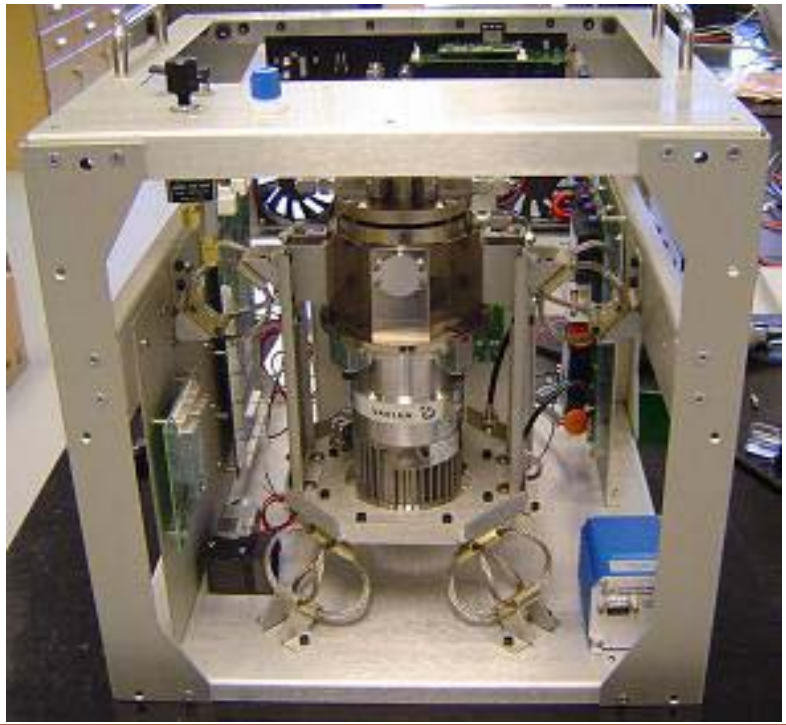
Instrument Specifications:

- MS/MS capable
- Picogram sensitivity (application dependent)
- Low thermal mass GC, SPME, MIMS, and direct SPME sample introduction available
- Multi-level software control
- Helium or ambient air bath gas
- Ruggedized and shock-mounted for outside-the-lab operation (Minotaur 400)
- Network communication compatible

Shock Mount platform



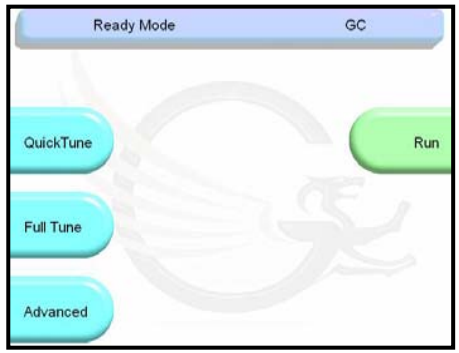




Wire-rope isolators are used to provide shock and vibration isolation of the most fragile components

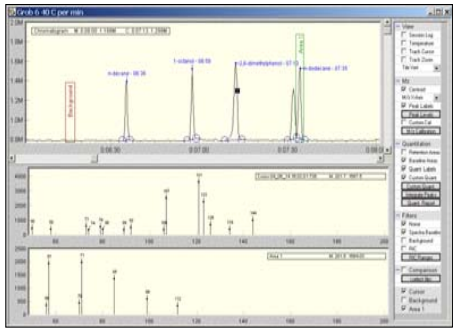
Layered Software Architecture

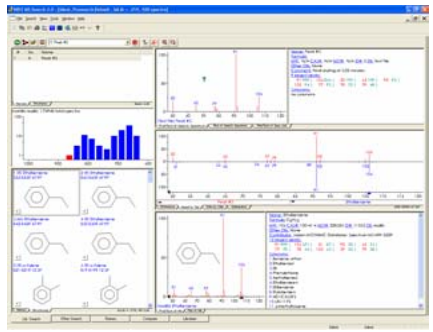
Level 1



simplified user interface

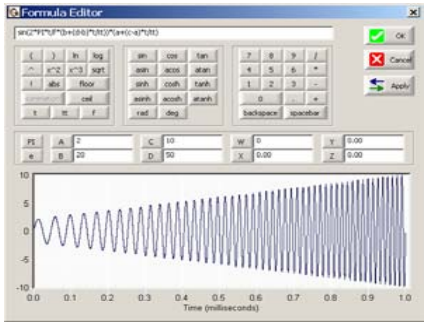
Level 2

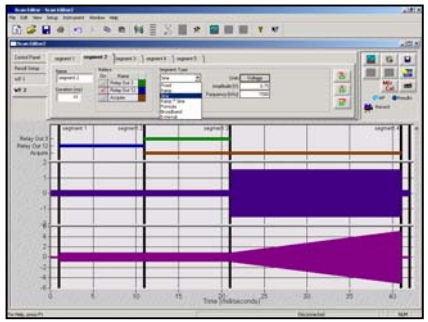


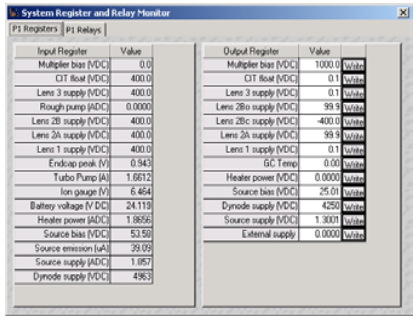


scientific user interface

Level 3



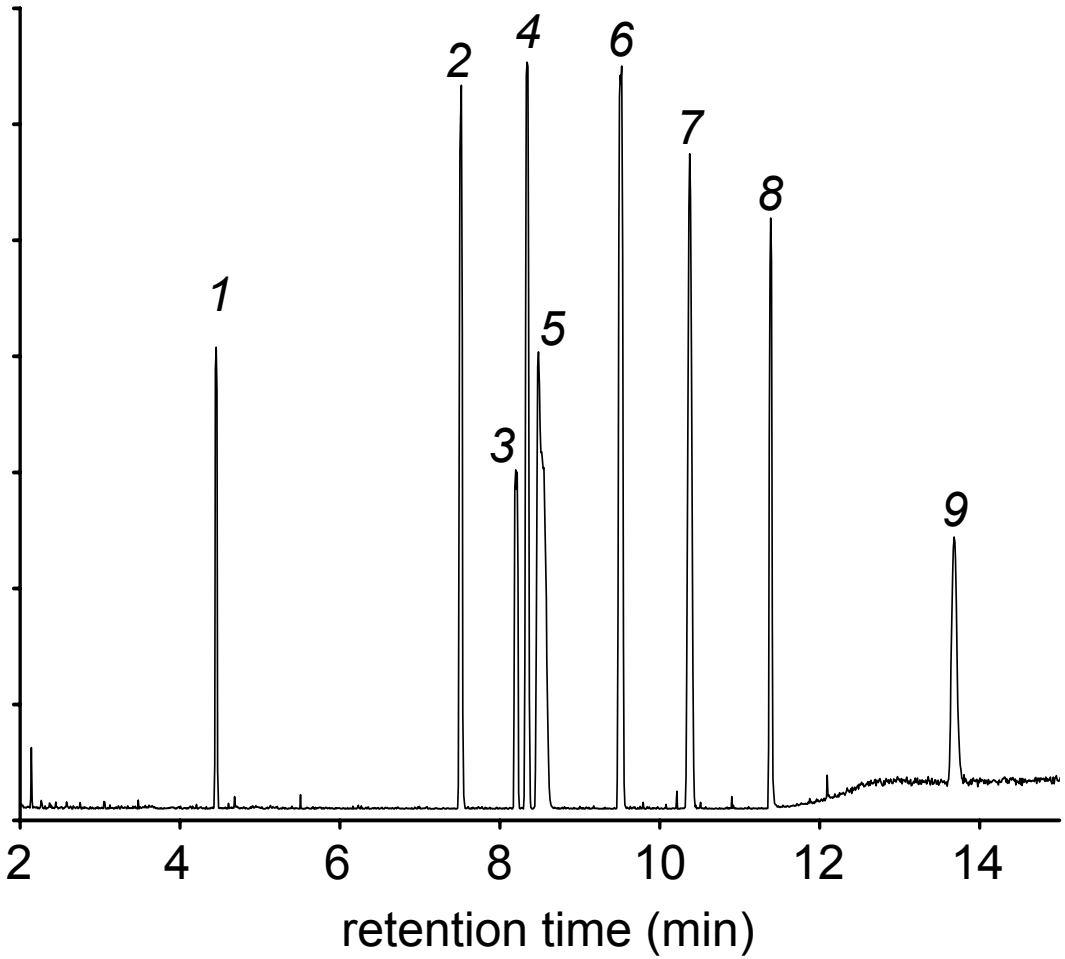




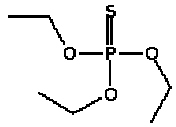
fundamental development tool and expert user interface

Analysis of CW agent simulants

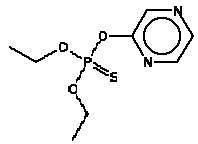
Separation of 9 organophosphorus pesticides
30 m x 0.25 mm Rtx-5MS column



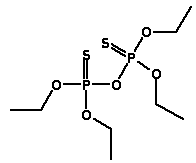
1) O,O,O-Triethyl Thiophosphate



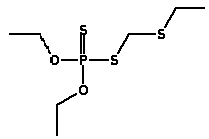
2) Thionazin



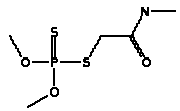
3) Sulfotep



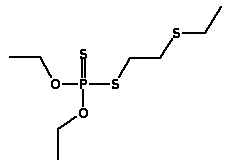
4) Phorate



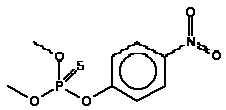
5) Dimethoate



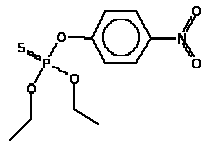
6) Disulfoton



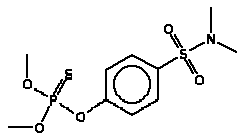
7) Methyl Parathion



8) Parathion

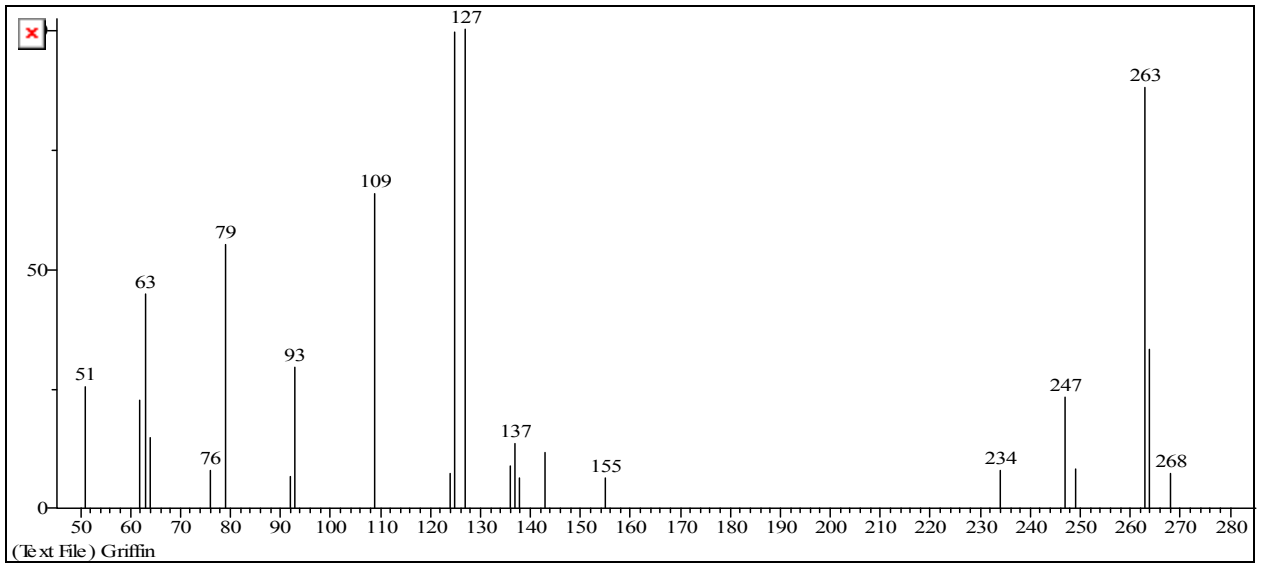


9) Famphur



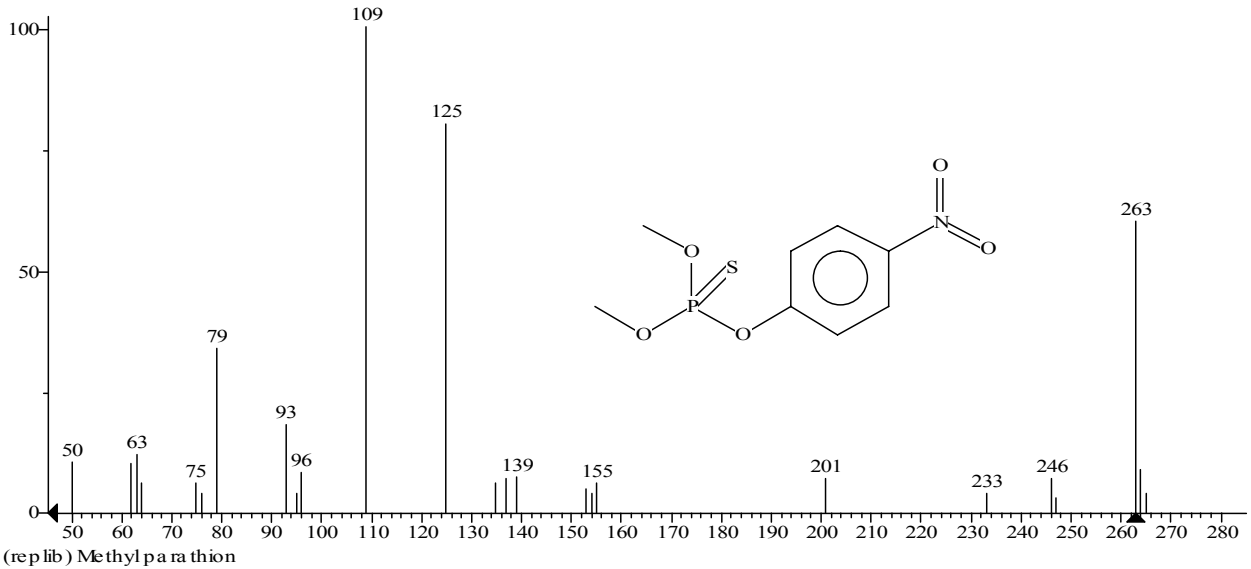
Analysis of CW agent simulants

MS of Peak #7



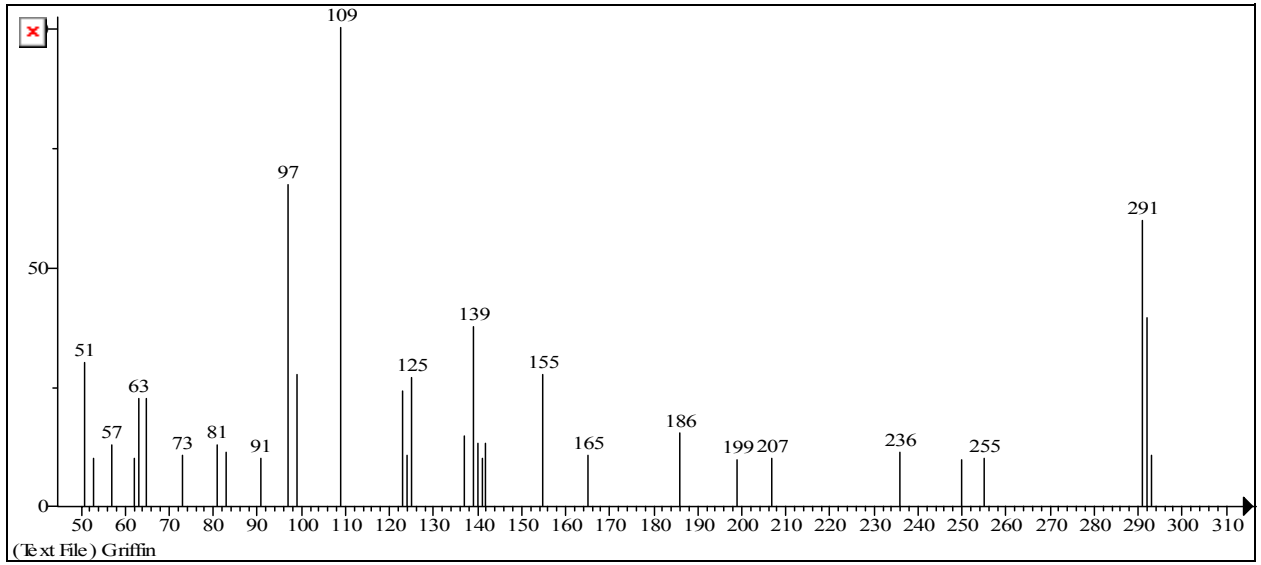
NIST MS of Methyl Parathion

Match Probability 88.5%



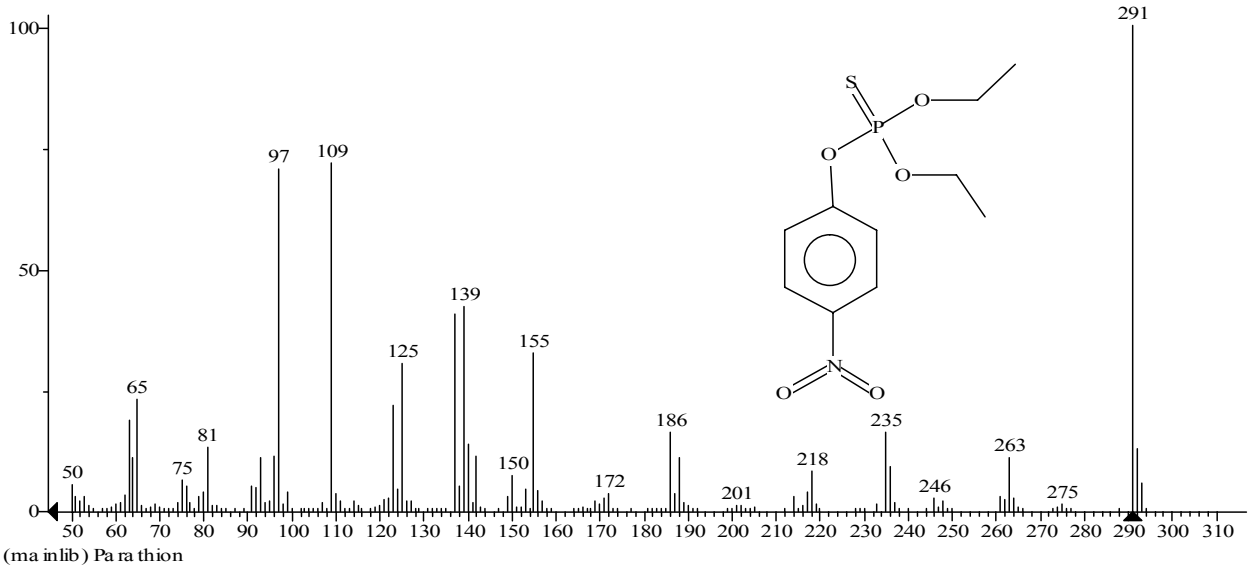
Analysis of CW agent simulants

MS of Peak #8



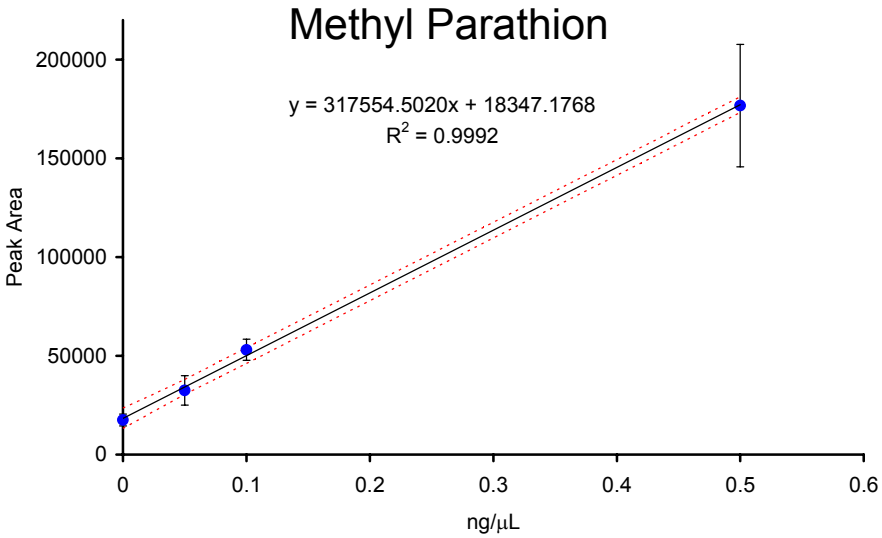
NIST MS of Parathion

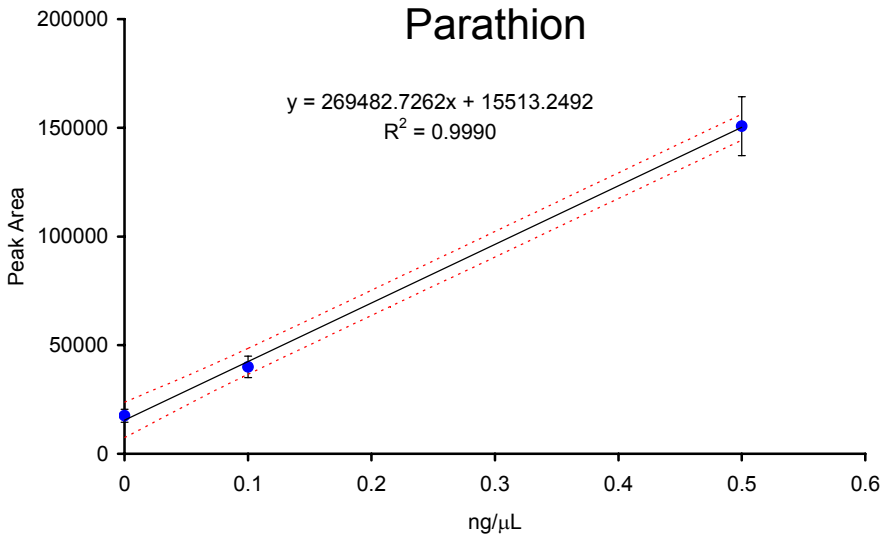
Match Probability 82.1%



Analysis of CW agent simulants

Pesticide Quantitation

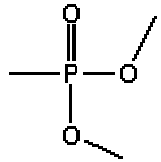




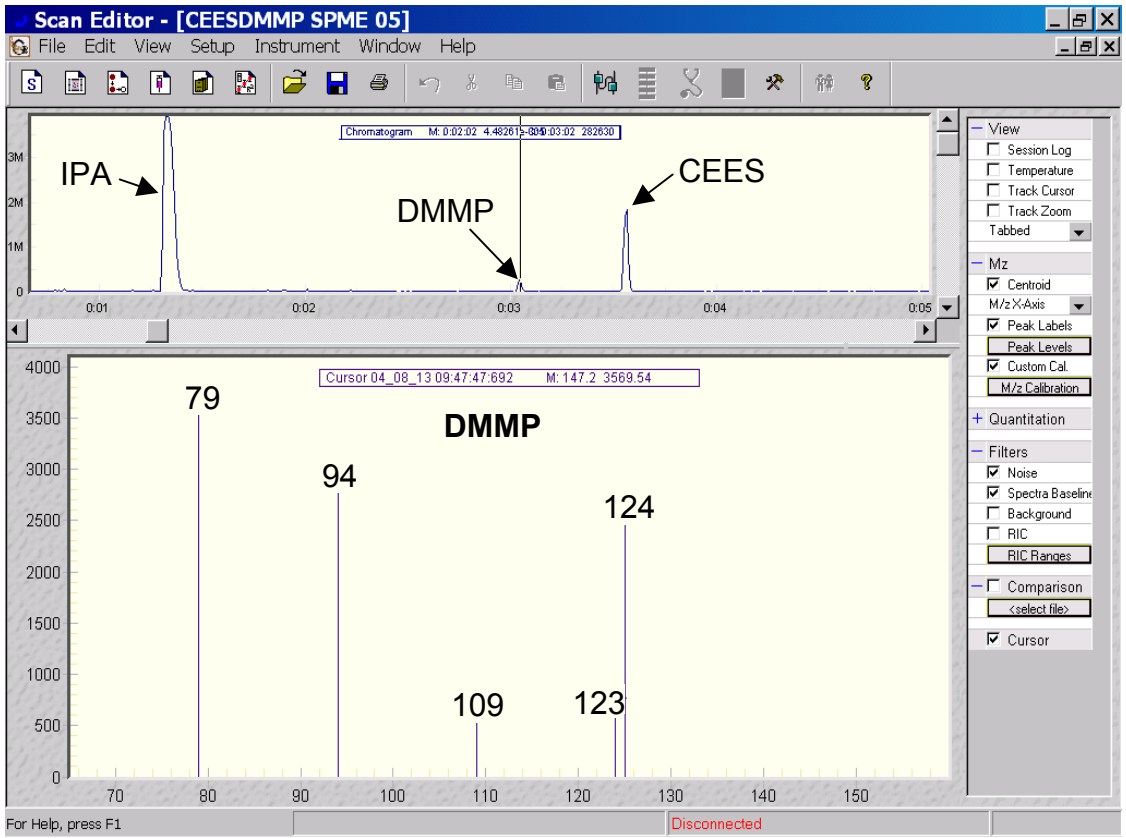
Compound	Limit of Detection (pg/µL)
O,O,O-triethylthiophosphate	9
thionazin	21
sulfotep	45
phorate	10
dimethoate	44
disulfoton	34
methyl parathion	26
parathion	41
famphur	~1000 pg (not detected in 500 pg/uL sample)

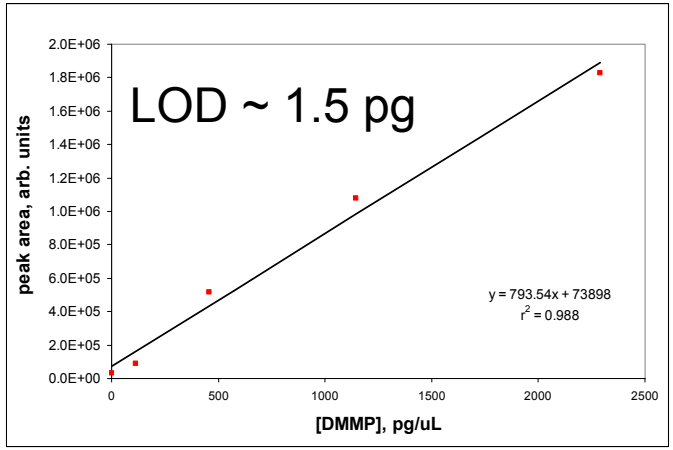
Analysis of CW agent simulants

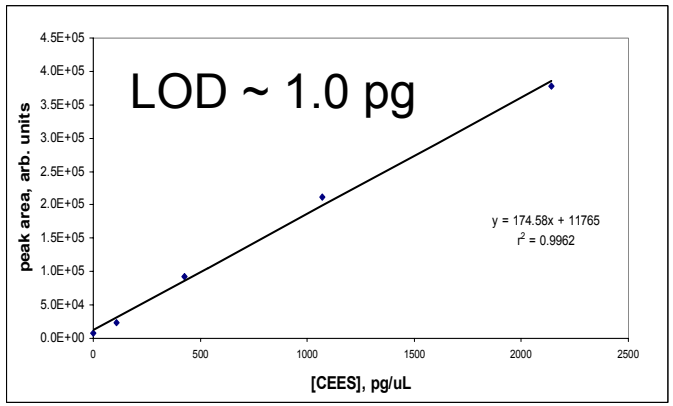
Dimethyl methylphosphonate (DMMP)



Chloroethyl ethyl sulfide (CEES)

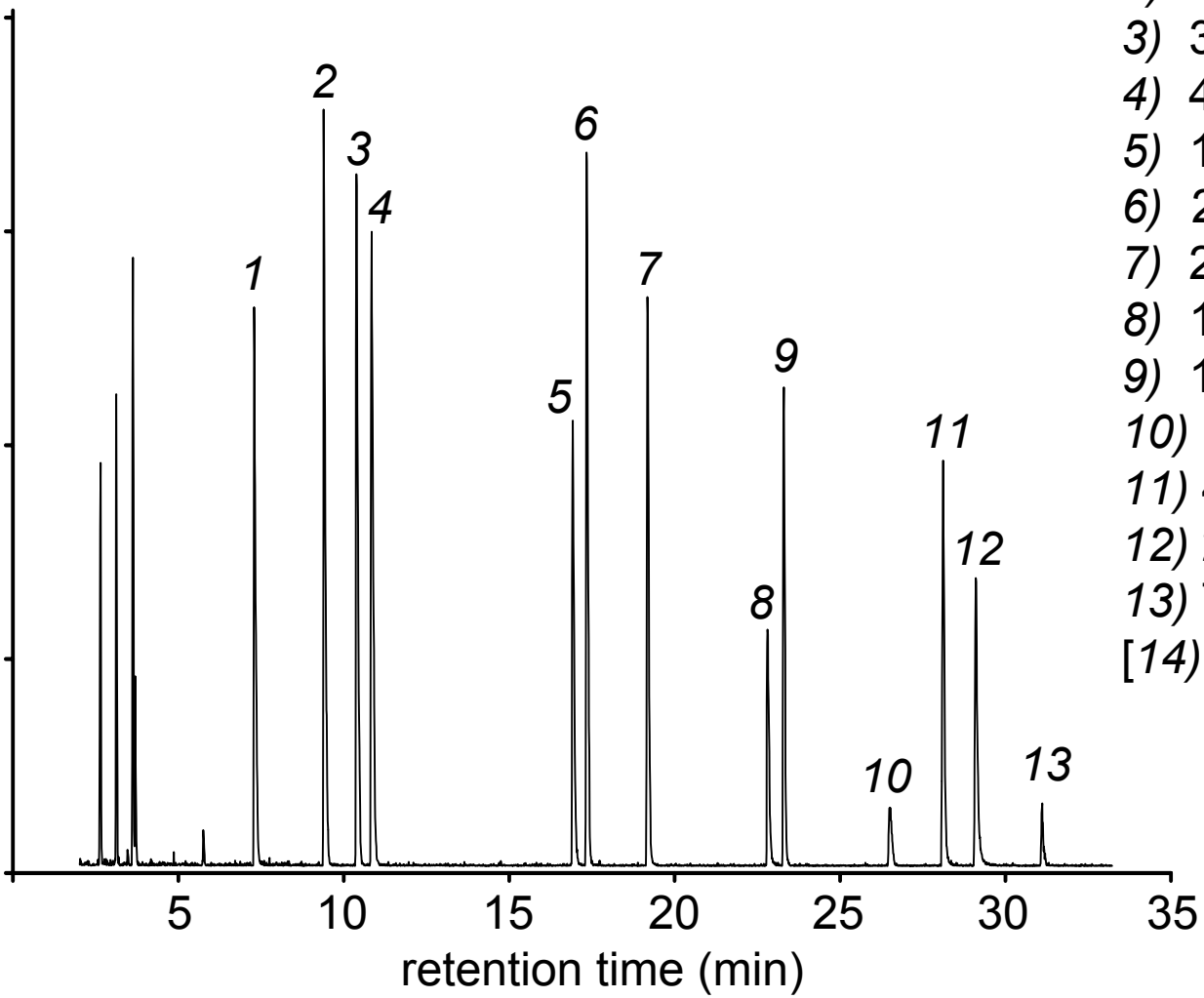






Analysis of Explosives

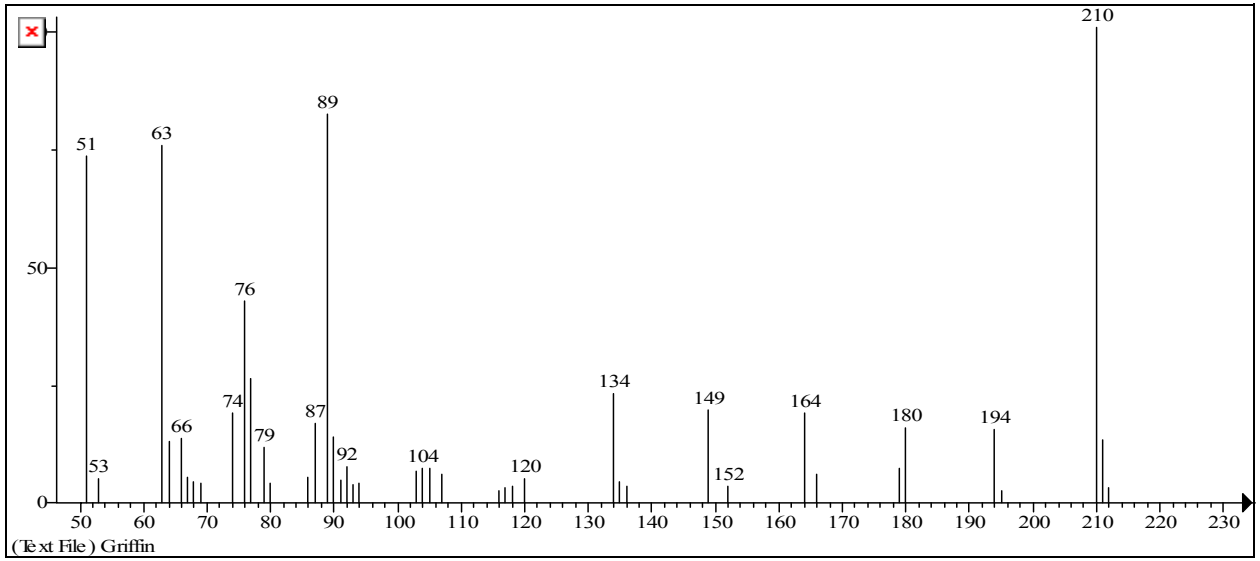
Separation of 14 nitrated organics
15 m x 0.25 mm Rtx-TNT1 column



1) Nitrobenzene
2) 2-Nitrotoluene
3) 3-Nitrotoluene
4) 4-Nitrotoluene
5) 1,3-Dinitrobenzene
6) 2,6-Dinitrotoluene
7) 2,4-Dinitrotoluene
8) 1,3,5-Trinitrobenzene
9) 1,3,5-Trinitrotoluene
10) RDX
11) 4-Amino-2,6-dinitrotoluene
12) 2-Amino-4,6-dinitrotoluene
13) Tetryl
[*14)* HMX not observed]

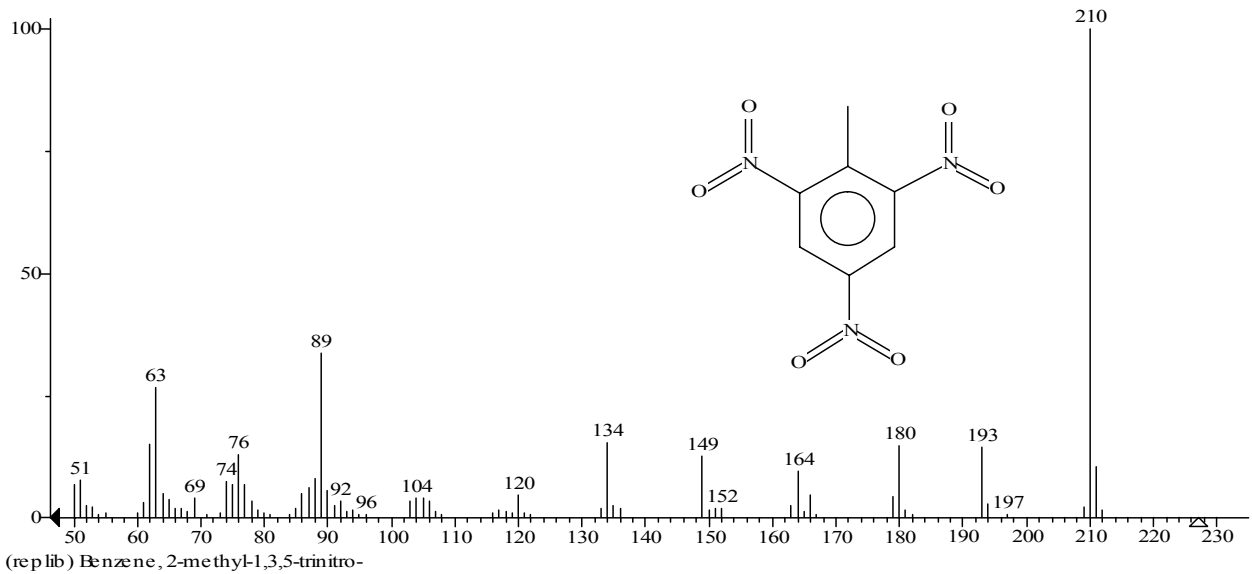
Analysis of Explosives

MS of Peak #9



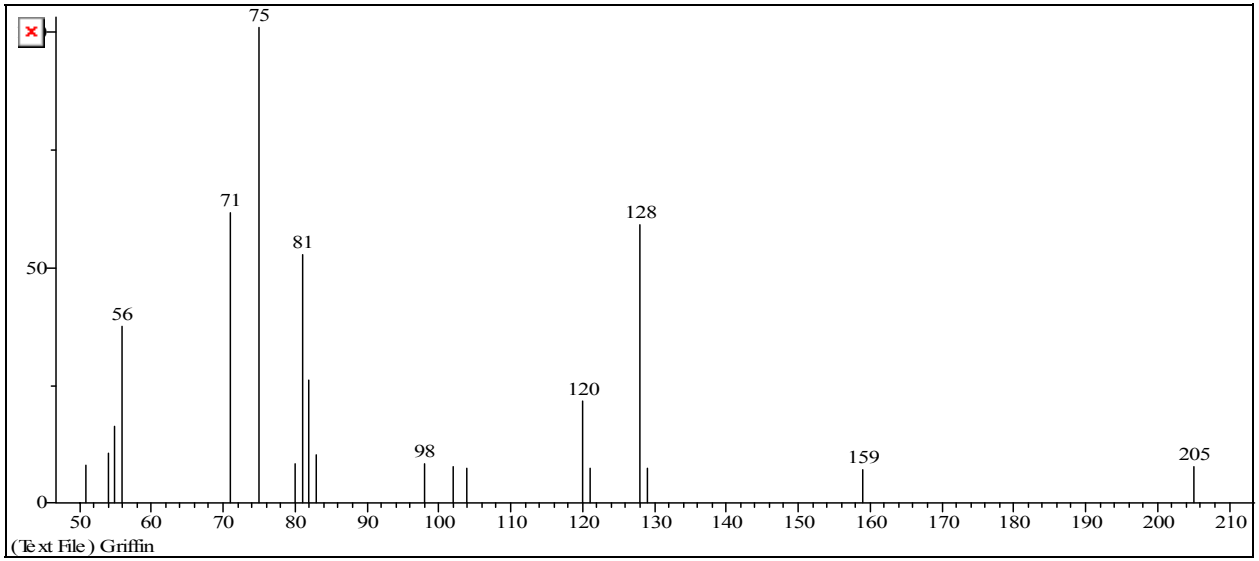
NIST MS of TNT

Match Probability 90.5%



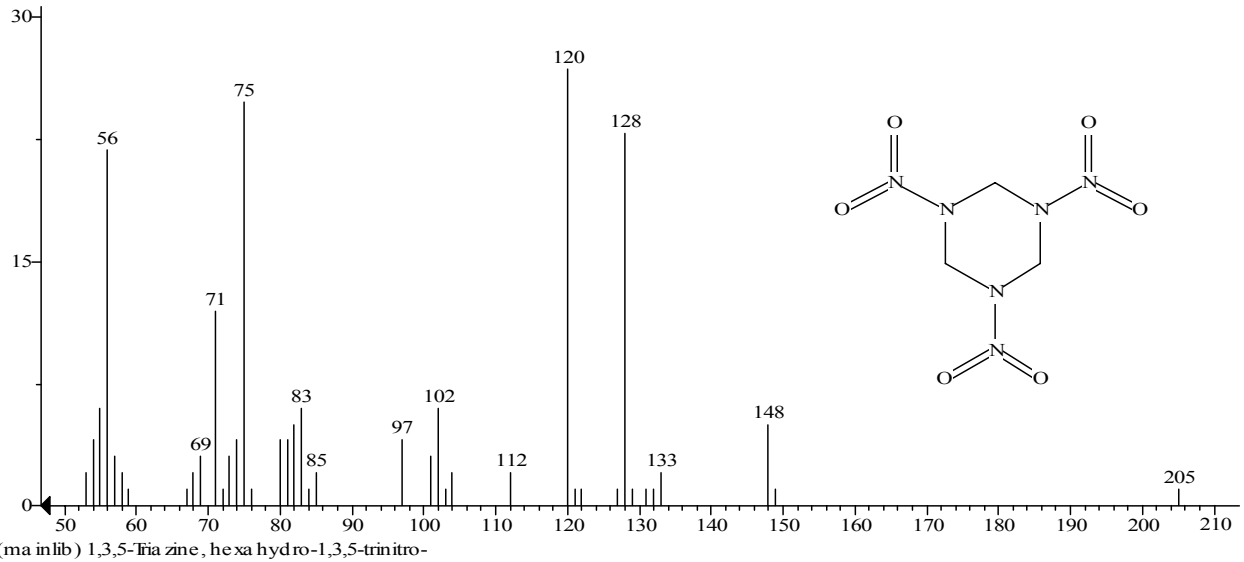
Analysis of Explosives

MS of Peak #10



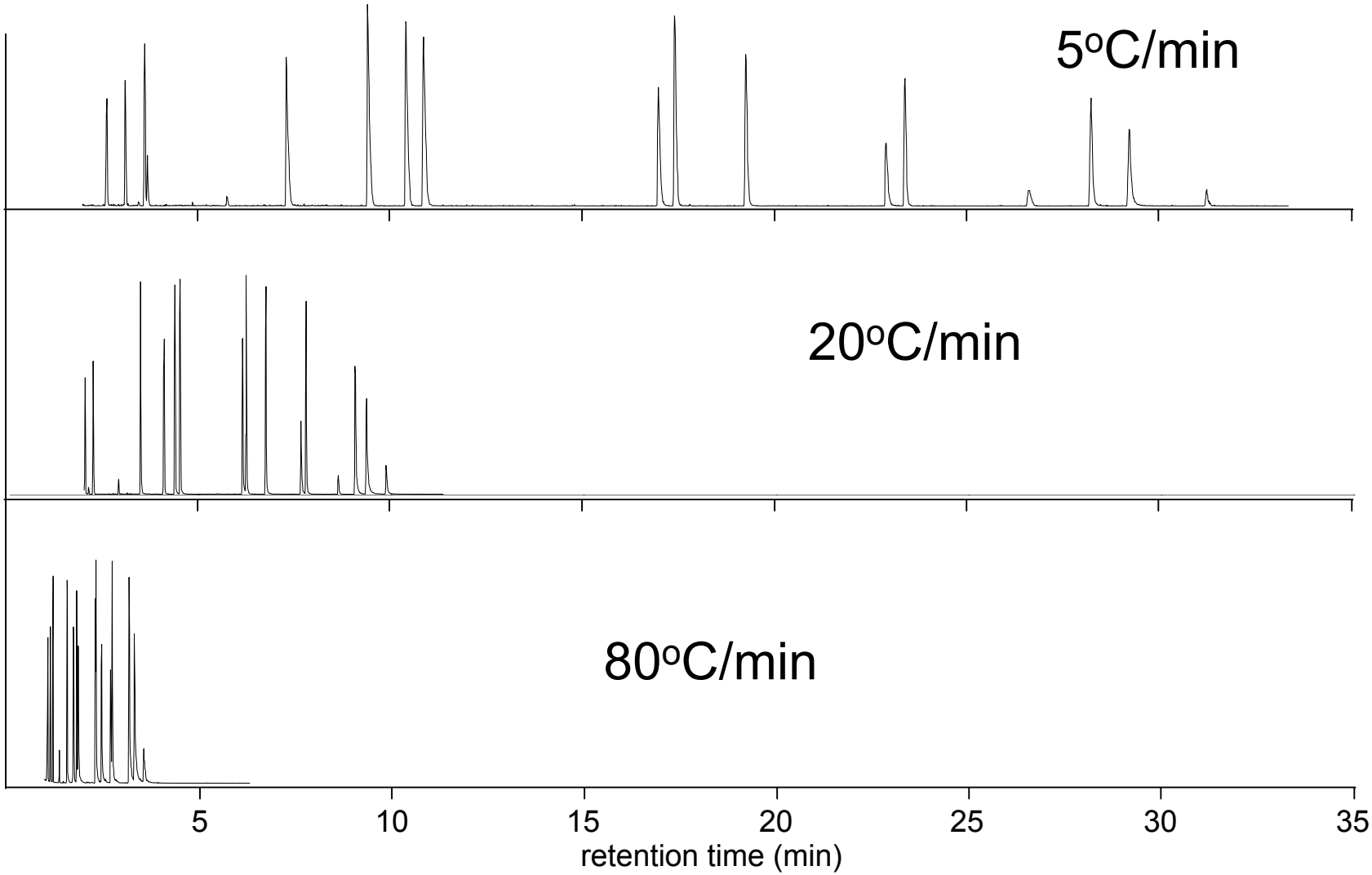
NIST MS of RDX

Match Probability 80.3%



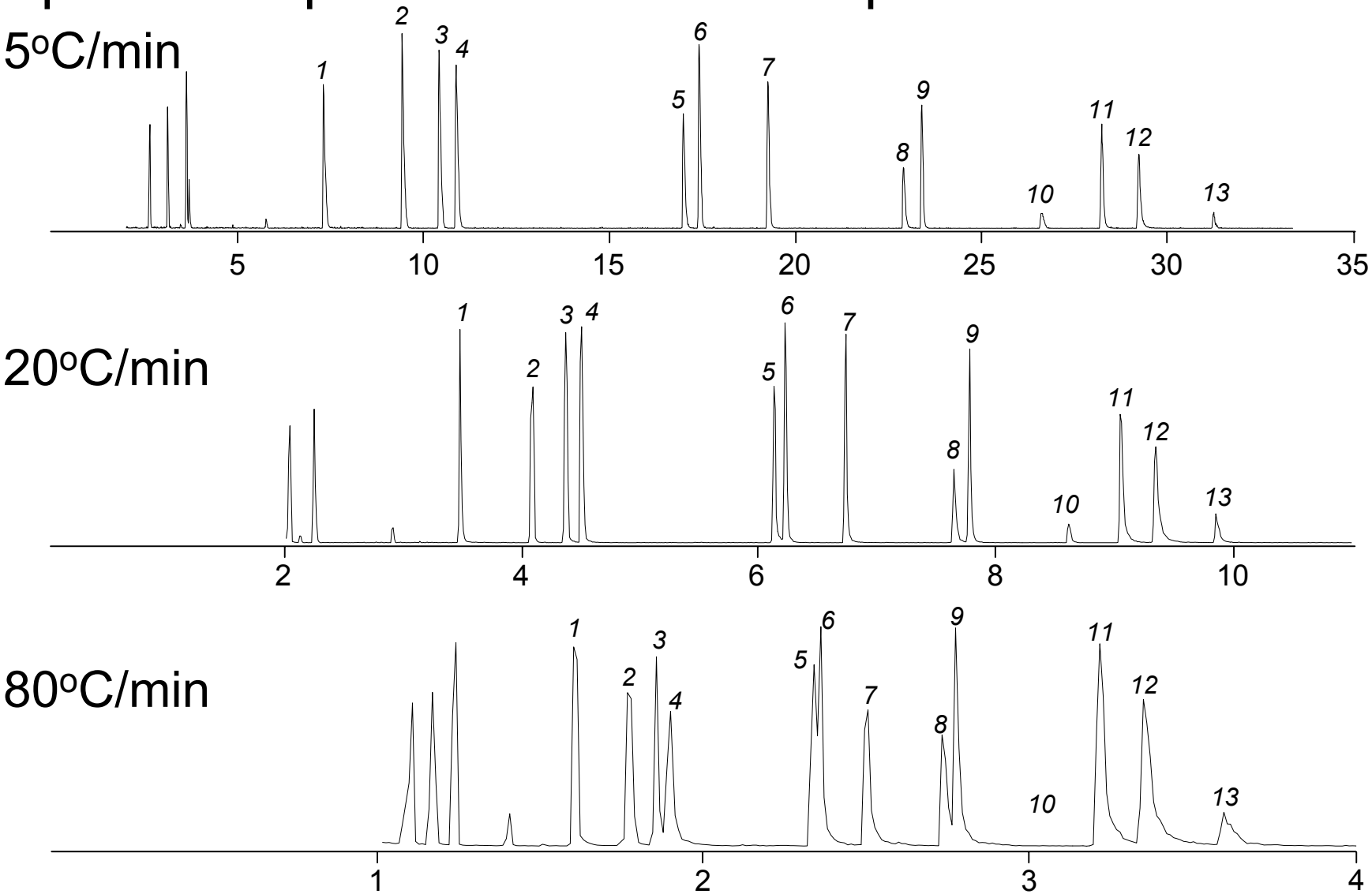
Analysis of Explosives

Explosive separation at faster ramp rates



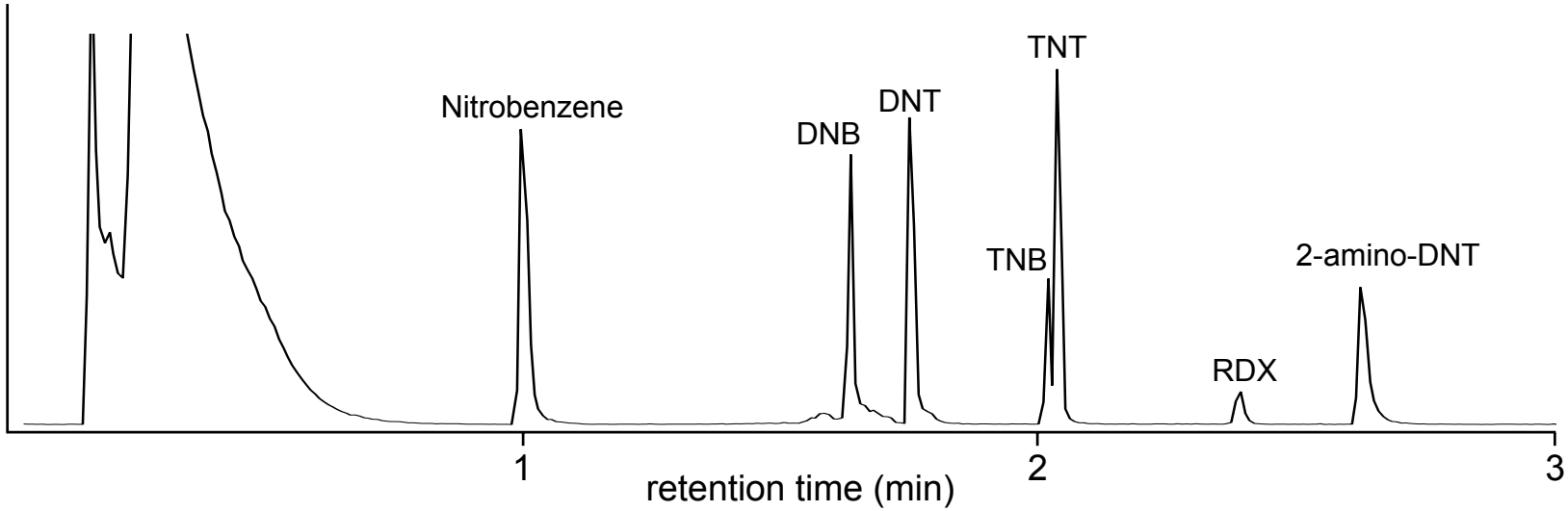
Analysis of Explosives

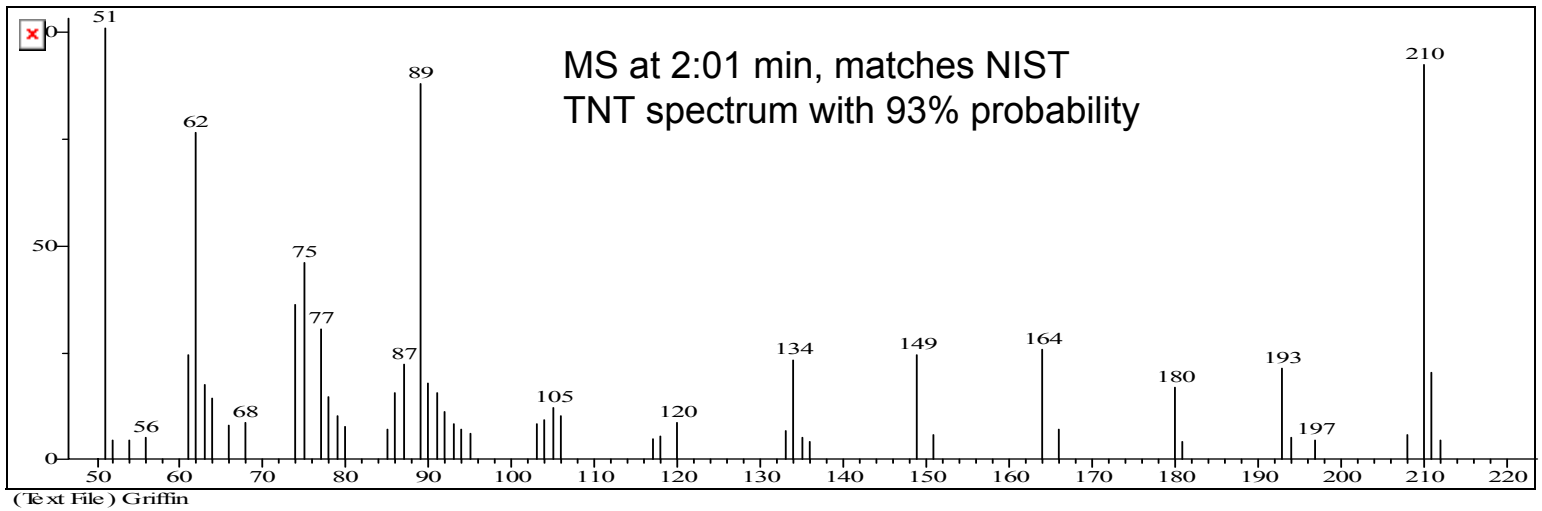
Explosive separation at faster ramp rates



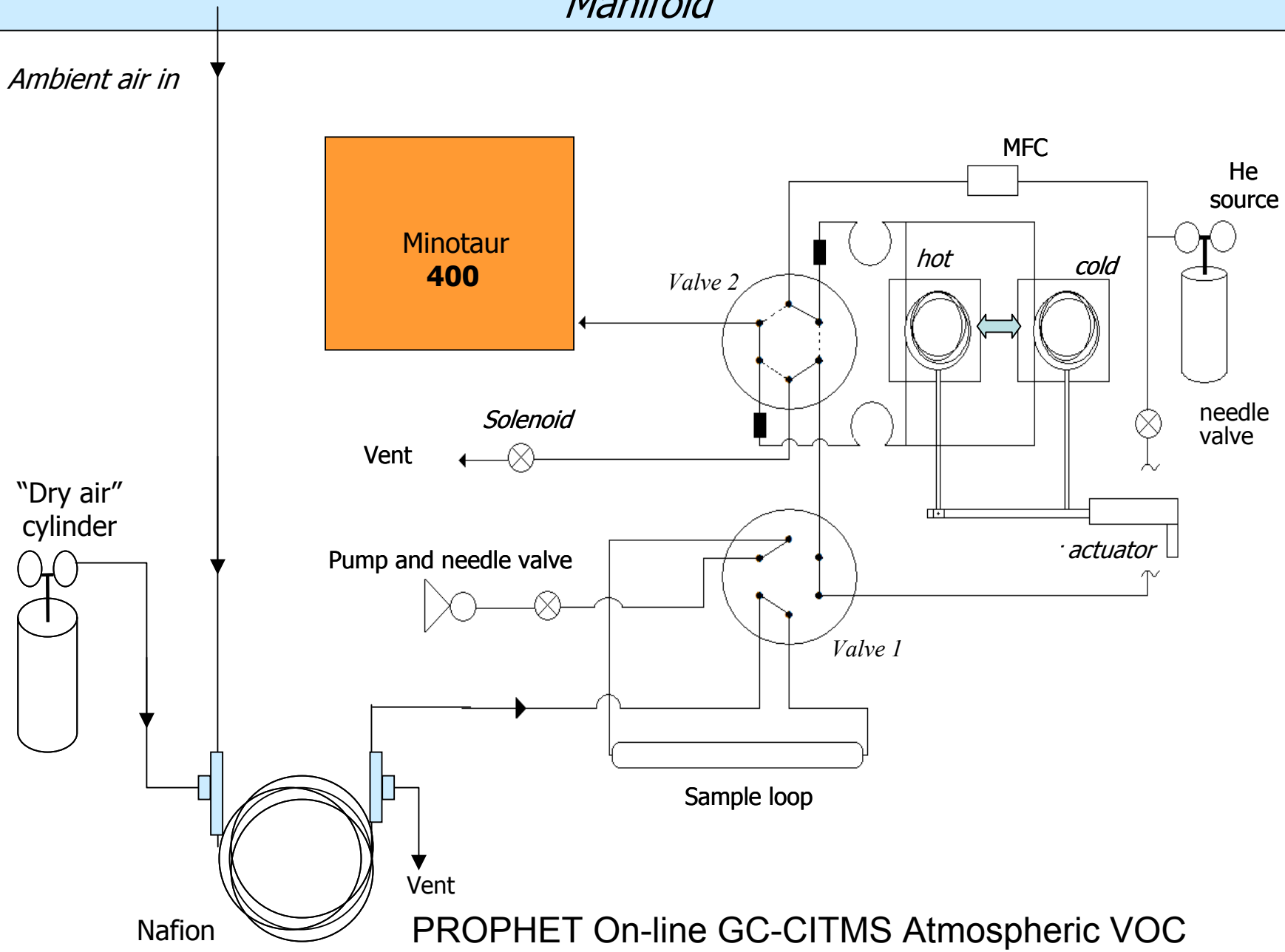
Analysis of Explosives

Explosive separation on a 5 m Rtx-5MS column



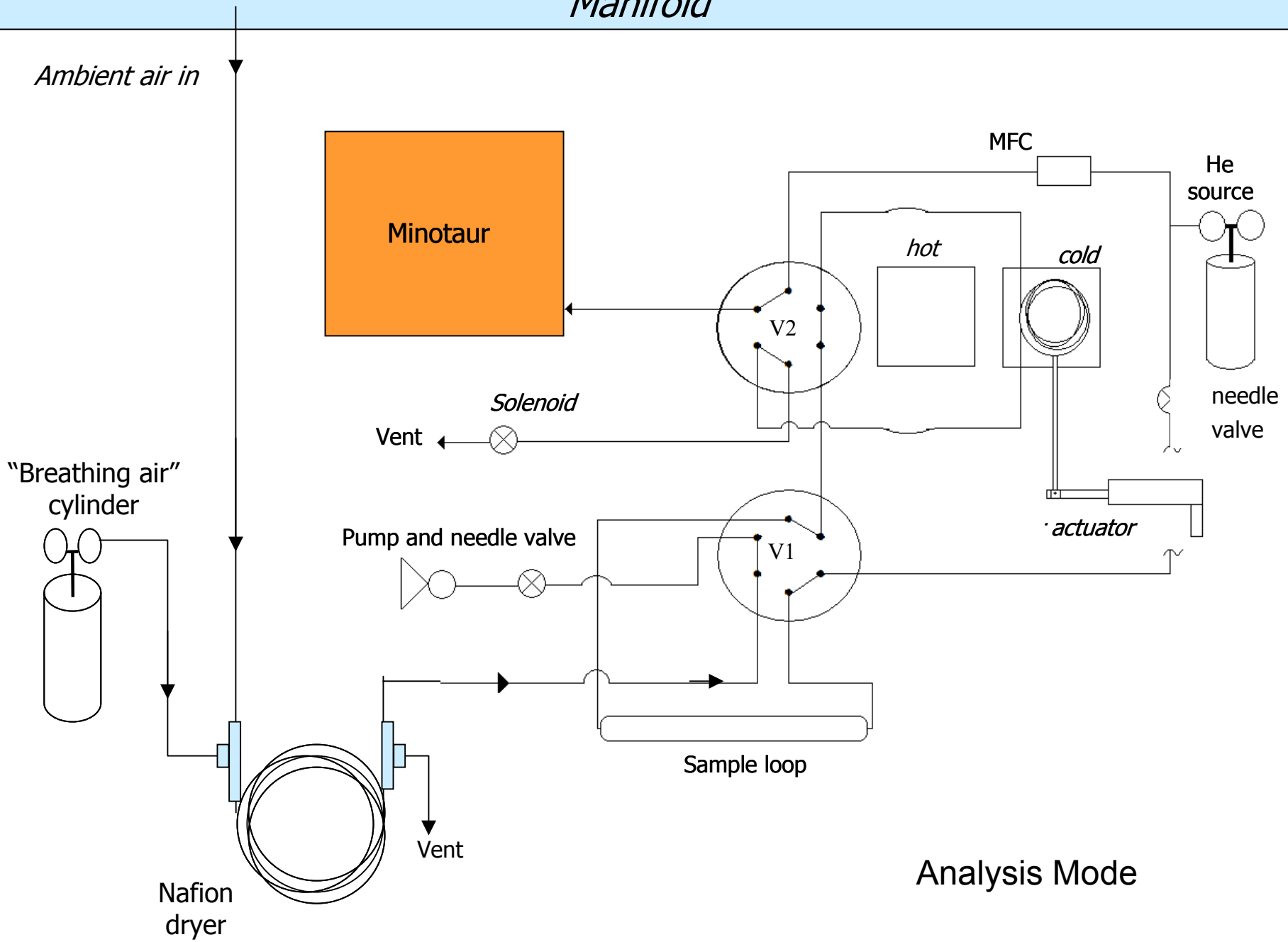


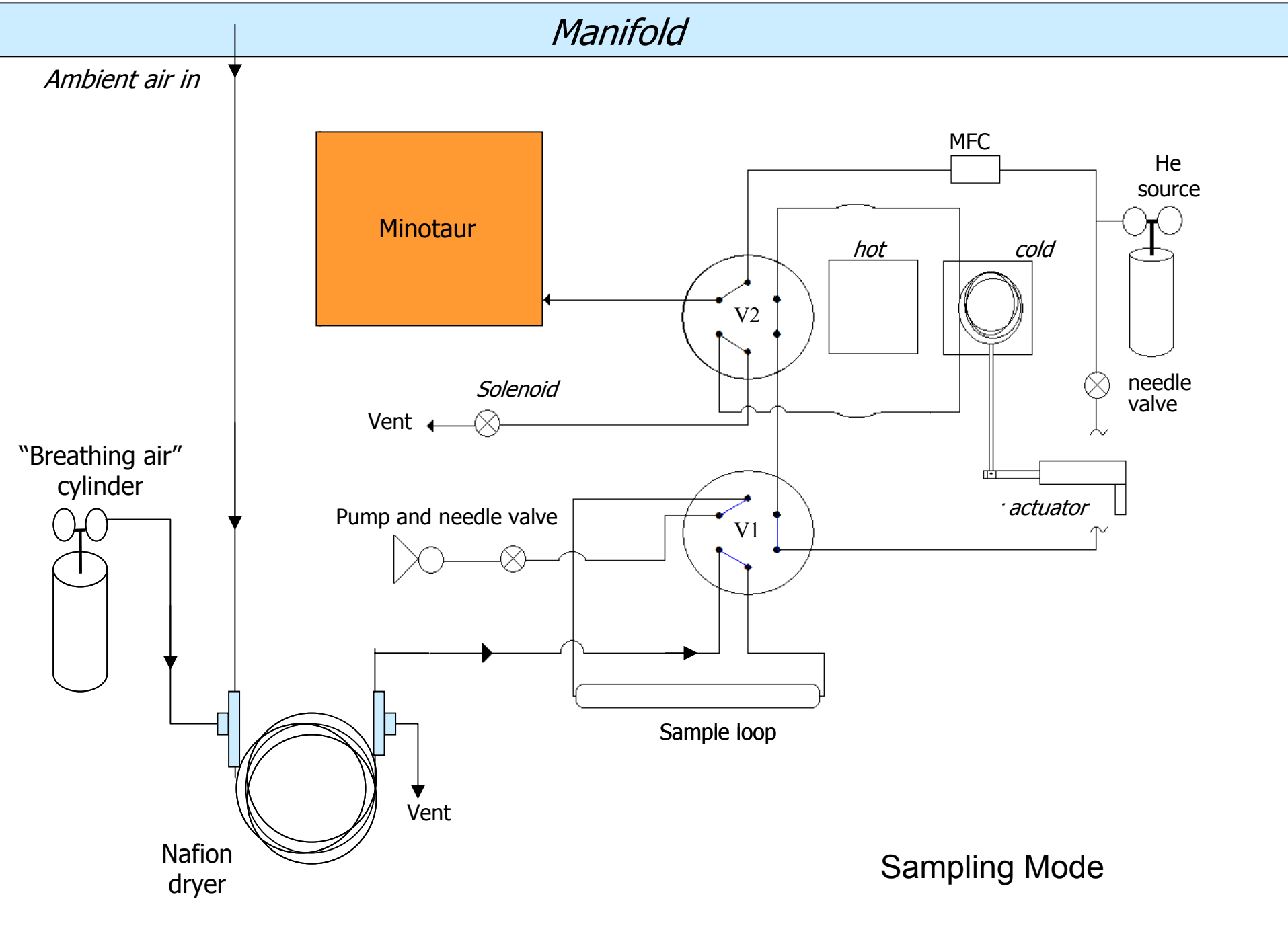
Manifold

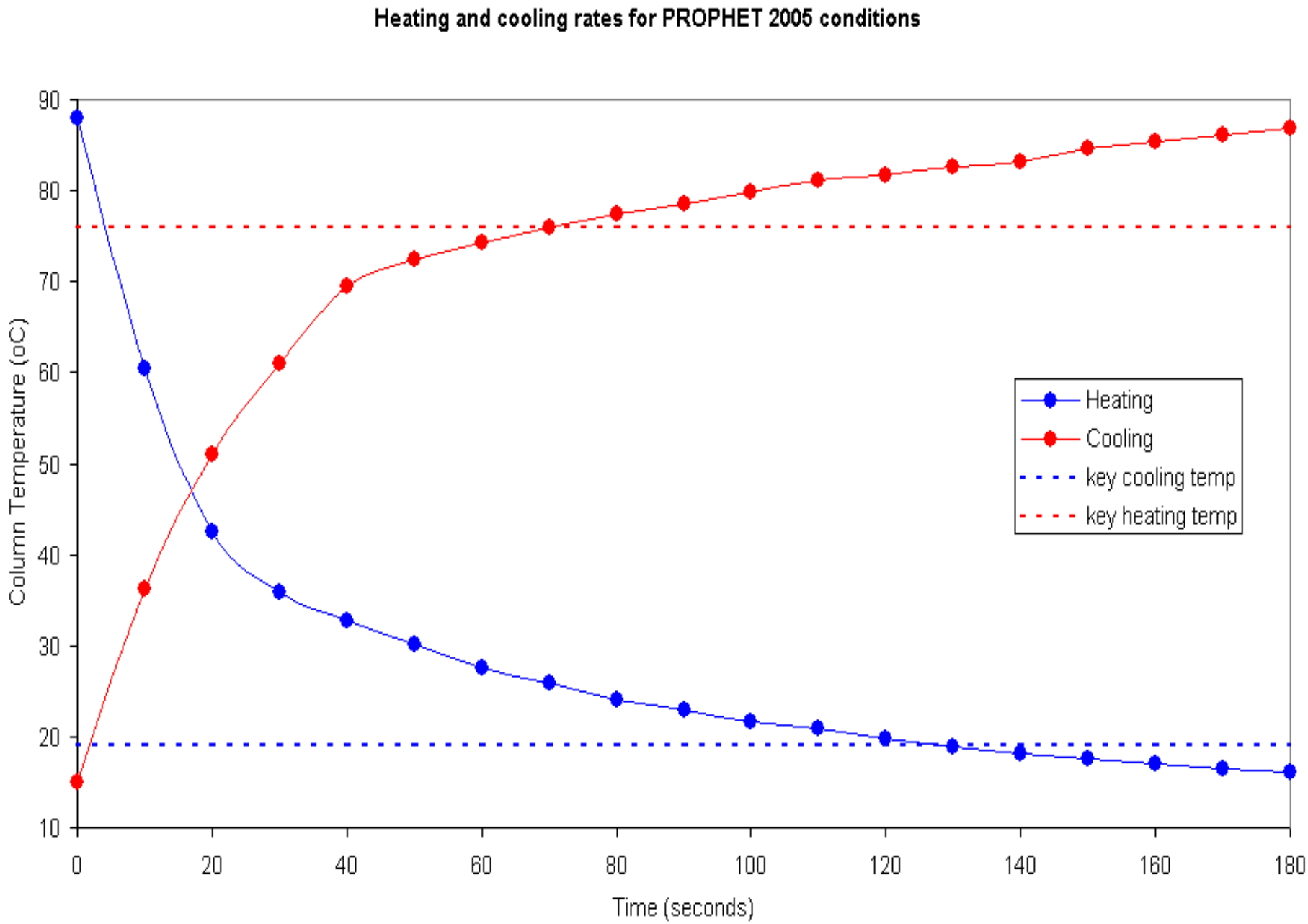


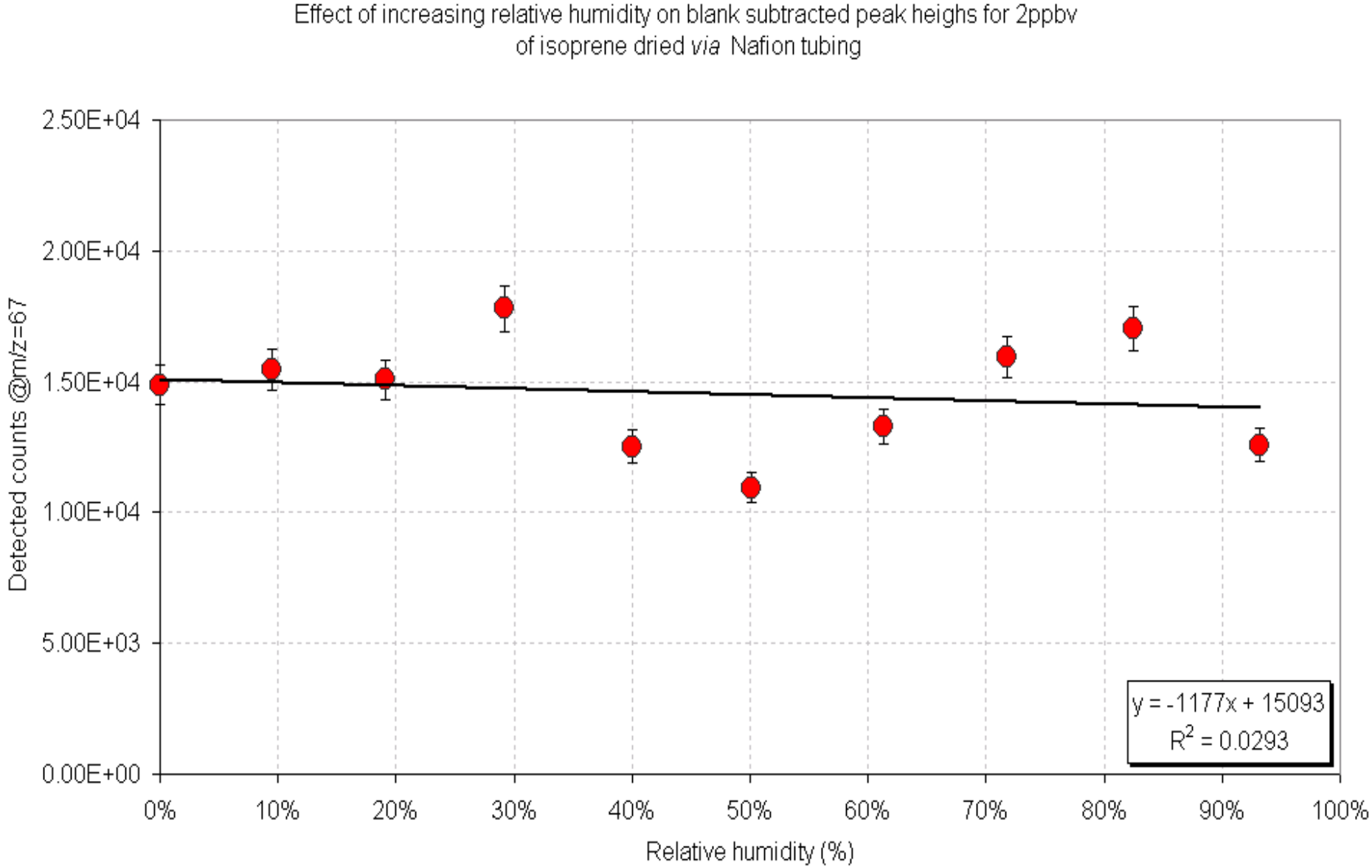
PROPHET On-line GC-CITMS Atmospheric VOC Monitoring System (Gavin Edwards, Paul Shepson)

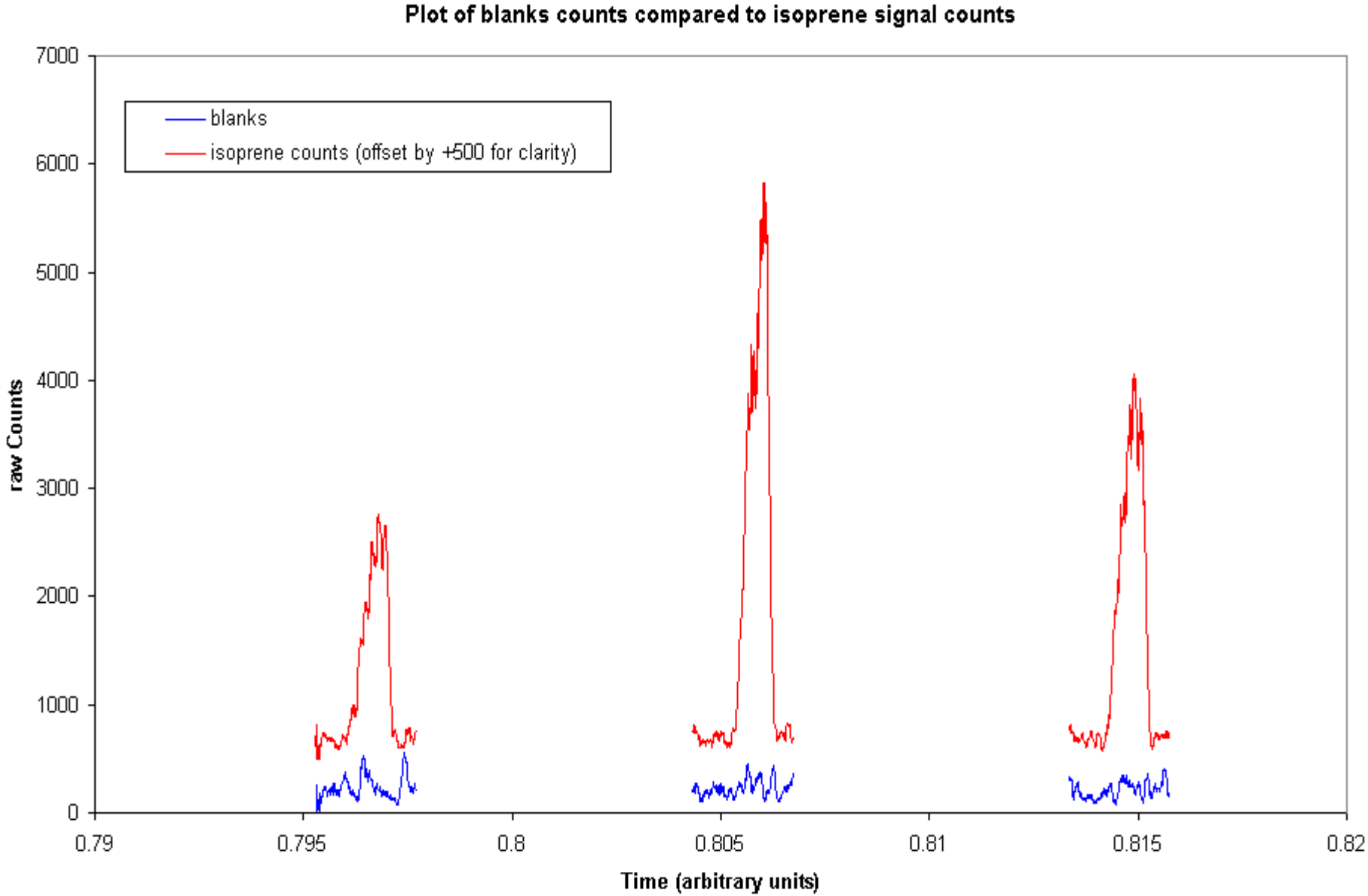
Manifold

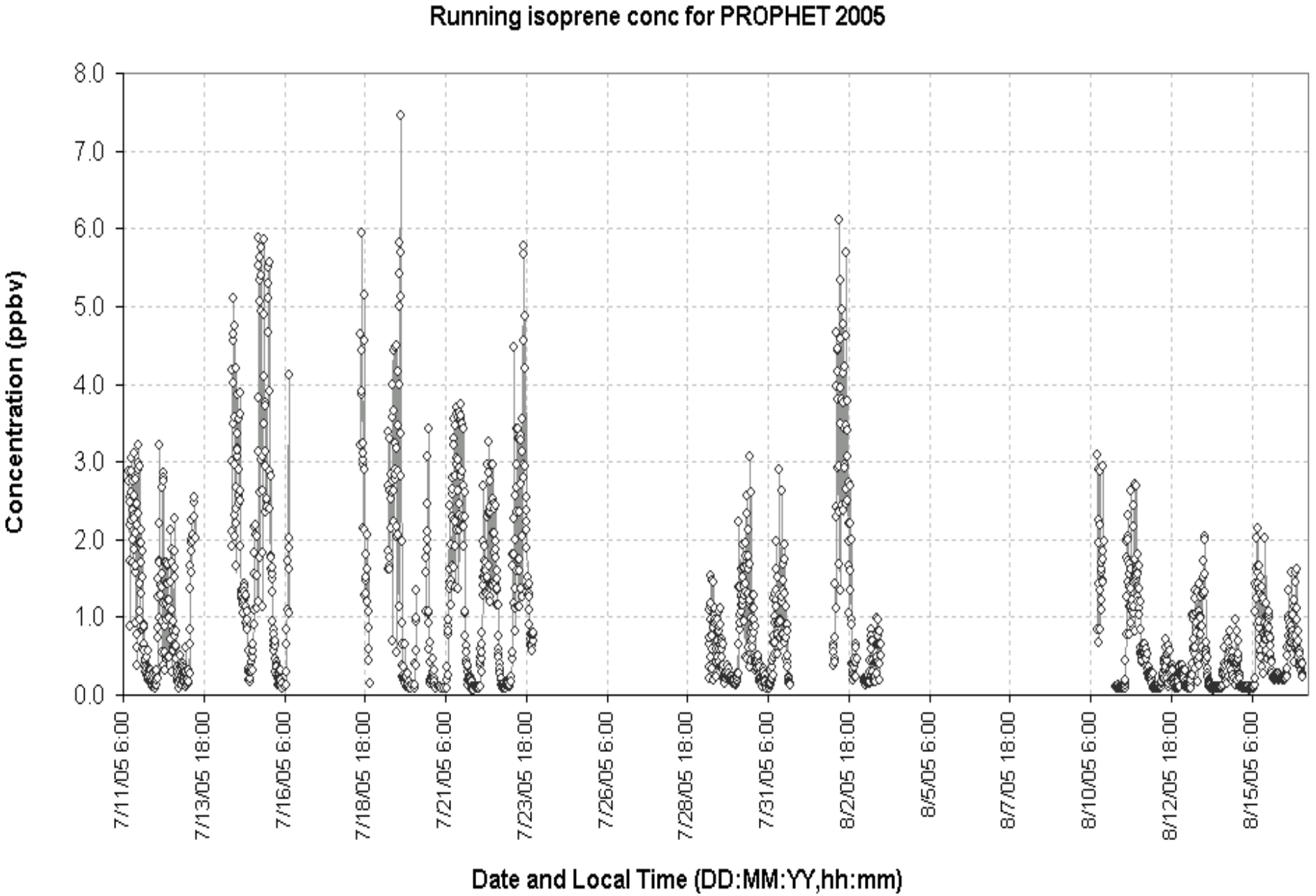

Ambient air in
Minotaur
MFC
He source
hot
cold
V2
Solenoid
Vent
needle valve
"Breathing air" cylinder
actuator
Pump and needle valve
V1
Sample loop
Vent
Nafion dryer
Analysis Mode

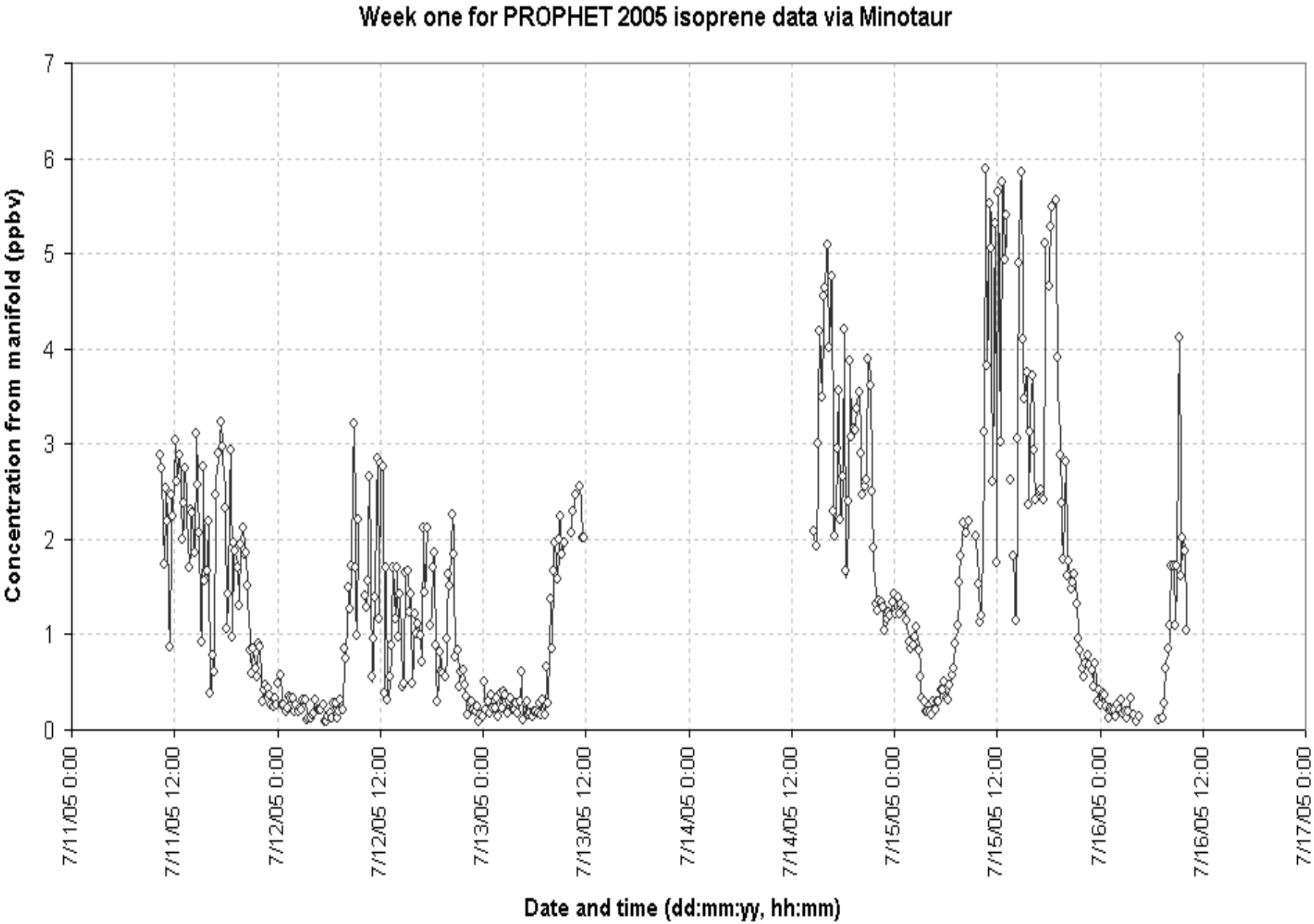

Manifold
Ambient air in
Minotaur
MFC
He source
hot
cold
V2
Solenoid
Vent
needle valve
"Breathing air" cylinder
actuator
Pump and needle valve
V1
Sample loop
Vent
Nafion dryer
Sampling Mode

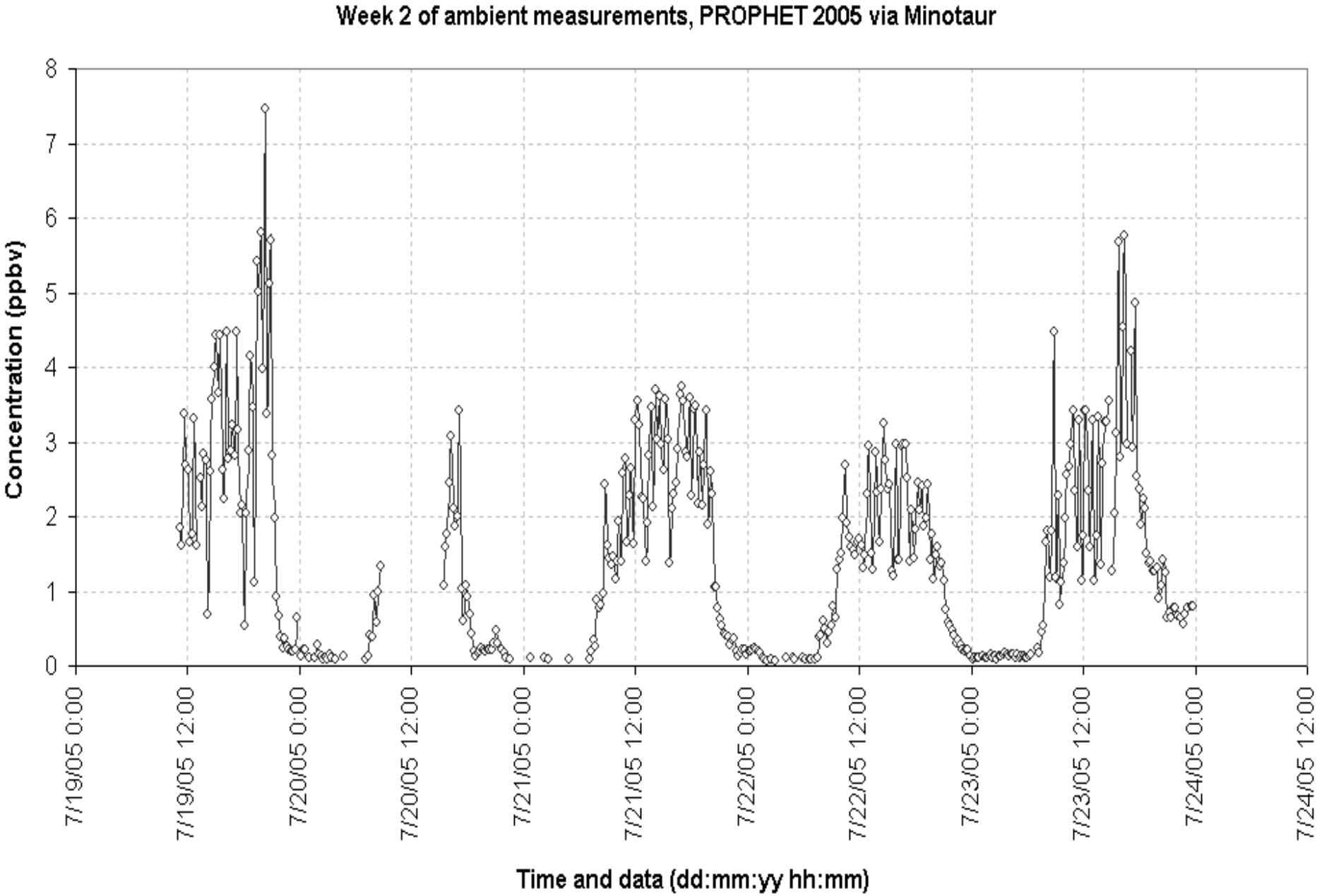

Heating and cooling rates for PROPHET 2005 conditions
Column Temperature (oC)
Time (seconds)
90
80
70
60
50
40
30
20
10
0
20
40
60
80
100
120
140
160
180
Heating
Cooling
key cooling temp
key heating temp

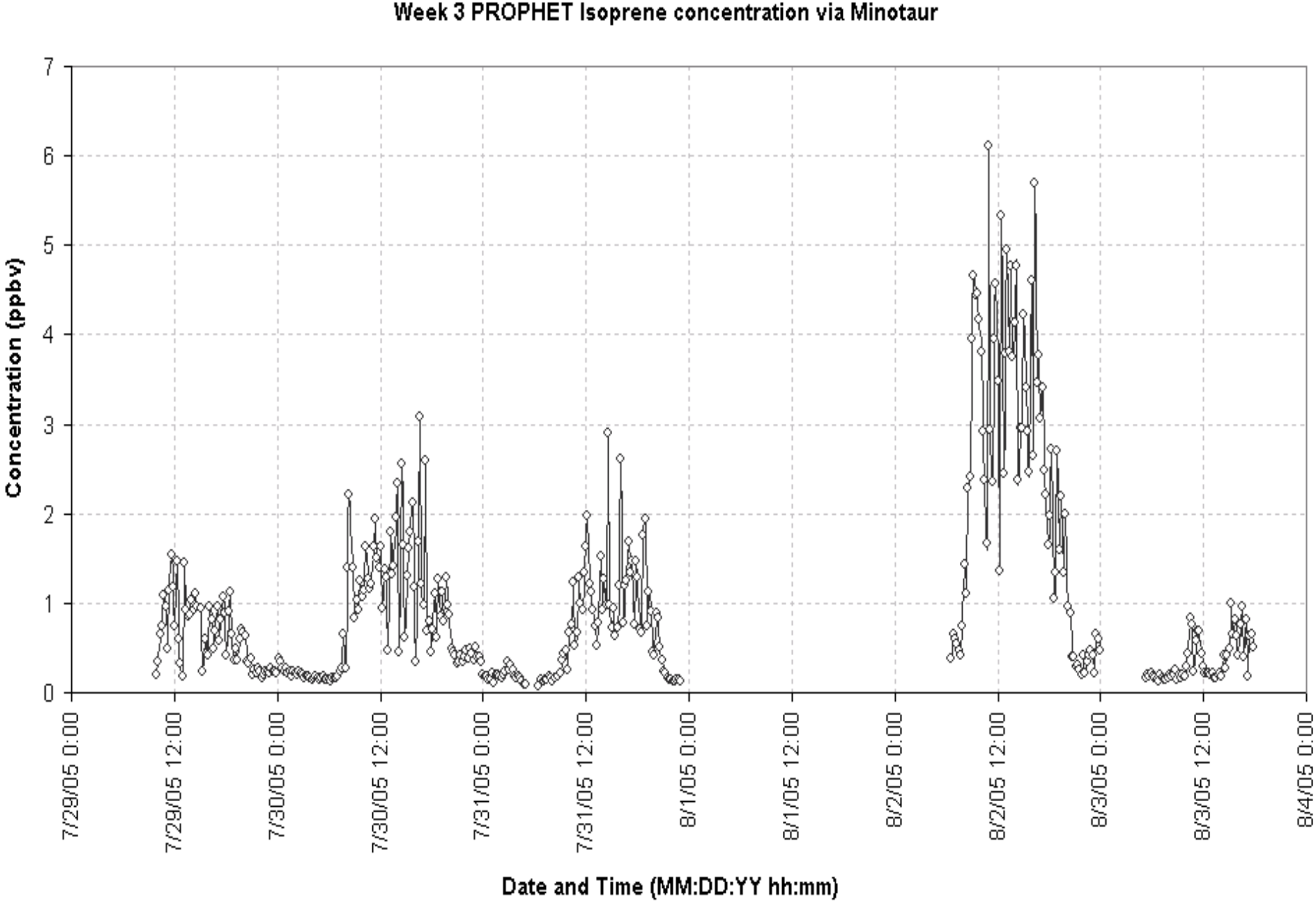

Effect of increasing relative humidity on blank subtracted peak heighs for 2ppbv of isoprene dried *via* Nafion tubing
2.50E+04
2.00E+04
1.50E+04
1.00E+04
5.00E+03
0.00E+00
Detected counts @m/z=67
0%
10%
20%
30%
40%
50%
60%
70%
80%
90%
100%
Relative humidity (%)
y = -1177x + 15093
R2 = 0.0293

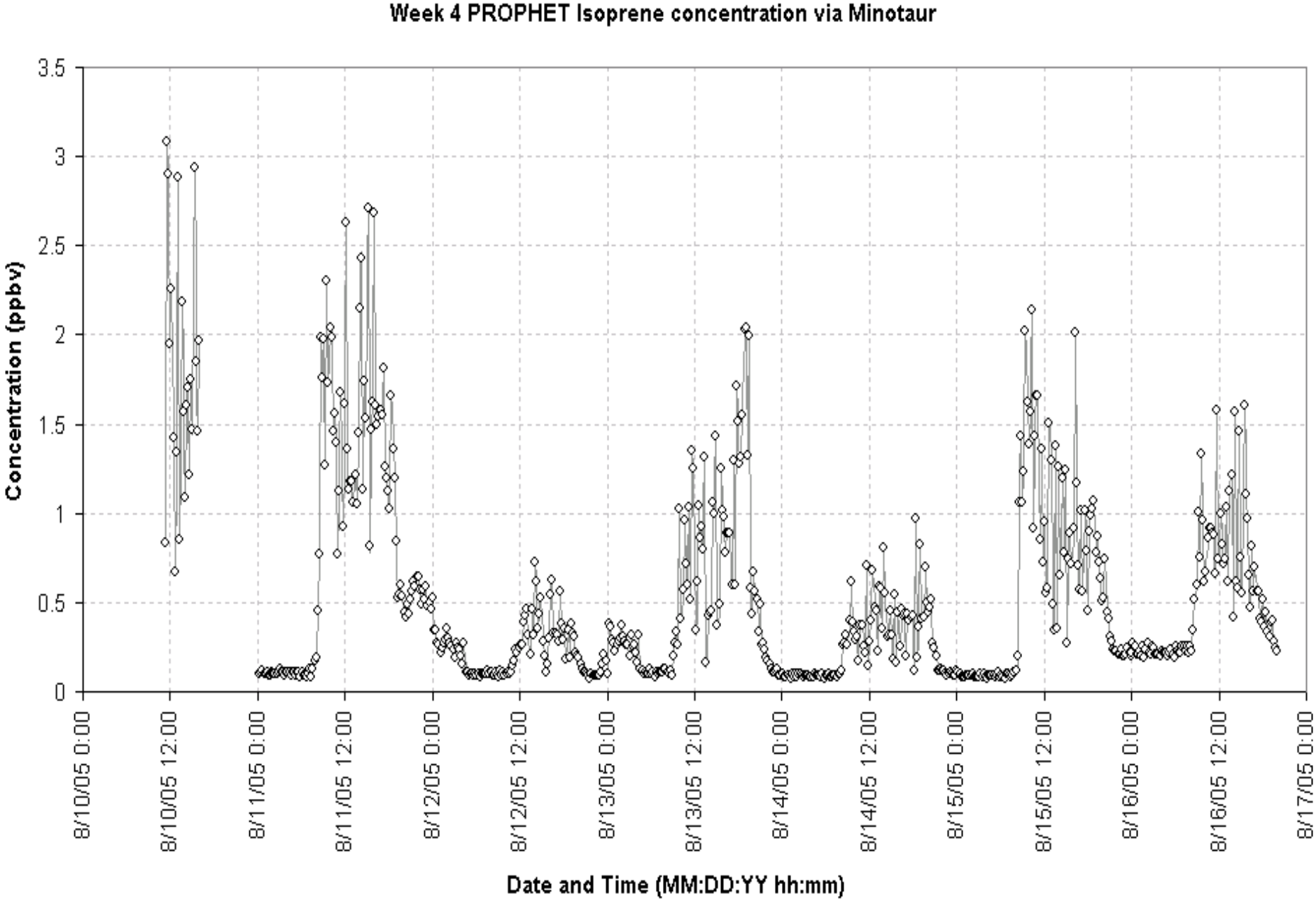

Plot of blanks counts compared to isoprene signal counts
blanks
isoprene counts (offset by +500 for clarity)
raw Counts
7000
6000
5000
4000
3000
2000
1000
0
0.79
0.795
0.8
0.805
0.81
0.815
0.82
Time (arbitrary units)

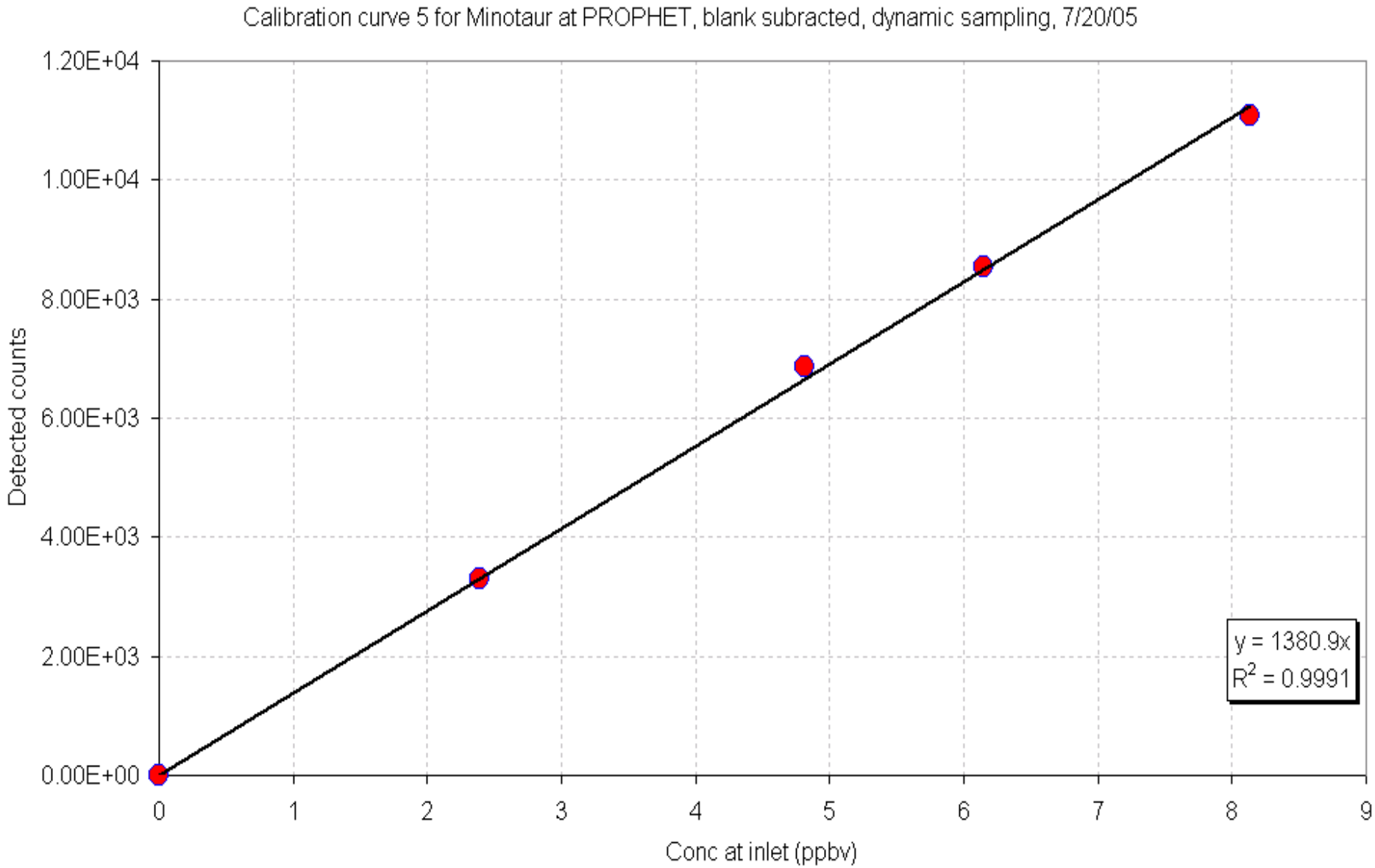

Running isoprene conc for PROPHET 2005
Concentration (ppbv)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
7/11/05 6:00
7/13/05 18:00
7/16/05 6:00
7/18/05 18:00
7/21/05 6:00
7/23/05 18:00
7/26/05 6:00
7/28/05 18:00
7/31/05 6:00
8/2/05 18:00
8/5/05 6:00
8/7/05 18:00
8/10/05 6:00
8/12/05 18:00
8/15/05 6:00
Date and Local Time (DD:MM:YY,hh:mm)


Week one for PROPHET 2005 isoprene data via Minotaur
Concentration from manifold (ppbv)
0
1
2
3
4
5
6
7
7/11/05 0:00
7/11/05 12:00
7/12/05 0:00
7/12/05 12:00
7/13/05 0:00
7/13/05 12:00
7/14/05 0:00
7/14/05 12:00
7/15/05 0:00
7/15/05 12:00
7/16/05 0:00
7/16/05 12:00
7/17/05 0:00
Date and time (dd:mm:yy, hh:mm)


Week 2 of ambient measurements, PROPHET 2005 via Minotaur
Concentration (ppbv)
0
1
2
3
4
5
6
7
8
7/19/05 0:00
7/19/05 12:00
7/20/05 0:00
7/20/05 12:00
7/21/05 0:00
7/21/05 12:00
7/22/05 0:00
7/22/05 12:00
7/23/05 0:00
7/23/05 12:00
7/24/05 0:00
7/24/05 12:00
Time and data (dd:mm:yy hh:mm)


Week 3 PROPHET Isoprene concentration via Minotaur
Concentration (ppbv)
0
1
2
3
4
5
6
7
7/29/05 0:00
7/29/05 12:00
7/30/05 0:00
7/30/05 12:00
7/31/05 0:00
7/31/05 12:00
8/1/05 0:00
8/1/05 12:00
8/2/05 0:00
8/2/05 12:00
8/3/05 0:00
8/3/05 12:00
8/4/05 0:00
Date and Time (MM:DD:YY hh:mm)


Week 4 PROPHET Isoprene concentration via Minotaur
Concentration (ppbv)
3.5
3
2.5
2
1.5
1
0.5
0
8/10/05 0:00
8/10/05 12:00
8/11/05 0:00
8/11/05 12:00
8/12/05 0:00
8/12/05 12:00
8/13/05 0:00
8/13/05 12:00
8/14/05 0:00
8/14/05 12:00
8/15/05 0:00
8/15/05 12:00
8/16/05 0:00
8/16/05 12:00
8/17/05 0:00
Date and Time (MM:DD:YY hh:mm)


Calibration curve 5 for Minotaur at PROPHET, blank subracted, dynamic sampling, 7/20/05
Detected counts
1.20E+04
1.00E+04
8.00E+03
6.00E+03
4.00E+03
2.00E+03
0.00E+00
0
1
2
3
4
5
6
7
8
9
Conc at inlet (ppbv)
y = 1380.9x
R2 = 0.9991

Field Study Summary

- Verified new inlet and MS system in an atmospheric chemistry field study

- 9 calibrations, humidity blanks evaluated

- >2000 measurements (7/11/2005-8/16/2005, off and on)

- 14 days with 24 hr coverage, 7 days with partial coverage

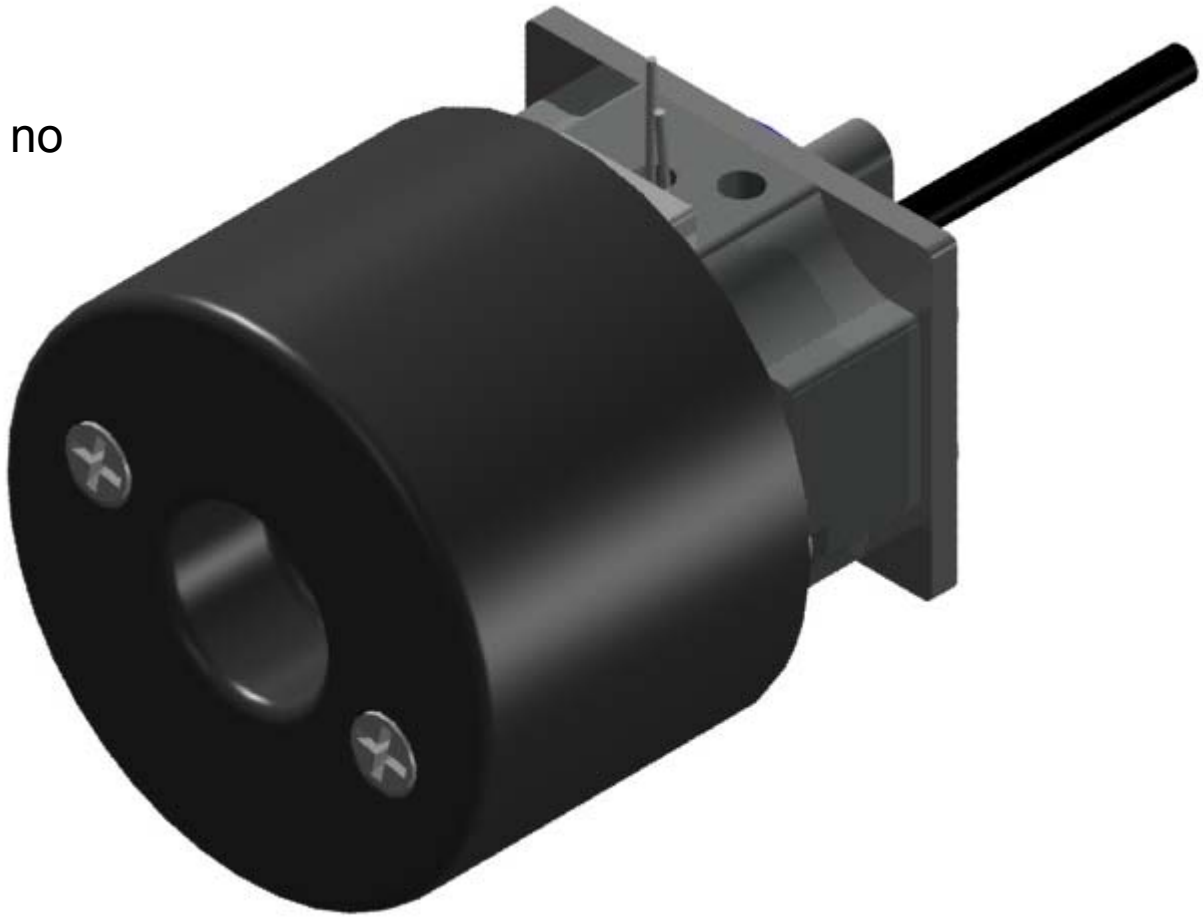
- Isoprene concentrations between <70 ppt (LOD) and >8 ppb

- Potential for intercomparison with other techniques such as FIS, DEA and PTRMS

Direct SPME

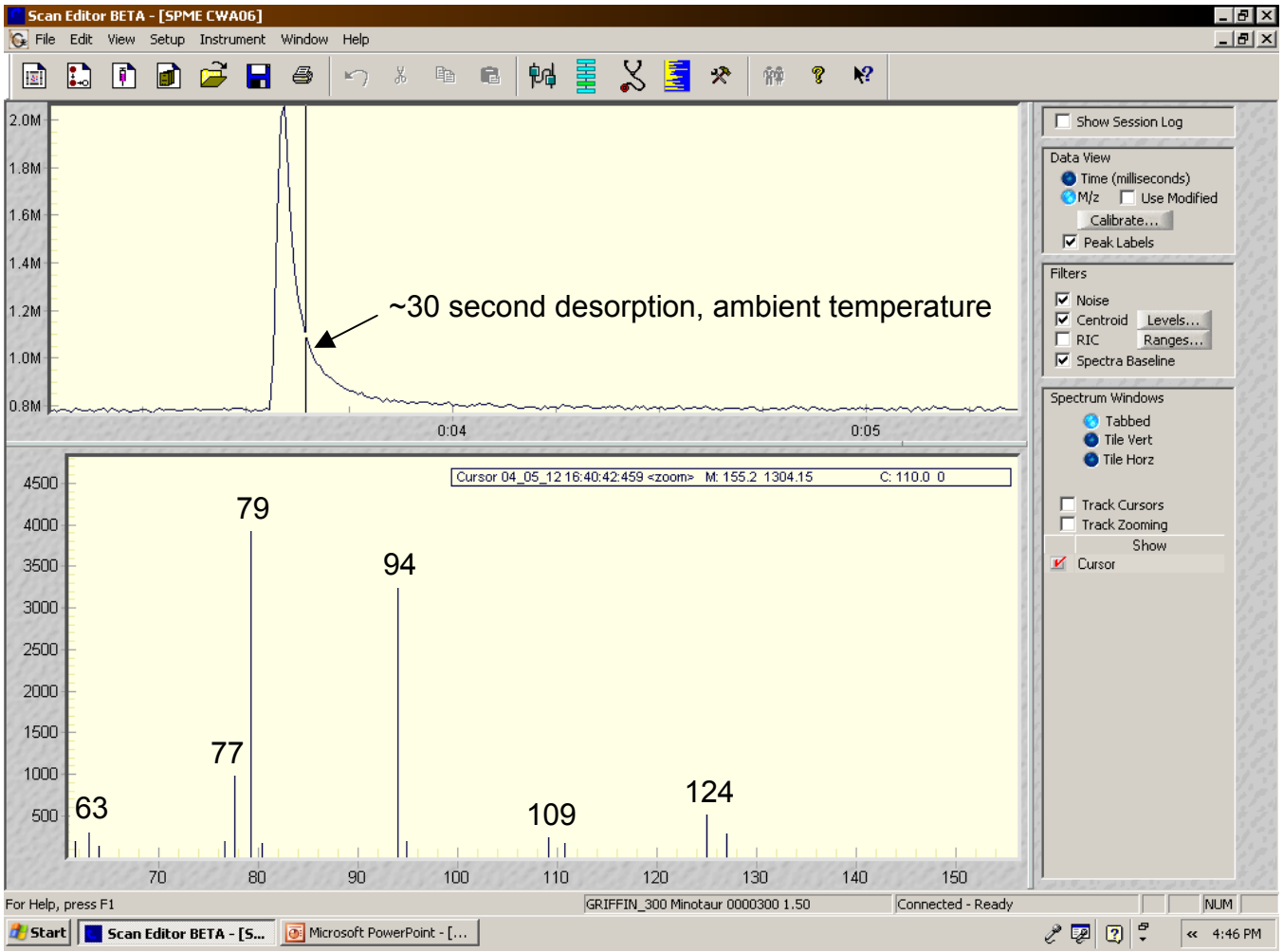
Direct Inject SPME
SPME fiber is directly inserted into
the vacuum chamber for rapid desorption

Max temp 300˚C
Replaceable septum with no
vacuum vent



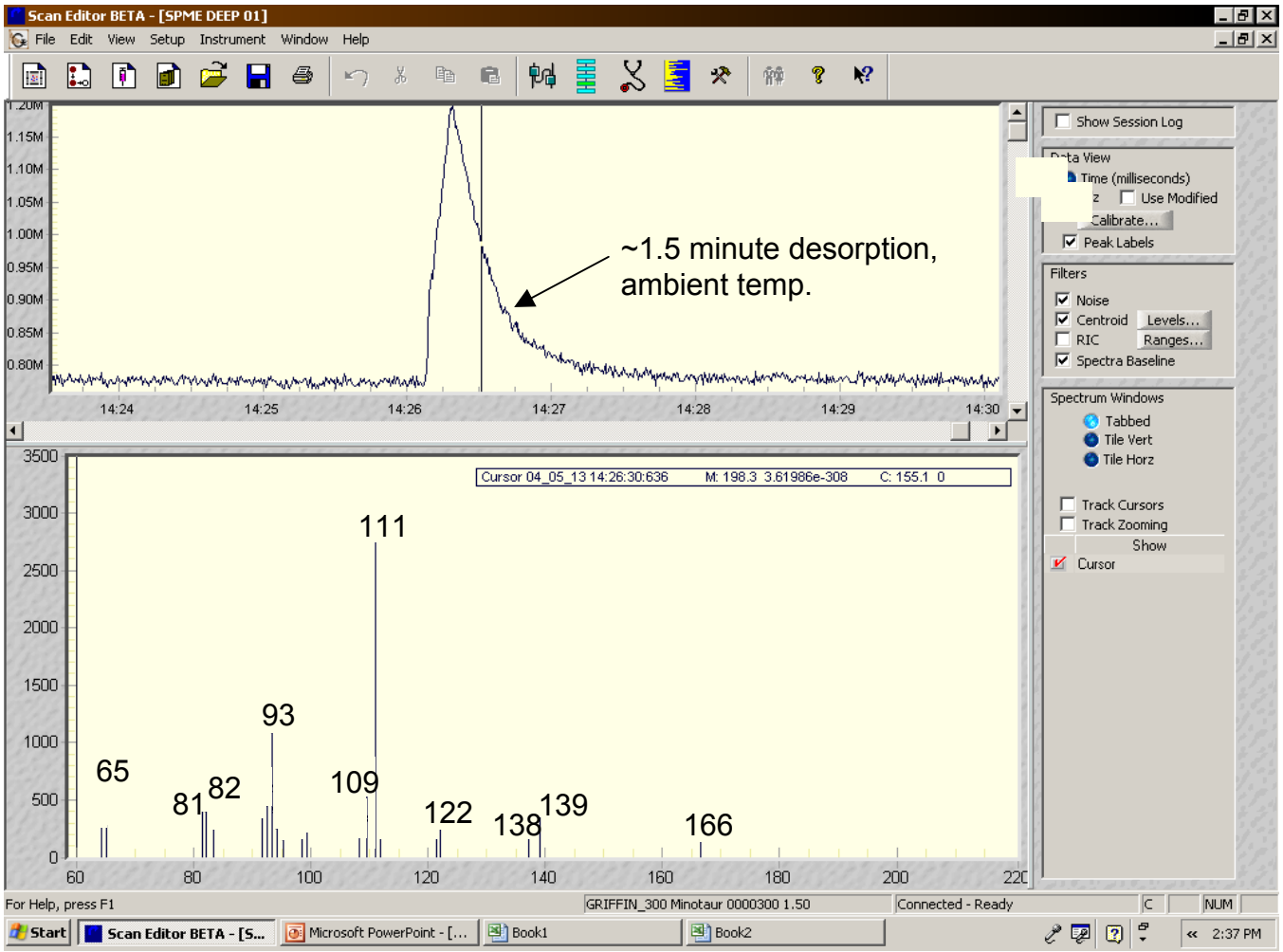
Direct SPME

Direct SPME analysis of DMMP, headspace sampling of neat compound



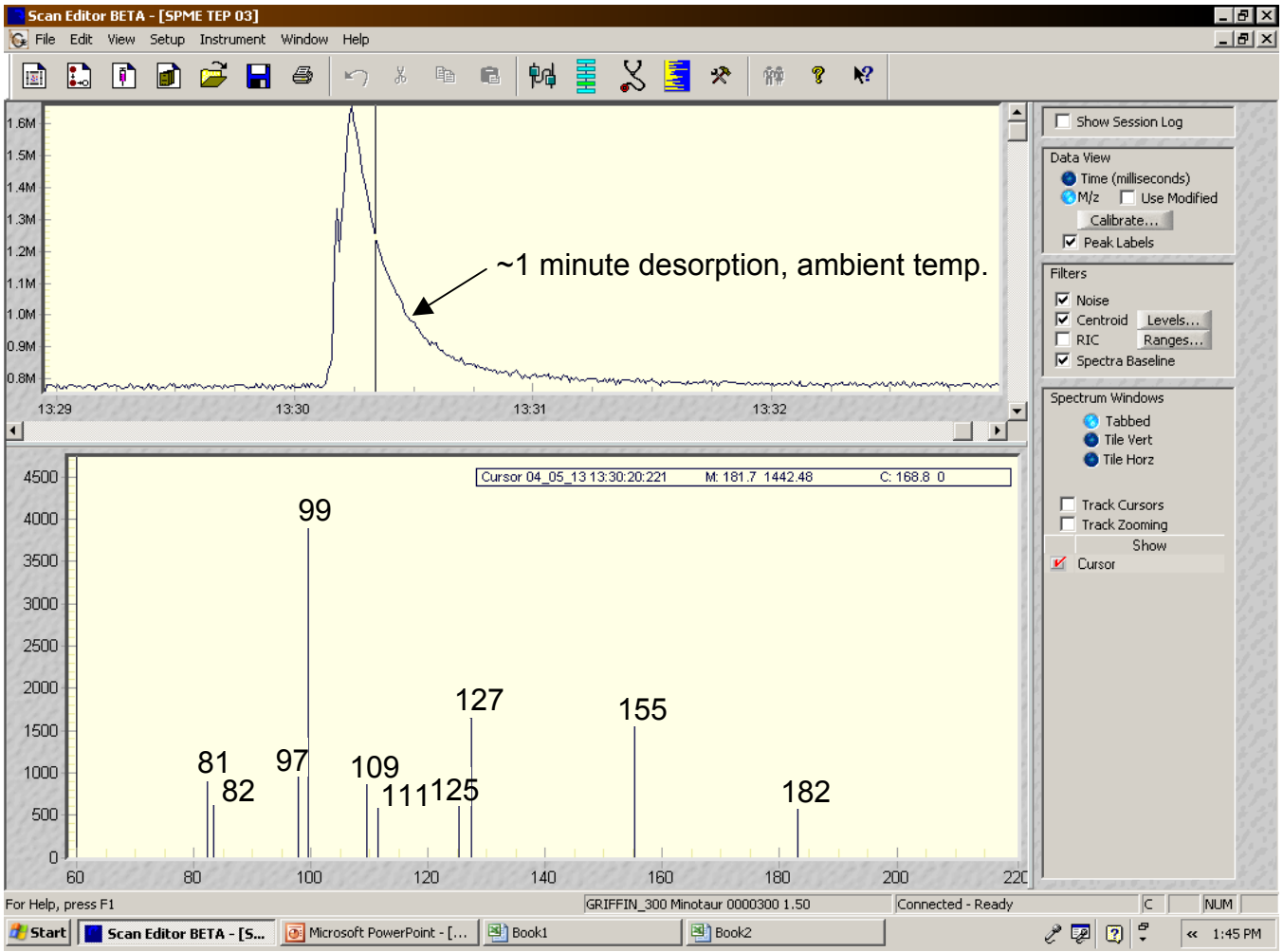
Direct SPME

Direct SPME analysis of DEEP, headspace sampling of neat compound



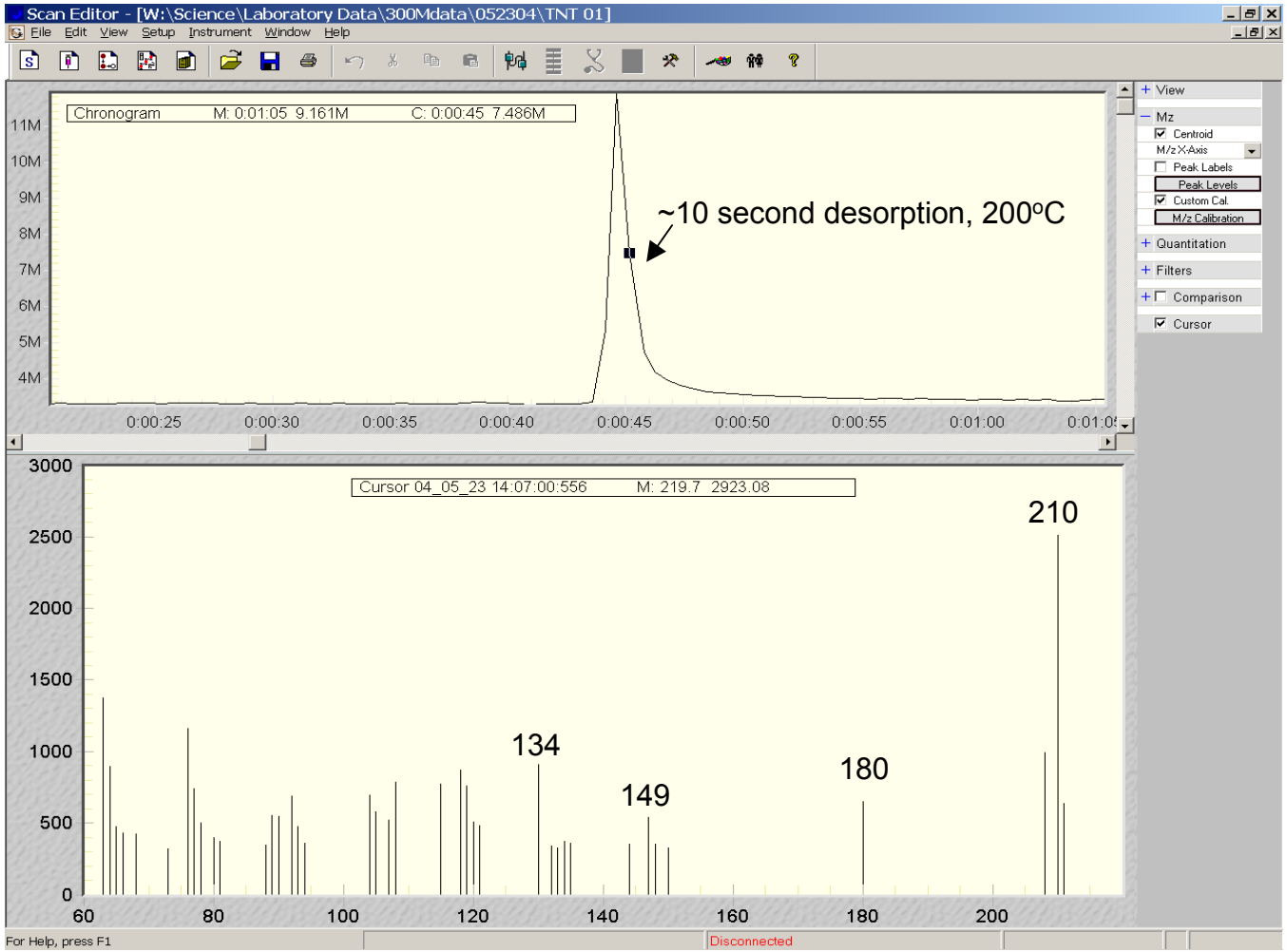
Direct SPME

Direct SPME analysis of TEP, headspace sampling of neat compound



Direct SPME

Direct SPME analysis of TNT, headspace sampling 100 ng/uL solution



Conclusions

- New GC/MS instruments based on LTM-GC and CIT mass analyzer provide:
  - Small footprint, ruggedized package for field use
  - Rapid separation of complex mixtures
  - MS analysis with good database matching (NIST and user-defined) for compound ID, MS/MS for ID confirmation
  - Quantitation to low pg (ppt-ppb) levels without preconcentration
  - Complementary sample inlets are also available

Acknowledgments

- Acknowledgements
  - Brad Rowland, US Army DPG
  - Prof. Graham Cooks, Purdue University
  - Prof. Paul Shepson, Purdue University

- The Griffin Team