From Helium to Hydrogen: GC-MS Case Study on SVOC's in water

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Helium and Profitability of Drinking Water Testing Labs

Helium to Hydrogen Migration: Practicalities

- **Hydrogen Carrier Gas:**
  - Van Deemter Curve
  - Thermo Scientific Position
  - Hydrogen Safety Points
  - Cylinders vs. Generators

- **GC-MS Tuning**
  - Baking & HC Background
  - Water Spectrum Check
  - Resolution Assurance
  - Target Tuning (DFTPP & BFBs)

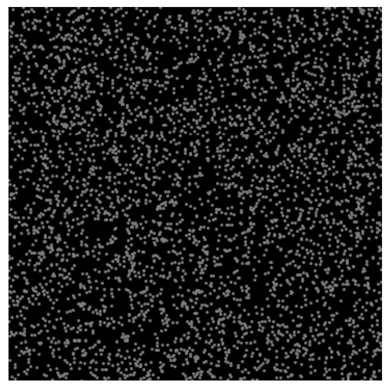
- **GC Inlet and Column**
  - Maintenance
  - Mode of Injection
  - Liner Selection
  - Column Selection

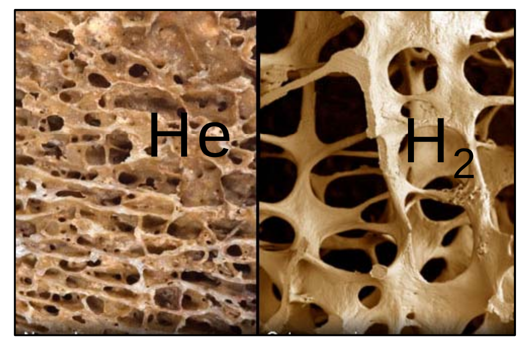
- **Performance: U.S. EPA Methods 524 & 525**
  - Linearity (measuring accuracy)
  - Sensitivity (MDLs)
  - Spectral Integrity
  - Ion Ratio Stability

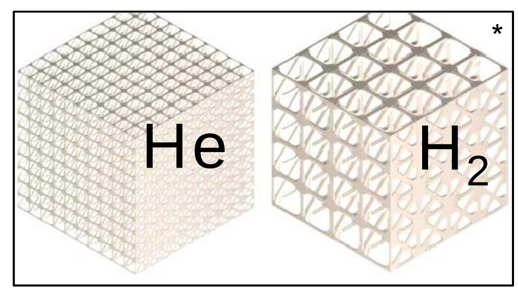
***Before* you migrate to hydrogen carrier gas on your GC-MS**

Carrier Gases: Physical Properties

Property	He	H_2	N_2	Ar
Molecular Mass, Da	4	2	28	40
Density, kg/m^3	0.18	0.09	1.25	1.78
Diffusion Coefficient, cm^2/s	0.55	0.60	0.15	0.20
Viscosity, Pa•s × 10^6	23.2	10.3	20.9	27.0
u_{opt}, cm/s	20	40	12	<10







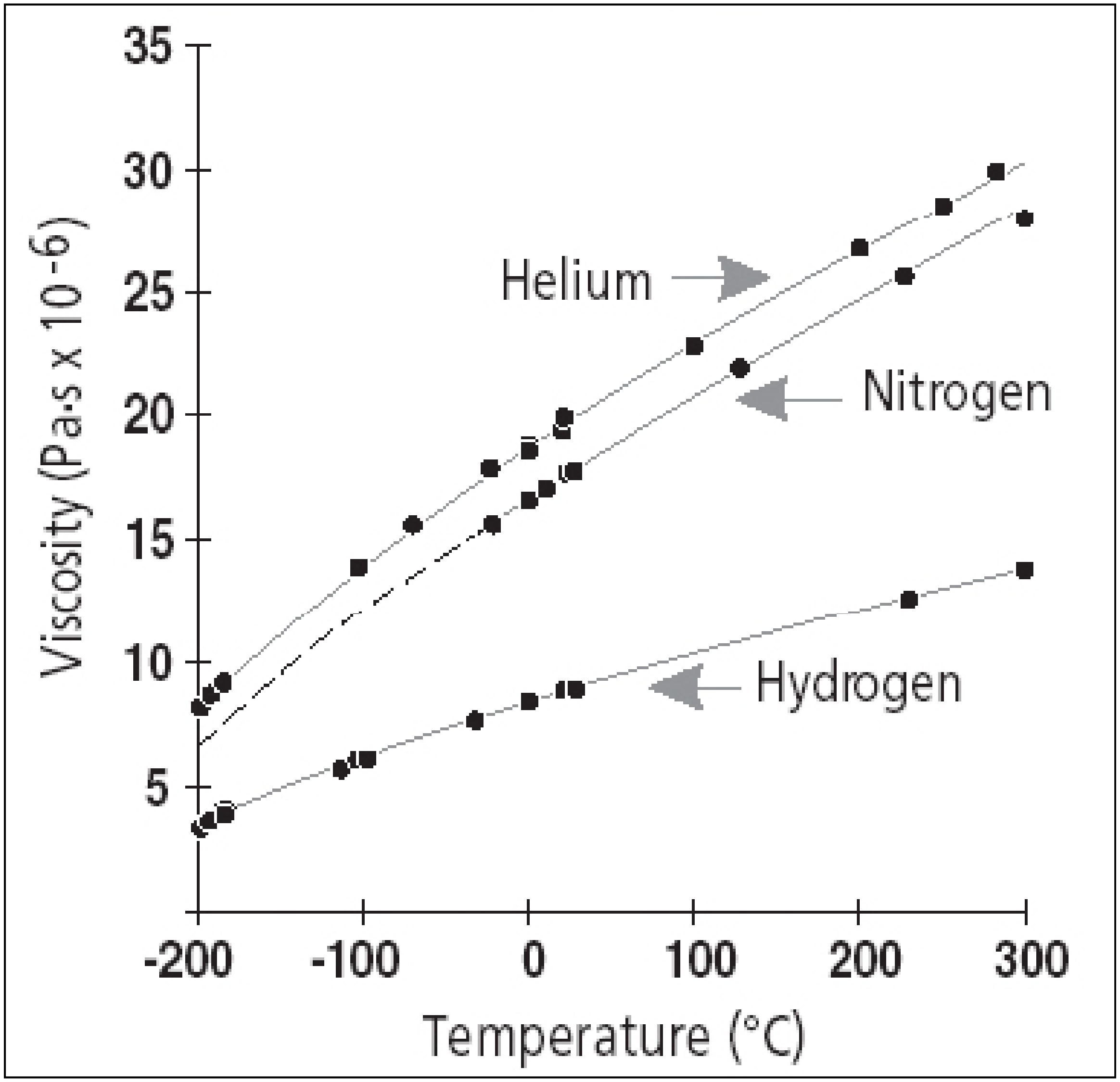
Carrier Gas **Density**: Less is Better

Diffusion Coefficient: More is Better**

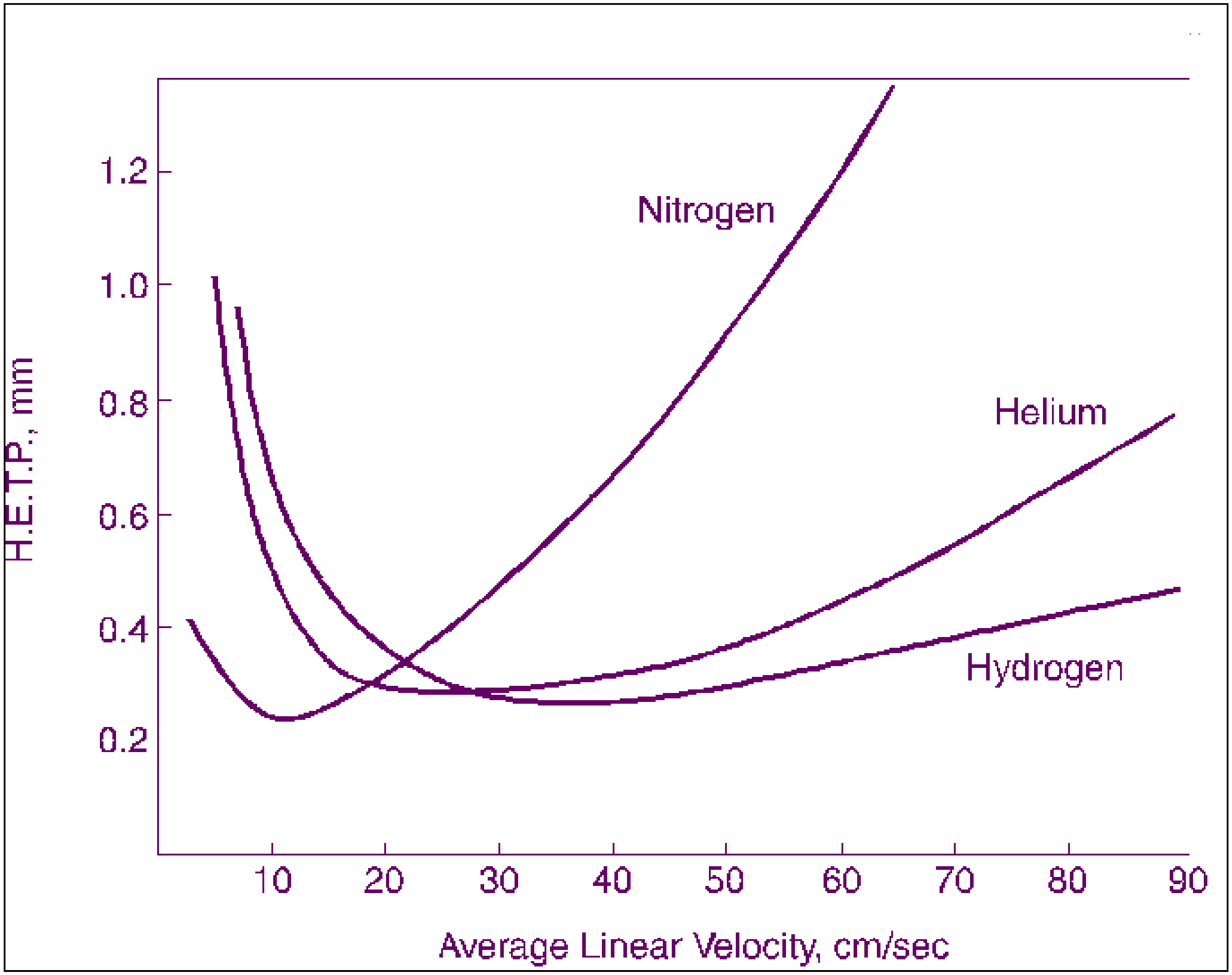
Dynamic **Viscosity** : Less is Better

* Crystal Lattice *Density Analogy* only, O.Fryazinov; A.Pasko; V.Adzhiev Computer-Aided Design 2011, 43(3) 265-277
** On the RHS of the van Demmter curve, that is.

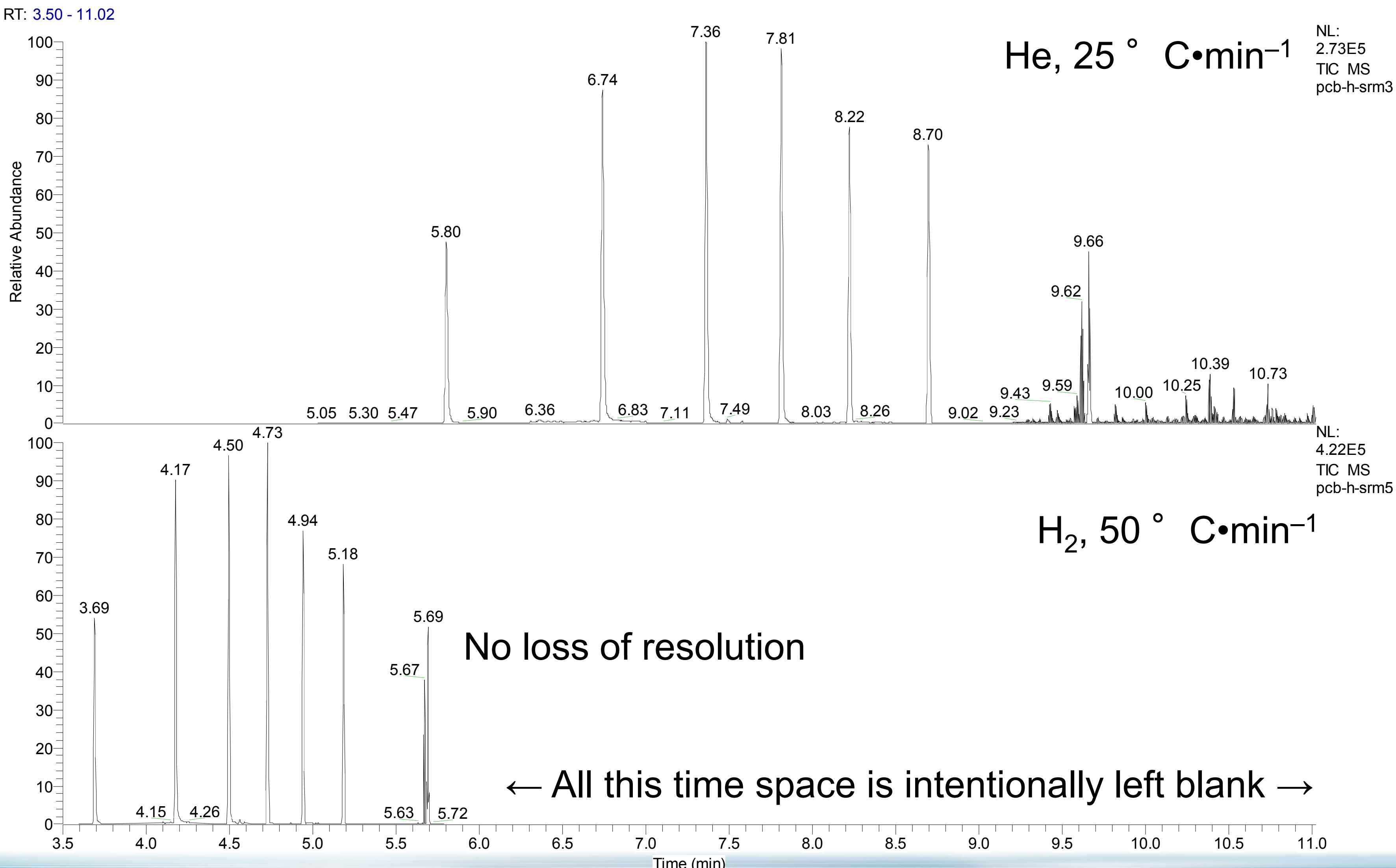
Dynamic Viscosity vs. Temperature



Carrier Gases Efficiencies: Van Deemter Plots

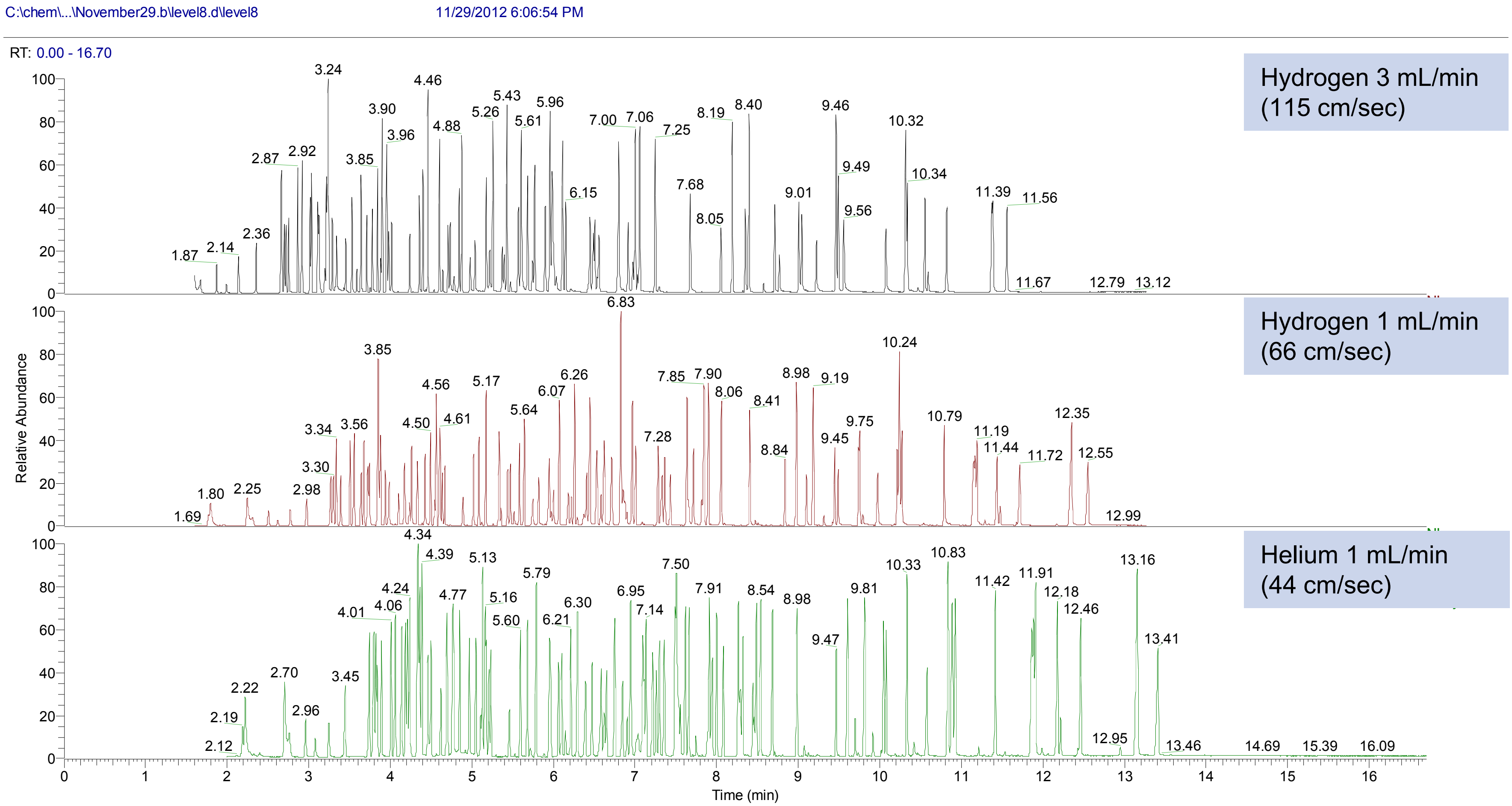


Effect of GC Oven Heating Rate: PCB Mix

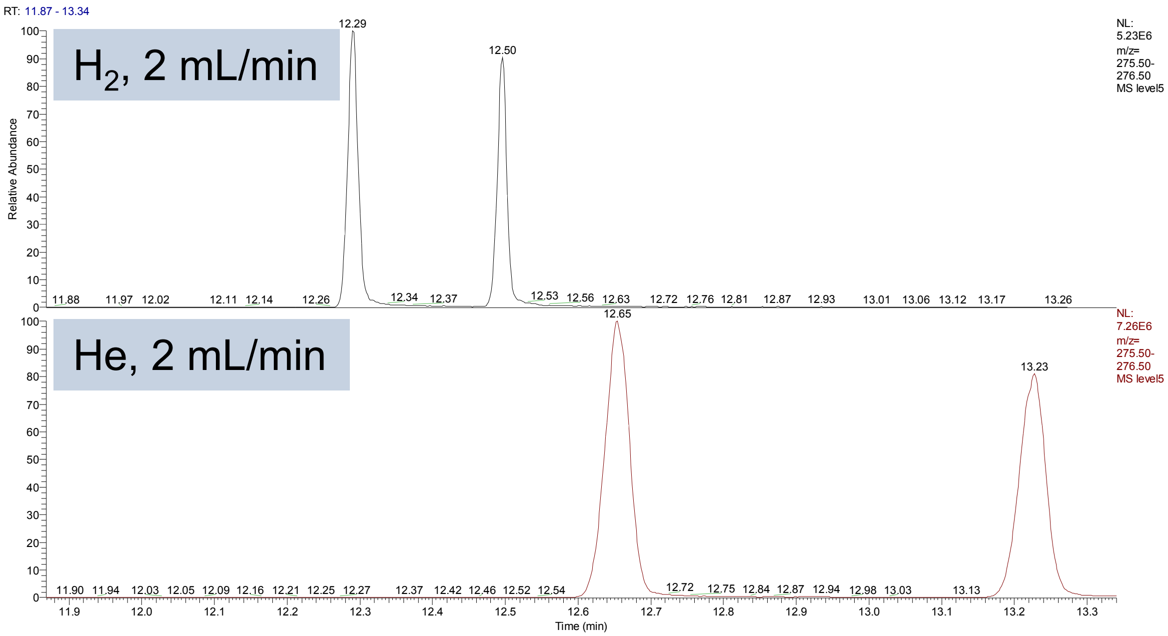


 Polychlorinated Biphenyls Mix ran by Dirk Claus, Interscience, www.is-x.be

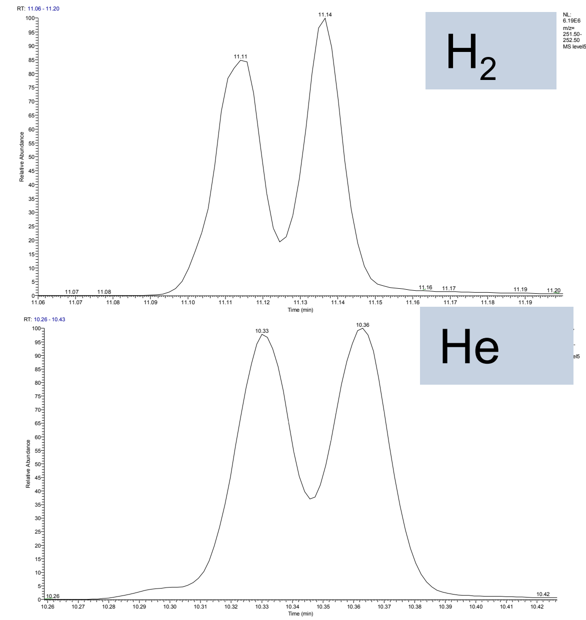
Effect of Flow Rate



Peak Shape and Critical Separations



Indeno[1,2,3-cd]pyrene & benzo[g,h,i]perylene



Benzo[b&k]fluoranthene

MINIMIZED BAND BROADENING OF PAHs

Thermo Scientific Position on Hydrogen with GC-MS

- **Hydrogen Kit**
  - Required to Run with H_2
  - Required for H_2 Specifications
  - Includes Hydrogen Sensor
  - Requires 300 L/s Turbo Pump

- **Thermo Scientific™ Hydrogen Systems**
  - ISQ™ Single Quadrupole GC-MS
  - TRACE™ 1300 Series GC
  - TSQ™ 8000 GC-MS/MS
  - Explosion Tested & Safety Certified

- **Upgrades in the Field**
  - All existing ISQ GC-MS Systems are either already H_2 capable or are upgradeable with 300 L/s Turbo and H_2 Kit
  - IF you decide to use your existing Thermo Scientific™ DSQ™, DSQ II & ITQ GC-MS with H_2, you do so at your own risk (no Safety Certification)

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ISQ GC-MS Hydrogen Carrier Installation Specifications

- **ISQ Single Quadrupole GC-MS**
  - Guaranteed installation specifications with hydrogen carrier gas
  - Choice of either He or H_2 on installation spec sign-off
  - PCI/NCI same S/N!

Standard Installation Specifications*

Mode / Concentration	He	H_2
In **EI** mode, 1 µL of 1 pg/µL octafluoronaphthalene (OFN) will produce the following minimum signal to noise for m/z 272 when scanning from 50 – 300 u:	1,500:1	100:1
In **PCI** mode, 1 µL of 100 pg/µL benzophenone will produce the following minimum signal to noise for m/z 183 when scanning from 80 – 230 u using methane reagent gas:	300:1	300:1
In **NCI** mode, 2 µL of 100 fg/µL of OFN will produce the following minimum signal to noise for m/z 272 when scanning from 50 – 300 u using methane reagent gas:	600:1	600:1

* Requires Hydrogen Kit for H_2.

ISQ GC-MS Technical Brief - AB52360

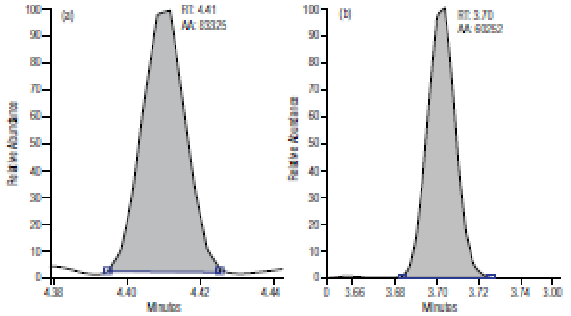

Figure 1. Injection of 1 pg/µL OFN in EI mode results in S/N of at least 100:1 for hydrogen (a.) vs. 600:1 for helium (b.).

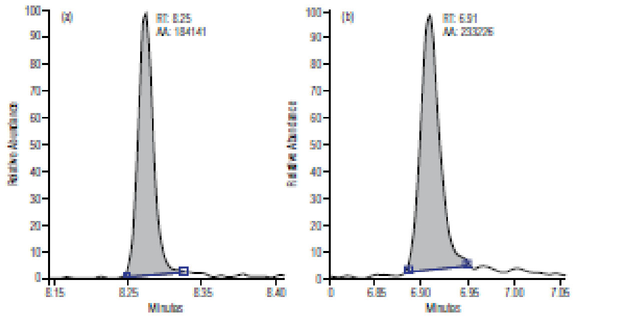

Figure 2. Injection of 100 pg/µL benzophenone in CI mode results in S/N of at least 300:1 for both hydrogen (a.) and helium (b.).

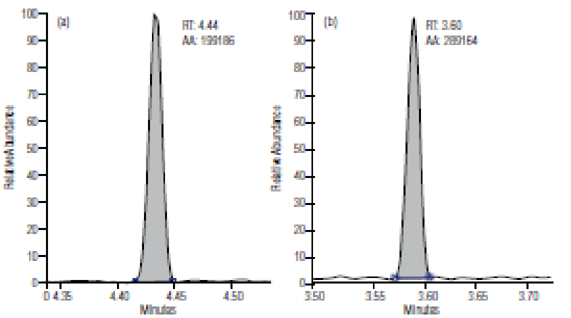

Figure 3. Injection of 200 fg/µL OFN in CI mode results in S/N of at least 600:1 for both hydrogen (a.) and helium (b.).

Application Brief 52360

Moving from Helium to Hydrogen as a Carrier Gas for the Thermo Scientific ISQ GC-MS System

Introduction

The purpose of this technical brief is to demonstrate the use of hydrogen as a carrier gas for GC-MS rather than the traditional use of helium. The world-wide shortage of helium has prompted many labs to investigate the feasibility of adopting the use of hydrogen as a carrier gas. The availability of hydrogen generators provides a safe, steady, and renewable supply of hydrogen for proper instrument operation. This brief summarizes what is needed to transition your ISQ™ GC-MS system to use hydrogen as a carrier gas. For more details and a discussion of how changing the carrier gas affects chromatography, please refer to the full application note (AN52362: Moving from Helium to Hydrogen as a Carrier Gas for GC-MS Applications: Practical Considerations).

Experimental Conditions

The ISQ GC-MS system used in this study was paired up with a Thermo Scientific TRACE 1300 GC equipped with a split/splitless injector and Thermo Scientific Xcalibur control software. The EI and CI modes with hydrogen carrier gas were both tested using a Thermo Scientific TG-SQC test column 30 m × 0.25 mm id × 0.25 um. (The comparative data with helium as carrier gas are shown for TG-SQC test column 15 m × 0.25 mm id × 0.25 µm).

The GC-MS parameters were set to typical operating conditions with only some slight changes to accommodate for the carrier gas differences. A Thermo Scientific hydrogen sensor was installed and set to alarm if a hydrogen leak occurred inside the GC oven. A cylinder of UHP hydrogen was used in this case, but a UHP hydrogen generator is recommended for safety reasons. For GC-MS, it is best to use a generator with a palladium diffusion dryer, otherwise the gas stream will have excessive water vapor. The maximum amount of moisture must be below 1 ppm. Examples of hydrogen generators may include, but are not limited to the following: Parker Balston® H2PD Series (http://www.labgasgenerators.com/hydrogengenerators) and F-DGS® WM-H2 Series (http://f-dgs.com/uk/hydrogen_uk.htm.)*

* Thermo Fisher Scientific does not endorse, guarantee, or warranty the use, suitability, or performance of any third party equipment. The models mentioned are examples only.

The system sensitivity was such that a 1 µL injection of 1 pg/µL standard yielded a signal-to-noise (S/N) ratio of at least 100:1 (Figure 1) for octafluoronaphthalene (OFN) in EI mode. Chemical Ionization (CI) yielded S/N of at least 300:1 (Figure 2) and 600:1 (Figure 3) for 100 pg benzophenone and 200 fg OFN respectively.

Results and Discussion

The sensitivity in the EI mode with the hydrogen carrier is 3–4 times lower than helium due to the increase in background noise. The CI mode, however, does not produce this effect and yields typical S/N values that are comparable to those observed with helium. Depending on the application used, the oven ramp and/or column dimensions will have to be adjusted to account for the lower viscosity of hydrogen.

Conclusion

It has been demonstrated that the ISQ GC-MS can be safely (taking all necessary safety precautions) converted to use hydrogen as carrier gas rather than helium. Depending on the complexity of the application, the GC parameters will require adjustment to accommodate the differences between the two gases. For a list of recommended part numbers for converting the system to use hydrogen and a detailed technical discussion, including van Deemter curve plots and comparisons of various chromatograms and conditions, please refer to the corresponding application note (AN52362: Moving from Helium to Hydrogen as a Carrier Gas for GC-MS Applications: Practical Considerations).

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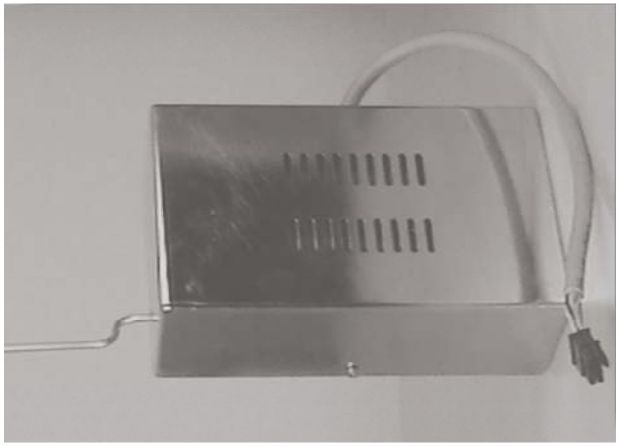
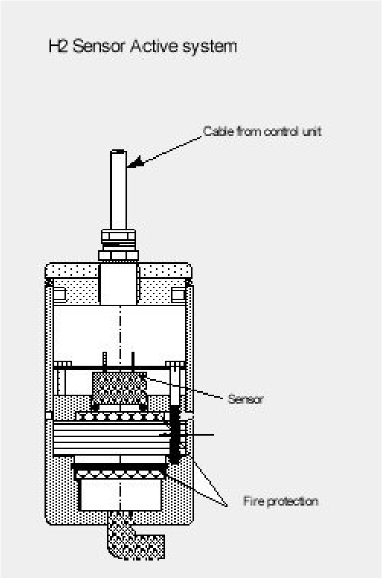
Hydrogen Gas Safety

- **Safety Precautions**
  - **Hydrogen sensor in GC oven**
  - **Venting hydrogen**
  - **The risk is minimal**
  - **Can be estimated & prevented**





- **Safety Facts on ISQ GC-MS and TSQ 8000 GC-MS**
  - **Explosion Tested & Explosion Certified**
  - **No detaching/flying pieces**
    ***even* IF hydrogen inside goes *boom*…**
  - **It is really hard in practice to reach explosive limits of hydrogen in the lab space …**
  - **… and the GC oven is monitored by the sensor**

Hydrogen Purity Specifications

- GC-MS Hydrogen Generator
- O_2 < 0.01 ppm
- H_2O < 1 ppm
- THC N/A

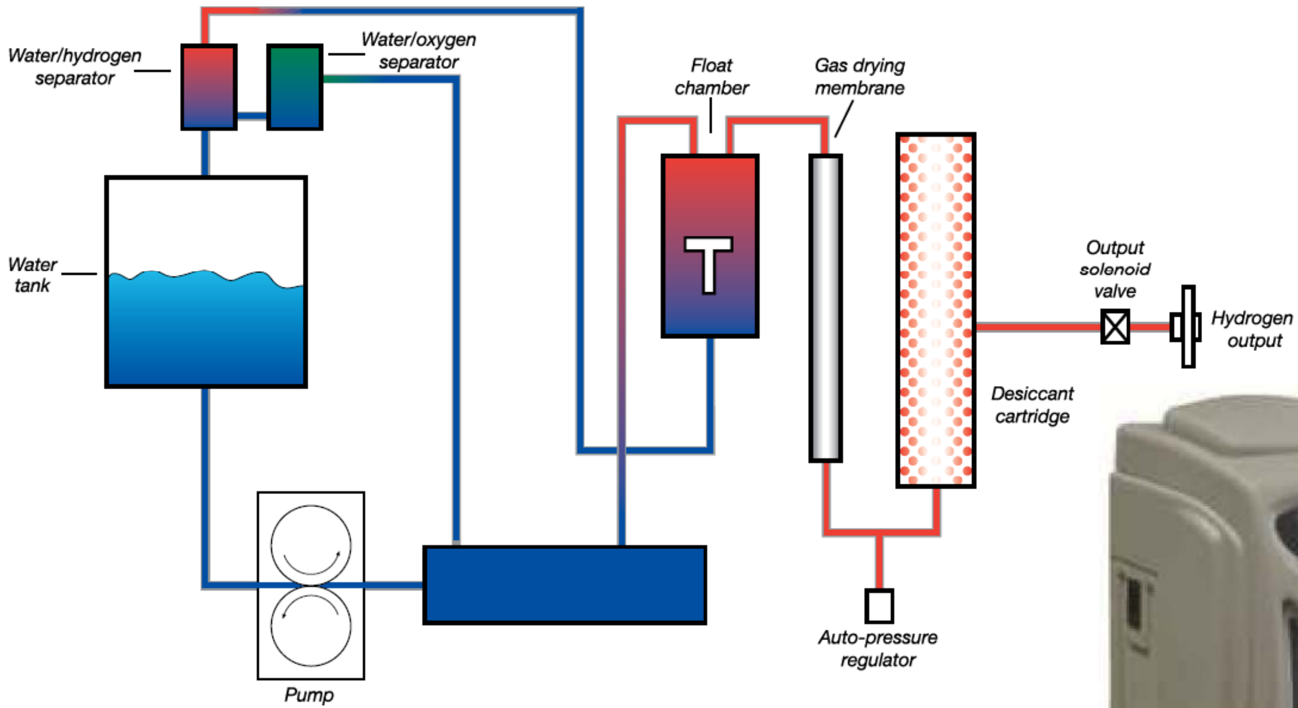
- Hydrogen Grade 5.0 UHP
- O_2 < 1 ppm
- H_2O < 3 ppm
- THC < 0.1 ppm

- Hydrogen FID Fuel Grade
- O_2 < 1 ppm
- H_2O < 3 ppm
- THC < 0.5 ppm

- Hydrogen Grade 6.0 UHP
- O_2 < 0.1 ppm
- H_2O < 0.5 ppm
- THC < 0.1 ppm

Hydrogen Generators: Choose the Best

How the generator works



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Understanding Hydrogen Plumbing

- Gas Filter, triple stage

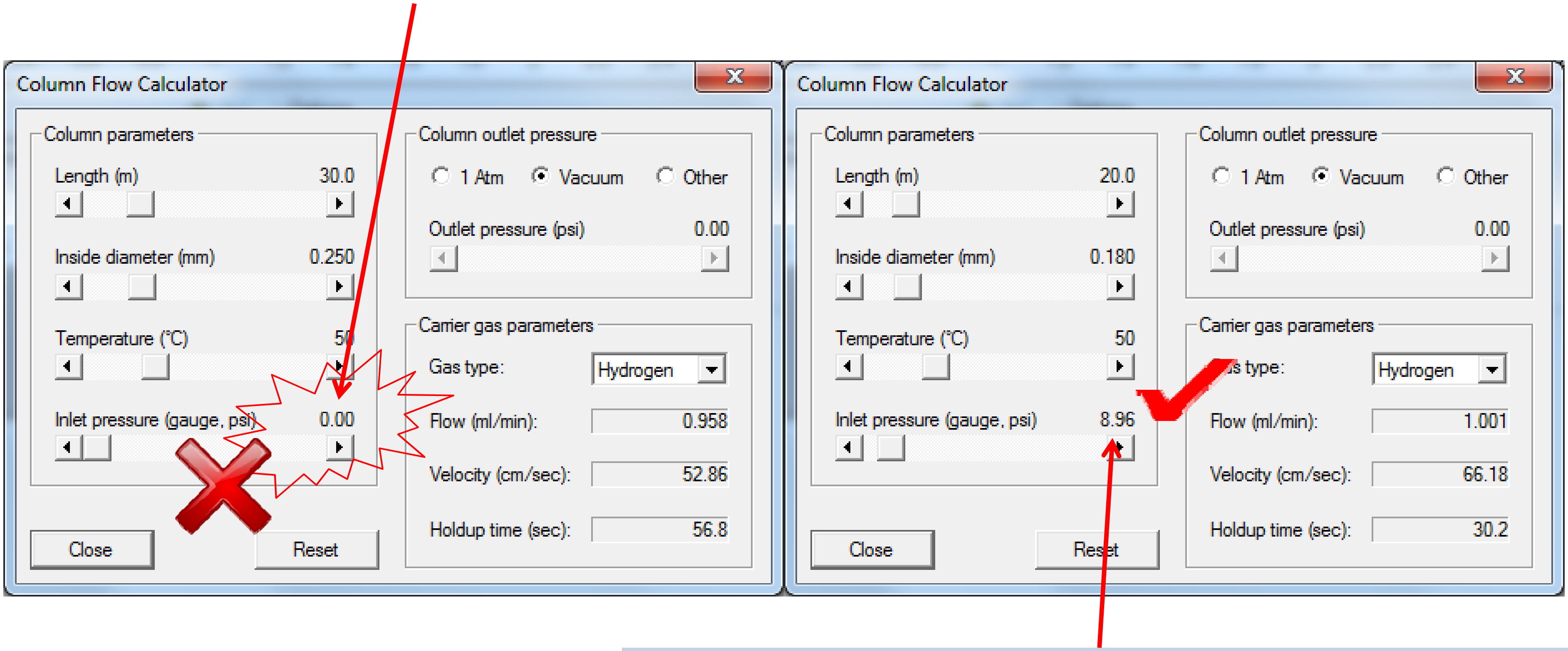
- Tubing
- Stainless steel preferred
- 1/8” pre-cleaned
- New tubing is preferred over used with He

- Filter – only need with tanks
- For removal of water, oxygen and hydrocarbons

What you need to know about plumbing up hydrogen to your GC

Limits on Hydrogen Flow and Column Selection

1 mL/min hydrogen on 0.25 mm × 30 m column



1 mL/min hydrogen on 0.18 mm × 20 meter column

GC-MS Tuning

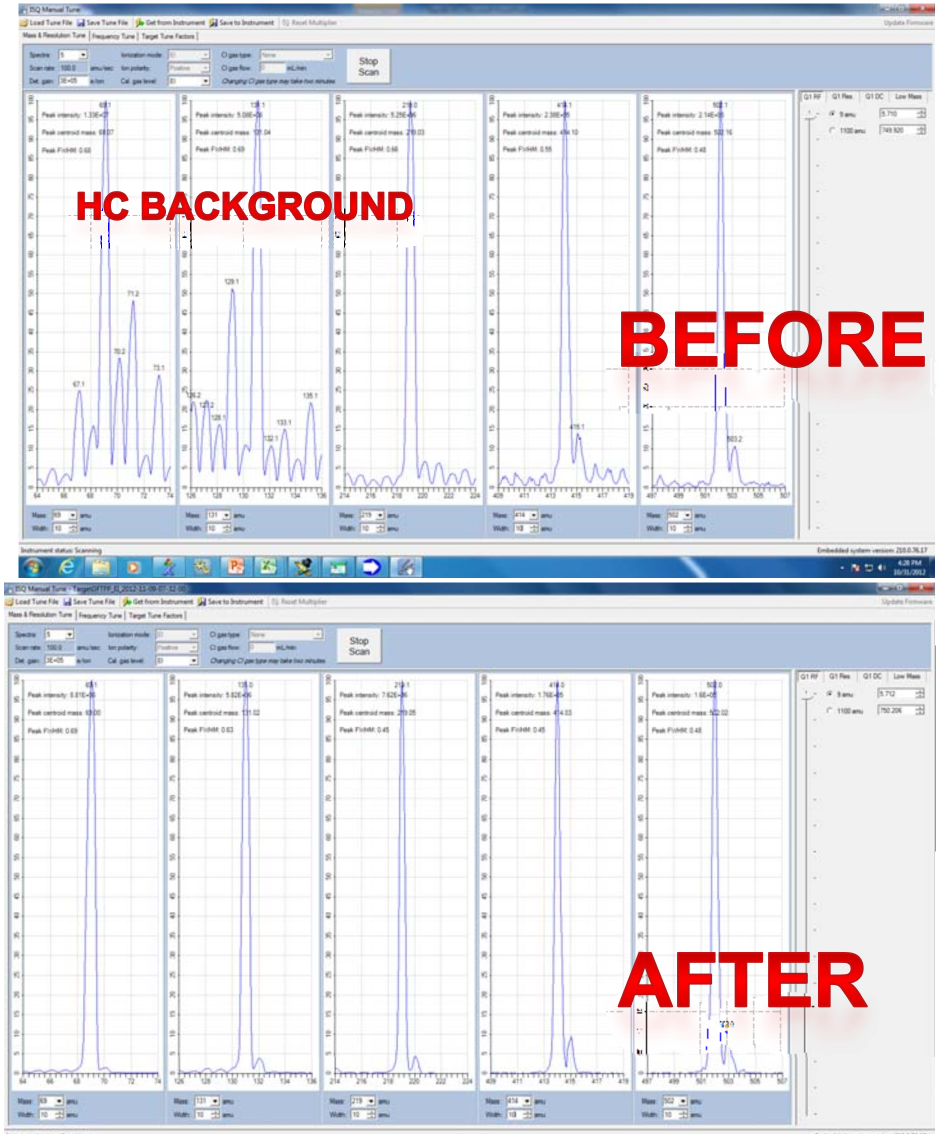
- Dealing with HC background ions and water
- Bake out at 350° C with hydrogen flow at 4 mL/min

- Mass Resolution: good

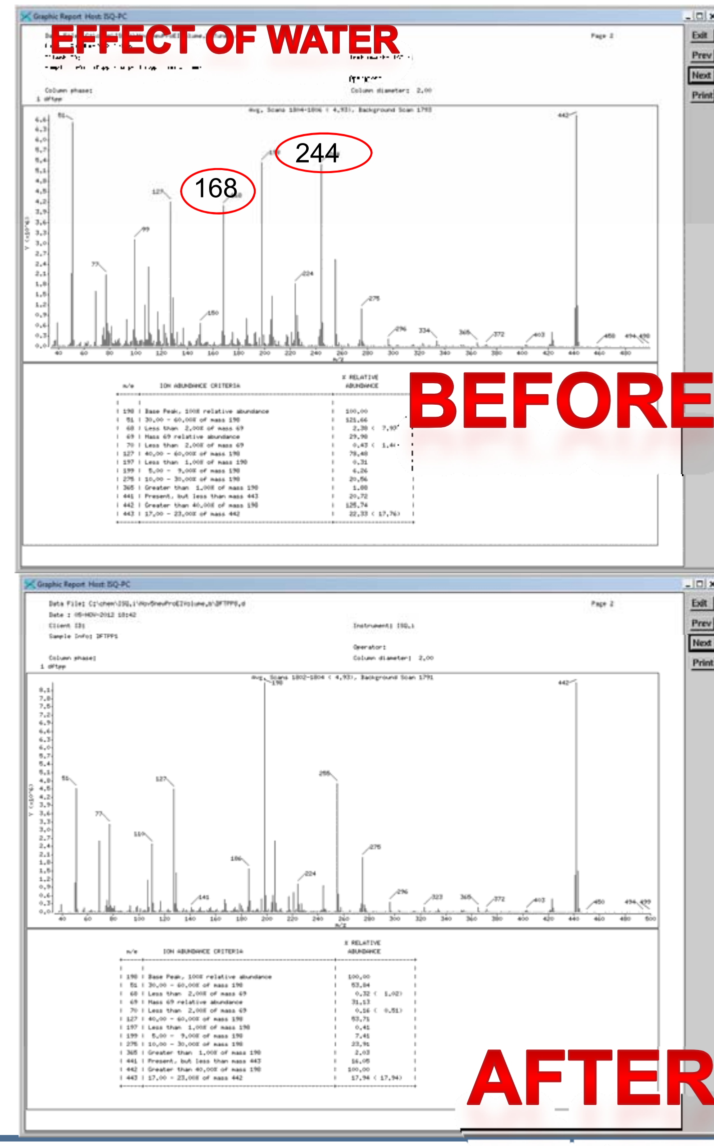
- Shows similar tune to that with helium

- Target Tuning for DFTPP and BFB
- Special tuning sequence to meet EPA tuning criteria

Bake-out at 350 ˚C with H_2 @ 4 mL/min for 1 hr

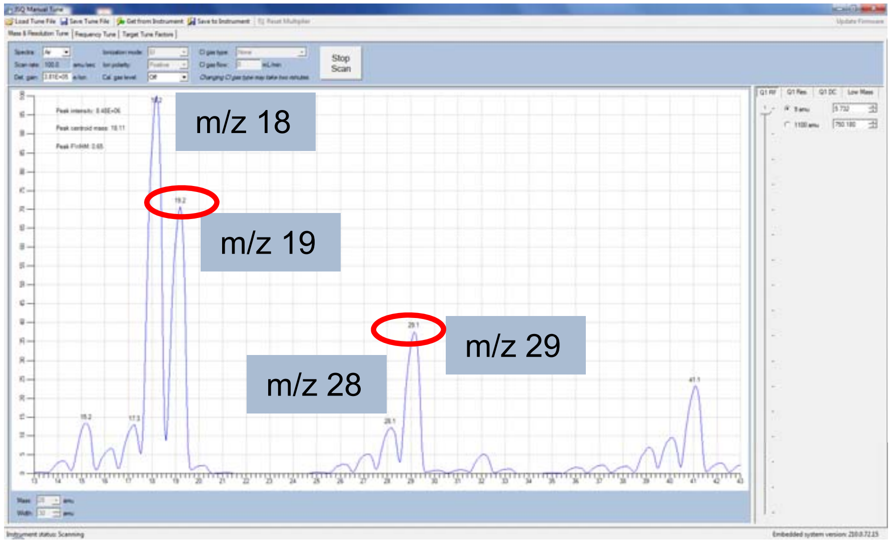


FC-43 n-$(C_4F_9)_3N$

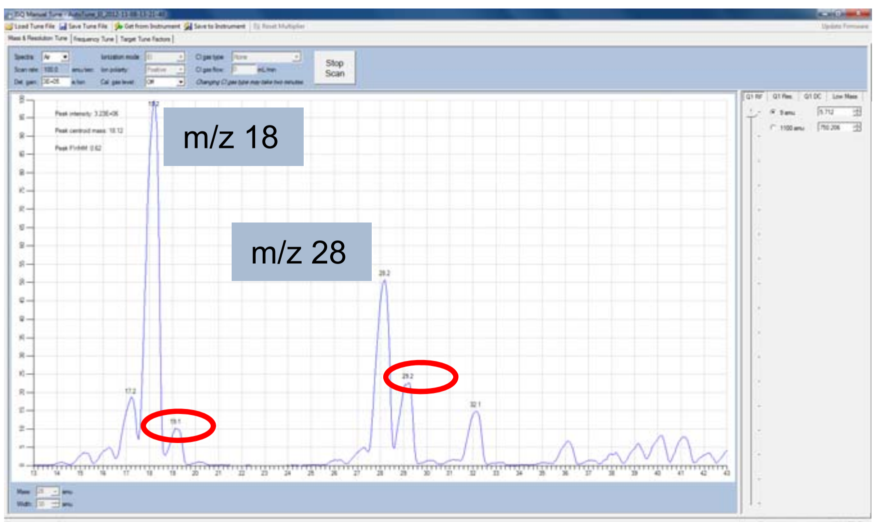


DFTPP

Air Water Spectra: *m/z* 29 and *m/z* 19

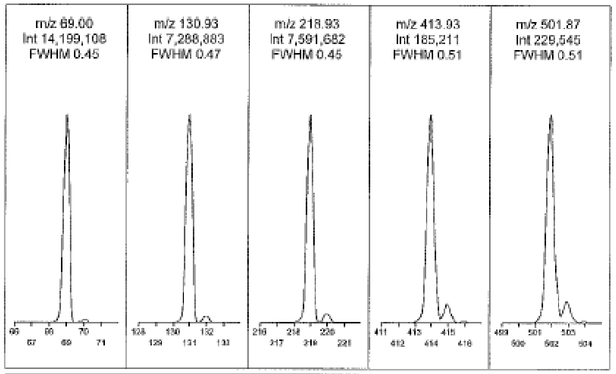


BEFORE



AFTER

Tuning Report: He vs. H_2



Polarity	Positive
Electron Lens Voltage	15 V
Electron Energy	70 eV
Emission Current	50 µA
Ion Guide Frequency	1618.6
Q1 Frequency	1080.9
Multiplier Voltage	1754.8 V
Detector Gain	3.0 × 10 ^ 5
MS Transfer Line	310.0 °C
Filament Selection	1
Ion Source Temperature	250.0 °C
Foreline Pressure	75 mTorr
Ion Gauge Pressure	1.3 × 10 ^ -5 Torr

Full Scan Spectrum Not Acquired

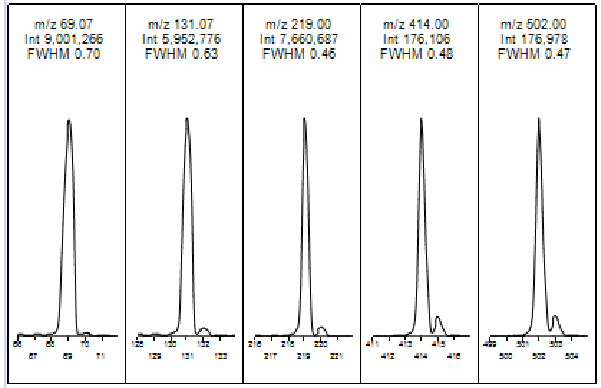
Mass	Theory	Abundance	Relative Abundance	Isotope Mass	Theoretical Isotope Mass	Isotope Abundance	Isotope Ratio
69.03	69.00	4,973,356	100.00	69.98	70.00	73,396	1.48
131.00	130.99	2,607,640	52.43	131.95	131.99	113,566	4.36
219.01	218.99	2,591,609	52.11	219.95	219.99	150,777	5.82
413.98	413.98	79,576	1.60	415.07	414.98	8,789	11.04
502.06	501.97	120,545	2.42	503.00	502.97	9,527	7.90

Air/ Water Check:			
H2O	(%18/69)	20.12	Leak check not performed
N2	(%28/69)	58.70	
O2	(%32/69)	4.24	
CO2	(%44/69)	2.20	
N2 / H2O	(%28/18)	2.92	

Target Tune Factors:

Mass	50	69	131	219	414	502
Scale	1	0.49	0.41	0.29	0.12	0.13

Signature: ____________________



EI Ion Source Type	EI
Polarity	Positive
Electron Lens Voltage	15 V
Electron Energy	70 eV
Emission Current	50 µA
Ion Guide Frequency	1599.5
Q1 Frequency	1097.1
Multiplier Voltage	1431.1 V
Detector Gain	3.0 × 10 ^ 5
MS Transfer Line	310.0 °C
Filament Selection	1
Ion Source Temperature	325.0 °C
Foreline Pressure	51 mTorr
Ion Gauge Pressure	9.6 × 10 ^ -6 Torr

Full Scan Spectrum Not Acquired

Mass	Theory	Abundance	Relative Abundance	Isotope Mass	Theoretical Isotope Mass	Isotope Abundance	Isotope Ratio
68.98	69.00	6,210,659	100.00	70.07	70.00	78,224	1.26
130.99	130.99	3,716,974	59.85	132.03	131.99	133,679	3.60
219.04	218.99	3,676,688	59.20	220.06	219.99	157,409	4.28
414.00	413.98	90,746	1.46	415.02	414.98	7,855	8.66
502.00	501.97	92,223	1.48	503.04	502.97	8,481	9.20

Air/ Water Check:			
H2O	(%18/69)	50.06	Leak check not performed
N2	(%28/69)	22.70	
O2	(%32/69)	6.34	
CO2	(%44/69)	5.42	
N2 / H2O	(%28/18)	0.45	

Target Tune Factors:

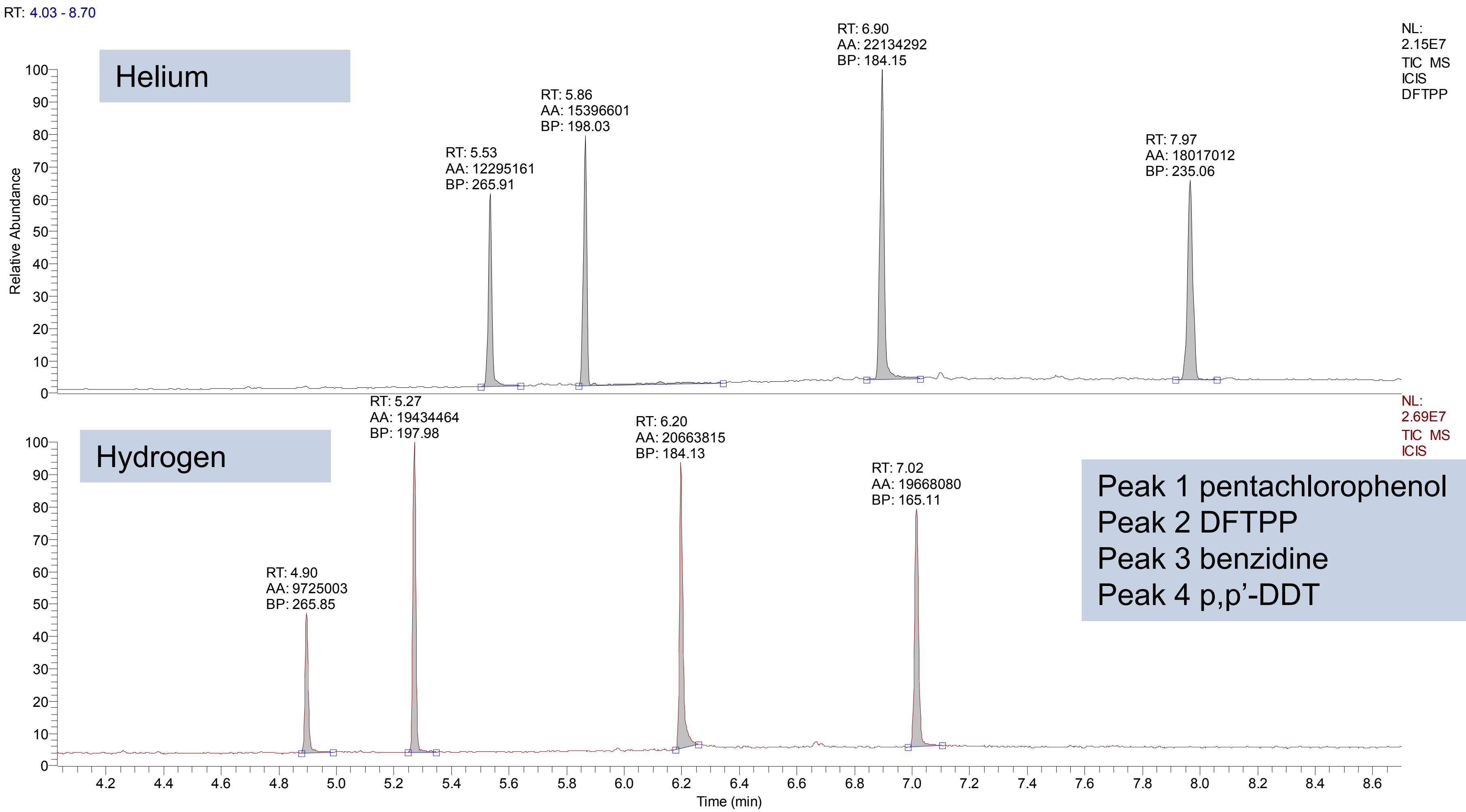
Mass	50	69	131	219	414	502
Scale	1	0.56	0.82	0.91	0.42	0.44

Signature: ____________________

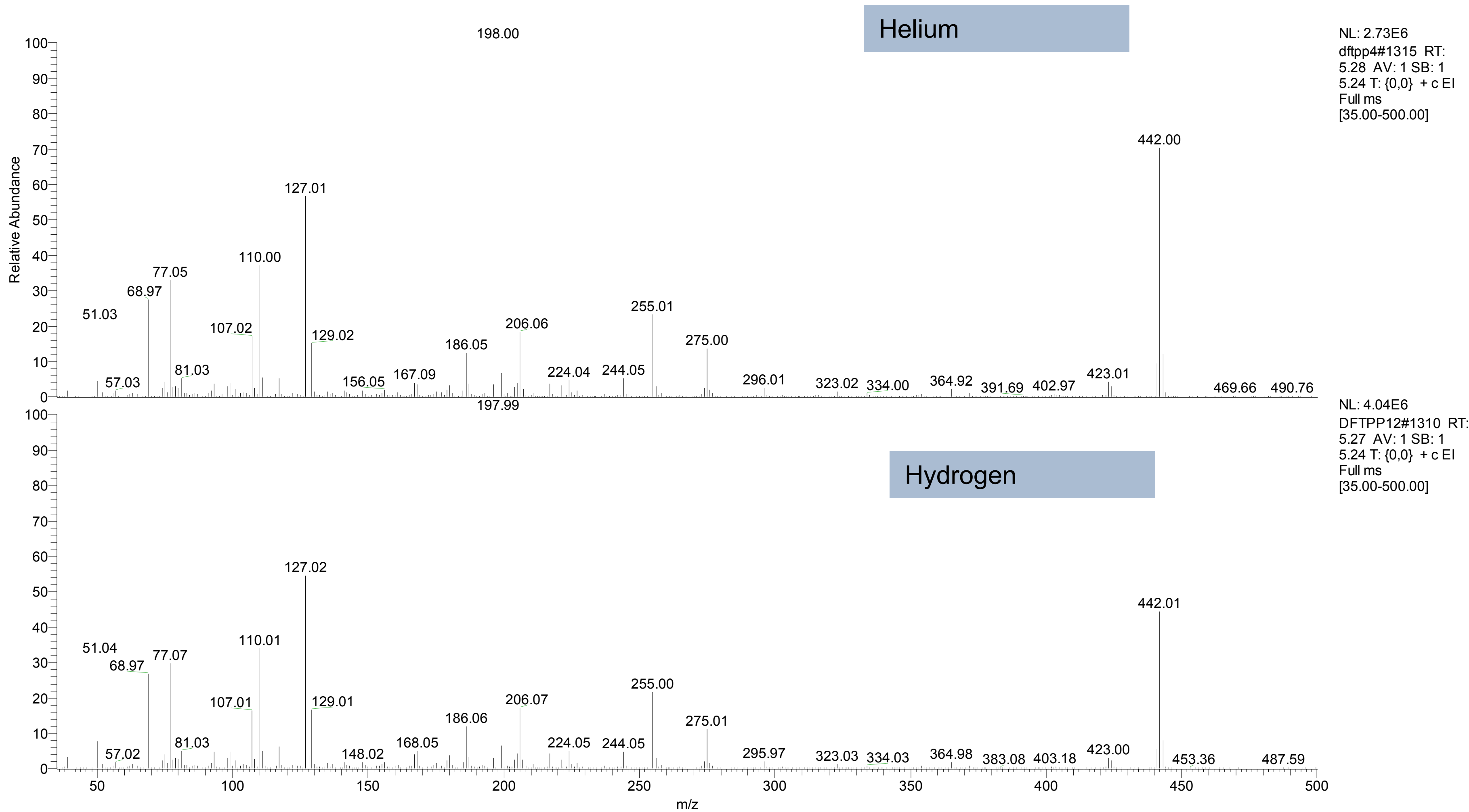
He

H_2

EPA Method 8270 Performance Mix (DFTPP)



DFTPP (Decafluorotriphenylphosphine) Spectrum



EPA Method 8270

- **EPA Method 8270**
  - Direct 1 µL liquid injection
  - Calibration (1 – 200 ppm)
  - More than 120 compounds
  - Surrogates = 40 ppm
  - Internal Standards = 40 ppm

- **Goals**
  - Improve peak shape and resolution
  - Improve method runtime (<15 minutes)

Semi-volatile Organics

Method Parameters: EPA Method 8270D

- **Standards**
  - Prepared in ethyl acetate
  - Calibration curve (1.0, 2.0, 5.0, 10, 50, 100, 200 ug/mL)
- **Inlet**
  - S/SL: 325° C
  - Split Mode: 15:1
- **Carrier: 1 mL/min H_2**
- **Column**
  - TG-5 SilMS 20m x 0.18mm x 0.36 µm

- **Oven:**

50°	1 min.	30°/min
150°	0 min	20°/min
200°	0 min	30°/min
300°	0 min	20°/min
350°	0.5 min	

- **MS**

Source	325°
EC	15 uamps
Full Scan	35-500
Scan speed	4,650 amu/ sec

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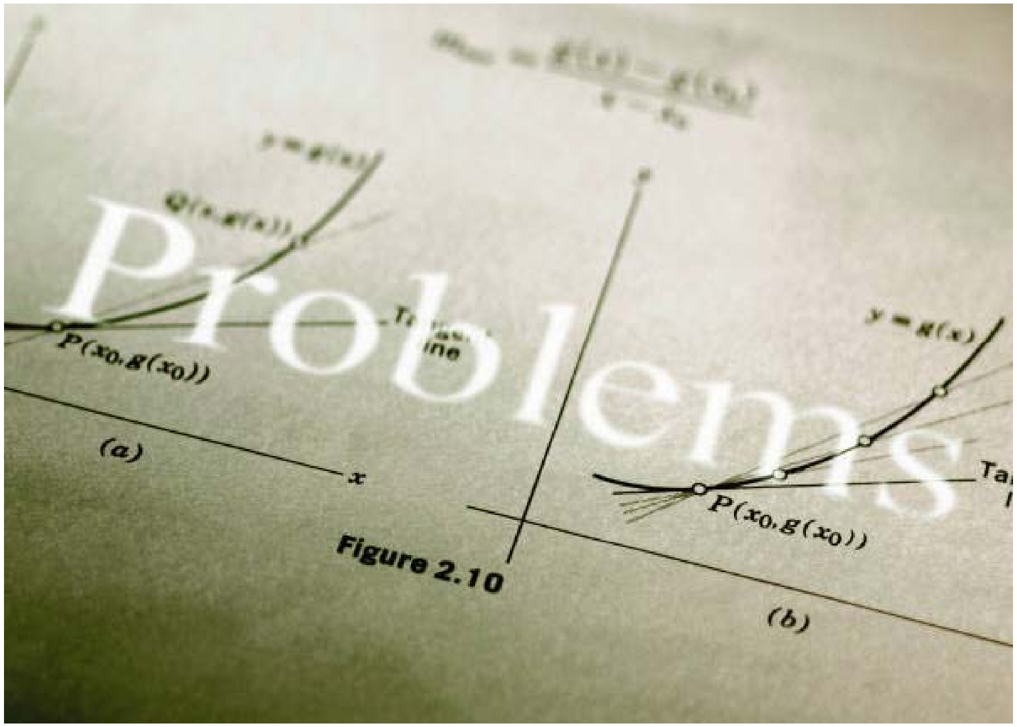
EPA Method Criteria

- **Tuning**
  - EPA Method 525/8270: DFTPP
  - EPA Method 524: BFB

- **Calibration**
  - Establish working range for each method (measurement of accuracy)
  - Method Detection Limits (MDLs)



Target DFTPP H_2 Tuning Report in Target Software

Graphic Report Host: ISQ-PC

Exit
Prev
Next
Print

Data File: C:\chem\ISQ.i\November16hy.b\dftpp5.d — Page 2

Date : 16-NOV-2012 18:18

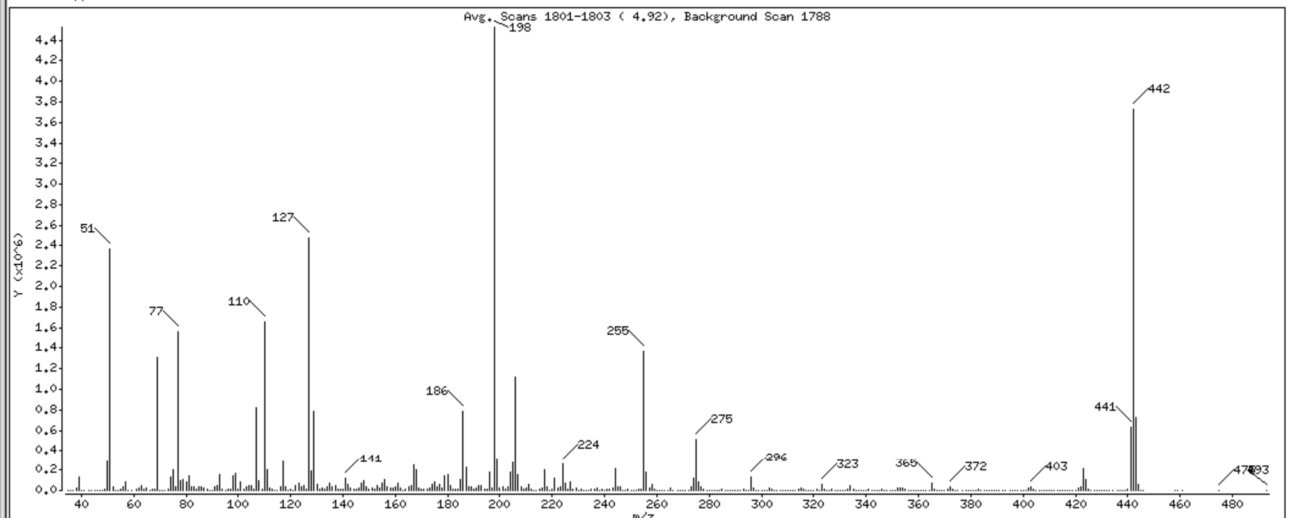
Client ID: — Instrument: ISQ.i

Sample Info: dftpp

Operator:

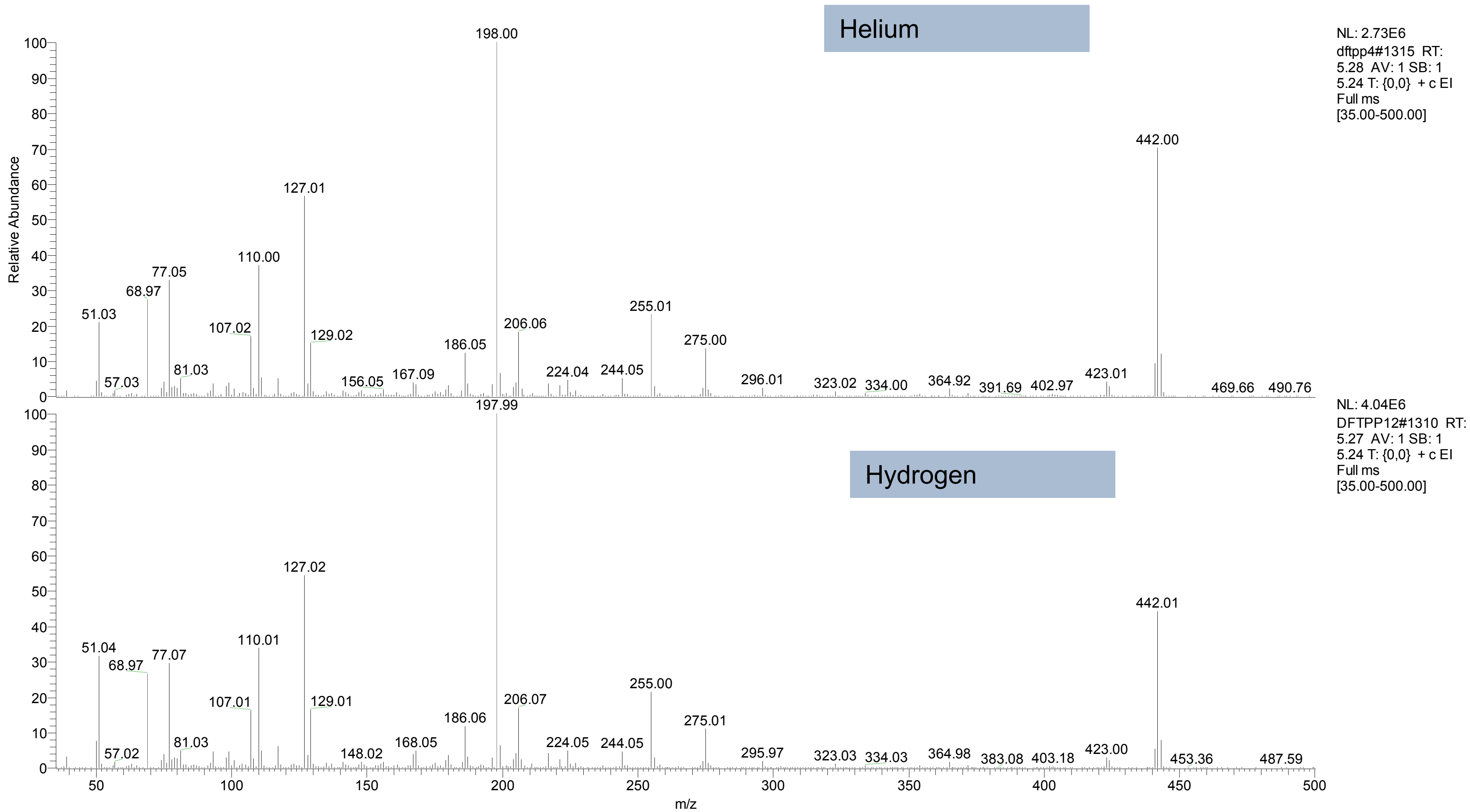
Column phase: — Column diameter: 2.00

1 dftpp

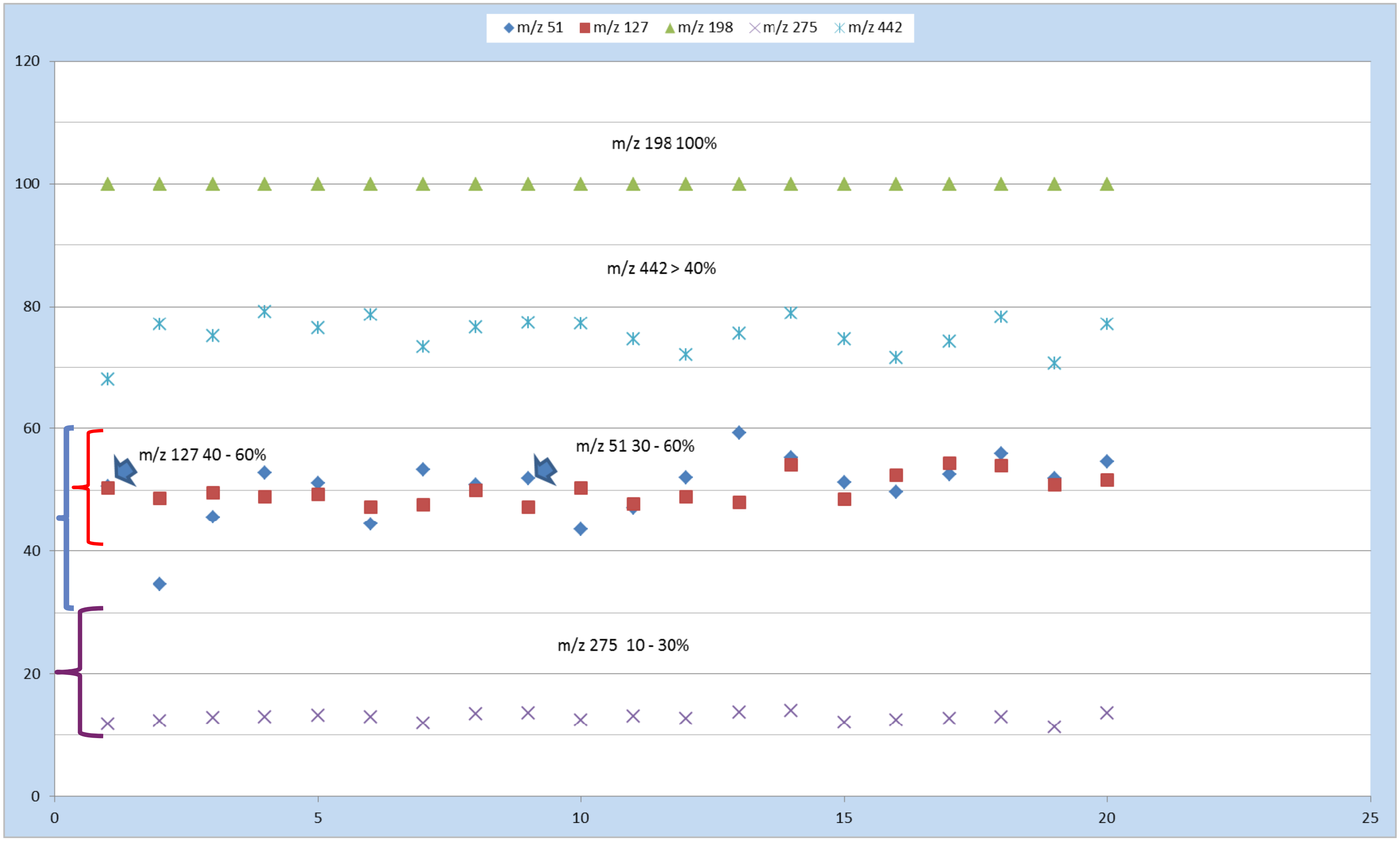


m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
198	Base Peak, 100% relative abundance	100.00
51	30.00 - 60.00% of mass 198	52.31
68	Less than 2.00% of mass 69	0.18 (0.63)
69	Mass 69 relative abundance	29.03
70	Less than 2.00% of mass 69	0.11 (0.39)
127	40.00 - 60.00% of mass 198	54.66
197	Less than 1.00% of mass 198	0.44
199	5.00 - 9.00% of mass 198	7.16
275	10.00 - 30.00% of mass 198	11.37
365	Greater than 1.00% of mass 198	1.53
441	Present, but less than mass 443	13.97
442	Greater than 40.00% of mass 198	82.44
443	17.00 - 23.00% of mass 442	15.97 (19.38)

DFTPP (Decafluorotriphenylphosphine) Spectrum



Ion Ratio Stability for DFTPP



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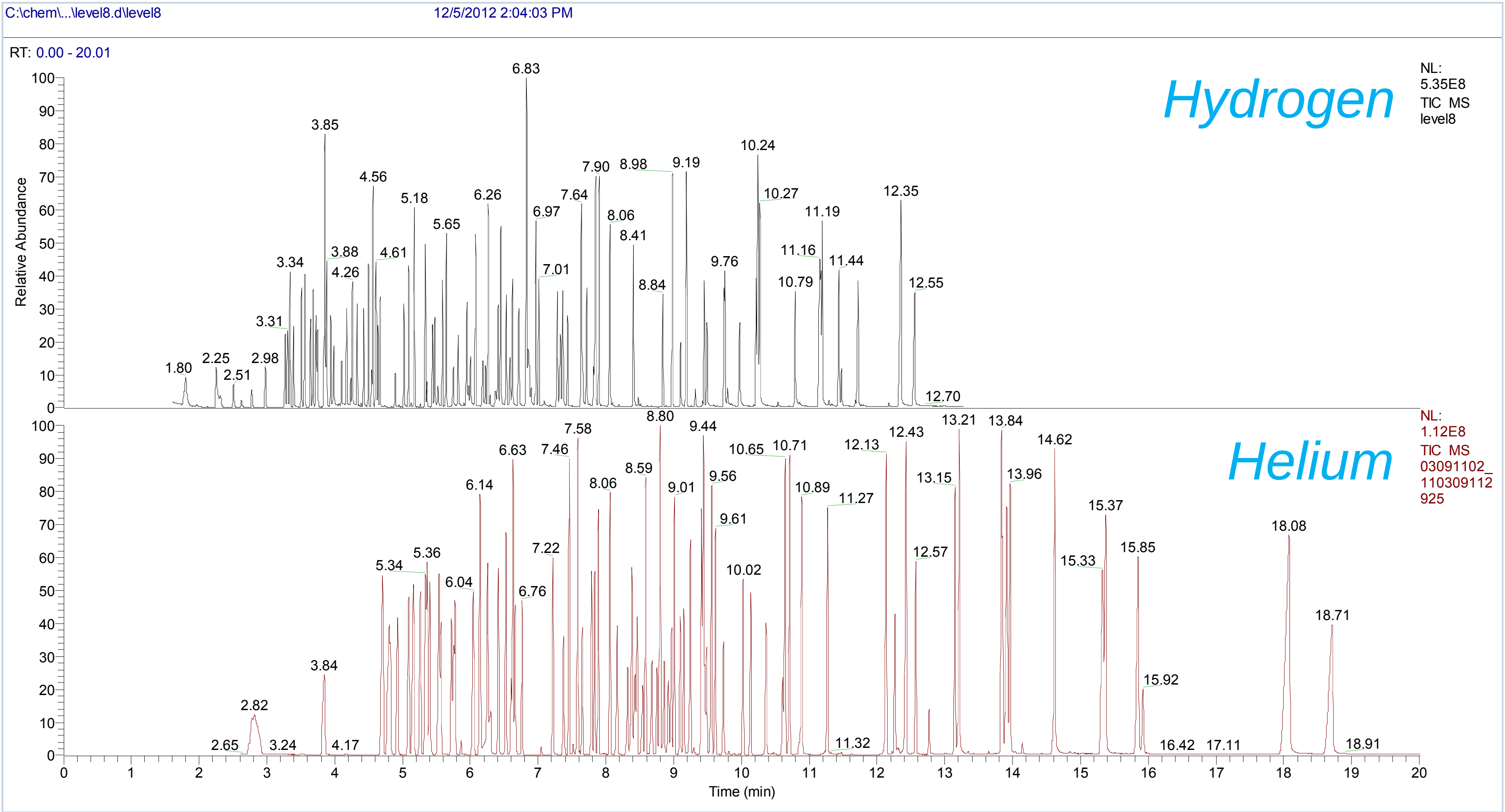
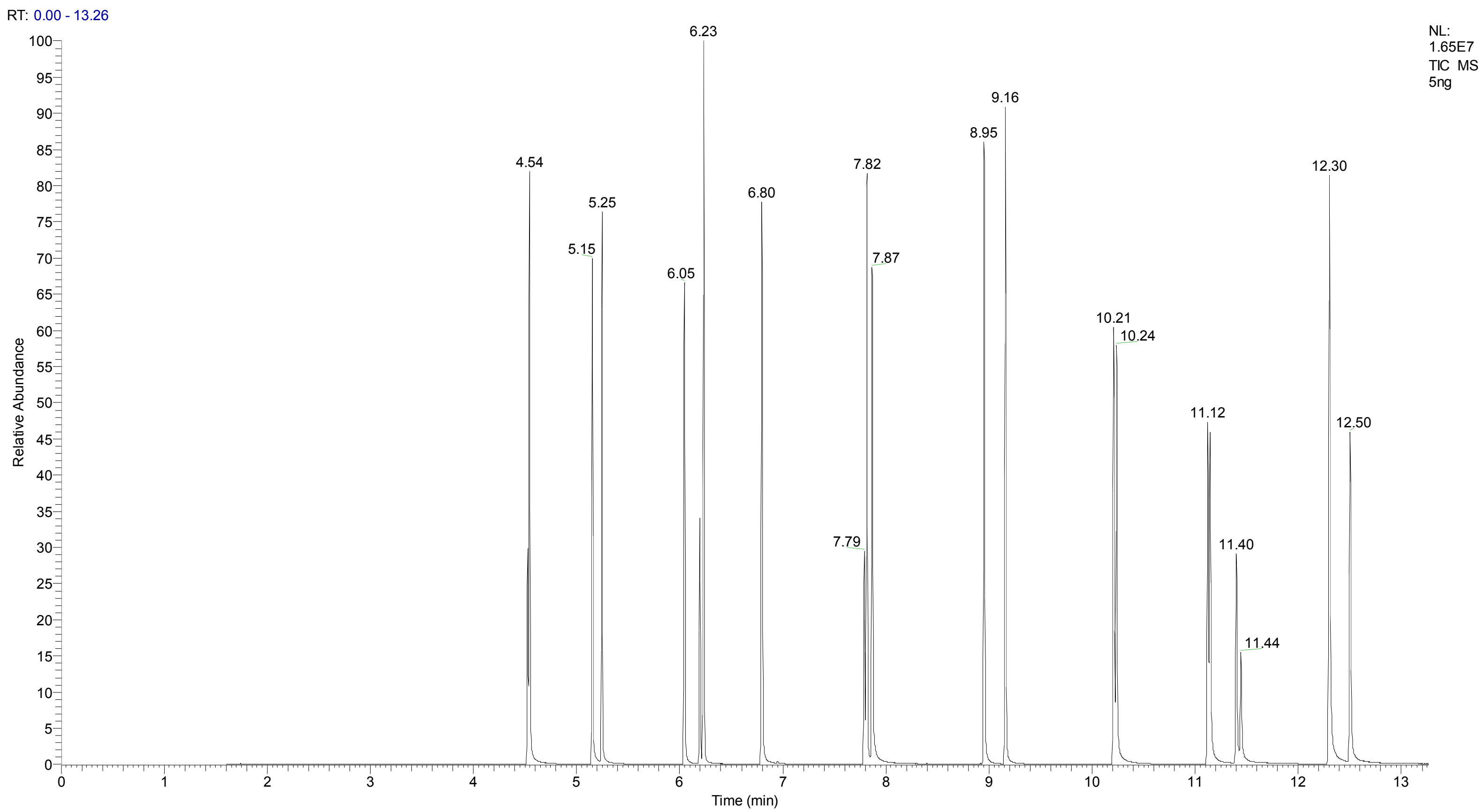
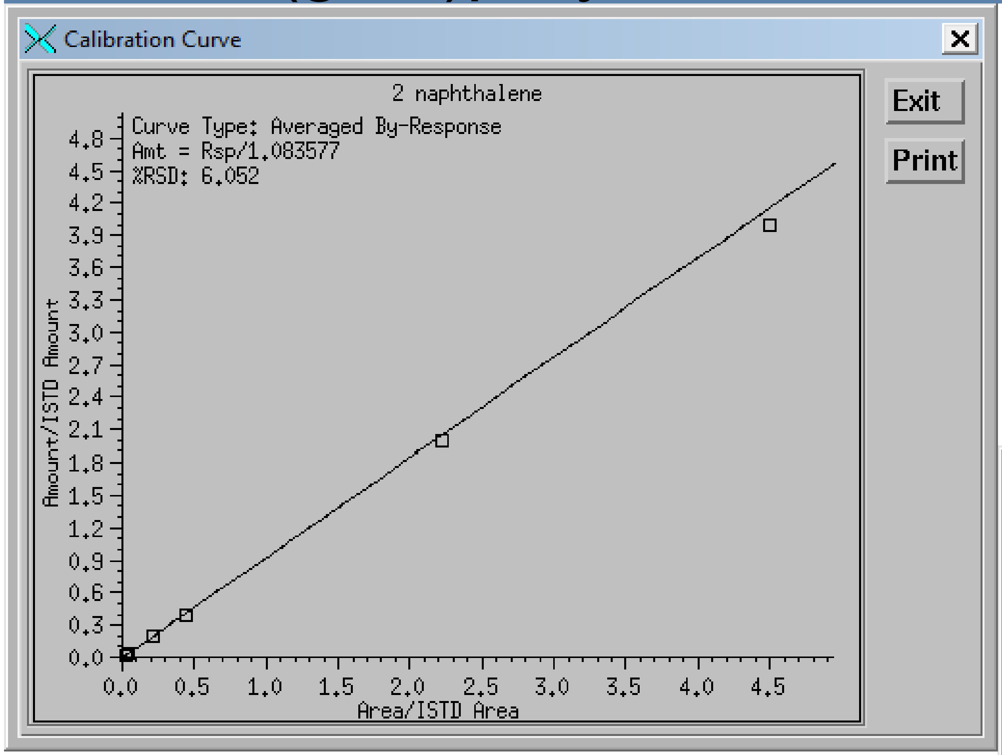
8270: Comparison of Hydrogen to Helium Run Times

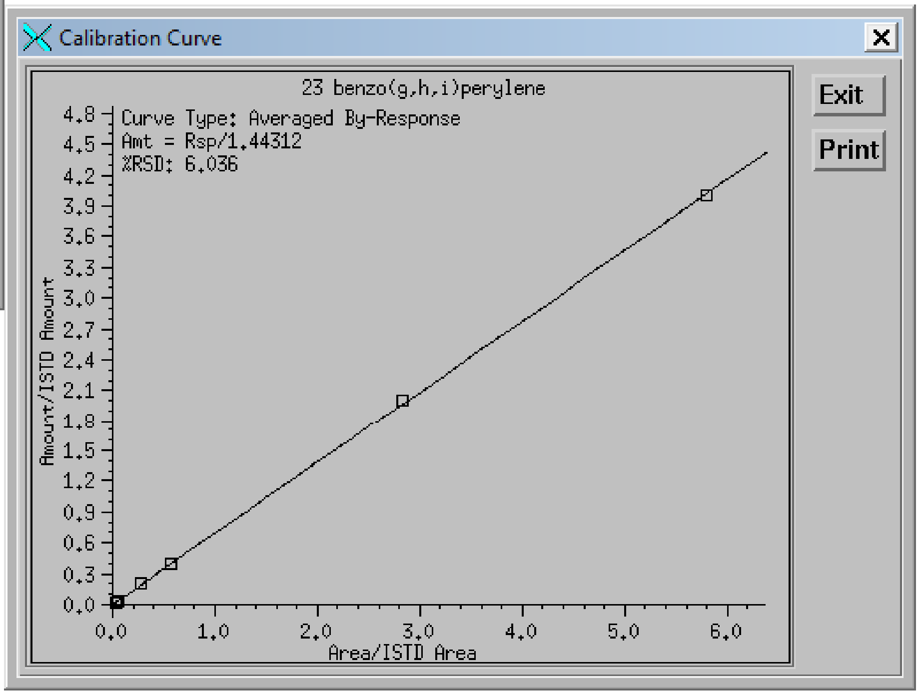


Low Level PNAs in SIM



Calibration Curves: Naphthalene & Benzo(g,h,i)perylene

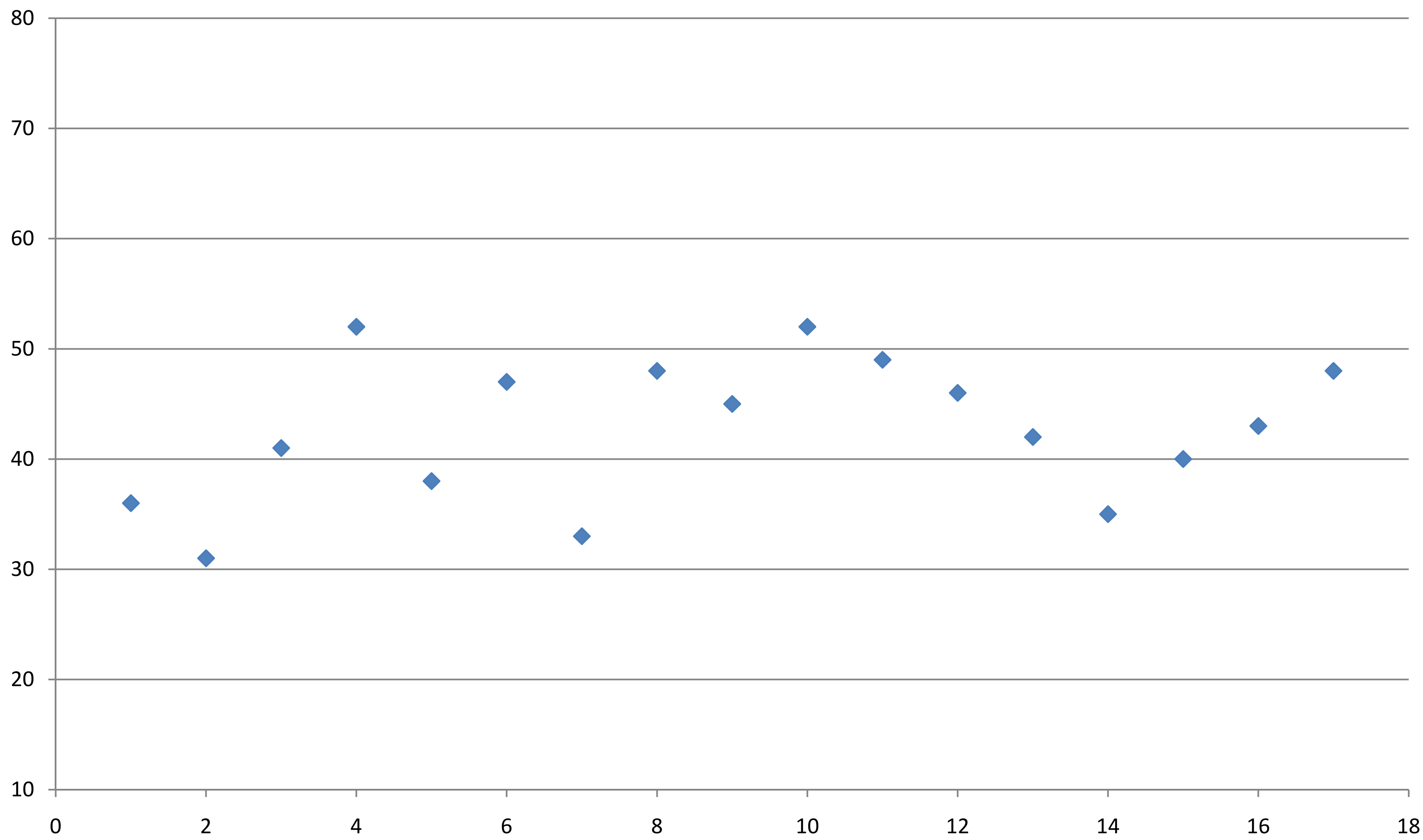




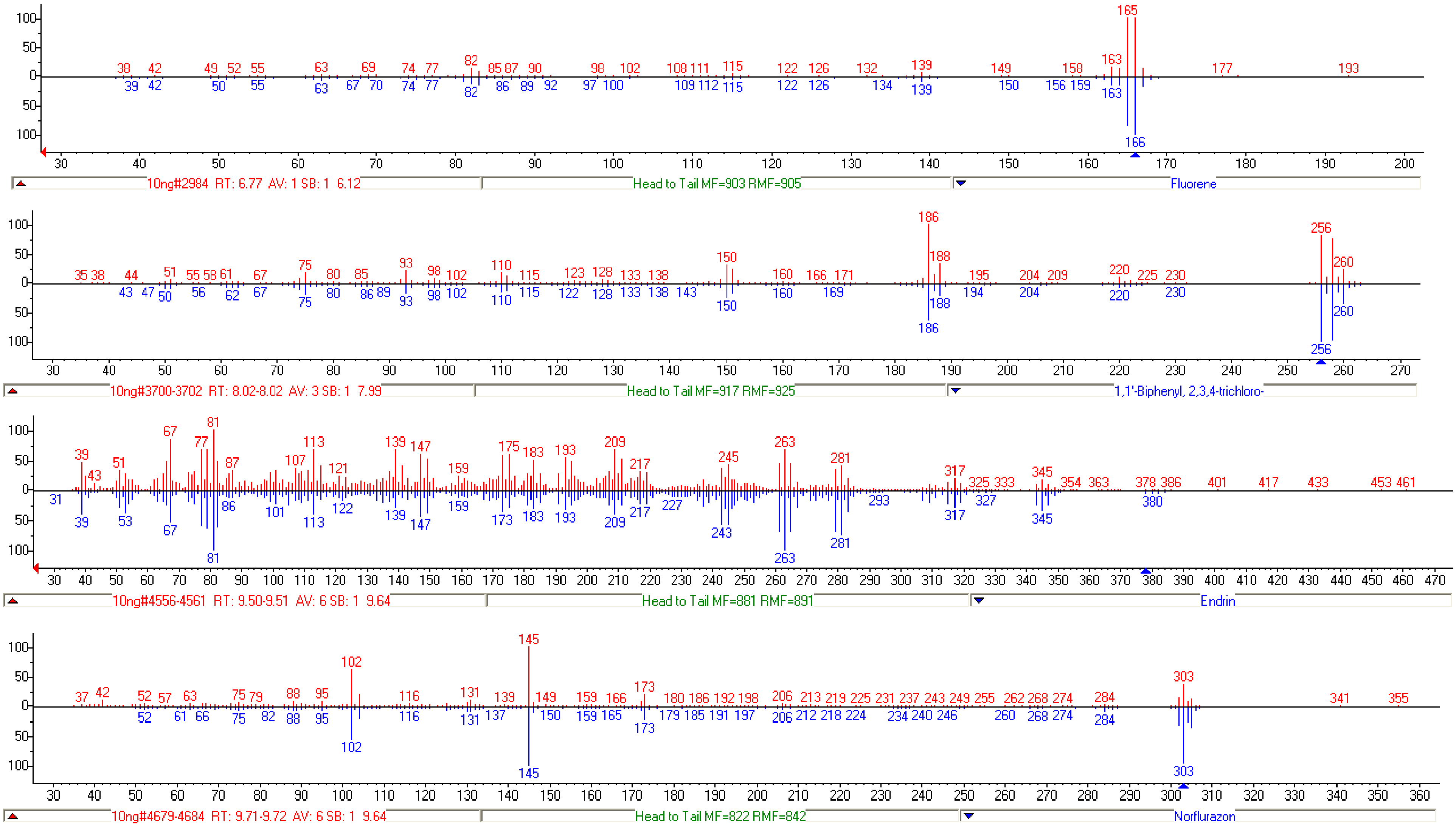
Linear Fit of Low Level PNAs in SIM (0.05 to 10 ppm)

Compound	%RSD	Compound	%RSD
naphthalene	6.1	flouranthene	6.7
2-methylnaphthalene	8.7	benzo(a)anthracene	8.2
1-methylnaphthalene	7.7	chrysene	5.9
acenaphthylene	8.5	benzo(b)fluoranthene	8.6
acenaphthene	8.5	benzo(k)fluoranthene	9.2
flourene	8.5	benzo(a)pyrene	7.8
phenanthene	3.8	indeno(1,2,3,c,d)pyrene	5.1
anthracene	7.0	dibenz(a,h)anthracene	5.4
pyrene	3.6	benzo(g,h,i)perylene	6.0
acenaphthene-d10	4.6	phenanthene-d10	4.5
chrysene-d12	9.7	perylene-d12	7.4

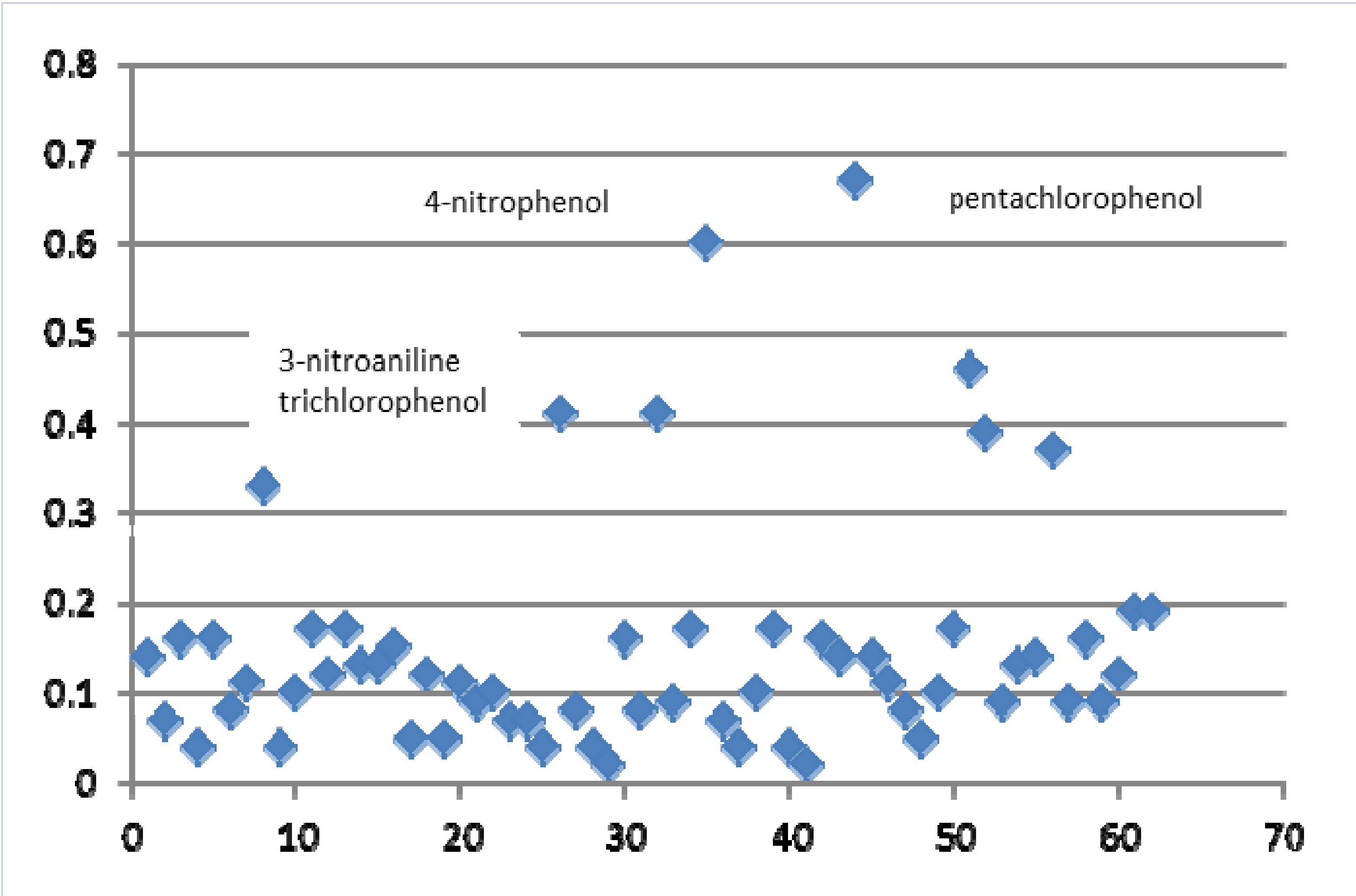
Low Level PNAs 50 ppb (+/- 50%) Accuracy



Spectral Purity: NIST Library Match



8270 Sensitivity and IDLs



EPA Methods 524 and 525 – Drinking water

- **EPA Method 524**
  - Purge & Trap
  - 5 mL sample volume
  - Calibration (0.4 – 200 ppb)

- **EPA Method 525**
  - Direct 1 µL liquid injection
  - 1 liter sample volume
  - Calibration (0.1 – 10 ppm)

Volatile and Semi-volatile Organics

Method Parameters: EPA Method 524.2

- **Purge & Trap: 5 mL sample**

VOCARB	Purge	11 min
	Dry Purge	1 min
Desorb	240°	2 min

- **Oven**

50°	1 min.	30°/min
150°	0 min	20°/min
200°	0 min	30°/min
300°	0 min	

- **Inlet**
  - S/SL: 200°
  - Split : 40:1
- **Carrier: 0.7 mL/min H_2**
- **Column: TG VMS 0.18 mm x 20 m x 1.0 µm**

- **MS**

Source	275°
EC	25 uamps
FS 0.8-1.6 1.6- 15 min	47-300 35-300
Scan Speed	1,687 amu/sec

Method Parameters: EPA Method 525.2

- **Standards**
  - Prepared in ethyl acetate
  - Calibration curve (0.1, 0.5, 1.0, 2.0, 5.0, 10 ug/mL)
- **Inlet**
  - S/SL: 325° C
  - Split Mode: 15:1
- **Carrier: 1 mL/min H_2**
- **Column**
  - TG-5 SilMS 20m x 0.18mm x 0.36 µm

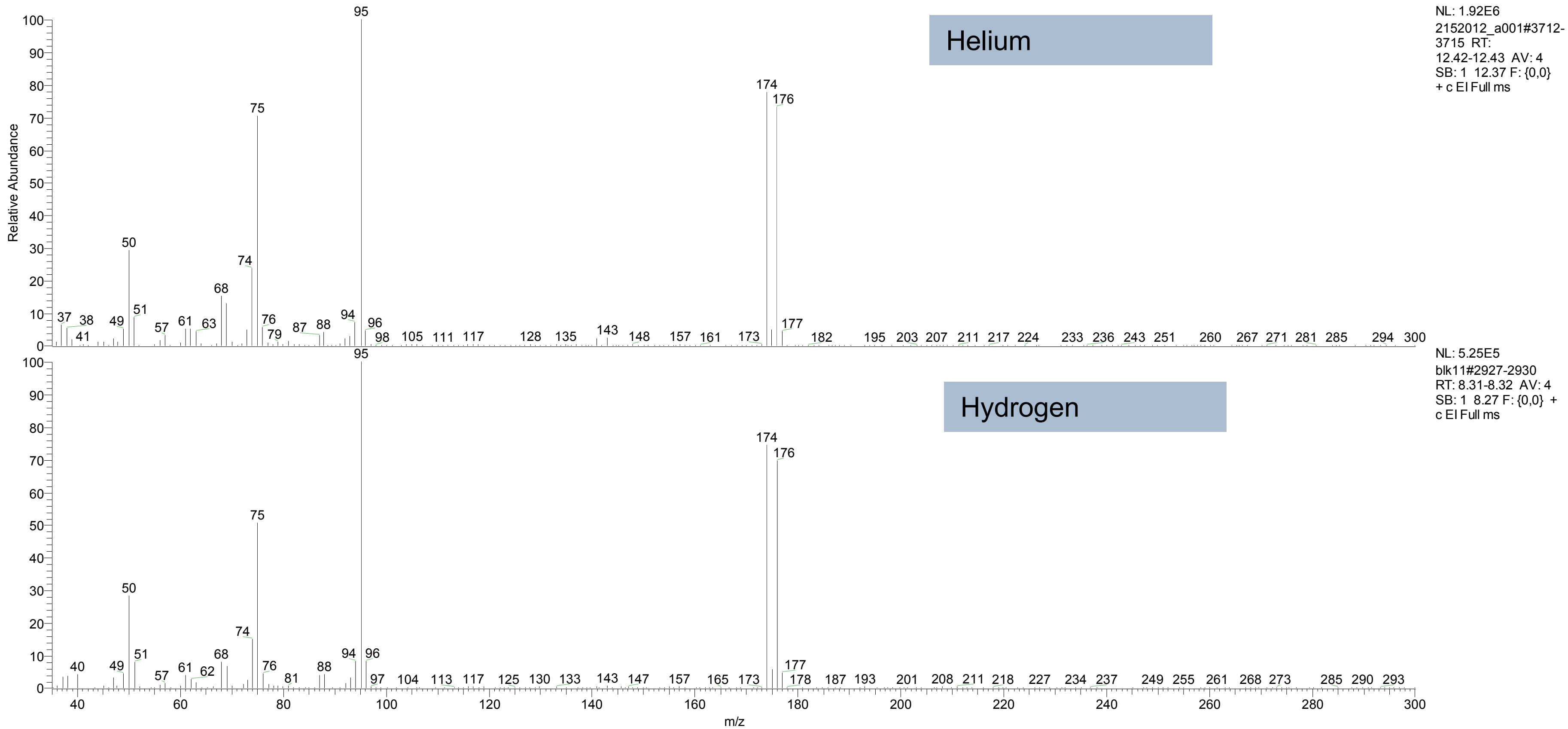
- **Oven:**

50°	1 min	30°/min
150°	0 min	20°/min
200°	0 min	30°/min
300°	0 min	20°/min

- **MS**

Source	325°
EC	15 uamps
Full Scan	35-500
Scan speed	4,650 amu/ sec

BFB (1-bromo-4-fluorobenzene)

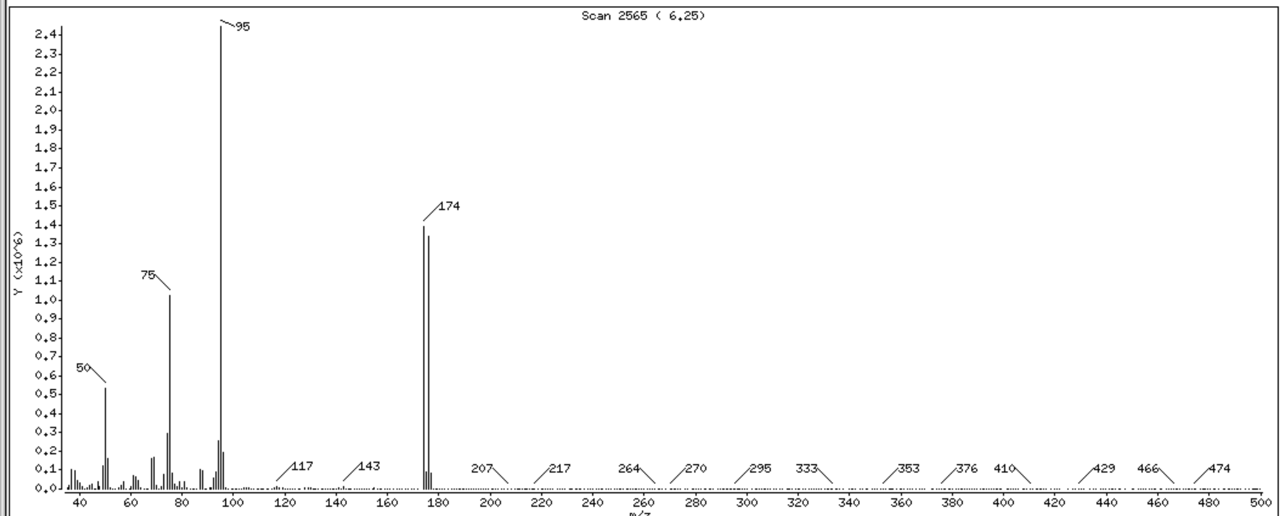


Target BFB (4-Bromofluorobenzene) H_2 Tuning Report

Graphic Report Host: ISQ-PC

Exit
Prev
Next
Print

Data File: C:\chem\ISQ.i\test.b\BFBtune.d
Page 2
Date : 14-NOV-2012 08:07
Client ID:
Instrument: ISQ.i
Sample Info: bfb 25ppm
Volume Injected (uL): 1.0
Operator:
Column phase:
Column diameter: 2.00
1 bfb

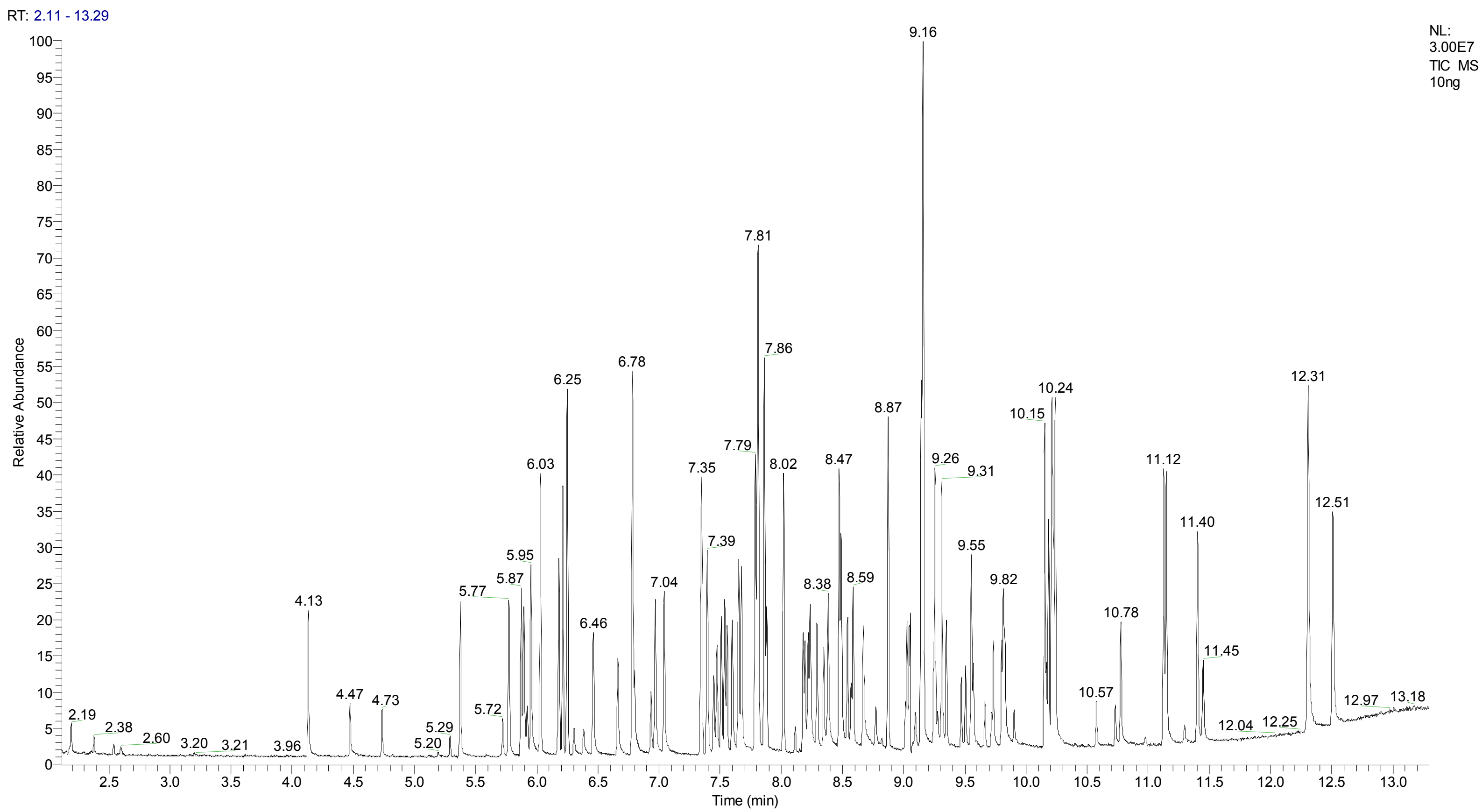


m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
95	Base Peak, 100% relative abundance	100.00
50	15.00 - 40.00% of mass 95	21.73
75	30.00 - 60.00% of mass 95	41.79
96	5.00 - 9.00% of mass 95	7.88
173	Less than 2.00% of mass 174	0.00 (0.00)
174	Greater than 50.00% of mass 95	56.83
175	4.00 - 9.00% of mass 174	3.81 (6.70)
176	95.00 - 101.00% of mass 174	54.87 (96.54)
177	5.00 - 9.00% of mass 176	3.39 (6.18)

BFB Ion Ratio Stability

m/z	Criteria	run01	run02	run03	run04	run05	run06	run07	run08	run09	run10	run11
50	15-40%	28	26	26	24	27	26	25	26	28	25	25
75	>30%	52	51	48	48	51	52	49	48	48	50	53
95	100%	100	100	100	100	100	100	100	100	100	100	100
96	5-9%	8.0	7.3	7.9	7.7	8.2	7.2	7.7	7.4	7.8	7.5	7.7
173	< 2% of 174	0.4	0.6	0.7	1.5	0.4	0.4	0.8	0.7	0.5	1.1	0.3
174	>50%	76	75	69	69	75	70	72	72	71	74	72
175	5-9% of 174	7.3	7.4	7.4	5.9	7.6	8.5	6.4	7.7	6.8	6.4	6.7
176	95-101% of 174	96	95	100	96	93	96	98	95	96	101	96
177	5-9% of 176	5.1	6.3	6.6	5.2	6.4	6	5.1	5.5	6.6	6.5	6

Performance: EPA Method 525 10 ppm Standard



Thermo Fisher SCIENTIFIC

Performance: EPA Method 524.2

- Method parameters

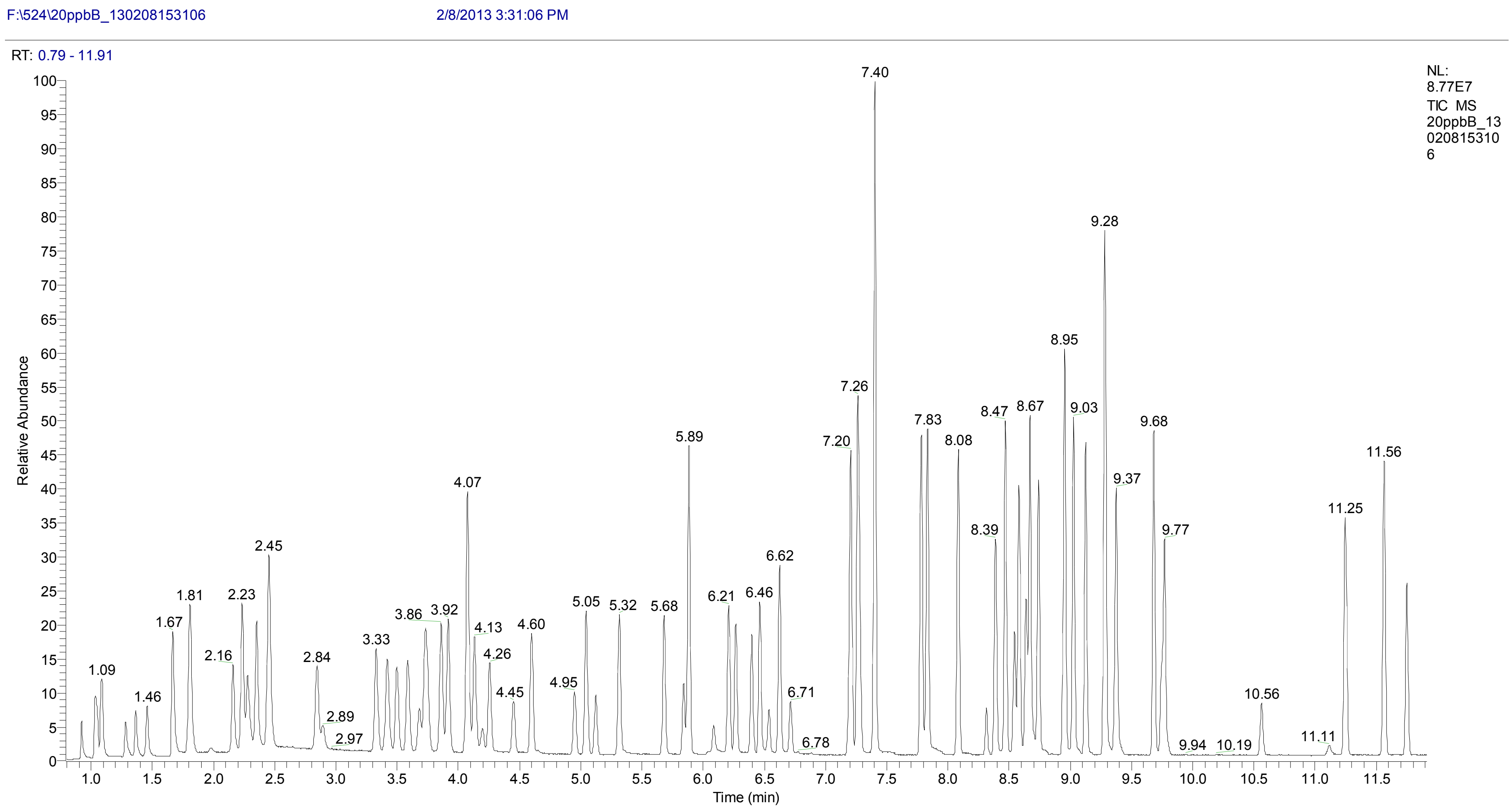
- Spectral integrity
  - #1 Match to NIST

- Accuracy(0.4 – 200 ppb):
  - Passed at all levels
- **Average MDL: 0.074 ppb vs 0.048 ppb in helium**

- BFB Ion ratio stabile

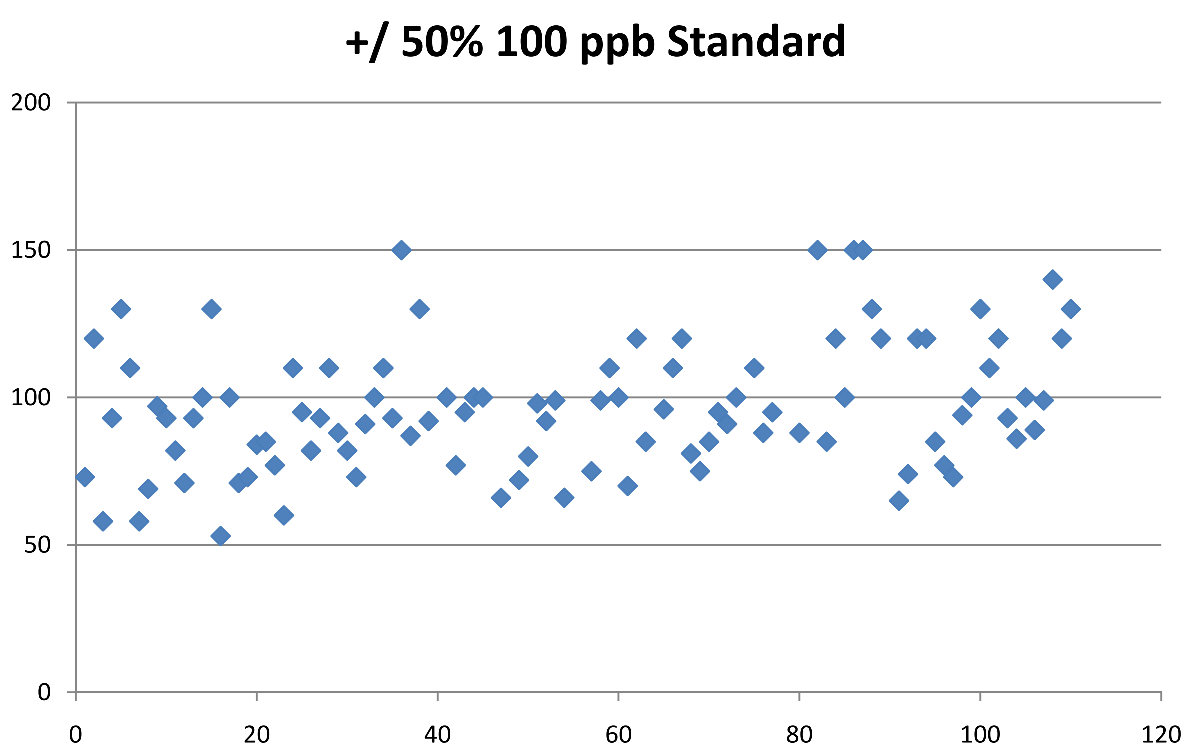
Passes QC for EPA Method 524.2

Performance: EPA Method 524 - 20 ppb Standard



Thermo Fisher SCIENTIFIC

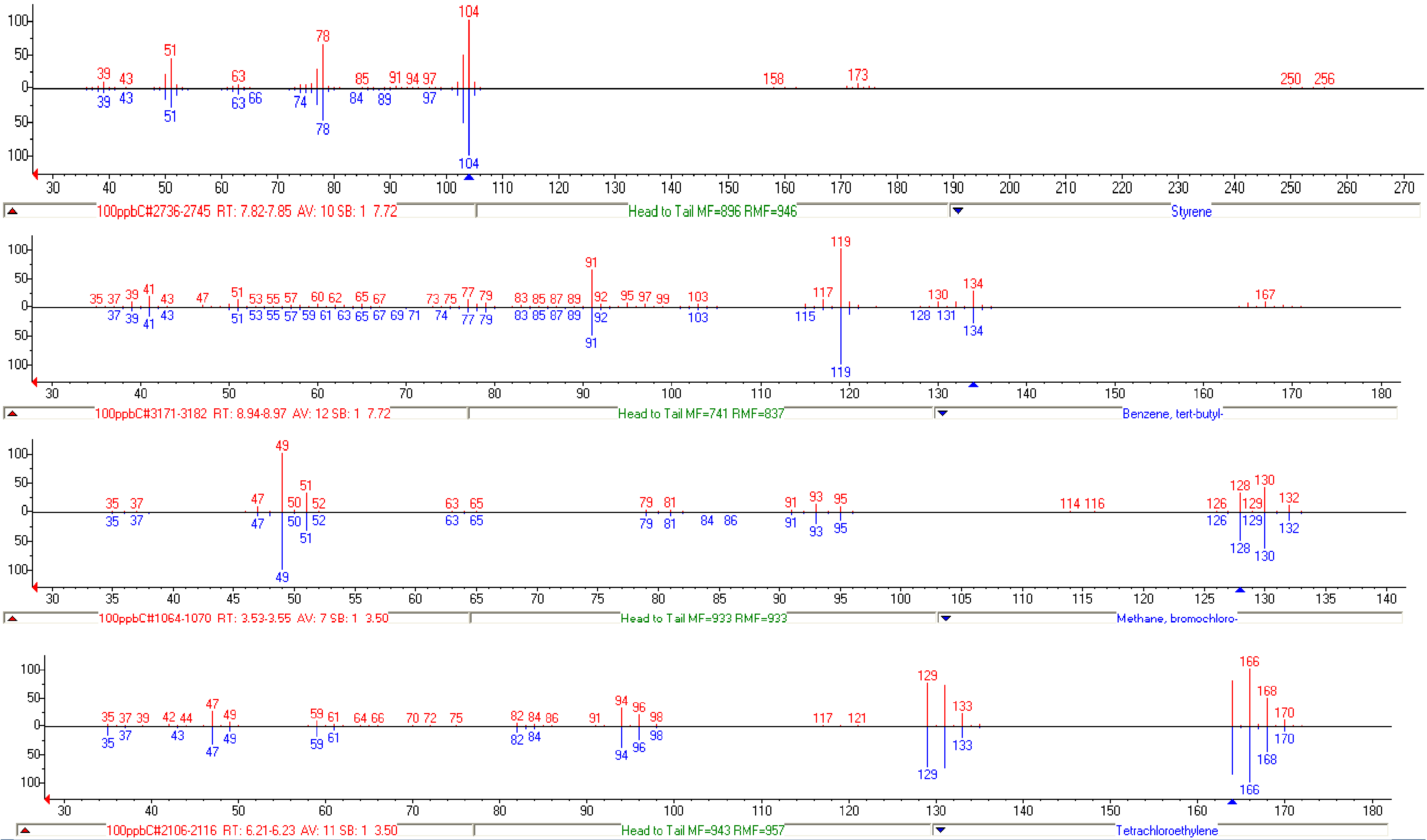
EPA Method 525: Measurement of Accuracy (+/- 50%)



Failed
pentachlorophenol
terbacil
metribuzin
bromacil
chlorpyrifos
trans-nonachlor
carboxon
Dieldrin
tricyclazole

Calibration: 0.1,0.5,1,2,5,10 ppm in Full Scan

Spectral Purity NIST Library Match



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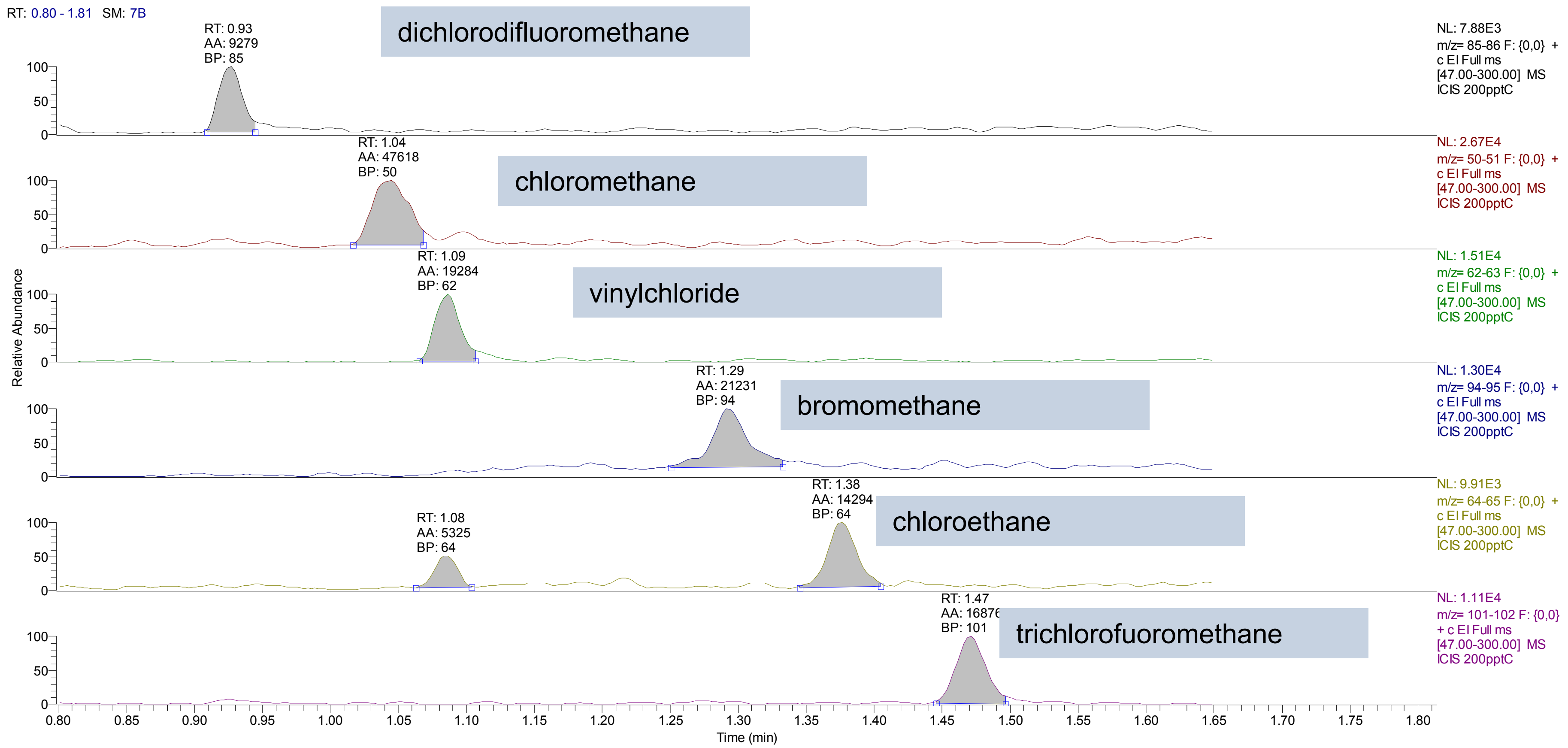
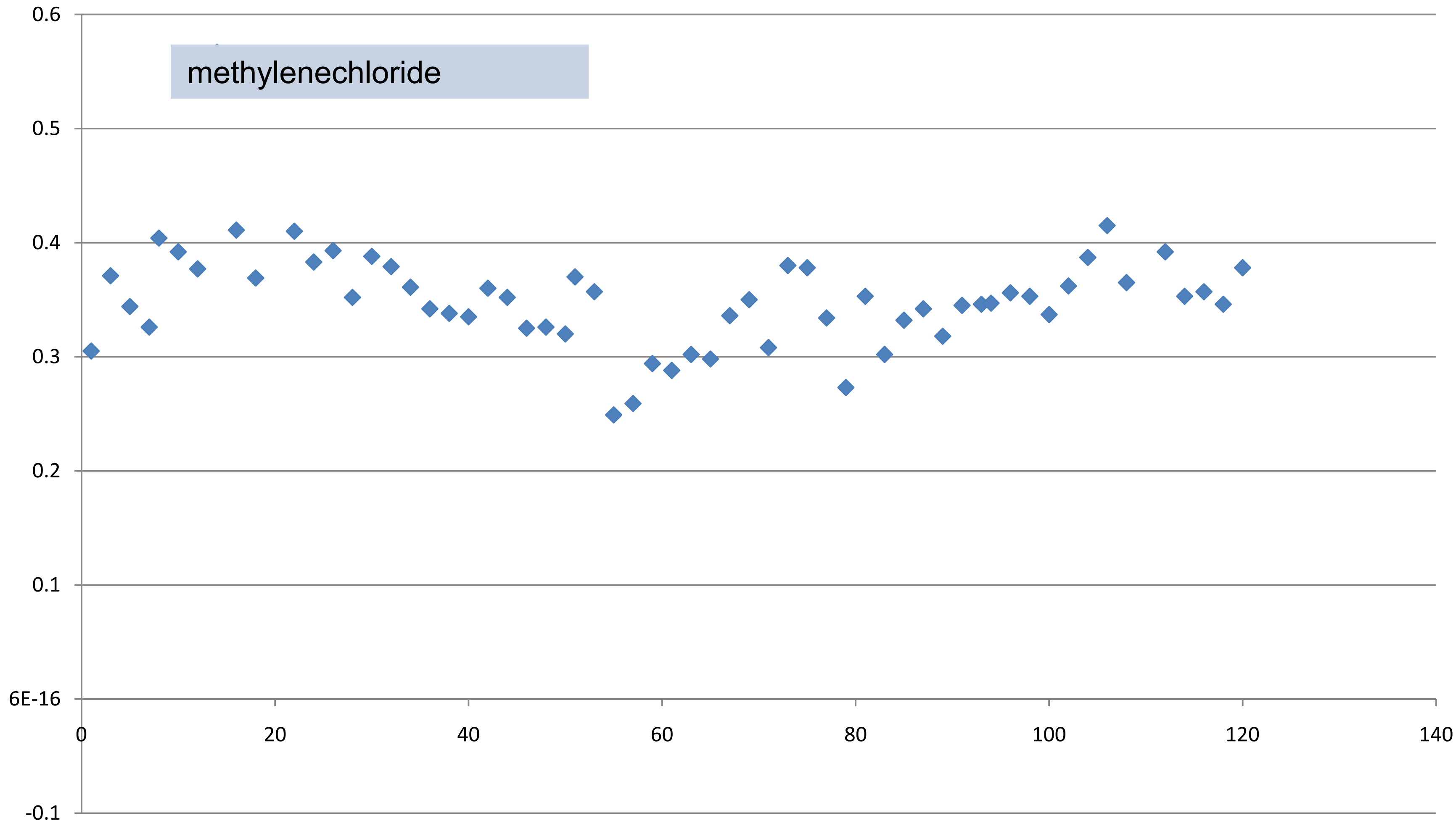
Gases at 200 ppt



Accuracy of 400 ppt - Passed +/- 50%



Thermo Fisher
SCIENTIFIC

Moving to Hydrogen for your Environmental Analyses

The world leader in serving science

Moving To Hydrogen on the GC Side

- **Hydrogen Sensor is a *Must***
  - In GC oven
  - Possible on the hydrogen generator
  - May also be required at the ceiling of the lab
- **Expect Lower Inlet Pressure**
  - Due to viscosity differences of H_2 and He
  - Move to a smaller id column

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Moving to Hydrogen on the MS Side

- ***NO*** **MS hardware change required to meet H_2 Installation Specs**
- **Maintain good vacuum**
  - Extended performance turbomolecular pumps
  - 9.8 x 10^{-6} Torr (1 ml/min hydrogen flow)
- **Higher initial background**
  - Minimized with stainless steel pre-cleaned tubing
  - UHP Grade 5.0 or better Hydrogen Source
  - Bake out source at 350° C for one hour with filament on
- **CI Effect on some compounds**
  - Require linear or quadratic fit
  - Reduce flow rate of H_2 into MS
- **Pressure dependency:**
  - Minimize solvent vapor with smaller id columns

ISQ Off-Axis Single Quadrupole GC-MS

Conclusion: Moving to Hydrogen Carrier Gas

- Considerations
  - Gas purity
  - Safety
  - Column selection
  - Column flow rate
  - Mass spectrometer modifications:
    - Hydrogen kit

Meets QC requirements for EPA Method 524 and 525

Thank you for your attention!

Questions?

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