The Agilent 5975 inert MSD: New Tools for the Forensic Analyst

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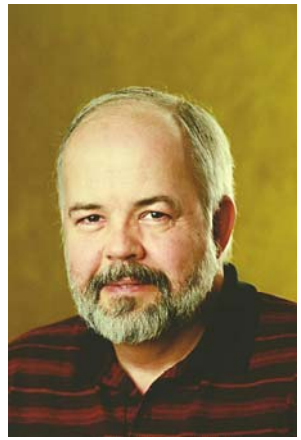
The Speaker

Bruce Quimby is a Senior Applications Chemist in the Chemical Analysis Solutions unit of Agilent Technologies in Wilmington, Delaware. He received a Ph.D. in analytical chemistry from the University of Massachusetts (Amherst) in 1980 and a batchelors degree in chemistry from Mansfield State College (PA) in 1974 . He has been at Agilent Technologies (formerly Hewlett-Packard) since 1979, working the first 10 years in research and development. He has authored or co-authored 18 journal articles and 10 patents in the field of gas chromatography. He is currently working in Forensic and Homeland Security applications.

R&D ID Photo



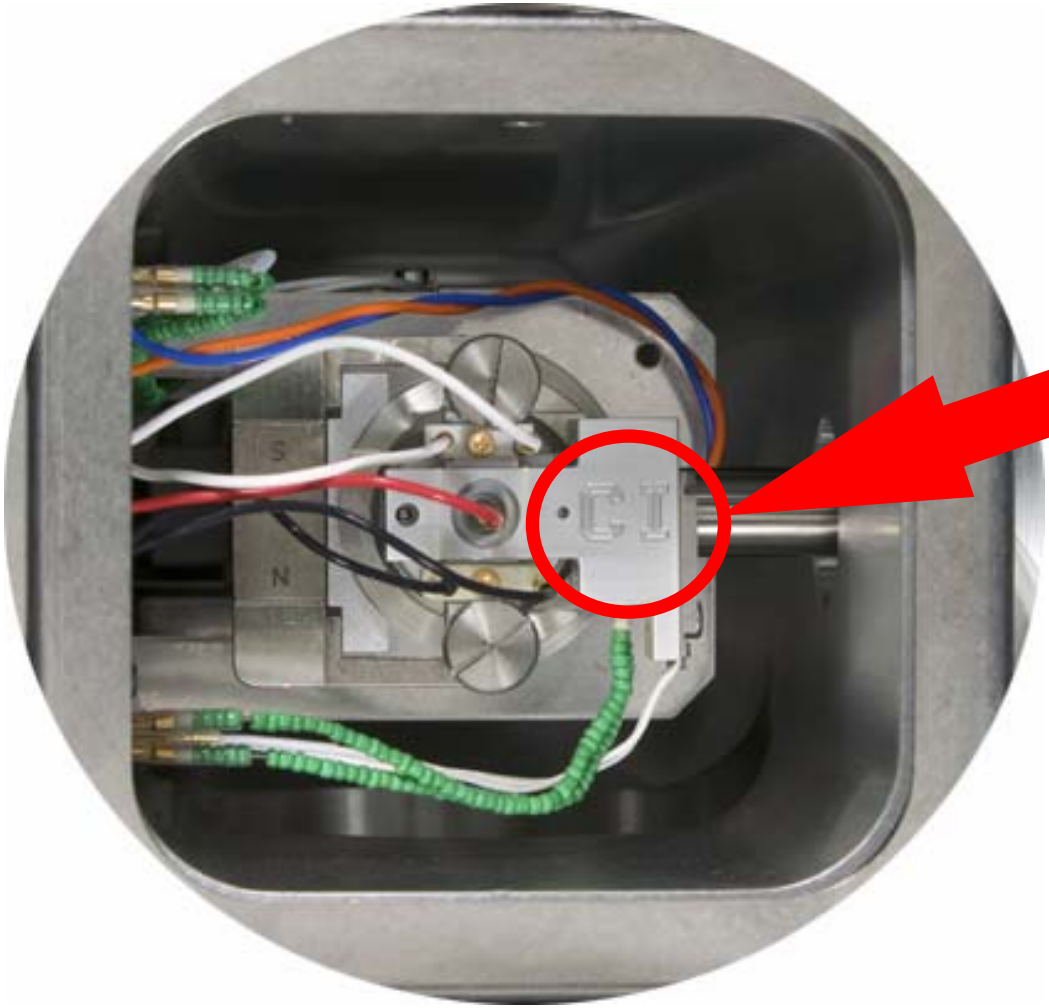
Marketing ID Photo

Introducing the 5975 inert MSD

What's Different?

- Does everything a 5973 does, plus…
- Synchronous SIM/Scan
- Extended mass range
- AutoCI and EI with CI source
- 6850 GC control
- QuickSwap MSD interface
- Permanent Effluent Splitters
- Deconvolution Reporting Software

Functional Front Window – Ease of Use



- ***Wire connections***
- ***Filament operation***
- ***Column insertion***
- ***CI Source identification***

Quick Access – Ease of Use

Easy analyzer access – hinged side cover

Extended Mass Range – Performance

1050 m/z for 5975 inert MSD

- Quadrupole modified
- New electronics

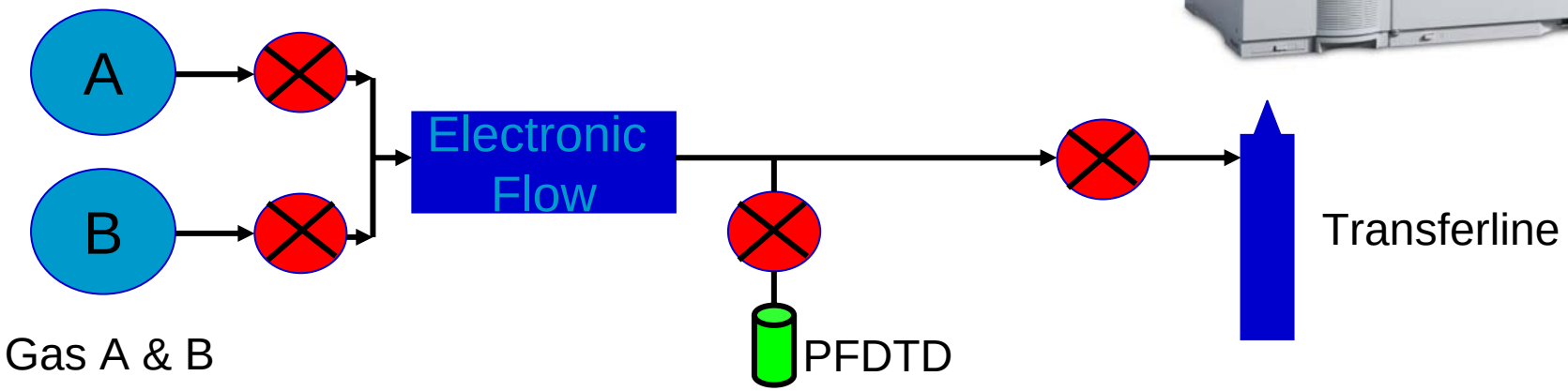
- 800 m/z for all 5973 MSDs
  (Software upgrade does not enable 1050 m/z)

AutoCI – Performance

- No external manual control
- Fully automated setup/tuning
- Flexible Configuration
- EI capability with CI Source
- AutoCI – as easy as EI

AutoCI Electronic Flow Control – Ease of Use

- **Reagent gas automatically adjusted by tune**
  - Ammonia compatibility
- **Ensures easy reproduction of application**



Ion Source – Improved Response

Data from a production forensic drug lab…

Inert Ion source – Improved response

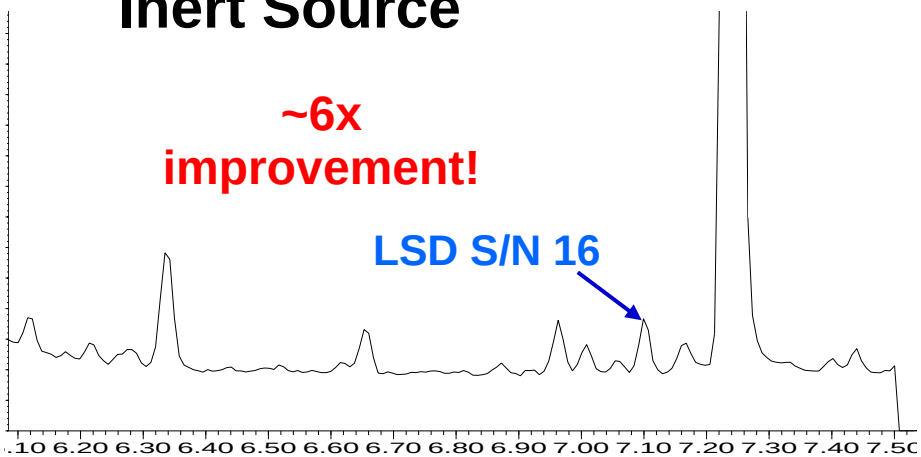
50pg LSD

Extracted Ion 253 m/z

Inert Source

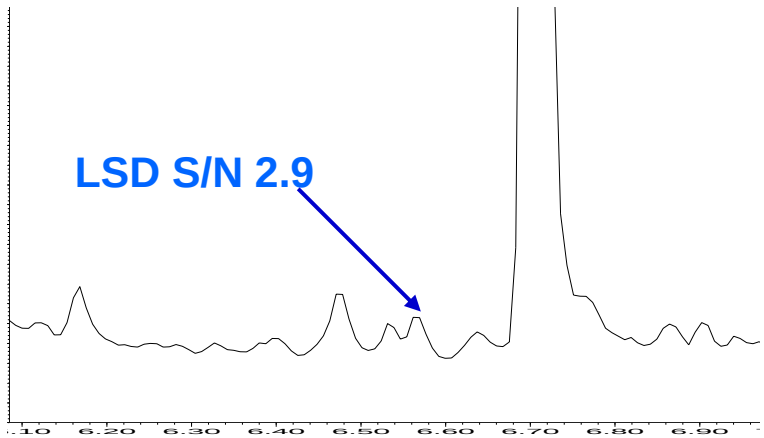
~6x improvement!

LSD S/N 16



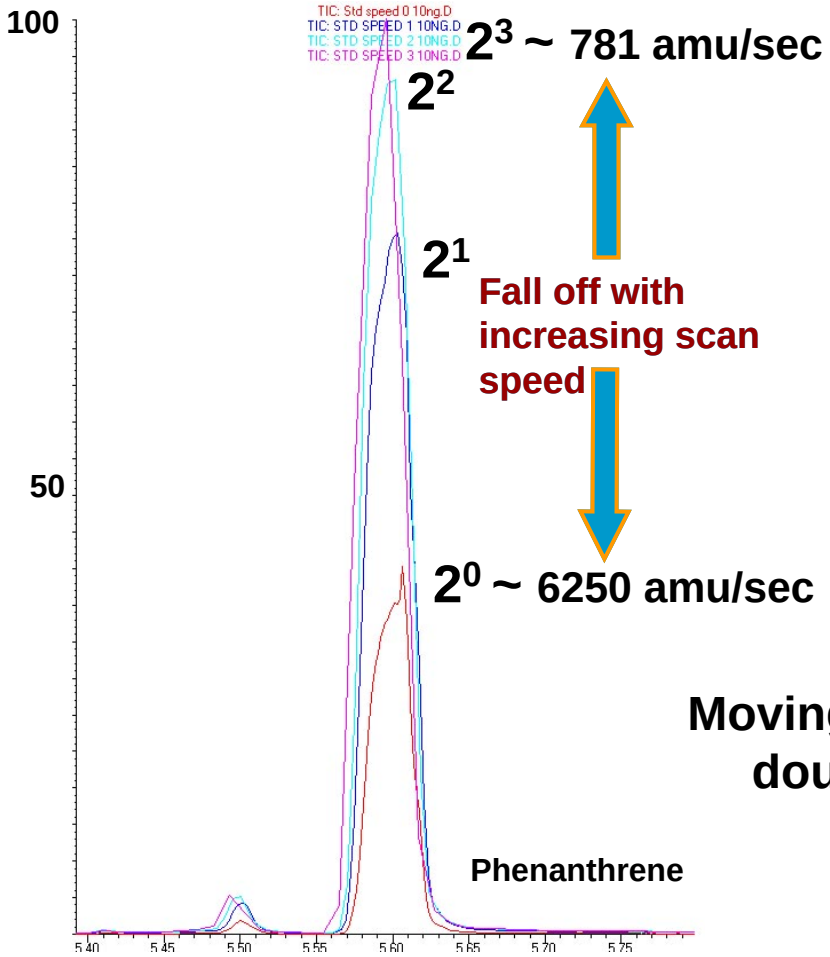
SS Source

LSD S/N 2.9

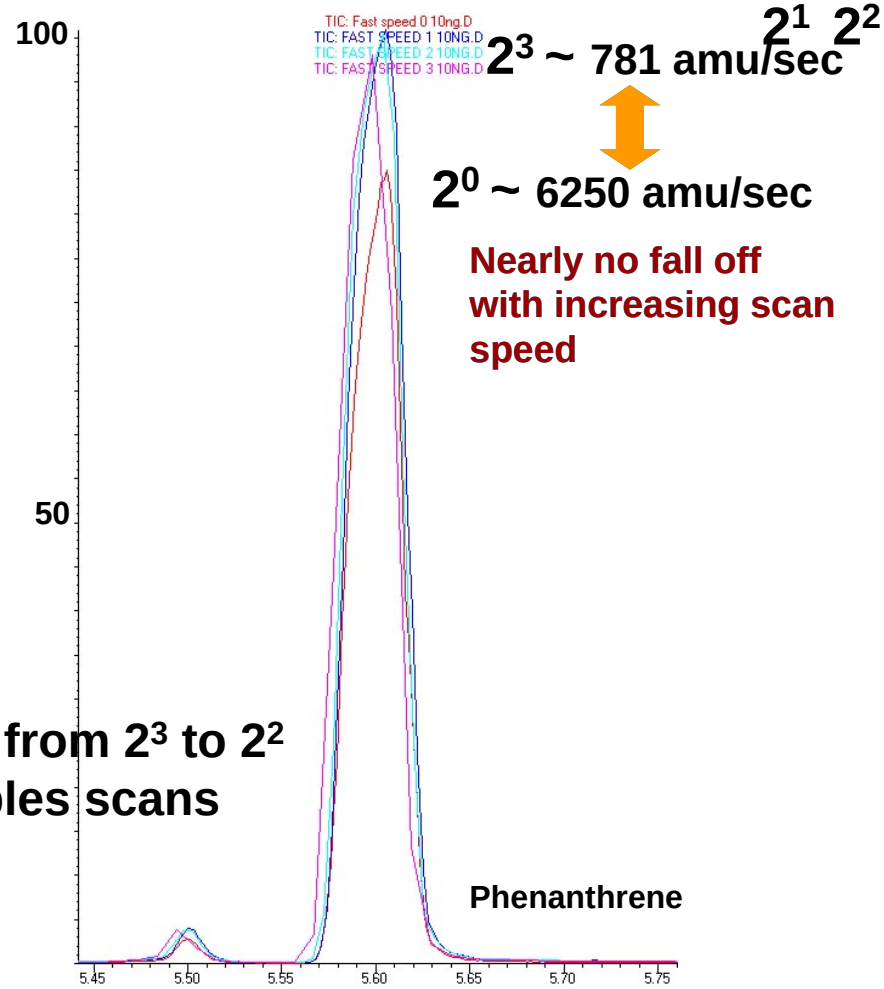


Faster Scan Speeds and 5 msec SIM Dwell

Standard Electronics

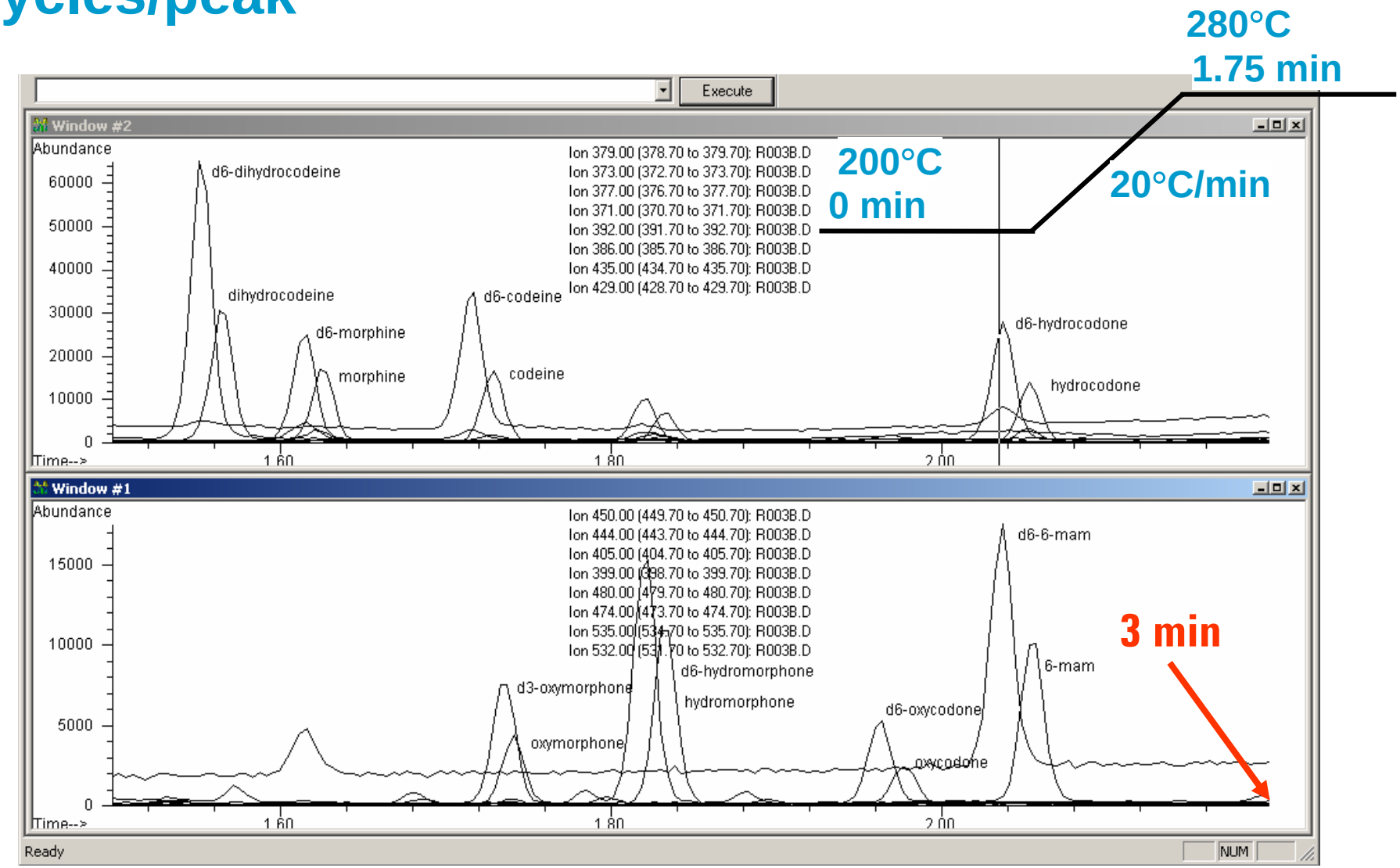


Performance Electronics



Moving from 2^3 to 2^2 doubles scans

Opiates, 38 ions (5 msec dwell) in 1 group, 10 SIM cycles/peak



Synchronous SIM/Scan

What is Synchronous SIM/Scan?

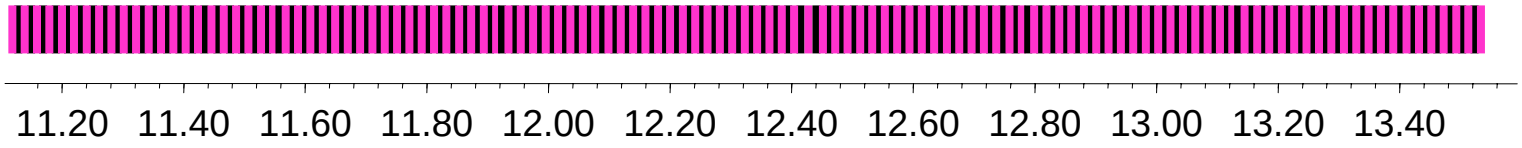
Agilent's Synchronous SIM/Scan allows for the continuous collection *of both SIM and Scan data in the SAME acquisition. More data without sacrificing sensitivity or library searching capability*

Great for target analysis (SIM) and identifying unknowns (Scan)

- SIM data for quantitation of targets
- Scan data for confirmation of unknowns

Agilent Synchronous SIM/Scan

Alternating SIM (black) and scan (magenta) data collection



SIM + scan in one cycle, alternating rapidly

High sensitivity + high information throughout the whole chromatogram

Agilent Instrumentation Used

Gas Chromatograph

- 6890N with Autosampler (tray & injector) and split/splitless inlet
- Capillary NPD with extended jet
- G3180B - Microfluidic Splitter with Makeup Gas

Mass Spectrometer

- 5975 inert MSD with EI source

Software

- GC/MSD Chemstation G1701 DA version D.02.00 or higher (includes RTL and Screener)

Column

- DB-17MS 15m x 250µm x 0.25µm part # 122-4712

Method Parameters

GC	Agilent 6890N		
Oven			
Ramp	'C/min	'C	Hold min
Initial		80	0.00
Ramp 1	20	320	5.00
Runtime	17 min		

Inlet	Split/Splitless
Temp	280 'C
Mode	Splitless
Pressure	23.16 psi
Purge Flow	50 mL/min
Purge time	2.00 min
Total Flow	56.3 mL/min

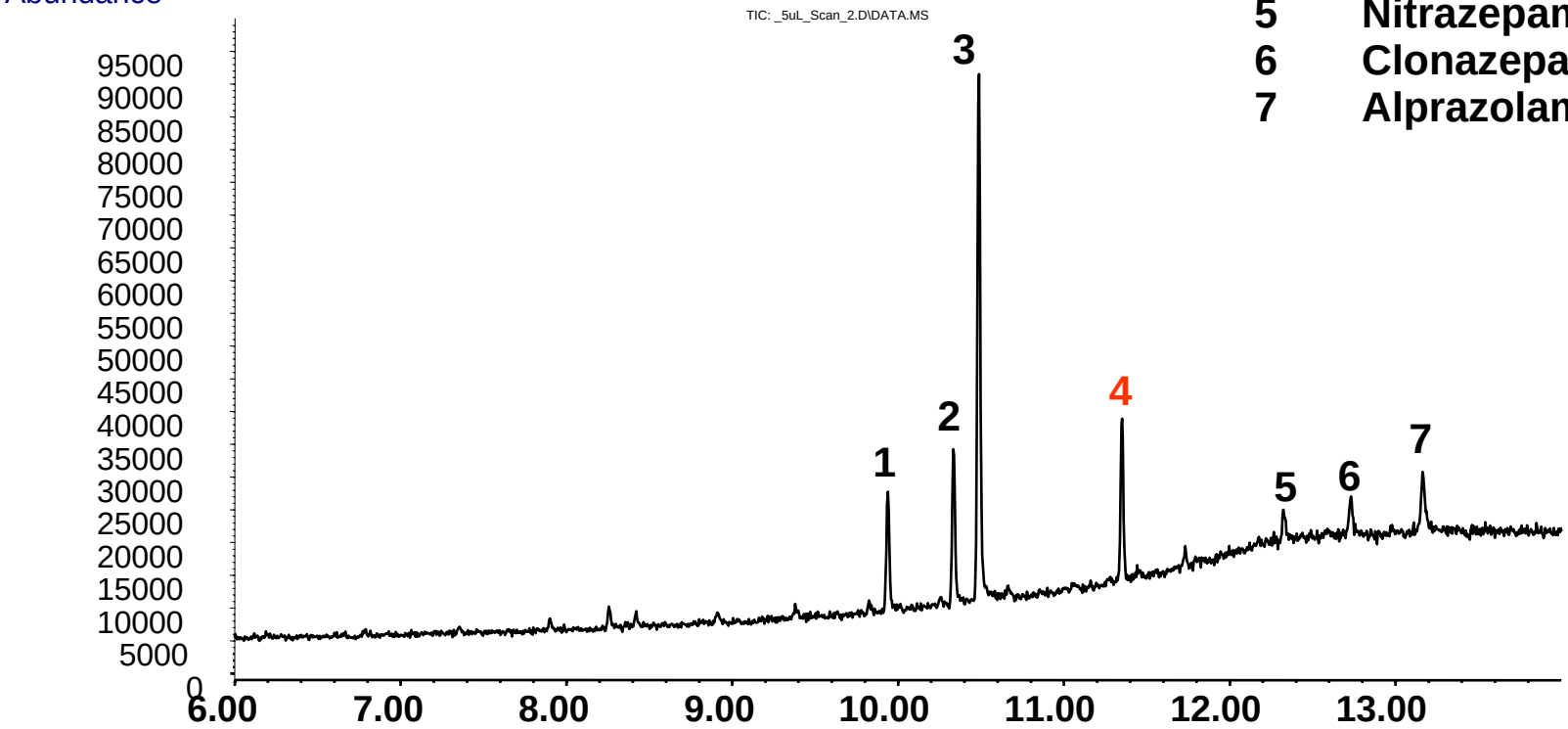
Column	DB-17MS part # 122-4712
Mode	Constant pressure
Pressure	23.16 psi
Initial Flow	3.8 mL/min
Outlet	Splitter
Outlet Pressure	3.8 psig

MSD	Agilent 5975inert
Solvent Delay	2.5 min
Scan Range	42 to 450
Threshold	0
Sampling	2^2 for scan, 2^1 for SIMSCAN
Quad Temp	150 'C
Source Temp	280 'C
Transfer Line	280 'C

Injector	
Sample Washes	0
Sample Pumps	6
Injection volume	2 uL
Solv A washes	4
Solv B washes	4
Viscosity Delay	3
Plunger Speed	Fast

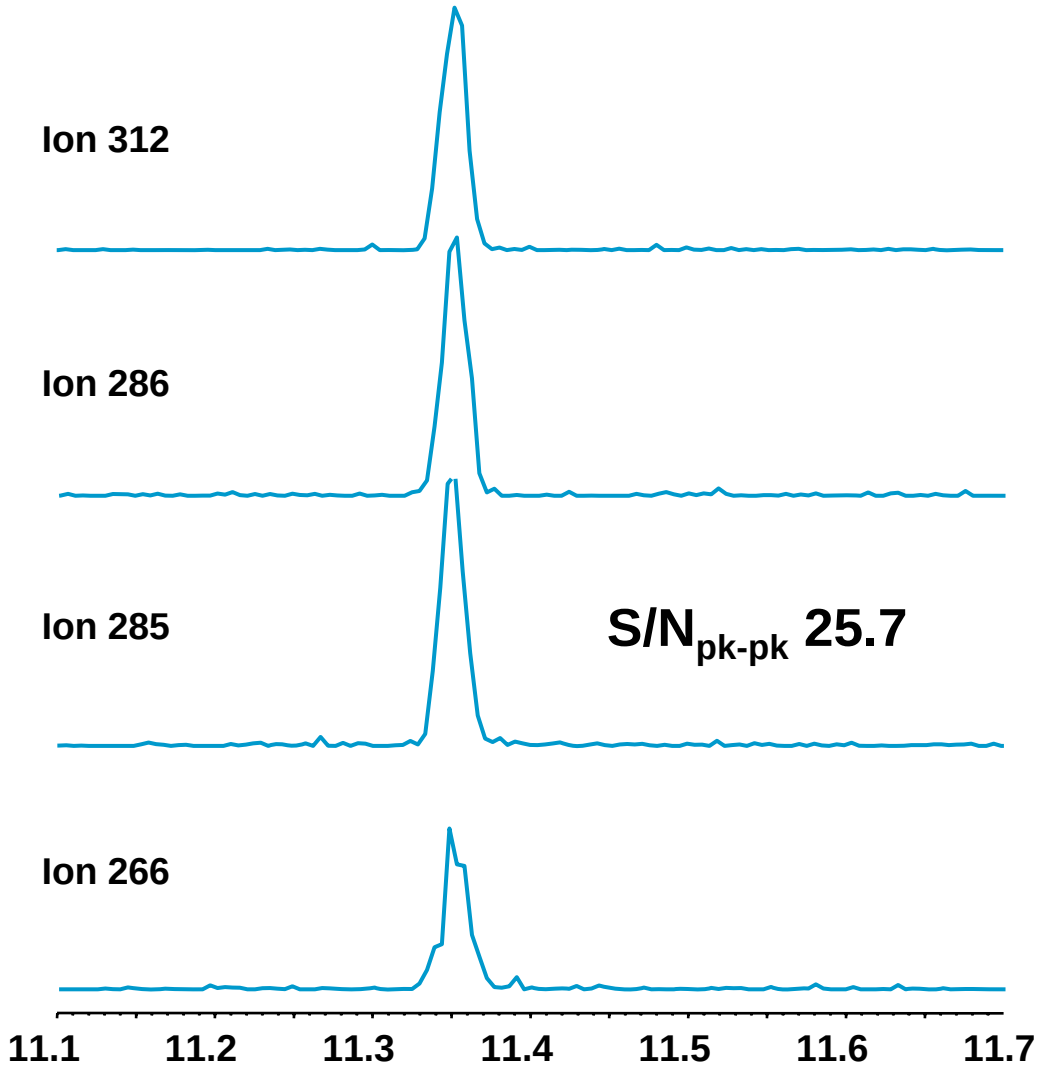
Benzodiazepines
0.63 ng each to MSD

1 Oxazepam
2 Lorazepam
3 Diazepam
4 Flunitazepam
5 Nitrazepam
6 Clonazepam
7 Alprazolam

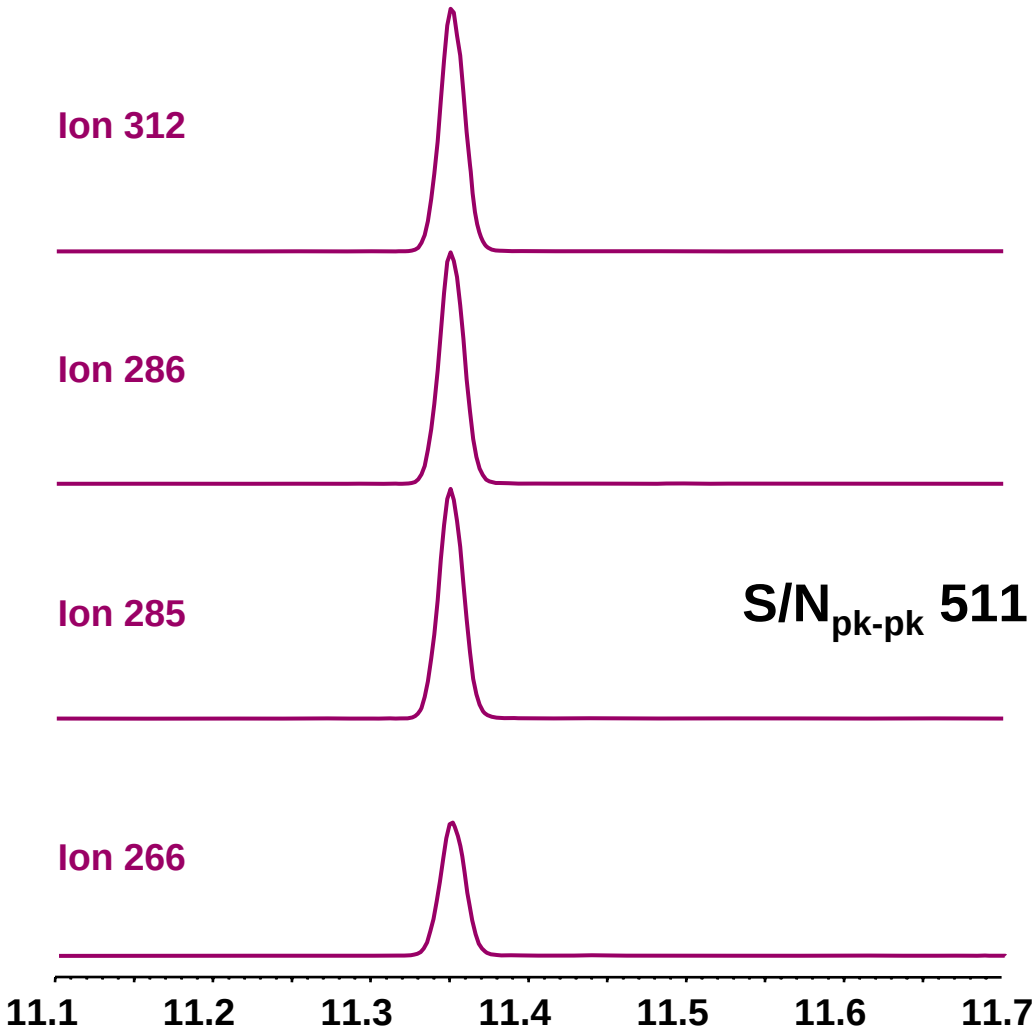


Agilent Technologies

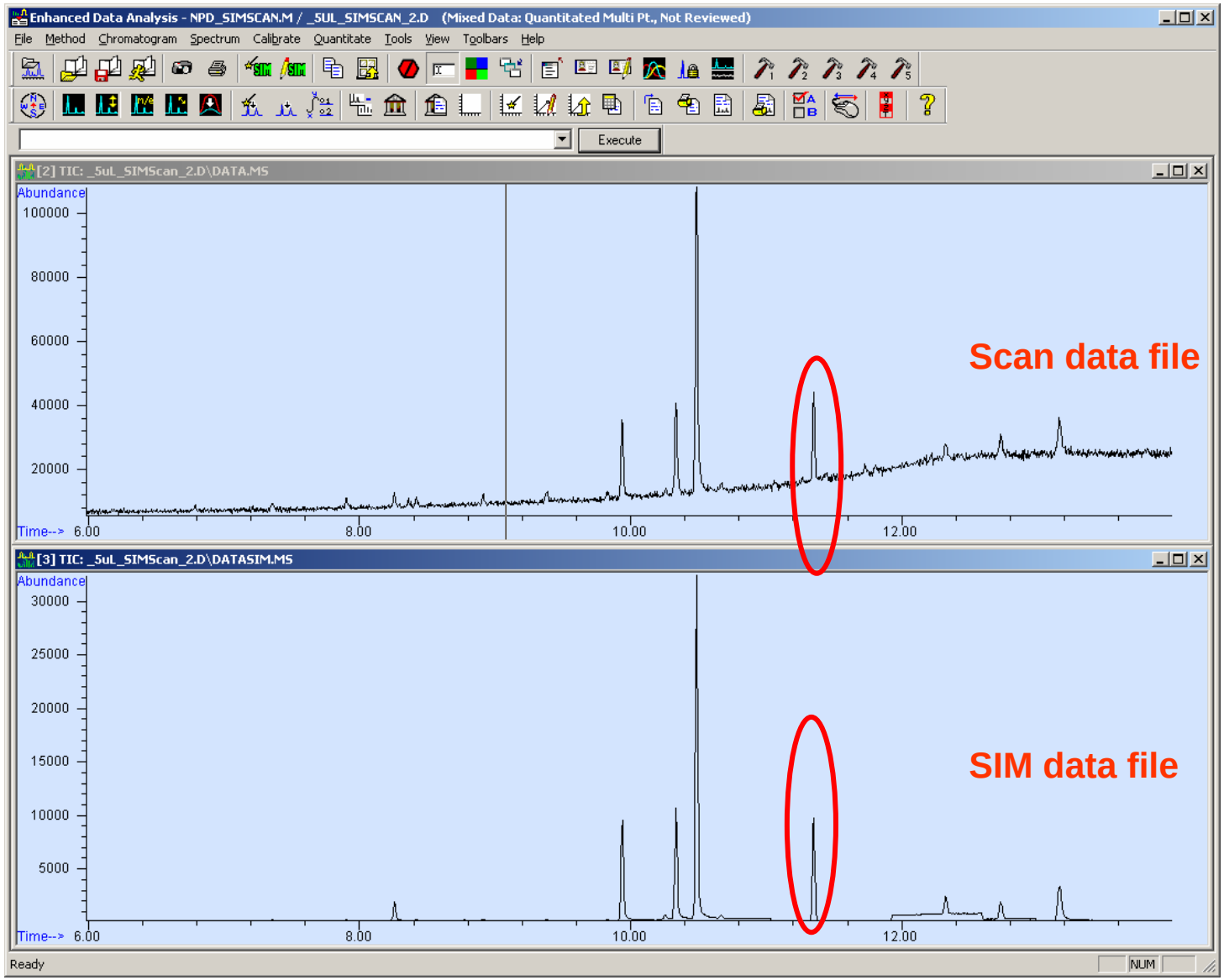
Flunitrazepam- Scan Only Mode
0.63 ng



Flunitrazepam- SIM From SIM Only Run 0.63 ng

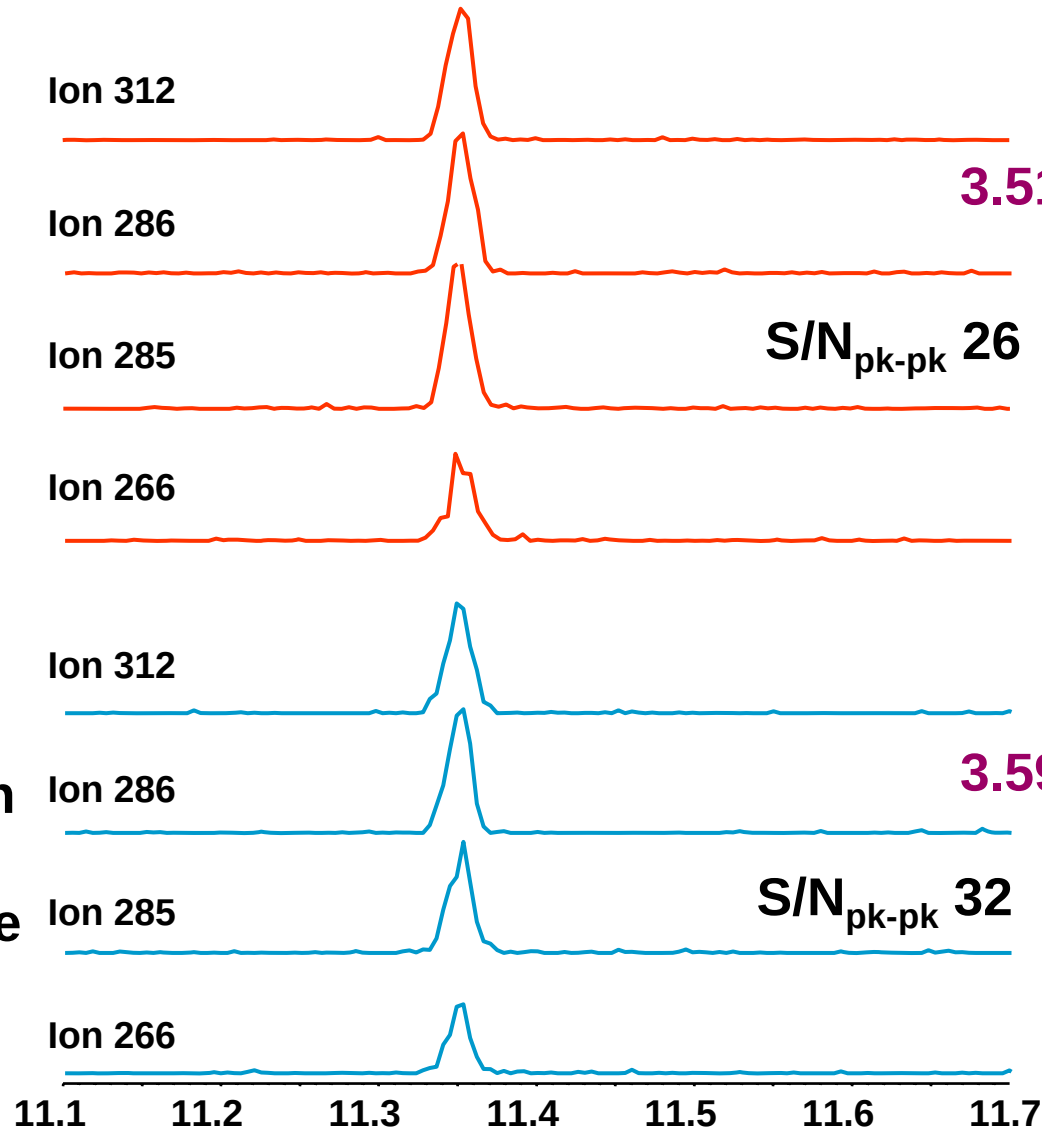


Synchronous Data



Comparison of Scan Only and Scan from SIM/Scan

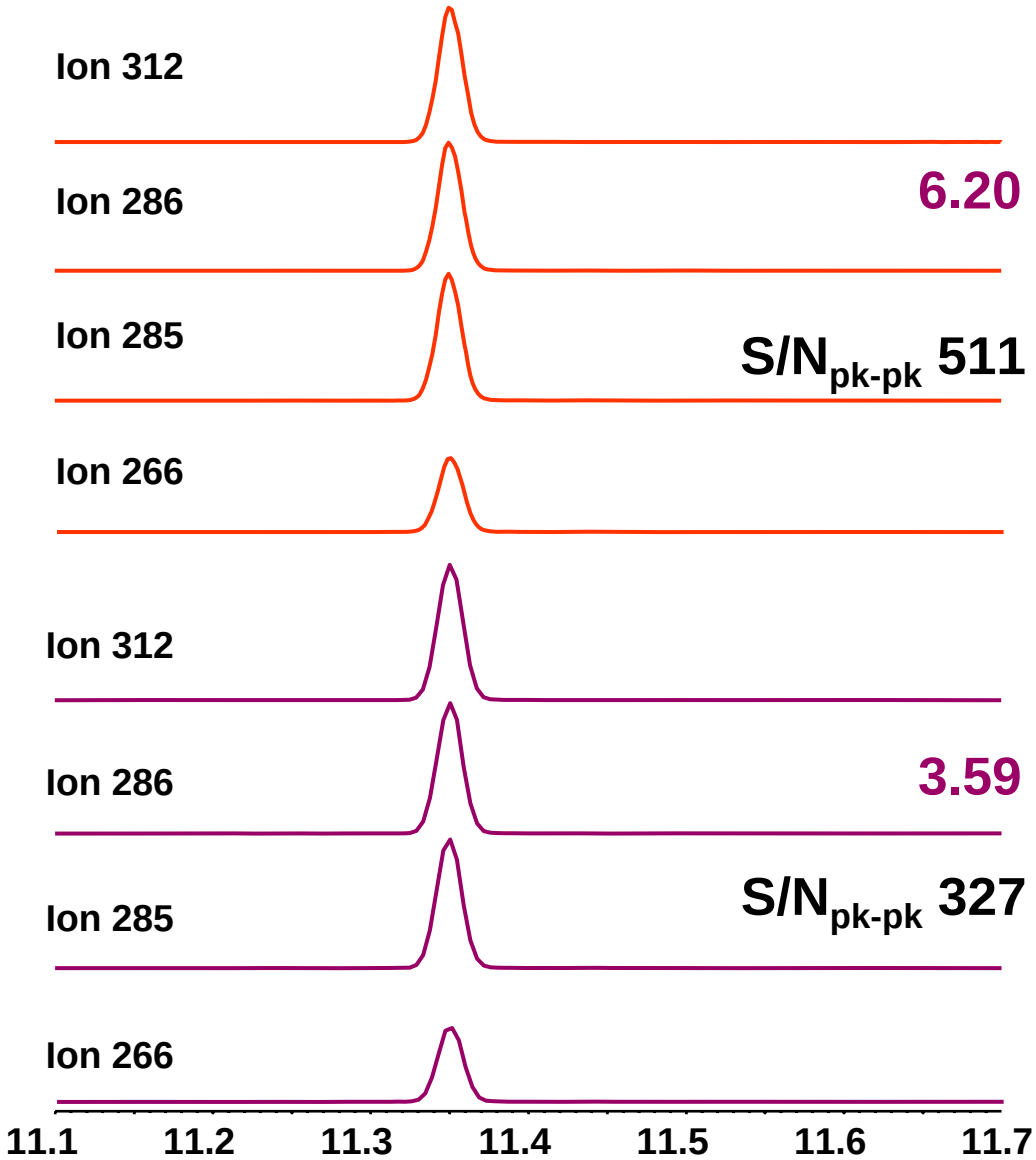
Extracted Ions From Scan Mode



Extracted Scan Ions From SIM/Scan Mode

Comparison of SIM Only and SIM from SIM/Scan

Extracted SIM Ions From Scan Mode
25 msec dwell

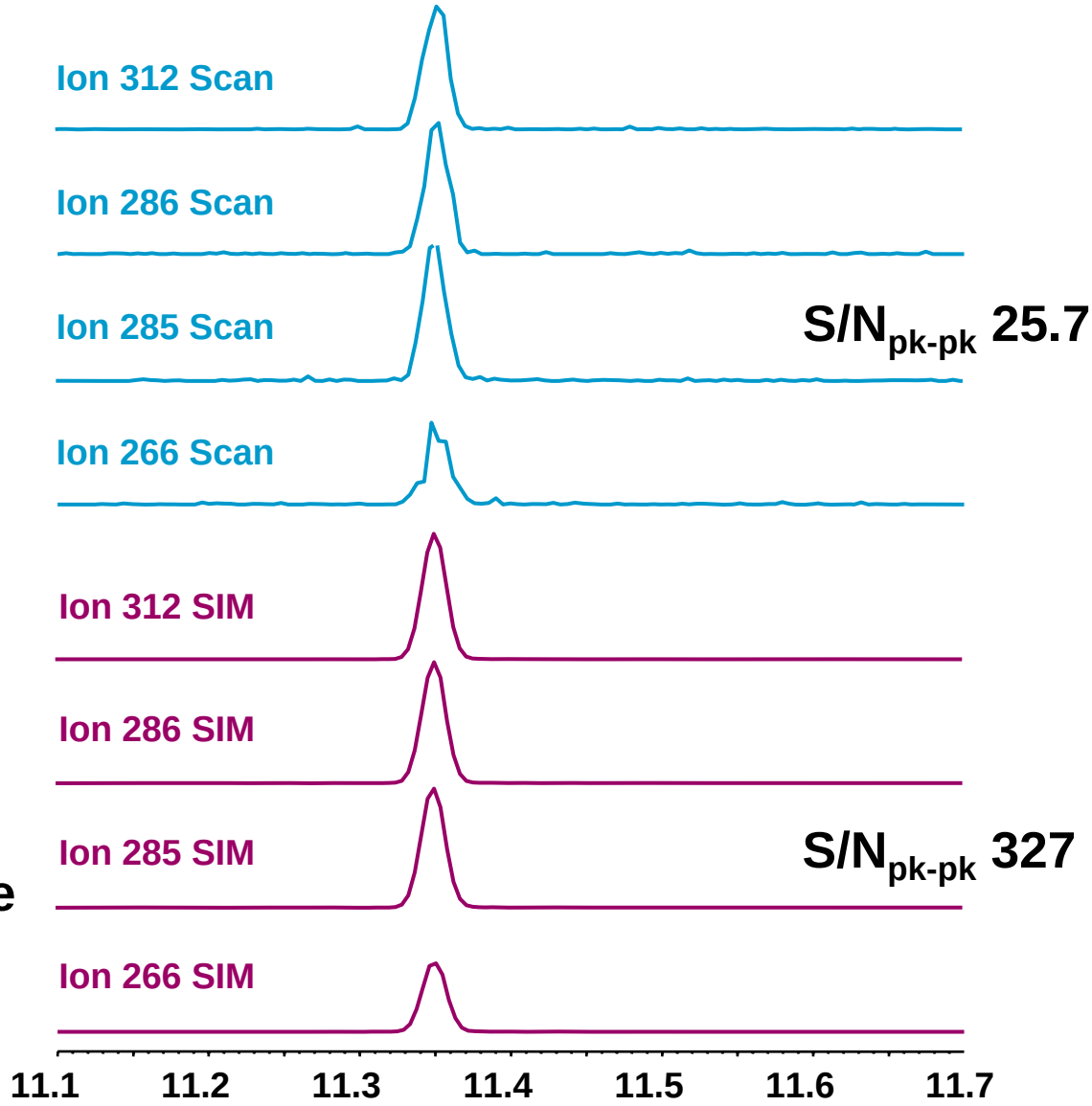


Extracted SIM Ions From SIM/Scan Mode
25 msec dwell

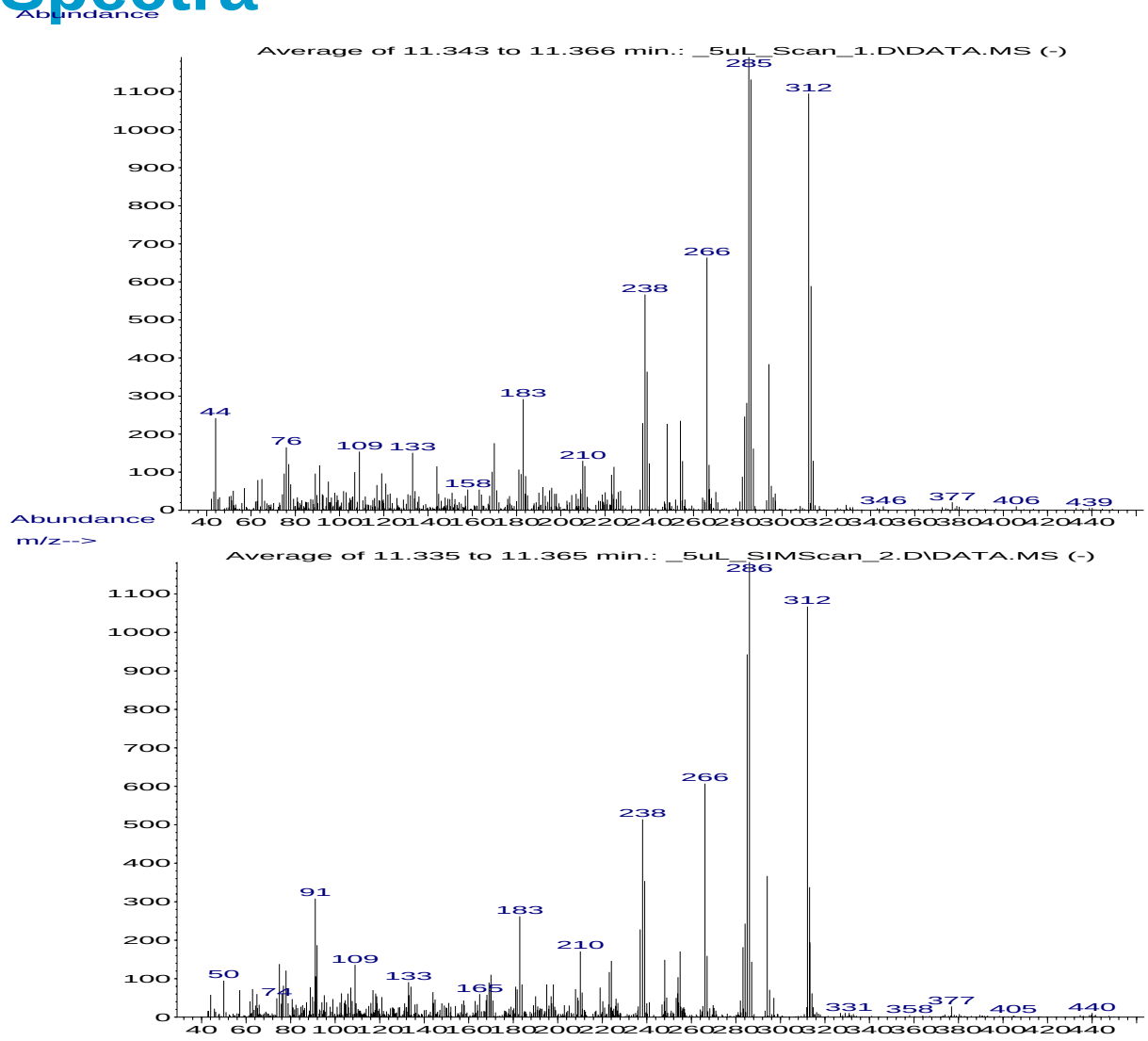
Flunitrazepam-Advantage for Quant with SIM/Scan 0.63 ng

Extracted Ions From Scan Mode

Extracted SIM Ions From SIM/Scan Mode



Synchronous SIM/Scan – Library Searchable Spectra



Flunitrazepam

Scan Only Spectra

NIST Forward Match 837

Spectra from SIM/Scan

NIST Forward Match 826

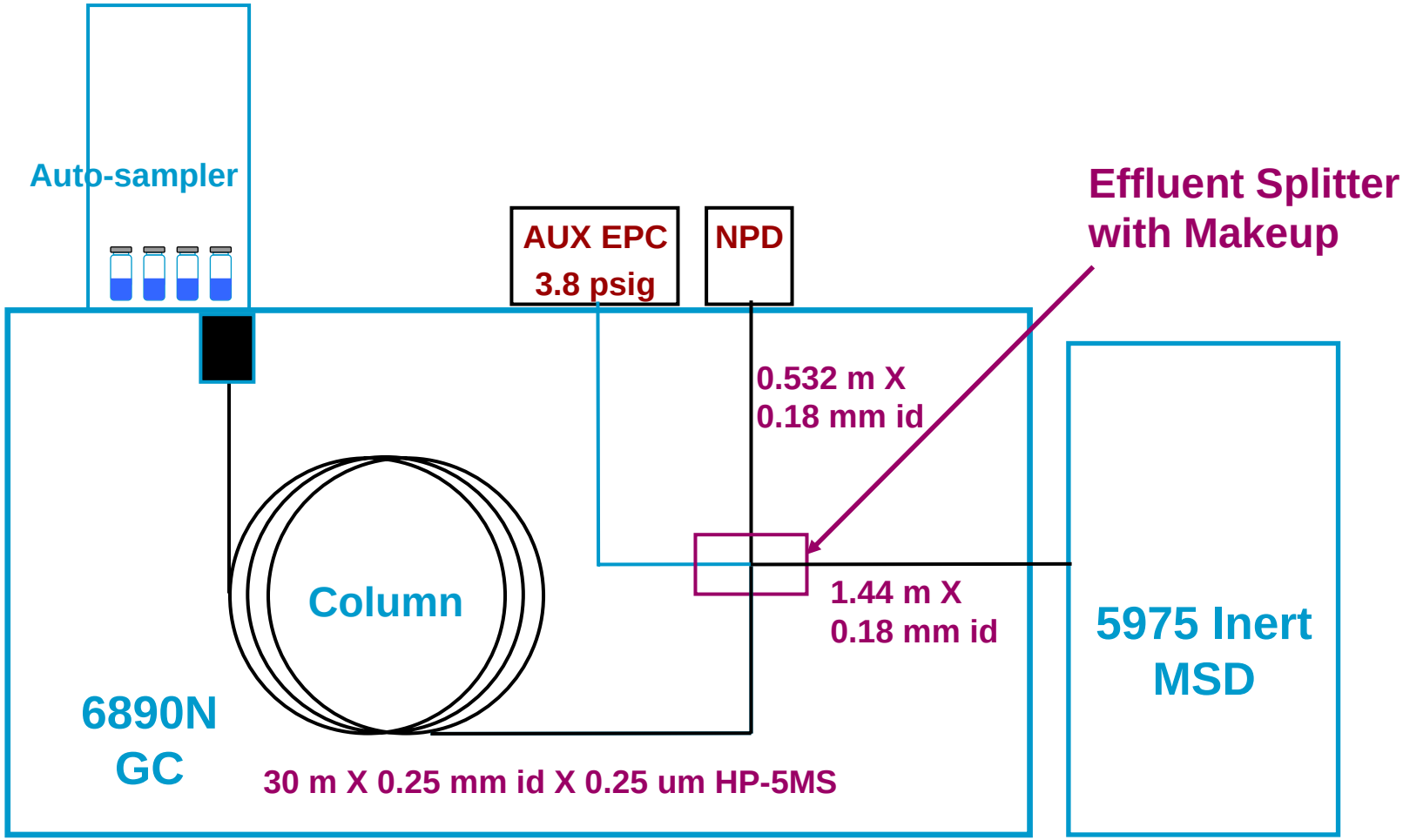
Signal to Noise Improvement with SIM/Scan

			A	B	C	D	D/A	
Ret Time	Compound	Tgt Ion	Scan Only S/N pk-pk	SIM/Scan Scan S/N pk-pk	SIM only S/N pk-pk	SIM/Scan SIM S/N pk-pk	Improvement factor	# ions in SIM
9.940	Oxazepam	267	43	49	687	676	16	4
10.338	Lorazepam	239	87	60	787	1098	13	7
10.490	Diazepam	256	242	388	2002	2638	11	7
11.353	Flunitazepam	285	50	42	461	597	12	4
12.327	Nitrazepam	280	6	12	115	116	18	8
12.733	Clonazepam	280	8	11	128	123	15	4
13.168	Alprazolam	308	18	24	173	192	11	4
							avg 13.6	

SIM and Scan performance of SIM/Scan is comparable to SIM only and Scan only modes

Permanent Splitter System GC/NPD/MSD

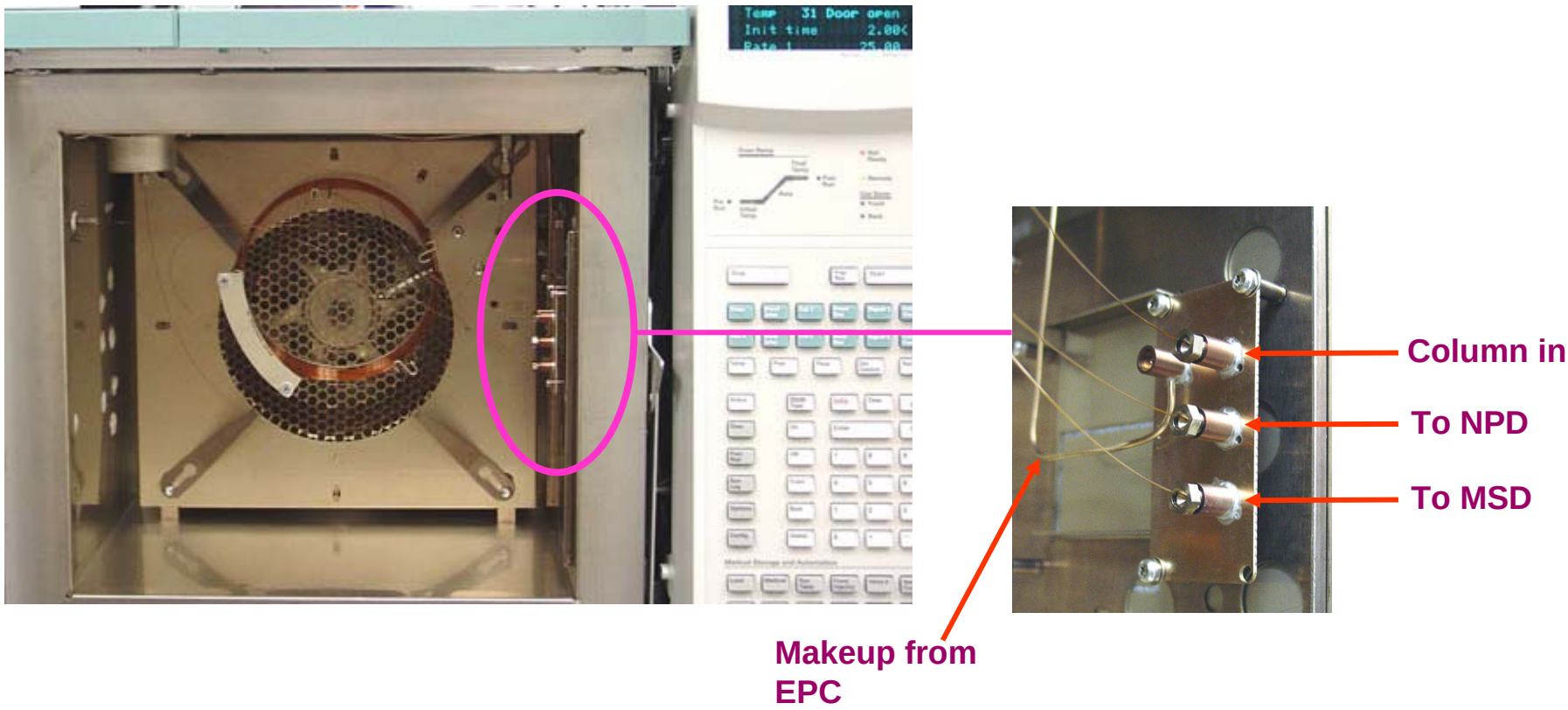
1:1 split NPD:MSD



uFluidic Splitter Hardware

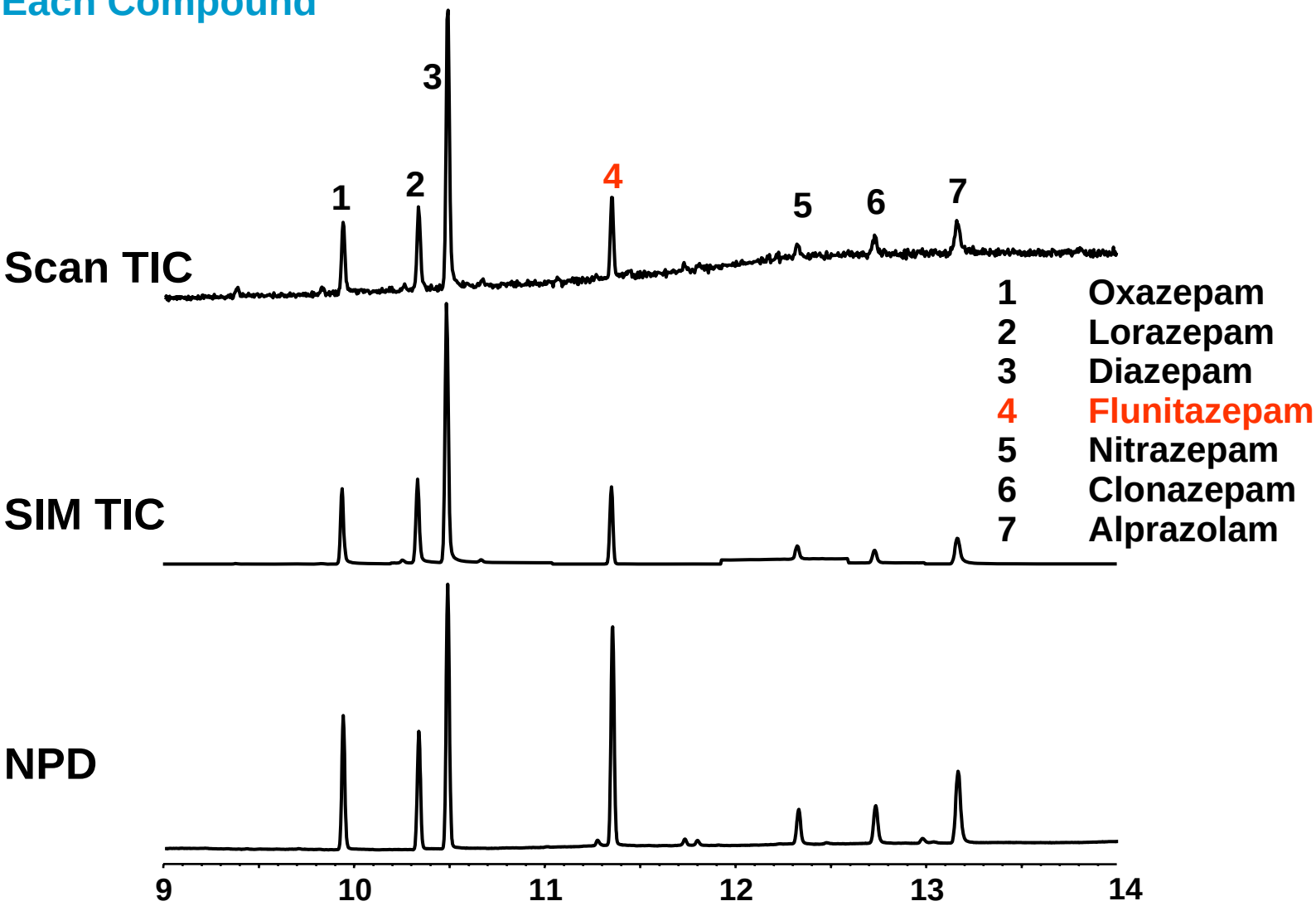
Deactivated microfluidic splitter operates to 350 C.

Uses metal ferrules to eliminate leaks and retightening



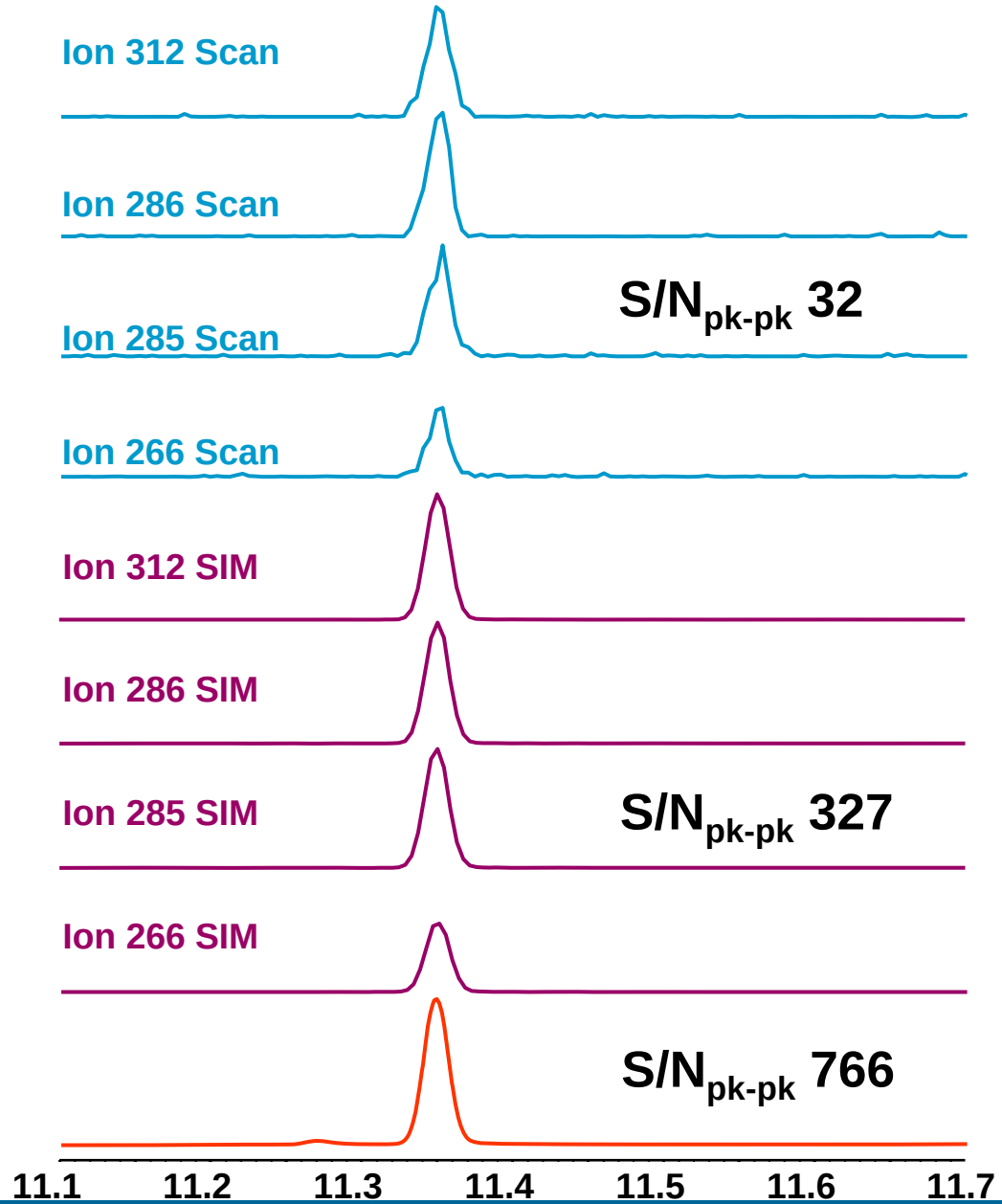
Three Signals from 1 Injection: Scan, SIM, and NPD

1.25 ng Each Compound



TIC: _5uL_SIMScan_2.D\DATA.MS

Flunitrazepam

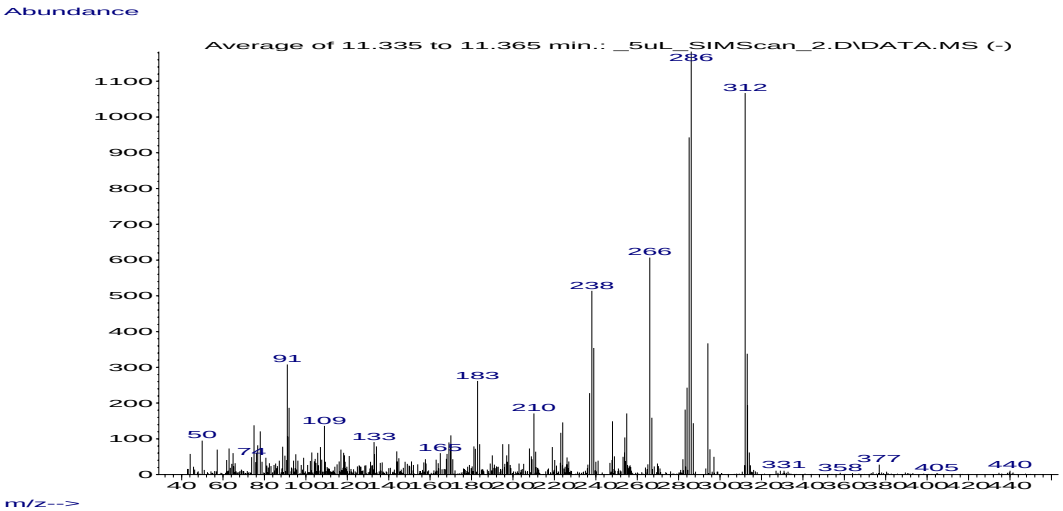


Extracted Ions From Scan Mode

Extracted SIM Ions From SIM/Scan Mode

NPD

Flunitrazepam 1.25 ng



Scan from SIM/Scan

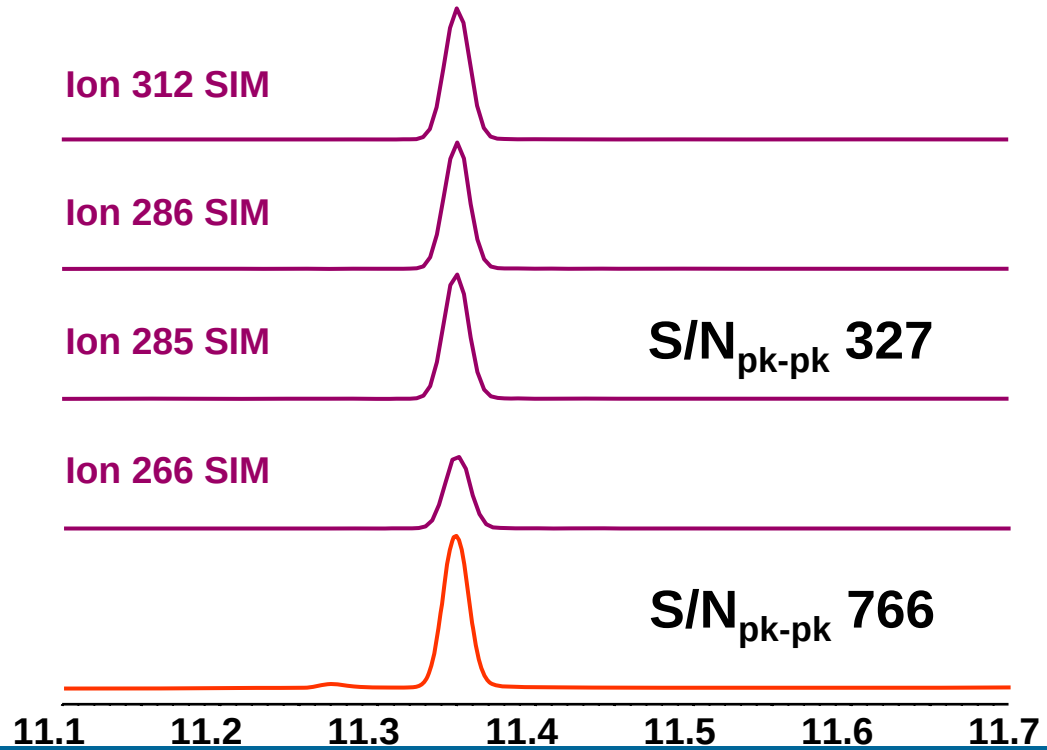
Use for confirmation

Extracted SIM Ions From SIM/Scan Mode

Use for quant

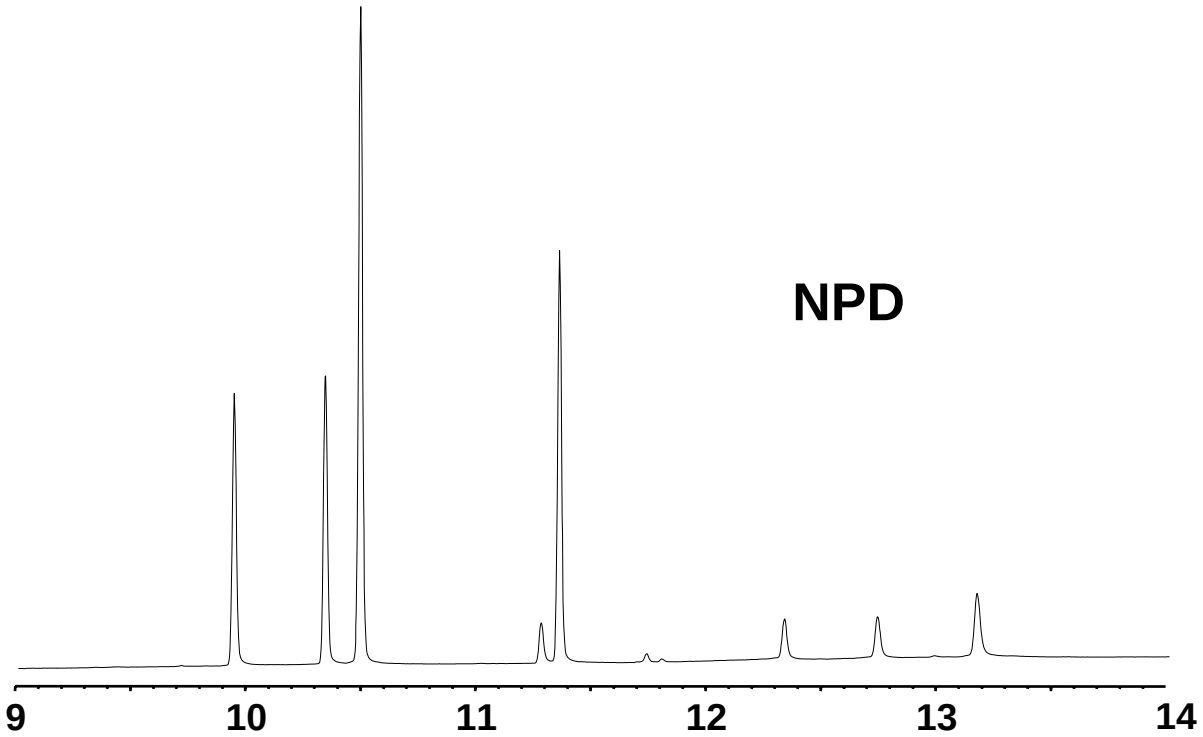
NPD

Use for confirmation and/or quant



Agilent Technologies

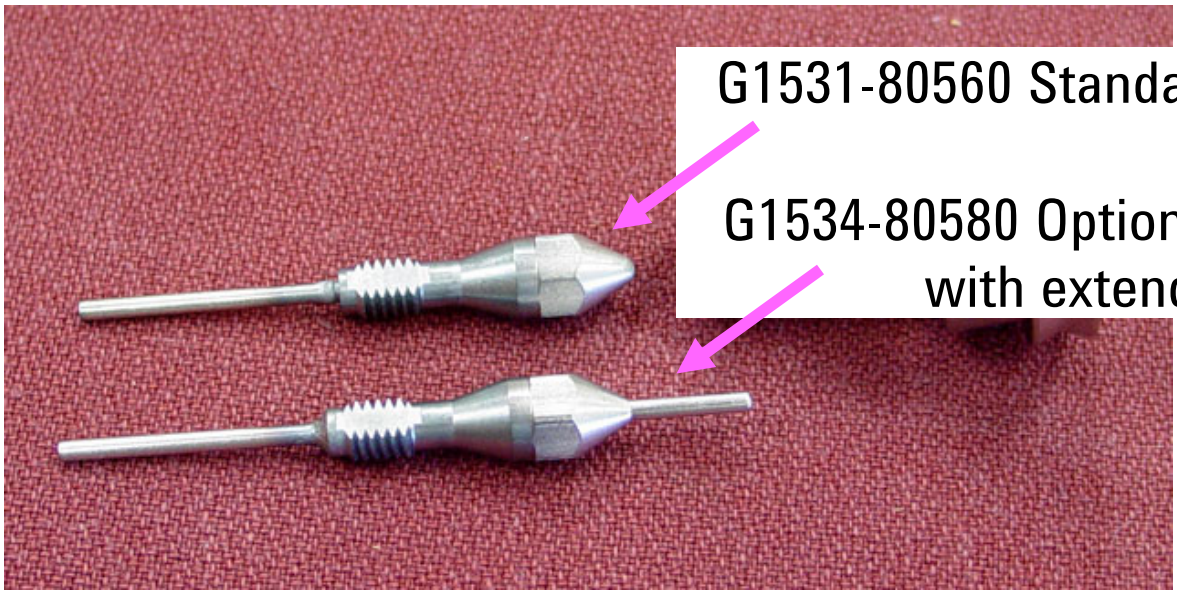
Why Does My NPD Peakshape Look So Good?



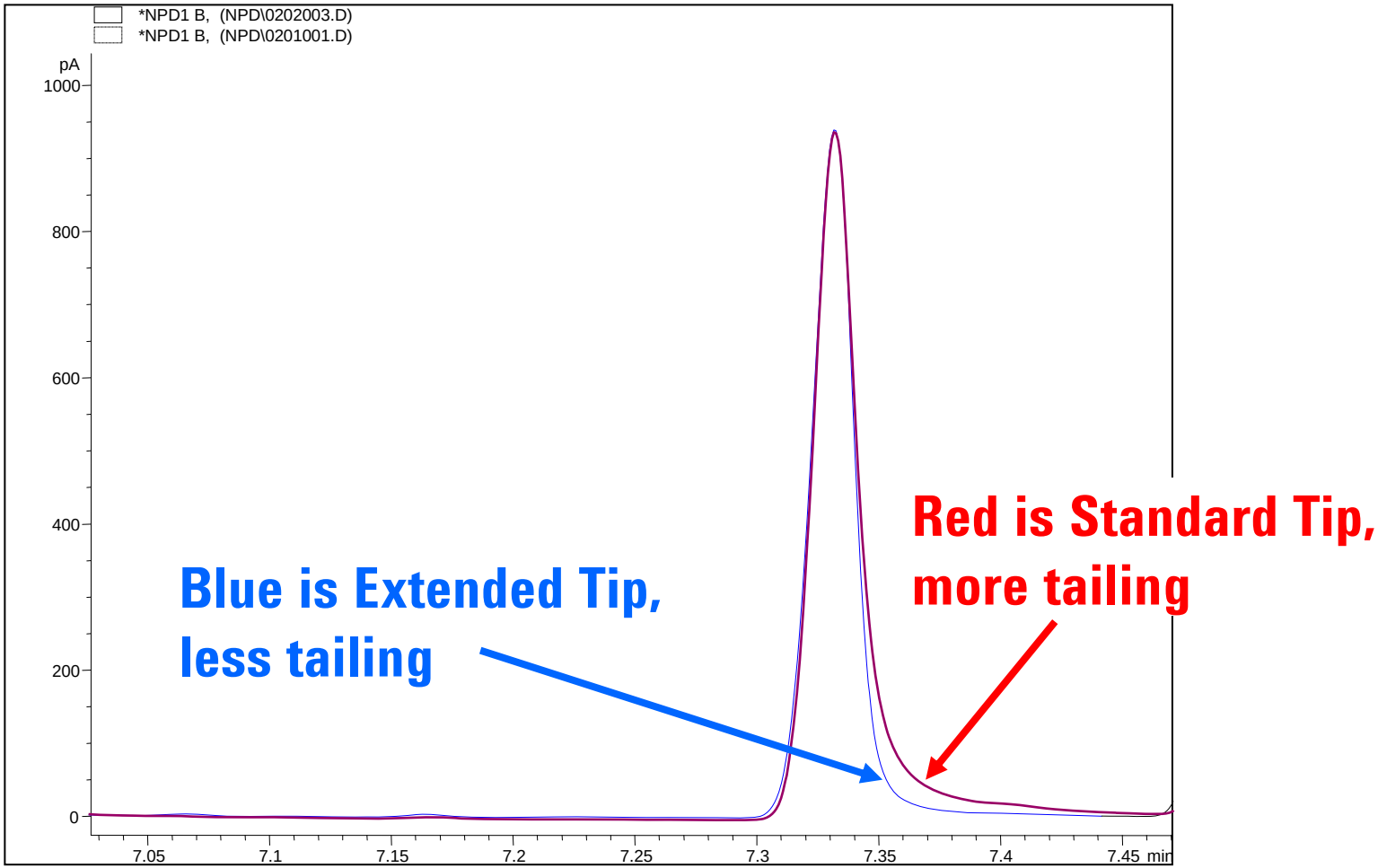
TIC: 25_NPDSIMScan2a.D\DATA.MS

NPD Jets, Standard vs Extended Tip

Extended Tip extends ~ 9mm
Analytes are positioned closer to the bead, minimizing tailing.



Drug Standard by NPD using Different Jets



NPD Jet For Improved Nitrogen on 6890

There is a much better jet for nitrogen detection with the NPD:

- G1534-80580 NPD Jet Capillary Det
- G1534-80590 NPD Jet Universal Det
- G1530A-29 Service Note that describes best practices for optimal NPD operation
- [note: disregard suggested use of narrower collector]

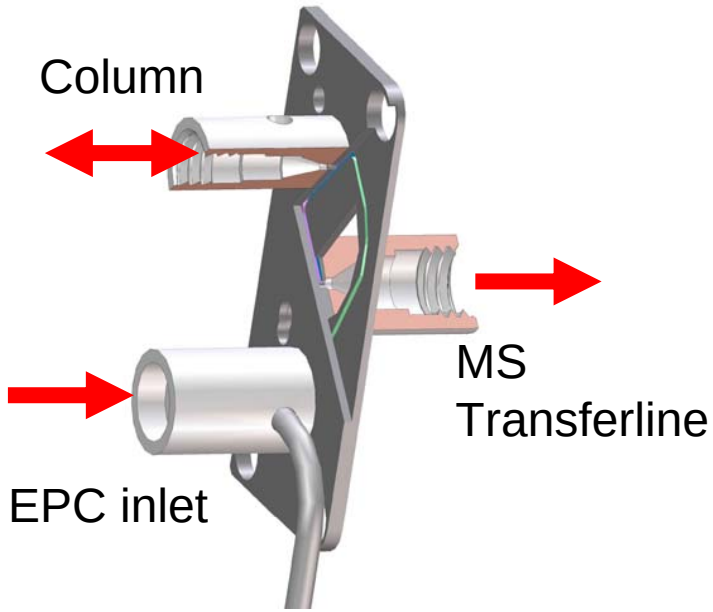
QuickSwap MSD Interface – Ease of Use

Remove column w/o venting

Backflush mode

- Inlet maintenance reverse flow
- Removes heavies from column

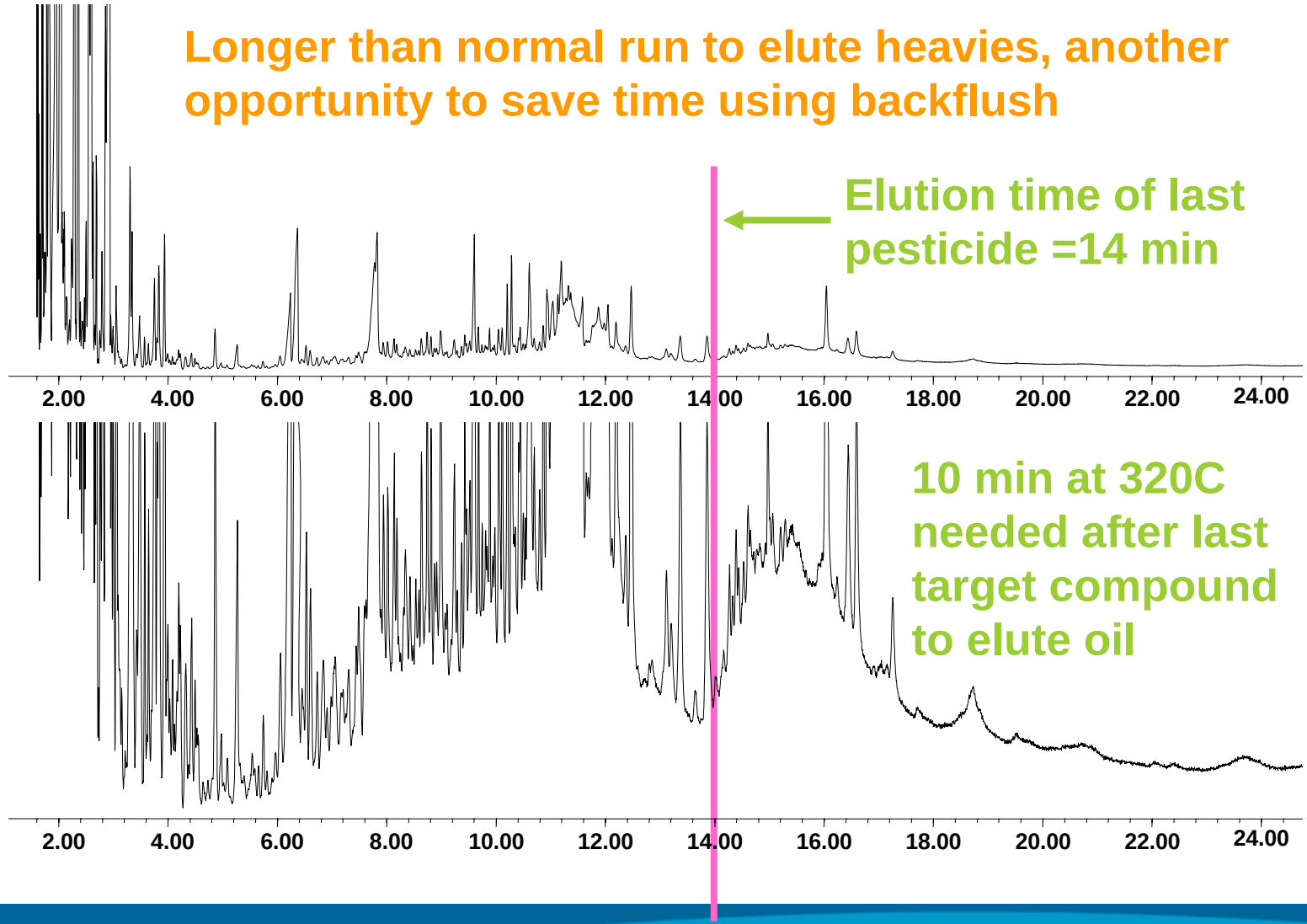
Maintain constant flow to MSD



Microfluidic technology

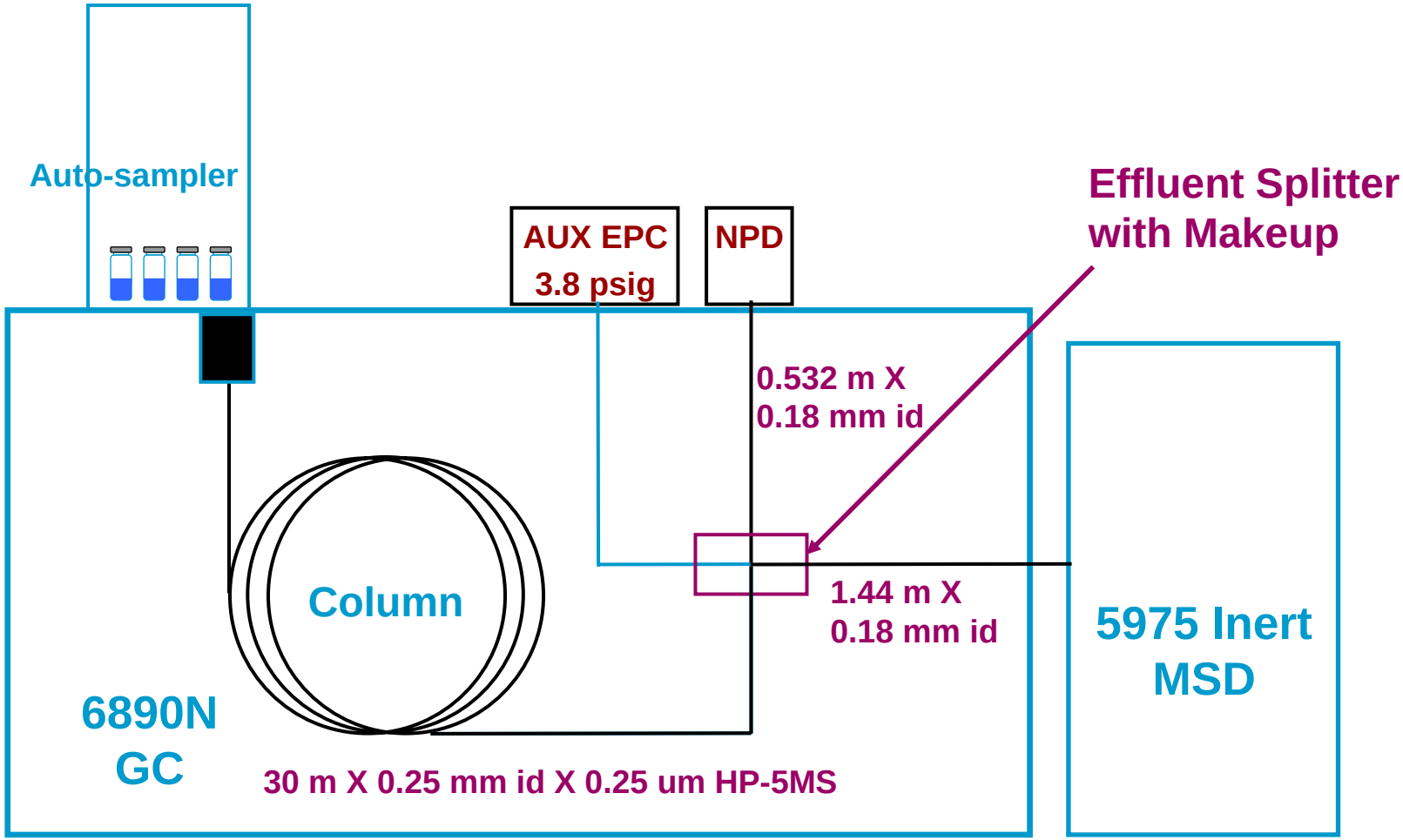
Requires performance turbo for flow rates > 2 cc/min

3x Speed, 15m, Lemon Oil TIC, no spike



Permanent Splitter System GC/NPD/MSD

1:1 split NPD:MSD



How Do You Backflush ?

Program inlet pressure to ~1.0 psi (may have to fine tune)

Program make-up pressure to calculated value after last target peak elution time

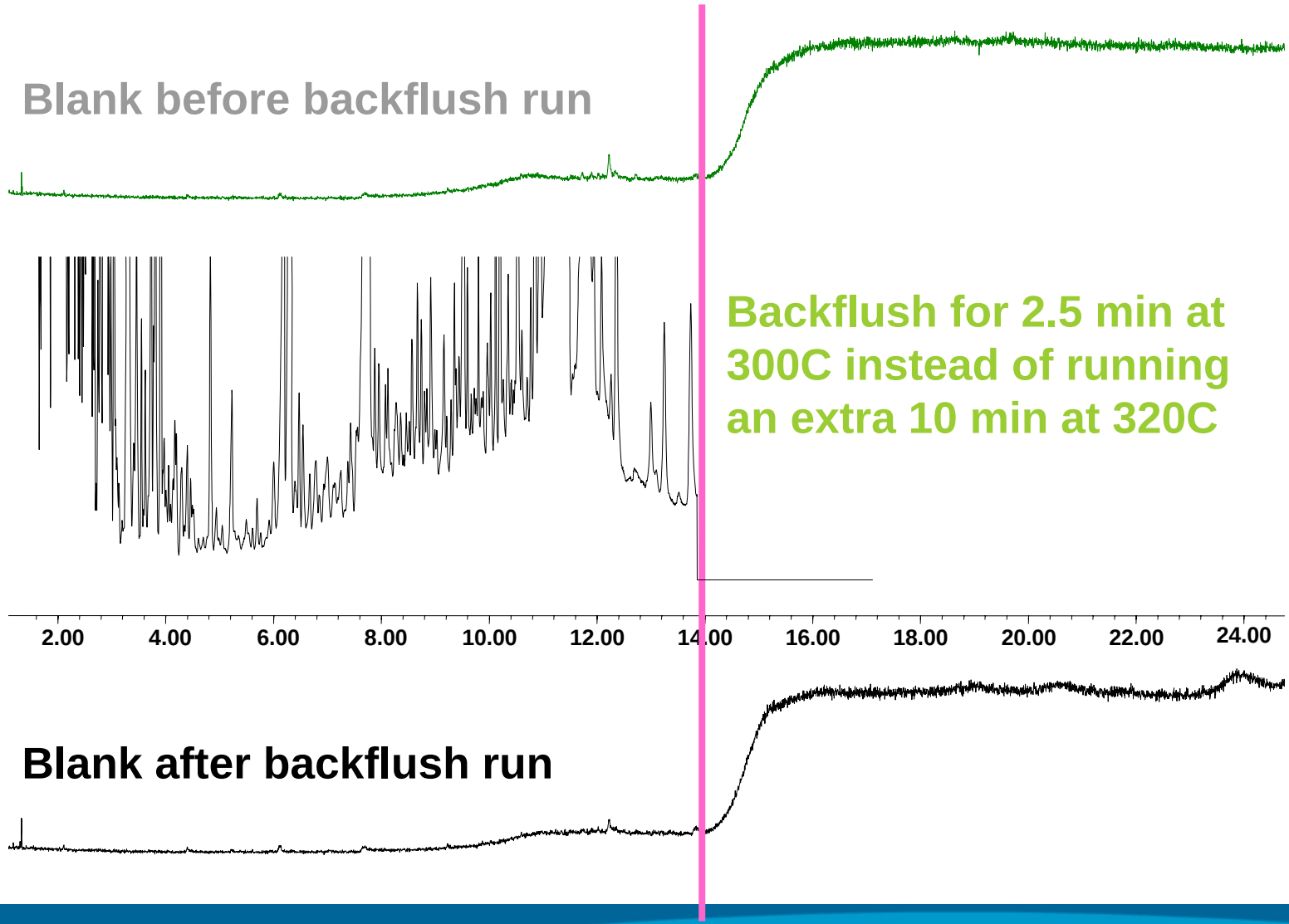
- Use flow calculator to get pressure for make-up to splitter (aux EPC or PCM). MS restrictor flow < 10 mL/min at backflush temp

Try a 3 void-times backflush, then run a blank for an extended time to make sure system is clean

Shorten this time if blank is clean, in 0.5 min steps

Performance Turbo a must

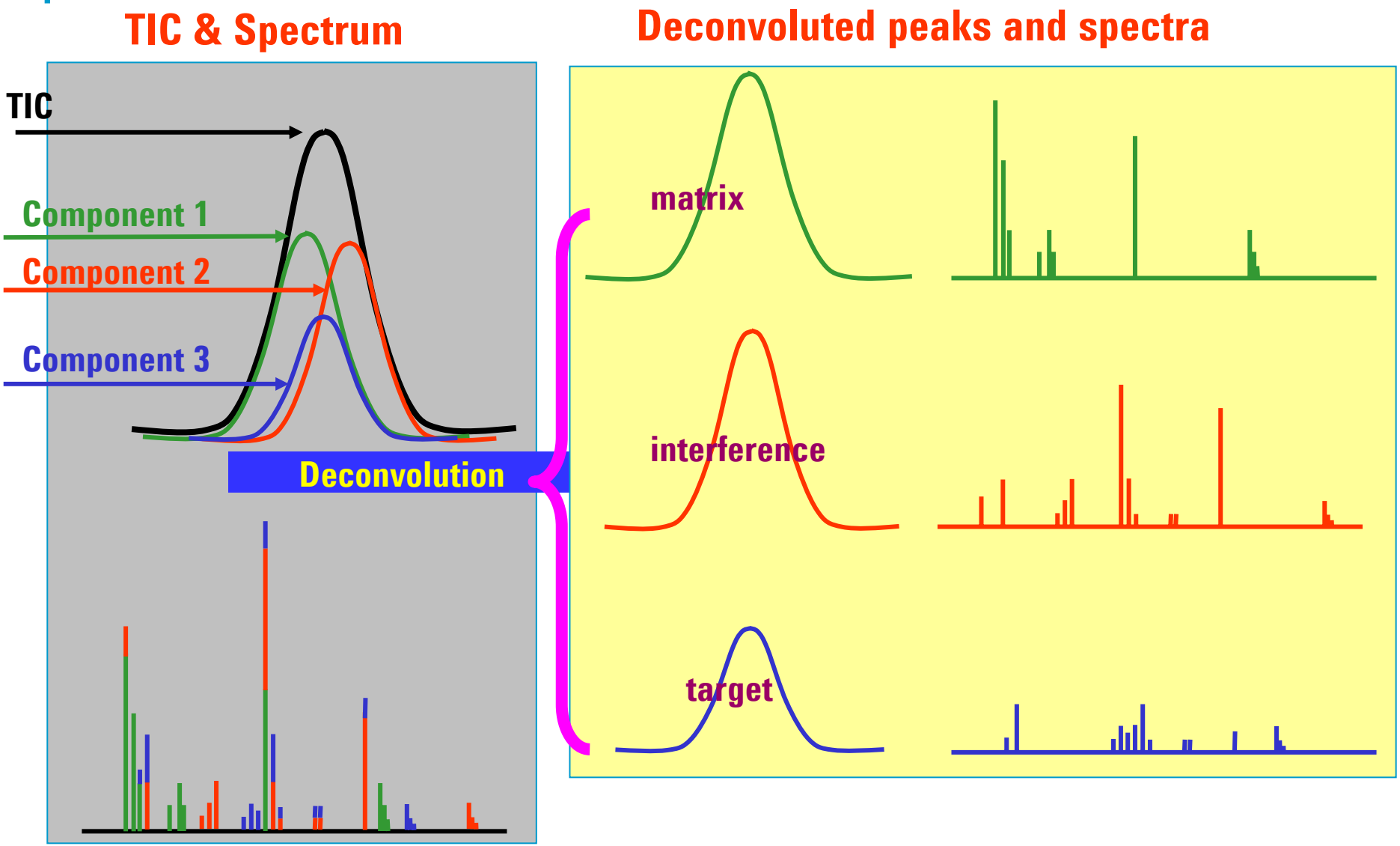
3x Speed, 15m, Lemon Oil TIC, no spike



Spectral Deconvolution

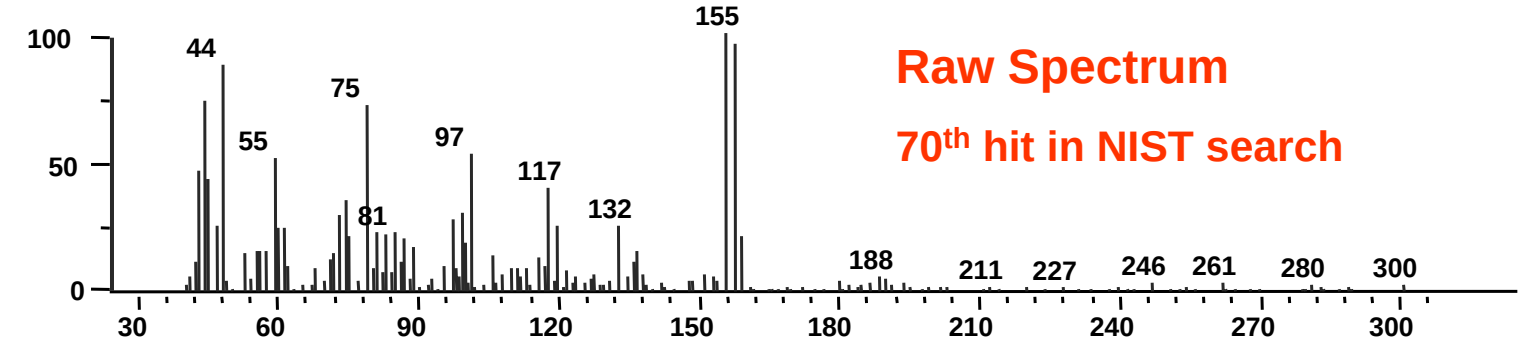
- Extracts cleaner mass spectra from overlap situations – easier, reliable identification
- Faster data review, a free second opinion from a mass spec expert
- When coupled with RTL and NPD data, gives higher quality identifications
- Can run searches of data not restricted by RT to find “spectral brethren” like designer drugs

AMDIS Deconvolution Pulls Out Individual Components and their Spectra



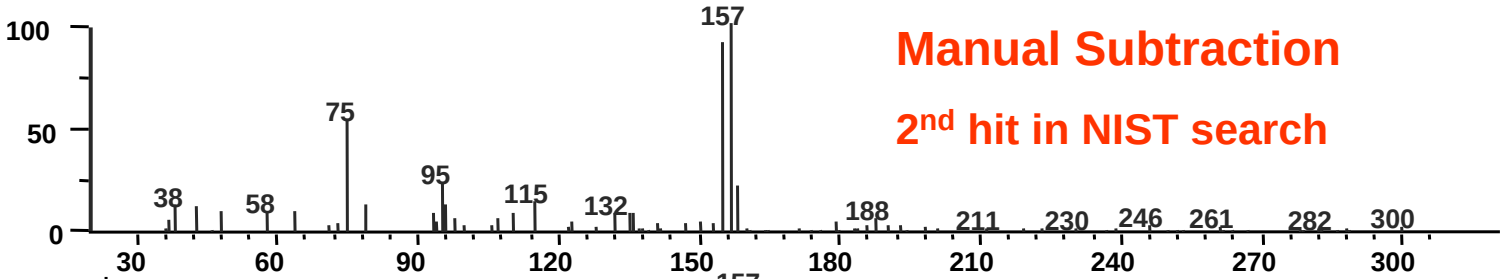
Deconvolution Removes Spectral Interferences

3-Chloro-1,2-dibromopropane overlapped with fuel components



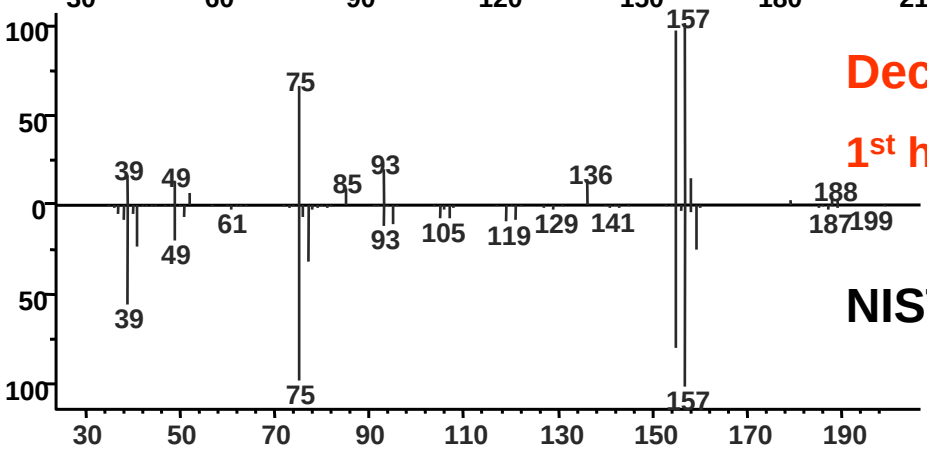
Raw Spectrum

70th hit in NIST search



Manual Subtraction

2nd hit in NIST search

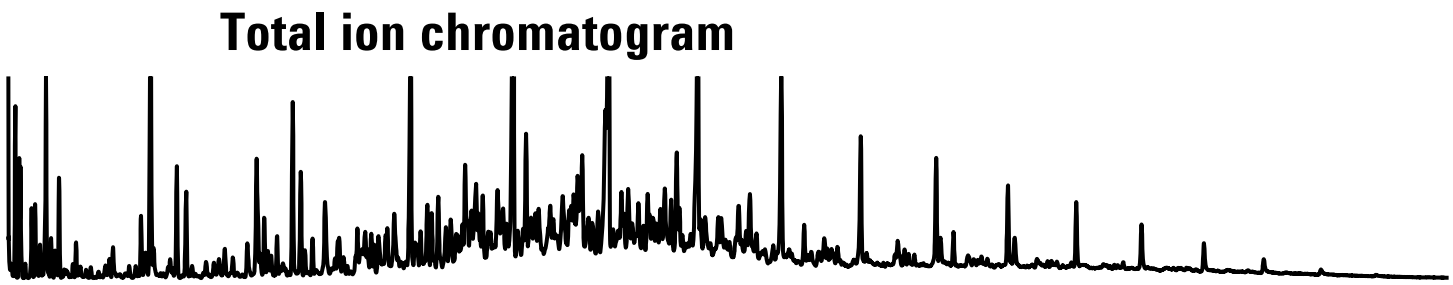


Deconvolved Spectrum

1st hit in NIST search

NIST 05 Library

DRS: 3 Integrated Processes



Targets are identified by locked R.T.s and 3 qualifier ions, then quantified using target ion area vs ISTD cal table

AMDIS 32 deconvolutes spectra and searches target MS database using locked RT as a qualifier

Deconvoluted Target spectra confirmed by AMDIS, searched against NIST02 MS database

Quant Results

Confirmed AMDIS hits

Confirmed NIST05 hits

Combined quantitative and qualitative HTML Summary report

DRS Report

MSD Deconvolution Report
Sample Name: 2 ppmVOA MIX 3 Only
Data File: C:\msdchem\1\DATA\042205AMHS_SimScan\2ppmMIX 3_Only_simscan.D
Date/Time: 05:13:03 PM Friday, Dec 9 2005

The NIST library was searched for the components that were found in the AMDIS target library.

			Agilent	AMDIS		NIST	
R.T.	Cas #	Compound Name	ChemStation Amount (ng)	Match	R.T. Diff sec.	Reverse Match	Hit Num.
1.497	107062	1,2-Dichloroethane	2.27	97	0.6	94	1
1.540	563586	1,1-Dichloropropylene	7.6	100	0.5	96	1
1.867	78875	1,2-Dichloropropane	4.92	95	0.7	90	1
1.871	79016	Trichloroethylene	7.58	99	0.6	91	1
2.330	10061015	cis-1,3-Dichloropropylene	4.39	98	1.0	92	2
2.677	10061026	trans-1,3-Dichloropropylene	3.3	97	1.5	94	1
2.758	79005	1,1,2-Trichloroethane	2.82	99	1.7	92	1
2.961	142289	1,3-Dichloropropane	3.39	98	1.5	92	1
3.273	106934	1,2-Dibromoethane	2.6	91	1.5	76	4
4.032	630206	1,1,1,2-Tetrachloroethane	5.15	100	1.9	94	1
5.187	79345	1,1,2,2-Tetrachloroethane	2.38	99	2.3	89	1
5.322	96184	1,2,3-Trichloropropane	1.89	98	2.5	94	1
6.323	76017	pentachloroethane	0.08	63	2.0	76	1
8.232	96128	3-Chloro-1,2-dibromopropane	1.62	93	1.6	87	1
10.439	87683	hexachlorobutadiene	16.46	94	0.9	95	1

Summary

The 5975 and associated new products bring several new tools to the forensic analyst:

- Auto-CI
- SIMScan
- Splitters
- DRS