7890-5975 MSD DRS Semivolatiles Analyzer, Minimizing Start-up Time

Hardware, Software and Methods Details

SP 7890-0459

Mike Szelewski

2010.09.28

What is an Analyzer ?

1. A complete system designed for an application
2. Analyzers available with GC only, GC-MSD and GC-QQQ
3. Includes hardware, software and consumables
4. Has a special configuration, such as backflushing
5. Pre-assembled at the manufacturing division
6. Pre-tested as a system, including GC and MSD
7. Retention time locked
8. Usually a Single Level Calibration
9. Service Engineers specifically trained in Analyzers
10. On-site installation more streamlined and foolproof
11. Start-up time minimized

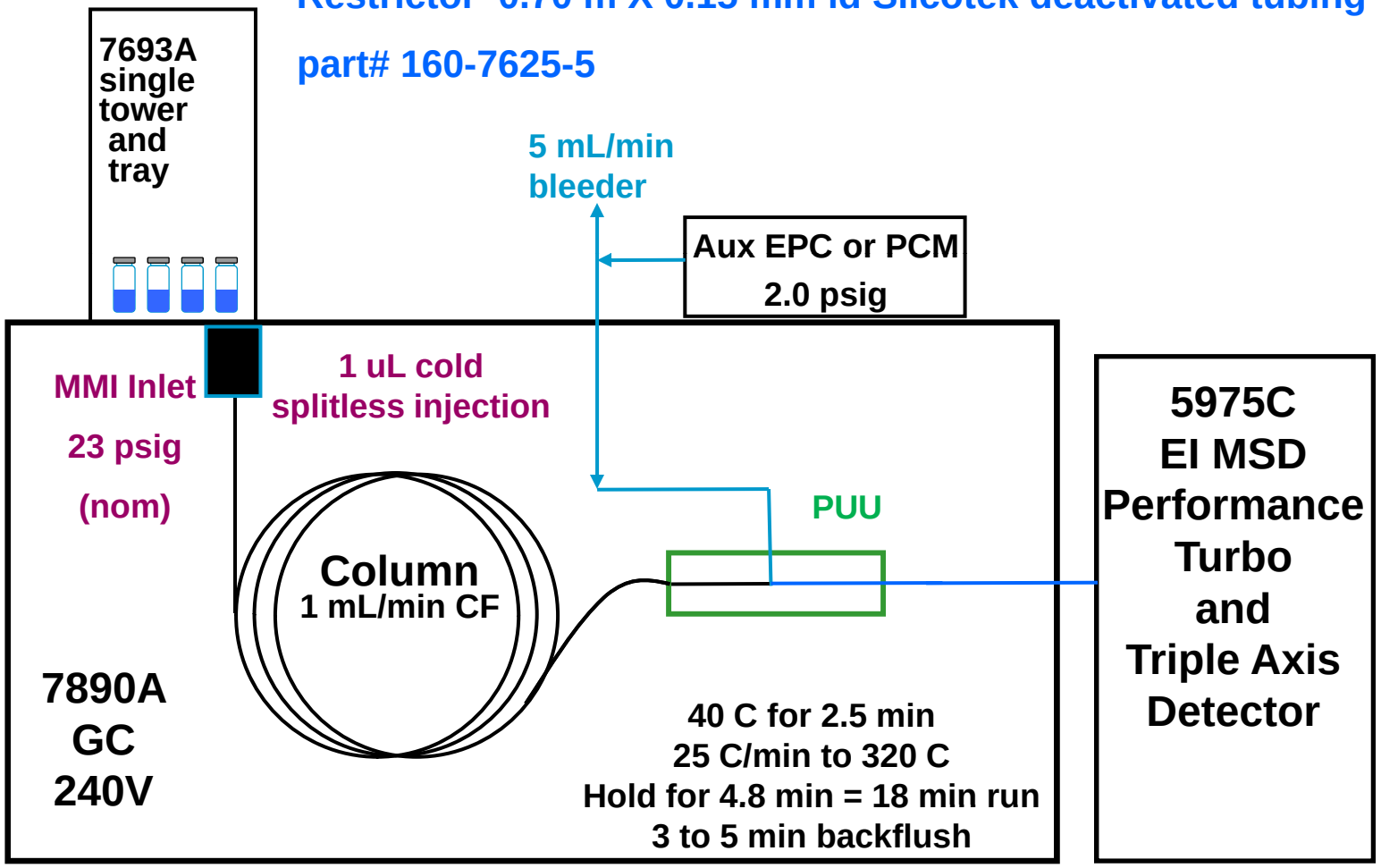
Semivolatiles Analyzer Overview

1. A generic system for the analysis of environmental semivolatile compounds (not USEPA 8270, 525 nor 625 method specific)
2. 7890A 5975C 7693A based
3. Multimode Inlet – best active compound performance
4. Post column Capillary Flow Technology - backflushing
5. Deconvolution Reporting Software for Identification and Quantitation
6. Semivolatiles Database/Library Includes spectra and locked retention times for 338 single component analytes
7. Single Level Calibration – 10 ppm (ug/mL, ng/uL)

Semivolatiles Analyzer Hardware

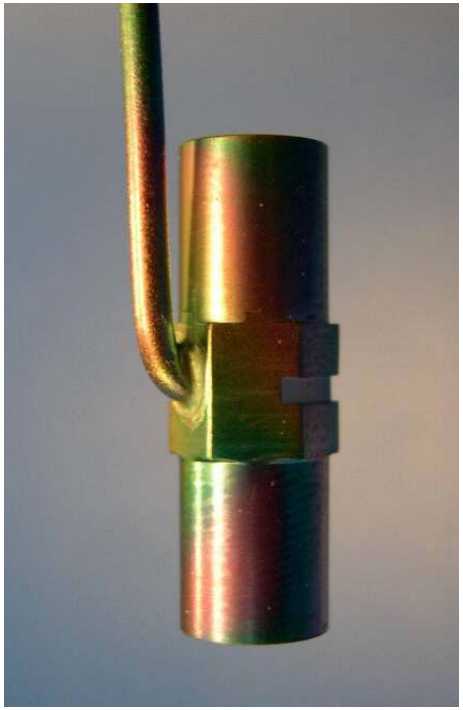
Column 20 m X 0.18 mm id X 0.36 um DB-5MS UI part# 122-5533 UI

Restrictor 0.70 m X 0.15 mm id Silcotek deactivated tubing part# 160-7625-5



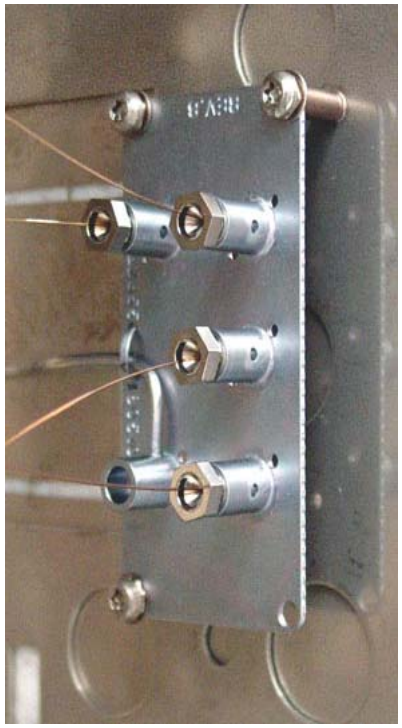
Any One of these three CFT Devices can be used in the Semivolatiles Analyzer

Purged Ultimate Union with Makeup



2-Way Splitter with Makeup

3-Way Splitter with Makeup



Semivolatiles Analyzer System Details

1. 7890A GC
   a. 240 volt
   b. Aux EPC or PCM for makeup at 2.0 psi constant pressure
   c. Additional GC detectors can be used with a splitter

2. 5975C MSD
   a. EI only
   b. Performance Turbo
   c. Recommend mounting MSD in rear position for easier access to MMI
   d. Triple-Axis detector used for some spectra
   e. Autotune used (not DFTPP tune which is USEPA specific)

3. 7693A Autosampler Tower and Tray
   a. Single tower only
   b. Fast injection
   c. Sample prep software is not yet released on MSD Chemstation

Semivolatiles Analyzer System Details

1. MMI (Multimode Inlet)
   a. 1 uL Cold Splitless injection for best active compound performance
      20°C-0.05 min-500°C/min-320°C hold
   b. Liquid CO_2 or liquid N_2 required for cooling
   c. Pulsed at 44 psi for 1.4 min – improves active compounds
   d. Septum purge off until 1.4 min, then 3 mL/min (run table event) - improves early eluter peak shape and response
   e. Splitless purge on time 1.4 min, 50mL/min
   f. Extra 1.1 minutes built in to allow large volume injection (LVI) - oven ramp starts at 2.5 minutes

2. CFT (Capillary Flow Technology)
   a. Purge Ultimate Union, but splitter also available
   b. Bleeder via tee on top of oven (outside) with fixed restrictor
      1. Acts as a pressure relief for RTLocking and/or pulsed injections
      2. Aux and PCM operate better with higher flows vs 0.5 mL/min makeup
   c. 80 psi backflush pressure for -2.5 mL/min at 320°C

MMI , N-nitrosodimethylamine, earliest eluter at 3.83 min

1 Hot Splitless

2 Pulsed Hot Splitless

3 Pulsed Hot Splitless with SPC

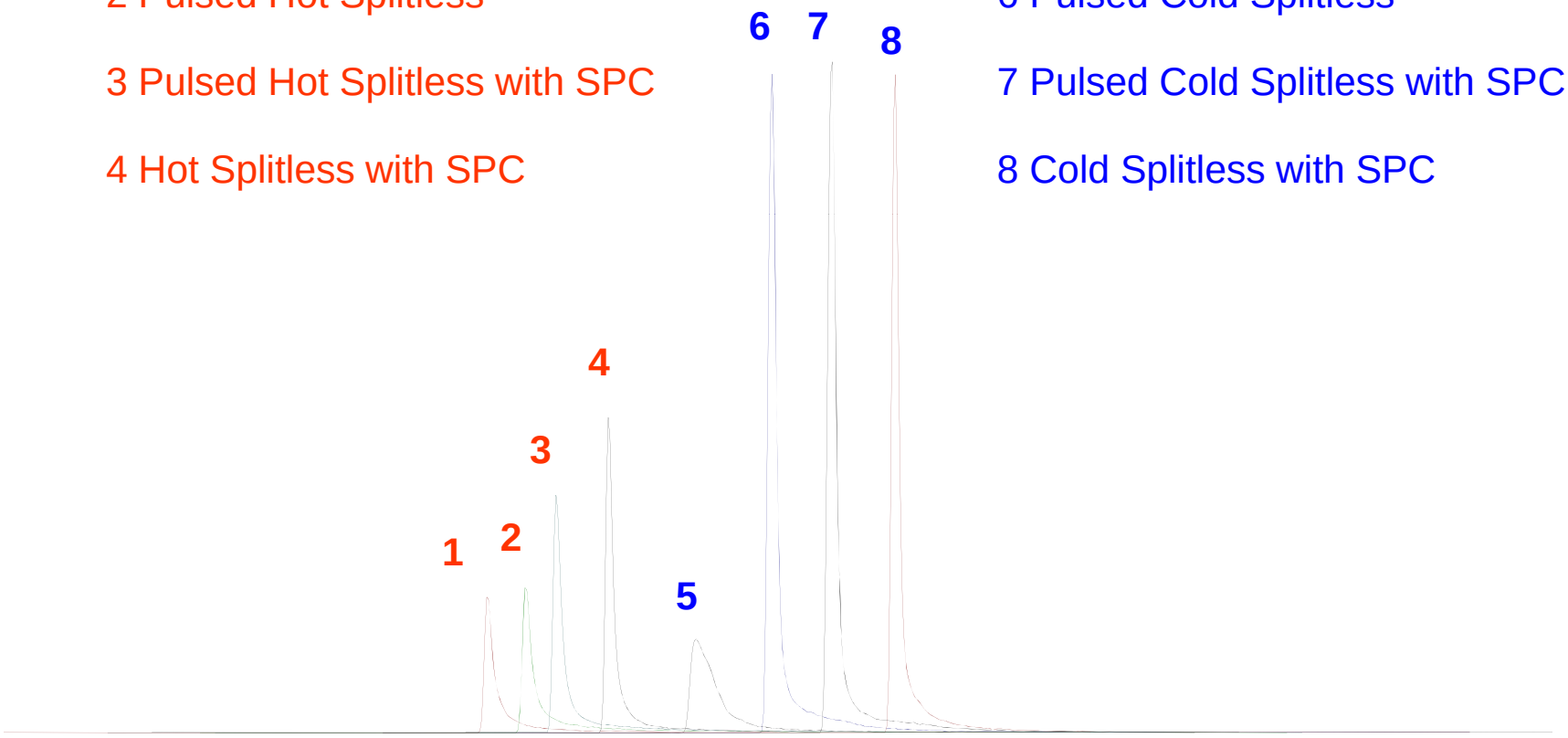
4 Hot Splitless with SPC

5 Cold Splitless

6 Pulsed Cold Splitless

7 Pulsed Cold Splitless with SPC

8 Cold Splitless with SPC



Data intentionally offset on y-axis

MMI , Pentachlorophenol, 10.8 min

1 Hot Splitless

2 Pulsed Hot Splitless

3 Pulsed Hot Splitless with SPC

4 Hot Splitless with SPC

5 Cold Splitless

6 Pulsed Cold Splitless

7 Pulsed Cold Splitless with SPC

8 Cold Splitless with SPC

An active compound where cold splitless is better than the best hot splitless

MMI , Perylene-d12, 15.3 min

1 Hot Splitless

2 Pulsed Hot Splitless

3 Pulsed Hot Splitless with SPC

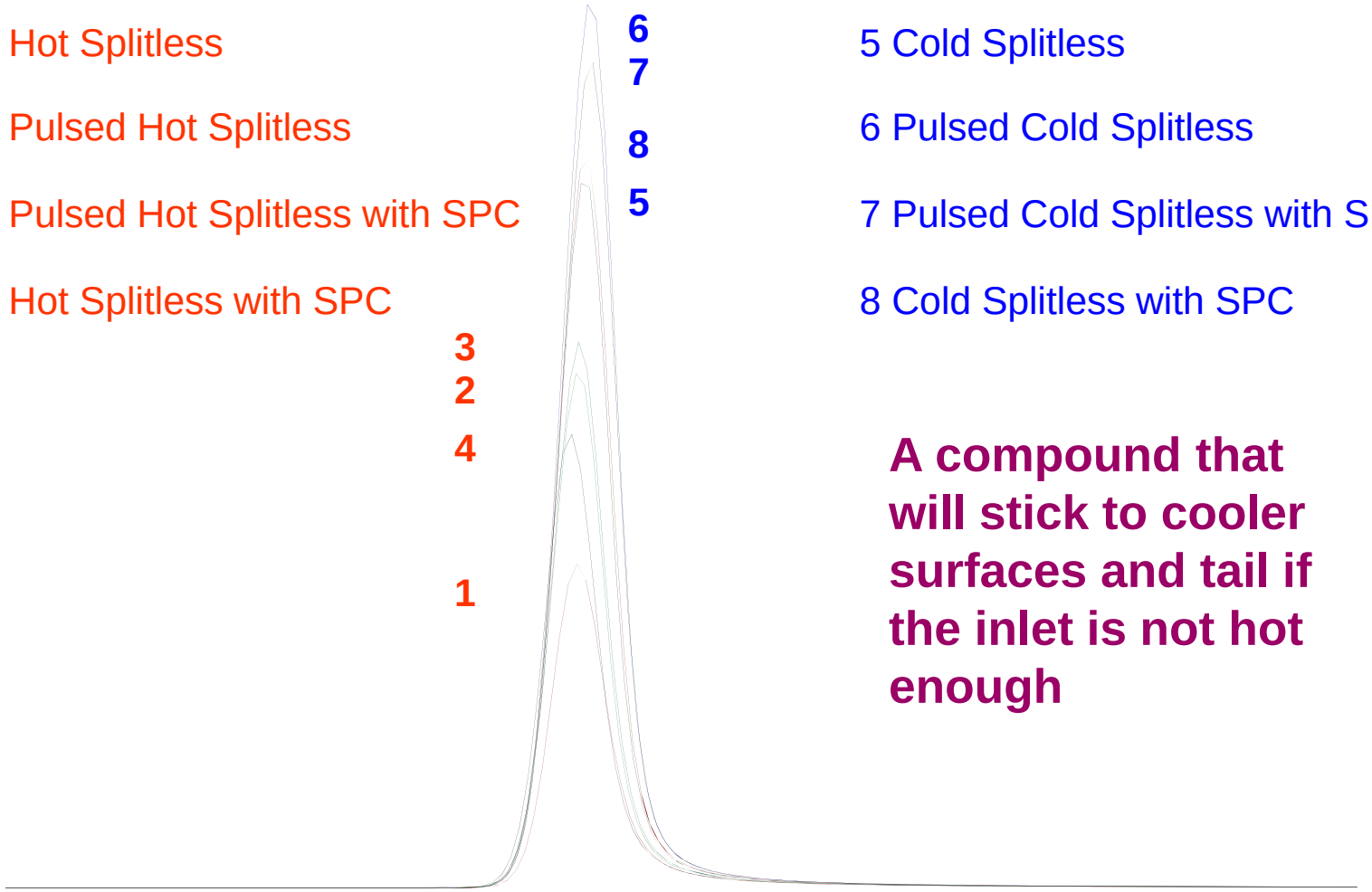
4 Hot Splitless with SPC

5 Cold Splitless

6 Pulsed Cold Splitless

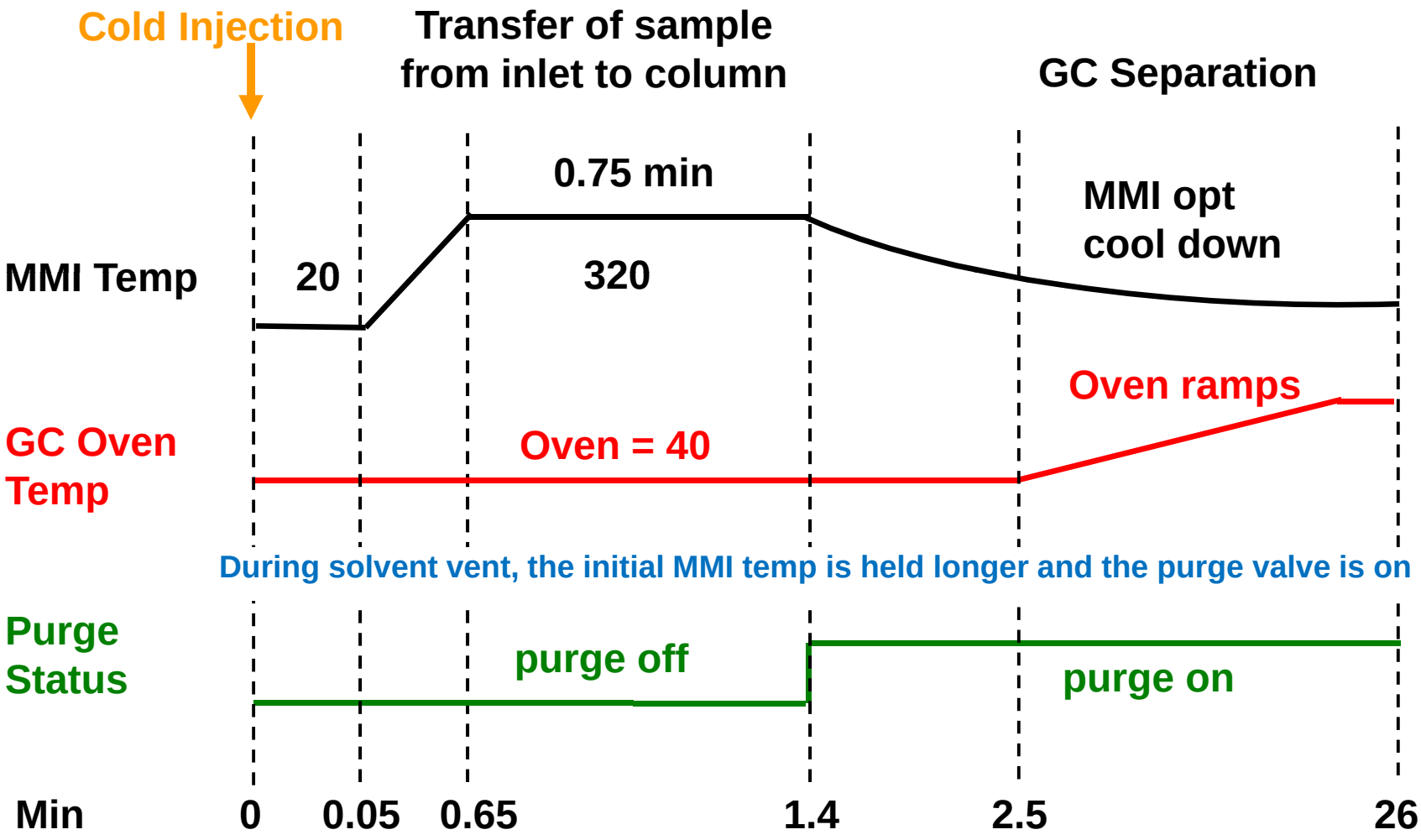
7 Pulsed Cold Splitless with SPC

8 Cold Splitless with SPC



A compound that will stick to cooler surfaces and tail if the inlet is not hot enough

MMI Cold Splitless and SV – Temperature and Flow Programs



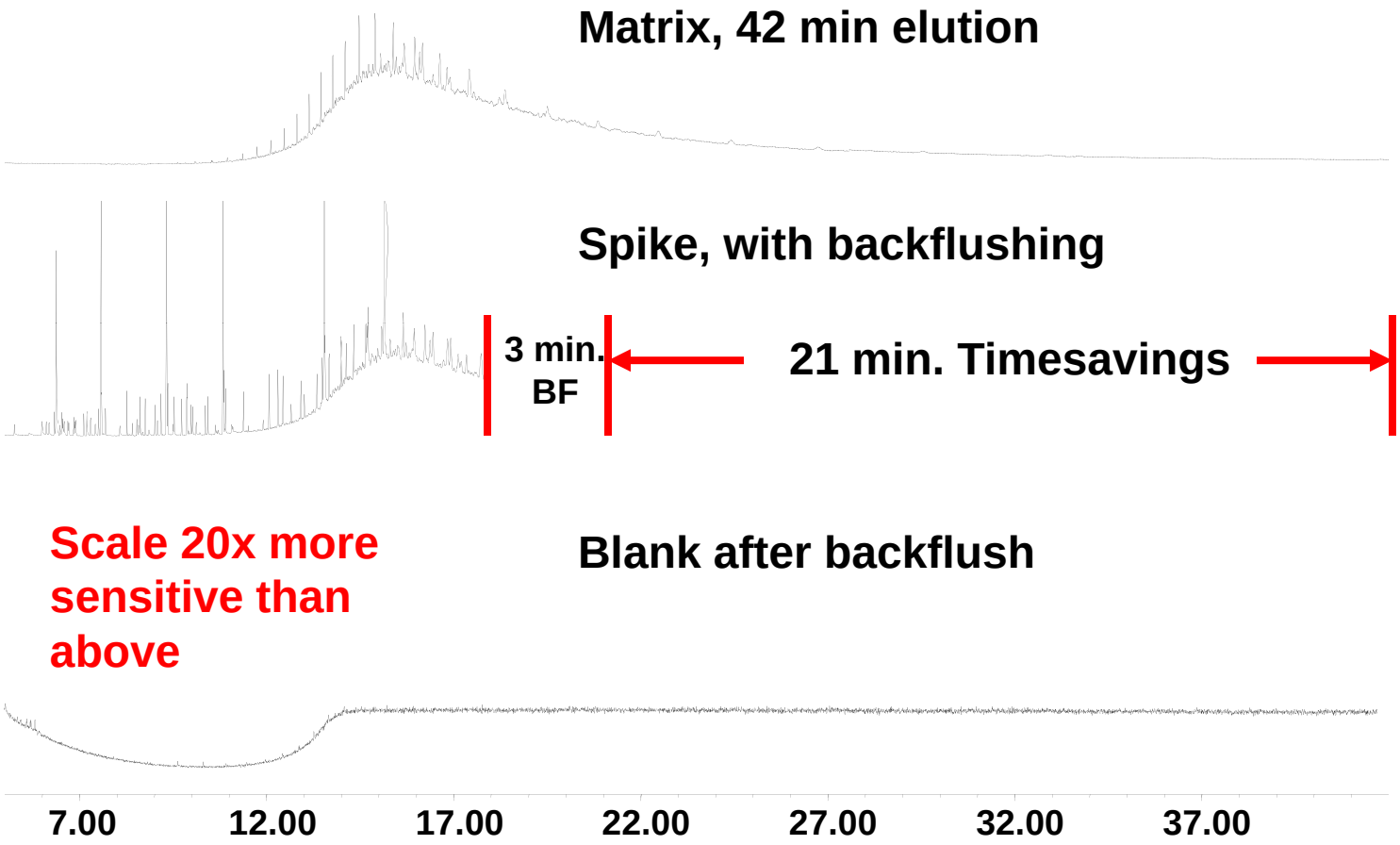
Semivolatiles Analyzer System Details

1. Column and oven
    a. 20 m x 0.18 mm x 0.36 um DB-5MS UI, part# 122-5533UI
    b. 40°C-2.5 min-25°C/min-320°C-4.8 min = 18.5 min acquisition
    c. Backflush for 3 to 5 min at -2.5 mL/min
        1. Inlet pressure during backflush set by system based on -2.5 mL/min flow
    d. RTLocked to Phenanthrene-d10 at 11.000 min, constant flow

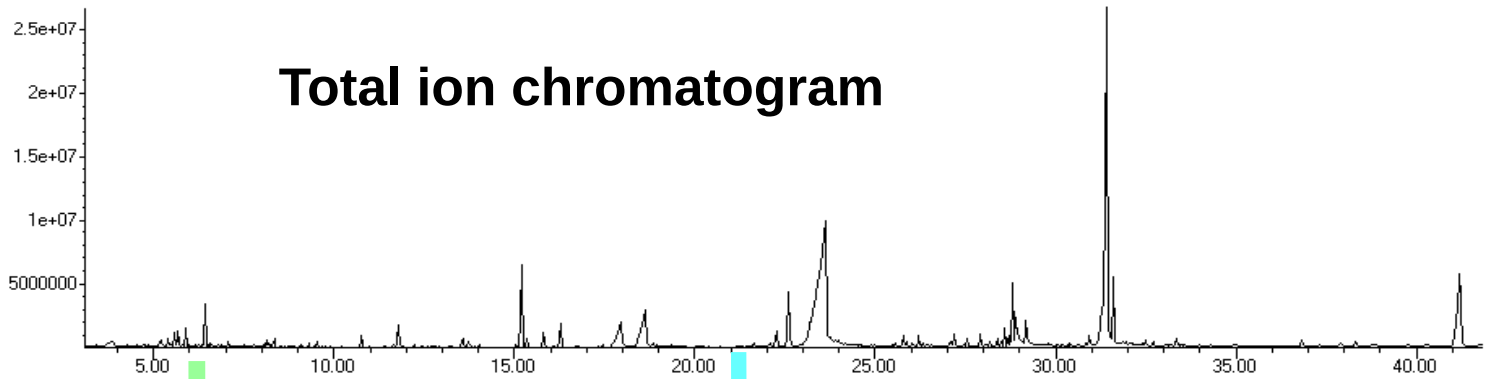
2. DRS, G1716AA Rev A.04, base product

3. Semivolatiles DBL (Database-Library) G1677AA Revision A.02
    4. 338 single component analytes including all of those from USEPA methods 8270 and 525 plus others
    5. One acquisition method for all analytes
    6. All analytes elute by 18 min except Dibenzo(a,e)pyrene, (CAS# 192654) which elutes at 23 min

Semivolatiles Analyzer Backflush Example



DRS Report from 3 Integrated Processes



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

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

Confirmed AMDIS hits

Deconvoluted Target spectra confirmed by AMDIS, searched against NIST08 MS database

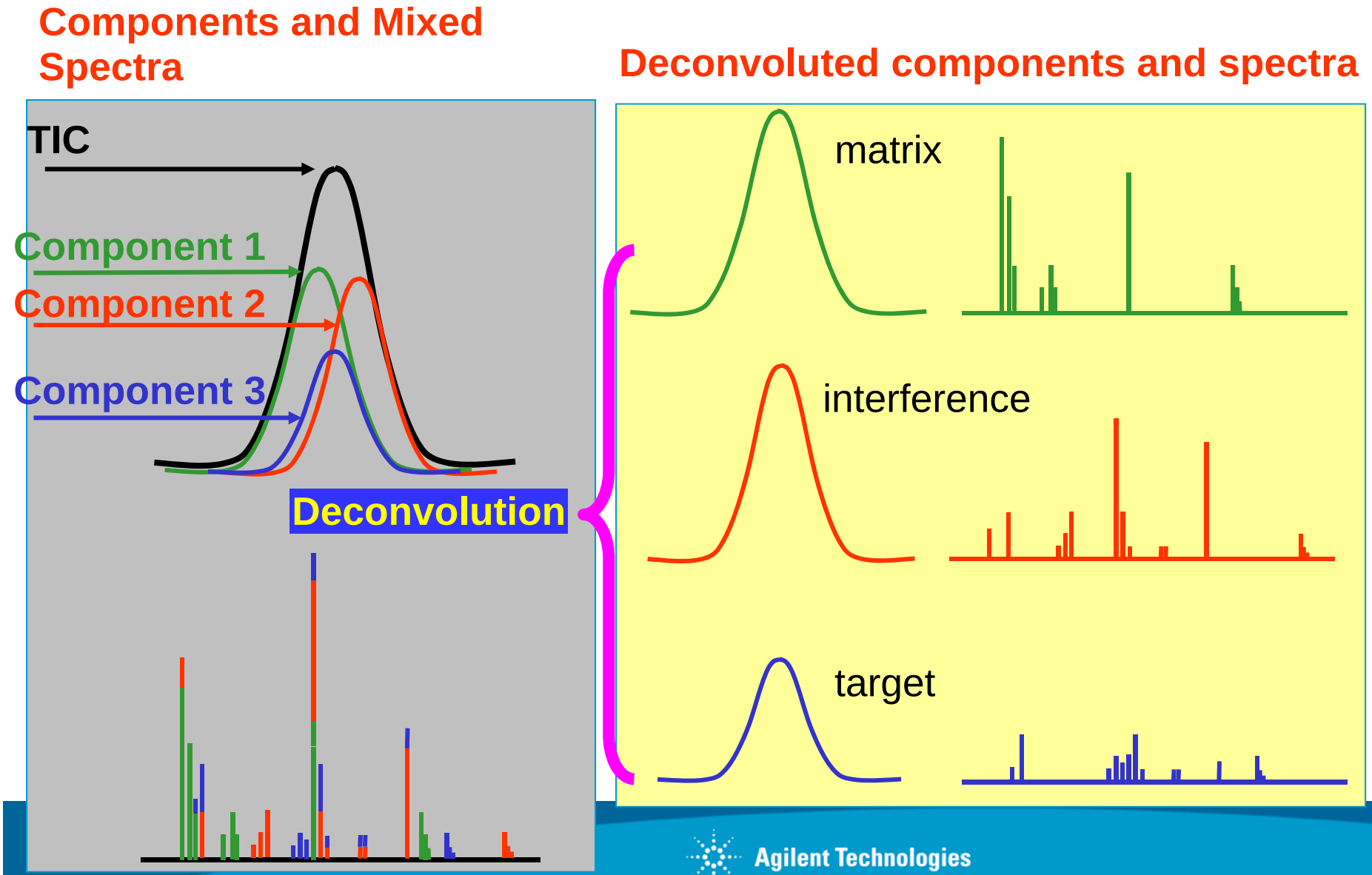
AMDIS Quant Result

Quant Results

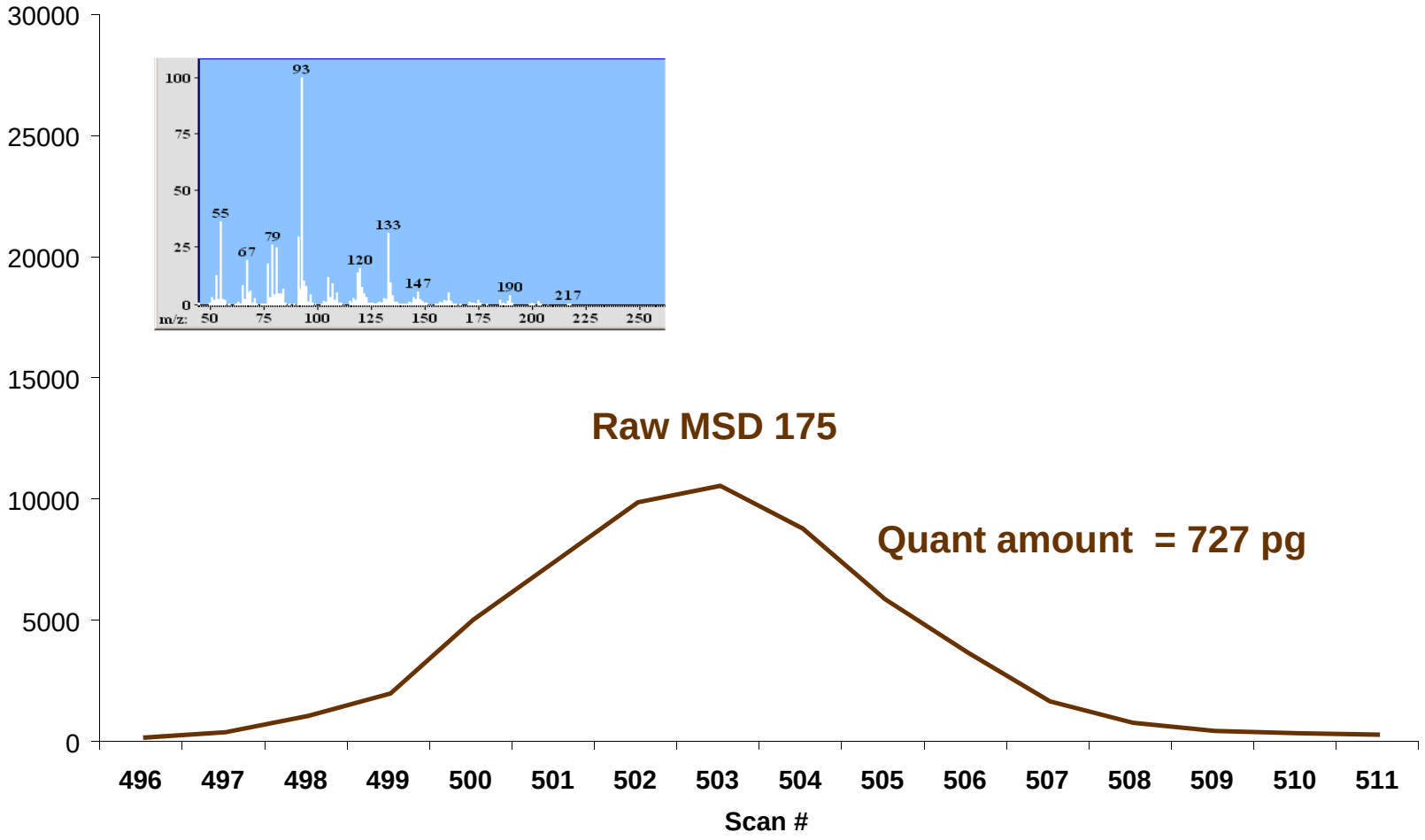
Confirmed NIST08 hits

Combined quantitative and qualitative HTML Summary Report

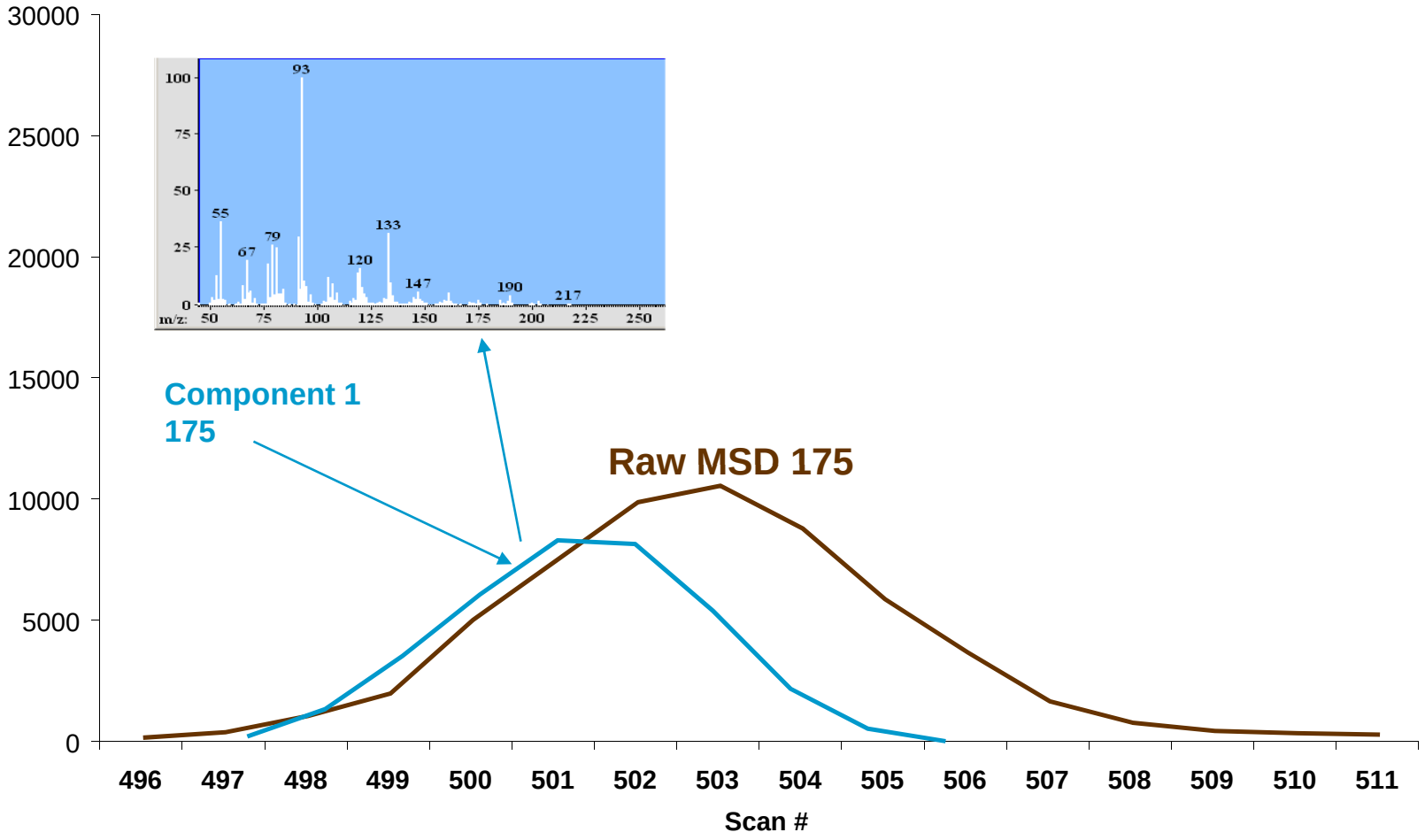
AMDIS Deconvolution Pulls Out Individual Components and their Spectra



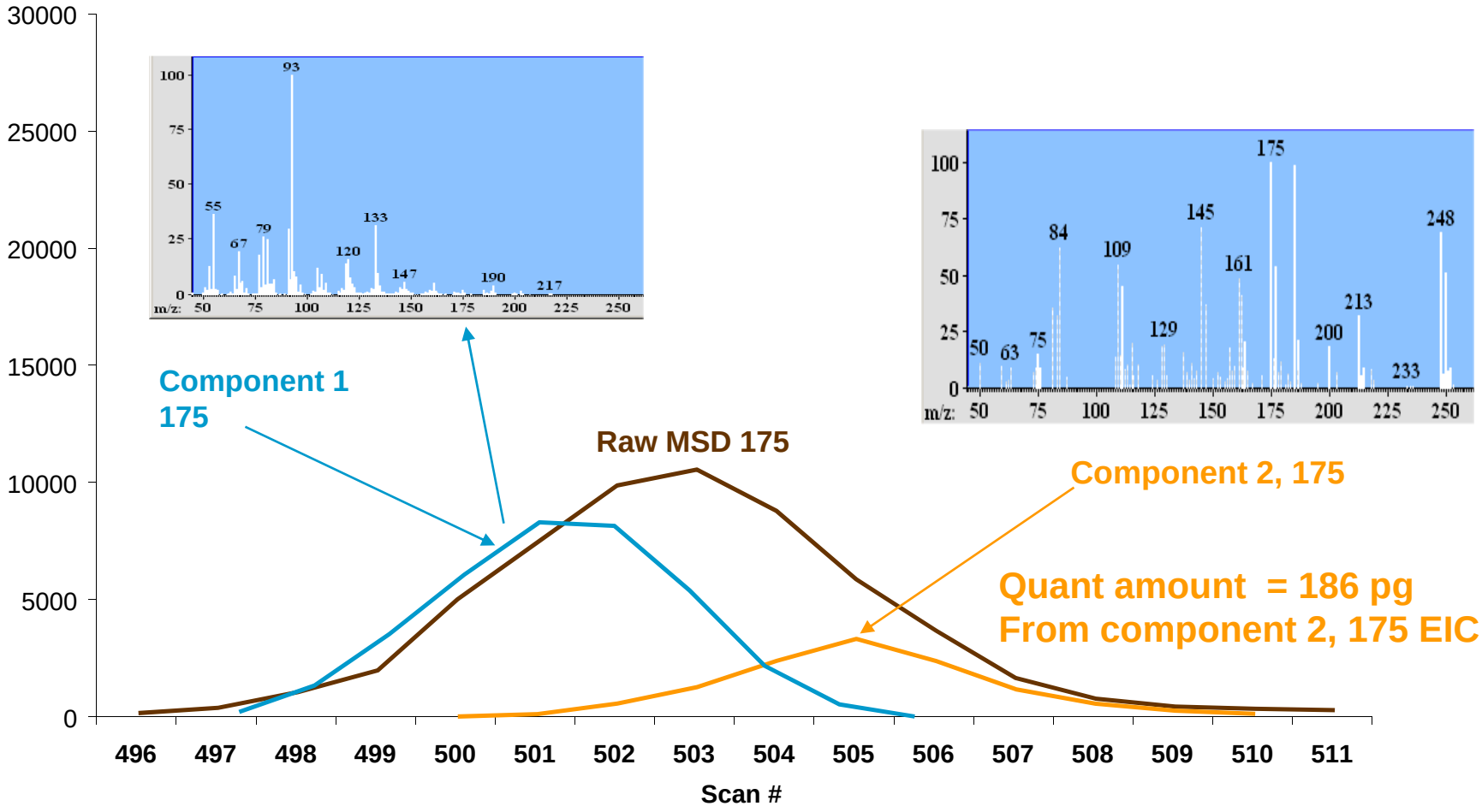
The extracted 175 ion appears in QEdit as this trace



Most of the 175 ion contribution comes from this component which is not visible nor the target



Each of the two components has a 175 ion and AMDIS separates the ion contribution from each



Semivolatiles Checkout Sample DRS Report

MSD Deconvolution Report
Sample Name: Semivoas Checkout
Data File: C:\msdchem\1
\DATA\Semivoas_Relock\Semivoas_Relock_2.D
Date/Time: 4:27:55 PM Monday, August 24, 2009

Adjacent Peak Subtraction = 2
Resolution = Medium
Sensitivity = High
Shape Requirements = Medium

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

			Amount (ng)		AMDIS		NIST	
R.T.	Cas #	Compound Name	Chem station	AMDIS	Match	R.T. Diff sec.	Reverse Match	Hit Num.
3.8773	62759	N-Nitrosodimethylamine	7.33	7	95	0.6	92	1
6.2524	62533	Aniline	10.21	8.4	99	0.3	96	1
6.569	3855821	1,4-Dichlorobenzene-d4	10	10	98	0.4	93	1
7.3429	78591	Isophorone	10.19	10.36	97	0.7	96	1
7.7494	81209	1,3-dimethyl-2-nitrobenzene (ss)	10.64	11.4	97	0.3	97	1
7.8060	1146652	Naphthalene-d8	10	10	100	0.4	90	1
8.6450	77474	Hexachlorocyclopentadiene	12.88	12.8	96	-1.0	85	1
9.0438	7786347	Mevinphos	11.96	11.36	96	-1.0	91	2
9.5301	15067262	Acenaphthene-d10	10	10	99	0.4	85	1
9.5652	51285	2,4-Dinitrophenol	18.86	17.57	91	-1.1	92	1
9.6000	100027	4-Nitrophenol	12.32	13.17	95	-0.8	91	1
9.6937	121142	2,4-dinitrotoluene	11.6	11.18	97	-0.9	92	1
10.0725	86737	Fluorene	10.33	9.22	98	-0.6	94	1
10.0944	534521	4,6-Dinitro-2-methylphenol	15.32	15.32	95	-0.6	93	1
10.2014	1582098	Trifluralin	11.65	11.93	97	-0.6	93	1
10.6528	122349	Simazine	12.47	10.77	93	-0.7	85	2
10.6773	1912249	Atrazine	11.21	11.11	96	-0.7	93	1
10.8004	87865	Pentachlorophenol	12.56	12.26	95	-0.1	90	1
10.8239	13071799	Terbufos	11.7	11.3	97	-0.1	91	1
10.9811	1897456	Chlorothalonil	10.76	10.29	97	0.0	93	1
11.0072	1517222	Phenanthrene-d10	10	10	96	0.4	85	2
11.8128	309002	Aldrin	10.5	9.86	95	0.4	92	1
12.1117	1024573	Heptachlor epoxide -isomer B	11.47	11.34	94	0.4	92	1
12.8189	72208	Endrin	13.13	13.36	94	0.6	94	1
13.1403	50293	4,4'-DDT	13.11	13.39	92	0.6	91	1
13.5762	91941	3,3'-Dichlorobenzidine	11.09	10.88	93	0.8	97	1
13.6369	1719035	Chrysene-d12	10	10	91	0.7	92	1
14.8057	205992	Benzo[b]fluoranthene	9.79	9.64	100	1.1	94	1
15.3217	1520963	Perylene-d12	10	10	97	1.3	84	1

DRS User Benefits, Time = $$$

Ease of use – no need to learn deconvolution software

Automation – part of a method or part of a sequence, analyst can be doing other things

Quality – program results are not subject to changes in mood/attention" of analyst

Sensitivity – AMDIS will find answers that an analyst might miss

Confidence – DRS will report the fewest false positive and false negatives in the shortest time

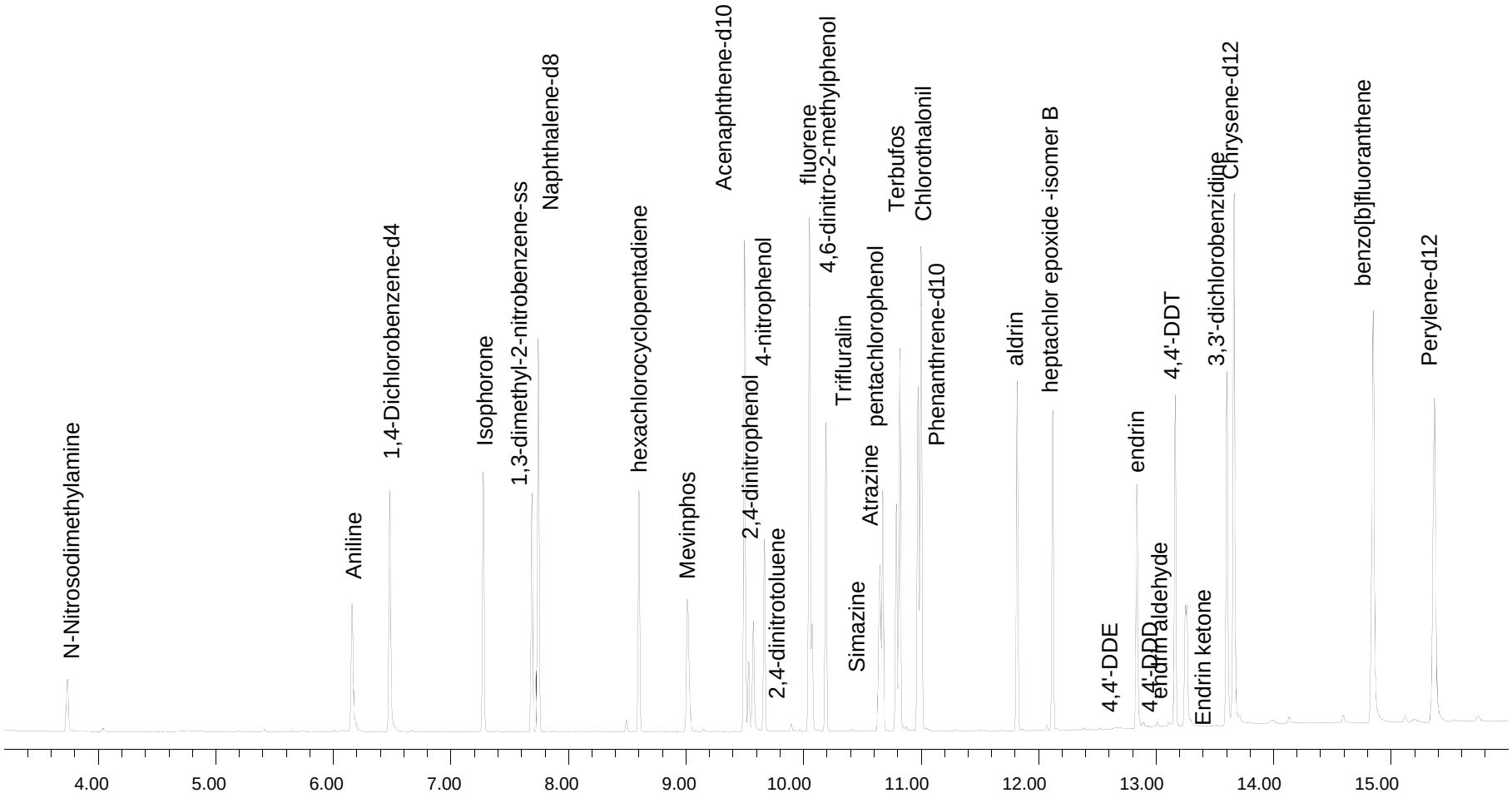
G1677AA Semivoas DBL A.02 Release

G1677AA Files Provided and Their Association

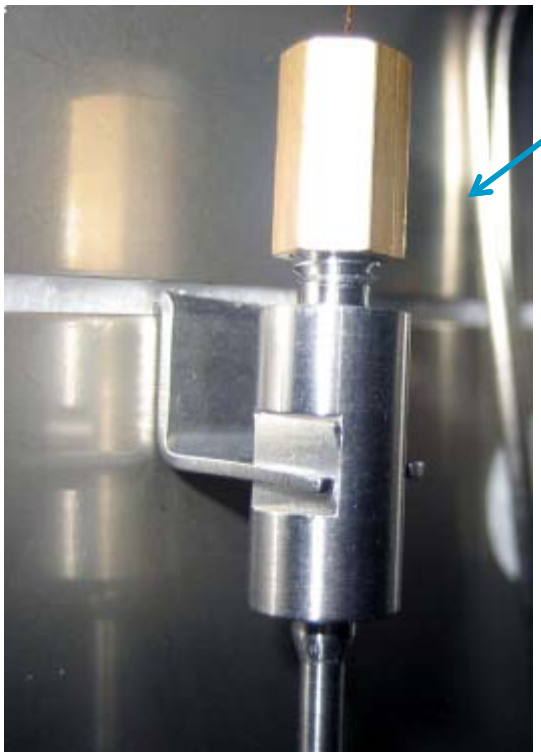
Revision	A.02.00	A.02.00 and A.01.00	A.02.00 and A.01.00	A.02.00 and A.01.00
GC/MSD Acqusition Method	**Semivoas_1_acq_BF.m**	manually entered	manually entered	manually entered
Run Time (min)	21.5	17.0	17.0	26.0
Inlet -mode	cold splitless	hot splitless	hot splitless	LVI
injection volume	1.0	0.5	0.5	25
Scaleable for LVI	Yes	no	no	n/a
Backflush and time	Yes - 3 min	no	no	no
Column	DB 5-MS UI	DB 5.625	DB 5.625	HP 5-MS
Dimensions	20m x 0.18m x 0.36um	20m x 0.18m x 0.36um	20m x 0.18m x 0.36um	30m x 0.25m x 0.25um
RTLock Compound	Phenanthrene -d10	Phenanthrene -d10	Phenanthrene -d10	Phenanthrene -d10
RTLock Time (min)	11.000	9.500	9.500	12.700
GC/MSD Data Analysis Method	**Semivoas_1_DA.m**	8270_RTL_DRS.m	525_RTL_DRS.m	525_Long_DRS.m
Number of Compounds	338	273	119	119
USEPA Method	8270 and 525 combined	8270 only	525 only	525 only
AMDIS msl (and cid)	**Semivoas_1.msl**	8270.msl	525.msl	525_Long.msl
AMDIS cal	**Semivoas_1.cal**	8270_cal_RT.cal	525_cal_RT.cal	525_Long_cal_RT.cal
Agilent Library in Method	8270_DRS.L and 525_DRS.L	8270_DRS.L	525_DRS.L	525_DRS.L
AMDIS csl	**Semivoas_1.csl**	8270_cal_RT.csl	525_cal_RT.csl	525_Long_cal_RT.csl
Demo Data File	**Semivoas_1 _Demo.D**	8270_DRS_Demo.D	525_DRS_Demo.D	525_Long_DRS_Demo.D
Data Analysis Checkout Method and AMDIS files for use with A.02 and Agilent Checkout Sample p/n 5190-0473	**Semivoas_1_DA-checkout.m** **Semivoas_1_checkout.msl** **Semivoas_1_checkout.cid**	n/a	n/a	n/a

Semivolatiles Checkout Sample – 29 Compounds

each at 10 ug/mL (ppm)

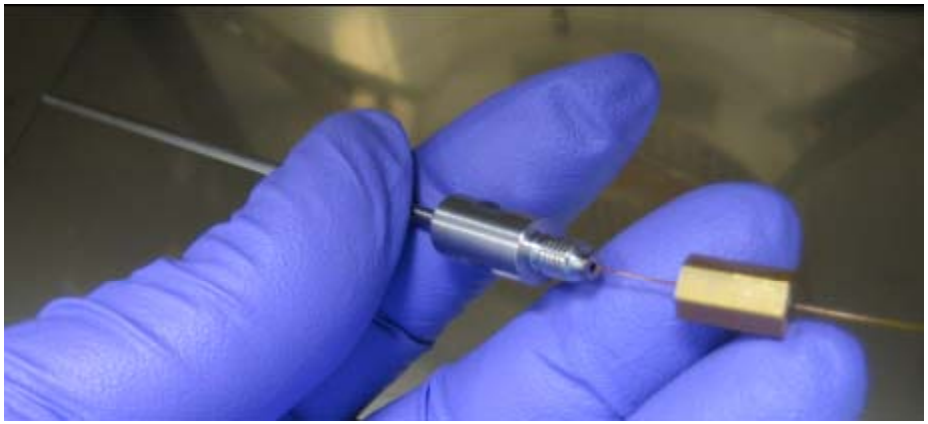


MSD column installation



Restrictor comes cut to length and mounted to the oven wall for shipping.

To install the restrictor column in the MS just remove restrictor from its holder and install it into the MS Interface. Do not re-cut the column.



The column installation tool from the 5973 is used to hold the column.

Generic checkout steps

1. Load the method specified in the CE guide provided on the CD
2. Resolve the method. Note: this step should not be needed since the methods provided were developed on the GCMS delivered to the customer.
3. Autotune and evaluate the tune
4. Relock the method.
5. Run the standard and produce a DRS report
6. Check the report against the report delivered with the system

Semivolatiles analyzer specific checkout steps

1. Load and resolve **Semivoas_1_acq_BF.m** method (resolution should not be needed)
2. Autotune MSD with Gain Normalized Autotune
3. Run 5190-0473, DRS Semivoas checkout sample twice
4. Relock retention time of method on second run
5. Rerun checkout sample with relocked method if needed
6. In DA, load method **Semivoas_1_DA-checkout.m** and run “Quant + DRS single” on checkout sample data file
7. Inspect report to assure: All peaks in checkout mix are found with match factor above minimum.
8. OFN **not** required - already run at Agilent

Familiarization key tasks

DRS

- Demonstrate how to run DRS
- Demonstrate how generate and evaluate a DRS report
- Explain how to use QEdit with DRS
- Demonstrate how to update the DRS calibration (Single level calibration shipped with system)
- DRS Videos for Training

7890

- Describe GC MMI (Multi-Mode Inlet) column installation
- Describe GC MMI maintenance, flows and parts replacement
- Describe the CFT column connections and review flow diagram
- Demonstrate how to make CFT connections using Siltite ferrules.

Minimizing Start-up Time with an Analyzer

1. The system is designed for a specific application
2. Pre-assembled and Pre-tested as a complete system
3. Column is Retention Time Locked
4. Method is built and shipped, ready for installation
5. Includes hardware, software and consumables
6. Backflush parameters set for maximum productivity
7. DRS configured for immediate use
8. Analyzer specific CD includes method(s), user Manual, application notes
9. Service Engineers specifically trained in Analyzers
10. Check out sample run to verify on-site performance