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

7693A single tower and tray

MMI Inlet 23 psig (nom)

7890A GC 240V

1 uL cold splitless injection

1 mL/min CF

5 mL/min bleeder

Aux EPC or PCM 2.0 psig

PUU

40 C for 2.5 min
25 C/min to 320 C
Hold for 4.8 min = 18 min run
3 to 5 min backflush

5975C EI MSD Performance Turbo and Triple Axis Detector
Any One of these three CFT Devices can be used in the Semivolatile Analyzer

- Purged Ultimate Union with Makeup
- 2-Way Splitter with Makeup
- 3-Way Splitter with Makeup
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
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 CO₂ or liquid N₂ 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
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

Cold Injection

Transfer of sample from inlet to column

GC Separation

During solvent vent, the initial MMI temp is held longer and the purge valve is on

MMI Temp

GC Oven Temp

Purge Status

Min

0 0.05 0.65 1.4 2.5 26

20 320

Oven = 40

purge off

purge on

purge on
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

Matrix, 42 min elution

Spike, with backflushing

Scale 20x more sensitive than above

Blank after backflush

21 min. Timesavings

3 min. BF

7.00 12.00 17.00 22.00 27.00 32.00 37.00
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

AMDIS Quant Result

Combined quantitative and qualitative HTML Summary Report

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

Confirmed NIST08 hits
AMDIS Deconvolution Pulls Out Individual Components and their Spectra

Components and Mixed Spectra

Deconvolution

TIC

Component 1

Component 2

Component 3

Deconvoluted components and spectra

matrix

interference

target
The extracted 175 ion appears in QEdit as this trace

Raw MSD 175

Quant amount  = 727 pg
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: Semivolatiles Checkout
- Data File: Coimbschem1
- DATE/SEMIVOLATILES_1/Semivolatiles_ReLock_2.D
- Date/Time: 4:27:55 PM Monday, August 24, 2009

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

<table>
<thead>
<tr>
<th>R.T.</th>
<th>Cas #</th>
<th>Compound Name</th>
<th>Chem station</th>
<th>AMDIS Match</th>
<th>AMDIS R T. Diff. sec.</th>
<th>Reverse Match</th>
<th>Hit Num</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.9773</td>
<td>62759</td>
<td>N-Nitrosodimethylamine</td>
<td>7.33</td>
<td>7</td>
<td>95</td>
<td>0.6</td>
<td>92.1</td>
</tr>
<tr>
<td>6.2524</td>
<td>62533</td>
<td>Aniline</td>
<td>10.21</td>
<td>8.4</td>
<td>89</td>
<td>0.3</td>
<td>96.1</td>
</tr>
<tr>
<td>6.568</td>
<td>3865621</td>
<td>1,4-Dichlorobenzene-d4</td>
<td>10</td>
<td>10</td>
<td>98</td>
<td>0.4</td>
<td>93.1</td>
</tr>
<tr>
<td>7.3439</td>
<td>78951</td>
<td>Isolexone</td>
<td>10.19</td>
<td>10.36</td>
<td>97</td>
<td>0.7</td>
<td>96.1</td>
</tr>
<tr>
<td>7.7404</td>
<td>31209</td>
<td>1,3-dimethyl-2-nitrobenzene (S)</td>
<td>10.64</td>
<td>11.4</td>
<td>97</td>
<td>0.3</td>
<td>96.1</td>
</tr>
<tr>
<td>7.7950</td>
<td>1146652</td>
<td>Naphthalene-98</td>
<td>10</td>
<td>10</td>
<td>100</td>
<td>0.4</td>
<td>90.1</td>
</tr>
<tr>
<td>8.450</td>
<td>77474</td>
<td>Hexachlorocyclopentadiene</td>
<td>12.28</td>
<td>12.8</td>
<td>96</td>
<td>1.0</td>
<td>86.1</td>
</tr>
<tr>
<td>9.0438</td>
<td>776347</td>
<td>Methylhexahexahexahexahexane</td>
<td>11.36</td>
<td>11.36</td>
<td>96</td>
<td>1.0</td>
<td>91.2</td>
</tr>
<tr>
<td>9.5301</td>
<td>15067262</td>
<td>Acenaphthene-d10</td>
<td>10</td>
<td>10</td>
<td>99</td>
<td>0.4</td>
<td>85.1</td>
</tr>
<tr>
<td>9.5852</td>
<td>51285</td>
<td>2,4-Dinitrophenol</td>
<td>18.36</td>
<td>17.57</td>
<td>91</td>
<td>1.1</td>
<td>92.1</td>
</tr>
<tr>
<td>9.6000</td>
<td>100037</td>
<td>4-Nitrophenol</td>
<td>12.32</td>
<td>13.17</td>
<td>96</td>
<td>0.8</td>
<td>91.1</td>
</tr>
<tr>
<td>9.6937</td>
<td>121142</td>
<td>2,4-Dinitrotoluene</td>
<td>11.6</td>
<td>11.18</td>
<td>97</td>
<td>0.9</td>
<td>92.1</td>
</tr>
<tr>
<td>10.0725</td>
<td>56373</td>
<td>Fluorene</td>
<td>10.33</td>
<td>9.22</td>
<td>98</td>
<td>0.6</td>
<td>94.1</td>
</tr>
<tr>
<td>10.0944</td>
<td>534521</td>
<td>4,6-Dinitro-2-methylphenol</td>
<td>15.32</td>
<td>15.32</td>
<td>95</td>
<td>0.6</td>
<td>93.1</td>
</tr>
<tr>
<td>10.2014</td>
<td>1550299</td>
<td>Trifluoromethane</td>
<td>11.65</td>
<td>11.93</td>
<td>97</td>
<td>0.6</td>
<td>93.1</td>
</tr>
<tr>
<td>10.6529</td>
<td>122349</td>
<td>Simazine</td>
<td>12.47</td>
<td>10.77</td>
<td>93</td>
<td>0.7</td>
<td>95.2</td>
</tr>
<tr>
<td>10.6773</td>
<td>1912249</td>
<td>Azulene</td>
<td>11.21</td>
<td>11.11</td>
<td>96</td>
<td>0.7</td>
<td>93.1</td>
</tr>
<tr>
<td>10.8004</td>
<td>78585</td>
<td>Pentachlorophenol</td>
<td>12.56</td>
<td>12.26</td>
<td>95</td>
<td>0.1</td>
<td>90.1</td>
</tr>
<tr>
<td>10.8239</td>
<td>1301799</td>
<td>Turibus</td>
<td>11.7</td>
<td>11.3</td>
<td>97</td>
<td>0.1</td>
<td>91.1</td>
</tr>
<tr>
<td>10.9811</td>
<td>1887456</td>
<td>Chlorothalonil</td>
<td>10.76</td>
<td>10.29</td>
<td>97</td>
<td>0.0</td>
<td>93.1</td>
</tr>
<tr>
<td>11.0072</td>
<td>1517222</td>
<td>Phenanthrene-d10</td>
<td>10</td>
<td>10</td>
<td>96</td>
<td>0.4</td>
<td>85.2</td>
</tr>
<tr>
<td>11.8129</td>
<td>300202</td>
<td>Adriene</td>
<td>10.5</td>
<td>9.86</td>
<td>95</td>
<td>0.4</td>
<td>92.1</td>
</tr>
<tr>
<td>12.1117</td>
<td>1024573</td>
<td>Methachlor isopropyl ester</td>
<td>11.47</td>
<td>11.34</td>
<td>94</td>
<td>0.4</td>
<td>92.1</td>
</tr>
<tr>
<td>12.8189</td>
<td>72208</td>
<td>Endrin</td>
<td>13.13</td>
<td>13.36</td>
<td>94</td>
<td>0.6</td>
<td>94.1</td>
</tr>
<tr>
<td>13.1403</td>
<td>50293</td>
<td>1,4-DOT</td>
<td>13.11</td>
<td>13.39</td>
<td>92</td>
<td>0.6</td>
<td>91.1</td>
</tr>
<tr>
<td>13.5962</td>
<td>91941</td>
<td>3,5-Dichlorobenzidine</td>
<td>11.59</td>
<td>10.58</td>
<td>93</td>
<td>0.5</td>
<td>97.1</td>
</tr>
<tr>
<td>13.6369</td>
<td>1719035</td>
<td>Chrysene-d12</td>
<td>10</td>
<td>10</td>
<td>91</td>
<td>0.7</td>
<td>92.1</td>
</tr>
<tr>
<td>14.8057</td>
<td>205952</td>
<td>Benzo[b]fluoranthene</td>
<td>9.73</td>
<td>9.64</td>
<td>100</td>
<td>1.1</td>
<td>94.1</td>
</tr>
<tr>
<td>15.3217</td>
<td>1620963</td>
<td>Pyrene-d12</td>
<td>10</td>
<td>10</td>
<td>97</td>
<td>1.3</td>
<td>84.1</td>
</tr>
</tbody>
</table>
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

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