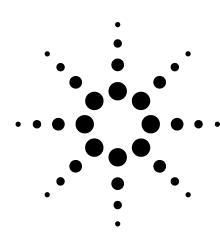
Significant Cycle Time Reduction Using the Agilent 7890A/5975C GC/MSD for EPA Method 8270

Application Brief

Mike Szelewski



U.S. EPA Method 8270 for semivolatiles analysis is used to concurrently measure a mix of 70–100 acids, bases, and neutrals. Laboratories want to reduce the typical 25–60-minute cycle time for productivity increases. The Agilent 7890A/5975C GC/MSD system meets this demand using a smaller id column, faster cooling oven, and backflushing. Criteria for system performance check compounds (SPCCs) and continuing calibration compounds (CCCs) are met using a calibration range of 1–200 ppm.

The system was calibrated at 10 levels using the conditions in Table 1. The SPCCs and CCCs all meet 8270 criteria, the results shown in Table 2. The overall average %RSD for all 77 analytes was 11%.

Cycle time savings are shown in Table 3. Historically, a 30 m \times 0.25 mm column is used in 6890 systems. The 20 m \times 0.180 mm column used here cuts the run time by 8 min, a 5 ppm standard shown in Figure 1A.

Highlights

- Productivity increases > 55% with the Agilent 7890A/5975C GC/ MSD system.
- The oven heats faster, cools down faster, and reduces cycle time.
- Backflushing reduces analysis time and increases column life while reducing maintenance time and frequency.

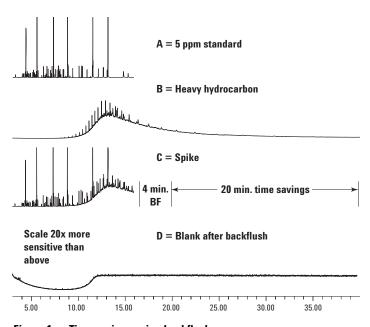


Figure 1. Time savings using backflush.



Bake-out of matrix often takes longer than the run time for analytes of interest. A heavy hydrocarbon, typical of an 8270-type extract, is shown in Figure 1B, eluting in \sim 40 min. The elution time on a 30-m column is > 50 min. All of this material is usually eluted into the MSD.

A backflush of the heavy material from a sample spiked with standard is shown in Figure 1C. The 4-min backflush is accomplished by raising the pressure in the QuickSwap and lowering the inlet pressure. The matrix elutes into the inlet and is swept out the split vent line.

Figure 1D shows a blank solvent run after the backflush. The heavy hydrocarbon matrix is not on the column nor in the liner.

The Agilent 7890A has faster oven cool-down, from 320–40 °C. Additionally, the MSD can be interfaced in a new rear oven position, along with the inlet in the rear. An oven insert, the "pillow," can occupy the front half of the oven. This allows even faster heating and cooling. The times are shown in Table 3.

Additional time savings are realized by using QuickSwap. Liner and column maintenance or changing can be done without venting the MSD.

Table 1. Gas Chromatograph and Mass Spectrometer Conditions

14510 11	eas sin sinategraph and mass spectrometer conditions
GC	Agilent Technologies 7890A
Inlet	EPC split/splitless
Mode	Splitless, 0.5 μL injected
Inlet temp	300 °C
Pressure	25.0 psi
Purge flow	30.0 mL/min
Purge time	0.75 min
Gas saver	Off
Gas type	Helium
Liner	Agilent Helix single taper liner with a narrow o.d. for both

Liner	Agilent Helix single taper liner with a narrow o.d. for both split
	and splitless, proprietary deactivation, Part # 5188-5397

Oven	240V		
Oven ramp	°C/min	Next °C	Hold min
Initial		40	1.00
Ramp 1	25	320	4.80
Total run time	17.0 min		
Equilibration time	0.5 min		
Oven max temp	325 °C		

Column	Agilent Technologies DB-5.625, Part # 121-5622
--------	--

Length	20.0 m
Diameter	0.18 mm
Film thickness	0.36 µm

Mode Constant flow = 1.1 mL/min

Inlet Front

Outlet QuickSwap, Agilent Part # G3185B

QuickSwap pressure 5.0 psi during acquisition, 80.0 psi during backflush with

inlet set to 1.0 psi during backflush

MSD Agilent Technologies 5975C, Performance Turbo
Drawout lens 6 mm large aperture drawout lens, Part # G2589-20045

Solvent delay 2.8 min
EM voltage Tune voltage
Mass range 35–500 amu

Sampling 1
Quad temp 180 °C
Source temp 300 °C
Transfer line temp 250 °C
Emission current 25 µamp

Calibration Standards

Accustandard, New Haven, CT. Part # M-8270-IS-WL-0.25x to 10x

77 compounds at 10 concentration levels with 6 internal standards at 40 ppm

Table 2. SPCC and CCC, Criteria and Results

	8270 criteria	7890A-5975C with QuickSwap (range)
4 SPCCs minimum average RRF	0.050	0.110-0.405
13 CCCs %RSD	< 30%	2% -20%

Table 3. Cycle Time Savings Using the 7890A-5975C

	Typical 6890	7890A	Minutes Saved
Run time without matrix bake-out, includes equib	25	17	8
Run time with matrix bake-out 6890 or QuickSwap 7890A	50	21	29
Cool down time from 320 to 40	7	4.3	2.7
Cool down time from 320 to 40 with pillow	n/a	3.3	3.7
Total time savings using a 7890A-5975C with the 20-m column, QuickSwap for backflush, rear position for MSD and pillow	57	24.3	32.7

Significant cycle time savings can be realized, depending on sample complexity and column and instrument configuration. Analyzing dirty samples on a 30-m column can take 57 minutes or more with a 6890. Using an Agilent 7890A, Quick-Swap, the 20-m column, and rear oven position, cycle time is < 25 minutes. This is a direct productivity increase of > 55%.

www.agilent.com/chem

Mike Szelewski is an application chemist based at Agilent Technologies, Wilmington, Delaware, USA.

For More Information

For more information on our products and services, visit our Web site at www.agilent.com/chem.

Agilent shall not be liable for errors contained herein or for incidental or consequential damages in connection with the furnishing, performance, or use of this material.

Information, descriptions, and specifications in this publication are subject to change without notice.

© Agilent Technologies, Inc. 2007

Printed in the USA January 10, 2007 5989-6026EN

