

# Increased Throughput in Method 8270 Analysis

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Method 8270 is a GC-MS method created by the U.S. EPA Office of Solid Waste for the analysis of semivolatile organics by gas chromatography-mass spectrometry. This is a common and difficult method performed in nearly all commercial environmental laboratories in the U.S. and in many other parts of the world. Following the creation of the EPA in 1970, the U.S. commercial laboratory industry experienced rapid growth throughout the 1980's. The 1990's through the present have brought an unsettled period of consolidation and adjustment. This period of decreasing profit margins has magnified the importance of quick and efficient production.

Method 8270 specifies that mass spectrometer tuning and calibration check samples must verify the integrity of the system every 12 hours.

These samples severely limit the production of each particular GC-MS system. Once the calibration check is completed, the instrument can run an unlimited number of samples in the next 12-hour clock. This results in the need for an analytical method with a short injection-to-injection time, maximizing the ratio of billable samples to nonbillable quality control samples.

The approach taken in this work was to use a standard 5% diphenyl/95% dimethyl polysiloxane column (Elite-5 MS 30 m × 0.25 mm × 0.250 μm, **PerkinElmer Life and Analytical Sciences** [Shelton, CT]), while reducing the nonproductive time between sample injections to increase the number of samples analyzed per hour. The novel GC

oven design utilized has allowed this new approach to increase analytical throughput.

## Experimental

The GC-MS system used was the Clarus® 600 GC-MS (**PerkinElmer Life and Analytical Sciences**). The GC has several options that were utilized to reduce the cycle time. The first is solvent prerinse; the auto-sampler performs all rinsing (solvent and sample) needed prior to injection, while the previous run is being acquired. This permits the autosampler to collect the final sample aliquot when the instrument becomes ready, injecting almost immediately. Secondly, the equilibration time for the GC oven was eliminated as a program step in favor of the opti-

**Table 1 GC experimental conditions**

GC conditions			
Total GC run time	15.5 min		
Injection-to-injection time	20.0 min		
Oven	Rate	Temp. (°C)	Hold
Initial	—	37	1
1	27	265	0
2	6	287	0
3	30	320	2.11
Injector	PSS		
Injector temperature	Rate	Temp. (°C)	Hold
Initial	—	120	0
1	999	320	5
2	999	120	999
Column phase	0.250 μm Elite-5 MS		
Column length	30 m		
Column diameter	0.250 mm		
Split	50.0 mL/min		
Syringe	5 μL		
Injection volume	1 μL		
Instrument timed events		Time (min)	Value (mL/min)
	Split flow	-0.51	0
	Split flow	1.00	50
	Split flow	5.00	20

**Table 2 MS experimental conditions**

MS settings	
Source temp.	280 °C
GC transfer line temp.	320 °C
Electron energy	70 eV
Photomultiplier voltage	450 V
Solvent delay	2.80 min
Cycle time	0.25 sec
Scan duration	0.20 sec
Inter-scan delay	0.05 sec
Ionization mode	EI+
Function type	Scan
Mass range	35–500 amu

mized timing set by the Clarus 600 firmware. Lastly, the system offers SOFTCooling™, which enables programmed cooling of the GC oven to reduce column bleed effects during ballistic cooling. Ballistic cooling was chosen in the interest of producing the fastest injection-to-injection time possible.

A temperature-programmed injection was utilized to allow for a more controlled expansion of the solvent within the injection port and better containment of the sample within the glass liner. The programmable split/splitless (PSS) injector used a 2-mm-i.d. injector port liner, packed with a thin plug of deactivated glass wool. Glass wool was used to minimize mass discrimination and ensure complete transfer of the heavier polycyclic aromatic hydrocarbons (PAH). Table 1 lists all of the gas chromatographic conditions used to collect these data.

Two Clarus 600 mass spectrometers were used in this study: a 600 D air-cooled oil diffusion pump and a 600 T 255-L/sec turbomolecular pump. Table 2 lists all of the mass spectrometer conditions used to collect these data. The ion source and transfer line of the MS were heated to prevent condensation and contamination. The photomultiplier tube (PMT) was adjusted to the midpoint in its range; the adjustment can be modified to meet differing needs of 8270 analysis. If low-level sensitivity is needed, the PMT voltage should be adjusted to a level where the high-

est concentration standard is just below the level of detector saturation. This will provide the highest signal possible for low-level standards while maintaining maximum linear dynamic range. If accurate estimations of components with concentrations above the calibration range are important, setting the PMT to a low level where sensitivity is just enough to measure the low standard will allow high standards to remain well within a linear portion of its range.

The mass spectrometer was set to acquire data in full-scan mode.

This can be modified to achieve low-level detection limits. Single ion monitoring (SIM) and single ion monitoring combined with full ion scanning (SIFI) functions can be added to the MS method, but were not needed for this routine analysis.

Calibration standards were diluted in methylene chloride from the 8270 Mega-Mix (Restek, Bellefonte, PA). The curves analyzed here were from 5 to 100 ppm, with internal standards at 25 ppm.

## Results

Following the guidelines of Method 8270, each 12-hour analysis

“clock” was initiated with an injection of 50 ng/μL of decafluorotriphenylphosphine (DFTPP) in methylene chloride. The ions of DFTPP consistently met the specified ratios for MS tune verification (Figure 1). When acquiring a tuning spectrum, Method 8270C recommends selecting three scans across the peak: one prior through one subsequent to the apex of the DFTPP peak, and subtracting a background scan within 20 scans of the peak. The background subtraction scan must not be chosen to remove specific ions to pull the tune within criteria.

The initial calibration of Method 8270 presented here was from 5 ppm to 100 ppm. The analyte list included the most commonly determined compounds under Method 8270. The initial calibration must be within 20% RSD (8270D) for the calibration to be considered acceptable. The calibration is allowed to have 10% of the compounds outside of the 20% RSD range. If more than 10% of the compounds are outside the 20% RSD range, then maintenance must be performed on the system and the initial calibration must be reanalyzed. Table 3 shows the calibration QC information for selected components, demonstrating compliance with the method requirements.

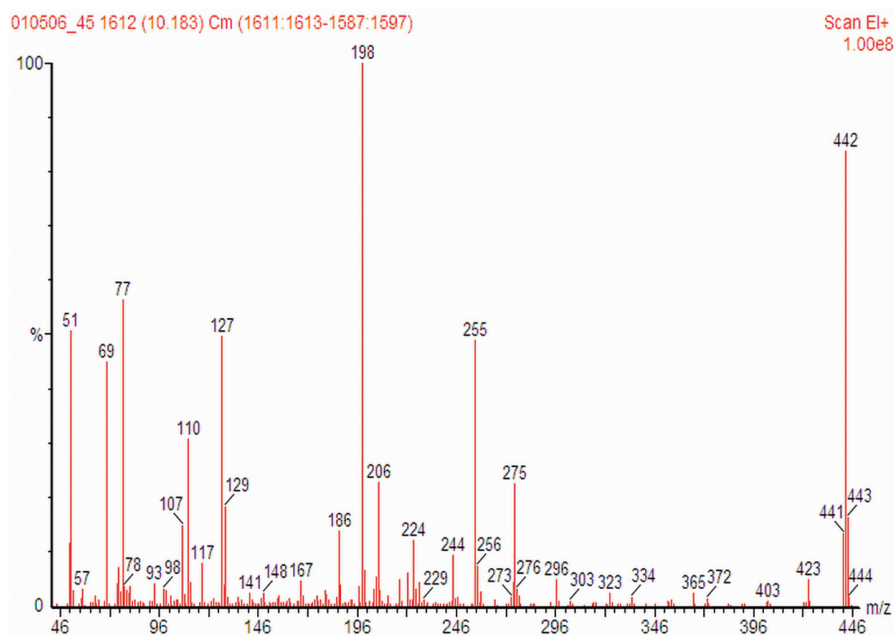


Figure 1 Background-subtracted spectra of DFTPP from the Clarus 600 D.

**Table 3 Example 8270 calibration data from the Clarus 600 D (the calibration range presented here is 5–100 ppm)**

Compound	% RSD
Phenol	8.96
Aniline	7.53
Bis(2-chloroethyl)ether	6.23
1,3-Dichlorobenzene	13.95
1,4-Dichlorobenzene	13.36
1,2-Dichlorobenzene	13.89
Naphthalene	10.6
Hexachlorobutadiene	2.52

Beyond calibration of the mass spectrometer, the chromatography contains a number of critical peak separations that must be maintained during this shorter chromatographic time method. Figure 2 demonstrates the separations of benzo(B) and (K)fluoranthene; phenol, aniline, and bis(2-chloroethyl)ether, showing the decreased run time and sufficient separation. The separations were improved with the ability of the GC oven to approach ambient temperatures in a short period of time. This allows for GC oven programs, which begin at 37 °C, while not significantly impacting the injection-to-injection time.

## Discussion

Method 8270 analysis was performed on both the diffusion and turbomolecular pumped systems, with statistically identical results. A difference between the two systems would be observed when the systems were pumped down from atmospheric pressure, i.e., the turbomolecular pump would more rapidly reach the vacuum level desired for analytical work.

As commonly implemented today, the injection-to-injection time for Method 8270 is approximately 30 min. Figure 2 shows a fully compliant 8270 analysis with a total injection-to-injection time of 20 min. This decrease in injection-to-injection

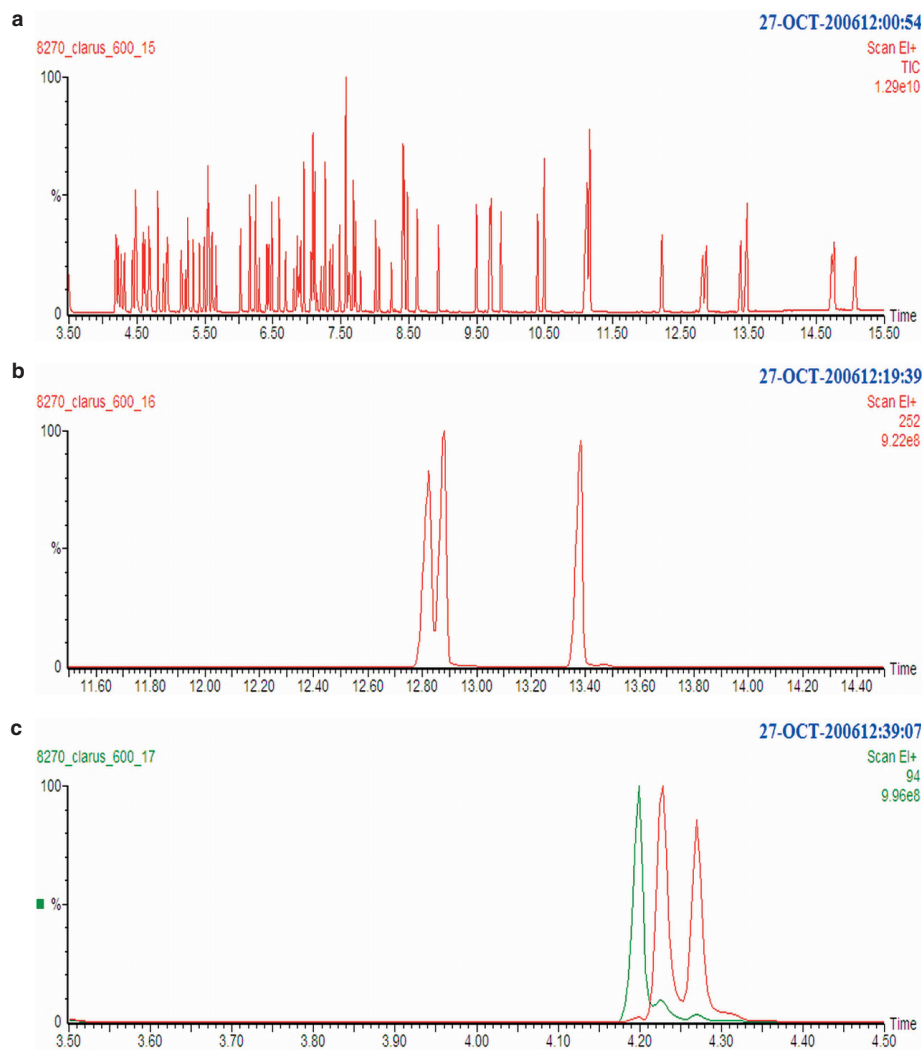


Figure 2 Chromatograms from three consecutive 8270 runs, illustrating the ~20-min injection-to-injection time, the total ion current chromatogram (a), and two key separations. Extracted ion  $m/z$  252 (b) shows the separation of benzo(B) and (K)fluoranthene at  $m/z$  94 and  $m/z$  93. Chromatogram (c) shows phenol, aniline, and bis(2-chloroethyl)ether.

time was achieved with a GC oven and does not require a narrow-bore, thin-film column. The column used was chosen because of its proven ability to withstand the rigors of environmental matrices.

Another way to speed up analysis is to reduce the chromatographic time by using a smaller-diameter column with a thin phase. Often, narrow-bore thin-film columns result in a higher column consumption and greater frequency of recalibration. The increased cost associated with the shorter life span and high frequency of calibration limits the profit increases associated with these columns.

The ability to reduce injection-to-injection time by 30% allows another sample to be run each hour, while maintaining chromatographic quality and column lifetime. Over the course of a 12-hour clock, an additional 12 samples can be run, significantly increasing productivity and potential profit.

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