

Gas Chromatography/  
Mass Spectrometry

## The Importance of High Speed Data Acquisition in the Design and Implementation of GC/MS Applications

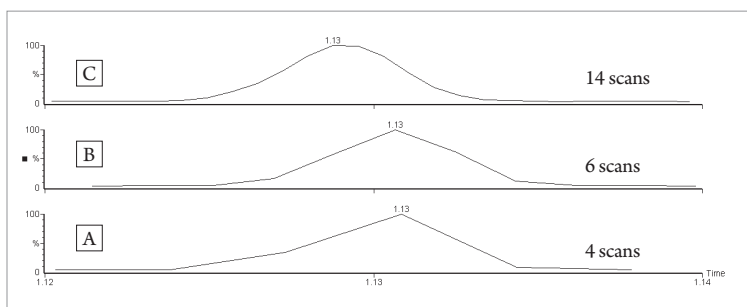


Figure 1. Consecutive 15 ng on column injections of n-decane to show the effect of scan speed on peak shape. In chromatogram 'A' 4 scans were collected, in chromatogram 'B' 6 scans were collected, and in chromatogram 'C' 14 scans were collected.

### Introduction

As gas chromatography (GC) methods and technology evolve and provide higher resolution and throughput, the resulting analyte peaks become ever narrower. This creates a greater need for fast data acquisition in order to achieve accurate and precise representation of a chromatographic data. In addition to fast GC, high speed MS acquisition is essential in applications such as semi-volatiles in environmental samples and pesticides in food, where the MS system is required to collect different types of data simultaneously.

A good practice in GC/MS is that 8 to 12 scans across a peak will give sufficient definition and reproducibility for quantitation. With slower scanning speeds, the representation of the peak may not be accurate, leading to reproducibility and quantitation problems. This is demonstrated in Figure 1.

The PerkinElmer® Clarus® 600 MS is capable of scanning at best-in-class speeds. This technical brief demonstrates the maximum scanning speeds in all three modes of MS operation: full scan, Selected Ion Recording (SIR, also called Selected Ion Monitoring, or SIM), and SIFI (Single Ion Full Ion). In full scan mode, the maximum scan speed is 12,500 Da/sec (also referred to as amu/sec or U/sec); the speed, and more importantly, the spectral fidelity of the data produced while operating at this speed will be demonstrated in the following pages. Sample rates of up to 100 scans/sec are achievable with SIR mode acquisition.

Fast scanning capability becomes extremely beneficial when applied to SIFI functions (SIFI data acquisition is further explained in the PerkinElmer Technical note “SIFI – Simultaneous Collection of Full Scan and Selected Ion Recording Mass Spectral Data”). SIFI functions make use of both the fast scan and SIR capabilities, allowing data collection with both the exceptional sensitivity of SIR and the library-searchable spectral data of full scan. Here, fast SIFI will be applied to a fast GC/MS analysis of aliphatic and aromatic hydrocarbons whose peak widths are 0.5 seconds wide at base, demonstrating at least 12 full spectrum scans and 12 SIR data points across each peak. This method simultaneously achieves good chromatographic peak-shape definition, full scan library-searchable spectral data, and SIR sensitivity.

## Experimental

To demonstrate the fast data acquisition capabilities of the Clarus 600 GC/MS, this analysis was carried out with a very short micro bore GC column, hydrogen carrier gas, and high oven heating rates (see Table 1). The chromatographic conditions used here result in peak widths of less than one second at base. The sample used here was a mixture of n-alkanes (c-10 through c-13): toluene, ethylbenzene, m-xylene, o-xylene, and p-xylene prepared in pentane at approximately 60 ppm ( $\mu\text{g/mL}$ ). It was necessary to use an extremely high split flow so as not to overload the column stationary phase.

All three modes of GC/MS data acquisition (full scan, SIFI, and SIR) were evaluated. Table 2 summarizes the scanning conditions used to demonstrate the full scan capabilities; this included operating the MS at five different scanning speeds.

Table 3 summarizes the mass spectrometer conditions used to acquire the high speed SIR data, and Table 4 summarizes the conditions utilized during the testing of SIFI analysis.

**Table 1. The gas chromatographic conditions used through this study.**

<b>Gas Chromatograph: PerkinElmer Clarus 600</b>			
Total GC Run Time:	3 min		
Analytical Column:	Elite-1 (5 m x 0.05 mm x 0.05 $\mu\text{m}$ )		
Injection Port Type:	PSSI		
Injector temperature:	320 °C		
Injection Type:	Split		
Syringe volume:	5 $\mu\text{L}$		
Injection volume:	0.5 $\mu\text{L}$		
Injection Speed:	Fast		
Rinse Solvent:	Pentane		
Carrier Gas Type:	Hydrogen		
Carrier Gas Program:	40 Psig (constant pressure)		
Split Flow:	400 mL/min		
Oven Program:	<b>Temperature</b>	<b>Hold Time</b>	<b>Rate</b>
	37 °C	0.5 min	140 °C/min
	70 °C	0 min	105 °C/min
	115 °C	0 min	85 °C/min
	220 °C	0.6 min	End

**Table 2. Full scan mass spectrometer method conditions at each of five different data acquisition speeds.**

<b>Mass Spectrometer:</b>	<b>PerkinElmer Clarus 600 T</b>
GC Inlet Line Temperature:	250 °C
Ion Source Temperature:	250 °C
Function Type:	Full Scan
Full Scan range:	48-173 $m/z$
Scan Speed:	<b>12,500 da/sec</b>
Full Scan Time:	0.01 sec
InterScan Delay:	0.006 sec
Scan Speed:	<b>6250 da/sec</b>
Full Scan Time:	0.02 sec
InterScan Delay:	0.006 sec
Scan Speed:	<b>3125 da/sec</b>
Full Scan Time:	0.04 sec
InterScan Delay:	0.006 sec
Scan Speed:	<b>1250 da/sec</b>
Full Scan Time:	0.1 sec
InterScan Delay:	0.01 sec
Scan Speed:	<b>625 da/sec</b>
Full Scan Time:	0.2 sec
InterScan Delay:	0.01 sec

**Table 3. Single ion recording mass spectrometer method conditions.**

Mass Spectrometer:	PerkinElmer Clarus 600 T
GC Inlet Line Temperature:	250 °C
Ion Source Temperature:	250 °C
Function Type:	SIR
Scan Speed:	100 scans/sec
SIR Dwell Time:	0.005 sec
SIR Interchannel Delay:	0.005 sec
Summary of SIR Functions:	1 ion/function
Function 2:	<i>m/z</i> 91
Function 3:	<i>m/z</i> 57

**Table 4. SIFI spectrometer method conditions.**

Mass Spectrometer:	PerkinElmer Clarus 600 T
GC Inlet Line Temperature:	250 °C
Ion Source Temperature:	250 °C
Function Type:	SIFI
Full Scan range:	48-173 <i>m/z</i>
Full Scan Time:	0.02 sec
InterScan Delay:	0.006 sec
SIR Dwell Time:	0.01 sec
SIR Interchannel Delay:	0.005 sec
Summary of SIR Functions:	3 ions/function
Function 2:	<i>m/z</i> 91, 92, 106
Function 3:	<i>m/z</i> 57, 71, 85

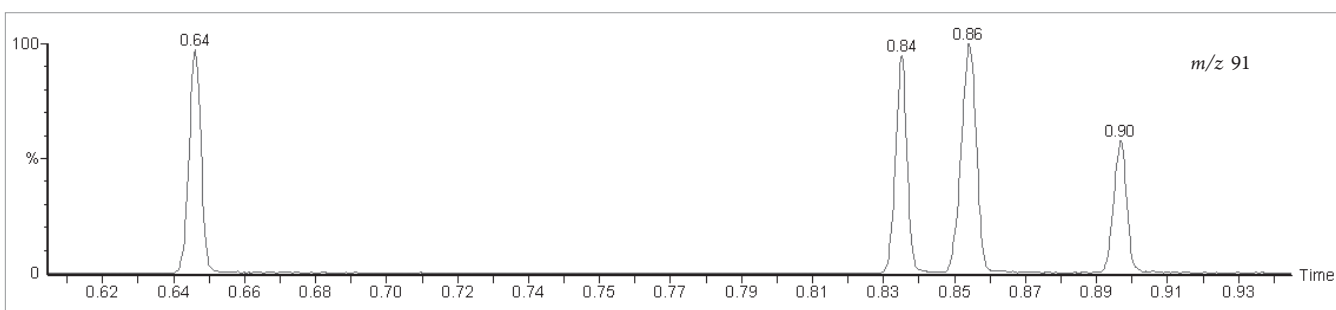


Figure 2. This chromatogram displays a 15 ng on-column injection of toluene, ethylbenzene, m&p-xylene, and o-xylene.

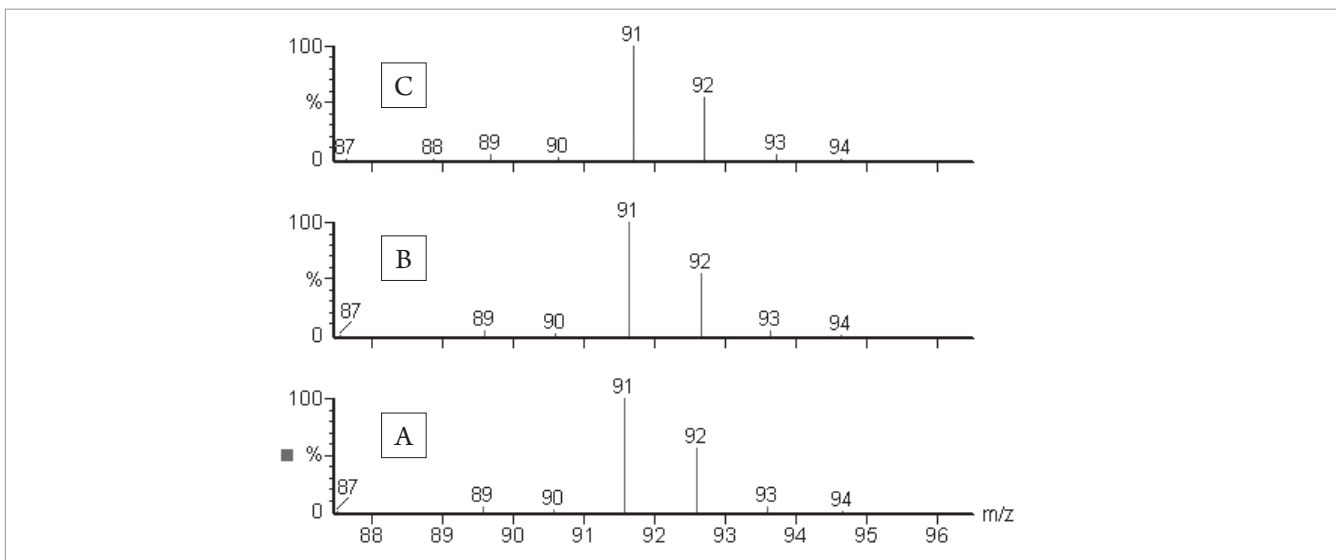


Figure 3. Background subtracted spectra of toluene, 'A' was collected at 3125 /u/sec, 'B' was collected at 6250 u/sec, 'C' was collected at 12,500 u/sec.

## Results

In the following sections, full scan, SIR, and SIFI, the various aspects of fast scanning will be demonstrated; data will be presented to verify that while operating at the top end speed, the Clarus 600 MS will remain precise and accurate.

### Full Scan

The Clarus 600 MS can acquire data at speeds up to 12,500 u/sec; the ability to reach these speeds while preserving spectral integrity is demonstrated in the following tests. Figure 2 demonstrates the analysis of volatile organic compounds (VOC's) under fast GC conditions, achieving a peak width of less than 1 second. In this case, approximately 40 scans were achieved across each peak.

While scanning in this mode it is necessary to show that spectral quality does not degrade over the scan speed range. To test this, multiple injections of the same standard were acquired at different scanning rates. Figure 3 depicts the spectra of toluene at 3125 u/sec, 6250 u/sec, and 12,500 u/sec. The ion ratios in Figure 3 are nearly identical across the range of scan speeds; demonstrating that spectral data remains constant across the scan range.

### SIR

The maximum speed of data acquisition in SIR mode is 100 scans/sec for a single mass; this was verified with the analysis of ethylbenzene and xylenes. Figure 4 demonstrates an example of this analysis, again the peak width is less than one second and the number of scans across each peak corresponds with the expectation of 100 points per second. For example, ethylbenzene has a peak width of 0.66 seconds and 66 scans, the scans across the xylene peaks follow suit.

### SIFI

The fast scanning capability can be combined with fast GC and SIFI acquisition to achieve high sensitivity of SIR and full spectral data of scanning acquisition. This demonstration achieves the necessary 8-12 scans per peak desired for precise and accurate peak definition. Figure 5 demonstrates the fast SIFI analysis of c-10 to c-13 alkanes. A minimum of 10 data points were collected across each peak, with each point containing both full scan and SIR data. This demonstrates that the electronics of the Clarus 600 MS are capable of providing sufficient speed to perform the most rigorous types of data collection under extremely fast GC conditions.

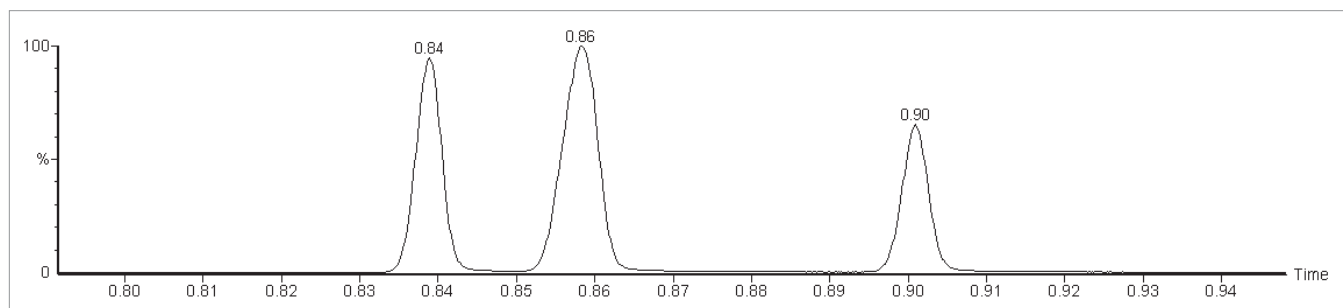


Figure 4. SIM mode data collection at 100 points/sec of 15 pg on column of ethylbenzene, m&p-xylene, and o-xylene.

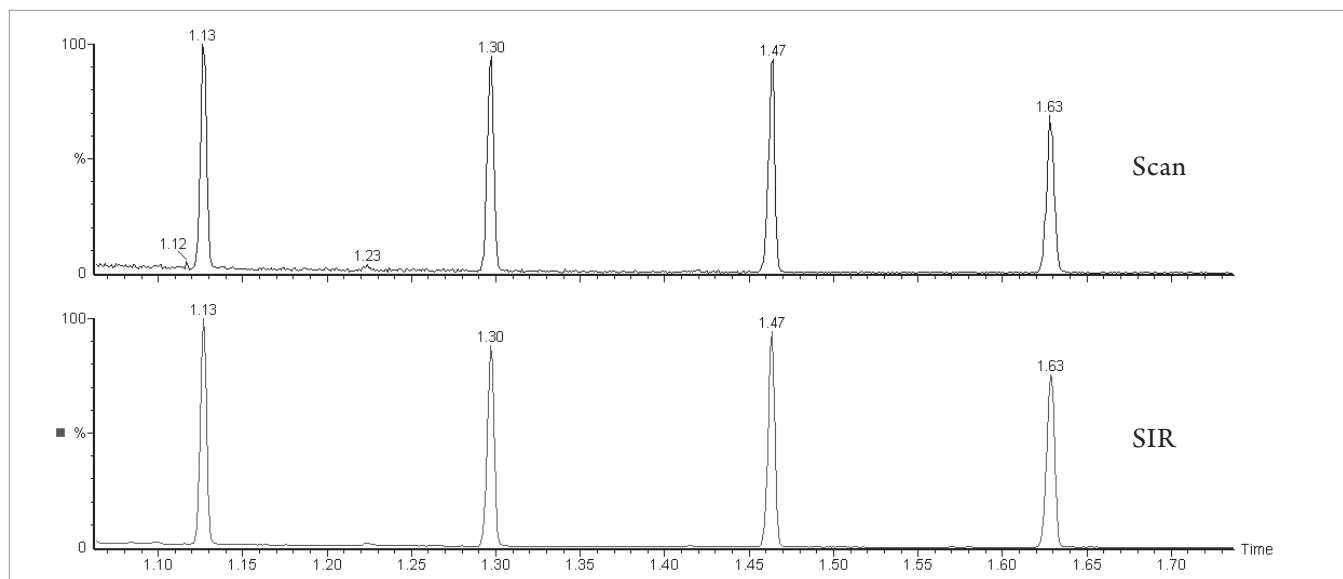


Figure 5. Full scan and SIR chromatograms obtained from a single SIFI analysis of 15 pg on column of c-10 through c-13 alkanes.

## Conclusion

The data acquisition capability of the Clarus 600 MS allows laboratories to push their chromatograph to achieve the narrowest peaks possible, while retaining the ability to collect sufficient data for peak shape definition and quantitation. The ability to collect both SIR and full scan data at high speeds allows outstanding flexibility in the creation of MS methods. It is used to detect low level banned pesticides in the presence of high level matrix and for quantitation of semi-volatile organics across a wide dynamic range in environmental samples.

In this example, the volatile organic components toluene, ethylbenzene, and xylene were analyzed under conditions which produced peaks less than one second wide at base. The data demonstrates that sufficient scans were collected across each peak and the mass spectral data remains consistent. Additionally, fast scanning was applied to SIFI data acquisition of extremely narrow GC peaks demonstrating the Clarus 600 MS will precisely and accurately record narrow GC peaks while collecting simultaneous full scan and SIR data.

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