

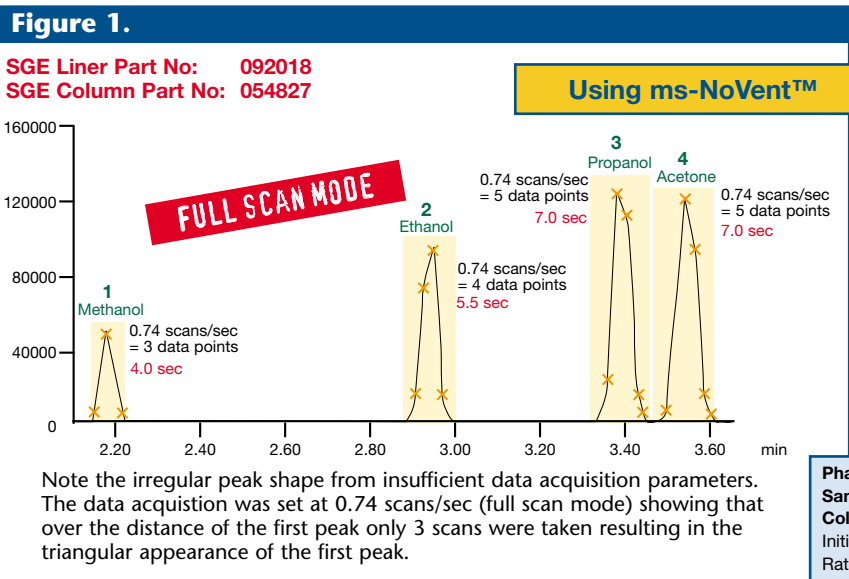
# HOW TO IMPROVE YOUR GC-MS ANALYSIS

## 1 SAMPLING RATE

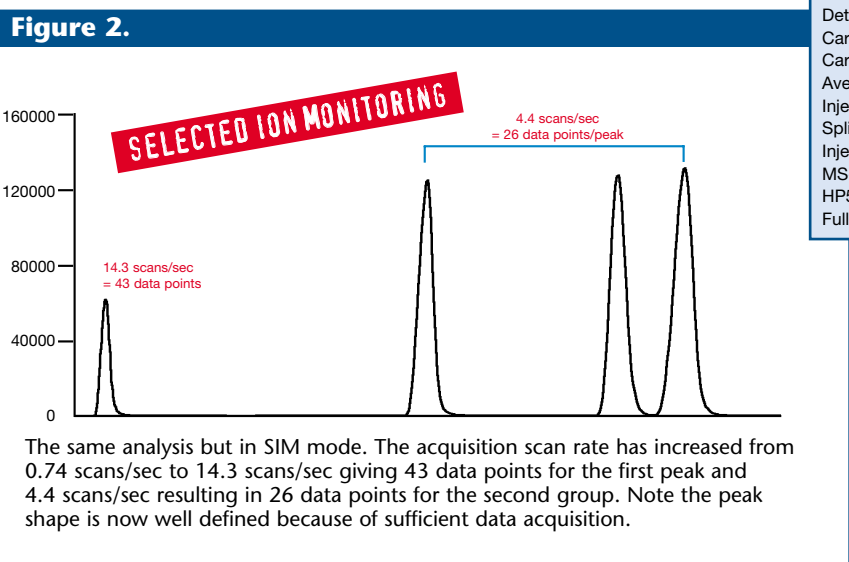
The data acquisition-sampling rate is important. Have you ever seen peaks like those shown in **Figure 1**? These sort of irregular peaks are most likely to happen at the start of the chromatogram and are caused by an incorrect setting of the mass spectrometer's sampling rate. If a peak is 2 seconds wide at its base, then the sampling rate needs to be set to 5 scans/sec to obtain enough data points across the peak for good quantitation. If this is not the case and the scan rate is too low, then peaks can 'look' quite irregular, sometime even triangular. In the worst case, if the scan rate is set too low, the peak can be 'missed' by the detector altogether resulting in no response.

**Figure 2** shows an analysis of the same chromatogram but instead of full scan mode, Selected Ion Monitoring (SIM) has been used. Note, immediately the improved peak shape of the early eluting peaks. The reason for this is that now the scan rate has been increased from 0.74 scans/sec to at least 4 scans/sec depending on the SIM window selected. Because in SIM mode the mass spectrometer is 'targeting a limited mass range', the number of scans across the peak has increased resulting in better peak shape. This is an easy solution for getting better quantitation for early eluting peaks.

Inspect the ions obtained for the peak in full scan mode and use at least one of the ions in SIM to obtain a better scan rate.



Phase:	BP624 1.2µm film
Sample:	500ppm in DMSO
Column:	30m x 0.22mm ID
Initial Temp:	37°C, 7min
Rate 1:	30°C/min to 190°C
Final Temp:	190°C, 2min
Detector Type:	MSD
Carrier Gas:	He, 6.5psi
Carrier Gas Flow:	1.3mL/min
Average Linear Velocity:	35cm/sec at 37°C
Injection Mode:	Split
Split Ratio:	150:1
Injection Volume:	1µL
MS-NoVent Part No:	113400
HP5973 Restrictor:	113409
Full Scan / SIM:	Full scan mode 45-450



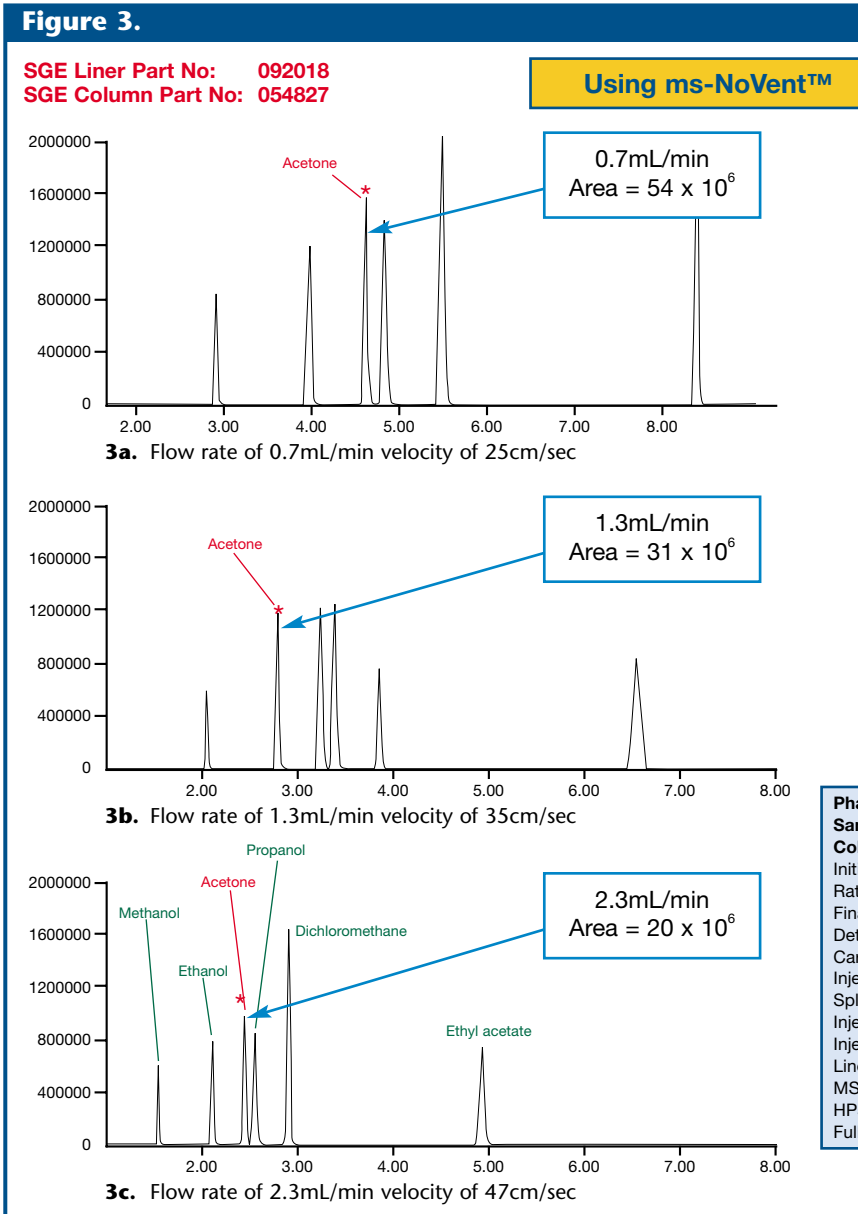
# 2 FLOW RATE

What about flow rate? Increasing the inlet pressure of the injector will result in faster chromatography but this can also have a detrimental effect on sensitivity. The reason for this is that the higher the flow rate of helium into the ionization chamber of the mass spectrometer, the greater the number of collisions between analyte molecules and helium. The result of this is a decrease in sensitivity. This can be clearly seen in **Figure 3**. As the flow rate is increased, the peak area of the labelled peak (acetone) decreases from a count of  $54 \times 10^6$  to  $20 \times 10^6$ .

This phenomenon described above can also be due to ① detuning of the source with more helium, ② lower analyte concentration to number of electrons and helium and ③ shorter residence time in the source.

# 3 AUTOTUNE

Most modern GC-MS benchtop instruments are very easy to operate. Operators will know they have to perform an Autotune periodically to maintain performance. What oven temperature should this tune be carried out at? Good chromatographers often choose a temperature at the midpoint of the method. For example, if the method is carried out from 100 to 300°C, then 200°C is chosen. It is worth remembering, however, that a higher oven temperature will result in less carrier gas flow into the mass spectrometer — gases become more viscous with increased temperature and the flow rate will decrease. Less flow will result in a higher efficiency of ionization of the calibration standard as explained earlier. This will result ultimately, in a lower electron multiplier voltage as a result of the autotune and will therefore increase the lifetime of your electron multiplier — the ‘detector’ in your mass spectrometer.



Phase:	BP624 1.2µm film
Sample:	10ppm in Methanol
Column:	30m x 0.22mm ID
Initial Temp:	37°C, 8min
Rate 1:	30°C/min to 190°C
Final Temp:	190°C, 2min
Detector Type:	MSD
Carrier Gas:	He
Injection Mode:	Split
Split Ratio:	100:1
Injection Volume:	1µL
Injection Temperature:	250°C
Liner Type:	4mm ID Double Taper Liner
MS-NoVent Part No:	113400
HP5973 Restrictor:	113409
Full Scan / SIM:	Full scan mode 45-450