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76% Increase in Throughput for Determination of Semi-Volatiles Using Narrow-Bore GC Columns and Rapid Data Acquisition with a Highly Sensitive Quadrupole GCMS System

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Introduction

Laboratories analyzing semi-volatile organics by methods such as Method 625 and Method 8270 typically use 0.25 mm ID capillary columns. Depending on method conditions, simply running the required quality control (QC) samples can consume a significant portion of a 12-hour shift, leaving less time for analysis of revenue-generating samples. A cycle time for a single injection on a standard single quadrupole Gas Chromatograph Mass Spectrometer (GCMS) may be as long as 30 minutes. Thus, in a 12-hour tune cycle with QC, including a continuing calibration verification (CCV), method blank (MB), laboratory control sample (LCS), laboratory control sample duplicate (LCSD), matrix spike (MS), and a matrix spike duplicate (MSD), per analytical batch you may only have time for 18 actual samples before having to verify the tune.

Fast GC uses narrow bore (0.15mm ID) columns, rapid oven heating and cooling, and rapid data acquisition to decrease runtime. A sensitive quadrupole mass spectrometer is necessary for identification and quantitation of target compounds at sub-nanogram levels. In addition, fast mass-spectral scanning and data acquisition is required because of the very narrow chromatographic peaks produced under these conditions.

The Shimadzu GCMS-QP2020 is a new, sensitive, fast-scanning single quadrupole GCMS system capable of significantly reducing the run time for EPA methods 625 and 8270.

Analysis with a 0.25mm x 30 m Column

The GCMS-QP2020 highly sensitive mass spectrometer provides excellent sensitivity in split injection mode. Split injection introduces less sample matrix into the column and detector, minimizing instrument maintenance.

The split injection improves productivity, allowing a higher starting temperature to shorten runtimes and the oven cooldown time (Figure 1).

Figure 1: GC oven cooldown from 330°C to 60°C in 4 minutes

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Taking advantage of rapid oven cooling alone shortens the GC cycle from 30 minutes to 25 minutes. This enables you to run 22 real samples with 10% QC in one 12-hour tune cycle. Raising the starting temperature to 60 °C instead of 40 °C shortens the run to 20 minutes allowing 27 samples in a 12-hour period.

Table 1 lists the GCMS operating conditions for the 0.25 mm ID column. Using the conditions described in Table 1, all compounds eluted in about 16.5 minutes. The first eluting compound, N-Nitrosodimethylamine, had a retention time of about 2 minutes. Figure 2 is an example TIC chromatogram. The patented, inert, high-efficiency Ion Shield source ensures maximum sensitivity and very little breakdown of even the most active analytes.

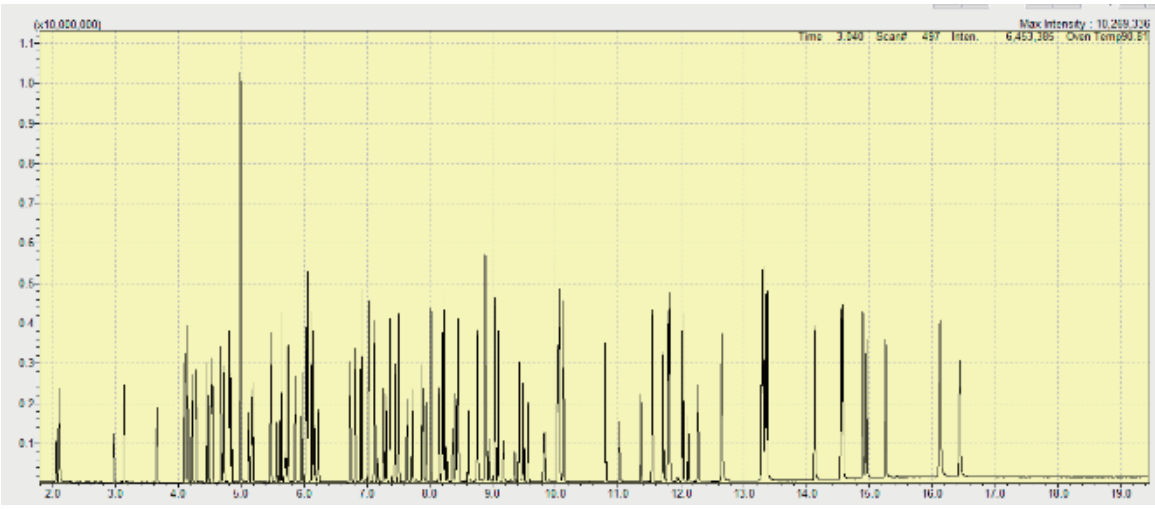

Figure 2: TIC Chromatogram using a 0.25mm x 30 m column

Table 1: GCMS Operating Conditions

Column	: 0.25mm x 0.25 mm x 30 m RTX-5Sil MS
Flow Control using constant linear velocity	: 45 cm/ second
Pressure	: 94.5 kPa
Injection Temperature	: 295 °C
Total Flow	: 47.7 ml / minute
Column Flow	: 1.52 ml / minute
Split ratio	: 30:1
GC temperature program	: 60 °C for 1.5 minutes
	20 °C / minute to 330 °C
	Hold 4.5 minutes
Total GC program time	: 19.5 minutes
Ion Source temperature	: 210 °C
Interface temperature	: 300 °C
Solvent cut time	: 1.75 minutes
Scan range	: 35 – 500 amu
Event time	: 0.15 seconds
Scan speed	: 3333
Start time	: 1.8 minutes
End time	: 19.45 minutes

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Figures 3, 4, and 5 are examples of the instrument calibration and peak shape. Peaks shown are at the lowest calibration level for that compound.

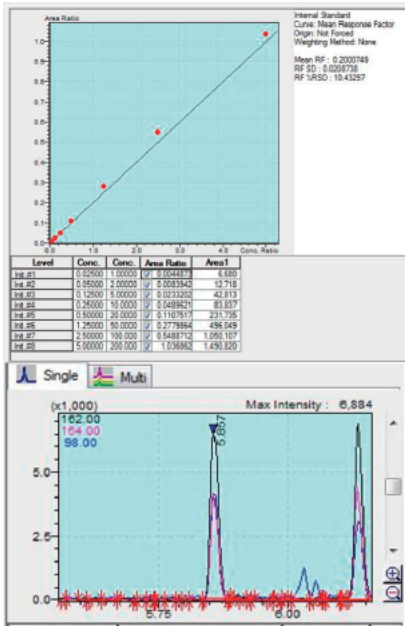

Figure 3: 2-Chlorophenol (0.2 µg/ml)

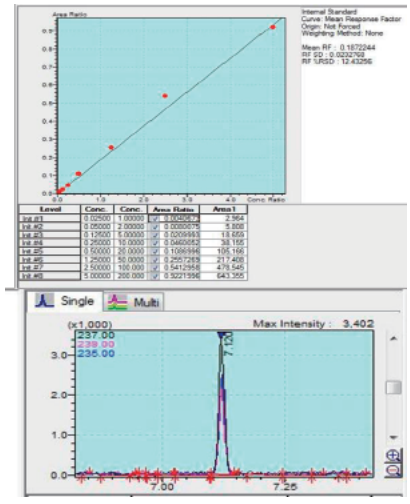

Figure 4: Hexachlorocyclopentadiene (0.5 µg/ml)

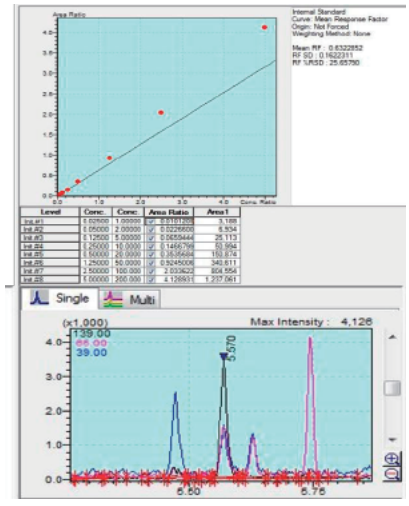

Figure 5: 2-Nitrophenol (1.0 µg/ml)

Analysis with a 0.15mm x 20 m Column

Changing to a smaller ID column results in narrower peaks and even faster analysis times. This can be a significant advantage, but it requires a fast scanning instrument. Resolution, precision, and peak shape vary depending on the number of scans (data points) across a GC peak (Figure 6); the more scans per peak the better the precision. With scan rates up to 20,000 u per second, the Shimadzu GCMS-QP2020 can easily meet these demands, resulting in faster analysis while maintaining high precision. To ensure adequate precision and accuracy, an event time of 0.1 seconds was necessary.

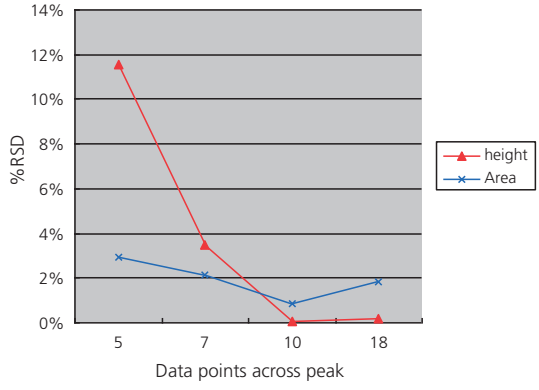

Figure 6: Increase in precision with more data points across a peak

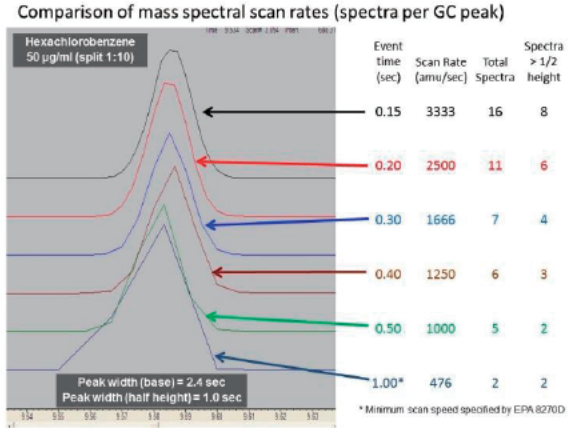

Figure 7: Peak shape improvement with more scans across a peak

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Table 2: GCMS Operating Conditions

Column	: 0.15mm x 0.15 mm x 20 m RTX-5Sil MS
Flow Control using constant linear velocity	: 45 cm/ second
Pressure	: 281.4 kPa
Injection temperature	: 295 °C
Total Flow	: 47.7 ml / minute
Column Flow	: 1.01 ml / minute
Split ratio	: 30:1
GC temperature program	: 60 °C for 1.0 minutes
	32.5 °C / minute to 330 °C
	Hold 2.69 minutes
Total GC program time	: 12.0 minutes
Ion Source temperature	: 220 °C
Interface temperature	: 320 °C
Solvent cut time	: 1.0 minute
Scan range	: 35 – 500 amu
Event time	: 0.10 seconds
Scan speed	: 5000
Start time	: 1.05 minutes
End time	: 11.95 minutes

Using the conditions described in Table 2, all compounds eluted within 12 minutes.

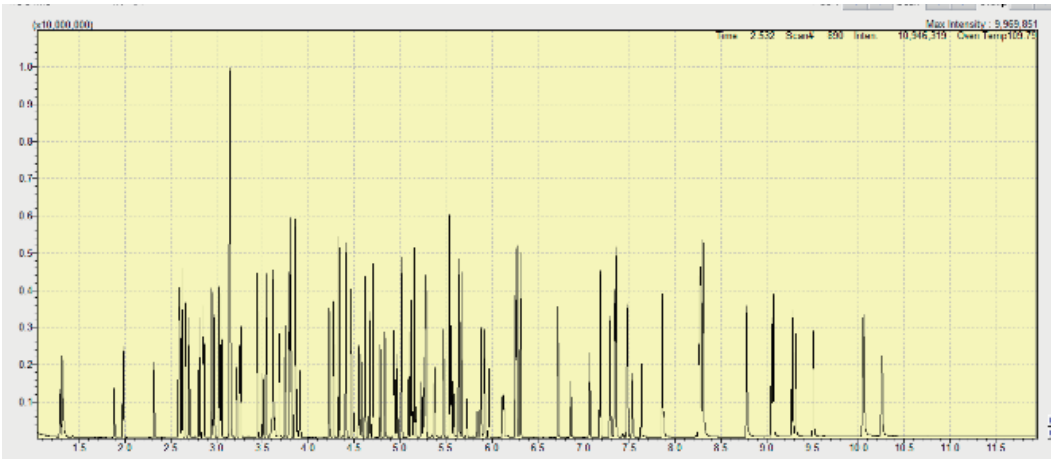

Figure 8: TIC chromatogram using a 0.15 mm x 20 m column

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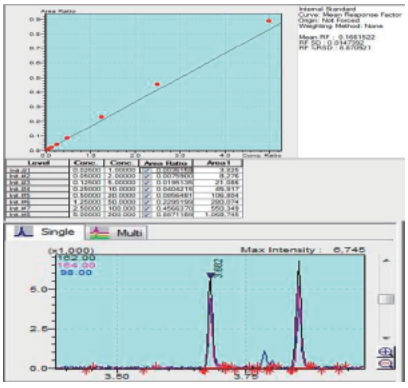

Figure 9: 2-Chlorophenol at 1.0 µg/ml

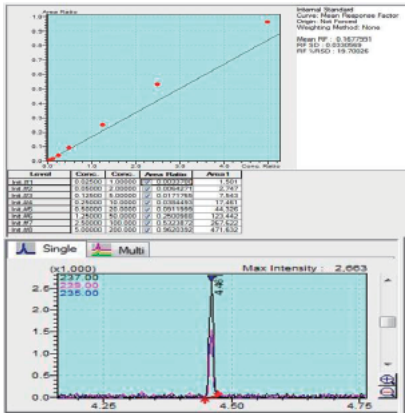

Figure 10: Hexachlorocyclopentadiene 1.0 µg/ml

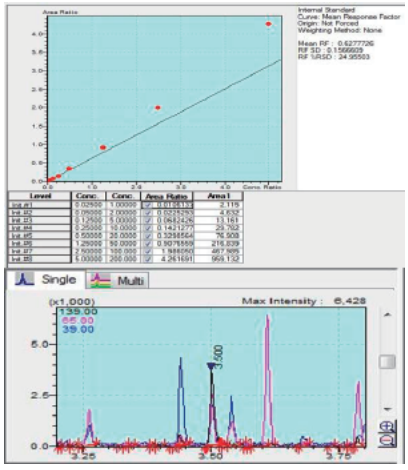

Figure 11: 2-Nitrophenol at 1.0 µg/ml

Discussion

The original chromatography conditions allowed analysis of only 18 samples during a 12-hour tune interval. Taking advantage of rapid oven cooling increased throughput to from 18 to 22 samples during a 12-hour tune cycle. With a new oven program, 27 samples is possible per 12-hour shift. A narrower bore column decreased the total cycle time from 24 minutes to 17 minutes, increasing throughput to 32 samples. The combination of the fast cooling oven, narrow bore column, and rapid mass spectrometer scanning results in an increase of 14 more samples per 12-hour shift. (See Figure 12).

Chromatography does not suffer with the narrow bore column; peaks are simply narrower (Figure 13).

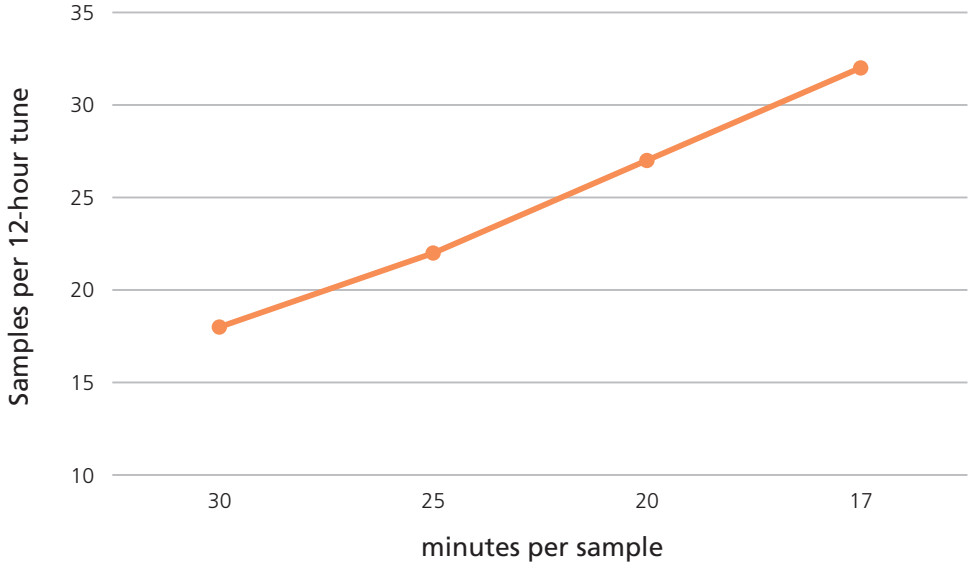

Figure 12: CGC run times showing samples run per 12-hour tune interval

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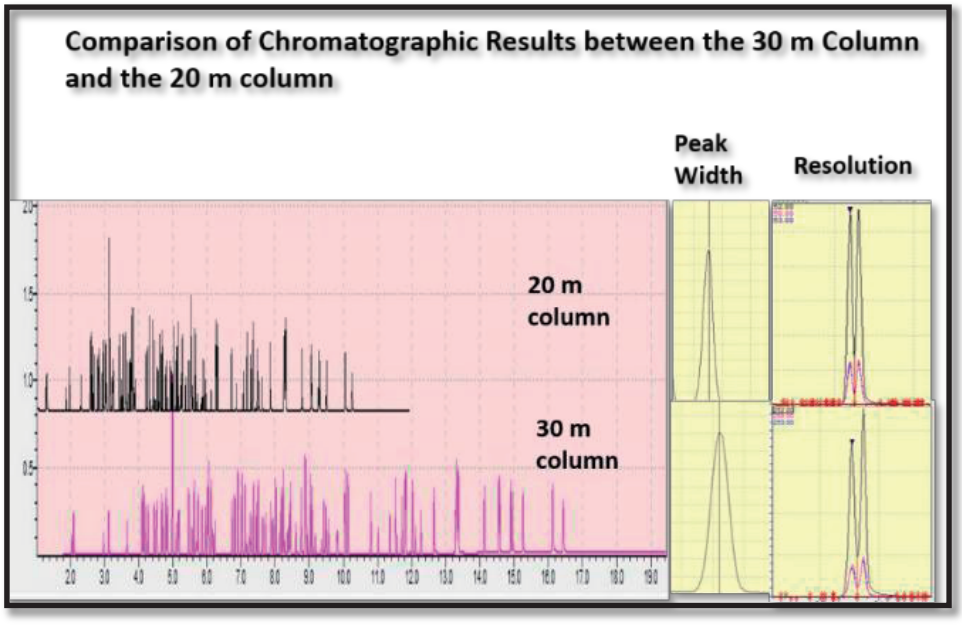

Figure 13: CGC run times showing samples run per 12-hour tune interval

Detection limits with the 0.15 mm ID column showed no statistical difference compared to the 0.25 mm ID column. (Figure 14 T Test)
Overall precision (Figure 15 T Test) was slightly better with the 30-meter column, however, the precision for both columns are well within the specifications of EPA Methods 625 and 8270.

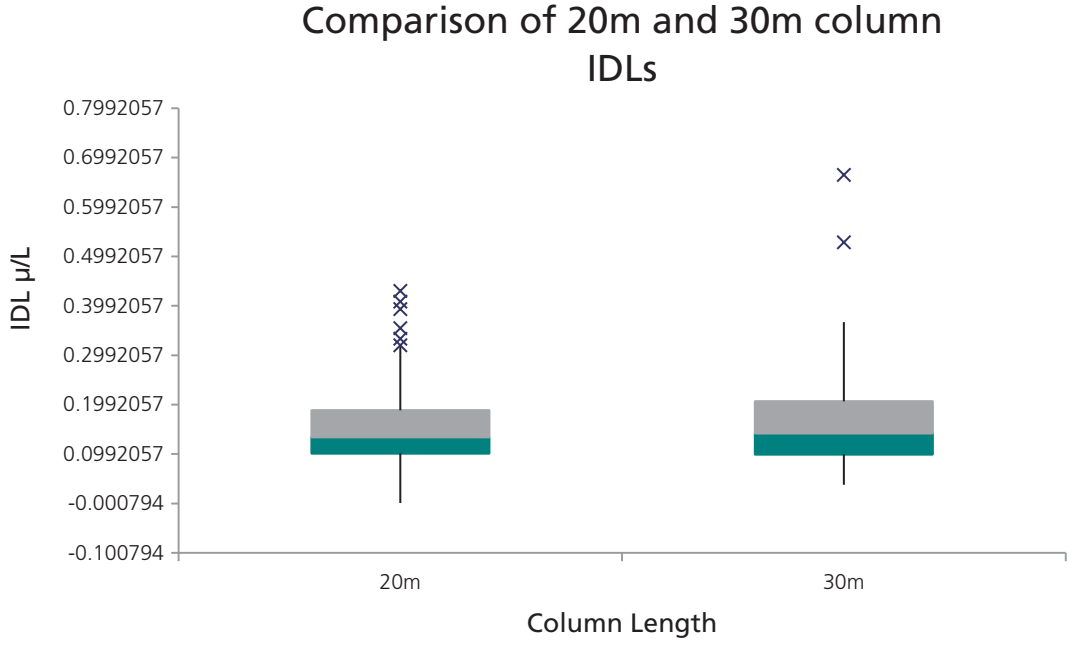

Figure 14: T Test comparing 20 meter and 30 meter column IDLs

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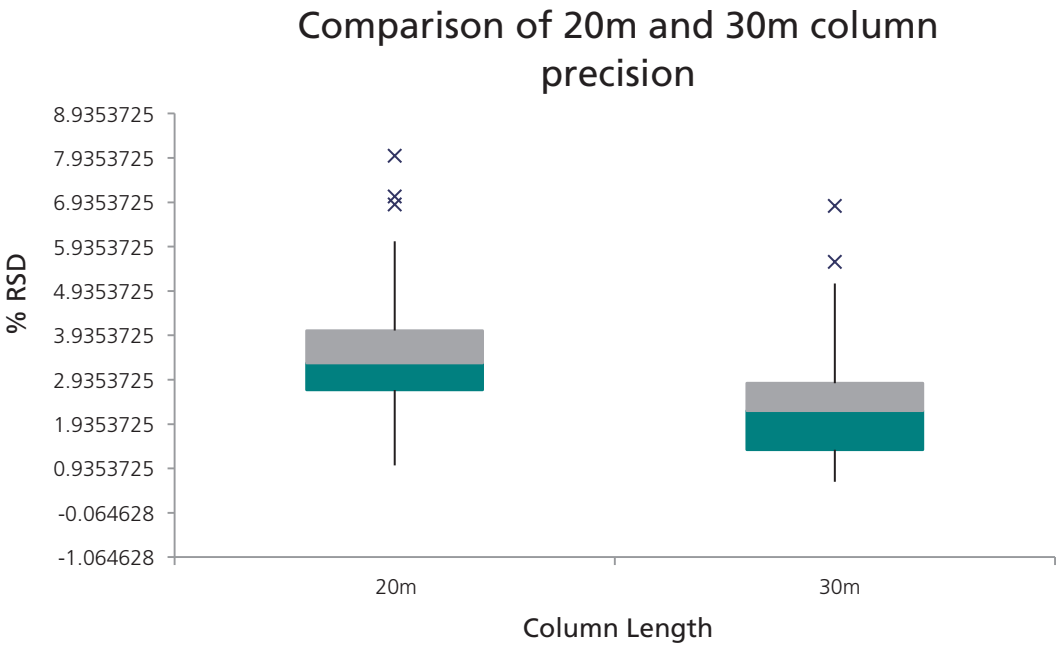

Figure 15: T Test comparing 20 meter and 30 meter column %RSD

Benefits of the Shimadzu GCMS-QP2020

Assume the laboratory work hours enable analysis of samples during two 12-hour tune intervals every day, five days per week. Also, assume that there is 35% downtime due to calibration, quality control, maintenance, and re-runs, with an efficiency of 85%. Decreasing the column ID from 0.25 mm to 0.15 mm and taking advantage of rapid oven cooling shortens the sample run time from 30 minutes to 17 minutes. This allowed method modification enables the laboratory to increase throughput from 133 samples to 234 samples per week. The 76% increase in throughput was calculated using the following formula:

$$\% \text{ increase} = \left(\frac{[234-133]}{133} \right) \times 100$$

These examples illustrate the benefit of fast GCMS to increase your labs productivity enabling you to run more samples in less time and make more money.

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Conclusion

Fast oven cooling combined with the rapid scanning mass spectrometer available on the Shimadzu GCMS-QP2020 allows you to shorten analysis times compared to other GCMS instruments. The GCMS-QP2020 is highly precise, highly accurate, and meets or exceeds U.S EPA Method 625 or 8270 requirements.

For further information

For a more complete discussion of the topics described here, please leave your card or send a request for the full technical report via www.ssi.shimadzu.com

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