

Fast GC–TOF MS for sensitivity and efficiency improvements in the analysis of SVOCs

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Introduction

The evaluation of semi-volatile organic compounds (SVOCs) in environmental matrices, such as that detailed by US EPA Method 8270, poses many challenges for the analyst, not least the vast number that may be encountered in any given analysis.

In addition, analyte reactivity and the potential for multiple analytes to co-elute can make achieving the required method detection limits quickly and cost-effectively a formidable task – which is especially the case when using quadrupole mass spectrometry (qMS) in ‘scan’ mode. Selected ion monitoring (SIM) can be used to improve detection limits, but it limits speed of analysis due to SIM window restrictions and prevents retrospective searching of the data for new emerging contaminants.

This study demonstrates improvements to the analysis of SVOCs following US EPA Method 8270, using GC–TOF MS for increased sensitivity, a wide dynamic range and data-rich chromatograms containing high-quality, library-searchable spectra.

Experimental

- Samples:** A 10-point calibration series was prepared from 2–2000 ppb using an 85-component standard mix of SVOCs and pesticides.
- Injection:** 1 µL pulsed splitless liquid injection.
- Sample throughput:** 13 minute injection-to-injection cycle time.
- MS:** BenchTOF-HD™ (SepSolve Analytical)
- Software:** Instrument control and data processing using ChromSpace®.



Figure 1: Photograph of the GC–TOF MS used in this study.

Results and discussion

DFTPP tuning criteria

Table 1: DFTPP tuning result for GC–TOF MS.

Ion ratio	Lower limit	Upper limit	Result	Pass/Fail
51:442	10%	80%	23.60%	Pass
68:69	0%	2%	0.59%	Pass
70:69	0%	2%	1.05%	Pass
127:442	10%	80%	32.00%	Pass
199:198	5%	9%	6.62%	Pass
275:442	10%	60%	22.50%	Pass
365:198	1%	100%	3.08%	Pass
441:442	1%	24%	11.51%	Pass
442:198	50%	200%	172.58%	Pass
443:442	15%	24%	20.03%	Pass

DFTPP tuning was performed at the start of the study to show that all of the instrument tuning and performance criteria for US EPA Method 8270 were met.

Fast GC–TOF MS analysis

Figure 2 shows the separation achieved for fast GC–TOF MS of the 85-component mix, with an injection-to-injection run time of just 13 minutes. The BenchTOF MS used in this study, provides reference-quality spectra, as seen in the examples shown in Figure 3, with excellent matches to the NIST library for confident identification.

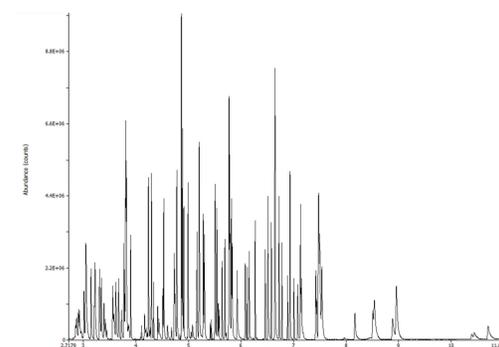


Figure 2: GC–TOF MS (TIC) chromatogram for the 100 ppb standard mix, with all 85 components separated in under 11 minutes.

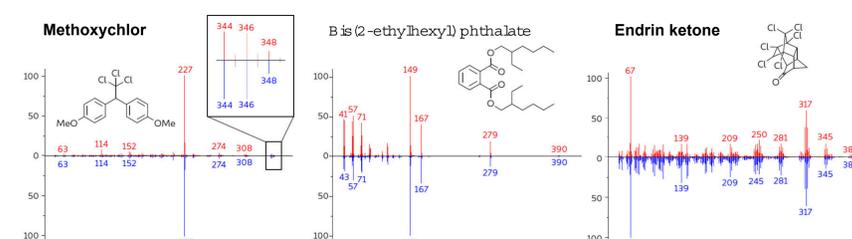


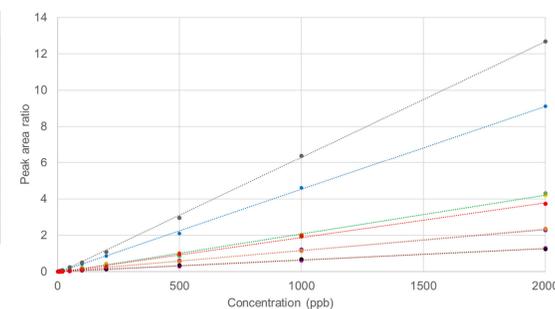
Figure 3: Reference-quality spectra generated by BenchTOF MS for three analytes in the standard.

Linearity

Linearities were calculated using a calibration series from 2–2000 ppb (Figure 4). The R² values were excellent – all were found to be >0.995. Repeat analysis (n = 5) and quantitation of a mid-point standard gave RSDs <15% for all targets.

Compound	R ²
Phenol	0.9985
N-Nitroso-n-propylamine	0.9994
Hexachlorobutadiene	0.9997
4-Nitrophenol	0.9959
β-BHC	0.9990
Fluorene	0.9996
Heptachlor	0.9978
Di-n-octyl phthalate	0.9996
Dibenz[a,h]anthracene	0.9977

Figure 4: Calibration curves for a selection of compounds in the standard (2–2000 ppb).



Improved productivity

Not only does the fast injection-to-injection time of 13 minutes increase productivity in this study, software workflows can also provide substantial time savings. ChromSpace software enabled real-time data processing to be employed during analysis. This allowed background-subtraction, integration, identification and quantitation to be performed automatically as part of the acquisition sequence, all while the samples were still being acquired. This means that the results are ready to review and report as soon as the analysis is complete, for improved productivity.

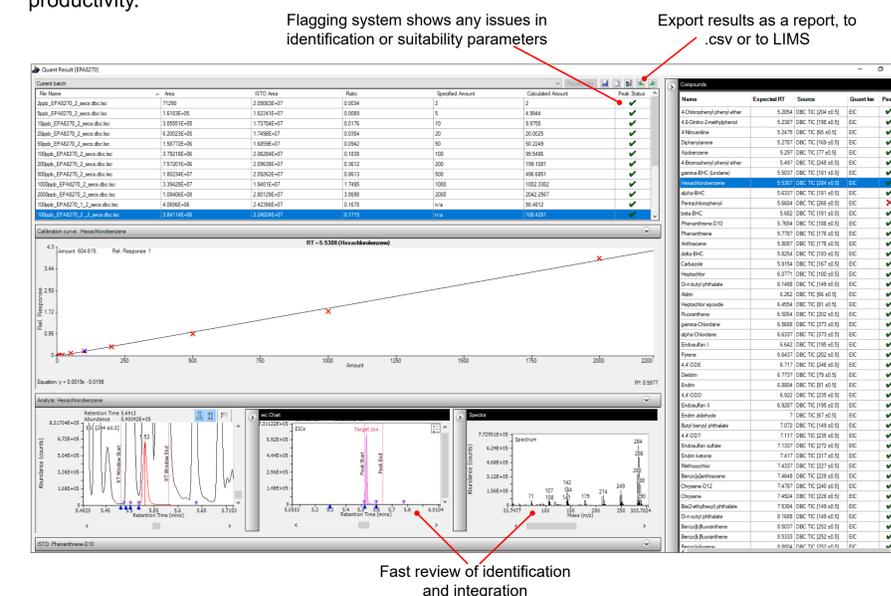


Figure 5: Screenshot of quantitative results browser in ChromSpace software, showing calibration results for hexachlorobenzene.

Quantitative results can be reviewed effortlessly in the results browser in ChromSpace – with simple, easy-to-use tools for further time savings (Figure 5).

Conclusions

In this poster, we have demonstrated how GC–TOF MS can provide:

- Confident identification using reference-quality spectra from BenchTOF mass spectrometers.
- Low detection limits for targets, while also allowing retrospective searching for non-targets.
- Excellent reproducibility and linearity across wide-ranging target compounds.
- Streamlined workflows are possible using ChromSpace software, with full instrument control and real-time data processing.