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Automated Sample Preparation using PAL3 RTC System for EPA 8270E Semi-volatile Organic Analysis by GC/TQ

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Introduction

Semi-volatile organic compounds (SVOC) analysis is widely implemented in analytical laboratories. In contrast, growing demand for this analysis reveals bottlenecks, e.g., a lack of practical sample preparation experience, low sample throughput, and high consumption of chemical solvents. Online automated sample preparation is gaining attention as a solution to address these laboratory challenges. This study presents a proof-of-concept on a novel automated sample preparation workflow modified based on the EPA 3510C method followed by gas chromatography/triple quadrupole mass spectrometry (GC/TQ) analysis according to EPA 8270E.[1, 2] 100 analytes of SOVC were tested in surface water samples and the analytical result was evaluated in terms of calibration linearity, method blank (MB), method sensitivity, and matrix-spiked QC recovery.

Experimental

Instrumentation

An Agilent 7000 series GC/TQ coupled to an Agilent 8890 GC together with PAL3 Series II RTC system was used for the calibration/sample preparation and injection. The integrated PAL3-GC/TQ system was controlled by Agilent MassHunter Workstation GC/MS Data Acquisition software.

Figure 1. Agilent PAL3 RTC System on Agilent 7000 Series GC/TQ

Experimental

Method Parameters

The GC was operated in pulsed splitless mode and chromatographic separation was performed on an Agilent J&W DB-UI8270D column (30 m x 250 µm x 0.25 µm). Table 1 contains the operating parameters for both GC and MS.[3]

Table 1. Method Parameters

GC Parameters			
Injection Volume	2.0 µL		
Inlet Temp.	250 °C		
Carrier Gas	He, constant flow, 1.2 mL/min		
Transfer Line	320 °C		
Oven Program	Initial Temp: 40 °C for 0.5 min		
	Rate (°C/min)	Final Temp (°C)	Hold Time (min)
	25	260	9.3
	5	280	13.3
	25	320	18.9
TQ Parameters (EI Mode)			
Acquisition Mode	dMRM		
Ion Source Temp.	320 °C		
Quadrupole Temp.	150 °C		
EMV Mode	Gain factor (10)		
Solvent Delay	1.5 minutes		

Automated Calibrators and Sample Preparation

Calibration levels, method blank, and spiked QCs were prepared automatically using PAL3 system. Liquid-liquid extraction (LLE) per EPA 3510C was followed. The only manual step involved was addition of 15 mL of the water sample into a 20 mL vial followed by adding 1g NaCl. The rest of the sample preparation was automated (Figure 2).

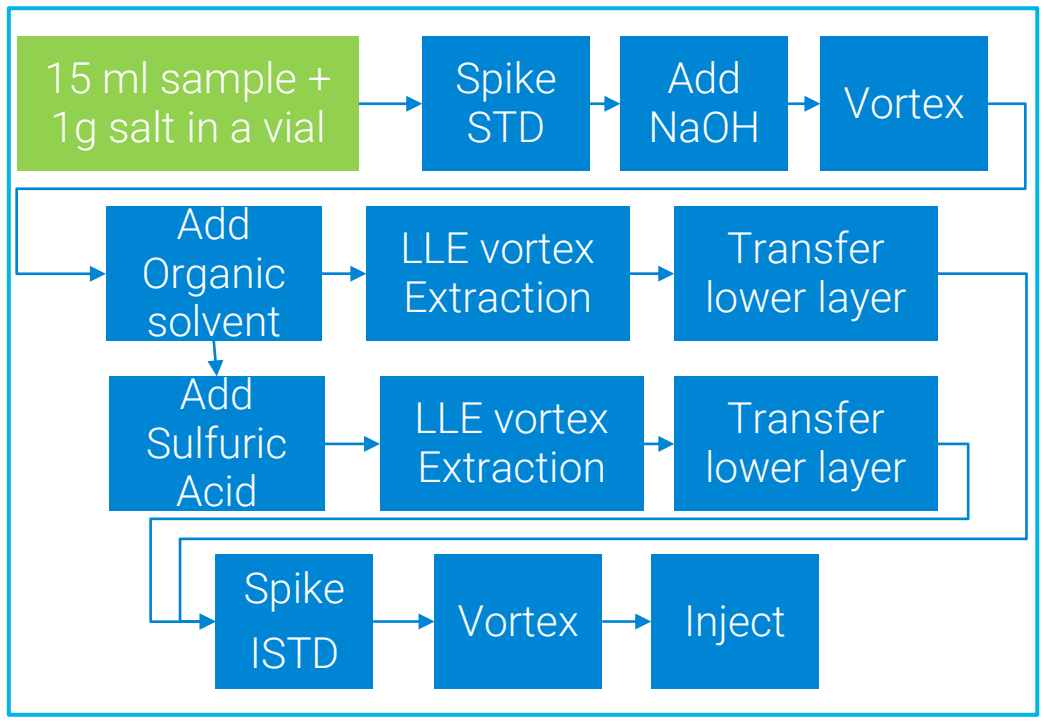

Figure 2. Automated sample preparation by the PAL3 system.

Online Analysis Sequence for Lab Productivity

Online analysis sequence was established covering 10 working calibration standards (0.01, 0.02, 0.05, 0.1, 0.2, 0.5, 1.0, 5.0, 10.0, and 20.0 ppm), MB, and matrix-spiked QC samples. Figure 3 shows online sample preparation and sample analysis proceeding in a parallel mode on the integrated PAL3-GC/TQ system. Thus, the lab productivity was increased through automation and eliminating waiting time between runs. MB was carried out through all stages of sample preparation and analyzed for the compounds of interest as a safeguard against lab contamination caused by the sample, the reagents used, and the preparation workflow.

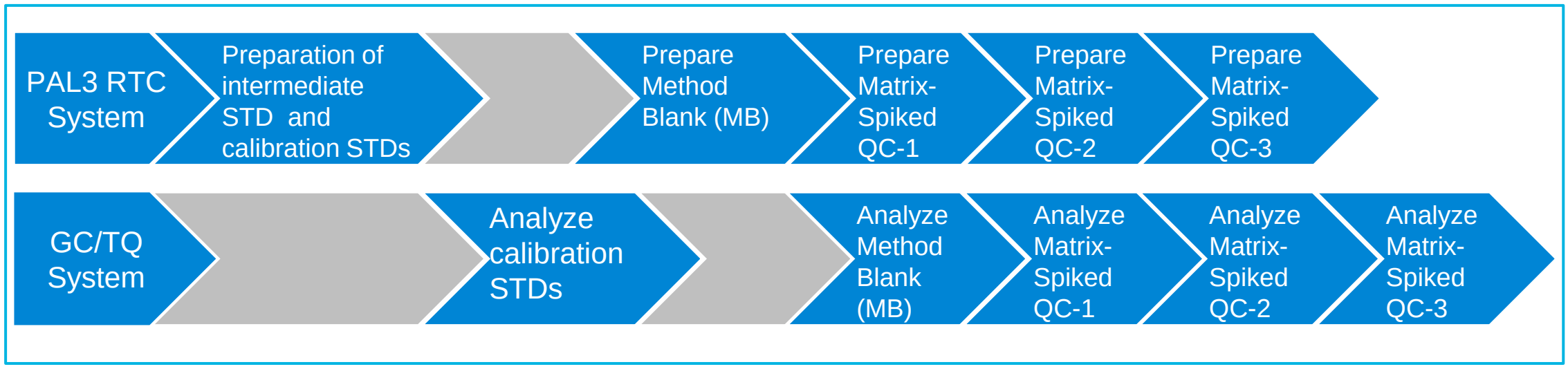

Figure 3. Online analysis sequence on the integrated PAL3-GC/TQ system

Compound Identification

Figure 4 shows a representative MRM chromatogram of the 100 analytes at 5 µg/mL prepared by the PAL3 system. The symmetric sharp peaks demonstrate the efficient chromatographic separation of targets within the relation time window.

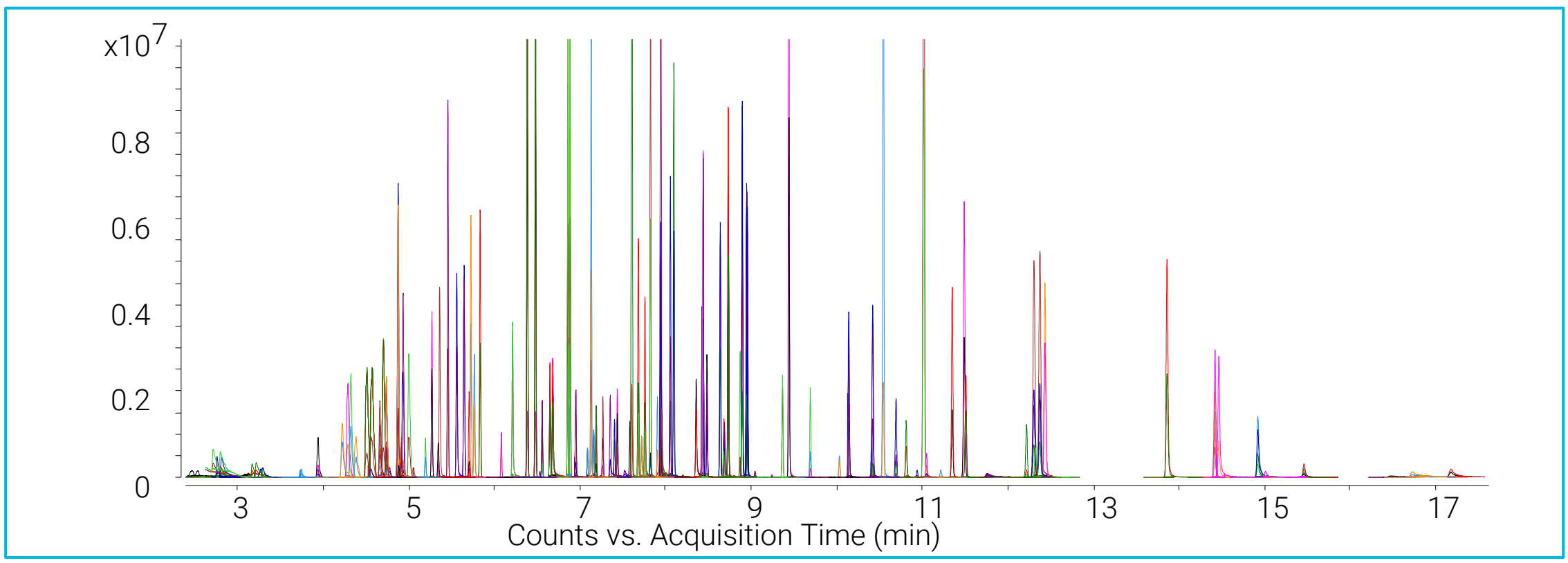

Figure 4. Representative MRM overlay for 100 analytes at 5 µg/mL and ISTDs at 2 µg/mL in DCM

Calibration Range

About 96% of compounds achieved R>0.995 with a minimum of 5 points, and 97% of compounds met the accuracy requirement for each calibration level.

Internal standards (ISTD) including 1,4-Dichlorobenzene-d4, Acenaphthene-d10, Chrysene-d12, Naphthalene-d8, Perylene-d12 and Phenanthrene-d10 were also assessed. The absolute RT change for ISTDs was within the regulatory recommendation of ≤ 30 secs. The response of all ISTDs in the individual standard was obtained within 70 to 150% of the average response throughout the final calibration range.

Results and Discussion

Sensitivity

The LLOQ of 100 analytes was distributed across 0.01 to 0.5 µg/mL (Figure 5).

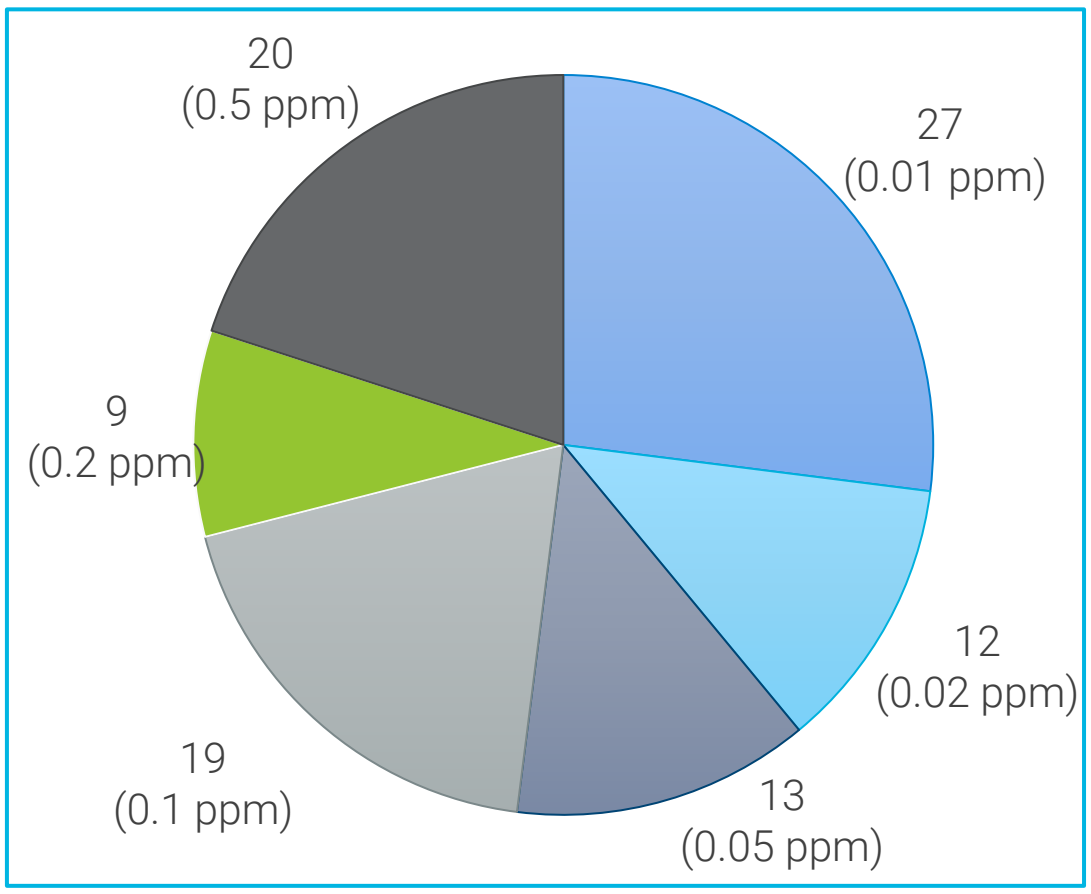

Figure 5. LLOQ distribution of 100 compounds

Matrix-spiked QC Recovery

Three technical replicates of matrix-spiked QC (n=3, 2 µg/mL in the final extract) were prepared by the PAL3 system to evaluate the reproducibility and robustness of the automated sample preparation. Each QC was analyzed by GC/TQ in duplicates to account for the homogeneity of the QC solution and the repeatability of spiked recovery. Overall, 96% of compounds met recovery 50 to 150%, and 98% of compounds obtained RSD of recovery ≤20% (Figures 6 and 7).

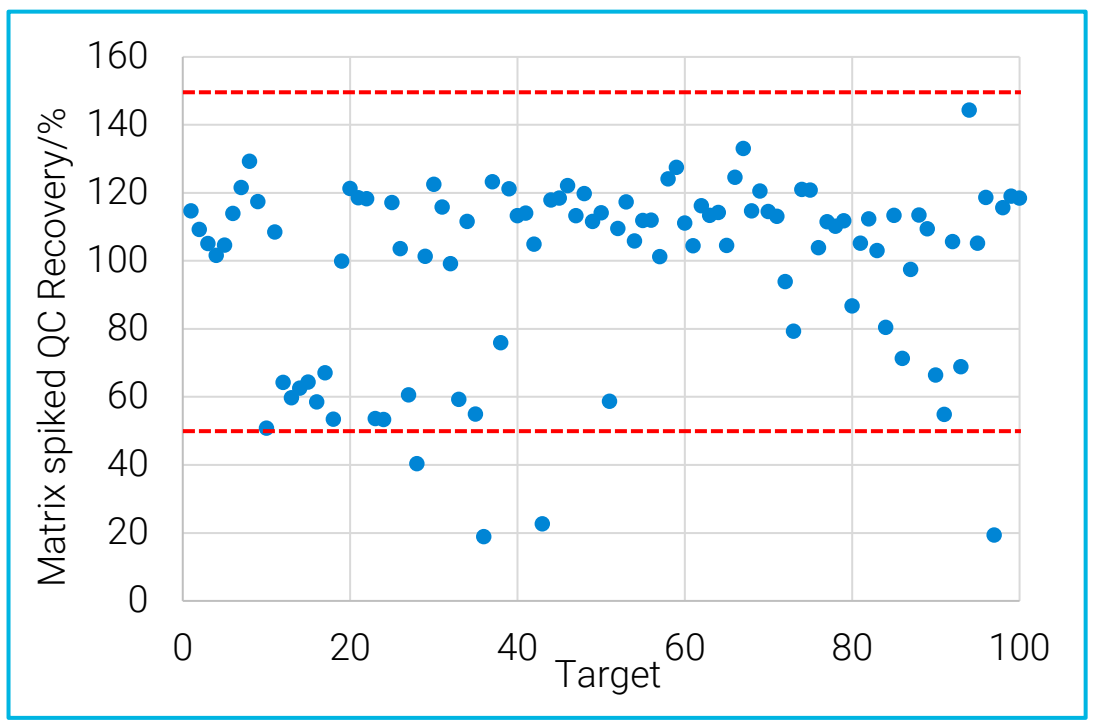

Figure 6. Matrix-spiked QC recovery at 2 µg/mL in the final extract

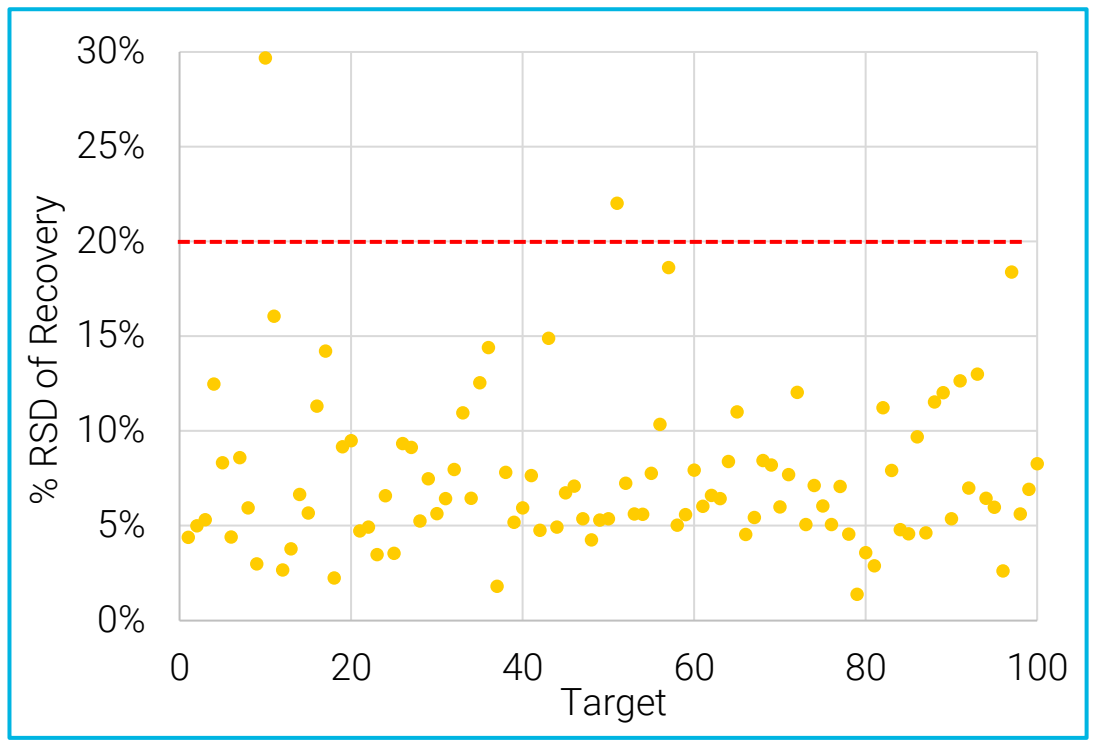

Figure 7. %RSD of recovery at 2 µg/mL in the final extract

Conclusions

- An automated Semi-Volatile Organic Compounds Analysis addressing regulatory needs which enhances lab productivity and cost saving was developed using Agilent GC/TQ and PAL3 RTC system.
- The analytical performance parameters were evaluated based on EPA 8270E, meeting acceptance criteria for more than 90 out of 100 compounds.
- Agilent 7000 series triple quadrupole mass spectrometer coupled to 8890 GC offered excellent selectivity and sensitivity to target analytes.
- This newly developed automated workflow on the integrated PAL3-GC/TQ system offers an easy-to-use and more environmentally friendly solution by reducing chemicals/standards consumption as well as waste.

(For details, refer to Agilent publication 5994-7138EN)

References

1. Method EPA 8270E: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectroscopy, Revision 6, June 2018.

2. EPA Method 3510C: Separatory Funnel Liquid-Liquid Extraction

3. A Fast Method for EPA 8270 in MRM Mode Using the 7000 Series Triple Quadrupole GC/MS, Agilent Publication, 5994-0691EN

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