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Quantitative screening of 68 Semi-volatile Organic Compounds residues in water with Automated Liquid-Liquid Extraction and large volume injection by GC-MS

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Introduction

The China Ministry of Ecological Environment (MEE) has released the updated standards of pollutants analysis in drinking water [1], regulating analytical methods for multiple types of semi-volatile organic compounds (SVOCs), including pesticides, nitrosamines, polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs) and pthalates. Sample preparation is a significant aspect of the analysis. Most of the standard methods applies conventional preparation workflow such as liquid-liquid extraction (LLE) and Solid phase extraction (SPE), needing a lot of manpower and solvent consumption. To help operators obtain enhanced efficiency in water SVOCs analysis, a fully automation solution including preparation (automated LLE) and determination (GC-MS) was developed for 68 targets quantitative screening.

Experimental

Figure 1. CTC PAL RTC with Agilent 8890GC-5977B MSD.

CTC PAL RTC with vortex module was brought into this solution for automated LLE. Agilent 8890 GC with Multi-mode Injection (MMI) and 5977B MSD were utilized as analytic platform. 15 mL water sample was transferred into 20 mL headspace vial with 1.5 g NaCl. The following preparation would be performed automatically including: 1 mL hexane addition, vortex, phase separation, supernatant transfer, internal standard addition and homogeneous mixing. After preparation, 10 µL sample was injected into MMI under room temperature for concentration. With specific script, determination of sample (N) and preparation of sample (N+1) can be perfectly overlapped. With this solution, operators can obtain simultaneous screening results of 68 SVOCs (31 analytes indicators in Chinese drinking water quality standard) within 1 hour.

Experimental

GC	8890
Mode	MMI Splitless
liner	Ultra Inert, splitless, single taper, glass wool, PN: 5190-2293
Heater	60 °C (10 µL injection) / 280 °C (1 µL injection)
MMI program	10 µLinjection rate / temp / Hold time / 60 °C / 2 min 720 °C/min / 280 °C / 20 min 720 °C/min / 60 °C / 7 min 1 µLinjection rate / temp / Hold time / 280 °C /
Flow	1.5 mL/min, constant flow
Purge flow to split vent	10 µL injection: 60 mL/min @ 2.5 min 1 µL injection: 60 mL/min @ 0.8 min
Oven program	rate / temp / Hold time / 40 °C / 4 min 10 °C/min / 300 °C / 6 min
MSD Transfer Line	280 °C
Column	HP-5MS UI, 30 m x 250 µm x 0.25 µm, PN: 19191S-433UI

MS	5977B
MS Source	230 °C
MS Quad	150 °C
Solvent Delay	5 min
Acquisition type	Select Ion Monitor (SIM)

Figure 2. GC and MS system settings for 68 SVOCs automated analysis.

Figure 3. Left: automated vortex at 2000 r/m; Right: transfer 200 uL supernatant automatically.

Results and Discussion

For each analyte, calibration curve was built with at least 6 concentration levels (0.1-200 µg/L). Linearity coefficients were established greater than 0.99 for all curves. Accuracy and reproducibility of the workflow were evaluated with spiked drinking water matrix (pure water, tap water and reservoir water) at low and high concentration levels (10-100 µg/L). More than 95% analytes had average recoveries (n=6) within 70-130%; relative standard deviation (RSD%) of all the targets was lower than 20% (n=6), showing excellent repeatability of method. The lower quantitation limits (LOQ) were determinate with 7 replicates of spiked samples (1 µg/L) and calculated as lower than 0.1 µg/L. For all the 31 indicator compounds of drinking water quality, method LOQ was lower than minimum requirement limitation (MRL) in government regulation. This workflow solution reduces ~95% of manual operation and organic solvent consumption, in the meantime, provides comparable data performance as conventional methods.

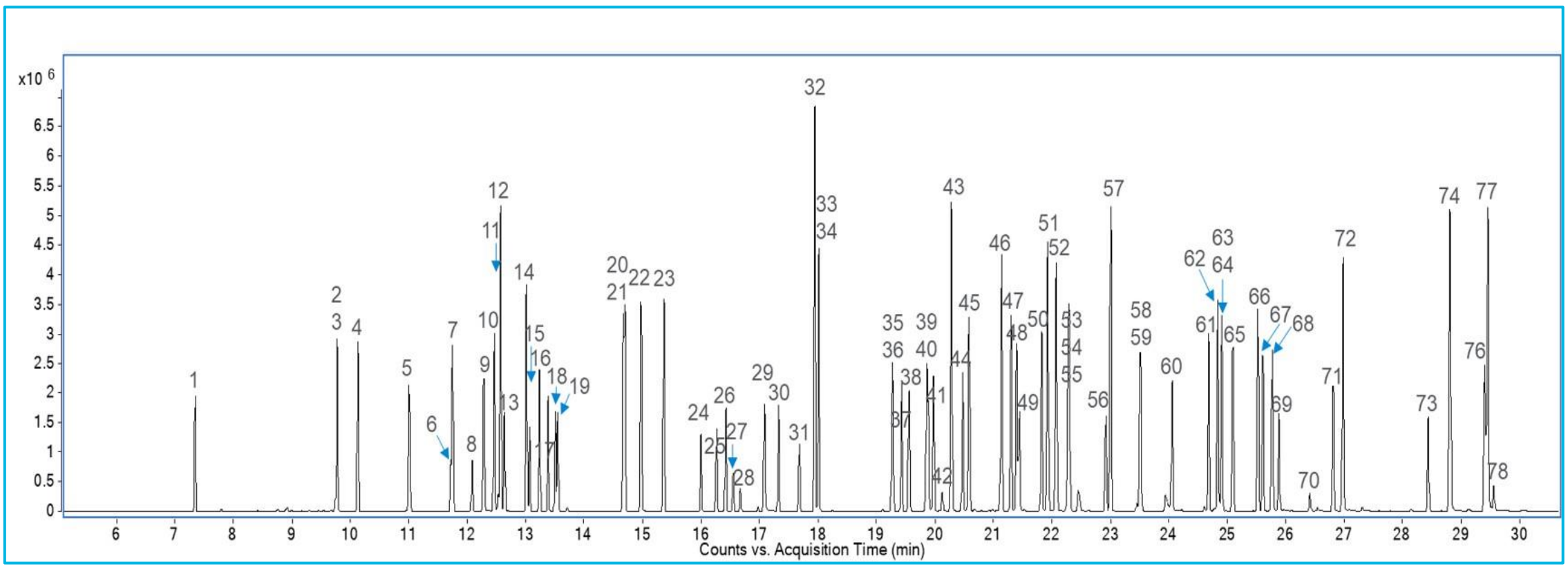

Figure 4. 68 target SVOC analytes with 4 surrogates and 6 I.S..

Conclusion

A full automated workflow for screening of 68 SVOCs (31 analytes indicators in Chinese drinking water quality standard) was done within 1 hour. It resulted in ~95% reduced manual operation and organic solvent consumption, while still maintaining efficient and good analytical performance.

References

[1]Standards for Drinking Water Quality (GB 5749-2022).

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