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Fully Automated Workflow for Volatile PFAS Analysis in Food Contact Materials Using GC-Triple Quadrupole

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Introduction

The analysis of per- and polyfluoroalkyl substances (PFAS) in food contact materials (FCMs) is crucial due to potential health and environmental concerns. Increased PFAS testing is needed to meet public demands and new regulations. Gas Chromatography coupled with Triple Quadrupole Mass Spectrometry (GC/TQ) offers a robust and sensitive method for detecting volatile PFAS. However, manual sample preparation could pose a bottleneck, impacting lab productivity. This study presents a robust and fully automated method using the PAL3 platform with GC/TQ for quantifying more than 30 volatile PFAS in FCMs, such as paper coffee cups. The automation approach minimizes human error, improving reliability and reproducibility for routine analysis and safety assessments of PFAS in FCMs.

Experimental

Automated Sample Preparation

The integrated 120 cm PAL3 Series 2 RTC autosampler, paired with the Agilent 7010D GC/TQ, facilitated a fully automated PFAS quantitation workflow using a paper cup matrix. It served as an automated liquid handling platform for preparing calibration standards, extracting samples, and injecting them into the GC/TQ system, equipped with essential tools and modules.



Figure 1: PAL3 Series 2 RTC autosampler with Agilent 7010D triple quadrupole GC/MS

Experimental

GCMS Instrumentation

The 7010D GC/TQ with HES 2.0 ion source was coupled to an Agilent 8890 GC system equipped with an MMI inlet and splitless liner (p/n 5190-2293). SWARM autotune optimized the instrument settings. The Agilent 8890 GC, operated in pulsed splitless mode with a splitless inlet liner, was used for analysis. The PAL3-GC/TQ system, managed by MassHunter Acquisition 13.0 software, provided an integrated single-software experience. MassHunter Optimizer was used to obtain MRM transitions for 34 targets, along with collision energies¹. Instrument operating conditions and parameters are given in the table below.

GC Parameters	
Injection Mode	Pulsed splitless
Carrier Gas	Helium, constant flow, 1 mL/min
Transfer Line Temp.	240 °C
Oven Program	45 °C hold for 2 min; 5 °C /min to 75 °C, hold for 1 min; 5 °C /min to 110 °C, hold for 1 min; 10 °C /min to 190 °C; 5 °C /min to 210 °C; 2 °C /min to 216 °C; 10 °C /min to 236 °C, hold 1 min
MS Parameters- EI mode (Solvent Delay: 5 min)	
Acquisition Mode	dMRM
Ion Source Temp.	280 °C
Quadrupole Temp.	150 °C
Gain	10

Workflow

The PAL system automated the preparation of nine calibrators (1 to 500 ng/mL), solvent-based extraction for paper cup sample blanks, and pre-spiked QCs. The entire automated analytical workflow is shown in Figure 2.

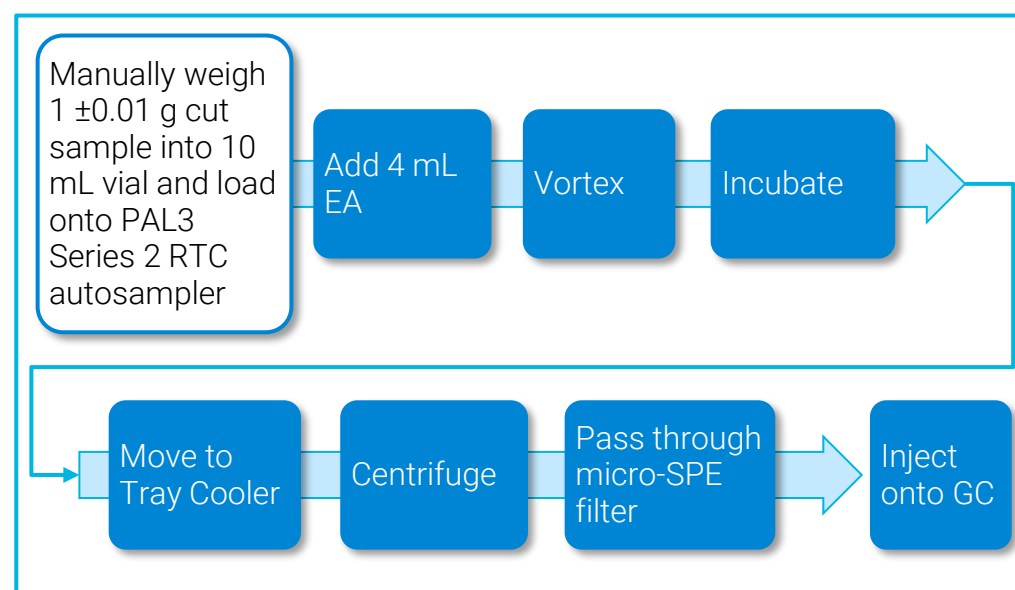


Figure 2: Automated sample preparation using the PAL3 Series 2 RTC autosampler

Results and Discussion

Chromatographic Separation

The fully automated workflow enabled sample preparation and data acquisition in a concurrent mode. The Agilent J&W DB-624 column (30 m x 0.25 mm x 1.40 μm) offered optimal separation of all 34 targets for precise quantitation.

Figure 3 shows a representative MRM chromatogram of the 34 analytes. Typical volatile PFAS, such as 4:2 FTOH, 6:2 FTOH, 8:2 FTOH, and 10:2 FTOH are labeled in Figure 3, with their zoomed-in chromatograms shown in the inset. The symmetric, sharp peaks confirm efficient chromatographic separation for accurate and reliable target quantitation.

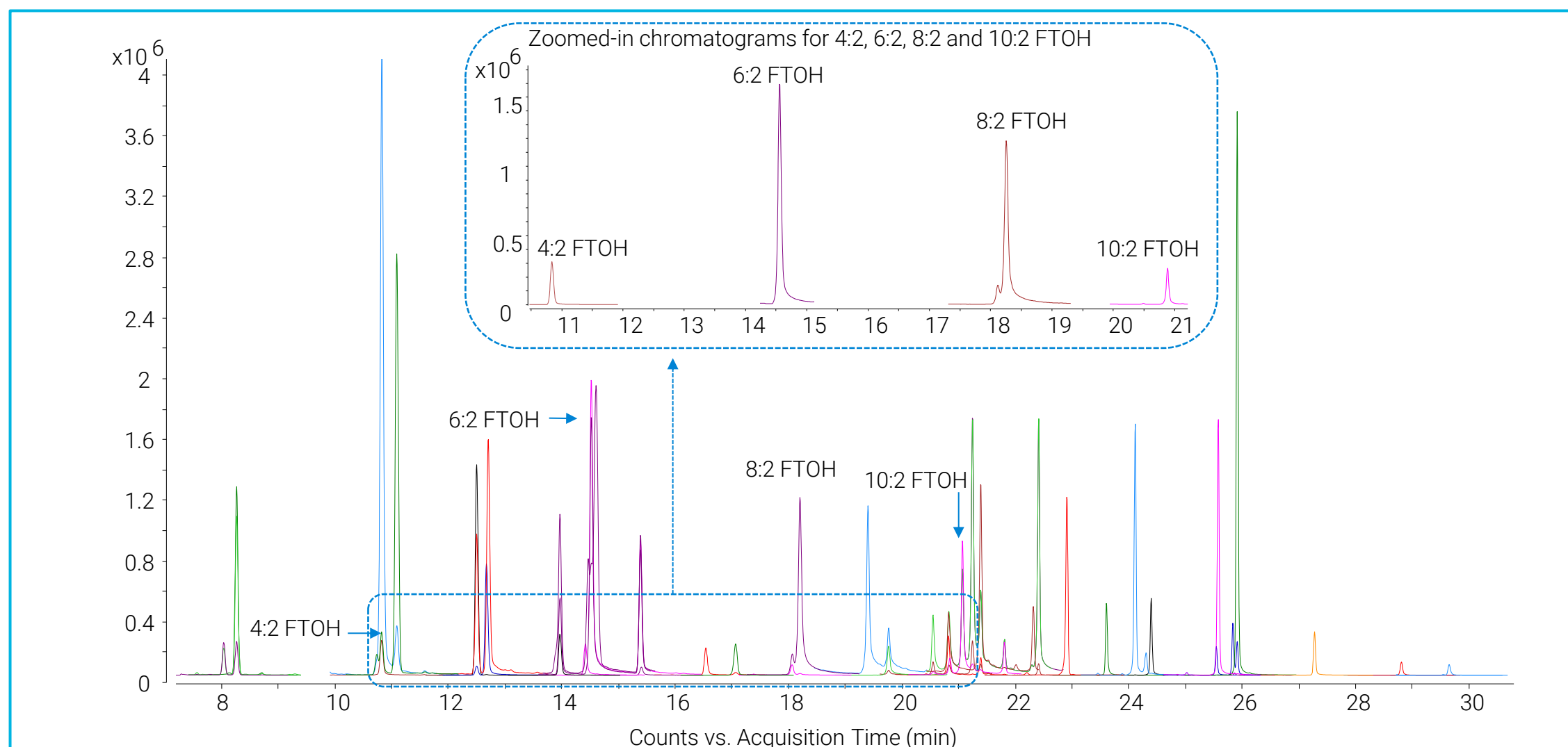


Figure 3: Representative MRM trace of all 34 analytes at 0.25 $\mu\text{g}/\text{mL}$ and 4 ISTDs at 0.50 $\mu\text{g}/\text{mL}$ in ethyl acetate

Sensitivity and Linearity

The method demonstrated excellent sensitivity, with method detection limits (MDLs) $< 2 \mu\text{g}/\text{kg}$ for most PFAS compounds. The method LOQ_{vali} for 24 out of 34 analytes, including 4:2 FTOH, 6:2 FTOH, 8:2 FTOH, 10:2 FTOH, and PFOSA, achieved an LOQ of 20 $\mu\text{g}/\text{kg}$.

Calibration curves for all targets from 1 to 500 ng/mL (9 levels: 1, 2.5, 5, 10, 25, 50, 100, 250, and 500 ng/mL) showed strong linearity, with correlation coefficients (R^2) greater than 0.99.

Figure 4 shows the linearity of 8:2 FTOH over the entire calibration range. Figure 5 displays the MRM overlay of $^{13}\text{C}_2$ -6:2 FTOH (ISTD of 8:2 FTOH) across the same range. These results confirm the reliability of automated calibration standard preparation using the PAL3 Series 2 RTC autosampler.

Precision and Accuracy

RT precision RSDs was $< 0.05\%$, indicating consistent chromatographic performance. Method precision was assessed based on the %RSD of QC recoveries ($n=8$). Overall, a %RSD of less than 20 was achieved for all target analytes across three QC levels..

Reagent Blank (RB) and Matrix Blank (MB)

RB was processed to evaluate contamination from consumables and reagents. The concentration of all targets in the RB was less than 25% of MB, indicating minimal PFAS background. Certain PFAS, including PFOSA and MeFOSE, were detected above MDL levels in the MB, confirming effective PFAS leaching from paper coffee cup samples. These findings can help establish regulatory limits for PFAS in food contact materials (FCMs).

Results and Discussion

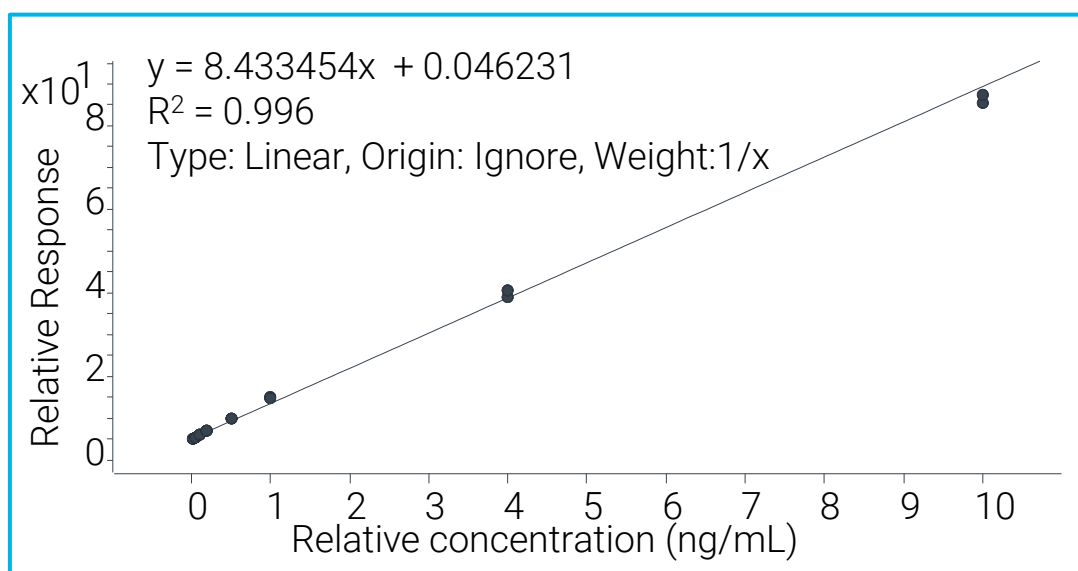


Figure 4: Linearity of 8:2 FTOH over calibration levels 1 to 9.

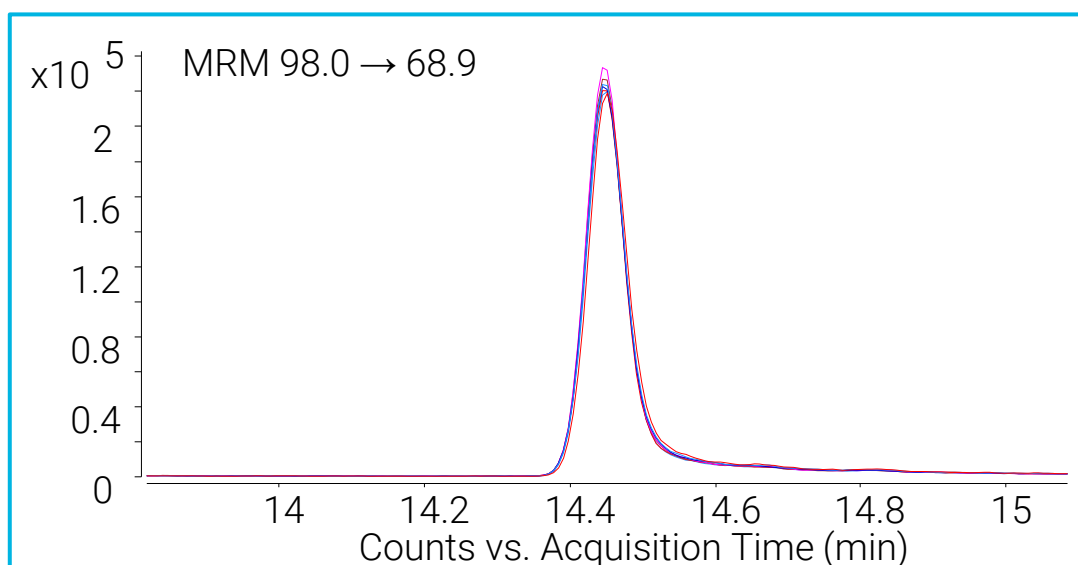


Figure 5: MRM overlay for $^{13}\text{C}_2$ -6:2 FTOH from calibration levels 1 to 9.

Recovery

Pre-spiked QC recovery evaluated the extraction efficiency and accuracy of the automated workflow. The PAL3 Series 2 RTC autosampler prepared three levels of pre-spiked QC samples (LSQ: 20 $\mu\text{g}/\text{kg}$, MSQ: 100 $\mu\text{g}/\text{kg}$ and HSQ: 200 $\mu\text{g}/\text{kg}$).

Target recovery for each QC level was calculated from the average of eight analyses (two injections per technical preparation). Overall, 71% of analytes at the LSQ, 85% at the MSQ, and 85% at the HSQ had a recovery range of 65 to 135%, meeting the general performance criteria set by the AOAC for PFAS in food.

Method precision was assessed based on the %RSD of QC recoveries ($n=8$). Overall, a %RSD of less than 20 was achieved for all target analytes across three QC levels.

Figure 6 shows the %RSD of recoveries at the MSQ for all targets, demonstrating the good precision of the automated analytical workflow for quantifying volatile PFAS in FCMs. Recovery precision and inter-batch reproducibility confirmed the excellent performance of automated workflow for routine PFAS analysis in FCM.

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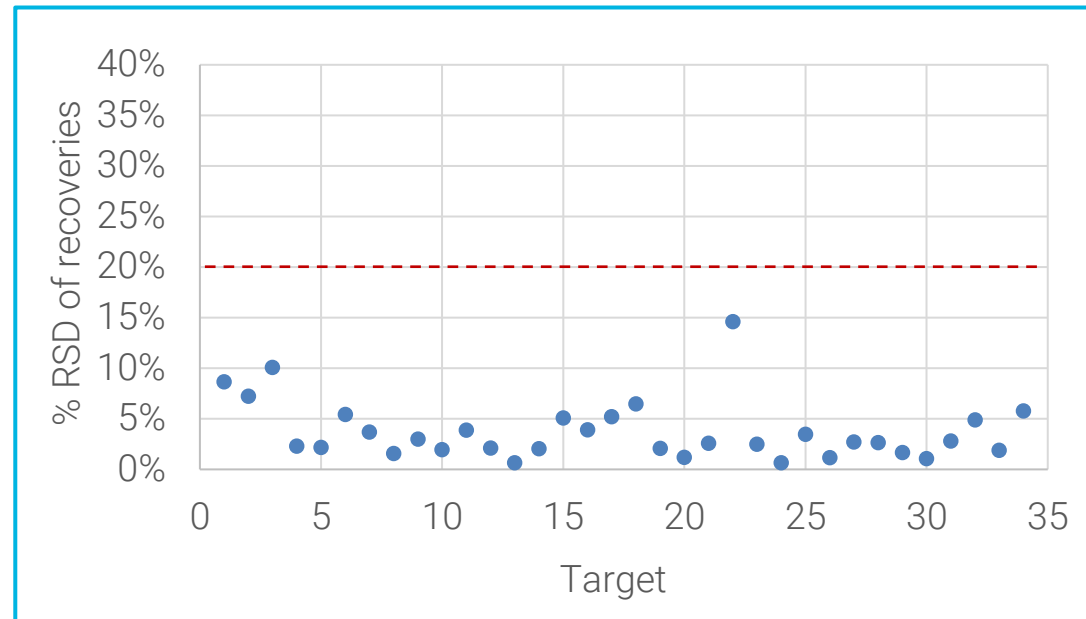


Figure 6: %RSD of recoveries at the MSQ for all targets from intra batch analyses

Conclusions

- This study demonstrated a robust, fully automated method for analyzing volatile PFAS in FCMs using the PAL3 Series 2 RTC autosampler and Agilent 7010D GC/TQ system.
- Method detection limits (MDL), linearity, retention time (RT) precision, area precision, and QC recovery was performed to assess the workflow performance.
- The MassHunter Optimizer simplified method development, while the HES 2.0 ion source provided exceptional sensitivity, achieving sub-ppb MDLs.
- For all FTOHs, an LOQ of 20 $\mu\text{g}/\text{kg}$ with recoveries of 65% to 135% and %RSD < 10% were achieved, highlighting the high extraction efficiency, accuracy and reliability of the automated workflow.
- Automation reduces human errors, decreases chemical usage, and enhances productivity, offering reliable trace-level volatile PFAS monitoring in FCMs for regulatory guidance.

References

- 1 For more details, refer to the Agilent publication titled "Fully Automated Workflow for Volatile PFAS Analysis in Food Contact Materials Using GC-Triple Quadrupole MS" (Publication Number: 5994-8295 EN).

PAL SYSTEM

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