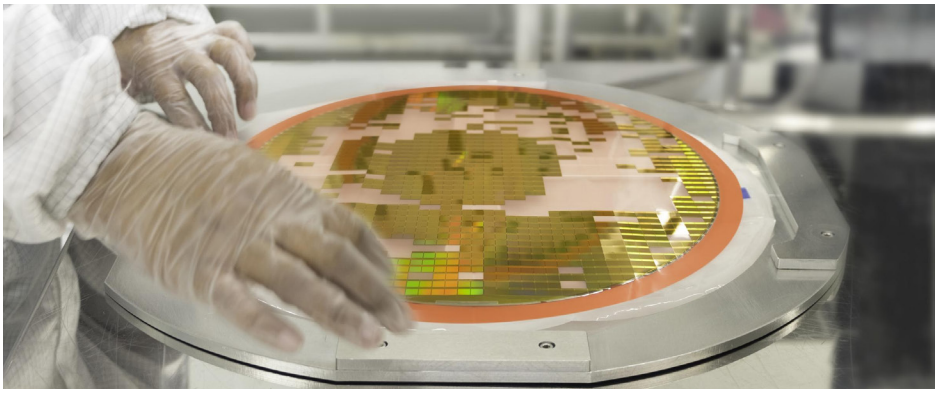
Application Note

Advanced Materials

Quantitative Analysis of Legacy and Emerging PFAS in Semiconductor Lubricant Using Agilent 6475 Triple Quadrupole LC/MS



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Abstract

Per-and polyfluoroalkyl substances (PFAS)-containing lubricants are known to be present in the semiconductor manufacturing process, where perfluoropolyether (PFPE), polytetrafluoroethylene (PTFE), and similar fluoropolymers are the main substances. Having a sensitive method for precise quantitation of trace amount of PFAS from lubricants would be beneficial for semiconductor industry. This study established a workflow based on solid phase extraction (SPE) and liquid chromatography/triple quadrupole mass spectrometry (LC/TQ). The Agilent 1290 Infinity II LC system and Agilent 6475 LC/TQ were used for quantitative analysis of legacy and emerging PFAS from a semiconductor lubricant matrix. The acquisition method, covering 100 plus native and isotopically labeled PFAS, used multiple reaction monitoring (MRM) and was built from the Agilent PFAS MRM Database. The extraction method used a weak anion exchange (WAX) method with Agilent Bond Elut PFAS WAX SPE cartridges that were applied for lubricant sample extraction. The method performance was evaluated in terms of linearity, sensitivity, recovery, and reproducibility. There were 15 PFAS detected above the limit of quantitation (LOQ), including PFOA, PFBA, PFPeA, PFHxA, PFHpA, PFDA, PFMPA, etc. that are currently regulated by EPA 1633, EPA 533, EPA 537.1, ASTM, ISO 21675, and SW-846 8327.

Introduction

In recent decades, the semiconductor industry has emerged as a pivotal player in the global technological landscape, driving innovations that shape our modern world. However, with rapid growth and increasing complexity, the industry faces new challenges related to environmental sustainability and safety. One such challenge is PFAS-based chemicals and additives used in the semiconductor manufacturing process, such as PFAS-containing lubricants.[1,2] Lubricant manufacturing suppliers assert that PFAS, such as PFPE and PTFE, are relatively low or nontoxic under normal circumstances.[3,4] These substances are the primary ingredients in their chemicals and additives. Global agencies have expressed skepticism regarding the potential presence of legacy PFAS produced as by-products or contaminants during the synthesis process. The environmental release and discharge of those PFAS from the semiconductor industry are highly concerning due to their persistence, bio-accumulative nature, and potential for widespread ecological and human health impacts. The European Commission has announced its intention in the recently published Chemicals Strategy for Sustainability to restrict the use of the most harmful chemicals, and PFAS are the first group of chemicals facing regulatory scrutiny on this basis.[5] Thus, many lubricant manufacturers have been working on quality control for PFAS in lubricant products or developing PFAS-free alternative formulations of lubricants. Also, semiconductor manufacturing is increasingly interested in testing the trace levels of PFAS in various products across the entire supply chain.

In this study, a comprehensive workflow using a 1290 Infinity II LC interfaced with a 6475 LC/TQ has been developed. This workflow is based on the Agilent PFAS MRM Database for the quantitative analysis of over 100 native and isotopically labeled PFAS in a lubricant matrix. Organic solvent extraction was performed to remove the fat from the matrix, followed by solid phase extraction with an Agilent Bond Elut PFAS WAX SPE cartridge.

Experimental

Chemicals and reagents

GC grade dichloromethane (DCM), LC/MS grade ammonium acetate, and ammonium hydroxide (28% ammonia in water, ≥ 99.99%) were purchased from Sigma-Aldrich (St. Louis, MO, USA). Optima LC/MS grade acetone nitrile (ACN) and 2-propanol were from Fisher Scientific (Waltham, MA, USA). Methanol (LC/MS grade) was obtained from Agilent. Ultrapure water was used from a Milli-Q water system.

Standards and calibration preparation

Native and isotopically labeled PFAS analytical standards were purchased as individual stock solutions, solution mixes, or powdered standards from Wellington Laboratories Inc. (Guelph, ON, Canada) and Toronto Research Chemicals (Toronto, ON, Canada).

The preparation of calibration standards is aligned with the workflow guide in the Agilent PFAS eMethod solution (part number G5285AA). Calibration standards (except for the calibration blank) were prepared in methanol:water (80:20, v:v) with the addition of a constant amount of surrogate mix and isotope performance standard (IPS) mix into each level.

Instrumentation

Chromatographic separation was achieved using an Agilent ZORBAX RRHD Eclipse Plus C18, 2.1 × 100 mm, 1.8 µm column (part number 959-758-902). The column was installed on an Agilent 1290 Infinity II LC system with the Agilent polyfluorinated compound (PFC)-free HPLC conversion kit (part number 5004-0006) to minimize background PFAS contamination. This kit includes substitutes for all critical LC system parts containing organic fluorine compounds and a PFC delay column for delaying potential PFAS impurities from the mobile phases. A 12-minute gradient as per the workflow guide was performed with 5 mM ammonium acetate in water (mobile phase A) and methanol (mobile phase B) at 0.4 mL/min. The total run time was approximately 18 minutes (injection to injection).

Dynamic MRM (dMRM) analysis was performed using a 6475 LC/TQ with an Agilent Jet Stream (AJS) ion source operated in negative ionization mode. The LC/TQ autotune was performed in unit mode. The acquisition method used in this application note was built based on the commercial Agilent PFAS MRM Database (part number G1736AA) and is also available as an electronic eMethod (part number G5285AA). Data processing was performed using Agilent MassHunter LC/MS Acquisition software version 12.0 and Quantitative Analysis software version 12.0.

Sample extraction

The lubricant sample used for this study is unique to semiconductor manufacturing. The sample contained oil as a main component, and other additives, which brought many difficulties to quantitation of PFAS due to the heavy matrix. Thus, organic solvent extraction was applied to remove fat content from the matrix prior to SPE.[6] Figure 1 illustrates the sample preparation in detail. A 1 ± 0.1 g of sample was weighted into a 15 mL polypropylene (PP) conical tube (part number 5610-2039), fortified with surrogate mix at defined concentrations. To prepare matrix spike quality control (QC) samples, an appropriate amount of native PFAS mix solution was added at two different concentration levels to make low spike QCs (LSQ) and high spike QCs (HSQ). The matrix blank was prepared without the addition of native PFAS standards. Then, 3 mL of methanol:DCM (50:50, v:v) was added into the sample tube followed by vigorous shaking using a Gino/Grinder for 10 minutes at 1,200 rpm. Then, the sample was centrifuged at 4,200 rpm for 10 minutes, and the extract was transferred into a new 15 mL PP tube. The previously described solvent extraction was then repeated three times, and the extract was combined into the same 15 mL PP tube. This mixture was concentrated to near-dryness under a gentle stream of nitrogen gas in a water bath at 50 to 55 °C, then redissolved in 5 mL of water for further SPE.

SPE was performed using Agilent Bond Elut PFAS WAX, 6 mL, 500 mg cartridges (part number 5610-2152). The cartridges were preconditioned with 5 mL of methanol containing 0.1% ammonium hydroxide, 5 mL of methanol, and 5 mL of water. Then, 5 mL of sample extract was loaded onto the cartridge under vacuum (≤ 2 in Hg) at approximately 2 to 3 mL/min. The cartridge was washed with 5 mL of water and dried under high vacuum for 2 minutes. The analytes were eluted from the SPE cartridge using 2.5 mL of methanol followed by 2.5 mL of 0.1% ammonium hydroxide in methanol. The eluates were concentrated to near-dryness under a gentle nitrogen stream followed by reconstituted to 0.5 mL of methanol/water (80:20, v:v) with the addition of isotope performance standard (IPS) mix. These samples were vortexed well and transferred into an autosampler vial (part number 5190-2242) for LC/TQ analysis. The result was a sample preconcentration factor of 2-fold.

1. Solvent Extraction	– Weigh 1 ± 0.1 g of lubricant sample into tube – Add 3 mL of methanol/DCM – Gino/Grinder for 10 minutes – Centrifuge for 10 minutes – Transfer the supernatant to a new sample tube – Repeat the above solvent extraction three times – Combine the extract into the same sample tube
2. Dryness and Reconstitution	– Dry the sample tube under a gentle nitrogen stream – Redissolve into 5 mL of water and mix well
3. Setup SPE	– Connect SPE manifold to the vacuum trap – Place sample tube in the collection rack – Assemble stopcock, WAX cartridge, adaptor, and sample reservoir
4. Condition SPE	– 5 mL of 0.1% methanolic ammonium hydroxide – 5 mL of methanol – 5 mL of water
5. Load Sample	– Pour the samples into the reservoir – Adjust vacuum and stopcock to flow rate at ~2 mL/min
6. Rinse Sample Tube and Reservoir	– 5 mL of water – Dry the cartridge under vacuum for 2 minutes
7. Elute Sample	– Rinse the sample tube and reservoir with 2.5 mL of methanol and transfer to SPE cartridge – Rinse the sample tube and reservoir with 2.5 mL of 0.1% methanolic ammonium hydroxide and transfer it to SPE cartridge – Flute the cartridge by gravity and dry it
8. Concentrate the Eluate	– Concentrate the eluate to near-dryness under a gentle nitrogen stream
9. Reconstitute and Add IPS	– Reconstitute with methanol/water and spike IPS – Vortex well
10. Analyze Sample	– Transfer an aliquot into poly ALS vial for LC/TQ analysis – Store any remaining solution at –20 °C

Figure 1. Lubricant sample extraction procedure.

Result and discussion

Calibration performance

The calibration curve was generated based on linear regression with 1/x weighing for all 71 analytes except for FTSAs, where quadratic regression was used. Excellent R^2 values of greater than 0.995 were achieved for all target analytes with a wide analytical range of at least three orders of magnitude. The accuracy and precision of each calibration standard met the typical acceptable limits of 70 to 130% and ≤ 20% (n = 3), respectively.

Method sensitivity

Method sensitivity was assessed based on the LOQ of target analytes in the sample matrix. The LOQ is the lowest concentration of mass of the analyte in the test material that has been validated with acceptable recovery and repeatability by applying the entire workflow method and identification criteria as described in the general guidance document.[7] The LOQ for each analyte is summarized in Table 1. The LOQ distribution for 71 PFAS analytes is mapped in Figure 2. Overall, 32 and 50 compounds obtained LOQ ≤ 0.1 and 1.0 µg/kg, respectively, indicating the excellent sensitivity of the method developed for PFAS analysis in lubricant samples using the 6475 LC/TQ. Furthermore, the LOQs obtained for PFOA, PFOS, PFNA, and PFHxS from this study are 0.01, 0.025, 0.01, and 0.05 µg/kg, which are about 10 times lower than the typical regulatory requirements from similar market spaces.[8]

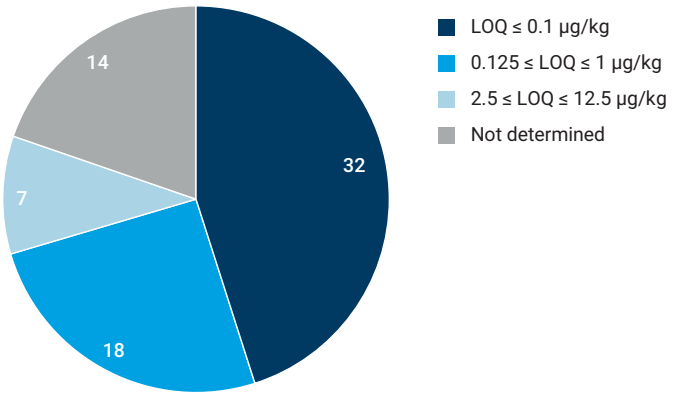

Figure 2. Distribution of LOQ for the 71 PFAS analytes in a lubricant sample.

Method recovery and precision

Matrix spiked QC recovery was used to evaluate the method accuracy in this study. Lubricant samples were fortified with PFAS spike mix, including a surrogate mix and an analyte mix, and were extracted following the entire sample preparation procedure. Three technical preparations were performed for both low spiked QC (LSQ, concentration range from 0.05 to 12.5 µg/kg) and high spiked QC (HSQ, concentration range from 0.2 to 50 µg/kg). The measured concentration of each analyte in the spiked QC sample was corrected by subtracting its native level present in the unspiked lubricant sample. The method recovery was calculated based on the mean percent recovery, while the method precision was assessed from the %RSD of recoveries.

Recovery values and %RSD for all analytes are summarized in Table 1. For LSQ samples, 44 out of 71 analytes met recovery 70 to 130% with %RSD ≤ 20. For HSQ samples, 57 compounds met this condition, which demonstrated the excellent efficiency and reproducibility of the Agilent WAX cartridge used for PFAS extraction in this study. HSQ recoveries for nine more compounds were within 50 to 61%, which could be acceptable by users according to the specific study purpose. PFOA and PFOS are high priority PFAS measurements across various regulatory guidelines. Figures 3A and 3B show the chromatogram overlay of triplicate preparations of LSQ for PFOA and PFOS, respectively. The results demonstrate the consistency within each technical preparation and thus confirm the accuracy and reliability of the workflow that was developed for PFAS analysis in the lubricant sample, a complicated, and fatty matrix.

Lubricant sample analysis result

The native level of PFAS present in the lubricant sample was also studied. To confirm the reliability of the measured concentration for target analytes, unspiked lubricant samples (matrix blanks) were extracted in triplicate using the same preparation procedure and analyzed by LC/TQ. Figure 4 shows the chromatogram of compounds determined above the LOQ level in an unspiked lubricant sample. Approximately 15 native PFAS were observed to be present in sub-ppb levels from a lubricant sample, such as PFBA, PFBS, PFDA, HFPO-TA, PFOA, PFNA, PFHxA, PFPeA, etc., which are concerned substances in global regulations including EPA 1633, EPA 533, EPA 537.1, ASTM, ISO 21675, SW-846 8327, and EU 2022/2388. For those compounds detected above the LOQ, the spiked QC recoveries were well within 70 to 130%, confirming the reliability and accuracy of the developed method protocol for PFAS analysis in the semiconductor lubricant matrix.

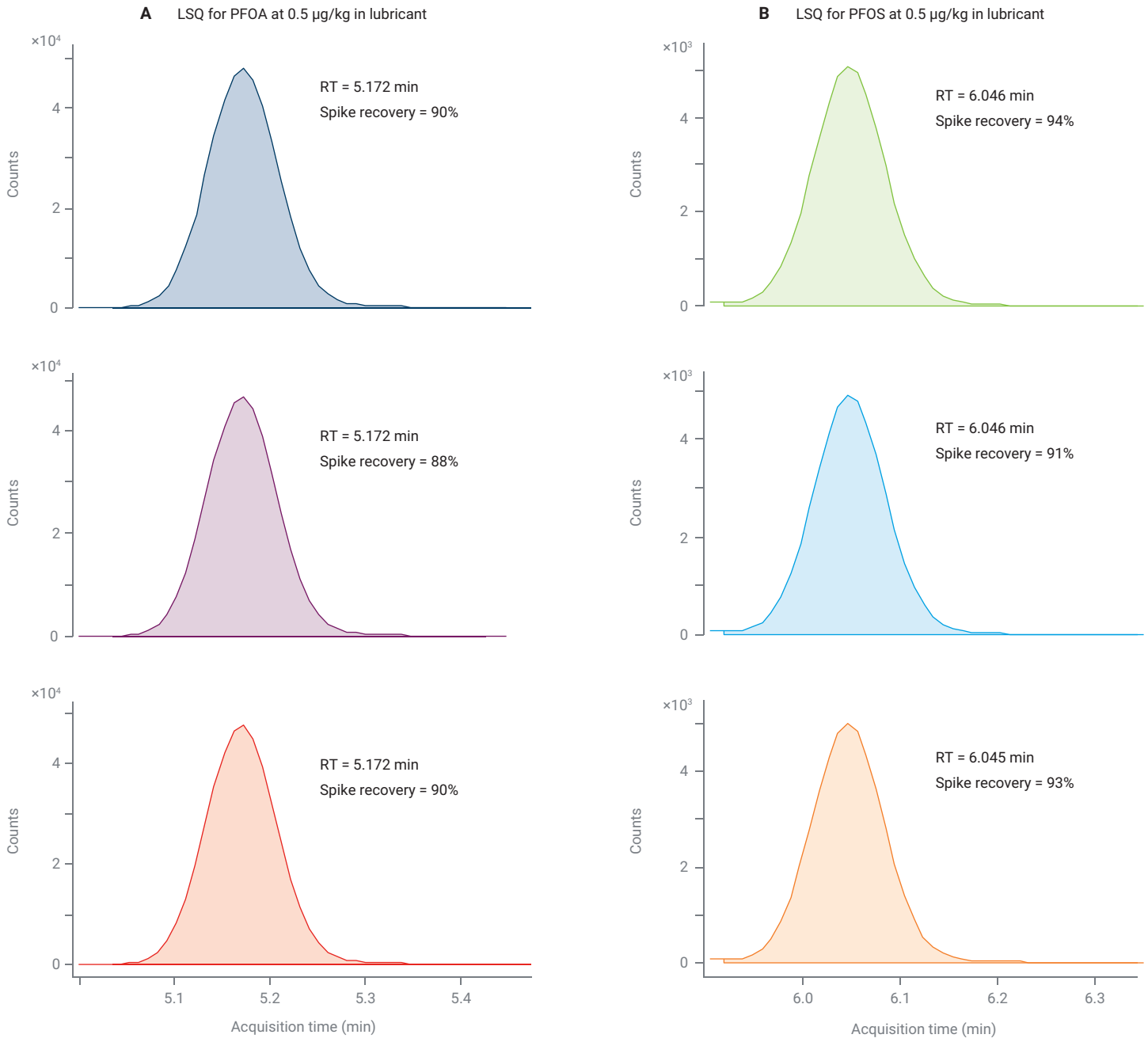

Figure 3. The chromatogram overlay of triplicate preparations of LSQ for PFOA (A) and PFOS (B) at 0.5 µg/kg.

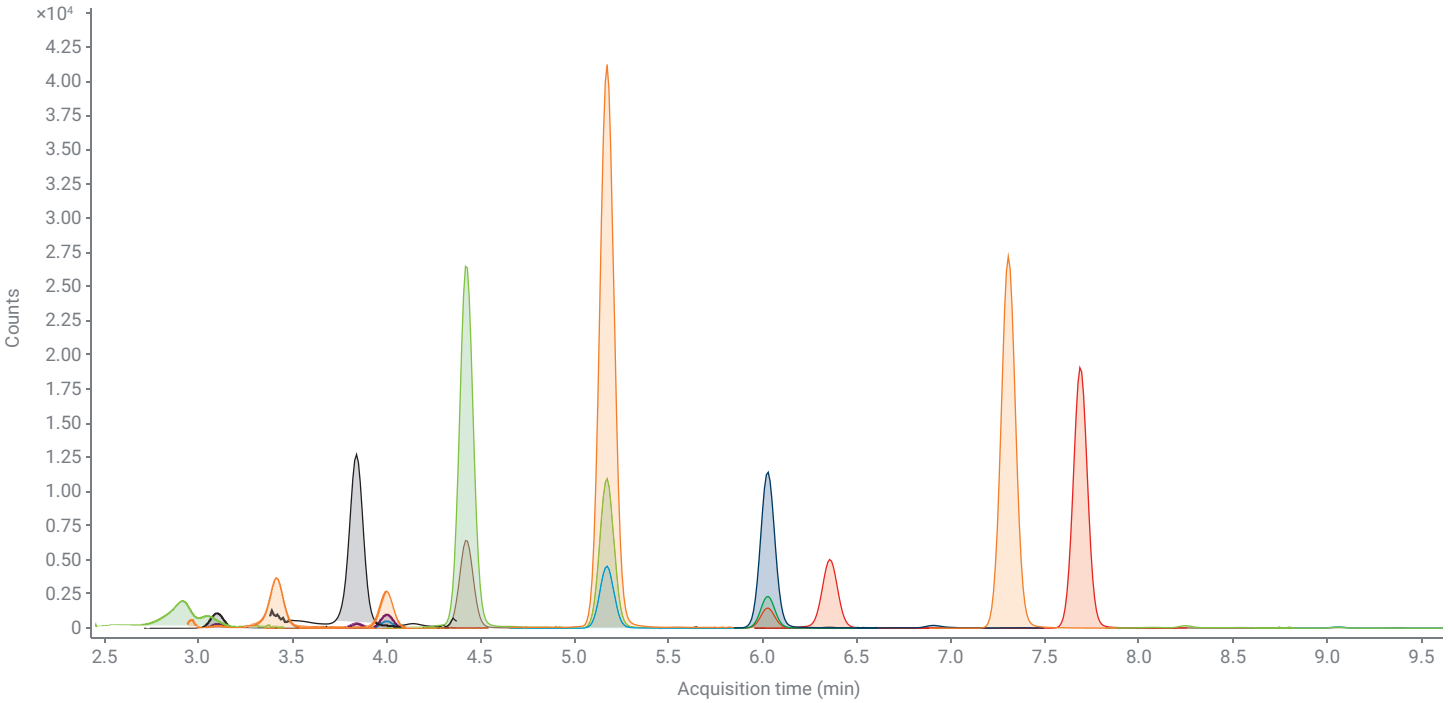

Figure 4. The MRM chromatogram of the unspiked lubricant sample extract (matrix blank).

Conclusion

This study presented an example of workflow performance for PFAS analysis in semiconductor lubricant using an Agilent 1290 Infinity II LC coupled to an Agilent 6475 LC/TQ system. Based on the Agilent PFAS MRM Database and Agilent eMethod, a comprehensive target list with 108 PFAS (including native and labeled) was tested.

The Agilent Bond Elut PFAS WAX cartridge offered selective and effective extraction for sample cleanup and preconcentration of PFAS analytes in the lubricant sample, demonstrating the capability of a WAX cartridge used for oil-based matrix extraction. The analytical performance of the workflow was evaluated in terms of calibration linearity, LOQ, spiked QC recovery, and method precision. LOQ ≤ 0.1 µg/kg was achieved for 45% of analytes. Spiked QC recovery met 70 to 130% with an %RSD ≤ 20 for the majority of compounds, confirming that the workflow developed on 6475 LC/TQ is applicable for the quantitative analysis of PFAS in a high oil content matrix. This workflow enables a ready-to-use protocol for lubricant suppliers and semiconductor manufactures to perform routine monitoring of trace levels of critical PFAS in lubricants.

Table 1. Summary of analytical performance.

No.	Compound	PFAS Group	CAS Number	Surrogate	LOQ (µg/kg)	LSQ (%)		HSQ (%)	
						Recovery	Precision (n = 3)	Recovery	Precision (n = 3)
1	PFUnDA	PFCA	2058-94-8	$^{13}C_7$-PFUnDA	0.025	114	5	95	6
2	PFTrDA	PFCA	72629-94-8	$^{13}C_2$-PFDoDA	0.025	117	4	102	8
3	PFTDA	PFCA	376-06-7	$^{13}C_2$-PFTDA	0.025	105	3	98	6
4	PFPeS	PFSA	2706-91-4	$^{13}C_3$-PFHxS	0.05	106	5	96	7
5	PFPeA	PFCA	2706-90-3	$^{13}C_5$-PFPeA	0.125	106	6	105	4
6	PFOSA	FASA	754-91-6	$^{13}C_8$-PFOSA	0.25	62	7	113	10
7	PFOS	PFSA	1763-23-1	$^{13}C_8$-PFOS	0.025	93	5	94	4
8	PFOPA	PFPA	40143-78-0	Cl-PFOPA	N.D.	58	19	52	20
9	PFODA	PFCA	16517-11-6	$^{13}C_2$-PFHxDA	0.025	82	6	87	11
10	PFOA	PFCA	335-67-1	$^{13}C_8$-PFOA	0.01	89	4	93	3
11	PFNS	PFSA	68259-12-1	$^{13}C_8$-PFOS	0.1	107	6	99	5
12	PFNA	PFCA	375-95-1	$^{13}C_9$-PFNA	0.01	81	15	108	5
13	PFMPA	PFECA	377-73-1	$^{13}C_4$-PFBA	0.125	87	13	128	2
14	PFMBA	PFECA	863090-89-5	$^{13}C_5$-PFPeA	0.05	104	5	90	8
15	PFHxS	PFSA	355-46-4	$^{13}C_3$-PFHxS	0.05	91	3	74	6
16	PFHxPA	PFPA	40143-76-8	Cl-PFOPA	N.D.	48	22	51	16
17	PFHxDA	PFCA	67905-19-5	$^{13}C_2$-PFHxDA	0.01	102	1	108	6
18	PFHxA	PFCA	307-24-4	$^{13}C_5$-PFHxA	0.01	106	9	114	6
19	PFHpS	PFSA	375-92-8	$^{13}C_8$-PFOS	0.05	112	7	100	3
20	PFHpA	PFCA	375-85-9	$^{13}C_4$-PFHpA	2.5	15	17	105	3
21	PFEESA	PFESA	113507-82-7	$^{13}C_3$-PFBS	0.05	98	4	105	7
22	PFDS	PFSA	335-77-3	$^{13}C_8$-PFOS	0.25	100	8	105	4
23	PFDPA	PFPA	52299-26-0	Cl-PFOPA	N.D.	52	10	50	11
24	PFDoS	PFSA	79780-39-5	$^{13}C_8$-PFOS	0.25	92	8	92	3
25	PFDoDA	PFCA	307-55-1	$^{13}C_2$-PFDoDA	0.01	112	4	98	6
26	PFDA	PFCA	335-76-2	$^{13}C_6$-PFDA	0.01	110	5	96	6
27	PFBS	PFSA	375-73-5	$^{13}C_3$-PFBS	1	107	3	89	6
28	PFBPA	PFPA	52299-24-8	Cl-PFOPA	2.5	41	16	93	19
29	PFBA	PFCA	375-22-4	$^{13}C_4$-PFBA	1	111	7	104	1
30	P5MeODIOXOAc	PFECA	1190931-41-9	$^{13}C_3$-HFPO-DA	0.5	108	12	85	5
31	N-MeFOSAA	FASAA	2355-31-9	$^{2}H_3$-N-MeFOSAA	0.05	94	5	77	2
32	N-MeFOSA	FASA	31506-32-8	$^{2}H_3$-N-MeFOSA	N.D.	51	14	50	9
33	NFDHA	PFECA	151772-58-6	$^{13}C_5$-PFHxA	0.125	70	11	74	11
34	N-EtFOSAA	FASAA	2991-50-6	$^{2}H_5$-N-EtFOSAA	0.05	92	5	78	7
35	N-EtFOSA	FASA	4151-50-2	$^{2}H_5$-N-EtFOSA	N.D.	45	23	42	16
36	MeFOSE	FASE	24448-09-7	$^{2}H_7$-MeFOSE	5	53	42	90	15
37	MeFHxSA	FASA	68259-15-4	$^{13}C_8$-PFOSA	N.D.	52	16	55	16
38	MeFBSA	FASA	68298-12-4	$^{13}C_8$-PFOSA	N.D.	41	19	50	11
39	HFPO-TA	PFECA	13252-14-7	$^{13}C_9$-PFNA	0.1	86	17	83	8
40	HFPO-DA	PFECA	13252-13-6	$^{13}C_3$-HFPO-DA	1	91	7	110	2
41	FOSAA	FASAA	2806-24-8	$^{2}H_3$-N-MeFOSAA	1	78	22	105	2
42	FHxSA	FASA	41997-13-1	$^{13}C_8$-PFOS	N.D.	38	25	45	13
43	FDSA	FASA	NA	$^{13}C_8$-PFOSA	0.125	97	7	109	6

N.D.: Not determined. LOQ for 14 compounds were not determined due to the challenges associated with sample preparation. More studies could be done to further improve the sample preparation efficiency for such a complex matrix.

No.	Compound	PFAS Group	CAS Number	Surrogate	LOQ (μg/Kg)	LSQ (%)		HSQ (%)	
						Recovery	Precision (n = 3)	Recovery	Precision (n = 3)
44	FBSA	FASA	30334-69-1	$^{13}C_3$-PFHxS	0.5	38	27	94	4
45	EtFOSE	FASE	1691-99-2	$^{2}H_9$-EtFOSE	N.D.	25	30	35	18
46	DONA	PFECA	919005-14-4	$^{13}C_4$-PFHpA	0.025	83	8	81	1
47	diSAmPAP	SAmPAP	2965-52-8	$(^{13}C_2)_2$-8:2 diPAP	0.025	71	5	126	3
48	Cl-PFHxPA	PFPA	NA	Cl-PFOPA	N.D.	27	9	34	12
49	9Cl-PF3ONS	PFESA	756426-58-1	$^{13}C_8$-PFOS	0.25	98	6	96	1
50	8:8 PFPi	PFPiA	40143-79-1	$(^{13}C_2)_2$-6:2 diPAP	0.25	100	6	92	9
51	8:3 FTCA	FTCA	34598-33-9	$^{13}C_6$-PFDA	N.D.	40	21	61	15
52	8:2 FTUCA	FTUCA	70887-84-2	$^{13}C_2$-8:2 FTUCA	0.025	51	17	103	6
53	8:2 FTSA	FTSA	39108-34-4	$^{13}C_2$-8:2 FTSA	0.1	101	5	97	8
54	8:2 FTCA	FTCA	27854-31-5	$^{13}C_2$-8:2 FTCA	2.5	109	21	106	11
55	8:2 diPAP	diPAP	678-41-1	$(^{13}C_2)_2$-8:2 diPAP	0.1	100	8	102	7
56	7:3 FTCA	FTCA	812-70-4	$^{13}C_2$-8:2 FTUCA	N.D.	33	16	50	17
57	6:8 PFPi	PFPiA	610800-34-5	$(^{13}C_2)_2$-6:2 diPAP	0.25	97	6	103	9
58	6:6 PFPi	PFPiA	40143-77-9	$^{13}C_2$-PFDoDA	0.05	102	14	115	13
59	6:2/8:2 diPAP	diPAP	943913-15-3	$(^{13}C_2)_2$-6:2 diPAP	0.1	59	10	77	13
60	6:2 FTUCA	FTUCA	70887-88-6	$^{13}C_2$-6:2 FTUCA	0.05	98	25	91	7
61	6:2 FTSA	FTSA	27619-97-2	$^{13}C_2$-6:2 FTSA	0.05	97	5	97	5
62	6:2 FTCA	FTCA	53826-12-3	$^{13}C_2$-6:2 FTCA	5	48	17	98	14
63	6:2 diPAP	diPAP	57677-95-9	$(^{13}C_2)_2$-6:2 diPAP	0.25	105	4	95	1
64	5:3 FTCA	FTCA	914637-49-3	$^{13}C_2$-6:2 FTUCA	N.D.	29	26	51	18
65	4-PFecHS	PFSA	646-83-3	$^{13}C_8$-PFOS	0.025	118	5	88	2
66	4:2 FTSA	FTSA	757124-72-4	$^{13}C_2$-4:2 FTSA	0.1	104	3	94	1
67	3:3 FTCA	FTCA	356-02-5	$^{13}C_5$-PFPeA	N.D.	22	17	47	22
68	11Cl-PF3OUdS	PFESA	763051-92-9	$^{13}C_8$-PFOS	0.05	95	6	94	5
69	10:2 FTUCA	FTUCA	70887-94-4	$^{13}C_2$-10:2 FTUCA	2.5	27	35	90	10
70	10:2 FTSA	FTSA	120226-60-0	$^{13}C_2$-8:2 FTSA	0.25	122	6	121	11
71	10:2 FTCA	FTCA	53826-13-4	$^{13}C_2$-10:2 FTCA	12.5	57	35	105	19

N.D.: Not determined. LOQ for 14 compounds were not determined due to the challenges associated with sample preparation. More studies could be done to further improve the sample preparation efficiency for such a complex matrix.

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