Application brief | 003596

Environmental

Streamlining environmental monitoring: Advanced analysis of PBDEs using triple quadrupole GC-MS/MS

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Keywords

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Goal

To enhance the efficiency of environmental laboratories by implementing a unified configuration that addresses all regulatory requirements for PBDEs in environmental analysis worldwide.

Thermo Fisher Scientific presented a new analytical approach using a single column to streamline laboratory operations with a unified configuration in Application Brief 002866.

This application brief outlines how the proposed GC-MS/MS configuration can effectively analyze PBDEs, ensuring compliance with regulatory standards such as the European Standard 16694:2025.

Introduction

Polybrominated diphenyl ethers (PBDEs) are a class of brominated hydrocarbons commonly used as additive flame retardants in various materials. These compounds can leach into the environment, where they persist and bioaccumulate, posing potential risks to human health and ecosystems. Structurally, PBDEs consist of two phenyl rings linked by an oxygen atom. The environmental analysis of PBDEs, as outlined in the European Standard 16694:2025, necessitates the determination of specific congeners such as BDE-28, -47, -99, -100, -153, and -154.

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Advanced laboratory techniques, such as the use of triple quadrupole GC-MS/MS systems, have been developed to efficiently analyze these compounds. These systems offer high selectivity and sensitivity, enabling the accurate detection and quantification of PBDEs alongside other environmental contaminants like pesticides and micropollutants. Implementing a single GC-MS/MS configuration can streamline laboratory processes, enhancing productivity and reducing operational costs by consolidating multiple analytical methods into one robust system.

This application brief describes an analytical method for the trace analysis of PBDEs with the Thermo Scientific™ TraceGOLD™ TG-Contaminants GC column and the Thermo Scientific™ TSQ™ 9610 Triple Quadrupole GC-MS/MS System equipped with Advance electron ionization (AEI) source. This method complies with all the expectations of laboratories—precision, sensitivity, reliability, and robustness.

Experimental

To develop the analytical method, a Thermo Scientific™ TRACE™ 1610 GC paired with a Thermo Scientific™ TSQ™ 9610 GC-MS/MS was employed. The system was outfitted with a Thermo Scientific™ iConnect™ Programmable Temperature Vaporizing (PTV) injector. Analytical separation was performed on a TraceGOLD TG-Contaminants GC column. These highly inert capillary columns provide thermal stability and unique selectivity, ensuring excellent chromatographic peak shapes and sensitivity, which facilitates the chromatography of both semi-volatile compounds and late-eluting compounds.

System components

- Thermo Scientific™ TriPlus™ RSH SMART autosampler (P/N 1R77010-2003)
- TRACE 1610 GC (P/N MI-148000-0007)
- iConnect PTV injector module (P/N 19070020)
- TSQ 9610 mass spectrometer with Thermo Scientific™ NeverVent™ technology and Advanced Electron Ionization (AEI™) source (P/N TSQ9610-NV-AEI)
- TraceGOLD TG-Contaminants GC column 15 m × 0.25 mm × 0.1 µm (P/N 26056-0350)
- Thermo Scientific™ LinerGOLD™ PTV 5 Baffles Liner (P/N 453T2171-UI)
- iConnect High Temperature Transfer Line Nut (P/N 1R120434-0040)
- Thermo Scientific™ SureSTART™ 2 mL Glass Screw Top Vials (P/N CHSV9-20P) and SureSTART™ 9 mm Screw Caps (P/N 6ASC9ST1)

Figure 1. Configuration utilized for analysis: the TRACE 1610 GC with the TSQ 9610 NV-AEI GC-MS/MS

Focus on our new iConnect High Temperature Transfer Line Nut

Use the Thermo Scientific high-temperature nut for leak-free results in high-temperature transfer line settings on your GC-MS/MS

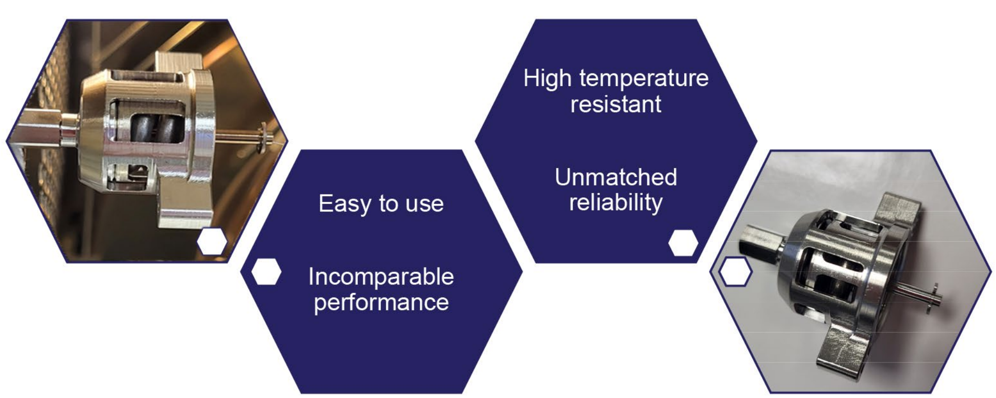


Figure 2. Details of the new iConnect High Temperature Transfer Line Nut

Table 1. Instrument parameters

Parameter	
Injection mode	Large volume
Injection volume (µL)	5
GC run time (min)	17
Number of compounds monitored	25
Calibration range in solvents (µg/L)	0.02–50
Acquisition mode	SRM
Carrier gas	Helium
Solvent	IsoOctane + analytical protectant

For further information, contact your local commercial representative.

PBDEs are a class of brominated flame retardants that have been widely used in various consumer products. Due to their harmful effects on the environment and human health, several PBDEs are now regulated and monitored. Table 2 contains a list of the main PBDE congeners that are often analyzed in the environment or food due to their regulatory importance.

Table 2. List of PBDEs monitored according to different regulations

Matrix	PBDEs
Clean water (drinking and environmental)	PBDE 28/47/99/100/153/154
Soil	PBDE 209
Wastewater	PBDE 28/47/99/100/153/154/183/209
Food	PBDE 28/47/49/99/100/138/153/154/183/209

To avoid limiting this method to the currently regulated PBDEs, Thermo Fisher Scientific has developed a comprehensive approach that includes other PBDE congeners as well as novel brominated flame retardants (NBFRs). See the complete list included in the analytical method in Table 3.

Results and discussion

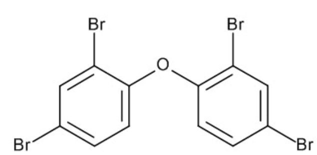
Linearity

In this study, the aim was to evaluate the linearity of the measurement system by analyzing five distinct calibration curves over a period of five different days as a typical method validation process. Linearity is a crucial parameter that ensures the accuracy and reliability of the analytical methods. By conducting these assessments over multiple days, any potential day-to-day variability is accounted for, thus providing a comprehensive understanding of the system's performance. The following sections present the detailed results of the linearity tests, highlighting the consistency and precision of the calibration standard. All PBDEs are corrected by an internal standard. Results are shown in Table 4.

The linearity of each compound was validated using five different calibration curves. Each calibration curve met the two validation criteria, R^2>0.990 and a relative amount deviation of less than 40% for the LOQ and less than 20% for the other levels.

Figure 3 shows three examples of compounds with the superposition of the five calibration curves.

Table 3. List of PBDEs included in the method according to different regulations



Compound	CAS Number
BDE 17	147217-75-2
BDE 28	41318-75-6
BDE 37	147217-81-0
BDE 49	243982-82-3
BDE 71	189084-62-6
BDE 47	5436-43-1
BDE 66	189084-61-5
BDE 77	93703-48-1
BDE 100	189084-64-8
BDE 99	60348-60-9
BDE 85	182346-21-0
BDE 154	207122-15-4
BDE 153	68631-49-2

Compound	CAS Number
BDE 138	182677-30-1
BDE 183	207122-16-5
BTBPE	37853-59-1
BDE 181	189084-67-1
BDE 190	189084-68-2
syn-Dechloran Plus	135821-03-3
anti-Dechloran Plus	135821-74-8
BDE 203	337513-72-1
BDE 205	446255-56-7
OBIND (OBTMPI)	155613-93-7
BDE 209	1163-19-5
DBDPE	84852-53-9

Table 4. Results of the linearity study

Compound	ISTD	Calibration type	LOQ (µg/L)	Range (µg/L)	R^2 for 5 calibration standards	Relative amount deviation (%) for 5 calibration standards
BDE17	BDE 28*C13	Lin, 1/A	0.05	0.05–50		
BDE28	BDE 28*C13	Lin, 1/A	0.02	0.02–50		
BDE37	BDE 28*C13	Lin, 1/A	0.02	0.02–50		
BDE49	BDE 47*C13	Lin, 1/A	0.02	0.02–50		
BDE71	BDE 47*C13	Lin, 1/A	0.02	0.0–50		
BDE47	BDE 47*C13	Lin, 1/A	0.02	0.02–50		
BDE66	BDE 47*C13	Lin, 1/A	0.02	0.02–50		
BDE77	BDE 47*C13	Lin, 1/A	0.02	0.0–50		
BDE100	BDE 100*C13	Lin, WithOffset, 1/A	0.02	0.02–50		
BDE99	BDE 99*C13	Lin, WithOffset, 1/A	0.02	0.05–50		
BDE85	BDE 99*C13	Lin, 1/A	0.05	0.05–50		
BDE154	BDE 154*C13	Lin, 1/A	0.02	0.02–50		
BDE153	BDE 153*C13	Lin, WithOffset, 1/A	0.02	0.02–50	>0.990	<40% at the LOQ <20% for all other levels
BDE138	BDE 153*C13	Quad, 1/A	0.05	0.05–50		
BDE183	BDE 153*C13	Lin, 1/A	0.02	0.02–50		
BTBPE	BDE 183*C13	Quad, WithOffset, 1/A	0.05	0.05–50		
BDE181	BDE 183*C13	Quad, 1/A	0.05	0.05–50		
BDE190	BDE 183*C13	Quad, WithOffset, 1/A	0.05	0.05–50		
syn-Dechloran-Plus	BDE 183*C13	Quad, WithOffset, 1/A	0.02	0.02–50		
anti-Dechloran-Plus	BDE 183*C13	Quad, WithOffset, 1/A	0.02	0.02–50		
BDE203	BDE 183*C13	Quad, WithOffset, 1/A	0.10	0.10–50		
BDE205	BDE 183*C13	Quad, WithOffset, 1/A	0.50	0.50–50		
OBIND	BDE 209*C13	Quad, WithOffset, 1/A	0.50	0.50–50		
BDE209	BDE 209*C13	Lin, WithOffset, 1/A	0.50	0.50–50		
DBDPE	BDE 209*C13	Quad, WithOffset, 1/A	2.00	2–50		

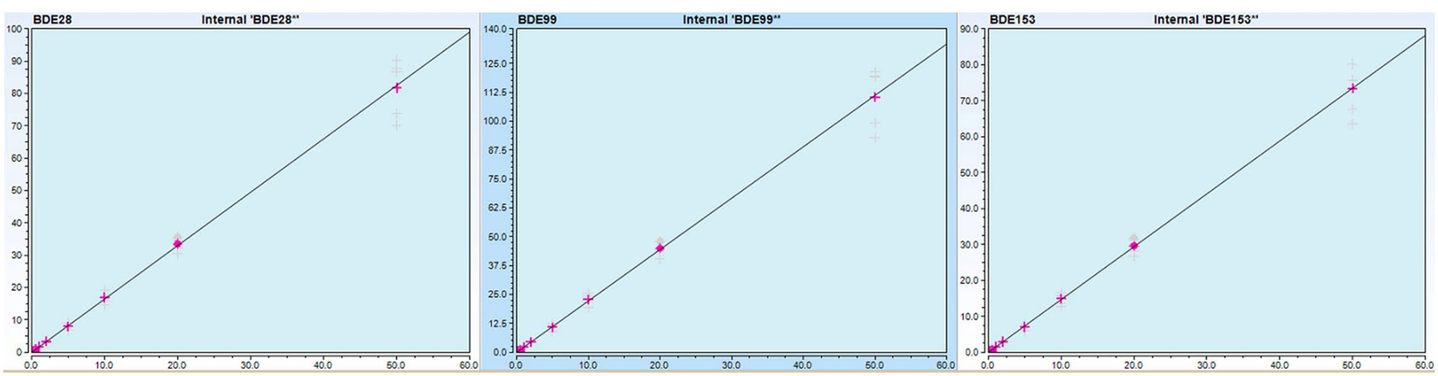


Figure 3. Example of superposition of five calibration curves for PBDE 28, PBDE99, and PBDE 153

Figure 4 shows the relative amount deviation for each calibration level (green points) across the repeated calibration curves. The white area represents the acceptable limit of relative amount deviation (%), whereas the red area indicates when outside the tolerance limit. The acceptance criteria of relative amount deviation of less than 40% at the LOQ level and less than 20% for the other levels were constantly met for all five calibration curves.

Sensitivity

The chromatograms in Figures 5–8 illustrate examples of six PBDEs at the LOQ in solvent, showcasing excellent resolution, peak shape, and signal-to-noise ratio.

The sensitivity of the analytical method demonstrates its capability to detect all compounds at very low levels, ensuring precise and reliable measurements.

All target congeners were successfully separated in under 17 minutes, achieving excellent resolution of the critical pair PBDE 49 and PBDE 71 (Figure 5). The use of the TraceGOLD TG-Contaminants capillary column ensured good chromatographic peak shapes for all compounds, including PBDE 209 and DBDPE, which are particularly difficult to analyze due to their tendency to break down and exhibit peak tailing.

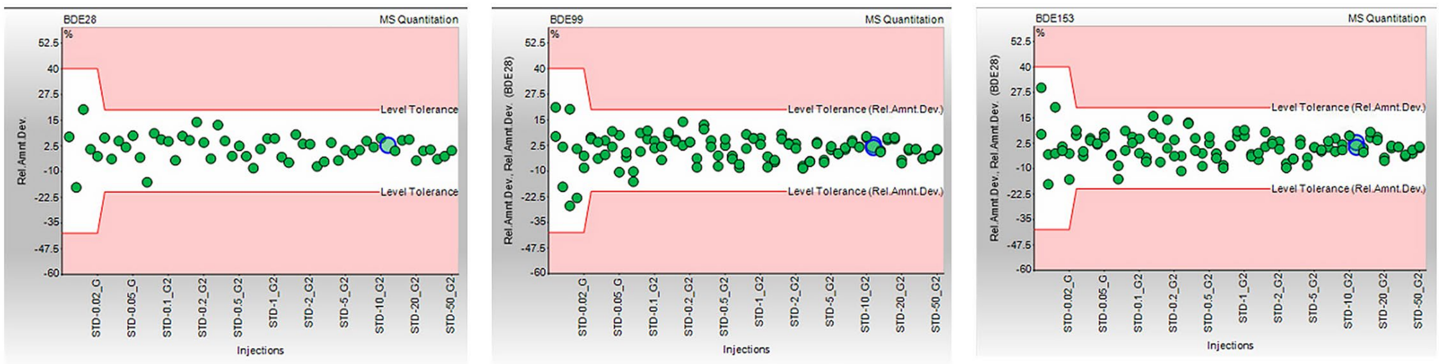


Figure 4. Example of charts to assess the relative amount deviation according to criteria for PBDE 28, PBDE99, and PBDE 153

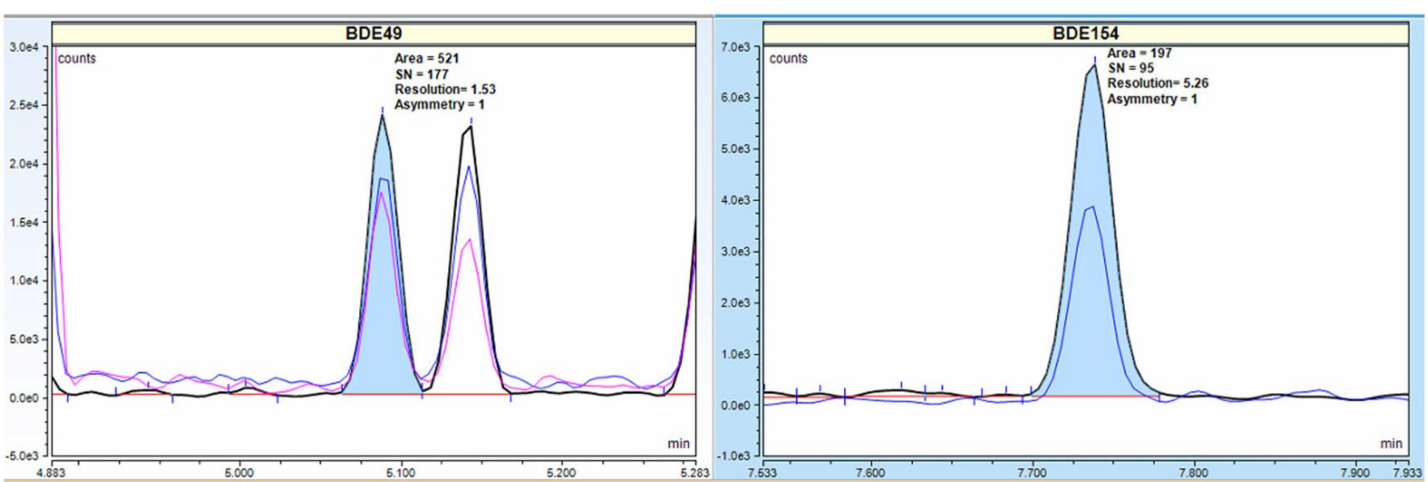


Figure 5. Overlaid SRM (quantifier and qualifier) at 0.02 µg/L for PBDE 49 and PBDE 154

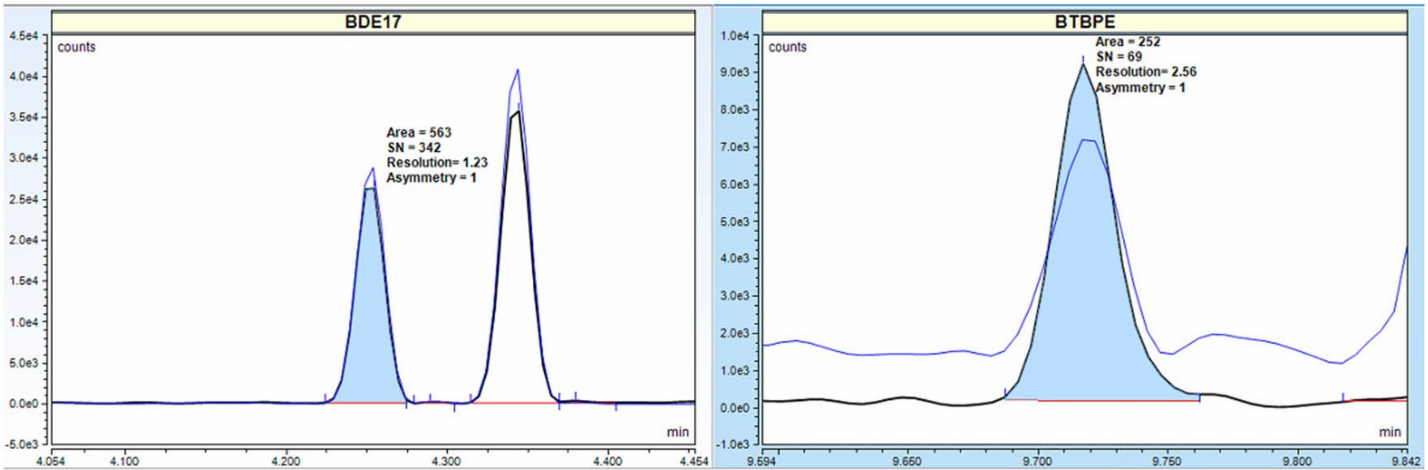


Figure 6. Overlaid SRM (quantifier and qualifier) at 0.05 µg/L for PBDE 17 and BTPE

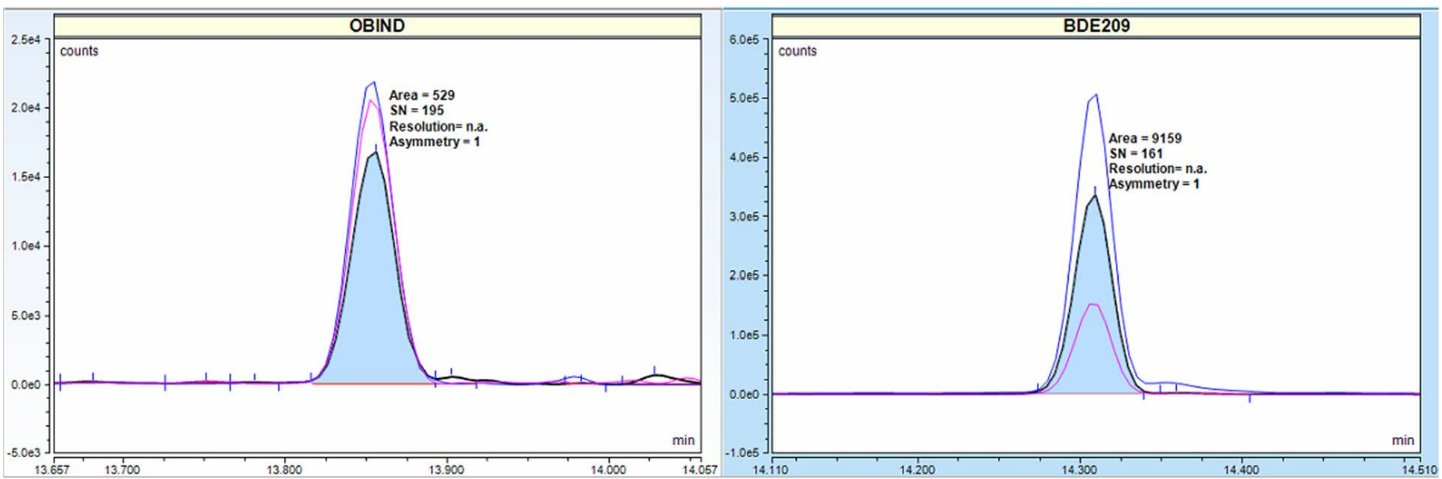


Figure 7. Overlaid SRM (quantifier and qualifier) at 0.5 µg/L for PBDE 209 and OBIND

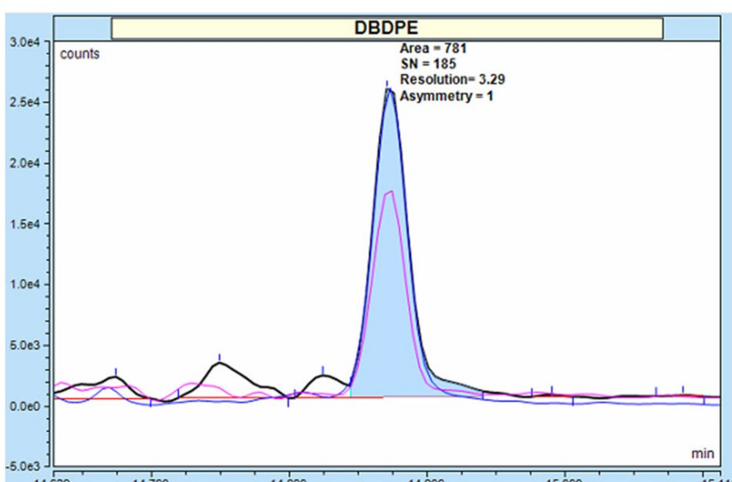


Figure 8. Overlaid SRM (quantifier and qualifier) at 2 µg/L for DBDPE

Conclusion

The developed analytical method combined with the unique features of the TSQ 9610 GC-MS/MS allow for method consolidation on a single platform while ensuring compliance with current regulations:

- **Single configuration for efficiency:** Thermo Fisher Scientific's analytical approach using a single column and triple quadrupole GC-MS/MS (TSQ 9610 system) streamlines the analysis of PBDEs, ensuring compliance with global regulatory standards such as European Standard 16694:2025.
- **Comprehensive analysis capability:** The TraceGOLD TG-Contaminants column provided exceptional chromatographic peak shapes and resolution, even for challenging compounds like PBDE 209 and DBDPE, ensuring precise and reliable measurements at very low levels (LOQ) for multiple PBDE congeners, with relative amount deviations consistently meeting stringent acceptance criteria, all within 17 minutes run time.
- **Exceeding regulatory requirements for PBDEs:** The method achieved LOQs surpassing those required by regulations.
- **Linearity and sensitivity validation:** The system demonstrated excellent linearity ($R^2 > 0.990$) and low limits of quantification.

The possibility of applying the same analytical method on a single analytical platform, without the need to change configuration and consumables, would allow contract testing laboratories to improve productivity, reduce the costs of ownership, and expand the list of analytes for future-proof performance.

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