Environmental

A streamlined laboratory workflow for the analysis of common contaminants according to the U.S. EPA 8270E and 8081B methods using GC-MS/MS

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Abstract

Purpose: The use of different instrument configurations and settings for analysis of various compound classes in environmental samples poses some challenges in terms of reduced productivity and sample throughput as well as increased time and costs for multiple platform maintenance, dedicated consumable usage as well as staff training. The adoption of a single instrument configuration would therefore greatly improve laboratory capacity, instrument- and consumables management, as well as streamlining staff training. .

Methods: The analysis of semi volatile compounds (SVOCs) and organochlorine pesticides(OCPs) was consolidated on a single analytical platform consisting of a triple quadrupole GC-MS/MS equipped with a SSL Injector.

Results: The utilization of a single instrument configuration for the analysis of SVOCs and OCPs in environmental samples allowed to meet the regulatory requirements while reducing the operational costs, including reduced training needs and centralization of consumables, as well as increasing the productivity.

Introduction

Contract testing laboratories dealing with environmental analysis must monitor diverse compound classes (SVOCs, pesticides, PCBs, etc.) in multiple matrices (drinking water, surface water, wastewater, soils, sludges) often requiring different instrument configurations and settings. This could lead to reduced productivity and sample throughput as well as increased time and costs. The adoption of a single platform based on triple quadrupole GC-MS/MS would streamline laboratory workflow allowing for confident detection, identification, and quantitation of hundreds of analytes within the same chromatographic run therefore improving laboratories sample throughput and productivity while reducing the overall costs.

Materials and methods

Test Method(s)

A Thermo Scientific™ TSQ 9610 triple quadrupole mass spectrometer equipped with Thermo Scientific™ NeverVent™ AEI ion source was coupled to a Thermo Scientific™ TRACE 1610 gas chromatograph equipped with a Thermo Scientific™ iConnect™ split/splitless (iConnect-SSL) injector and a Thermo Scientific™ AI/AS 1610 liquid autosampler. The same instrument configuration, chromatographic column and consumables were used for assessing instrument compliance to EPA 8270E and EPA 8081B methods (Figure 1). A Thermo Scientific™ TriPlus™ RSH SMART autosampler was placed on the bench and used as off-line sample preparation station for calibration curve dilution and internal standard addition. The use of an automated approach improved analyst's safety by reducing exposure to toxic chemicals such as dichloromethane (DCM).

Data Analysis

Data was acquired, processed and reported using the Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) software, version 7.3.2. Integrated instrument control ensures full automation of the analytical workflow combined with an intuitive user interface for data analysis, processing, customizable reporting, and storage in compliance with the Federal Drug Administration Title 21 Code of Federal Regulations Part 11 (Title 21 CFR Part 11). The Chromeleon Environmental Analysis Extension Pack for U.S. EPA-based environmental applications provide a comprehensive set of instrument methods, processing methods, and reports, designed for quick sequence set-up and reporting to allow rapid implementation of new instruments with ease.

Figure 1. Unified environmental method schematic for analysis of multiple compound classes using a single analytical platform.

EPA 625
(Base/Neutral/Acids)

EPA 8270E
(SVOC's)

Appendix IX
(OP Pesticides)

EPA 8081B
(OC Pesticides)

EPA 8141B
(OP Pesticides)

EPA 8082A
(Araclors/Congeners/
Chlordane/Toxaphene)

Unified Environmental Method (UEM)

- Same hardware
- Same column
- Same solvent
- Same method
- Speed
- Sensitivity

Results

EPA Method 8270E

U.S. EPA 8270E is the method used to determine the concentration of semi volatile organic compounds such as polycyclic aromatic hydrocarbons (PAHs) in many types of solid waste matrices, soils, air sampling media, and water samples by using gas chromatography coupled to mass spectrometry (GC-MS). The EPA 8270E method requires calculations of chromatographic resolution for these "critical pairs" to ensure that the fundamental chromatographic separation is adequate for analysis, so that for example analytical columns can be exchanged before degradation may affect data quality. An example of typical chromatograms for a reference standard mix prepared in DCM at a concentration of 500 µg/L (ppb) is shown in Figure 2. A very wide linear range can be easily achieved with the NeverVent™ AEI source coupled to the XLXR detector as demonstrated in Figure 3. The instrument detection limits (IDLs) were determined for all the target compounds by preparing n=7 solvent standard in DCM at 1 µg/L. Calculated values ranged from 5 to 45 µg/L with peak area RSD at the IDL level < 10% for most of the target compounds (Figure 4).

Figure 2. Example of chromatography achieved for a solvent standard spiked at 500 µg/L. The insets show examples of resolution for some critical pairs in compliance with the EPA 8270E method.

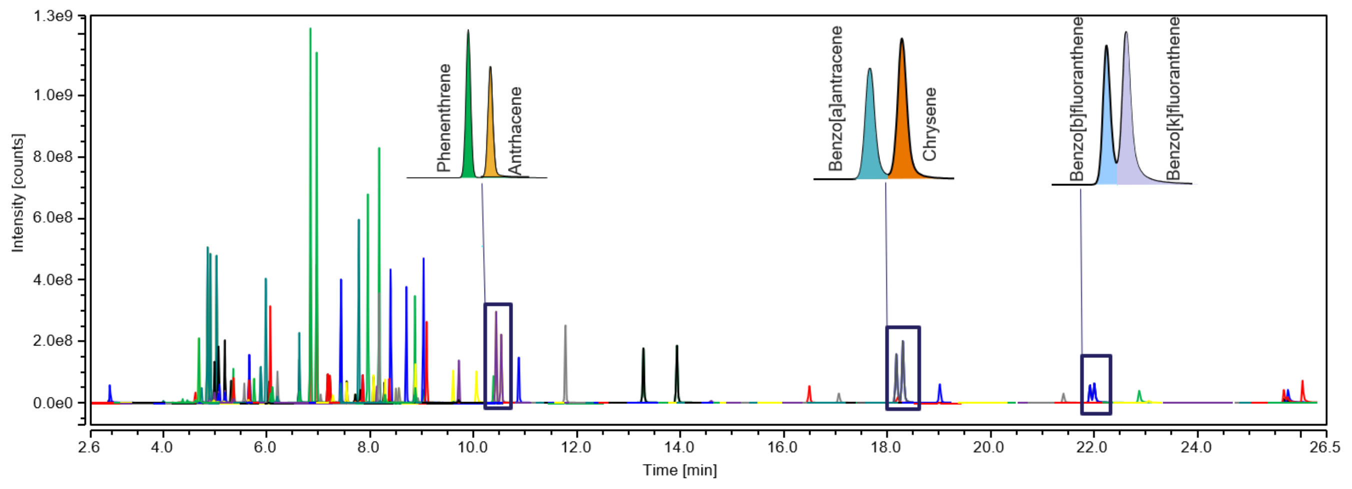


Figure 3. Example of calibration curves prepared in DCM (range:0.5-250 µg/L) for some selected target analytes as well as quantifier and qualifier ions at 0.5 µg/L.

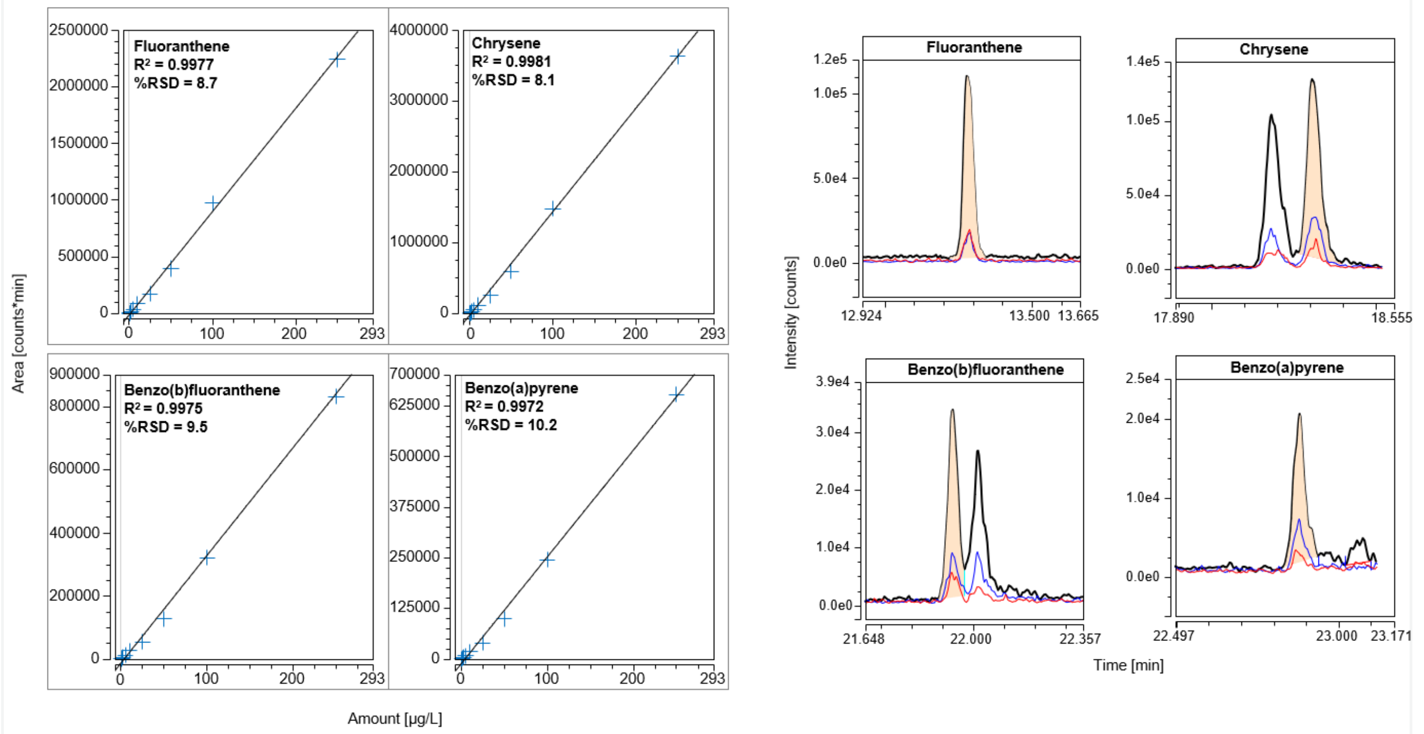
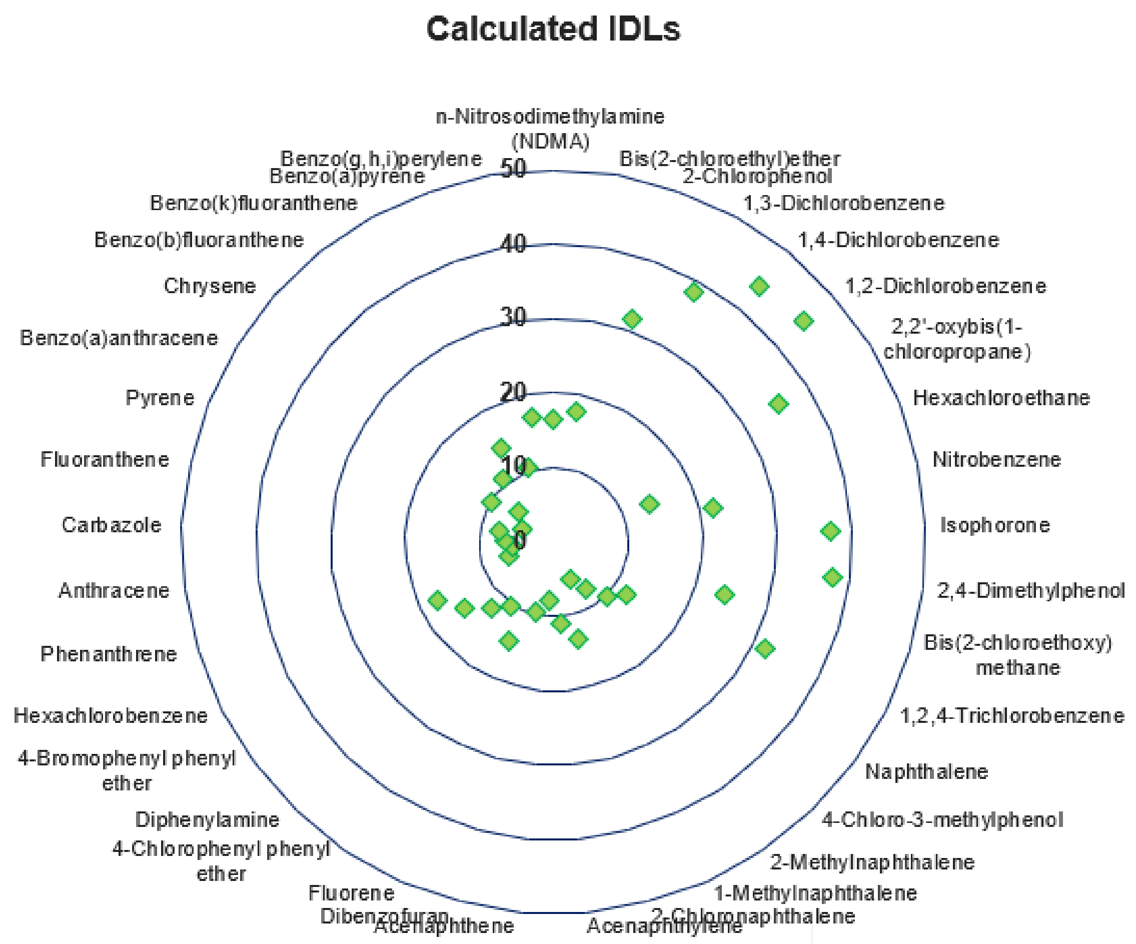


Figure 4. Examples of calculated IDLs for some selected compounds covering the whole volatility range.



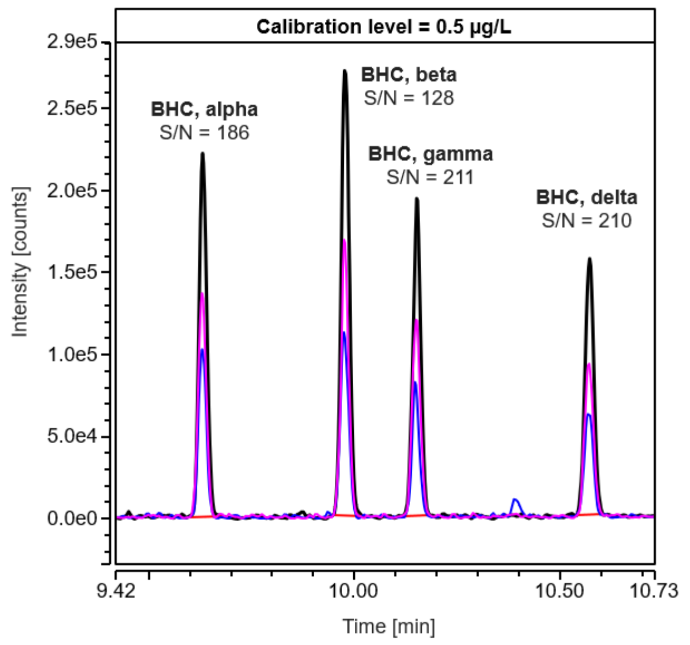
EPA Method 8081B

U.S. EPA method 8081B provides validated procedures for determination of organochlorine pesticides (OCPs). The use GC-MS/MS provided sensitivity and selectivity for confident identification and quantitation of analytes. The TSQ 9610 XLXR detector, offering extended detector lifetime and dynamic range, combined with the ultra-sensitive AEI and the best-in-class t-SRM acquisition mode allowed to achieve a liner range for the investigated compounds between 0.5 to 1000 µg/L (Figure 5) with calculated IDLs within 0.10 to 0.68 µg/L (Table 1).

Table 1. Calculated IDLs, peak area %RSD as well as average calibration factor %RSD for the investigated pesticides

Compound	RT (min)	Average RF %RSD	Calculated IDL (µg/L)	Peak area %RSD
BHC, alpha	9.63	5.2	0.18	6.5
BHC, beta	9.98	7.8	0.31	11.4
BHC, gamma	10.15	5.9	0.16	5.8
BHC, delta	10.58	9.1	0.12	4.2
Heptachlor	11.49	9.2	0.35	11.6
Aldrin	12.23	8.6	0.33	11.3
Heptachlor epoxide	13.08	8.7	0.32	11.3
Chlordane, gamma-*trans*	13.64	7.3	0.18	6.6
Endosulfan, peak1	13.64	17.7	0.68	23.7
Chlordane, alpha-*cis*	13.94	9.3	0.28	10.0
4,4'-DDE	14.48	9.4	0.10	3.7
Endrin	14.66	8.5	0.18	6.6
Dieldrin	14.68	14.4	0.35	16.4
Endosulfan, peak 2	15.50	15.9	0.43	12.5
4,4'-DDD	15.61	15.7	0.14	4.9
Endrin aldehyde	15.89	19.2	0.50	16.7
Endosulfan sulfate	16.57	9.8	0.16	6.5
4,4'-DDT	16.70	11.7	0.22	7.4
Endrin ketone	17.96	13.0	0.26	9.2
Metoxychlor	18.36	15.5	0.48	17.0

Figure 5. Intensity signal obtained for alpha, beta, gamma, and delta BHC at the lowest calibration point (0.5 µg/L).



Conclusions

Modern environmental laboratories benefit greatly from the next generation of gas chromatography mass spectrometry instrumentation that allows analysts to be more productive with simpler to use, more rugged, and more sensitive instrumentation. Advances in the Thermo Scientific line of GC-MS/MS allow for modernization of common environmental workflows for the analysis of SVOCs and OCPs, as well as many more target analytes, all from a single, easy-to-use platform.

- Using a single hardware platform translates into increased sample throughput and potential for consolidating multiple methods in one single GC run for streamlined operations, harmonization of consumables and supplies, as well as more efficient laboratory operations and better ROI.
- Accelerated routine maintenance operation through the Thermo Scientific™ NeverVent™ technology, allows a user to maintain the system without breaking the vacuum, and the modular concept of the TRACE 1610 GC, allows for flexible configurations and reduced instrument downtime
- Future proof analysis with the GC-MS/MS timed-SRM acquisition and the AEI ion source for lower limits of detection and confident compound identification and quantitation, far surpassing current regulations

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