

Optimize Critical Separations in the Simultaneous Analysis of PCBs and PAHs

Polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) are two critical classes of environmental contaminants that pose significant risks to human health and the environment. Europe has established comprehensive regulatory frameworks and monitoring programs to address these pollutants. Continued vigilance and research are essential to mitigate their impacts and protect public health and the environment. Please see references at the end of the article for information on the specific regulations.

Excellent resolution and selectivity for closely eluting compounds is essential for environmental analysis where complex mixtures require high-resolution separations to identify and quantify individual contaminants accurately. The Rxi-XLB, Rtx-440 and the Rxi-SVOCms GC columns were evaluated for optimal separation of select PAHs and PCBs in one method. This evaluation was done using a 30 m x 0.25 mm ID x 0.25 μ m column format. Standards were prepared at a concentration of 5 μ g/mL.

For this evaluation, the three columns produced varying results. The Rxi-SVOCms and Rtx-440 GC columns produced good resolution for the PAHs, but they did not separate the PCBs as well as the Rxi-XLB GC column (See Table 1; poor or no separation of PCB 28/31 on the Rxi-SVOCms and Rtx-440 GC columns). The Rxi-XLB GC column produced the best overall resolution for critical pair separations. Notably, excellent separation was achieved for PCB 31 and PCB 28 (peaks 8 & 9) and phenanthrene and anthracene (peaks 6 & 7) as shown in Figure 1. Other critical separations of interest include benzo[b]fluoranthene and benzo[k]fluoranthene (peaks 23 & 24) and indeno[1,2,3-cd] pyrene, dibenzo[a,h]anthracene (peaks 27 & 28), which the Rxi-XLB GC column was able to resolve to the requirements for regulatory compliance. PCB 180 and benzo[a]anthracene also exhibited the best separation using the Rxi-XLB compared to the other columns that were tested (peaks 18 & 19). The total analysis time using the Rxi-XLB GC column was 20 minutes.

Whether analyzing air, water, soil, or sediment methods, the Rxi-XLB GC column provides the resolution necessary to ensure compliance with environmental regulations and to support the protection of public health and the environment.

Table	e I. Com	narison (of Critical	Pair Resolution	nn* Values in th	e Analysis of PAH and PCBs	
Iabii		Dalison (я списан	raii nesoluli	an values in m	e Aliaivsis oi fan aliu funs	

Peak Pair	Peak Numbers	Rxi-XLB (30m x 0.25 mm ID x 0.25μm)	Rtx-440 (30m x 0.25 mm ID x 0.25μm)	Rxi-SVOCms (30m x 0.25 mm ID x 0.25 μm)
Phenanthrene Anthracene	6 7	2.92	2.57	2.26
PCB 31 PCB 28	8 9	1.28	0.71	0.00
PCB 180 Benzo[a]anthracene	18 19	2.30	1.00	1.16
Triphenylene Chrysene	20 21	0.71	0.56	3.23
Benzo[b]fluoranthene Benzo[k]fluoranthene	23 24	0.76	1.20	1.39
Indeno[1,2,3-cd]pyrene Dibenzo[a,h]anthracene	27 28	0.67	0.89	1.38
Run Time:		20 792 min	20 667 min	21 667 min

*Note - Baseline resolution = 1.50



Figure 1: Excellent separations were achieved for critical separations in the analysis of PAHs and PCBs on the Rxi-XLB GC column. 18 16.74 16.8 16.86 15 15.06 15.12 15.18 15.24 15.3 PCB 28 (#9) PCB 31 (#8) 11.48 11.36 11.4 11.44 20 23 24 19 10.9 10.96 11.02 11.08 11 | 13 8.9 18 14 1516 10 10 10.5 11 11.5 12 12.5 13 13.5 8.5 9 9.5 14 14.5 15 15.5 16 16.5 17 17.5 18 18.5 Time (min) GC EV1520 Column Rxi-XLB, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13723) Standard/Sample Peaks t_R (min) (µg/mL) 2-Methylnaphthalene (cat.# 31285) SV calibration mix #5/610 PAH (cat.# 31011) 1. Naphthalene 6 134 5 2. 2-Methylnaphthalene PCB congener mix #2 (cat.# 32294) 7.051 5 Diluent: Methylene chloride 3. Acenaphthylene 8.353 Conc.: 5 μg/mL 4. Acenaphthene 8.617 Injection 5. Fluorene 9.435 5 1 uL splitless Ini. Vol.: 6. Phenanthrene 10.946 5 5 5 Liner: Topaz Precision inlet liner, 4.0 mm x 6.3 x 78.5 (cat.# 23305) 7. Anthracene 8. PCB 31 11 045 Inj. Temp.: Oven 11 412 5 9. PCB 28 11.451 Oven Temp.: 40 °C (hold 1 min) to 120 °C at 30 °C/min to 330 °C at 16 °C/min (hold 4 min) 5 10. PCB 52 11.799 Carrier Gas He, constant flow 1 mL/min 11. Fluoranthene 12.868 Flow Rate: 12. PCB 101 12.902 5 5 5 5 Detector 13. Pyrene 13.235 Scan Scan Program: 14. 2-Methylfluoranthene 13 586 15. PCB 118 13.765 Group Start Time Scan Range Scan Rate 16. PCB 153 13.937 (min) (amu) (scans/sec) 17. PCB 138 14.289 50-550 10 18. PCB 180 15.07 5 19. Benzo[a]anthracene 15 152 5 5 5 Transfer Line Temp.: 250°C 20. Triphenylene 15 197 Source Temp.: 300°C 21. Chrysene 15.227 Ouad Temp.: 180°C 5 22. PCB 194 16.127 Agilent 7890A GC & 5975C MSD Instrument 23. Benzo[b]fluoranthene 16.799 **Sample Preparation** The sample was in a 2 mL short-cap, screw-thread vial (cat.# 21143) and capped 24. Benzo[k]fluoranthene 16.832 with a short-cap, screw-vial closure (cat.# 24495) 25. Benzo[e]pyrene 17.211 5 5 26. Benzo[a]pyrene 17 295

For an optimized analysis of PAHs on the Rxi-SVOCms, see our application at https://www.restek.com/articles/optimized-polycyclic-aromatic-hydrocarbon-pah-analysis-by-gc-ms.

5

5

19.161

19.209

References:

^{2.} International Organization for Standardization, ISO 18475:2023, Environmental solid matrices - Determination of polychlorinated biphenyls (PCB) by gas chromatography-mass selective detection (GC-MS) or electron-capture detection (GC-ECD), October 2023. https://www.iso.org/standard/84177.html



27. Indeno[1,2,3-cd]pyrene 28. Dibenzo[a,h]anthracene

29. Benzo[ghi]perylene

^{1.} iTeh, Inc., EN 17322:2020, Environmental Solid Matrices-Determination of polychlorinated biphenyls (PCB) by gas chromatography-mass selective detection (GC-MS) or electron-capture detection (GC-ECD), 28 July 2020. https://standards.iteh.ai/catalog/standards/cen/bd16cd16-70b0-4f08-8ad6-e3d3e125c05b/en-17322-2020

Rxi-XLB GC Capillary Column

Improvements in polymer synthesis and tubing deactivation enable us to make inert, stable Rxi-XLB columns especially well-suited for analyzing active, high molecular weight compounds with sensitive GC-MS systems, including ion trap detectors. Excellent efficiency, coupled with inertness, low bleed, and high thermal stability, make Rxi-XLB columns ideal for analyzing semivolatile compounds in drinking water.

Rxi-XLB columns are a great choice for semivolatile organics analysis by EPA Method 525.2. An Rxi-XLB column plus some simple adjustments to the injection conditions can greatly improve sensitivity for active and high molecular weight Method 525.2 target compounds.

- General-purpose columns with unique selectivity. Ideal for many GC-MS applications, including pesticides, PCB congeners (e.g., Aroclor mixes), PAHs.
- High thermal stability; exhibits extremely low bleed.
- Temperature range: 30 °C to 360 °C.

Catalog No.	Product Name	Units
13723	Rxi-XLB GC Capillary Column, 30 m, 0.25 mm ID, 0.25 μm	ea.



Topaz Precision Inlet Liner

Topaz GC inlet liners feature revolutionary technology and inertness to deliver you the next level of True Blue Performance:

- **Deactivation**—unbelievably low breakdown for accurate and precise low-level GC analyses.
- **Reproducibility**—unbeatable manufacturing controls and QC testing for superior reliability across compound classes.
- **Productivity**—unparalleled cleanliness for maximized GC uptime and lab throughput.
- 100% Satisfaction—if a liner doesn't perform to your expectations, we will replace it or credit your account.

Patented

Catalog No.	Product Name	Units
23305	Topaz, Precision Inlet Liner, 4.0 mm x 6.3 x 78.5, for Agilent GCs, w/Quartz Wool, Premium Deactivation	5-pk.





2-Methylnaphthalene Standard

2-Methylnaphthalene (91-57-6)

Catalog No.	Product Name	Units
31285	2-Methylnaphthalene Standard, 1000 μg/mL, Methylene Chloride, 1 mL/ampul	ea.



Acenaphthene (83-32-9) Acenaphthylene (208-96-8) Anthracene (120-12-7) Benz[a]anthracene (56-55-3) Benzo[a]pyrene (50-32-8) Benzo[b]fluoranthene (205-99-2) Benzo[k]fluoranthene (207-08-9) Benzo[g,h,i]perylene (191-24-2) Chrysene (218-01-9)
Dibenz[a,h]anthracene (53-70-3)
Fluoranthene (206-44-0)
Fluorene (86-73-7)
Indeno[1,2,3-cd]pyrene (193-39-5)
Naphthalene (91-20-3)
Phenanthrene (85-01-8)
Pyrene (129-00-0)

Catalog No.	Product Name	Units
31011	SV Calibration Mix #5/610 PAH, 2000 μg/mL, Methylene Chloride, 1 mL/ampul	ea.



2,4,4'-Trichlorobiphenyl (BZ #28) (7012-37-5) 2,2',5,5'-Tetrachlorobiphenyl (BZ #52) (35693-99-3) 2,2',4,5,5'-Pentachlorobiphenyl (BZ #101) (37680-73-2) 2,3',4,4',5-Pentachlorobiphenyl (BZ #118) (31508-00-6) 2,2',3,4,4',5'-Hexachlorobiphenyl (BZ #138) (35065-28-2) 2,2',4,4',5,5'-Hexachlorobiphenyl (BZ #153) (35065-27-1) 2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ #180) (35065-29-3)

Catalog No.	Product Name	Units
32294	PCB Congener Mix #2, 10 μg/mL, Isooctane, 1 mL/ampul	ea.

Vials and Caps

Catalog No.	Product Name	Units
21143	Short-Cap Vial with Grad Marking Spot, 9-425 Screw-Thread, 2.0 mL, 9 mm, 12 x 32 (vial only), Amber	1000-pk.
24495	Short Screw Caps, Polypropylene, Screw-Thread, PTFE/Silicone/PTFE Septa, Black, Preassembled, 2.0 mL, 9 mm	100-pk.



EZ No-Vent GC Column-Mass Spectrometer Connectors

Topaz GC inlet liners feature revolutionary technology and inertness to deliver you the next level of True Blue Performance:

- \bullet Change GC-MS columns in minutes without venting—100 μm transfer line maintains vacuum and eliminates the need to vent.
- Easy to install and maintain—no special tools or plumbing required.
- Gold-plated body for inertness.
- High-temperature polyimide ferrules eliminate leaks at the problematic transfer line fitting.
- Lower cost than other "no-vent" fittings.

Catalog No.	Product Name	Units
21323	EZ No-Vent GC Column-Mass Spectrometer Connector, for Agilent GCs with 5971/5972, 5973, 5975, or 5977 GC-MS	kit
22431	EZ No-Vent GC Column-Mass Spectrometer Connector, for Shimadzu QP2010, QP2010 Plus, and QP2020, kit	kit





Questions? Contact us or your local Restek representative (www.restek.com/contact-us).

Restek patents and trademarks are the property of Restek Corporation. (See www.restek.com/Patents-Trademarks for full list.) Other trademarks in Restek literature or on its website are the property of their respective owners. Restek registered trademarks are registered in the U.S. and may also be registered in other countries. To unsubscribe from future Restek communications or to update your preferences, visit www.restek.com/subscribe To update your status with an authorized Restek distributor or instrument channel partner, please contact them directly.

© 2024 Restek Corporation. All rights reserved. Printed in the U.S.A.

