Fast GC-MS/MS Analysis of PCBs and Dioxins on a Single Zebron[™] ZB-Dioxin GC Column

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Overview

Polychlorinated Biphenyls (PCBs) are synthetic chemical compounds that are persistent in nature, as are dioxins. Persistent pollutants are constantly monitored in environmental and food samples. Both of these analyte classes have numerous congeners that are similar in structure and need high chromatographic selectivity to resolve the most toxic isomer from the other. Commonly, two different GC column selectivities are utilized for PCBs and dioxin analysis by GC-HRMS or GC-MS/MS. The column swap between the two selectivities involves venting the MS, re-establishing vacuum, and tuning the mass spec, which causes a lot of instrument down time. In this application note, we present ZB-Dioxin as a single GC solution for both PCB and Dioxins by GC-MS/MS. This not only improves lab productivity by cutting the instrument down time but also provides a single method with short run time to process multiple Dioxin and PCB samples.

GC-MS/MS Conditions

Column: Zebron ZB-Dioxin Dimension: 40 meter x 0.18 mm x 0.14 μ m Part No.: 7PD-G045-47 Injection: Splitless for 1.5 min@ 290 °C, 1 µL Recommended Liner: Zebron PLUS Z-Liner™ (Compatible with Agilent[®] & Thermo[®] GC instrument) Part No.: AG2-0A13-05 **Carrier Gas:** Helium @ 0.8 mL/min (constant flow) Oven Program: 45 °C for 0 min to 175 °C @ 50 °C/min, to 220 °C @ 15 °C/min, to 250 °C @ 5 °C/min for 3 min, to 300 °C @ 50 °C/min for 10 min Detector: GC-MS/MS Transfer Line Temperature: 300 °C Mode: Scan (100-450 m/z) Source Temperature: 300 °C Quad Temperature: 150 °C Solvent Delay: 8.0 min

14.

17.

PCB-167

PCB-157

15. PCB-128

16. PCB-156

18. PCB-180

19. PCB-169

20. PCB-189

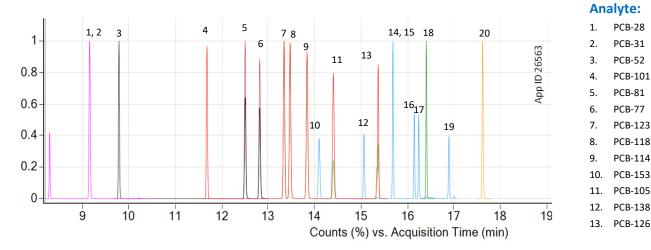


Figure 1. GC-MS/MS Analysis of PCBs on a 40 Meter Zebron ZB-Dioxin GC Column

Refer to Table 1 on pages 4 & 5 for MS/MS parameters

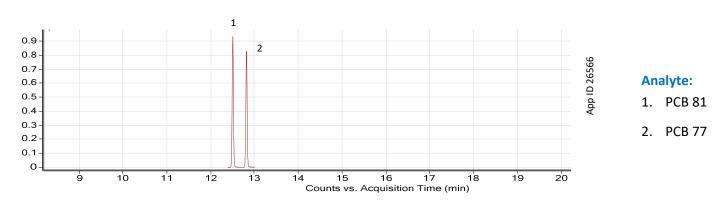
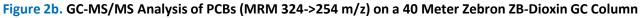
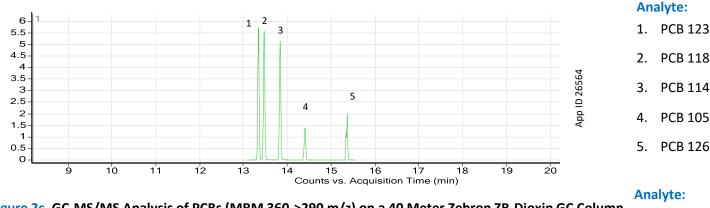
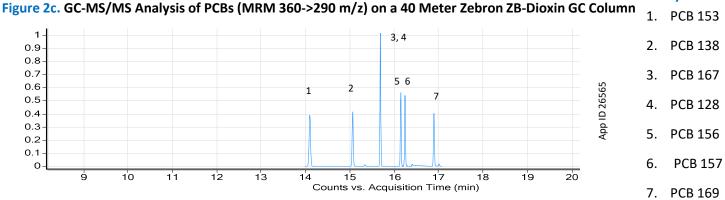
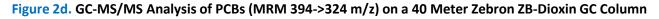


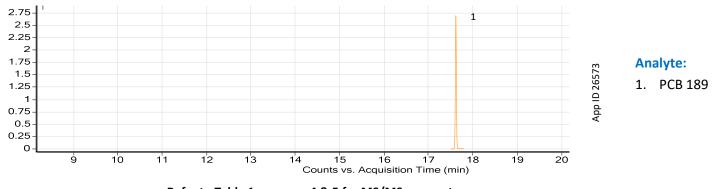
Figure 2a. GC-MS/MS Analysis of PCBs (MRM 290->220 m/z) on a 40 Meter Zebron ZB-Dioxin GC Column

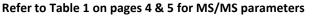






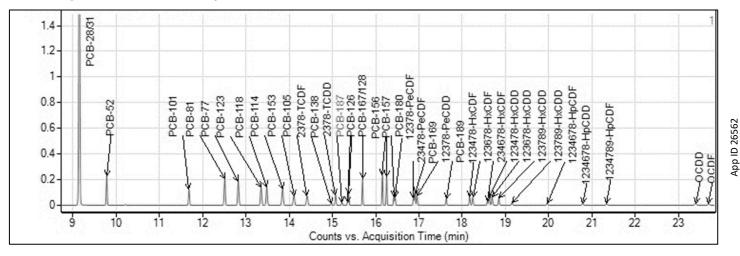






Have questions or want more details on implementing this method? We would love to help! Visit **www.phenomenex.com/Chat** to get in touch with one of our Technical Specialists









Analyte:

GC-MS/MS Conditions

| | | And | ycc. | | |
|----------------------------|---|---------|--------|---------------------|--|
| | | PCB-28 | 3 | PCB-157 | |
| | Zebron ZB-Dioxin 40 meter x 0.18 mm x 0.14 μm <u>7PD-G045-47</u> Splitless for 1.5 min@ 290 °C, 1 μL Zebron PLUS Z-Liner™ (Compatible with Agilent® & Thermo® GC instrument) <u>AG2-0A13-05</u> Helium @ 0.8 mL/min (constant flow) 45 °C for 0 min to 175 °C @ 50 °C/min, | PCB-3 | 1 | PCB-180 | |
| | | PCB-52 | 2 | 1,2,3,7,8-PeCDF | |
| | | PCB-10 | 01 | 2,3,4,7,8-PeCDF | |
| | | PCB-8 | 1 | PCB-169 | |
| | | PCB-7 | 7 | 1,2,3,7,8-PeCDD | |
| Recommended Liner: | | PCB-12 | 23 | PCB-189 | |
| | | PCB-1 | 18 | 1,2,3,4,7,8-HxCDF | |
| | | PCB-1 | 14 | 1,2,3,6,7,8-HxCDF | |
| | | PCB-1 | 53 | 2,3,4,7,8-HxCDF | |
| 0 | | PCB-10 |)5 | 1,2,3,4,7,8-HxCDD | |
| | to 220 °C @ 15 °C/min, to 250 °C @ 5 | 2,3,7,8 | B-TCDF | 1,2,3,6,7,8-HxCDD | |
| | °C/min for 3 min, to 300 °C @ 50 °C/min for | PCB-1 | 38 | 2,3,4,7,8-HxCDD | |
| | 10 min | 2,3,7,8 | B-TCDD | 1,2,3,4,6,7,8-HpCDD | |
| Detector: | GC-MS/MS | PCB-1 | 37 | 1,2,3,4,6,7,8-HpCDF | |
| Transfer Line Temperature: | 300 °C | PCB-12 | 26 | OCDD | |
| Mode: | Scan (100-450 m/z) | PCB-1 | 57 | OCDF | |
| Source Temperature: | 300 °C | PCB-12 | 28 | | |
| Quad Temperature: | 150 °C | PCB-1 | 56 | | |
| Solvent Delay: | 8.0 min | | | | |

Our Customer Says Yes!

"The ZB-Dioxin achieves superior resolution for both 2,3,7,8-TCDD and 2,3,7,8-TCDF while not only maintaining chromatography for the hexes but improving it. This is all performed while reducing the overall runtime over traditional 5ms dioxin columns by as much as 25%. The ZB-Dioxin increases throughput by not only eliminating the need for a second column confirmation, but also by allowing additional samples to be analyzed in each 12-hour analytical sequence" Andrew Patterson, Technical Director, Eurofins Specialty Services, USA

The opinions stated herein are solely those of the speaker and not necessarily those of any company or organization.



Table 1. MS/MS Method Parameters for Dioxin and PCB Separation

| | _ | | F | RT | ~ " | |
|--------------|-----------|---------|-------|-------|-------|-----|
| Analyte Name | Precursor | Product | 40 | 60 | Dwell | CE |
| | ion | ion | meter | meter | (ms) | (V) |
| | 258 | 186 | 9.19 | 10.77 | 72.1 | 25 |
| PCB 28 | 256 | 186 | 9.19 | 10.77 | 72.1 | 25 |
| РСВ 31 | 258 | 186 | 9.5 | 10.77 | 72.1 | 25 |
| | 256 | 186 | 9.5 | 10.77 | 72.1 | 25 |
| | 292 | 220 | 9.82 | 11.48 | 40.8 | 27 |
| PCB 52 | 220 | 150 | 9.82 | 11.48 | 40.8 | 35 |
| DOD 404 | 326 | 256 | 11.71 | 13.79 | 124 | 35 |
| PCB 101 | 254 | 184 | 11.71 | 13.79 | 124 | 35 |
| DOD 04 | 292 | 220 | 12.54 | 14.87 | 124.1 | 27 |
| PCB 81 | 290 | 220 | 12.54 | 14.87 | 124.1 | 24 |
| DCD 77 | 292 | 220 | 12.86 | 15.16 | 124.1 | 27 |
| PCB 77 | 290 | 220 | 12.86 | 15.16 | 124.1 | 24 |
| 000 400 /440 | 326 | 256 | 13.36 | 15.56 | 186.7 | 25 |
| PCB 123/118 | 324 | 254 | 13.38 | 15.64 | 186.7 | 25 |
| DC 444 | 326 | 256 | 13.47 | 15.88 | 92.9 | 25 |
| PC 114 | 324 | 254 | 13.47 | 15.88 | 92.9 | 25 |
| DCD 452 | 362 | 290 | 14.14 | 16.03 | 92.9 | 30 |
| PCB 153 | 360 | 290 | 14.14 | 16.03 | 92.9 | 30 |
| | 326 | 256 | 14.45 | 16.23 | 124.1 | 25 |
| PCB 105 | 324 | 254 | 14.45 | 16.23 | 124.1 | 45 |
| 2220 7005 | 306 | 243 | 15 | 16.61 | 75.5 | 40 |
| 2378-TCDF | 304 | 241 | 15 | 16.61 | 75.5 | 40 |
| 120 | 362 | 290 | 15.1 | 16.65 | 51.2 | 30 |
| 138 | 360 | 290 | 15.1 | 16.65 | 51.2 | 30 |
| 2220 7000 | 322 | 259 | 15.25 | 16.8 | 47.7 | 26 |
| 2378-TCDD | 320 | 257 | 15.25 | 16.8 | 47.7 | 26 |
| DCD 420 | 326 | 256 | 15.4 | 16.9 | 82.5 | 35 |
| PCB 126 | 324 | 254 | 15.4 | 16.9 | 82.5 | 45 |
| 120/167 | 358 | 288 | 15.71 | 17.25 | 124.1 | 24 |
| 128/167 | 360 | 290 | 15.71 | 17.25 | 124.1 | 30 |
| 450 | 360 | 290 | 16.18 | 17.79 | 64.3 | 30 |
| 156 | 358 | 288 | 16.18 | 17.79 | 64.3 | 24 |
| 1 - 7 | 360 | 290 | 16.27 | 17.9 | 44.3 | 30 |
| 157 | 358 | 288 | 16.27 | 17.9 | 44.3 | 24 |
| 190 | 396 | 324 | 16.43 | 18.08 | 44.3 | 25 |
| 180 | 394 | 359 | 16.43 | 18.08 | 44.3 | 15 |

| Total MRMs | 70 |
|---------------------------|-------|
| Minimum Concurrent MRMs | 1 |
| Maximum Concurrent MRMs | 8 |
| Min Dwell (group average) | 40.8 |
| Max Dwell (group average) | 186.7 |
| Estimated Cycle Time | 250 |
| IonSource | EI |
| Source Temp. (°C) | 300 |
| Solvent Delay (min) | 8 |

Table 1. MS/MS Method Parameters for Dioxin and PCB Separation (Continued)

| | Broquisor | Product | R | r | Dwell | CE | |
|---------------|------------------|---------|-------------|-------|-------|-----|--|
| Analyte Name | Precursor ion | ion | 40 meter | | | (V) | |
| 12270 0-005 | 340 | 277 | 16.46 | 18.18 | 72 | 40 | |
| 12378-PeCDF | 338 | 275 | 16.46 | 18.18 | 72 | 40 | |
| | 340 | 277 | 16.89 | 18.67 | 75.5 | 40 | |
| 23478-PeCDF | 338 | 275 | 16.89 | 18.67 | 75.5 | 40 | |
| 100 | 360 | 290 | 16.92 | 18.68 | 54.7 | 30 | |
| 169 | 358 | 288 | 16.92 | 18.68 | 54.7 | 24 | |
| 42270 0.000 | 356 | 293 | 16.97 | 18.76 | 75.5 | 26 | |
| 12378-PeCDD | 354 | 291 | 16.97 | 18.76 | 75.5 | 26 | |
| 100 | 394 | 324 | 17.65 | 19.55 | 124.1 | 34 | |
| 189 | 392 | 322 | 17.65 | 19.55 | 124.1 | 28 | |
| | 376 | 313 | 18.19 | 20.28 | 92.9 | 40 | |
| 123478-HxCDF | 374 | 311 | 18.19 | 20.28 | 92.9 | 40 | |
| | 376 | 313 | 18.26 | 20.37 | 92.9 | 40 | |
| 123678-HxCDF | 374 | 311 | 18.26 | 20.37 | 92.9 | 40 | |
| | 376 | 313 | 18.6 | 20.79 | 64.3 | 40 | |
| 234678-HxCDF | 374 | 311 | 18.6 | 20.79 | 64.3 | 40 | |
| | 392 | 329 | 18.64 | 20.84 | 43.5 | 25 | |
| 123478-HxCDD | 390 | 327 | 18.64 | 20.84 | 43.5 | 25 | |
| | 392 | 329 | 18.71 | 20.92 | 43.5 | 25 | |
| 123678-HxCDD | 390 | 327 | 18.71 | 20.92 | 43.5 | 25 | |
| | 392 | 329 | 18.86 | 21.13 | 64.3 | 25 | |
| 123789-HxCDD | 390 | 327 | 18.86 | 21.13 | 64.3 | 25 | |
| | 376 | 313 | 19.17 | 21.56 | 124.1 | 40 | |
| 123789-HxCDF | 374 | 311 | 19.17 | 21.56 | 124.1 | 40 | |
| | 410 | 347 | 19.98 | 22.59 | 124.1 | 40 | |
| 1234678-HpCDF | 408 | 345 | 19.98 | 22.59 | 124.1 | 40 | |
| | 426 | 363 | 20.81 | 23.67 | 124.1 | 24 | |
| 1234678-HpCDD | 424 | 361 | 20.81 | 23.67 | 124.1 | 24 | |
| | 410 | 347 | 21.37 | 24.45 | 124.1 | 40 | |
| 1234789-HpCDF | 408 | 345 | 21.37 | 24.45 | 124.1 | 40 | |
| | 460 | 397 | 23.43 | 27.21 | 92.9 | 24 | |
| OCDD | 458 | 395 | 23.43 | 27.21 | 92.9 | 24 | |
| o oo - | 444 | 381 | 23.73 | 27.67 | 61.6 | 40 | |
| OCDF | 442 | 379 | 23.73 | 27.67 | 61.6 | 40 | |

APPLICATIONS

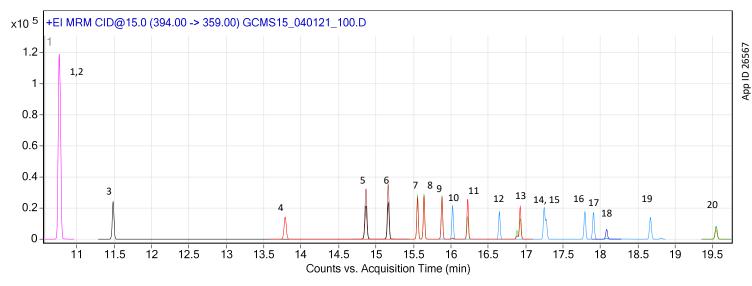
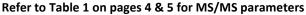


Figure 4. GC-MS/MS Analysis of PCBs on a 60 Meter ZB-Dioxin GC column



| GC-MS/MS Condition | Analyte: | | | | |
|---|---|--|--|---------------------------------|---|
| Column: Dimension: Part No.: Injection: Recommended Liner: Part No.: Carrier Gas: | Zebron ZB-Dioxin 60 meter x 0.25 mm x 0.20 μm 7KG-G045-10 Splitless for 1.5 min@ 290 °C, 1 μL Zebron PLUS Z-Liner™ (Compatible with Agilent® & Thermo® GC instrument) AG2-0A13-05 Helium @ 1.5 mL/min (constant flow) 45 °C for 0 min to 175 °C @ 50 °C/min, to 220 °C @ 15 °C/min, to 250 °C @ 5 °C/min for 3 min, to 300 °C @ 50 °C/min for | 1. 2. 3. 4. 5. 6. 7. 8. 9. 10. 11. | PCB-28 PCB-31 PCB-52 PCB-101 PCB-81 PCB-77 PCB-123 PCB-118 PCB-114 PCB-153 PCB-105 PCB-105 PCB-138 | 15. 16. 17. 18. 19. | PCB-167 PCB-128 PCB-156 PCB-157 PCB-180 PCB-169 PCB-189 |
| Detector: | 15 min GC-MS/MS | 13. | PCB-126 | | |
| Transfer Line Temperature: | 300 °C | | | | |
| Mode: | Scan (100-450 m/z) | | | | |
| Source Temperature: | 300 °C | | | | |
| Quad Temperature: | 150 °C | | | | |
| Solvent Delay: | 8.0 min | | | | |

Our Customer Says Yes!

"The ZB-Dioxin column better resolution in all congeners and in the critical separations of the standard mixture compared to the one normally used (5% Phenyl Arylene)." **Dr. Fabio Busico, Istituto Zooprofilattico Sperimentale del Lazio e Toscana M.Aleandri**

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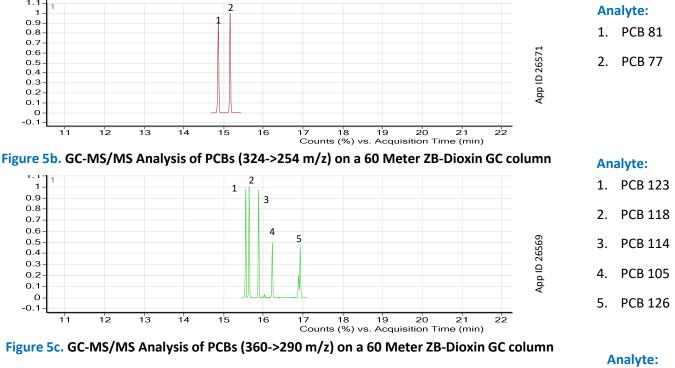


Figure 5a. GC-MS/MS Analysis of PCBs (290->222 m/z) on a 60 Meter ZB-Dioxin GC column

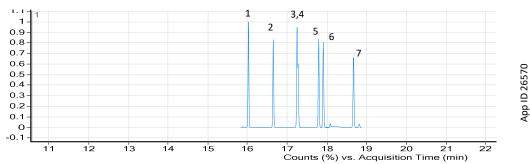
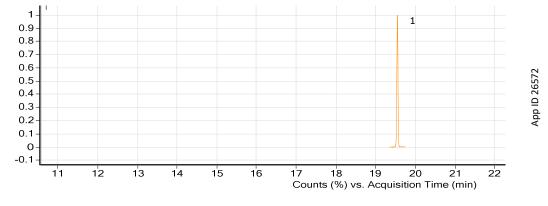


Figure 5d. GC-MS/MS Analysis of PCBs (392->322 m/z) on a 60 Meter ZB-Dioxin GC column





1. PCB 189

PCB 153

PCB 138

PCB 167

PCB 128

PCB 156

PCB 157

PCB 169

1.

2.

3.

4.

5.

6.

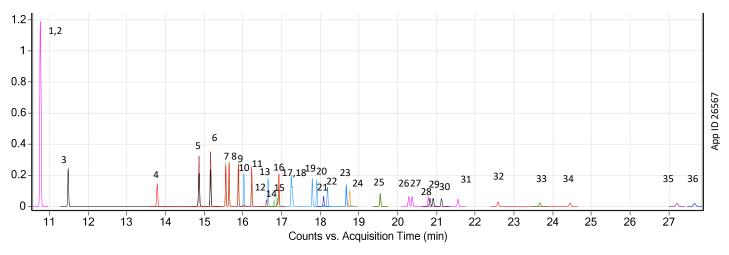
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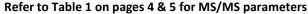
Refer to Table 1 on pages 4 & 5 for MS/MS parameters



APPLICATIONS

Figure 6. GC-MS/MS Analysis of PCBs and Dioxins on a 60 Meter ZB-Dioxin GC column





GC-MS/MS Conditions

Column: Zebron ZB-Dioxin Dimension: 60 meter x 0.25 mm x 0.20 μm Part No.: 7KG-G045-10 Injection: Splitless for 1.5 min@ 290 °C, 1 µL Recommended Liner: Zebron PLUS Z-Liner[™] (Compatible with Agilent[®] & Thermo[®] GC instrument) Part No.: AG2-0A13-05 Carrier Gas: Helium @ 1.5 mL/min (constant flow) Oven Program: 45 °C for 0 min to 175 °C @ 50 °C/min, to 220 °C @ 15 °C/min, to 250 °C @ 5 °C/min for 3 min, to 300 °C @ 50 °C/min for 15 min Detector: GC-MS/MS Transfer Line Temperature: 300 °C Mode: Scan (100-450 m/z) Source Temperature: 300 °C Quad Temperature: 150 °C Solvent Delay: 8.0 min

Analyte:

| 1. | PCB-28 | 20. | PCB-157 |
|-----|--------------|-----|---------------------|
| 2. | PCB-31 | 21. | PCB-180 |
| 3. | PCB-52 | 22. | 1,2,3,7,8-PeCDF |
| 4. | PCB-101 | 23. | 2,3,4,7,8-PeCDF |
| 5. | PCB-81 | 24. | PCB-169 |
| 6. | PCB-77 | 25. | 1,2,3,7,8-PeCDD |
| 7. | PCB-123 | 26. | PCB-189 |
| 8. | PCB-118 | 27. | 1,2,3,4,7,8-HxCDF |
| 9. | PCB-114 | 28. | 1,2,3,6,7,8-HxCDF |
| 10. | PCB-153 | 29. | 2,3,4,7,8-HxCDF |
| 11. | PCB-105 | 30. | 1,2,3,4,7,8-HxCDD |
| 12. | 2,3,7,8-TCDF | 31. | 1,2,3,6,7,8-HxCDD |
| 13. | PCB-138 | 32. | 2,3,4,7,8-HxCDD |
| 14. | 2,3,7,8-TCDD | 33. | 1,2,3,4,6,7,8-HpCDD |
| 15. | PCB-187 | 34. | 1,2,3,4,6,7,8-HpCDF |
| 16. | PCB-126 | 35. | OCDD |
| 17. | PCB-167 | 36. | OCDF |
| 18. | PCB-128 | | |
| 19. | PCB-156 | | |
| | | | |



By structure, Dioxins and Furans have a hetero atom while PCBs have a biphenyl ring with substituted chlorine group at different position. So, the EI source fragmentation as well as MS/MS transition can spectrally resolve Dioxins from PCBs. For this experiment, mixture of 36 components including PCBs and Dioxins and Furans were analyzed on a 40m and 60m ZB-Dioxin GC Column. **Figure 1** represents the separation of 20 PCBs on a ZB-Dioxin GC column with 20 PCBs run within 19 min. **Figure 2a to 2d** represents individual PCBs that are quantified by their precursor to product transitions. This includes commonly analyzed critical pairs in PCB including PCB 123 and PCB 118, PCB 105 and 153 and PCB 156/157 that are completely resolved from each other. **Figure 3** represents the separation of 36 Dioxins , furans and PCBs on 40m ZB-Dioxin using the same method parameters within 24 min run time. Thus, the developed GC-MS/MS method provided fast analysis of Dioxins and PCBs within 24 min on a 40 meter ZB-Dioxin GC column. Traditionally, Dioxin and PCB analysis can take up to 60 minutes run time. With the optimal selectivity and column dimensions, such short run time is realized.

Generally, laboratories analyzing Dioxins perform their analysis on a 60 meter 5ms selectivity GC column and swap a different selectivity for their PCB analysis. Presented in **Figure 4** is the separation of 20 PCBs on a 60 meter ZB-Dioxin GC column. Figure **5a to d** represents individual critical PCBs by MS/MS transitions and **Figure 6** represents 36 Dioxins and PCBs on a single method. In addition to providing fast overall run time, 60 meter ZB-Dioxin GC column provides optimal separation of critical pairs of Dioxins and PCBs. The complete list of PCBs, Dioxins and furans along with MS transitions and their retention times on 40m and 60m ZB-Dioxin GC columns are presented in **Table 1**.

Notably, both 60 meter and 40 meter ZB-Dioxin provides similar profile due to the equivalent phase volume ratio in these two columns. While 60 meter ZB-Dioxin column dimension provides loadability, the 40 meter x 0.18 mm x 0.14 μ m provides similar profile and resolution with much shorter analysis time. The versatile selectivity of ZB-Dioxin GC column allows it to analyze PCBs and dioxins in a single method without the necessity to change columns, in addition, this column also provides fast analysis, thus reducing down time and improving lab productivity.

Conclusion

Zebron[™] ZB-Dioxin GC column provides optimal selectivity to analyze Dioxins and PCBs on a single column. With optimal dimension, a runtime as short as 24 min was achieved to separate 36 PCBs and Dioxins mixture.



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