

Separation of a Mixture of PCBs Using an Accucore C18 HPLC Column

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Key Words

Accucore, Core Enhanced Technology, solid core, polychlorinated biphenyls, PCB, environment, HPLC

Abstract

Polychlorinated biphenyls (PCBs) are man-made organic compounds composed of two phenyl (benzene ring) groups with multiple chlorine atoms attached. The heat-resistant and non-conductive properties of PCBs led to their extensive use as coolants and lubricants in electrical equipment until the late 1970s when their toxicity was recognized and their usage restricted. However, PCBs are very persistent environmental contaminants. Improper disposal and storage of old electrical equipment continue to allow the chemicals to leach into the environment. Soil and water samples are routinely tested for the presence of PCBs, and a number of EPA methods exist for their analysis.^{1,2} This HPLC method shows the use of the Thermo Scientific Accucore C18 column for the separation of PCBs in a commercial mixture. The 20 minute analysis time provides good resolution between all components.

Introduction

Environmental monitoring of chemicals is becoming more widespread. Traditionally these analyses have been carried out using GC. In laboratories where access to GC equipment is not readily available, alternative approaches such as HPLC should be considered.

Accucore™ HPLC columns use Core Enhanced Technology™ to facilitate fast and highly efficient separations. The 2.6 µm diameter particles are not totally porous but have a solid core and a porous outer layer. The optimized phase bonding creates a series of high coverage, robust phases. The tightly controlled 2.6 µm diameter of the Accucore particles results in performance typically seen with sub-2 µm materials but at much lower backpressures.



Experimental Details

| Consumables | Part Number |
|---|-------------|
| The PCB congener mixture of chlorinated biphenyls was obtained from a commercial supplier | |
| Fisher Scientific HPLC grade water | W/0106/17 |
| Fisher Scientific HPLC grade acetonitrile | A/0626/17 |
| Thermo Scientific Premium 2 mL vial convenience kit | 60180-600 |

Sample Preparation

A 200 μ L aliquot of the PCB congener mixture was dissolved in 800 μ L methanol.

Separation Conditions

| | | | |
|-------------------------|--|--------------|----|
| Instrumentation: | Thermo Scientific Accela UHPLC system | | |
| Column: | Accucore C18 2.6 μ m, 150 x 2.1 mm | 17126-152130 | |
| Mobile phase A: | Water | | |
| Mobile phase B: | Acetonitrile | | |
| Gradient: | Time | %A | %B |
| | 0 | 50 | 50 |
| | 8 | 30 | 70 |
| | 16 | 20 | 80 |
| | 19 | 10 | 90 |
| | 20 | 10 | 90 |
| | 21 | 50 | 50 |
| Column temperature: | 65 °C | | |
| Injection details: | 2 μ L partial loop | | |
| Injection wash solvent: | Water / acetonitrile (80:20 v/v) | | |
| UV detector wavelength: | 205 nm | | |
| Backpressure: | 230 bar at 400 μ L/min | | |

Results

Sixteen components were successfully separated with good resolution except for the heptachloro isomers, which are peaks 13 and 14 in Figure 1. Peak class identities were established by correlation of compound hydrophobicity with LC-MS analysis of isotopic distributions and confirmatory mass ions. The isobaric components were not individually identified but treated as groups based on the number of chlorine atoms present in the compound (Table 1).

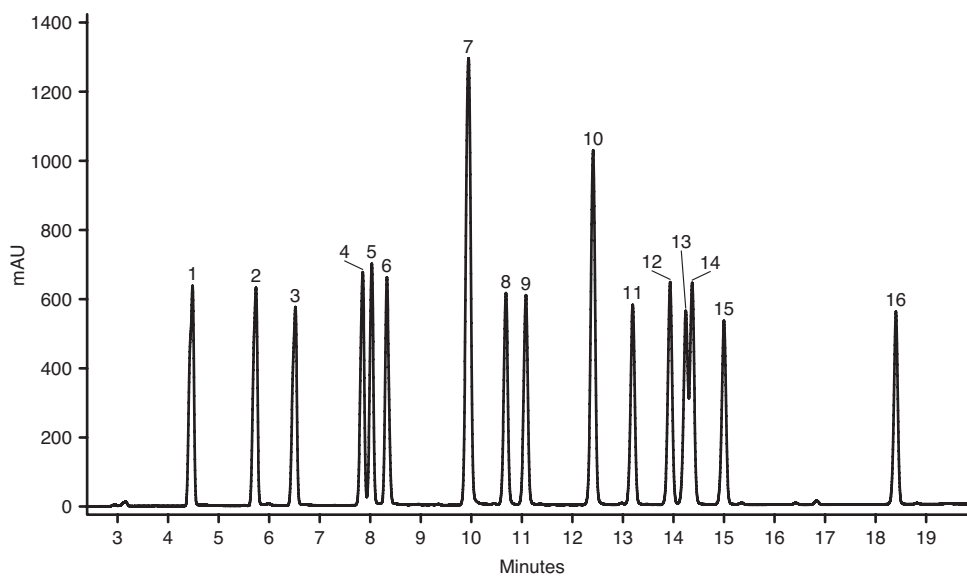


Figure 1: UV 205 nm chromatogram of the HPLC analysis of a commercial mixture of polychlorinated biphenyls

| Peak # | Class | Retention Time | | Peak Asymmetry | | Peak Resolution | |
|--------|-----------------|----------------|------|--|------|-----------------|------|
| | | Mean | % CV | Mean | % CV | Mean | % CV |
| 1 | not determined | 4.45 | 0.35 | 0.76 | 2.11 | | |
| 2 | Cl ₃ | 5.72 | 0.22 | 0.81 | 3.07 | 8.30 | 4.10 |
| 3 | Cl ₃ | 6.49 | 0.15 | 0.80 | 2.78 | 5.13 | 4.34 |
| 4 | Cl ₄ | 7.82 | 0.15 | 0.87 | 2.36 | 9.11 | 4.48 |
| 5 | Cl ₄ | 8.00 | 0.14 | 0.90 | 2.46 | 1.36 | 4.00 |
| 6 | Cl ₄ | 8.30 | 0.18 | 1.11 | 3.16 | 2.26 | 3.50 |
| 7 | Cl ₅ | 9.89 | 0.30 | 0.96 | 2.29 | 10.44 | 3.06 |
| 8 | Cl ₅ | 10.61 | 0.34 | 0.94 | 1.01 | 4.69 | 2.81 |
| 9 | Cl ₆ | 11.01 | 0.37 | 0.93 | 1.19 | 2.68 | 2.89 |
| 10 | Cl ₆ | 12.33 | 0.35 | 0.94 | 0.86 | 8.68 | 2.31 |
| 11 | Cl ₆ | 13.11 | 0.31 | 0.96 | 2.18 | 5.05 | 2.41 |
| 12 | Cl ₇ | 13.87 | 0.34 | 0.95 | 0.50 | 4.84 | 1.41 |
| 13 | Cl ₇ | 14.18 | 0.34 | Not calculated due to partial resolution | | 0.72 | 5.20 |
| 14 | Cl ₇ | 14.31 | 0.31 | | | 1.88 | 2.21 |
| 15 | Cl ₇ | 14.94 | 0.29 | 0.96 | 1.12 | 3.94 | 1.32 |
| 16 | Cl ₉ | 18.34 | 0.20 | 0.93 | 0.94 | 22.99 | 0.74 |

Table 1: Retention time, peak asymmetry and resolution data from the HPLC analysis of eight replicate injections of a commercial mixture of polychlorinated biphenyls

Conclusion

The use of an Accucore C18 column allowed the separation of different PCB classes in less than 20 minutes at standard HPLC backpressures. Accucore C18 columns are therefore an excellent alternative choice for the analysis of PCBs where access to the more traditional GC instrumentation is not available.

References

1. EPA Method 1668: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS, November 2008, EPA-821-R-08-020.
2. EPA SW 846 Method 8082A: Polychlorinated Biphenyls (PCBs) by Gas Chromatography, February 2007.

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