

Achieve Exceptional Resolution of PAHs, Including Several Isomer Sets Using SLB®-ILPAH Capillary GC Columns

By: Lisa McCombie, Product Manager GC, and Len Sidisky R&D Manager Gas Chromatography

Polycyclic aromatic hydrocarbons (PAHs) are ubiquitous in the environment. They belong to a group known as persistent organic pollutants (POPs). Monitoring is important because they are identified as carcinogens. Multiple isomers exist, which are difficult to resolve chromatographically. SLB®-ILPAH is a special purpose column based on an ionic liquid stationary phase. A distinct combination of stationary phase selectivity and efficient column dimensions allow exceptional resolution of PAHs, including several isomer sets. Complete column specifications are listed in Table 1.

Table 1. SLB®-ILPAH Column Specifications

| | |
|---------------|---|
| Application: | This special purpose and specially tested capillary GC column is designed for the analysis of polycyclic aromatic hydrocarbons (PAHs). It incorporates an ionic liquid stationary phase. Each column is individually tested to ensure resolution of several key sets (phenanthrene / anthracene, benzo[a]anthracene / chrysene / triphenylene, and benzo[b]fluoranthene / benzo[k]fluoranthene / benzo[j]fluoranthene). |
| USP Code: | None |
| Phase: | Non-bonded; 1,12-Di(triisopropylphosphonium) dodecane bis(trifluoromethanesulfonyl)imide |
| Temp. Limits: | Subambient to 300 °C (isothermal or programmed) |

Conditions

| | |
|--------------|---|
| column: | SLB-ILPAH, 20 m × 0.18 mm I.D., 0.05 µm (29799-U) |
| oven: | 90 °C (6 min), 20 °C/min to 225 °C, 5 °C/min to 300 °C (10 min) |
| inj. temp.: | 300 °C |
| detector: | FID, 310 °C |
| carrier gas: | hydrogen, 1.3 mL/min, constant flow |
| injection: | 1 µL, 50:1 split |
| liner: | 4 mm I.D., split type, cup design |
| sample: | 10 PAHs, each at 100 µg/mL in methylene chloride |

Resolution Test

Every SLB®-ILPAH column is specially tested to ensure it meets stringent resolution requirements for several sets of PAHs. Figure 1 depicts a chromatogram obtained from analysis of the QC test mix. The resolution (R_S)

results obtained from this chromatogram are:

- 2.21 for phenanthrene/anthracene
- 1.82 for benzo[a]anthracene/chrysene
- 1.69 for chrysene/triphenylene
- 2.65 for benzo[b]fluoranthene/benzo[k]fluoranthene
- 1.52 for benzo[k]fluoranthene/benzo[j]fluoranthene

Column efficiency is also determined by measuring the theoretical plate value of naphthalene. For this chromatogram, it was good, based on the value of 109,480 plates that was obtained.

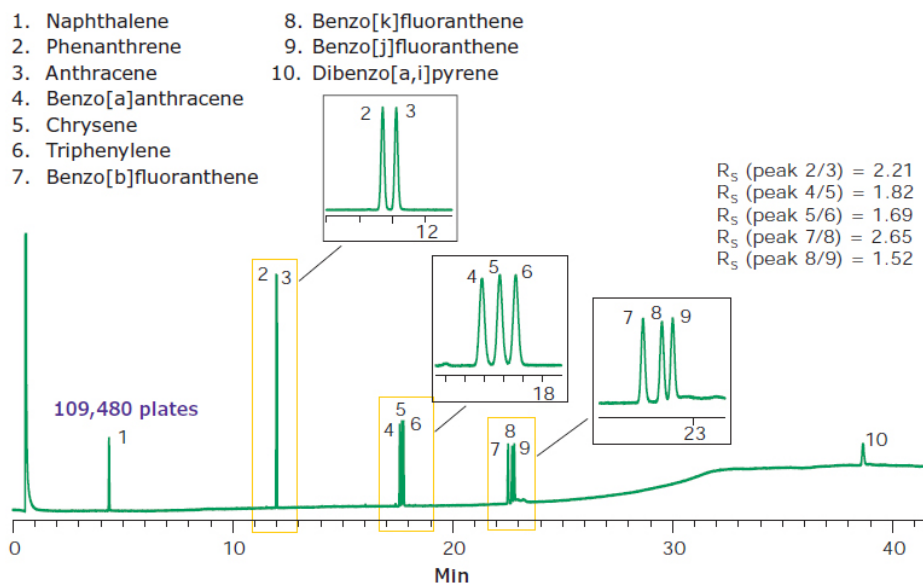


Figure 1. QC Test Mix

22-Component PAH Mix

Multiple regulatory agencies around the world have promulgated methodologies for the analysis of PAHs. The number of analytes listed in these methods ranges from 16 to 24. To present data more accurately aligned with real-world usage, a 22-component PAH mix which contains the most frequently listed PAHs was analyzed on a SLB®-ILPAH column. The resulting chromatogram is shown in Figure 2. The significant finding is that this column has the necessary selectivity to provide exceptional resolution for several sets of PAHs, such as peaks 5/6, peaks 9/10, and peaks 12/13/14. Also of great interest is that this column can provide baseline separation of dibenz[a,h]anthracene (peak 16) and indeno[1,2,3-cd]perylene (peak 17). This last pair typically co-elutes on other columns, and requires the use of mass spectrometry (MS) for proper identification.

Conditions

| | |
|--------------|--|
| column: | SLB®-ILPAH, 20 m × 0.18 mm I.D., 0.05 µm (29799-U) |
| oven: | 150 °C, 15 °C/min to 225 °C, 5 °C/min to 300 °C (15 min) |
| inj. temp.: | 300 °C |
| detector: | FID, 310 °C |
| carrier gas: | hydrogen, 1.3 mL/min, constant flow |
| injection: | 1 µL, 300:1 split |
| liner: | 2.3 mm I.D., split/splitless type, wool packed straight FocusLiner™ design |
| sample: | 22 analytes, each at 100 µg/mL in methylene chloride |

- | | |
|-----------------------|----------------------------|
| 1. Naphthalene | 12. Benzo[b]fluoranthene |
| 2. Acenaphthene | 13. Benzo[k]fluoranthene |
| 3. Acenaphthalene | 14. Benzo[j]fluoranthene |
| 4. Fluorene | 15. Benzo[a]pyrene |
| 5. Phenanthrene | 16. Dibenz[a,h]anthracene |
| 6. Anthracene | 17. Indeno[1,2,3-cd]pyrene |
| 7. Fluoranthene | 18. Benzo[g,h,i]perylene |
| 8. Pyrene | 19. Dibenzo[a,l]pyrene |
| 9. Benzo[a]anthracene | 20. Dibenzo[a,e]pyrene |
| 10. Chrysene | 21. Dibenzo[a,i]pyrene |
| 11. 5-Methylchrysene | 22. Dibenzo[a,h]pyrene |

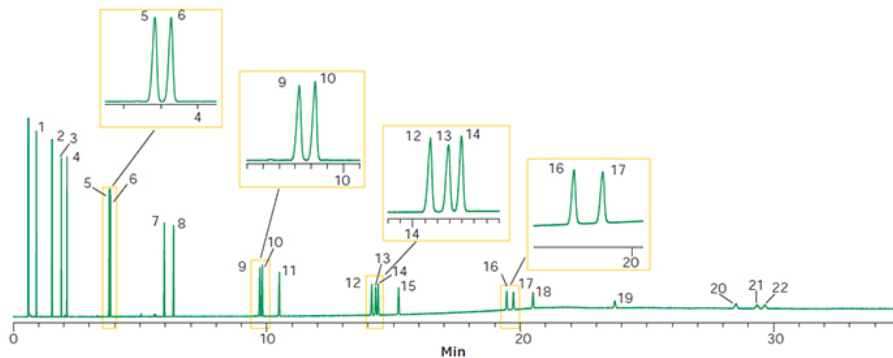


Figure 2. 22-Component PAH Mix

Conclusion

The main strength of ionic liquid GC columns is unique selectivity. This often results in increased resolution compared to columns made with polysiloxane polymer or polyethylene glycol columns. The analysis of PAHs is an example of how an ionic liquid column can achieve a level of separation not possible with other columns. In this case, it is the specially tested SLB®-ILPAH.

Multiple applications, product information, real-time availability, and ordering information is available 24 hours a day at SigmaAldrich.com/il-gc

We offer a wide variety of single-component and multi-component PAH mixtures available as certified reference materials (CRMs). Additionally, we have great custom capabilities. To learn more, visit SigmaAldrich.com/pahstandards

Materials

| Product # | Image | Description |
|-----------|-------|--|
| 29799-U | | SLB®-ILPAH Capillary GC Column L × I.D. 20 m × 0.18 mm, d _r 0.05 µm |