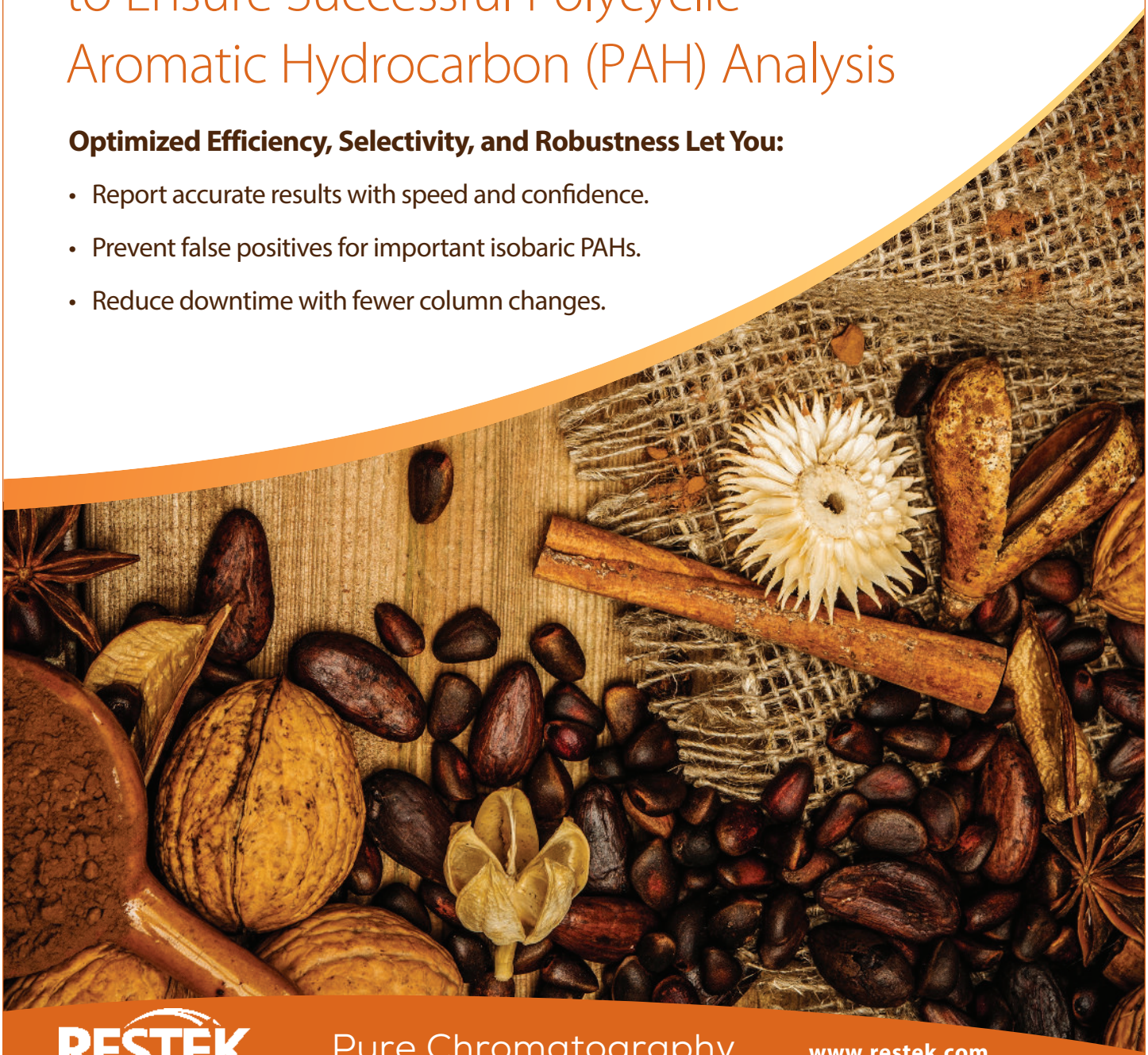
Rxi GCColumns

Rely on Rxi®-PAH Columns

to Ensure Successful Polycyclic Aromatic Hydrocarbon (PAH) Analysis

Optimized Efficiency, Selectivity, and Robustness Let You:

- Report accurate results with speed and confidence.
- Prevent false positives for important isobaric PAHs.
- Reduce downtime with fewer column changes.



RESTEK Pure Chromatography www.restek.com

Rely on Rxi®-PAH Columns to Ensure Successful Polycyclic Aromatic Hydrocarbon (PAH) Analysis

> Food can contain dozens of polycyclic aromatic hydrocarbons (PAHs) and, while research has shown that some are genotoxic and carcinogenic, others are not known to be harmful to human health. This creates one of the leading challenges for food safety laboratories: **how to accurately report toxic PAHs, without high bias or false positives caused by nontoxic PAHs.**

The main difficulty in determining if PAH concentrations exceed maximum levels is that less toxic PAHs coelute with harmful target compounds. Whether these PAH interferences are known and reported together or are unknown and contributing bias, these coelutions increase the risk of safe food being reported as containing PAHs above maximum levels. While mass spectrometry (MS) often can resolve compounds of interest from coeluting interferences, in PAH analysis there are isobaric interferences that are indistinguishable by MS. Because the EFSA PAH4 group [1], as well as other frequently analyzed PAH lists, includes isobars that must be separated chromatographically, column choice is an essential consideration.

The Rxi®-PAH column from Restek is designed specifically for comprehensive PAH analysis in food and is the best column on the market today for these applications. Column dimensions were chosen to maximize efficiency and the selectivity of the proprietary stationary phase has been optimized to maximize resolution between critical pairs. In addition, the stabilized, bonded, arylene phase provides high temperature stability and excellent robustness. This combination of efficiency, selectivity, and robustness makes the Rxi®-PAH column the best choice for successful PAH analysis.

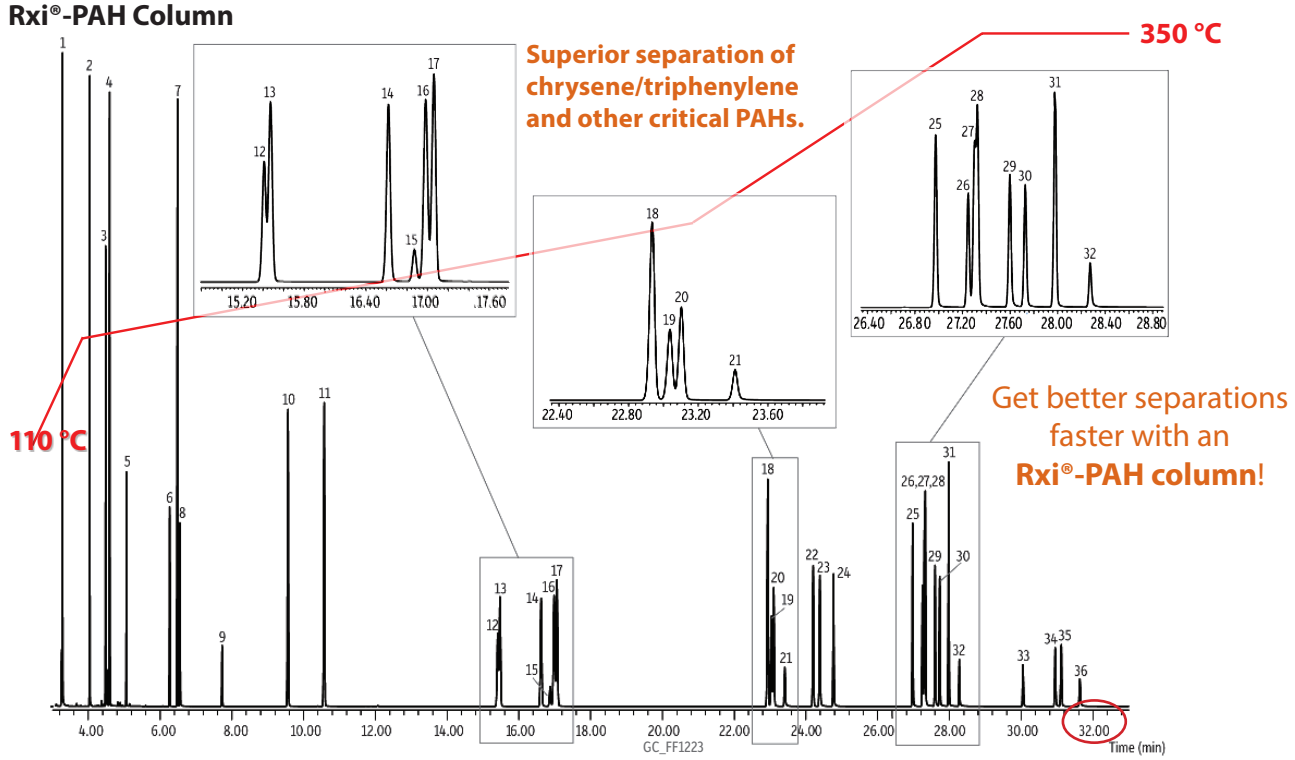
Generate Data With Greater Confidence and Speed Using an Rxi®-PAH Column

The optimized separating power of the Rxi®-PAH column provides the best performance for PAH analysis. Compared to other columns, the Rxi®-PAH column provides good separations faster and in a more straightforward analysis. Figure 1 contrasts the performance of an Rxi®-PAH column (40 m x 0.18 mm x 0.07 µm) and a competitor's PAH column (30 m x 0.25 mm x 0.15 µm) in an analysis optimized for EFSA PAH4 compounds. Although the competitor column is 10 m shorter than the Rxi®-PAH column, a ~30% faster analysis is achieved on the Restek® column with a simpler oven program (red overlay). This optimization is possible because the enhanced efficiency and selectivity of the Rxi®-PAH column allows for resolution of critical compounds, including the benzo [b], [k], and [j] fluoranthenes, while still eluting the heavier PAHs in a short analysis time. Because thinner films bleed less, there is very little interference from column bleed, making the Rxi®-PAH column an excellent choice for the low-level MS analyses of PAHs in food.

[1] Scientific Opinion of the Panel on Contaminants in the Food Chain on a request from the European Commission on Polycyclic Aromatic Hydrocarbons in Food, The EFSA Journal 724 (2008) 1.

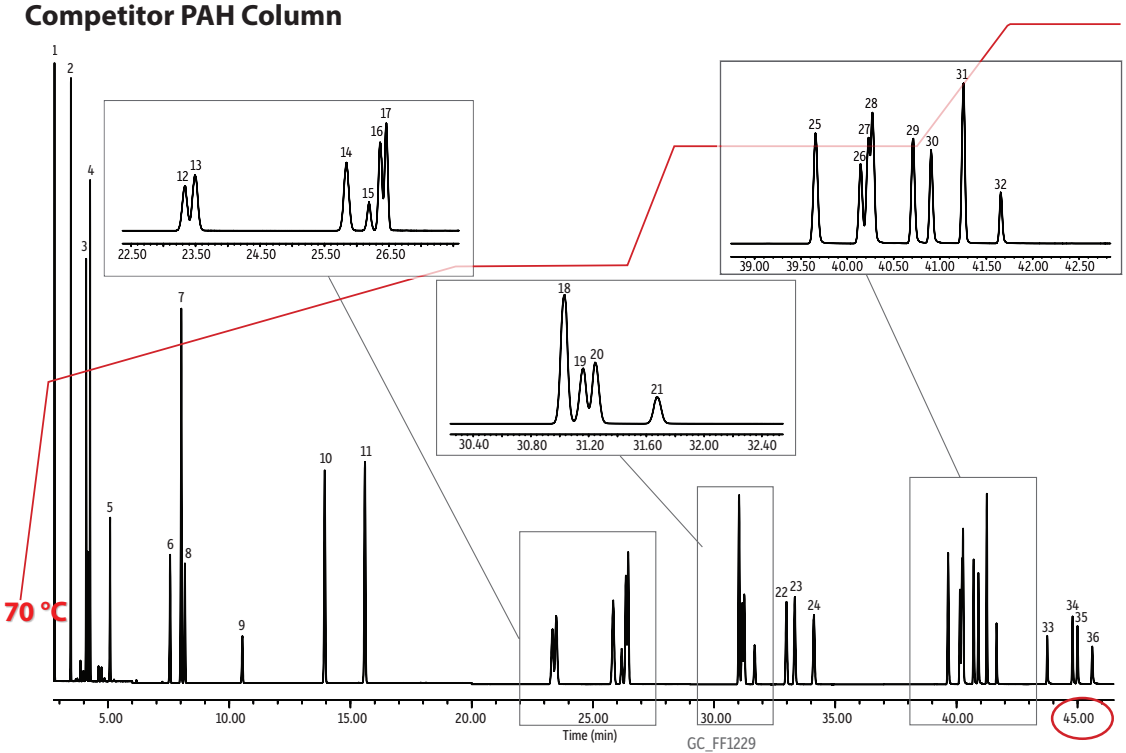
Figure 1: A faster and more straightforward analysis can be achieved with an Rxi®-PAH column due to its higher efficiency and optimized selectivity.

Rxi®-PAH Column



Column: Rxi®-PAH, 40 m, 0.18 mm ID, 0.07 µm (cat.# 49316); **Sample:** NIST SRM 2260a PAH mix; Diluent: Toluene; Conc.: 0.2 - 2 µg/mL (SRM 2260a PAH mix was diluted 5x in toluene); **Injection**: Inj. Vol.: 0.5 µL pulsed splitless (hold 0.58 min); Liner: Restek Premium 2 mm single taper w/wool (cat.# 23316.1); Inj. Temp.: 275 °C; Pulse Pressure: 80 psi (551.6 kPa); Pulse Time: 0.6 min; Purge Flow: 40 mL/min; **Oven:** Oven Temp.: 110 °C (hold 1 min) to 210 °C at 37 °C/min to 260 °C at 3 °C/min to 350 °C at 11 °C/min (hold 4.5 min); **Carrier Gas:** He, constant flow; Flow Rate: 1.4 mL/min; **Detector:** MS; Mode: SIM. For complete instrument conditions, visit www.restek.com and enter GC_FF1223 in the search. **Red line** = oven temperature program.

Competitor PAH Column



Peaks
1. Naphthalene
2. Biphenyl
3. Acenaphthylene
4. Acenaphthene
5. Fluorene
6. Dibenzothiophene
7. Phenanthrene
8. Anthracene
9. 4H-Cyclopenta[def] phenanthrene
10. Fluoranthene
11. Pyrene
12. Benzo[ghi]fluoranthene
13. Benzo[c]phenanthrene
14. Benz[a]anthracene
15. Cyclopenta[cd]pyrene
16. Triphenylene
17. Chrysene
18. Benzo[b]fluoranthene
19. Benzo[k]fluoranthene
20. Benzo[j]fluoranthene
21. Benzo[a]fluoranthene
22. Benzo[e]pyrene
23. Benzo[a]pyrene
24. Perylene
25. Dibenz[a,j]anthracene
26. Dibenz[a,c]anthracene
27. Indeno[1,2,3-cd]pyrene
28. Dibenz[a,h]anthracene
29. Benzo[b]chrysene
30. Picene
31. Benzo[ghi]perylene
32. Anthanthrene
33. Dibenzo[b,k]fluoranthene
34. Dibenzo[a,e]pyrene
35. Coronene
36. Dibenzo[a,h]pyrene

Conditions based on column manufacturer's recommendations. **Column:** Vendor A PAH Column, 30 m, 0.25 mm ID, 0.15 µm; **Injection:** Inj. Vol.: 1.0 µL splitless (hold 1.0 min); **Oven:** Oven Temp.: 70 °C (hold 0.7 min) to 180 °C at 85 °C/min to 230 °C at 3 °C/min (hold 7 min) to 280 °C at 28 °C/min (hold 10 min) to 350 °C at 14 °C/min (hold 4 min); **Carrier Gas:** He, constant flow; Flow Rate: 2.0 mL/min; **Detector:** MS. For complete instrument conditions, visit www.restek.com and enter GC_FF1229 in the search.

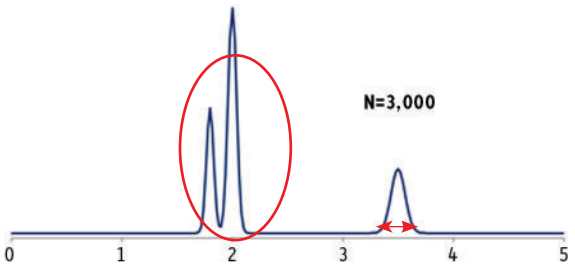
Get Better Separations of Key Food PAHs and Interferences With a Higher Efficiency Column

Maximum efficiency is absolutely required for any analysis involving many potentially isobaric interferences that elute closely with compounds of interest, as is the case with PAH analysis. Efficiency is a function of peak width, with more efficient columns producing narrower peaks. Narrower peaks are also taller peaks, which means that not only is resolution between closely eluting compounds increased, but sensitivity is also improved. As shown in Figure 2, columns with higher efficiency give much better separation of compounds independent of stationary phase selectivity, and they also provide improved peak responses, which is critical for trace analyses like PAHs in food.

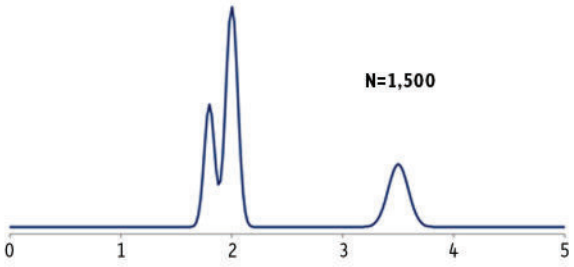
Figure 2: High efficiency helps to separate compounds independent of column selectivity. High efficiency Rxi®-PAH columns provide better separations and improved peak responses, which are required for successful PAH analysis.

A. Higher efficiency separation

Higher efficiency columns give tall, narrow peaks and better separations and responses



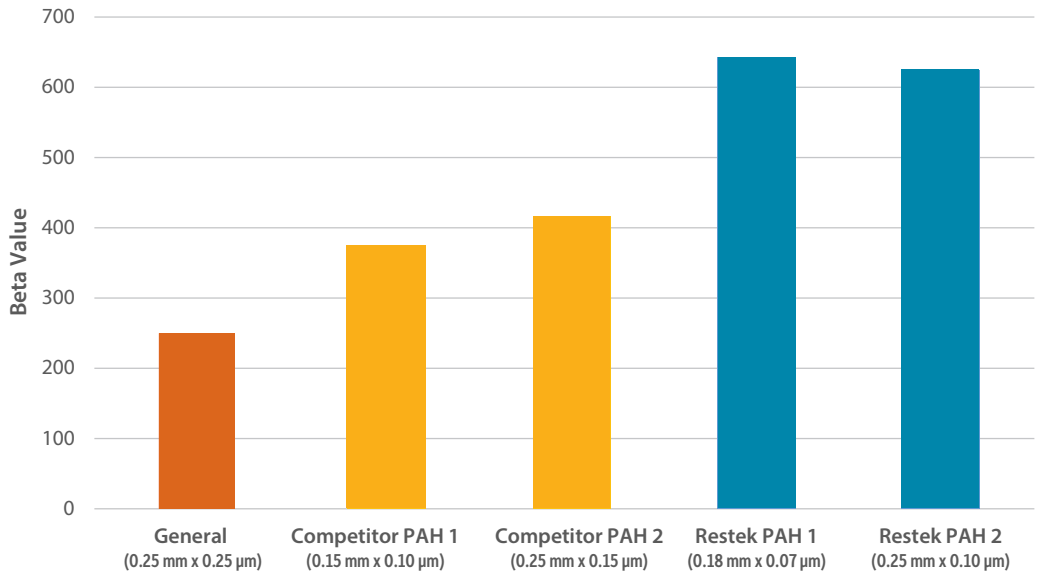
B. Lower efficiency separation



Restek's Rxi®-PAH columns were developed with stabilized thin films that are specifically optimized to increase efficiency. Thinner films result in more efficient columns because thin films have a higher rate of mass transfer than thicker films. Beta value, or phase ratio, is related to efficiency and is based on the ratio of column ID to film thickness. Thinner film columns have higher beta values, and thus higher efficiency. Restek developed the Rxi®-PAH column with a very thin film, while preserving the robustness required for difficult matrices (see robustness data on p. 7). The beta values for all configurations are > 600, which is significantly higher than PAH columns from other manufacturers and also, for reference, higher than general-purpose columns with a 0.25 mm ID and 0.25 µm film thickness (Figure 3).

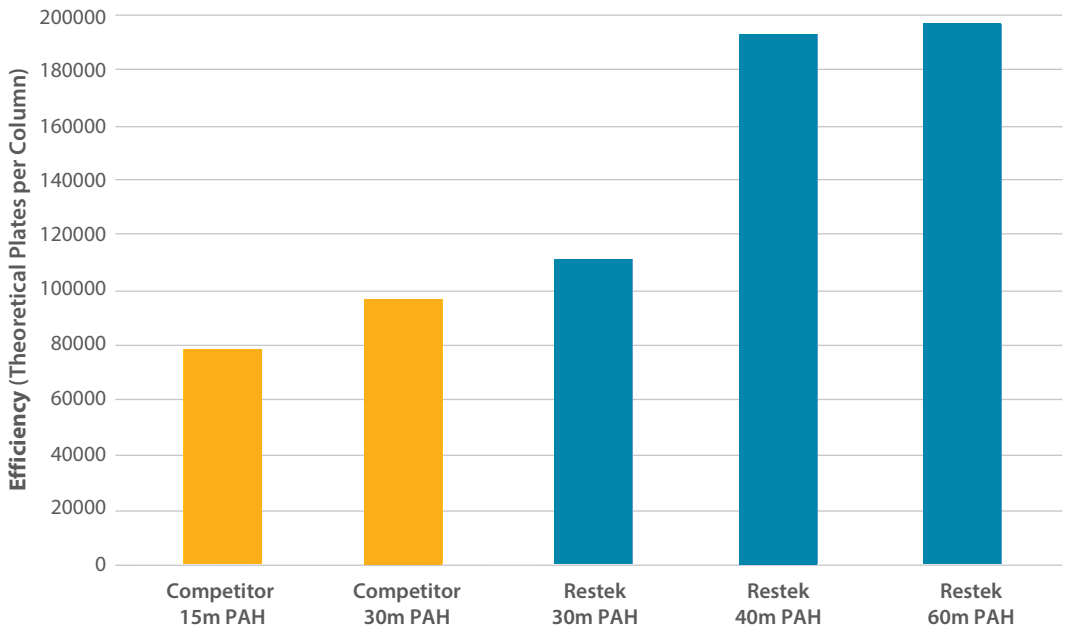
Rxi®-PAH columns are the highest efficiency PAH column available, and higher efficiency ensures better resolution of critical compounds.

Figure 3: Restek's Rxi®-PAH columns have higher beta values than competitor PAH columns for maximum efficiency.



Column efficiency can be measured using theoretical plate number, which is directly proportional to peak width. Because of their optimized film thicknesses and beta values, Restek® Rxi®-PAH columns have between 14,000 and 98,000 *more* theoretical plates per column than competitor columns, allowing for separations of critical closely-eluting compounds (Figure 4).

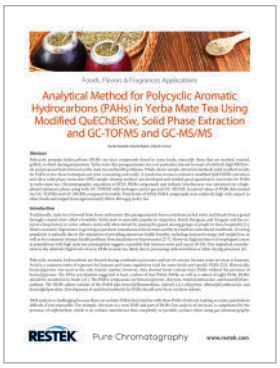
Figure 4: Restek's Rxi®-PAH columns have more theoretical plates than competitor PAH columns, which results in better separations.



For reference, a typical midpolarity column has 105,000 plates per column.

Optimized Rxi®-PAH Selectivity Separates all Priority EFSA PAH4 PAHs From Interferences

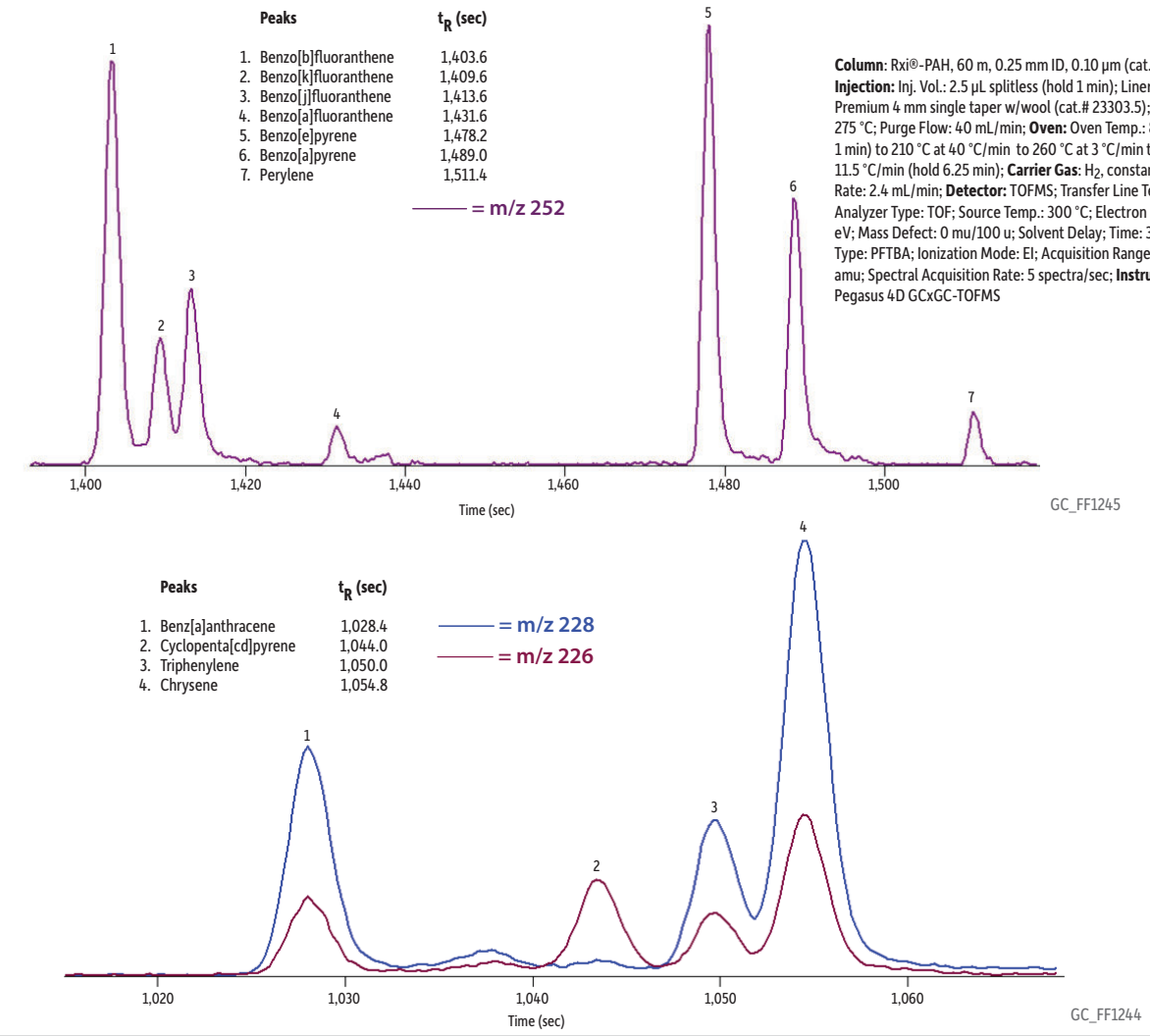
In addition to maximum efficiency, optimized selectivity is necessary for successful PAH analysis, especially when separating EFSA PAH4 compounds chrysene and benzo[b]fluoranthene from interfering isobaric PAHs. While some columns separate one of these key PAHs, no column separates both chrysene and benzo[b]fluoranthene from their interferences better than the Rxi®-PAH column. For example, low phenyl phases (e.g., 5-type columns) will resolve triphenylene and chrysene, but will not resolve benzo[b] fluoranthene and benzo[j]fluoranthene. Conversely, with higher phenyl content phases (e.g., 17-type columns), good separation can be achieved between benzo[b] fluoranthene and benzo[j]fluoranthene, but the triphenylene/chrysene separation is severely compromised. In contrast, the Rxi®-PAH column proves excellent separation of both triphenylene and chrysene, as well as all the benzo fluoranthenes. As shown in Figure 5, this column provides the outstanding separations needed for accurate quantification, even in complex matrices such as tea. With its optimized selectivity, reliable results can be obtained quickly and accurately using an Rxi®-PAH column and method, even in difficult food matrices that contain many PAH interferences.



Get Our Method

Download Restek's full application note on analyzing PAHs in tea. Visit **www.restek.com** and enter FFAN2086-UNV in the search.

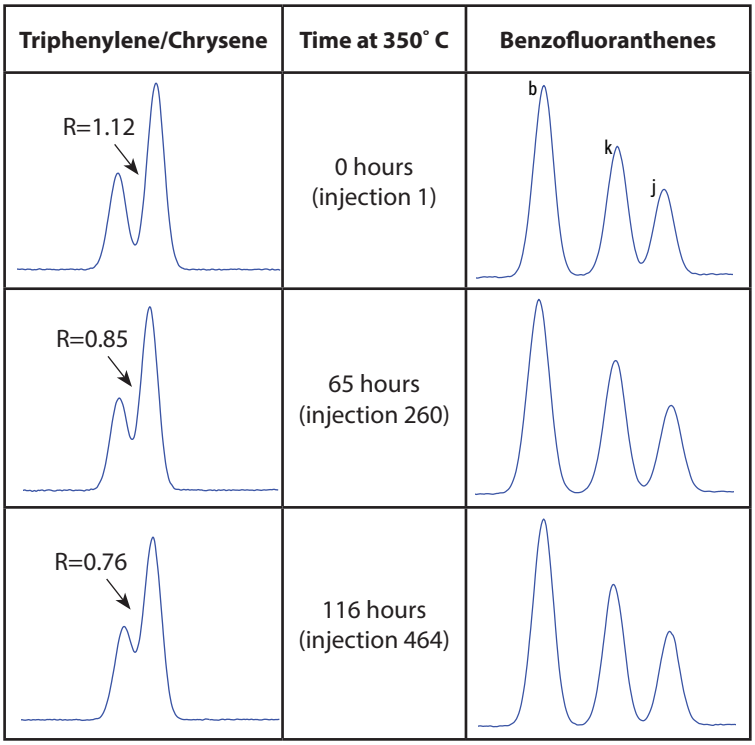
Figure 5: Excellent separation of important PAHs from interferences on an Rxi®-PAH column ensures accurate reporting.



Reduce Downtime With Fewer Column Changes—Robust Rxi®-PAH Columns Withstand High Temperatures and Harsh Matrices

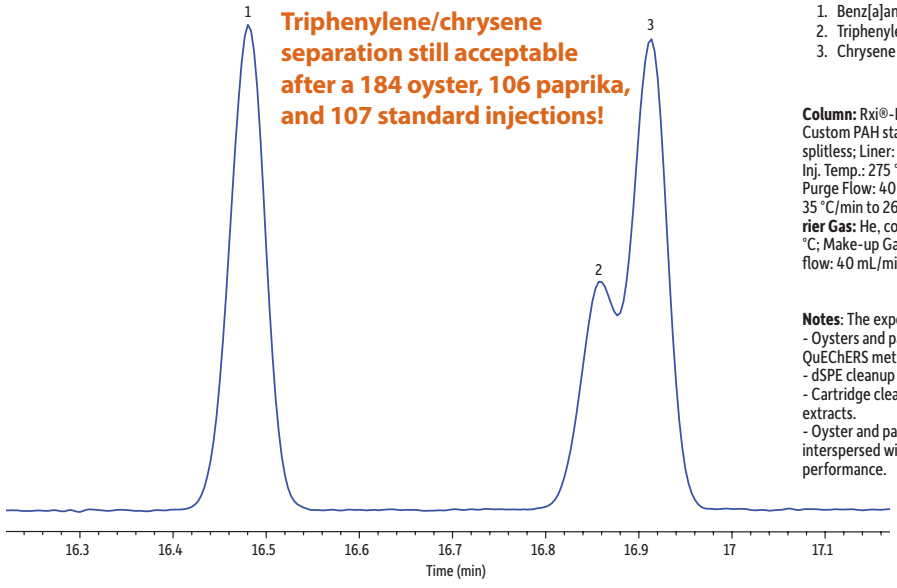
While thin film columns are very efficient, they may be less robust than thicker film columns under some conditions. In contrast, the Rxi®-PAH column can withstand the high temperatures necessary to elute the heavier (e.g., 302 m/z) PAHs due to the robustness of its bonded, arylene-stabilized stationary phase. To demonstrate this, repeated injections of PAH standard were performed, with a final isothermal hold of 4.5 minutes at 350 °C. Figure 6 shows that acceptable resolution between triphenylene/chrysene and the benzo fluoranthenes was maintained even after 464 injections and a total of 116 hours at 350 °C. In fact, after 464 injections, the Rxi®-PAH column is just beginning to approach the competitor's new, out-of-the-box resolution specification for triphenylene/chrysene. These columns are also robust enough to withstand repeated injections of dirty sample matrix. As shown in Figure 7, Rxi®-PAH columns maintain excellent performance for the triphenylene/chrysene separation, even after 184 injections of oyster extract and 106 injections of paprika extract (Figure 7).

Figure 6: Even after extended time at high temperatures, Rxi®-PAH columns provide excellent separation of key PAHs, meaning less time and expense is lost to column replacement and maintenance.



Column: Rxi®-PAH, 40 m, 0.18 mm ID, 0.07 µm (cat.# 49316); **Sample:** Mixed PAH standard prepared at 10–40 µg/mL from neat materials in toluene; **Injection:** 0.5 µL pulsed splitless (hold 0.58 min); Liner: Restek Premium 2 mm single taper w/wool (cat.# 23316.1); Inj. Temp.: 275 °C; Pulse Pressure: 80 psi (551.6kPa); Pulse Time: 0.6 min; Purge Flow: 40 mL/min; **Oven Temp:** 110 °C (hold 1 min) to 210 °C at 35 °C/min to 260 °C at 3 °C/min to 350 °C at 10 °C/min (hold 15 min); Carrier Gas: He, constant flow; Flow Rate: 1.4 mL/min; **Detector:** FID @ 360 °C.

Figure 7: Good separation of triphenylene and chrysene is maintained even after repeated injections of dirty extracts.



Peaks
1. Benz[a]anthracene
2. Triphenylene
3. Chrysene

Column: Rxi®-PAH, 40 m, 0.18 mm ID, 0.07 µm (cat.# 49316); **Sample:** Custom PAH standard; Diluent:Toluene; **Injection:** Inj Vol.: 0.5 µL pulsed splitless; Liner: Restek Premium 2 mm single taper w/wool (cat.# 23316.1); Inj. Temp.: 275 °C; Pulse Pressure: 80 psi (551.6kPa); Pulse Time: 0.6 min; Purge Flow: 40 mL/min; **Oven:** Oven Temp.: 110 °C (hold 1 min) to 210 °C at 35 °C/min to 260 °C at 3 °C/min to 350 °C at 10 °C/min (hold 4.5 min); **Carrier Gas:** He, constant flow; Flow Rate: 1.4 mL/min; **Detector:** FID @ 360 °C; Make-up Gas; Flow Rate: 45 mL/min; Make-up Gas Type: N_2; Hydrogen flow: 40 mL/min; Air flow: 450 mL/min; **Instrument**: Agilent 7890A GC

Notes: The experiment was carried out as follows:
- Oysters and paprika were extracted using QuEChERS methodology.
- dSPE cleanup was performed on oyster extracts.
- Cartridge cleanup was performed on paprika extracts.
- Oyster and paprika extracts were injected, interspersed with solvent standard, to monitor performance.

GC_FS0543

Ensure Successful PAH Analysis with the Rxi®-PAH Column

Whether you are analyzing comprehensive lists or focusing on the EFSA PAH4, the Rxi®-PAH column ensures your success by combining efficiency, selectivity, and robustness to produce a column that outperforms all competitors' columns. In addition, every Rxi®-PAH column is tested to ensure critical separations—your guarantee of consistent column performance. Chose the configuration that best suits your application and be confident that you are reporting accurate PAH results without bias from interferences.

Cat.#	Length	ID	df	Description
49316	40 m	0.18 mm	0.07 µm	Narrow inside diameter, thinner film, faster analysis, excellent separation of important PAHs, less sample loading capacity
49317	60 m	0.25 mm	0.10 µm	0.25 mm inner diameter, better sample loading capacity, highest resolution of important PAHs, longer analysis than 0.18 mm column, thin film allows elution of dibenzo pyrenes
49318	30 m	0.25 mm	0.10 µm	0.25 mm inside diameter, better sample loading capacity, faster analysis time than 60 m column, adequate resolution of important PAHs, lower cost column

Pair an Rxi®-PAH Column with Certified Reference Standards

EU 15+1 PAH Standard (16 components)

Benz(a)anthracene (56-55-3)
Benzo(a)pyrene (50-32-8)
Benzo(b)fluoranthene (205-99-2)
Benzo(c)fluorene (3,4-Benzofluorene) (205-12-9)
Benzo(g,h,i)perylene (191-24-2)
Benzo(j)fluoranthene (205-82-3)
Benzo(k)fluoranthene (207-08-9)
Chrysene (218-01-9)
Cyclopenta(c,d)pyrene (27208-37-3)
Dibenz(a,e)pyrene (192-65-4)
Dibenz(a,h)anthracene (53-70-3)
Dibenzo(a,h)pyrene (189-64-0)
Dibenzo(a,i)pyrene (189-55-9)
Dibenzo(a,l)pyrene (191-30-0)
Indeno(1,2,3-cd)pyrene (193-39-5)
5-Methylchrysene (3697-24-3)

100 µg/mL each in toluene, 1 mL/ampul
cat.# 32470 (ea.)

PAH Interferences Standard (5 components)

Benzo(j)fluoranthene (205-82-3)	750 µg/mL
Benzo(k)fluoranthene (207-08-9)	1,000
Benzo(e)pyrene (192-97-2)	500
Perylene (198-55-0)	250
Triphenylene (217-59-4)	750

In toluene, 1 mL/ampul
cat.# 32472 (ea.)

EFSA PAH4 Standard (4 components)

Benz[a]anthracene (56-55-3)
Benzo[a]pyrene (50-32-8)
Benzo[b]fluoranthene (205-99-2)
Chrysene (218-01-9)

1,000 µg/mL each in toluene, 1 mL/ampul
cat.# 32469 (ea.)

RESTEK
Pure Chromatography

Questions about this or any other Restek® product?
Contact us or your local Restek® representative (www.restek.com/contact-us).

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Lit. Cat.# FFSS2114-UNV