

Fast Dual-Column Analysis of Pesticides or PAHs

new!
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Using an Rtx®-440 Capillary GC Column

By Greg France, Innovations Chemist, and Gary Stidsen, Innovations Team Manager

- Analyze 20 organochlorine pesticides in less than 9 minutes.
- Analyze 16 PAHs in 22 minutes.
- New low-bleed, high-resolution column is ideal for dual-column analyses.

Assessments for organochlorine pesticides or polynuclear aromatic hydrocarbons (PAHs) are critical, frequently performed GC analyses—and they often are among the most challenging. Issues that can arise include analyte breakdown and poor linearity, and calibration times can be long. In addition to the problems inherent to the analysis, analysts must be concerned

with column reactivity and bleed, which affect sensitivity and reproducibility. In analyses of PAHs, there are critical pairs to resolve and, because the samples often include interfering hydrocarbons, a confirmation column typically is required. Compounding these problems is constant pressure to process more samples in less time.

Figure 1 Separate 20 organochlorine pesticides in 9 minutes, using an Rtx®-440 column.

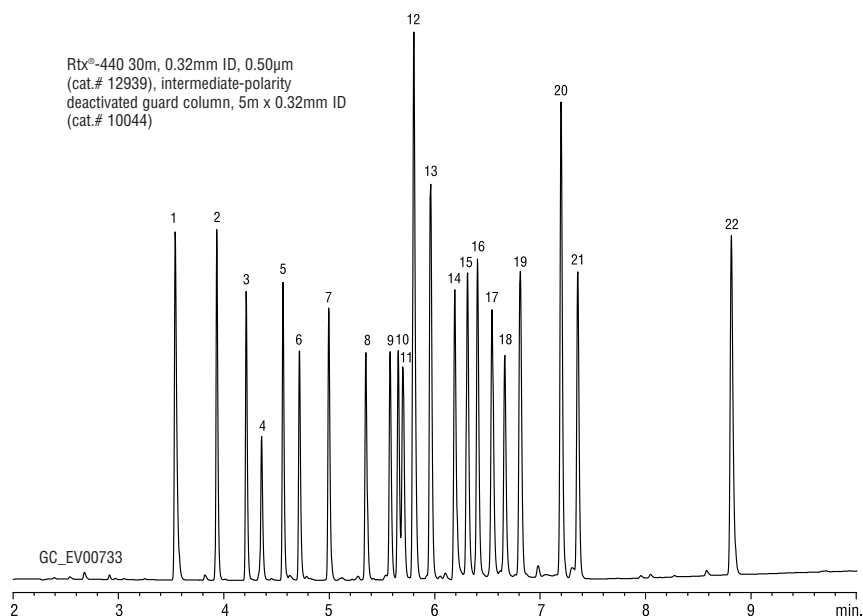
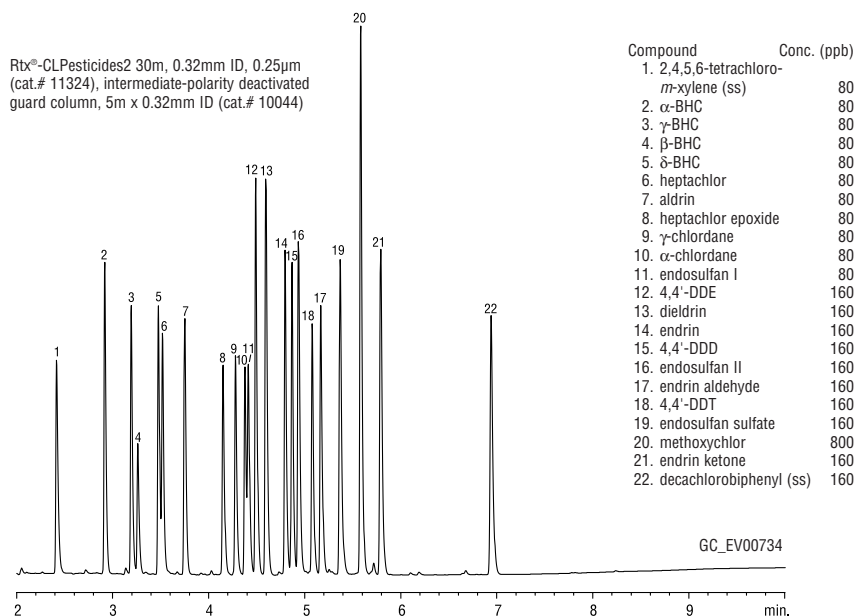


Figure 2 An Rtx®-CLPesticides2 column complements the Rtx®-440 column in dual-column analysis of organochlorine pesticides.



With the new Rtx®-440 column, Restek makes available an excellent choice for both of these demanding applications.

Organochlorine Pesticides: Sub-10-Minute Analyses

Figure 1 shows a separation of 20 commonly analyzed organochlorine pesticides, obtained in less than 10 minutes by using an Rtx®-440 column. Only α-chlordane and endosulfan I (peaks 10 & 11) are not separated to the baseline. The column's excellent thermal stability is indicated by a virtually flat baseline between the initial temperature and the maximum temperature of the program, 330°C. In a dual-column approach to this application, an Rtx®-440 column can be paired with an Rtx®-CLPesticides2 column. The latter column will provide an equally fast separation (Figure 2) and near-equivalent resolution, with a reverse in elution order for endrin aldehyde and 4,4'-DDT (peaks 17 & 18). By connecting the two columns to a "Y" connector and making the sample injection onto a 5-meter intermediate-polarity deactivated guard column, the two analyses can be conducted simultaneously.

Polynuclear Aromatic Hydrocarbons: Baseline Resolution of Critical Pairs

In Figure 3, 16 commonly encountered PAHs have eluted from an Rtx®-440 column in less than 18 minutes. Two critical pairs, phenanthrene/anthracene (peaks 5 and 6) and benzo(a)anthracene/chrysene (peaks 9 and 10), are resolved to baseline, and benzo(b)fluoranthene and benzo(k)fluoranthene (peaks 11 and 12) and indeno(1,2,3-cd)pyrene and dibenzo(a,h)anthracene (peaks 14 and 15) are almost completely separated. Also notice the excellent thermal stability—baseline rise is negligible even at 320°C. Similar results can be obtained by using an Rtx®-5Sil MS column or an Rtx®-CLPesticides2 column and constant flow, as shown in the Applications section of our general catalog. An Rtx®-440 column can be paired with either of these other columns, for a rapid, dual-column/FID analysis of commonly encountered PAHs.

Conditions for Figures 1 and 2

Sample: Organochlorine Pesticide Mix AB #2 (cat.# 32292), 2,4,5,6-tetrachloro-*m*-xylene (ss) (cat.# 32027), decachlorobiphenyl (ss) (cat.# 32029), diluted in hexane, on-column amounts listed on figure

Inj.: 1.0µL splitless (hold 0.75 min.), 4mm Drilled Uniliner® inlet liner (cat.# 21055)

Inj. temp.: 225°C

Carrier gas: hydrogen, constant pressure

Linear velocity: 73cm/sec. (Rtx®-440) or 77cm/sec. @ 140°C (Rtx®-CLPesticides2)

Oven temp.: 140°C (hold 0.5 min.) to 268°C @ 30°C/min., to 290°C @ 11°C/min., to 330°C @ 25°C/min. (hold 5 min.)

Det.: ECD @ 320°C

Conclusion

The new Rtx®-440 column is an excellent addition to the selection of innovative columns available from Restek. The column exhibits high thermal stability and, for organochlorine pesticides, a selectivity alternative to the Rtx®-CLPesticides2 column. An Rtx®-440 column can be paired with an Rtx®-CLPesticides2 column to ensure sub-10-minute analysis times for organochlorine pesticides, or can be used as a confirmation column, with an Rtx®-5Sil MS or an Rtx®-CLPesticides2 column, for GC/FID analysis of PAHs.

Organochlorine Pesticide Mix AB #2

	8µg/mL		
aldrin	8	dieldrin	16
α-BHC	8	endosulfan I	8
β-BHC	8	endosulfan II	16
δ-BHC	8	endosulfan sulfate	16
γ-BHC (lindane)	8	endrin	16
α-chlordane	8	endrin aldehyde	16
γ-chlordane	8	endrin ketone	16
4,4'-DDD	16	heptachlor	8
4,4'-DDE	16	heptachlor epoxide (B)	8
4,4'-DDT	16	methoxychlor	80

Each	5-pk.	10-pk.
In hexane:toluene (1:1), 1mL/ampul		
32292	32292-510	—
	w/data pack	
32292-500	32292-520	32392

2,4,5,6-Tetrachloro-m-xylene

Each	5-pk.	10-pk.
200µg/mL in acetone, 1mL/ampul		
32027	32027-510	—
	w/data pack	
32027-500	32027-520	32127
200µg/mL in acetone, 5mL/ampul		
32028	32028-510	—
	w/data pack	
32028-500	32028-520	32128

Decachlorobiphenyl (BZ #209)

Each	5-pk.	10-pk.
10µg/mL in isooctane, 1L/ampul		
32289	32289-510	—
	w/data pack	
32289-500	32289-520	32389
200µg/mL in acetone, 1mL/ampul		
32029	32029-510	—
	w/data pack	
32029-500	32029-520	32129
200µg/mL in acetone, 5mL/ampul		
32030	32030-510	—
	w/data pack	
32030-500	32030-520	32130

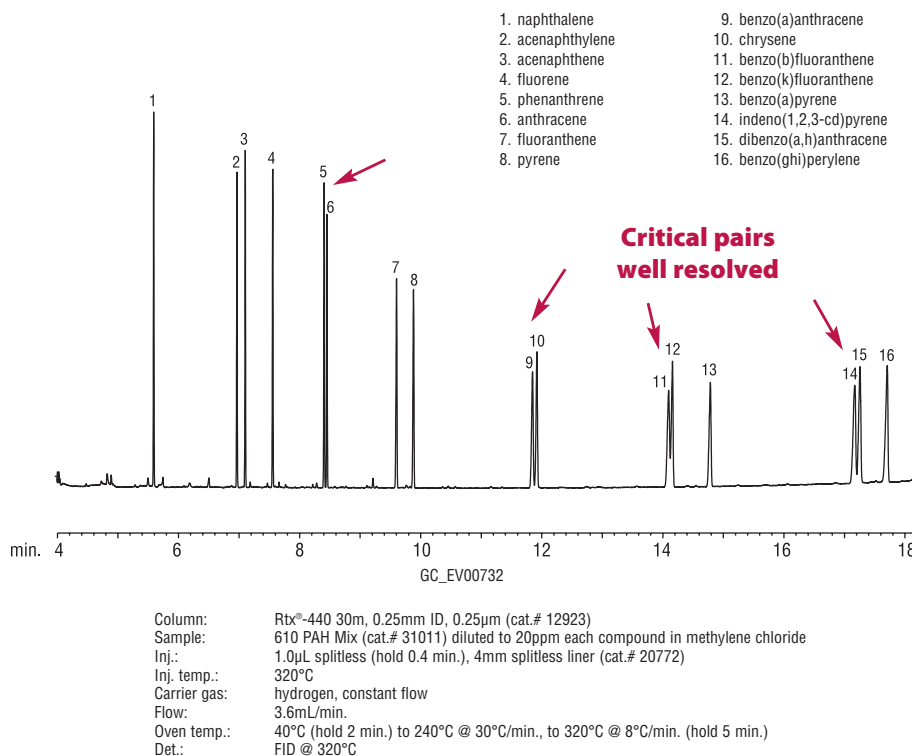
Rtx®-440 Columns (fused silica)

ID	df (µm)	temp. limits	30-Meter
0.25mm	0.25	20°C to 320/340°C	12923
	0.50	20°C to 320/340°C	12938
0.32mm	0.25	20°C to 320/340°C	12924
	0.50	20°C to 320/340°C	12939
0.53mm	0.50	20°C to 320/340°C	12940
	1.00	20°C to 320/340°C	12955

Rtx®-CLPesticides2 Columns (fused silica)

ID	df (µm)	temp. limits	10-Meter	15-Meter	20-Meter	30-Meter	60-Meter
0.10mm	0.10	-60 to 310/330°C	43301		43302		
0.18mm	0.14	-60 to 310/330°C	42301		42302		
0.25mm	0.20	-60 to 320/340°C		11320		11323	11326
0.32mm	0.25	-60 to 320/340°C		11321		11324	
0.53mm	0.42	-60 to 300/320°C		11337		11340	

Figure 3 Analyze 16 PAHs in 22 minutes, and resolve critical pairs, with an Rtx®-440 column.



Searching for a chromatogram?
www.restek.com

SV Calibration Mix #5 / 610 PAH Mix

Each	5-pk.	10-pk.
acenaphthene	chrysene	
acenaphthylene	dibenzo(a,h)anthracene	
anthracene	fluoranthene	
benzo(a)anthracene	fluorene	
benzo(a)pyrene	indeno(1,2,3-cd)pyrene	
benzo(b)fluoranthene	naphthalene	
benzo(k)fluoranthene	phenanthrene	
benzo(ghi)perylene	pyrene	

Each	5-pk.	10-pk.
2,000µg/mL each in methylene chloride, 1mL/ampul		
31011	31011-510	—
	w/data pack	
31011-500	31011-520	31111

SeCure™ "Y" Connectors

- Use standard "Y" Press-Tight® connectors and 1/16" graphite ferrules.
- Reliable seal integrity, will not unexpectedly disconnect during temperature-programmed analyses.
- Open design allows visual confirmation of the seal for added confidence in the connection.



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Kits include: SeCure™ "Y" connector body, 3 knurled nuts, 1 "Y" Universal Press-Tight® union, 3 ferrules

Description	Ferrules Fit Column ID (mm)	qty.	cat.#
SeCure™ "Y" Connector Kit	0.25/0.28	kit	20276
SeCure™ "Y" Connector Kit	0.28/0.32	kit	20277
SeCure™ "Y" Connector Kit	0.45/0.53	kit	20278
Knurled nut		3-pk.	20279