RESTEK environmental

Applications note

Higher Responses for Chlorinated Pesticides, Using a Drilled Uniliner® GC Inlet Liner and Rtx®-CLPesticides Columns

Inlet reactivity is the primary drawback to using hot flash injection when analyzing chlorinated pesticides by GC. Residues of heavier and non-volatile materials often build up on the inner surface of the injection port, leaving a reactive surface that can cause compounds such as endrin and DDT to break down. An inlet liner that makes a press-fit connection with the analytical column prevents analytes from coming in contact with a major portion of this reactive surface—the bottom of the injection port. The physical connection between the column and the liner also improves the accuracy of the analysis, by minimizing injection port discrimination.

Used together, a Drilled Uniliner® inlet liner (Figure 1), an Rtx®-CLPesticides column, and an Rtx®-CLPesticides2 column ensure excellent responses in analyses of chlorinated pesticides. Markedly better results are apparent from comparisons of calibration data obtained by using a Drilled Uniliner® inlet liner and an Rtx®-CLPesticides/Rtx®-CLPesticides2 column pair to data obtained by using a conventional splitless inlet liner, or by using a conventional splitless inlet liner packed with fused silica wool.

Figure 1—The drilled hole in a Uniliner® injection port liner makes direct injection possible with EPC systems by equalizing pressure in the injection port.

Table 1—RSD values for chlorinated pesticides are well within acceptable limits (20%) when the inlet is fitted with a Drilled Uniliner® inlet liner.

Analyte	% RSD Rtx®-CLPesticides Column	%RSD Rtx®-CLPesticides2 Column
TCX*	2.3	2.0
α-BHC	7.8	9.5
γ-BHC	6.3	5.1
β-BHC	1.5	2.5
δ-BHC	8.5	3.3
heptachlor	3.2	11.6
aldrin	2.2	5.6
heptachlor epoxide	4.4	1.2
γ-chlordane	2.5	1.4
α -chlordane	3.8	2.9
4,4'-DDE	2.6	5.0
endosulfan I	3.2	2.8
dieldrin	1.0	2.1
endrin	1.7	3.5
4,4'-DDD	4.9	3.9
endosulfan II	3.5	2.8
4,4'-DDT	2.1	1.5
endrin aldehyde	4.8	4.8
methoxychlor	12.5	14.2
endosulfan sulfate	3.4	2.5
endrin ketone	1.9	3.3
DCB**	6.7	6.5

*2,4,5,6-tetrachloro-m-xylene
**decachlorobiphenyl
Columns and conditions listed in Figure 2.

Laboratories following US EPA Method 8081 and 8000 must document the quantification results they generate, to ensure reliability, precision, and accuracy. Beginning with a five-point calibration curve, a calibration factor must be calculated for each analyte. The relative standard deviation (RSD) for each analyte should be no more than 20%. In evaluations made using a Drilled Uniliner® inlet liner, RSD values were between 1.0 and 12.5% for an Rtx®-CLPesticides column and between 1.2 and 14.2% for an Rtx®-CLPesticides2 column (Table 1).

Analysts following Method 8081 and 8000 also must regularly analyze and quantify a calibration standard. In the analysis, the concentration of each analyte in the calibration standard should be within ±15% of the "true" value. The accuracy of data obtained from a system that included a Drilled Uniliner® inlet liner was tested with a pesticide standard mix at the 20/40/200ng/mL concentration level (Figure 2). The percent difference from the "true" value for each analyte ranged from 0 to a maximum of only 6.2%, well within the acceptable limit. Table 2 summarizes the data.

Because some analytes readily break down as the injection port inlet becomes contaminated (e.g., endrin and DDT in this analysis), a performance evaluation mix must be analyzed and breakdown for each analyte calculated. Breakdown should not exceed 15%. The Drilled Uniliner® inlet liner reduced endrin and DDT breakdown, relative to the splitless or splitless/wool-packed liners (Table 3), because the latter liners allow the analytes to contact more of the reactive surface in the inlet. Wool packing in the splitless liner makes this problem worse; it greatly increases the surface area within the liner and introduces additional activity.

In addition to reducing variability and increasing the accuracy of calibration data, a Drilled Uniliner® inlet liner increases overall response for individual analytes, enhancing minimum detection levels compared to splitless or splitless/wool-packed inlet liners. This is most apparent from the area counts for the last eluting peak, decachlorobiphenyl, which were greater by 18–39%, relative to area counts for injections made on the splitless liners (Table 3). Finally, a Drilled Uniliner® inlet liner ensures greater sensitivity, because less of the injected sample remains in the inlet when the inlet is swept clean to prepare it for the next sample.

By eliminating the bottom of the injector from the sample pathway, a Drilled Uniliner® inlet liner makes the pathway more inert. This reduces breakdown of labile analytes, such as endrin and DDT, and increases accuracy and precision. For analysts using hot flash injection techniques in analyses of chlorinated pesticides, or other labile analytes, these results clearly show that a Drilled Uniliner® inlet liner is the liner of choice.

Table 2—Measured analyte concentrations are very near true values, using a Drilled Uniliner® inlet liner.

Analyte	True Value	Rtx®-CLPesticides Column Measured Value	Rtx®-CLPesticides Column % Difference*	Rtx®-CLPesticides2 Column Measured Value	Rtx®-CLPesticides2 Column % Difference*
TCX	20.0	20.4	2.0	20.4	2.0
α-BHC	20.0	20.0	0	20.5	2.5
γ-BHC	20.0	20.2	1.0	20.4	2.0
β-BHC	20.0	20.3	1.5	20.5	2.5
δ-BHC	20.0	20.0	0	20.7	3.5
heptachlor	20.0	20.3	1.5	19.0	5.0
aldrin	20.0	20.2	1.0	20.3	1.5
heptachlor epoxide	20.0	20.5	2.5	20.4	2.0
γ-chlordane	20.0	20.3	1.5	20.4	2.0
α-chlordane	20.0	20.4	2.0	20.6	3.0
4,4'-DDE	40.0	40.6	1.5	40.4	1.0
endosulfan I	20.0	20.2	1.0	20.7	3.5
dieldrin	40.0	40.7	1.8	41.5	3.8
endrin	40.0	40.6	1.5	42.4	6.0
4,4'-DDD	40.0	41.3	3.2	42.5	6.2
endosulfan II	40.0	40.6	1.5	42.2	5.5
4,4'-DDT	40.0	40.4	1.0	41.1	2.8
endrin aldehyde	40.0	41.0	2.5	40.9	2.2
methoxychlor	200.0	203.1	1.5	206.2	3.1
endosulfan sulfate	40.0	40.8	2.0	40.3	0.8
endrin ketone	40.0	41.2	3.0	41.6	4.0
DCB	20.0	20.7	3.5	20.6	3.0

*Allowed maximum = 15%. Columns and conditions listed in Figure 2.

Table 3—Lowest analyte breakdown, and highest responses, are obtained by using a Drilled Uniliner® inlet liner.

% Breakdown*

Analyte	Column	Drilled Uniliner®	4mm splitless	4mm splitless with wool
Endrin	Rtx®-CLPesticides	4.4	4.7	9.8
	Rtx®-CLPesticides2	4.9	6.9	8.3
DDT	Rtx®-CLPesticides	0.2	0.3	2.6
	Rtx®-CLPesticides2	0.3	0.9	3.1

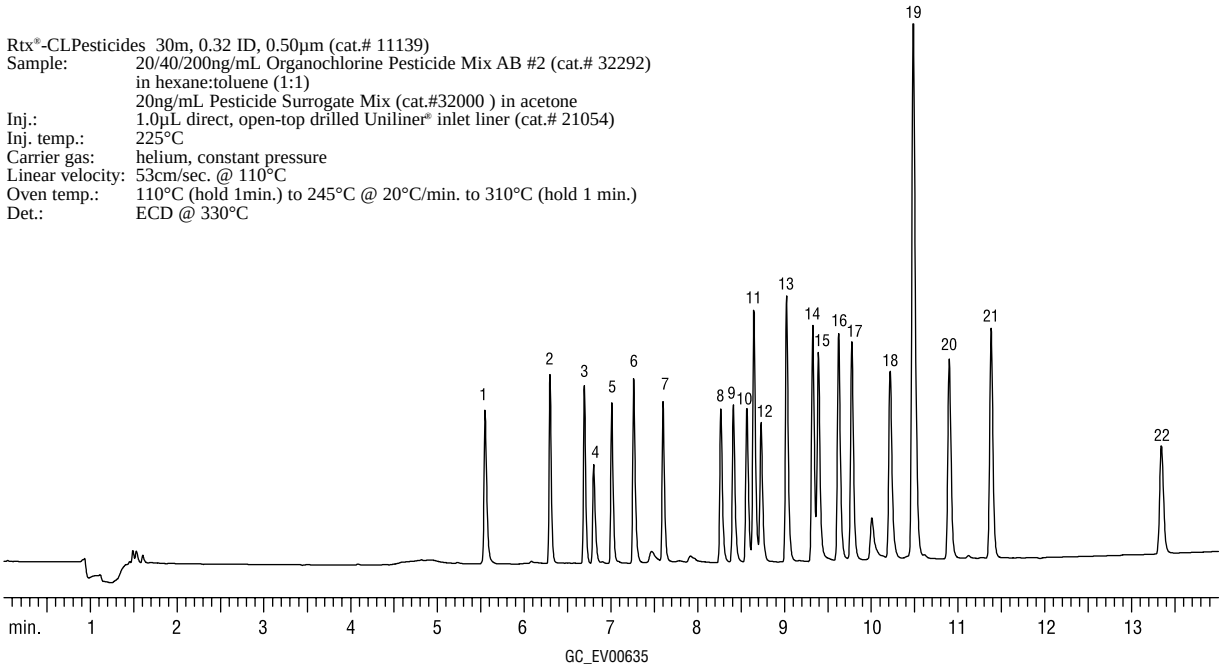
Area Response**

Analyte	Column	Drilled Uniliner®	4mm splitless with wool	4mm splitless
Tetrachloro-*m*-xylene (TCX)	Rtx®-CLPesticides	147	111	106
	Rtx®-CLPesticides2	191	167	162
Decachlorobiphenyl (DCB)	Rtx®-CLPesticides	150	119	108
	Rtx®-CLPesticides2	209	177	166

*Allowed maximum = 15%.
**Mean response (n=2); value in table x 10^3 = response units.
Columns and conditions listed in Figure 2, except:
Inj.: direct, open-top Drilled Uniliner® inlet liner (cat.# 21054)
splitless (hold 1 min.), 4mm single gooseneck inlet liner (cat.# 20798)
splitless (hold 1 min.), 4mm single gooseneck inlet liner w/ fused silica wool (cat.# 22405)

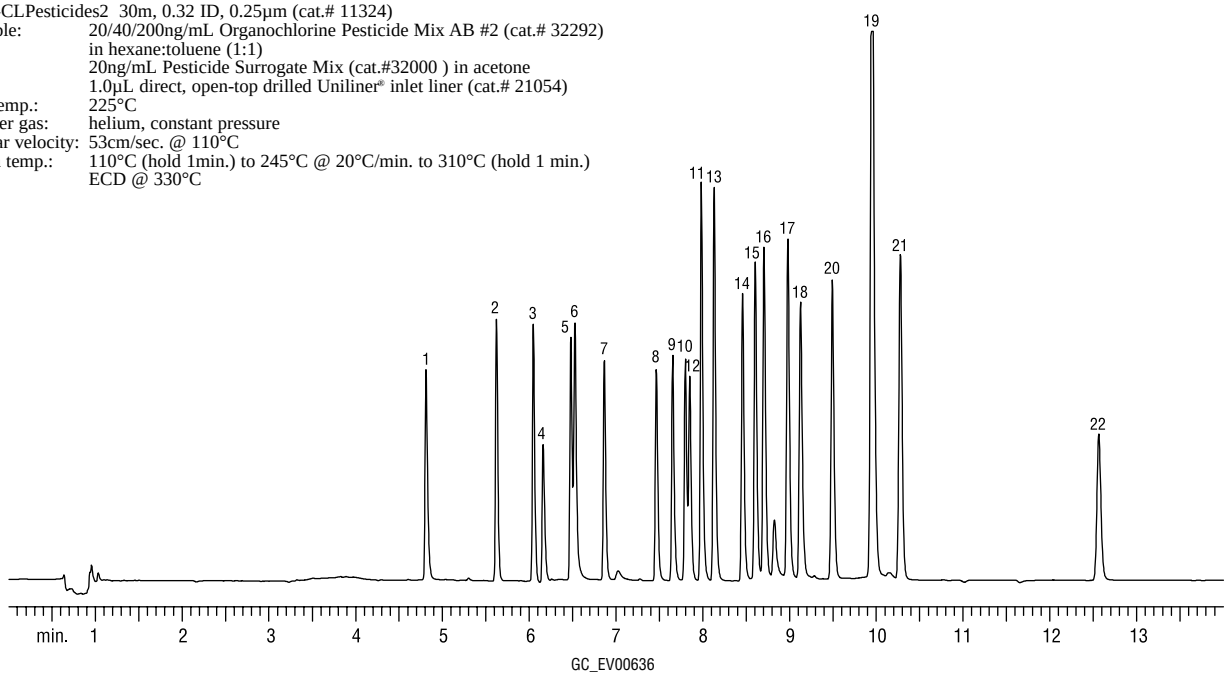
Figure 2—Chlorinated pesticides show excellent response, using the Drilled Uniliner® inlet liner and Rtx®-CLPesticides columns—even at the 20/40/200ng/mL concentration level.

Rtx®-CLPesticides 30m, 0.32 ID, 0.50µm (cat.# 11139)
Sample: 20/40/200ng/mL Organochlorine Pesticide Mix AB #2 (cat.# 32292)
in hexane:toluene (1:1)
20ng/mL Pesticide Surrogate Mix (cat.#32000) in acetone
Inj.: 1.0µL direct, open-top drilled Uniliner® inlet liner (cat.# 21054)
Inj. temp.: 225°C
Carrier gas: helium, constant pressure
Linear velocity: 53cm/sec. @ 110°C
Oven temp.: 110°C (hold 1min.) to 245°C @ 20°C/min. to 310°C (hold 1 min.)
Det.: ECD @ 330°C



1. 2,4,5,6-tetrachloro-*m*-xylene (TCX)
2. α-BHC
3. γ-BHC
4. β-BHC
5. δ-BHC
6. heptachlor
7. aldrin
8. heptachlor epoxide
9. γ-chlordane
10. α-chlordane
11. 4,4'-DDE
12. endosulfan I
13. dieldrin
14. endrin
15. 4,4'-DDD
16. endosulfan II
17. 4,4'-DDT
18. endrin aldehyde
19. methoxychlor
20. endosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl (DCB)

Rtx®-CLPesticides2 30m, 0.32 ID, 0.25µm (cat.# 11324)
Sample: 20/40/200ng/mL Organochlorine Pesticide Mix AB #2 (cat.# 32292)
in hexane:toluene (1:1)
20ng/mL Pesticide Surrogate Mix (cat.#32000) in acetone
Inj.: 1.0µL direct, open-top drilled Uniliner® inlet liner (cat.# 21054)
Inj. temp.: 225°C
Carrier gas: helium, constant pressure
Linear velocity: 53cm/sec. @ 110°C
Oven temp.: 110°C (hold 1min.) to 245°C @ 20°C/min. to 310°C (hold 1 min.)
Det.: ECD @ 330°C



Ordering Information | Rtx®-CLPesticides Columns (Fused Silica)

ID	df (µm)	temp. limits	10-Meter	15-Meter	20-Meter	30-Meter
0.10mm	0.10	-60 to 310/330°C	43101			
0.18mm	0.18	-60 to 310/330°C	42101		42102	
0.25mm	0.25	-60 to 320/340°C		11120		11123
0.32mm	0.50	-60 to 320/340°C		11136		11139
0.53mm	0.50	-60 to 300/320°C		11137		11140

Ordering Information | 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	

Ordering Information | Rtx®-CLPesticides Kits

0.25mm ID Rtx®-CLPesticides Kit cat.# 11199 (kit)

Includes:	cat.#
30m, 0.25mm ID, 0.25µm Rtx®-CLPesticides Column	11123
30m, 0.25mm ID, 0.20µm Rtx®-CLPesticides2 Column	11323
Siltek™ Universal Angled "Y" Press-Tight® Connector	20487
5m, 0.25mm ID Siltek™ Guard Column	10026

0.32mm ID Rtx®-CLPesticides Kit cat.# 11198 (kit)

Includes:	cat.#
30m, 0.32mm ID, 0.50µm Rtx®-CLPesticides Column	11139
30m, 0.32mm ID, 0.25µm Rtx®-CLPesticides2 Column	11324
Siltek™ Universal Angled "Y" Press-Tight® Connector	20487
5m, 0.32mm ID Siltek™ Guard Column	10027

0.53mm ID Rtx®-CLPesticides Kit cat.# 11197 (kit)

Includes:	cat.#
30m, 0.53mm ID, 0.50µm Rtx®-CLPesticides Column	11140
30m, 0.53mm ID, 0.42µm Rtx®-CLPesticides2 Column	11340
Siltek™ Universal Angled "Y" Press-Tight® Connector	20487
5m, 0.53mm ID Siltek™ Guard Column	10028

Columns are not pre-connected in these kits.

DI Liners for Agilent 5890 & 6890 GCs (For 0.25/0.32/0.53mm ID Columns)	ID*/OD & Length (mm)	cat.# ea.	cat.# 5-pk.
 Drilled Uniliner®	4.0 ID 6.3 OD x 78.5	21054	21055
 Siltek™ Drilled Uniliner®	4.0 ID 6.3 OD x 78.5	21054-214.1	21055-214.5
 Siltek™ 1mm Drilled Uniliner®	1.0 ID 6.3 OD x 78.5	21390-214.1	21391-214.5

**Nominal ID at syringe needle expulsion point.*

Allows splitless injections as well as direct injections with EPC-equipped GCs!

Pesticide Surrogate Mix

04.1, 3/90, 4/89, and 2/88 SOW

decachlorobiphenyl
2,4,5,6-tetrachloro-*m*-xylene

200µg/mL each in acetone, 1mL/ampul

	Each	5-pk.	10-pk.
	32000	32000-510	—
w/data pack	32000-500	32000-520	32100

Pesticide Performance Evaluation Mix w/Surrogates (8 components)

04.1 and 3/90 SOW

α-BHC	1µg/mL
β-BHC	1
γ-BHC (lindane)	1
4,4'-DDT	10
decachlorobiphenyl	2
endrin	5
methoxychlor	25
2,4,5,6-tetrachloro-*m*-xylene	2

In hexane, 1mL/ampul

	Each	5-pk.	10-pk.
	32074	32074-510	—
w/data pack	32074-500	32074-520	32174

Organochlorine Pesticide Mix AB #2

(20 components)

aldrin	8µg/mL	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

In hexane:toluene (1:1), 1mL/ampul

	Each	5-pk.	10-pk.
	32292	32292-510	—
w/data pack	32292-500	32292-520	32392

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