

Increase Sample Throughput for Complex Drinking Water Pesticides

Using Rtx[®]-CLPesticides and Rtx[®]-CLPesticides2 Capillary Columns

By Jason Thomas, Environmental Innovations Chemist

- Optimized conditions cut analysis time in half, for higher sample throughput.
- Unique selectivity fully resolves complex compound list.
- Meets all method QA requirements, reducing rework.

With the advent of modern agriculture, and its vast selection of chemical pest control measures, the farming community has made significant increases in productivity and efficiency. Crop yield per acre is at an all time high, due in part to the role of pesticides and herbicides in mitigating the devastating effects of many plant and insect pests.¹ However, the use of these chemicals can have drawbacks, including surface and ground water contamination. EPA Methods, such as 508.1, are used to monitor pesticides and herbicides in drinking and ground water. The optimized dual column method shown here satisfies all method requirements in half the analysis time, significantly improving sample throughput.

Continued on page 4.

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Continued from page 3.

EPA Method 508.1 includes many of the components as Method 505, a similar GC/ECD method, but also contains several others, expanding the list to 38 compounds. This method calls for solid phase extraction and extract concentration, followed by analysis using a GC/ECD system. In order to increase sample throughput, an optimized method was developed using a dual column configuration with the Rtx®-CLPesticides/Rtx®-CLPesticides2 column pair. These columns, used under the conditions shown, offer a unique selectivity that allows the target analytes to be resolved in approximately half the analysis time of the original method (Figure 1). There was one coelution on the primary column, but these compounds were separated on the second column. Both columns easily passed the comprehensive system performance criteria adapted from 508.1 (Table 1).²

In conclusion, due to the complexity of the compound list in Method 508.1, a very high degree of selectivity is required of the capillary column in order to achieve adequate resolution of all target analytes in a reasonable time. The optimized dual column method shown here offers a significantly faster analysis time, while maintaining excellent resolution of challenging drinking water pesticides and herbicides.

References

1. <http://www.usda.gov/nass/pubs/trackrec/track00a.htm#principal>
2. US EPA Method 508.1, James W Eichelberger Rev 1.0 1994.

Conditions for Figure 1

Column: Rtx®-CLPesticides2, 30m, 0.32mm ID, 0.25µm (cat.# 11324) and Rtx®-CLPesticides, 30m, 0.32mm ID, 0.32µm (cat.# 11141) with 5m x 0.32mm ID Rxi® deactivated guard tubing (cat.# 10039), connected using Universal "V" Press-Tight® Connector (cat.# 20405-261)

Sample: 50ng/mL 508.1 Calibration Mix #1 (cat.# 32094), 100ng/mL 508.1 Calibration Mix #2 (cat.# 32095), 100ng/mL 508.1 Calibration Mix #3 (cat.# 32096), 50ng/mL 508.1 Internal Standard (cat.# 32091), 250ng/mL 508.1 Surrogate (cat.# 32092), 500ng/mL Atrazine (cat.# 32208), 500ng/mL Simazine (cat.# 32236) in ethyl acetate

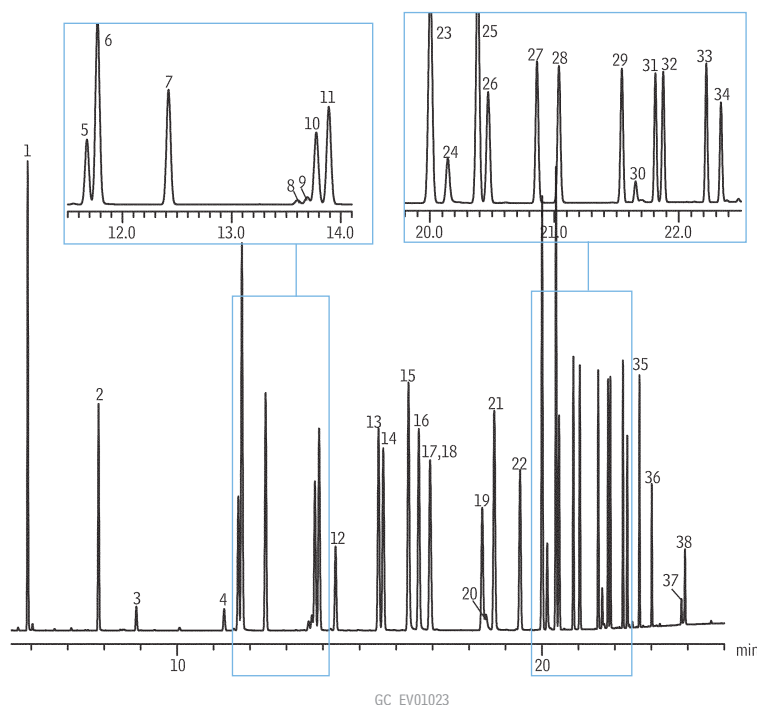
Inj.: 2µL splitless (hold 0.75 min.), 4mm cyclo double gooseneck liner (cat.# 20896)

Inj. temp.: 250°C
Carrier gas: helium, constant flow
Linear velocity: 26cm/sec. @ 80°C
Oven temp.: 80°C (hold 0.5 min.) to 155°C (hold 1 min.) @ 19°C/min. to 210°C @ 4°C/min. to 310°C (hold 0.5 min.) @ 25°C/min.
Detector temp.: ECD @ 325°C

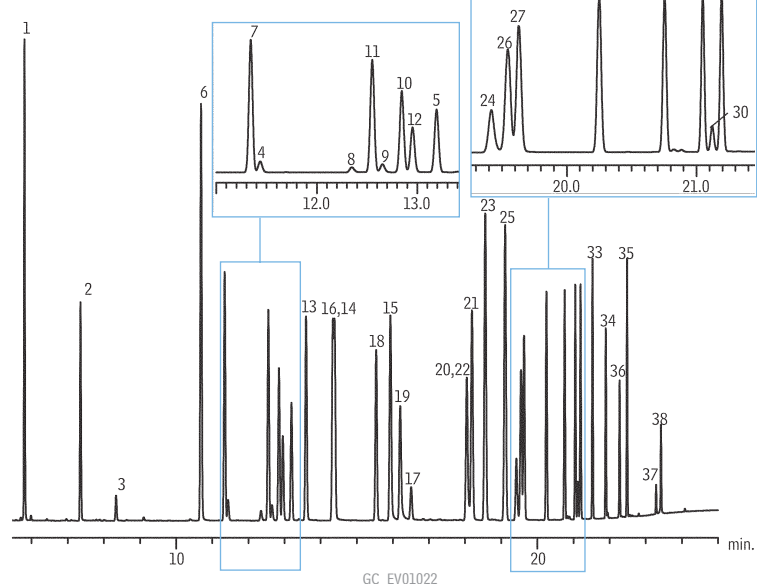
Figure 1 Resolve all critical pairs using Rtx®-CLPesticides and Rtx®-CLPesticides2 columns.

- | | | |
|----------------------------------|-------------------------------|------------------------|
| 1. hexachlorocyclopentadiene | 14. heptachlor | 27. 4,4'-DDE |
| 2. etridiazole | 15. chlorothalonil | 28. dieldrin |
| 3. chlordane | 16. metribuzin | 29. endrin |
| 4. propachlor | 17. alachlor | 30. chlorobenzilate |
| 5. trifluralin | 18. aldrin | 31. 4,4'-DDD |
| 6. hexachlorobenzene | 19. 4,4'-dibromobiphenyl (SS) | 32. endosulfan II |
| 7. α-BHC | 20. metachlor | 33. 4,4'-DDT |
| 8. simazine | 21. DCPA | 34. endrin aldehyde |
| 9. atrazine | 22. heptachlor epoxide | 35. endosulfan sulfate |
| 10. pentachloronitrobenzene (IS) | 23. γ-chlordane | 36. methoxychlor |
| 11. γ-BHC | 24. cyanazine | 37. cis-permethrin |
| 12. β-BHC | 25. α-chlordane | 38. trans-permethrin |
| 13. δ-BHC | 26. endosulfan I | |

Rtx®-CLPesticides2



Rtx®-CLPesticides



Satisfy all method requirements in half the time!

Table I Rtx®-CLPesticides and Rtx®-CLPesticides2 columns easily pass EPA Method 508.1 performance criteria.

Test/Requirement	Analyte	Concentration (ppb)	Rtx®-CLPesticides2	Rtx®-CLPesticides
Inertness (breakdown <20%)	endrin	50	0.9%	1.4%
Inertness (breakdown <20%)	4,4'-DDE	100	1.0%	1.1%
Sensitivity (S/N>3)	chlorpyrifos	2	12.0	6.2
Chromatographic performance (0.8<PGF<1.15)	DCPA	50	1.03	1.06
Column performance (resolution>0.50)	chlorothalonil	50	9.9	26.8
Column performance (resolution>0.50)	gamma-BHC	40	9.9	26.8

Rxi® Guard/Retention Gap Columns (fused silica)

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.	10-Meter	10-Meter/6-pk.
0.25mm	0.37 ± 0.04mm	10029	10029-600	10059	10059-600
0.32mm	0.45 ± 0.04mm	10039	10039-600	10064	10064-600
0.53mm	0.69 ± 0.05mm	10054	10054-600	10073	10073-600

Universal "Y" Press-Tight® Connectors

Description	ea.	3-pk.
Universal "Y" Press-Tight Connector	20405	20406
Deactivated Universal "Y" Press-Tight Connector	20405-261	20406-261
Siltek Treated Universal "Y" Press-Tight Connector	20485	20486

Rtx®-CLPesticides Columns (fused silica)

ID	df (µm)	temp. limits	length	cat. #
0.32mm	0.32	-60 to 320/340°C	30-Meter	11141

508.1 Calibration Mix #1 (17 components)

aldrin	endosulfan I
α-BHC	endosulfan II
β-BHC	endosulfan sulfate
δ-BHC	endrin
γ-BHC (lindane)	endrin aldehyde
4,4'-DDD	heptachlor
4,4'-DDE	heptachlor epoxide (isomer B)
4,4'-DDT	methoxychlor
dieldrin	

500µg/mL each in ethyl acetate, 1mL/ampul
cat. # 32094

508.1 Calibration Mix #2 (11 components)

chlorobenzilate	hexachlorobenzene
α-chlordane	cis-permethrin*
γ-chlordane	trans-permethrin*
chlorneb	propachlor
DCPA (Dacthal®)	trifluralin
etridiazole	

500µg/mL each in ethyl acetate, 1mL/ampul
cat. # 32095

*1000µg/mL total permethrin. Exact content of each isomer listed on certificate of analysis.

508.1 Calibration Mix #3 (8 components)

alachlor	hexachlorocyclopentadiene
atrazine	metolachlor
chlorthalonil	metribuzin
cyanazine	simazine

500µg/mL each in ethyl acetate, 1mL/ampul
cat. # 32096

Rtx®-CLPesticides2 Columns (fused silica)

ID	df (µm)	temp. limits	length	cat. #
0.32mm	0.25	-60 to 320/340°C	30-Meter	11324

508.1 Internal Standard

pentachloronitrobenzene
100µg/mL in ethyl acetate, 1mL/ampul
cat. # 32091

508.1 Surrogate

4,4'-dibromobiphenyl
500µg/mL in ethyl acetate, 1mL/ampul
cat. # 32092

Atrazine

1,000µg/mL in acetone, 1mL/ampul
cat. # 32208

Simazine

1,000µg/mL in acetone, 1mL/ampul
cat. # 32236

Splitless Liners for Agilent GC

ID* x OD & Length	qty.	cat.#
Cyclo Double Gooseneck (4mm)		
4.0mm x 6.5mm x 78.5mm	5-pk.	20896

*Nominal ID at syringe needle expulsion point.

Resprep™-C18 SPE Disks

Description	qty.	cat.#
Resprep-C18 47mm SPE Disks	20-pk.	24004