Application Note

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Retention Time Locking of Organochlorine Pesticides on an 8860 Using the OpenLab Retention Time Locking Wizard

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Abstract

The retention time locking (RTL) wizard (a feature of Agilent OpenLab CDS) was used to demonstrate retention time stability on an Agilent 8860 gas chromatograph. Using heptachlor epoxide as the locking compound, analysis of a standard containing 20 chlorinated pesticides showed excellent peak resolution and retention time repeatability before and after a column maintenance event.

Introduction

Maintaining consistent retention times after routine maintenance and across multiple instruments is a simple way to increase laboratory productivity. When retention times are consistent, there is no need to update calibration tables and integration events. Methods can also be shared across multiple instruments, and their results more easily compared.

The RTL wizard included in OpenLab CDS is designed to help guide new users through the locking process, but is still streamlined enough for experienced users. The wizard automates the process of locking a method by performing three injections of a standard with varying inlet pressures to build a retention time versus pressure (RT versus P) calibration. This calibration only needs to be run once, and will be used to relock retention times by automatically adjusting the method's inlet pressure. A method may need relocking after replacing or trimming columns, moving to a new system, or changing from an atmospheric to a vacuum detector. The relocking of a method requires only a single run.

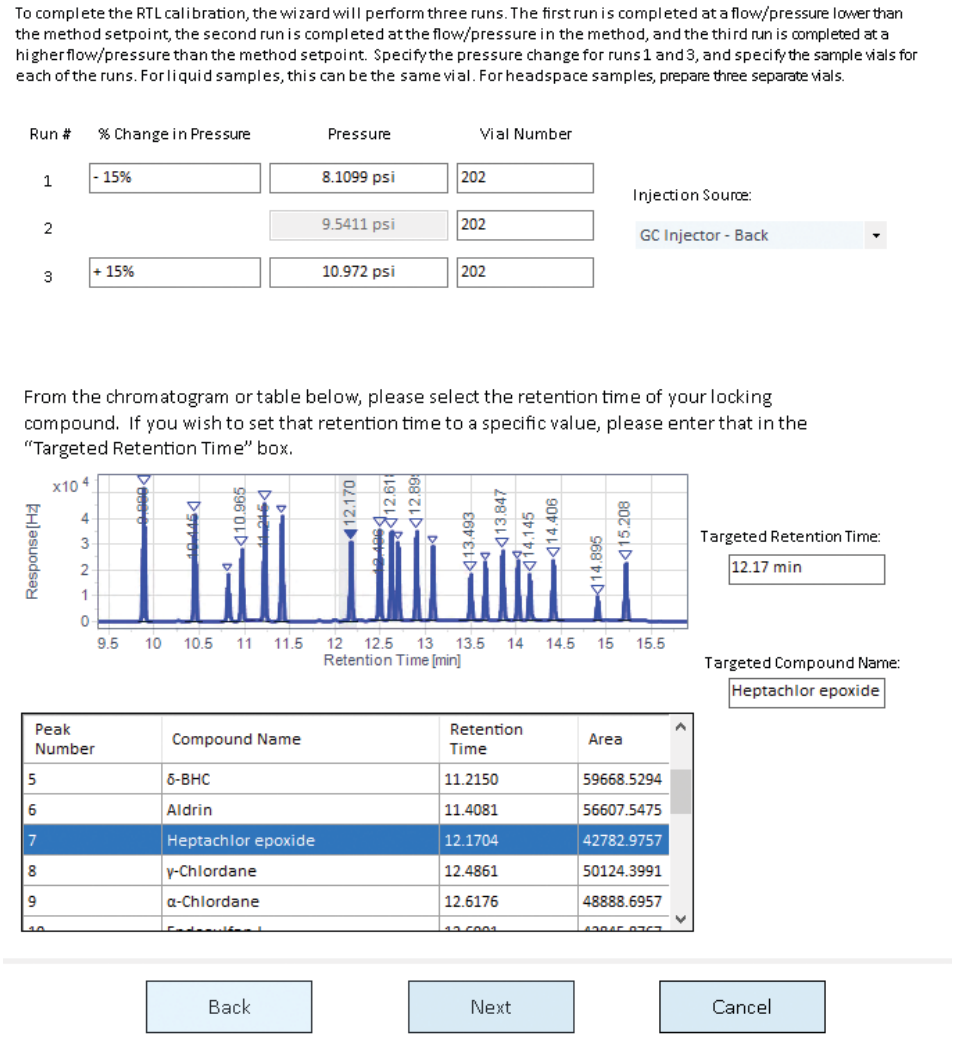

Figure 1. Retention time locking wizard setup screen.

After launching the RTL wizard, the user is prompted to select an established acquisition and processing method. The user then selects a chromatogram from a previously run standard, and chooses the locking compound (Figure 1). A good locking compound will have an easily identifiable, well resolved peak in a critical part of the chromatogram. Then, the upper and lower inlet pressures can be adjusted or left at default. Whether the column control mode is set to flow or pressure, the RTL wizard will always calculate from the initial inlet pressure. The RTL wizard will then perform the three injections, and prompt the user to identify the locking compound in each of the resulting chromatograms (Figure 2). Upon saving, the method is locked.

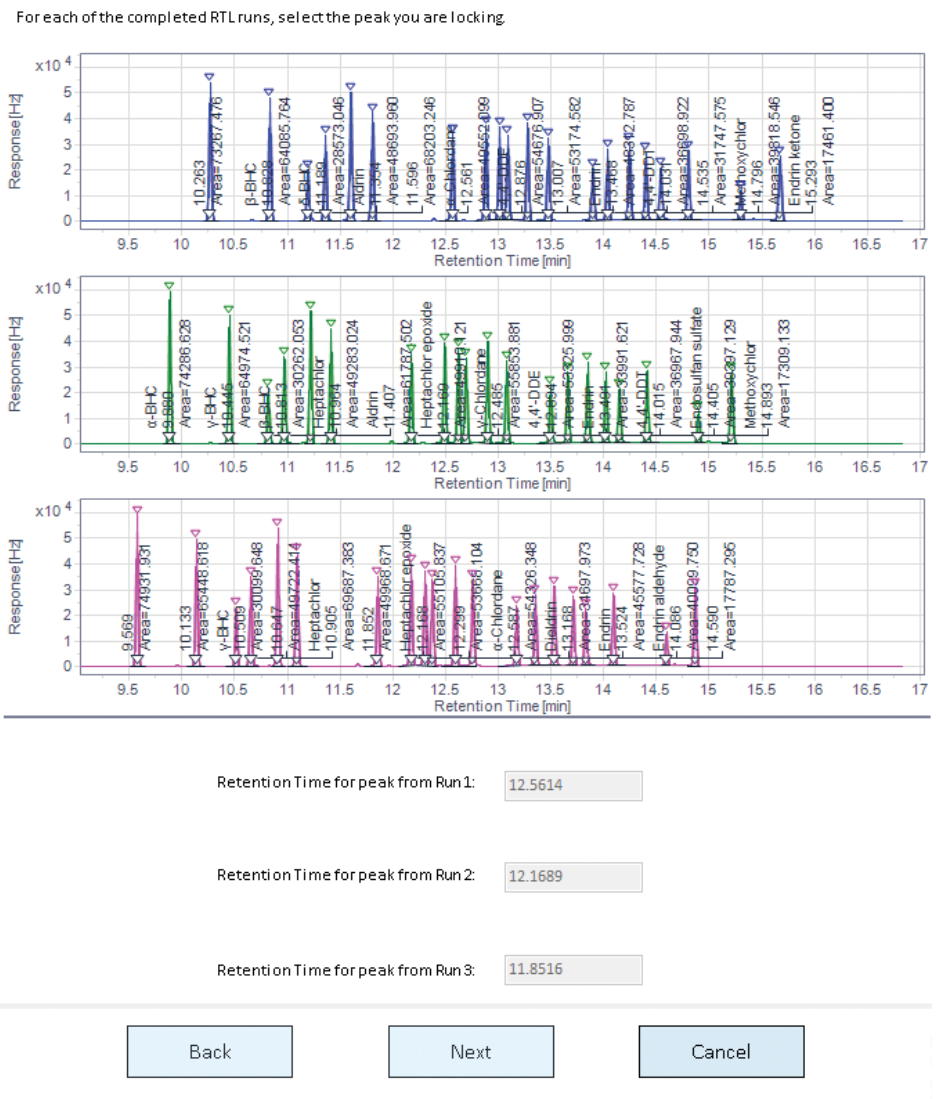

Figure 2. Target compound selection from locking runs.

Experimental

The OpenLab RTL wizard was demonstrated on an 8860 GC equipped with a split/splitless inlet and an Electron Capture detector. A standard containing 20 chlorinated pesticides was diluted to 2 ppm, and injected onto an Agilent DB-CLP1 column (p/n 123-8232). The injections were made under the method conditions listed in Table 1, and retention time repeatability was tested over 10 injections (Table 2). Heptachlor epoxide was selected as the locking compound, and the inlet pressure was varied by ±15 % of the method's setpoint. Column maintenance was simulated by trimming approximately 50 cm from the inlet end of the column. The standard was injected again, and the retention time shift after the column trimming was recorded. The RTL wizard was run to relock the method, and another run was made to verify successful retention time locking.

Table 1. Method parameters.

ALS	
Syringe	10 µL
Injection volume	0.5 µL
Inlet (SSL)	
Mode	Pulsed splitless
Heater	On, 250 °C
Pressure	9.5411 psi
Injection pulse pressure	60 psi for 0.3 minutes
Purge flow to split vent	75 mL/min at 0.5 minutes
Septum purge	3 mL/min
Liner	Splitless, single taper, Ultra Inert (p/n 5190-2292)
Column	
Column	Agilent DB-CLP1, 30 m × 320 µm, 0.25 µm (p/n 123-8232)
Carrier	Helium, 2 mL/min, constant flow
Oven	
	50 °C, 30 °C/min to 135 °C, 15 °C/min to 300 °C, hold two minutes
Detector (ECD)	
Heater	300 °C
Make up	N_2 at 60 mL/min

Table 2. Retention time repeatability results from 10 injections.

Compound	%RSD	RT Average	RT After maintenance	ΔRT After maintenance	RT After relocked	ΔRT After relocked
α-BHC	0.005	9.881	9.790	0.094	9.884	0.003
γ-BHC	0.007	10.445	10.354	0.094	10.448	0.003
β-BHC	0.008	10.814	10.724	0.092	10.816	0.002
Heptachlor	0.005	10.965	10.873	0.093	10.966	0.001
δ-BHC	0.007	11.216	11.124	0.092	11.216	0.000
Aldrin	0.007	11.408	11.313	0.096	11.409	0.001
Heptachlor epoxide	0.005	12.170	12.076	0.094	12.170	0.000
γ-Chlordane	0.006	12.487	12.394	0.093	12.487	0.000
α-Chlordane	0.005	12.617	12.523	0.095	12.618	0.001
Endosulfan I	0.007	12.690	12.595	0.097	12.692	0.002
4,4'-DDE	0.006	12.895	12.803	0.092	12.895	0.000
Dieldrin	0.007	13.075	12.980	0.094	13.074	0.001
Endrin	0.004	13.493	13.397	0.096	13.493	0.000
4,4'-DDD	0.005	13.655	13.563	0.091	13.654	0.001
Endosulfan II	0.004	13.847	13.750	0.096	13.846	0.001
4,4'-DDT	0.006	14.016	13.923	0.091	14.014	0.002
Endrin aldehyde	0.006	14.144	14.048	0.095	14.143	0.001
Endosulfan sulfate	0.004	14.406	14.311	0.094	14.405	0.001
Methoxychlor	0.007	14.895	14.802	0.090	14.892	0.003
Endrin ketone	0.006	15.208	15.102	0.104	15.206	0.002

Results and discussion

The 20 organochlorine pesticides were well resolved by the 8860 GC (Figure 3). Retention time stability was also excellent, with all 20 compounds showing less than 0.008 % RSD (Table 2). The RTL wizard was run at inlet pressures of 8.1, 9.5, and 10.9 psi. The resulting chromatograms were processed through the RTL wizard, and the RT versus P calibration achieved an R^2 of 0.999.

After trimming the column to simulate maintenance, the peaks eluted approximately 0.100 to 0.090 minutes earlier in the chromatogram (Figure 4). This was just enough of a shift to move some peaks out of OpenLab default retention time windows. This RT shift would have required adjustments to the calibration table, but in this case, the run was analyzed by the RTL Wizard. Using the RT versus P calibration, the RTL wizard automatically made the necessary adjustment to the inlet pressure, and applied the new setpoint of 9.1 psi to the method. A confirmation run with the new inlet pressure resulted in a shift of only 0.001 to 0.003 minutes from the premaintenance average retention times. All peaks in the confirmation run were identified by the processing method, and no retention times needed adjusting.

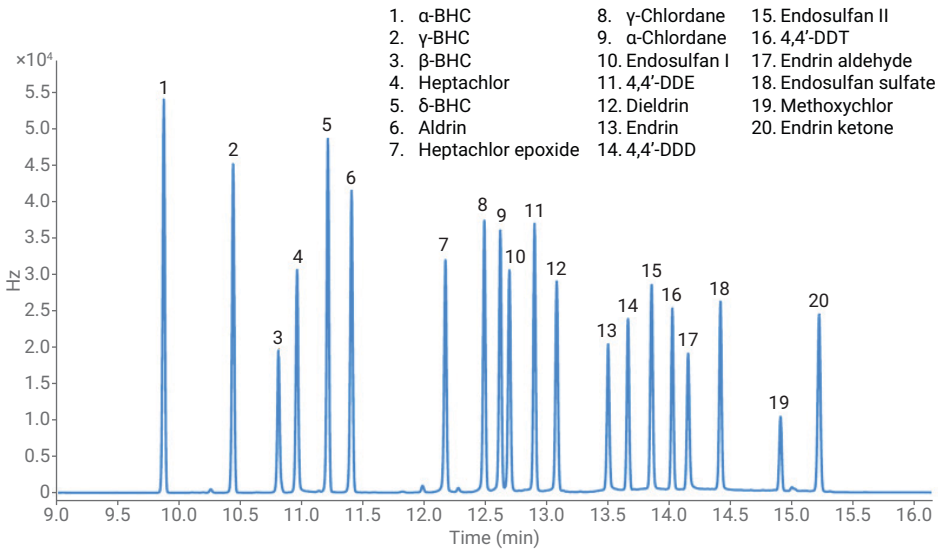

Figure 3. Twenty chlorinated pesticides separated on a DB-CLP1 column.

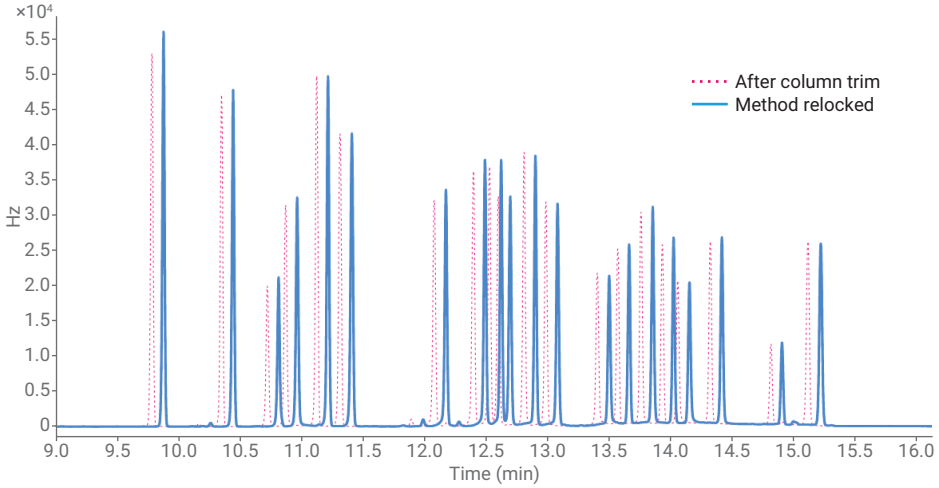

Figure 4. Retention time shift after column maintenance.

Conclusions

The combination of the 8860 GC with the retention time locking Wizard proved to be a simple way to maintain retention times after column maintenance. The retention times were relocked, and matched the premaintenance times to within 0.003 minutes with no need to make any further adjustments to the method.

References

1. Organochlorine Pesticide Analysis Using an Agilent Intuvo 9000 Dual ECD GC System. *Agilent Technologies*, publication number 5991-9000EN, February **2018**.
2. Giarrocco, V.; Quimby, B.; Klee, M. Retention Time Locking: Concepts and Applications. *Agilent Technologies*, publication number 5966-2469E, December **1997**.

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