

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

Author

Ian Eisele
Agilent Technologies, Inc.

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.

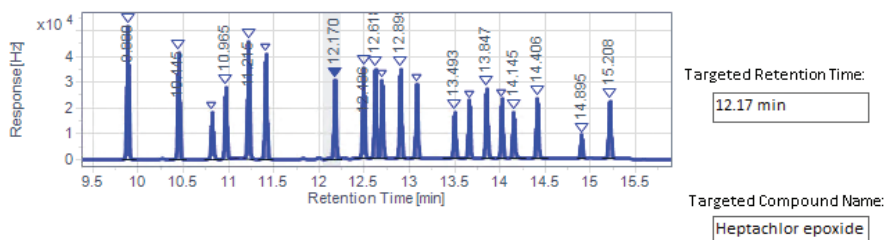
The RTL wizard included in OpenLab CDS is designed to help guide new users through the locking process, but is still streamlined enough for experienced users. The wizard automates the process of locking a method by performing three injections of a standard with varying inlet pressures to build a retention 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.

To complete the RTL calibration, the wizard will perform three runs. The first run is completed at a flow/pressure lower than the method setpoint, the second run is completed at the flow/pressure in the method, and the third run is completed at a higher flow/pressure than the method setpoint. Specify the pressure change for runs 1 and 3, and specify the sample vials for each of the runs. For liquid samples, this can be the same vial. For headspace samples, prepare three separate vials.

| Run # | % Change in Pressure | Pressure | Vial Number |
|-------|-----------------------------------|---|----------------------------------|
| 1 | <input type="text" value="-15%"/> | <input type="text" value="8.1099 psi"/> | <input type="text" value="202"/> |
| 2 | | <input type="text" value="9.5411 psi"/> | <input type="text" value="202"/> |
| 3 | <input type="text" value="+15%"/> | <input type="text" value="10.972 psi"/> | <input type="text" value="202"/> |

Injection Source:

From the chromatogram or table below, please select the retention time of your locking compound. If you wish to set that retention time to a specific value, please enter that in the "Targeted Retention Time" box.

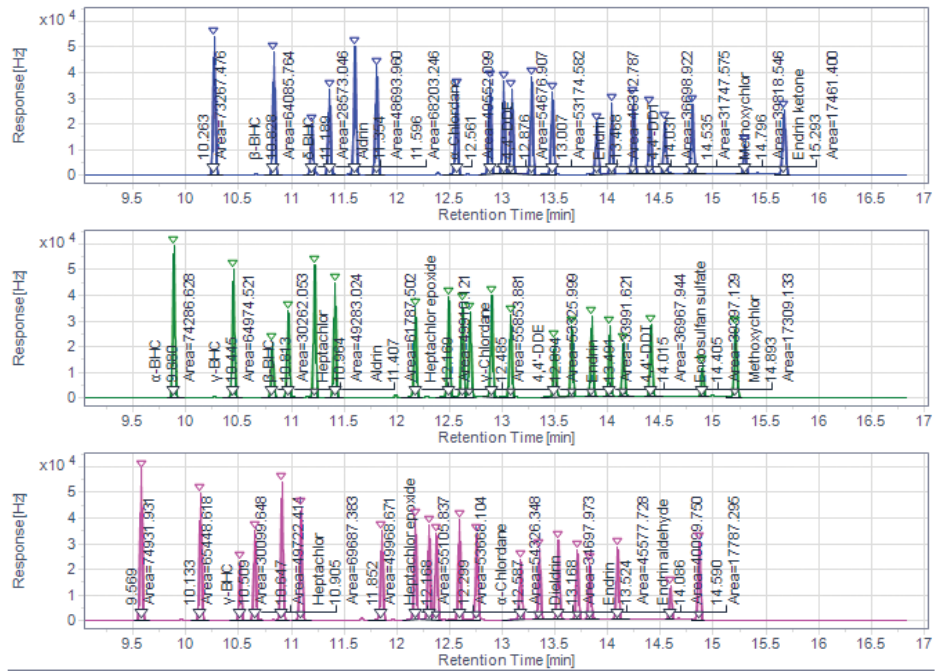


| Peak Number | Compound Name | Retention Time | Area |
|-------------|--------------------|----------------|------------|
| 5 | δ-BHC | 11.2150 | 59668.5294 |
| 6 | Aldrin | 11.4081 | 56607.5475 |
| 7 | Heptachlor epoxide | 12.1704 | 42782.9757 |
| 8 | γ-Chlordane | 12.4861 | 50124.3991 |
| 9 | α-Chlordane | 12.6176 | 48888.6957 |
| 10 | Endosulfan | 13.2991 | 43845.8757 |

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.

For each of the completed RTL runs, select the peak you are locking



Retention Time for peak from Run 1:

Retention Time for peak from Run 2:

Retention Time for peak from Run 3:

Back

Next

Cancel

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 $\pm 15\%$ 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 μ L |
| Injection volume | 0.5 μ L |
| 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 \times 320 μ m, 0.25 μ m (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 | Δ RT After maintenance | RT After relocked | Δ RT 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 pre-maintenance average retention times. All peaks in the confirmation run were identified by the processing method, and no retention times needed adjusting.

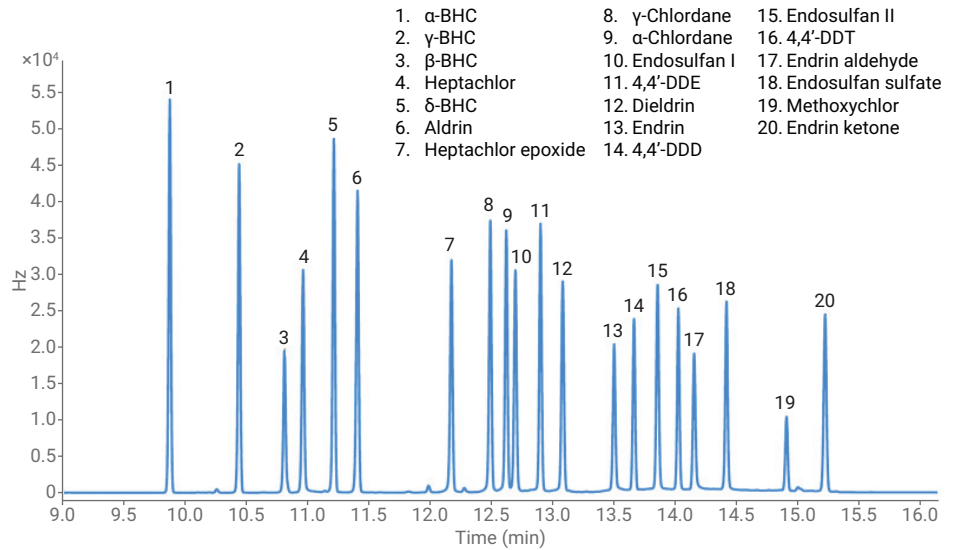


Figure 3. Twenty chlorinated pesticides separated on a DB-CLP1 column.

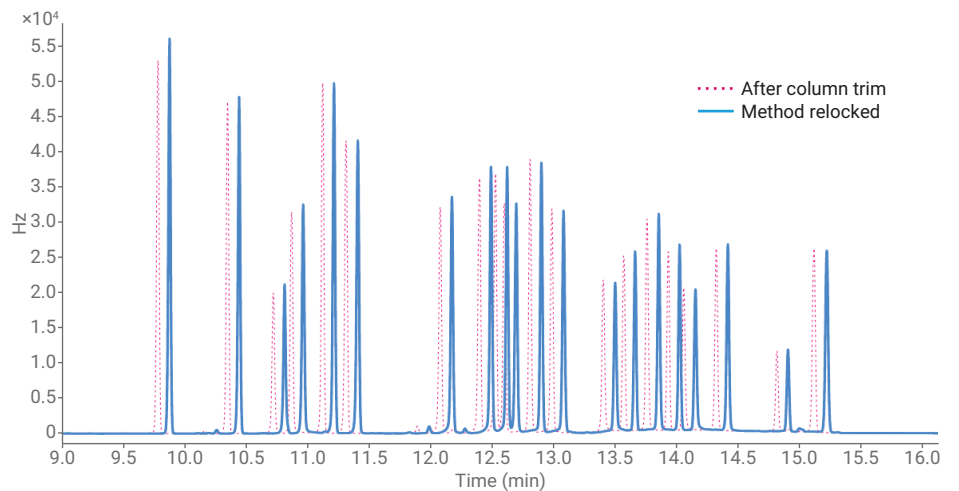


Figure 4. Retention time shift after column maintenance.

Conclusion

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 pre-maintenance 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**.

www.agilent.com/chem

This information is subject to change without notice.

© Agilent Technologies, Inc. 2019
Printed in the USA, June 25, 2019
5994-0551EN