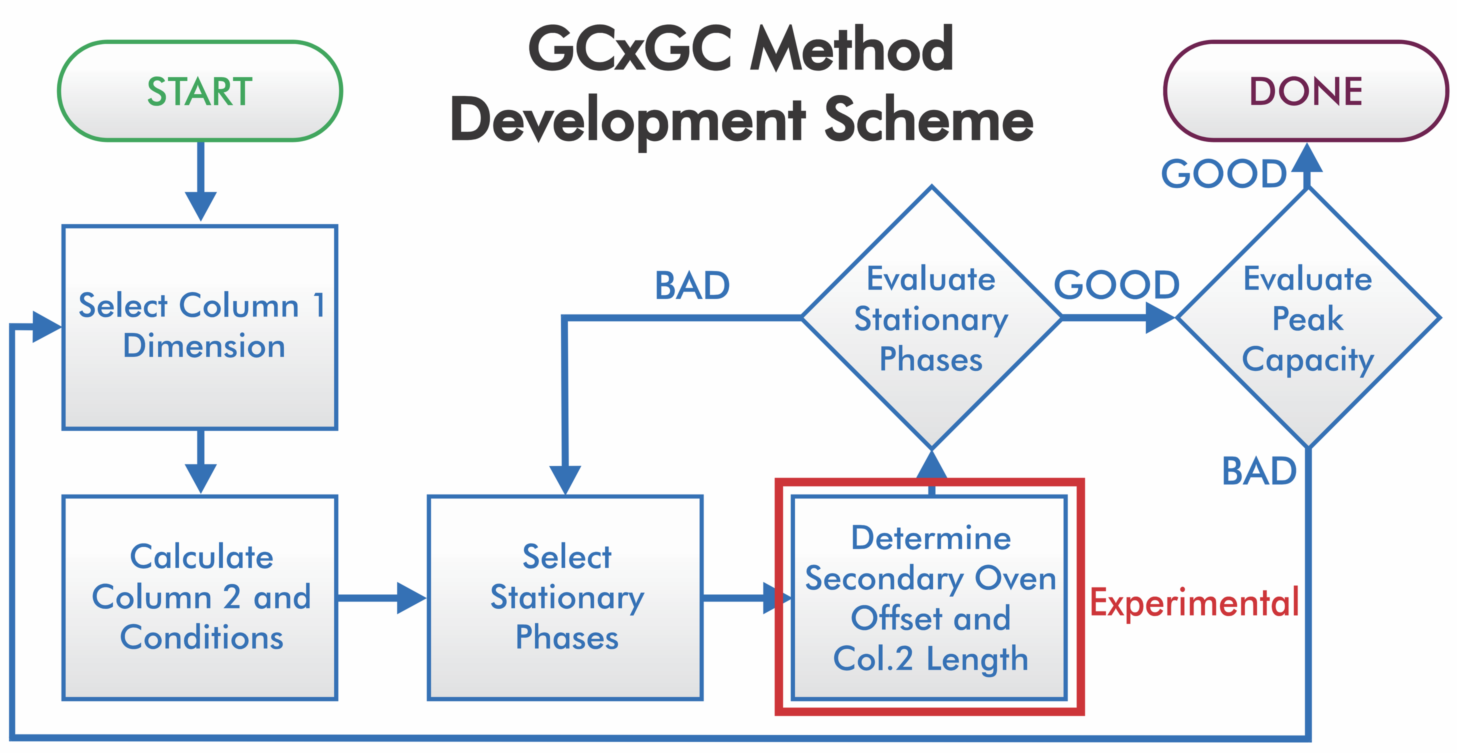
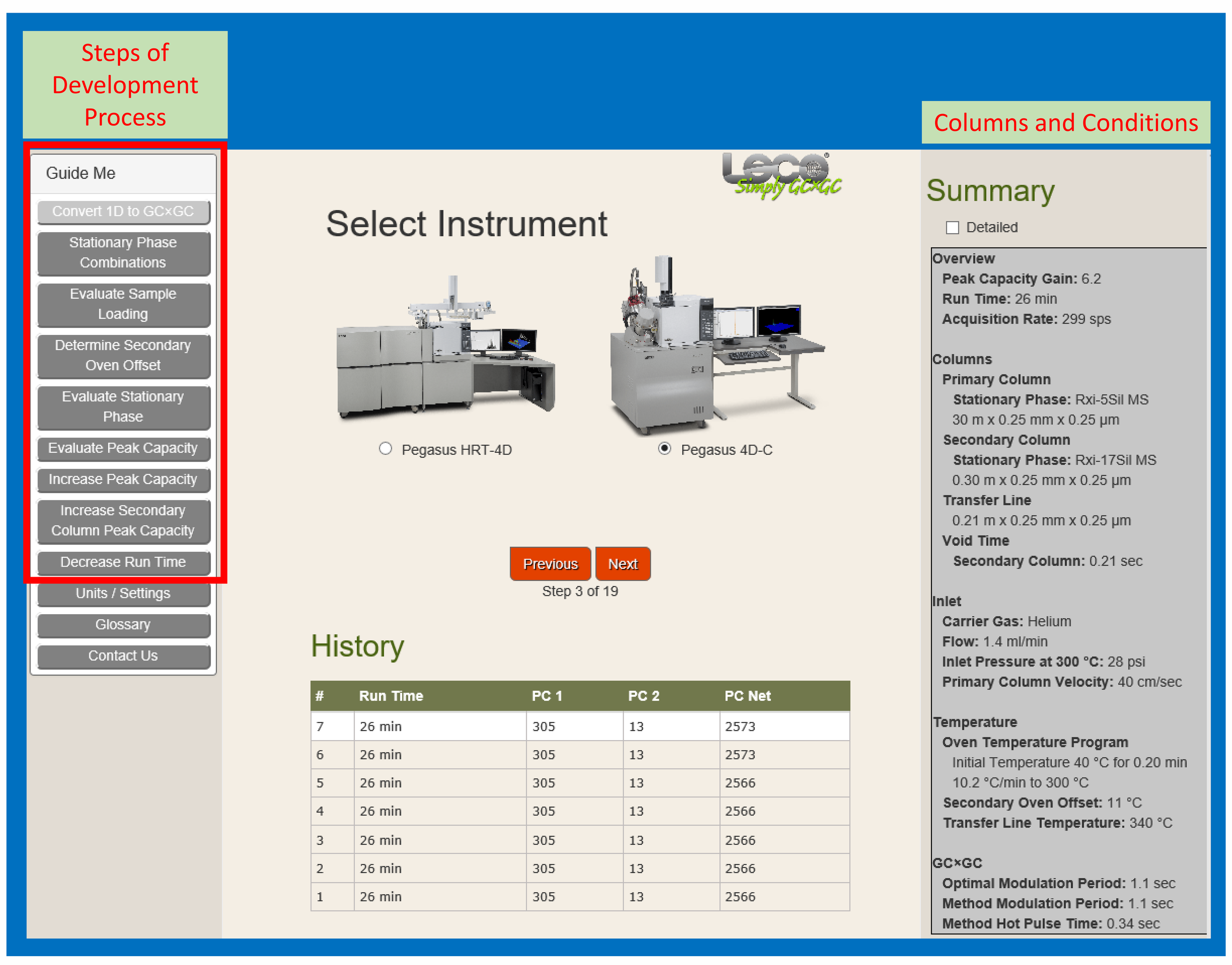
A GC×GC Method Development Scheme Utilizing a Software Tool

Mark F. Merrick;[1] Steve Robles;[1] Kevin McNitt;[1] Leonid M. Blumberg;[2] | [1] LECO Corporation, St. Joseph, MI USA; [2] Advachrom, Wilmington, DE USA

Introduction

Comprehensive two-dimensional gas chromatography/time-of-flight mass spectrometry (GC×GC-TOFMS) has been commercially available for 15 years. It is surprising that such a powerful and useful technique has not become more widespread. One reason offered for this lack of interest is that method development is too difficult. Last year at the 13th GC×GC Symposium we presented validation results for a computational engine which calculates the optimum column dimensions and operating conditions for a GCxGC method with respect to peak capacity. In this poster we present a GC×GC method development tool (wizard) utilizing this computational engine. This software tool, "Simply GCxGC™", is freely available on the internet at www.leco.com/simply-gcxgc .





GC×GC Method Development Tool – "Simply GCxGC"

Step 1 - Convert 1D to GC×GC

Information: General description of the tool.

Input: User selects Pegasus® HRT-4D or Pegasus 4D-C, carrier gas, primary column dimensions, start temperature, hold, end temperature, column flow (optimum recommended), and heating rate (optimum recommended).

Output: Starting GC×GC method including secondary column length with same ID, optimum flow if chosen, optimum heating rate if chosen, optimum modulation period, run time, recommended data acquisition rate, and predicted peak capacity (each column and net).

Step 2 - Stationary Phase Combinations

Information: General recommendations on selecting stationary phases and table of recommended combinations for selected applications.

Input: User selects stationary phase for each column. This is just to document selections. Tool does not use this information.

Output: Lists in summary of columns and conditions.

Step 3 - Evaluate Sample Loading

Information: General description of how to evaluate sample loading in GC×GC.

Input: User installs column set and makes analysis at recommended conditions with representative sample or standard. User evaluates data and makes adjustments in sample loading as needed.

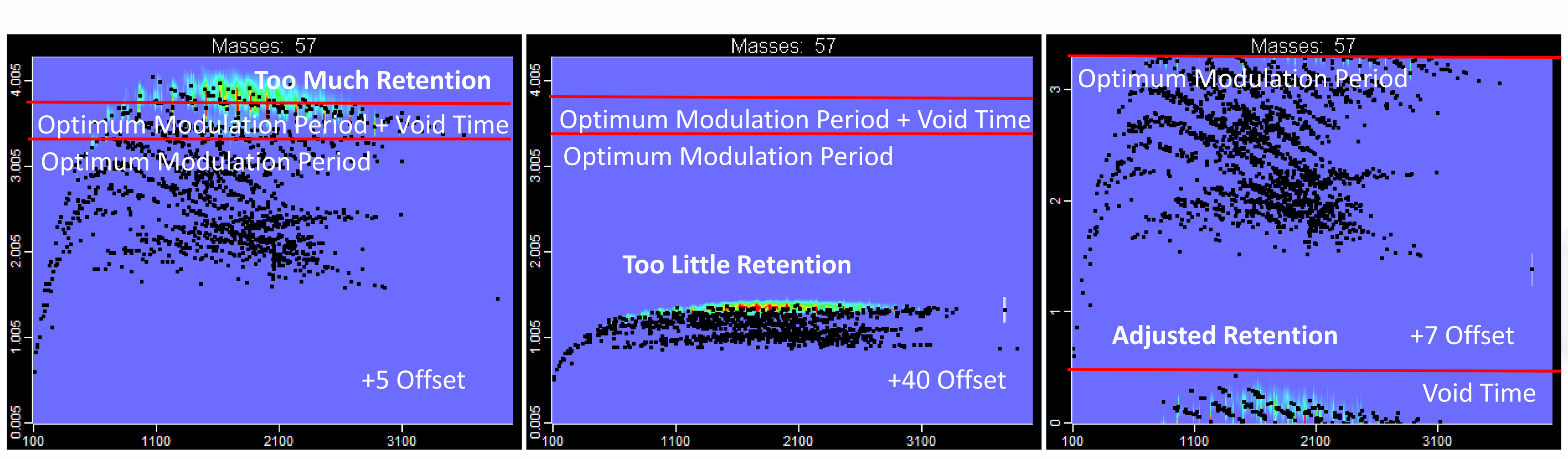
Output: Next step.

Step 4 - Determine Secondary Oven Offset

Information: Description of how to determine the appropriate secondary oven offset and adjust the secondary column length as needed. Note that depending on stationary phases, the desired adjustment may not be possible.

Input: User does two analyses with recommended column set and conditions at an offset of +5° and +40° with a long modulation period to avoid wrap around. The user identifies the last eluting peak in the second dimension and inputs the first and second dimension retention times for the two analyses.

Output: The tool directs the user to: 1) add a loop of Column 2 to the secondary oven if there is too little retention at +5° and +40°; 2) remove a loop of Column 2 from the secondary oven if there is too much retention at +5° and +40°; 3) analyze the sample at the calculated secondary oven offset and confirm the result; or 4) change stationary phases because the retention cannot be adjusted as desired.



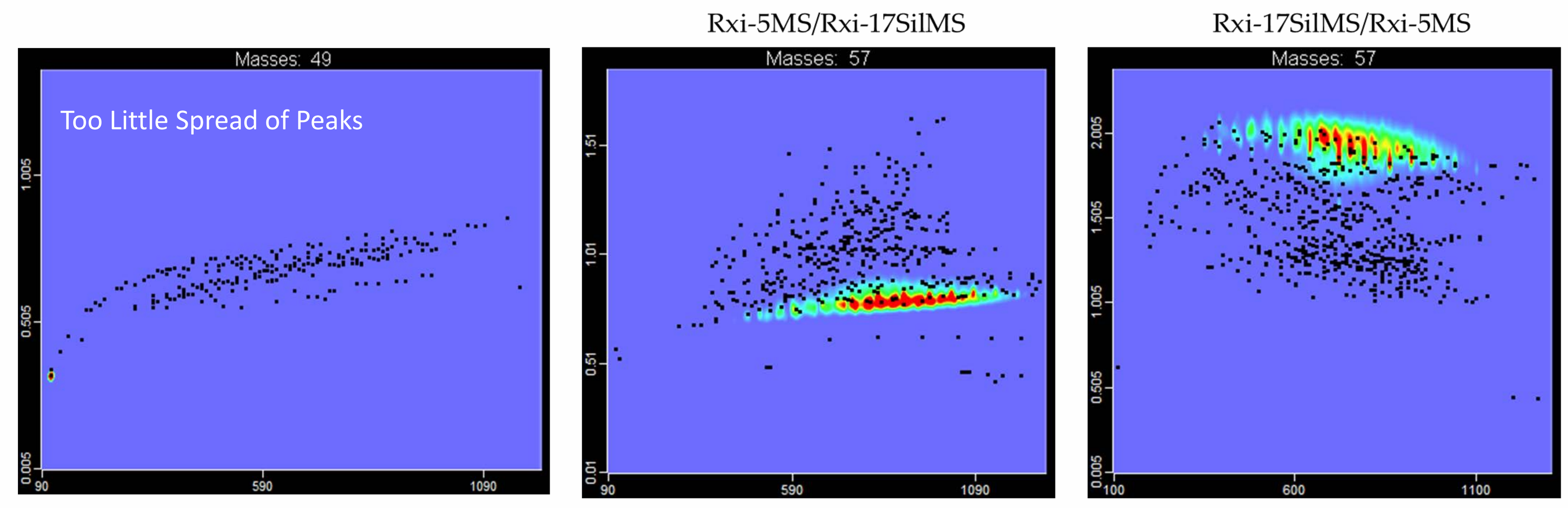
Determination of Secondary Oven Offset – example of desired retention between +5° and +40°.

Step 5 - Evaluate Stationary Phase

Information: Description of how to evaluate the stationary phase in each dimension and general guidelines with examples of good and bad results.

Input: User selects Good if the stationary phases are acceptable, or Bad if the stationary phases are not acceptable.

Output: The tool goes to the next step depending on the user's choice.



Poor stationary phase combination.

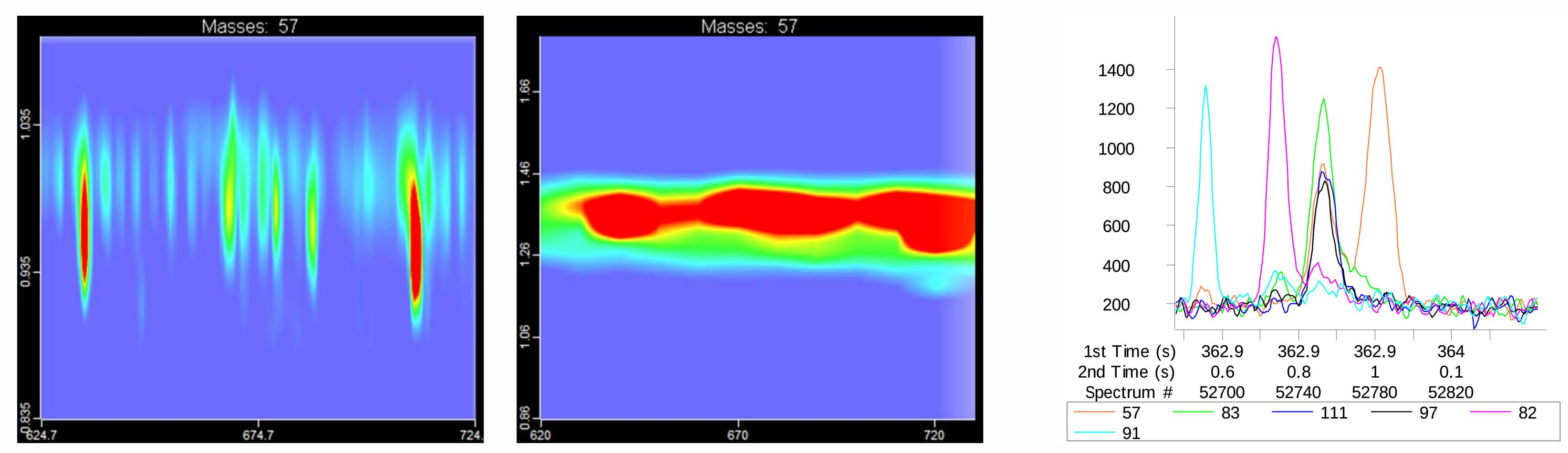
Good stationary phase combinations. Note difference between apolar/polar and polar/apolar.

Step 6 - Evaluate Peak Capacity

Information: Description of how to evaluate peak capacity in each dimension.

Input: Based on evaluation of the data, the user selects from three choices: 1) "Need More" total peak capacity; 2) "Need More" (peak capacity) in the Second dimension only; or 3) Good.

Output: The tool goes to the next step depending on the user's choice.



Evaluate first dimension. Is there enough resolution (peak capacity)?

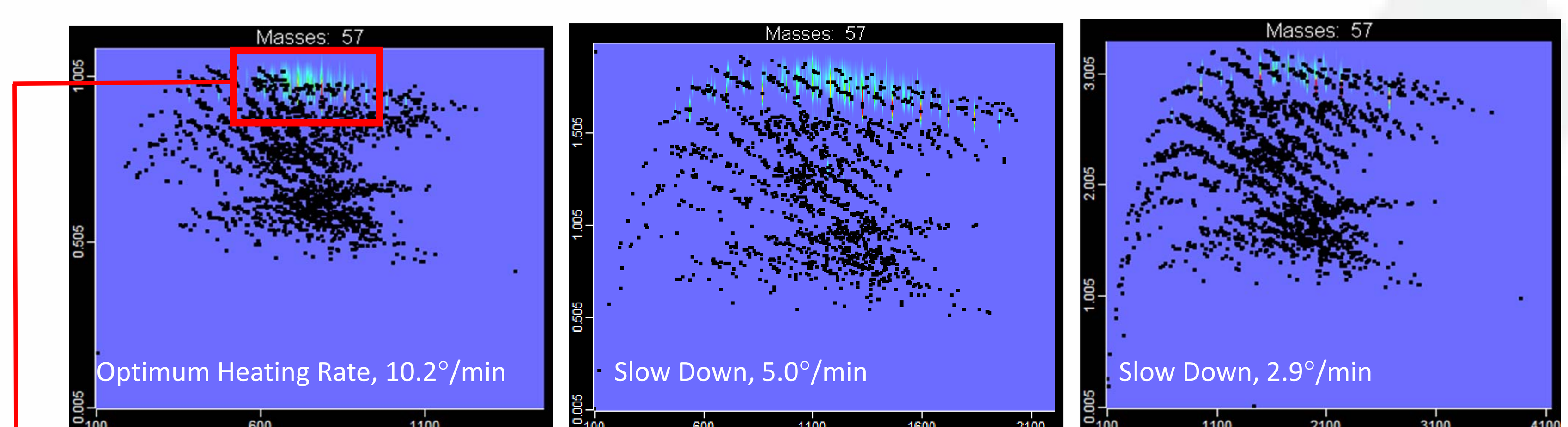
Evaluate second dimension. Is there enough resolution (peak capacity)?

Step 7 - Increase Peak Capacity

Information: Choices are provided which increase the total peak capacity: 1) lengthen columns; 2) slow down heating rate with peak capacity similar to lengthened columns; or 3) slow down heating rate not as much for intermediate increase of peak capacity. Note that increasing column lengths has a shorter run time, but requires changing the column set.

Input: User selects choice.

Output: The tool updates the column dimensions and conditions and takes the user back to determining the secondary oven offset and secondary column length.



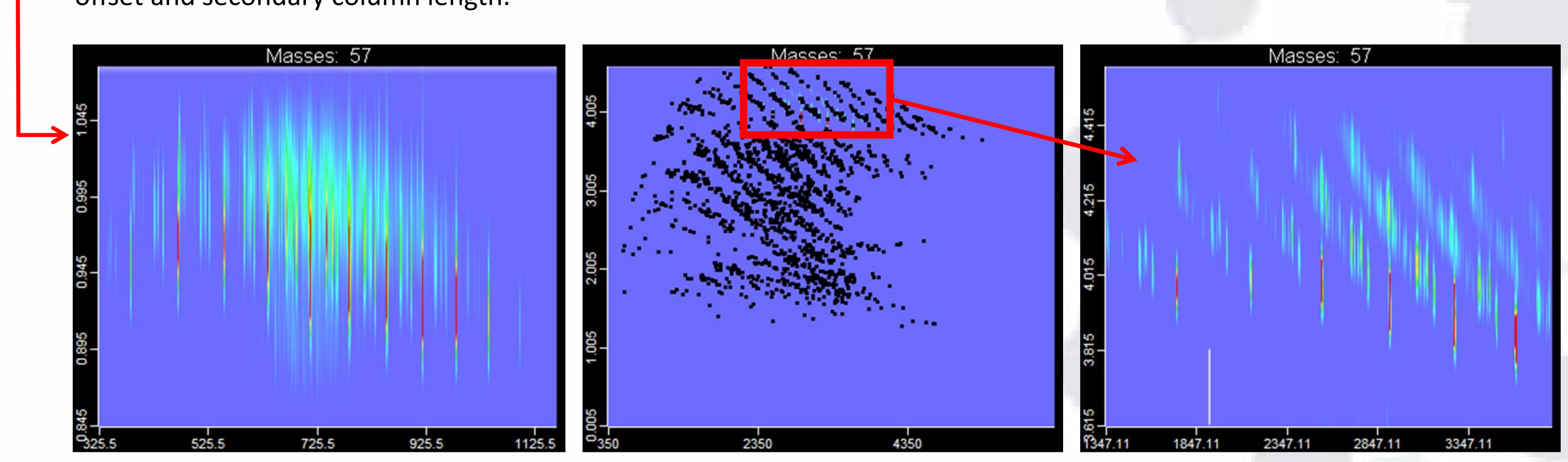
Diesel on 30 x 0.25 x 0.25 Rxi-17SilMS/0.6 x 0.25 x 0.25 Rxi-5MS

Step 8 - Increase Secondary Column Peak Capacity

Information: Choices are provided which increase peak capacity in the second dimension: 1) use narrower diameter; 2) use narrower diameter with slow down; or 3) use even narrower ID. These choices mean that the primary column ID will stay the same while the secondary column ID is decreased to the next smaller size, or decrease the ID further. The smaller the ID of the secondary column is relative to the primary column ID, the greater the increase in peak capacity in the second dimension.

Input: User selects choice.

Output: The tool updates the column dimensions and conditions and takes the user back to determining the secondary oven offset and secondary column length.



30 x 0.25 x 0.25/
0.6 x 0.25 x 0.25, 10.2°/min

30 x 0.25 x 0.25/0.9 x 0.1 x 0.1, 2.0°/min

Step 9 - Decrease Run Time

Information: Choices are provided: 1) keep current column dimensions and conditions; 2) shorten column (will decrease time and peak capacity); 3) ramp faster (will decrease time and peak capacity); or 3) use narrower diameter in both dimensions (will decrease time and keep peak capacity the same).

Input: User selects choice.

Output: The tool updates the column dimensions and conditions and takes the user back to determining the secondary oven offset and secondary column length.

Conclusion

A GC×GC Method Development software tool, "Simply GCxGC™", has been developed and is freely available on the internet at www.leco.com/simply-gcxgc . This tool provides the user with an easy step-by-step, logical process to an optimized GC×GC method.

Delivering the Right Results