How to determine non-volatile hydrocarbons according to DIN H53 (ISO 16703)?

The recommended procedure for H53 method integration in Clarity (method 1):

1. Use *Baseline – Lock* on the whole chromatogram.
2. Apply *Baseline – Allow Crossing* to the whole chromatogram with the option To Start/End.
3. Via *Peak – Add positive*, add the following peaks:
    - A peak with the start time just before the solvent peak (this is the initial point of the baseline) and the end time at the beginning of the first fraction.
    - The first fraction (C10) peak with the start time exactly at the end time of the previous peak and the end time at the end of the fraction.
    - In a similar manner, i.e., with non-overlapping time frames, add the other peaks for remaining fractions (optionally, C10–C20, C20–C30, and C30–C40).
4. Set *Baseline – Together* and *Baseline – Forward horizontal* over the entire chromatogram.
5. Assign peaks to respective fractions using *Force Peak Name* (exact times are filled automatically when selecting the peak from the graph).
6. Create a calibration file for the present fractions.

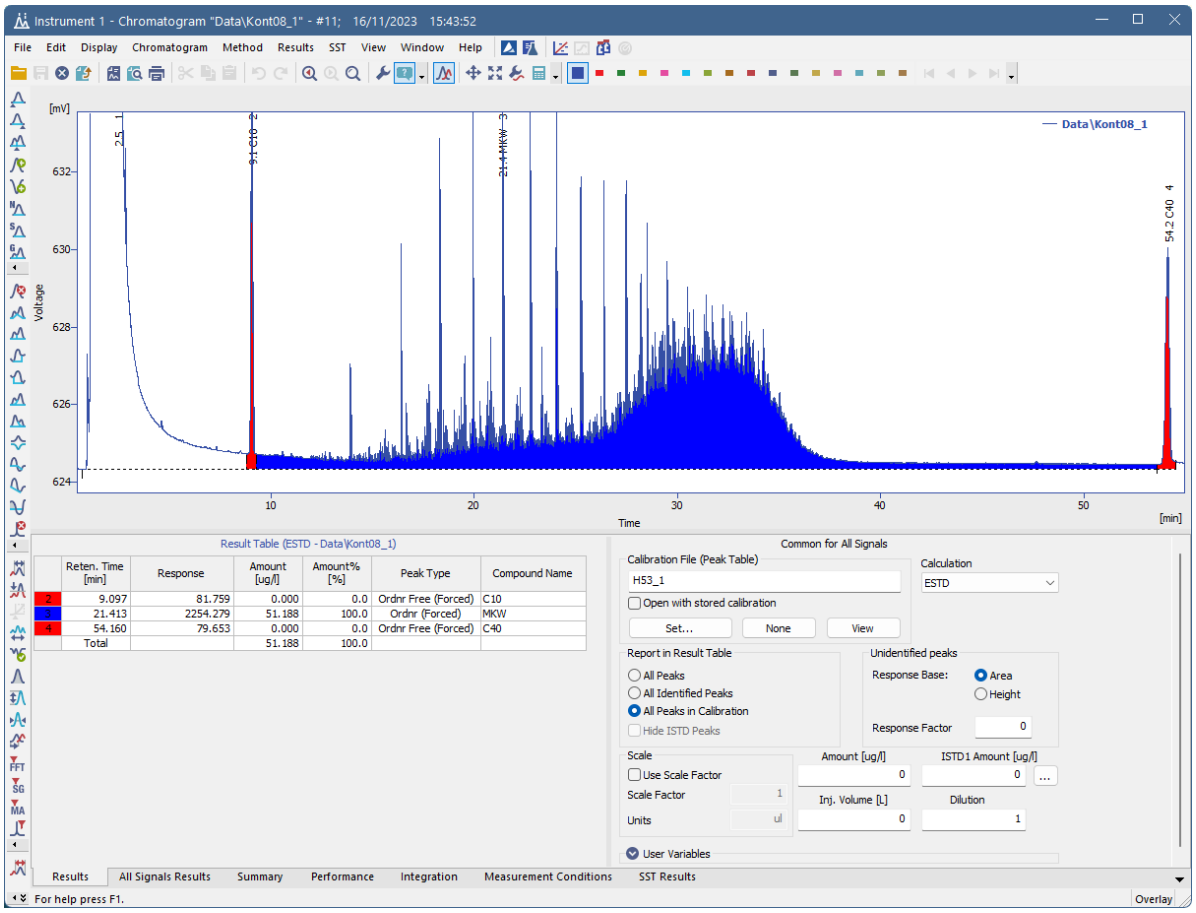
When the chromatogram is integrated appropriately, you can use the *Method - Save as Template* command from the *Chromatogram* window to save the method as a template method, so it can be used for further measurements. In the case you have already measured all the samples, you can apply the integration on all your chromatograms via *Analysis – Batch* reprocess by method.

Integration Table

Chromatogram Operation	Time A [min]	Time B [min]	Value
Global Peak Width			0.100 min
Global Threshold			0.1000
Global Filter - Bunching			2
Baseline - Lock	0.000	0.000	
Baseline - Allow Crossing	0.000	0.000	To Start/End
Peak - Add positive	0.700	8.800	
Peak - Add positive	8.800	9.300	
Peak - Add positive	9.300	53.600	
Peak - Add positive	53.600	54.500	
Baseline - Together	0.000	0.000	
Baseline - Forw. horizontal	0.000	0.000	
Peak - Force Peak Name	8.800	9.300	C10
Peak - Force Peak Name	9.300	53.600	MKW
Peak - Force Peak Name	53.600	54.500	C40

Note: In the case, that the amounts of some compounds in standards are not known, it is necessary to set the *Curve Fit Type* to *Free Calibration* on the tabs of these compounds in the *Calibration* window.

Example of an evaluated chromatogram



An alternative procedure for H53 method integration in Clarity (method 2):

1. Use the same *Peak Width* and *Threshold* as in standard integration.
2. Apply *Baseline – Together* to the whole chromatogram.
3. Apply *Baseline – Forward Horizontal* to the whole chromatogram.
4. Mark all the peaks between C10 and C40 as a group (the group boundary lines may exceed the C10 and C40 peaks, only fully contained peaks will be included).
5. Amend the peak starts and ends by manual peak tools if necessary for the C10 and C40 (use the Peak - Both, with the A time corresponding to the respective C10 or C40 time and B time positive or negative, respectively. The times are relative, i.e., will be shifted accordingly in other chromatograms).
6. Create calibration file for C10, C40, and Group A peaks.

If the integration is not correct at this point (e.g., the integrated area for Group A between C10 and C40 peaks does not start/end exactly where the C10/C40 peaks end/start respectively, as shown in the figure below), an additional step might be added after step 3.: Apply again *Baseline – Together* to the whole chromatogram. Continue with the remaining steps.

To subtract a baseline, in the *Method Setup – Advanced* dialog, set the baseline chromatogram, and in the *Matching*, set *No Change*. The baseline file should be acquired on the stabilized system and preferably without injection of solvent or at

least with a lower volume of the solvent. Otherwise, negative artifacts will appear, interfering with the correct baseline setting in Clarity later on.

Integration Table

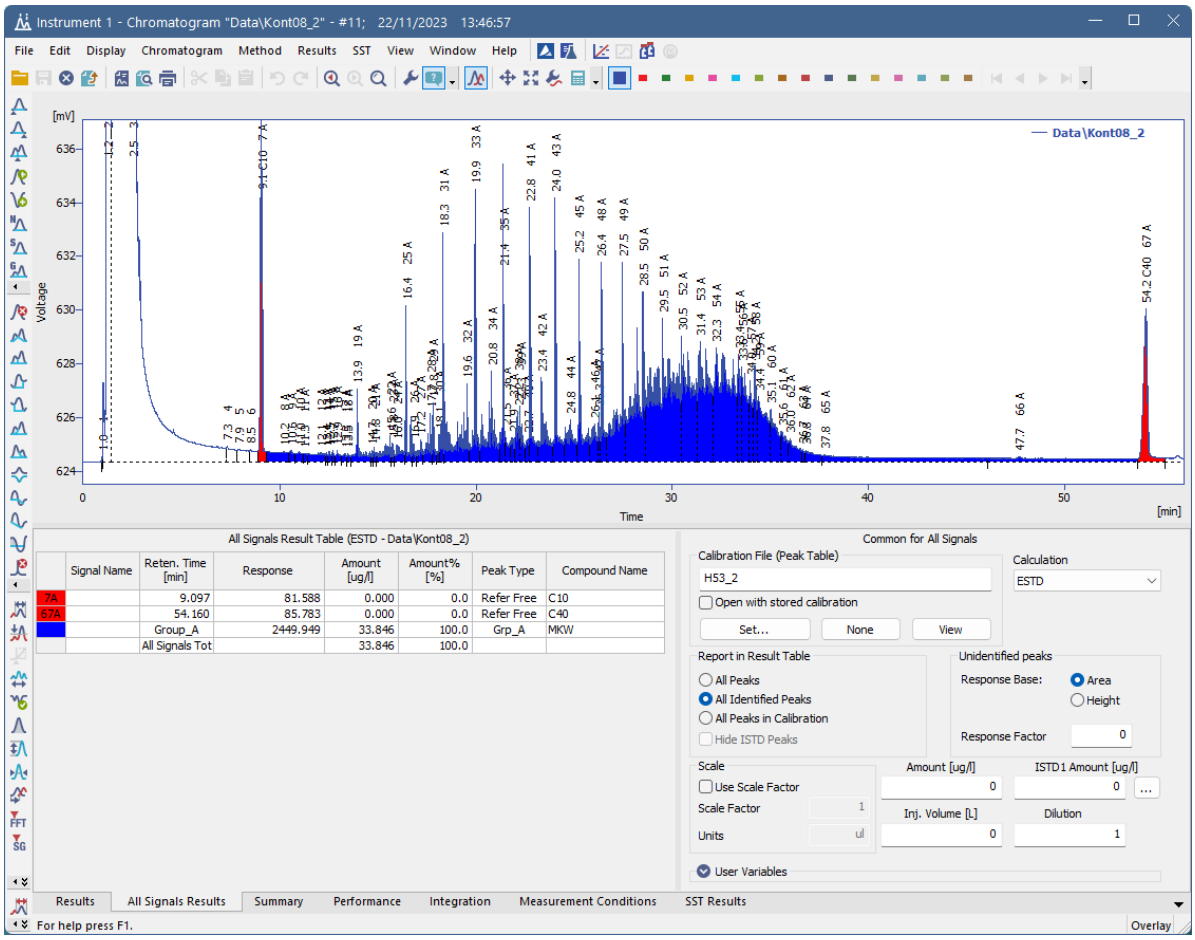
Chromatogram Operation	Time A [min]	Time B [min]	Value
Global Peak Width			0.100 min
Global Threshold			0.1000
Global Filter - Bunching			2
Group - Add group	9.061	54.490	A
Baseline - Together	0.000	0.000	
Baseline - Forw. horizontal	0.000	0.000	
Baseline - Together	0.000	0.000	
Peak - Both	12.520	-3.204	
Peak - Both	54.093	-0.454	

There is a condition that must be met:

- **The drawn baseline must not cross the chromatogram line (the negative areas arising do not have any physical meaning).**

This approach is generally less reliable and more sensitive to the choice of Integration Algorithm.

Example of an evaluated chromatogram



Sample data for Clarity

- DIN_H53.zip - zipped chromatograms, methods, and calibrations.
- Din-H53-processed.dgz - archived Clarity Project to restore with Clarity via the Archive/Restore function