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Two-Dimensional Preparative Chromatography: Development of CPC x SFC for the isolation of high molecular weight compounds from biomass

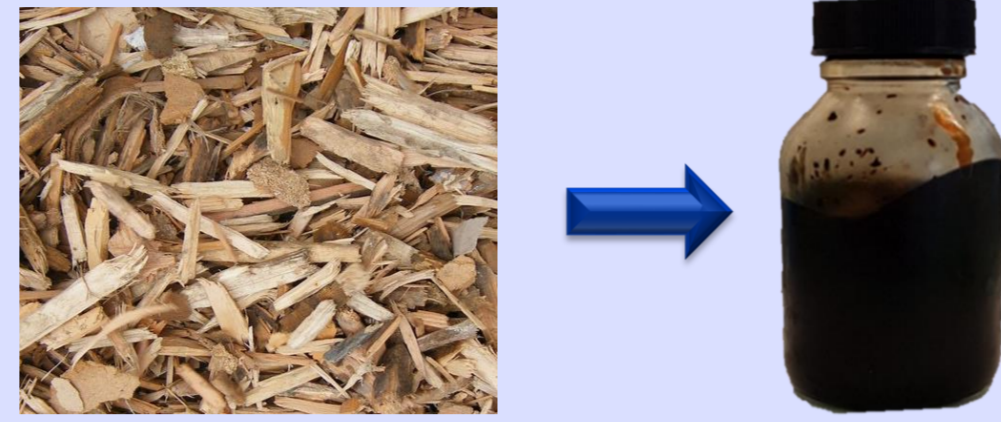
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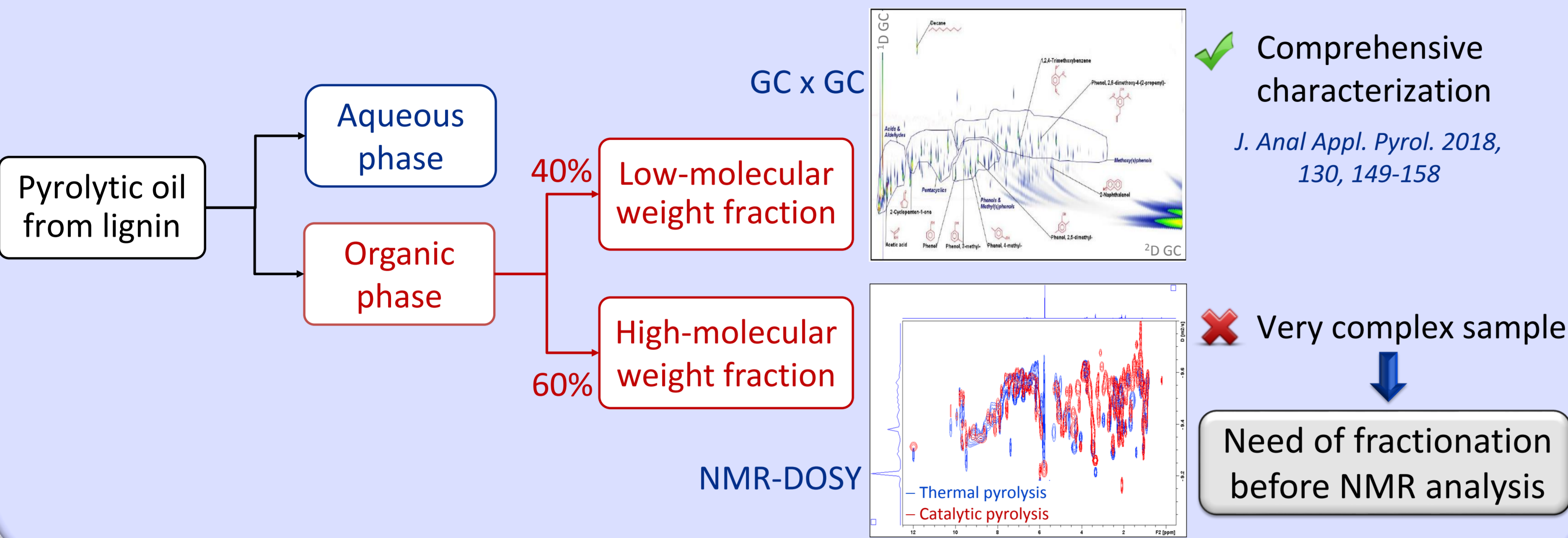
Bio-oil from biomass as an alternative to fossil fuels:

- due to rarefaction of crude oil
- contributes to diversify energy sources
- reduces greenhouse gas emissions

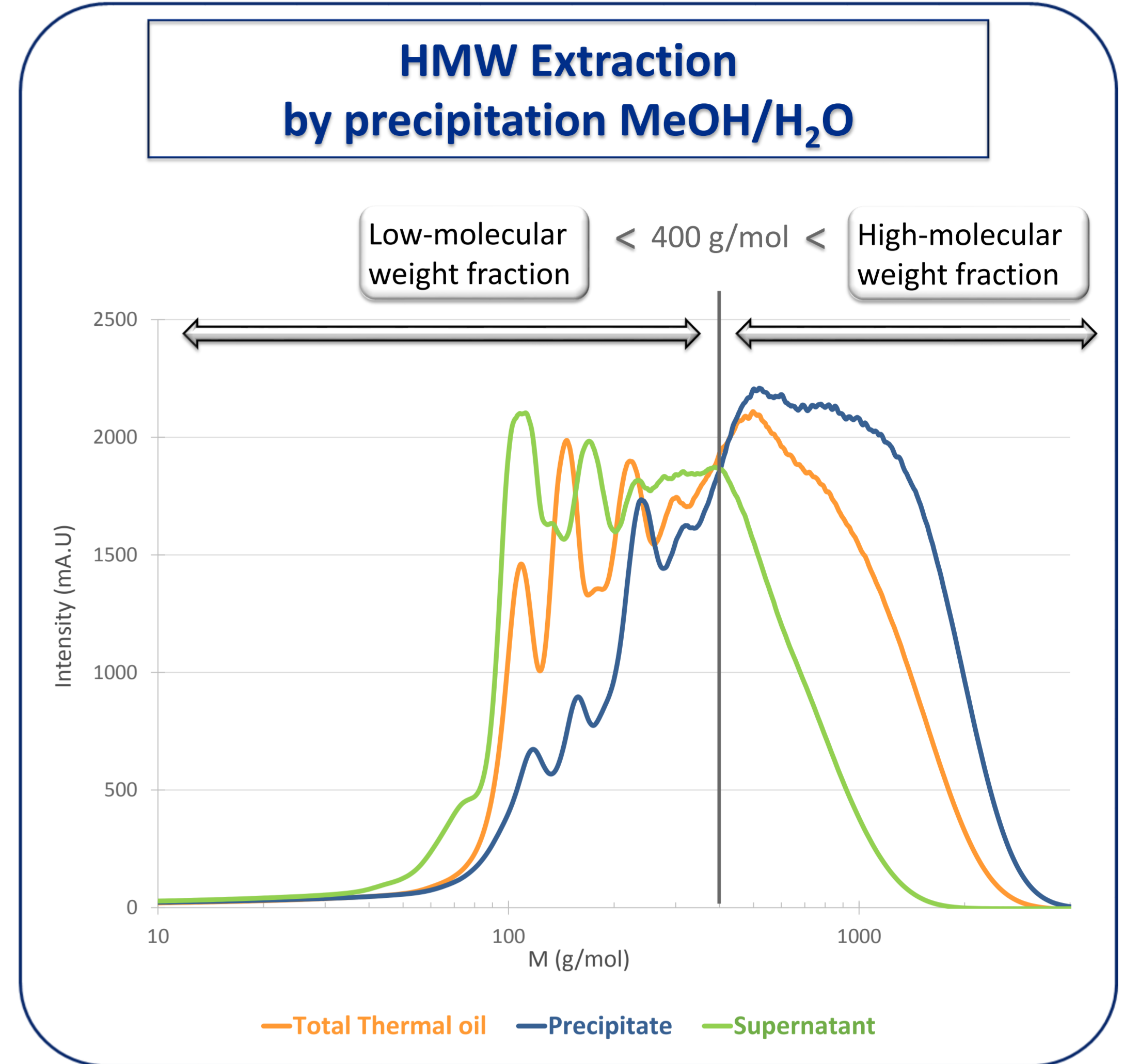


Valorization of lignocellulose biomass from forestry industry waste into energetic bio-oil by hydroconversion process (conventional thermal pyrolysis or catalytic pyrolysis)

- to optimize the catalyst → need to identify which molecules are concerned by catalysis



Sample Preparation

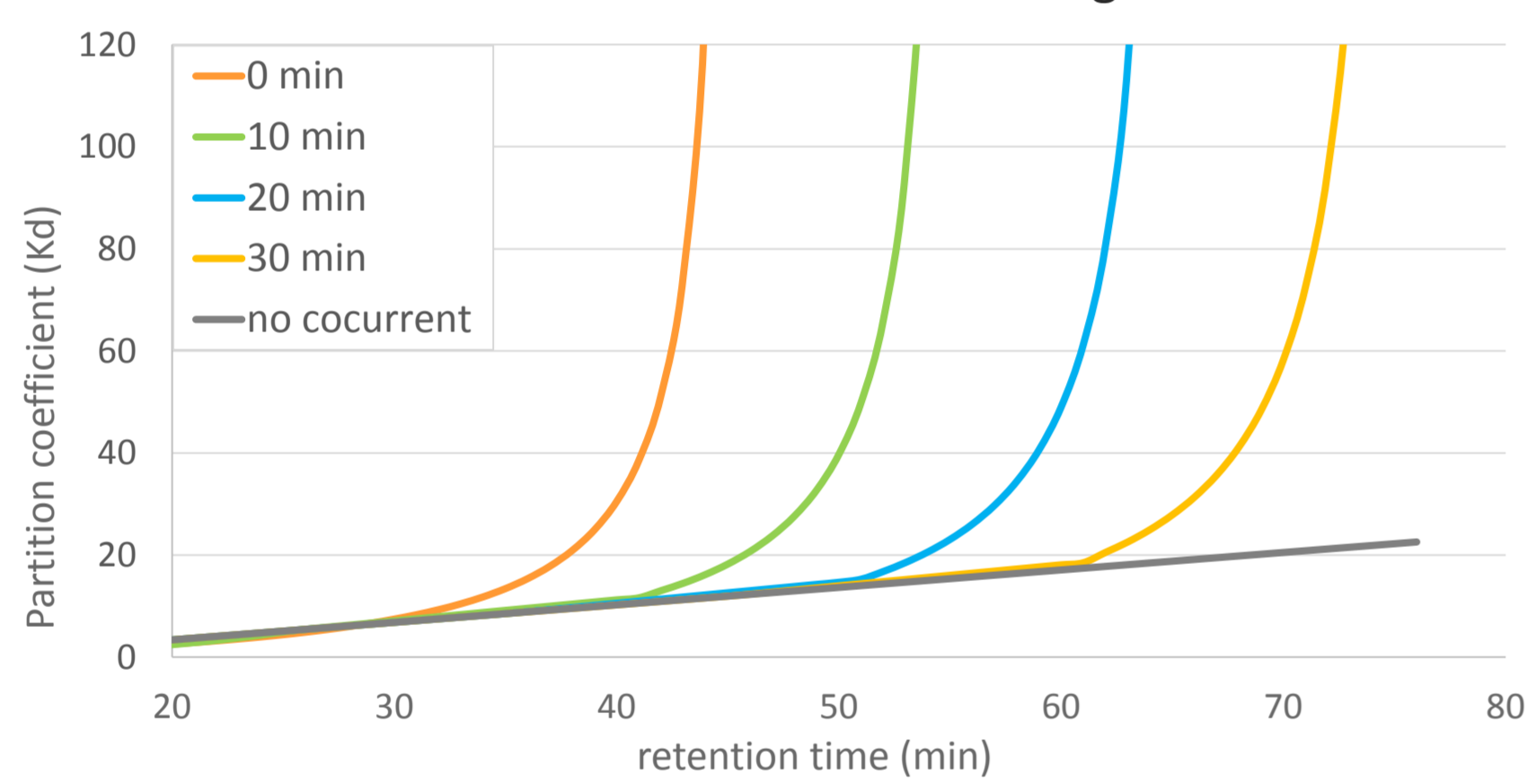


Development of the preparative CPC x SFC method

1D: Centrifugal Partition Chromatography

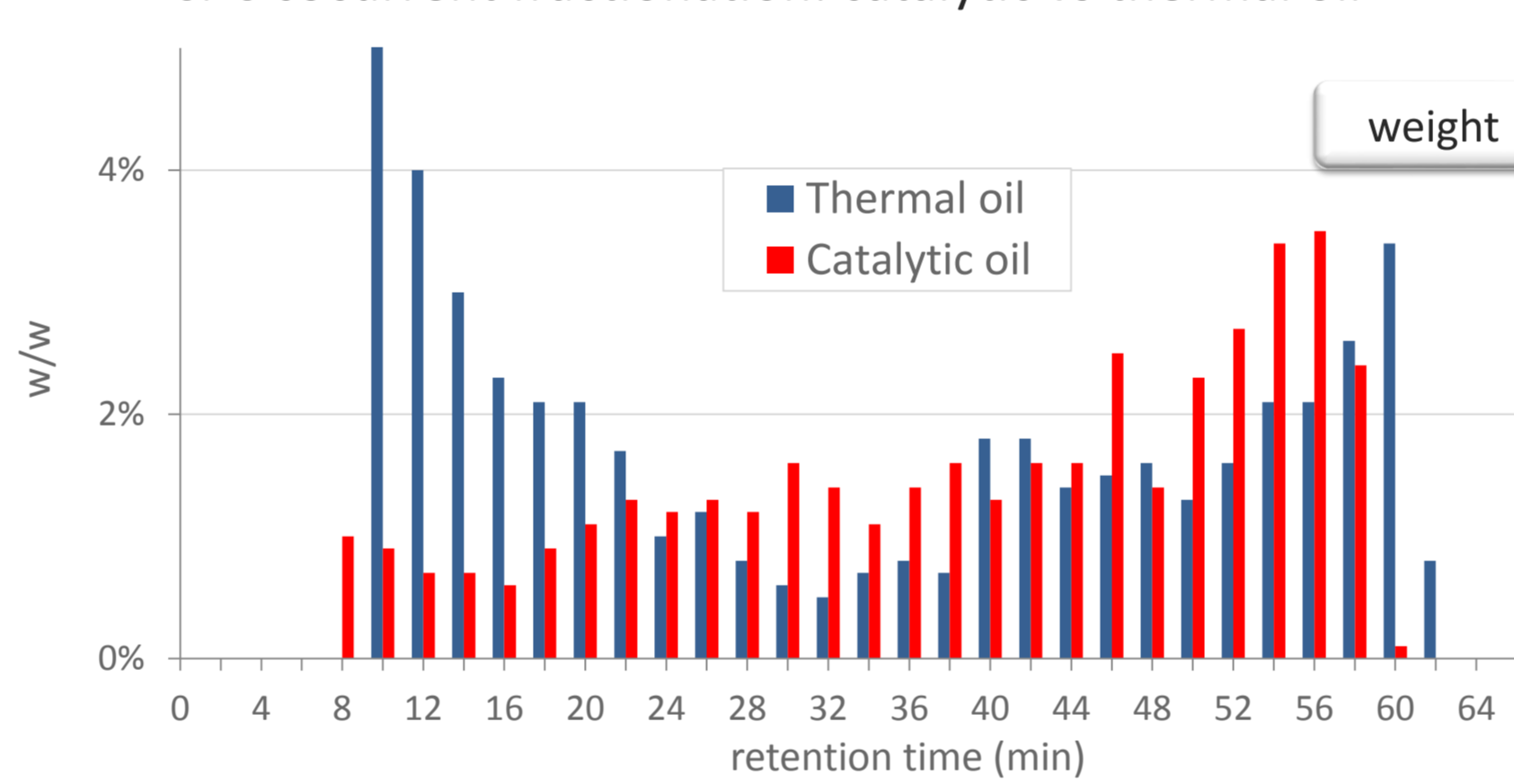
- **Very large retention range (K up to 200): "cocurrent elution" stationary phase gradient**
 - The liquid stationary phase is slowly pushed forward to speed up the separation

Simulation of cocurrent elution: effect of Φ_{stat} gradient start time



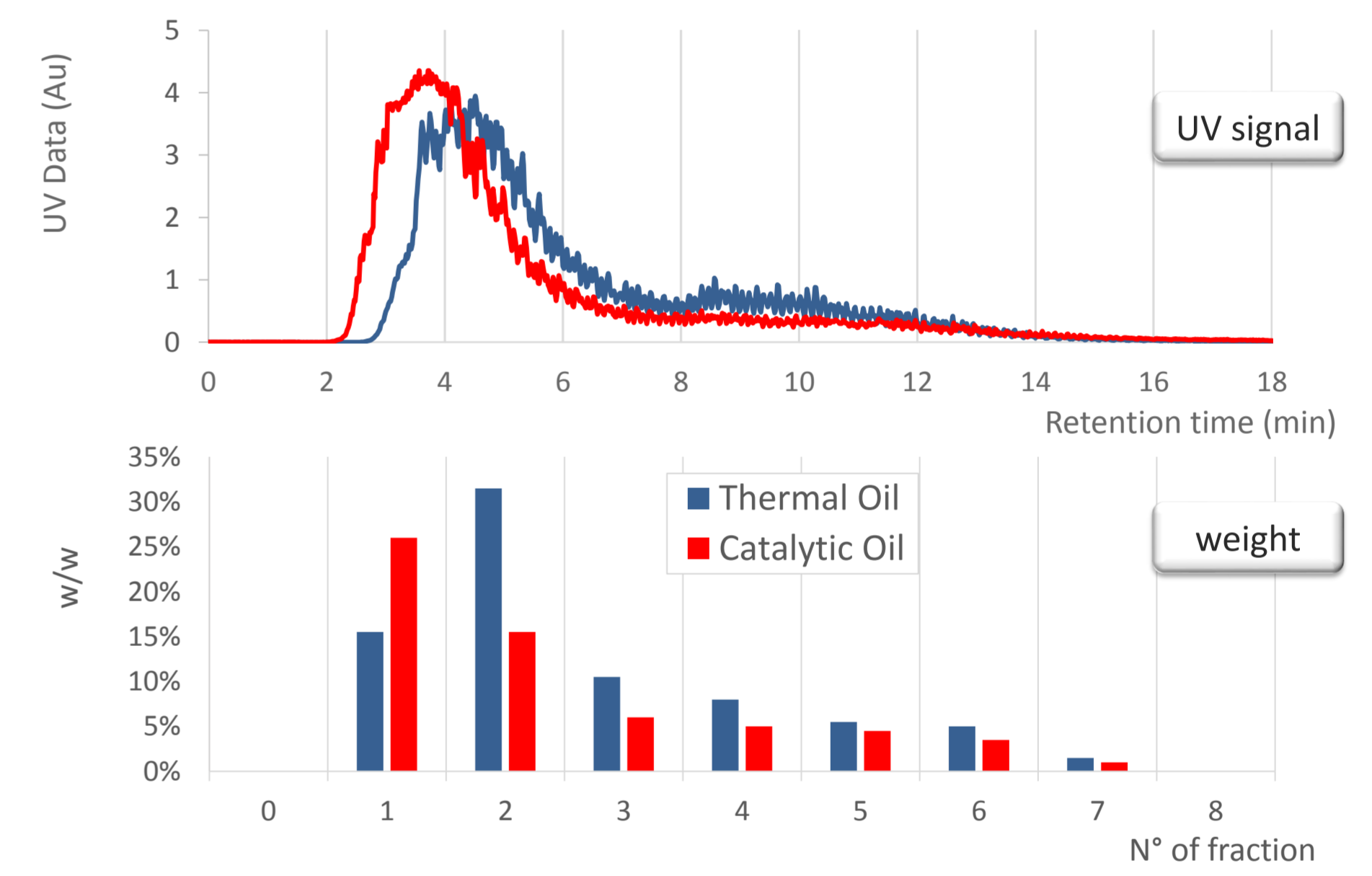
Vc 65.5 mL
Cocurrent elution: ϕ_{mob} 5 mL/min; ϕ_{stat} 1 mL/min
Gradient program: Vs initial 14.5 mL Vs final 30 mL

CPC cocurrent fractionation: catalytic vs thermal oil



SS: Toluene/Acetone/Water (descending mode)
Cocurrent elution: ϕ_{mob} 5 mL/min + ϕ_{stat} 1 mL/min
Start time = 20 min; Vinj = 1 mL (100 mg/mL) in ϕ_{stat}

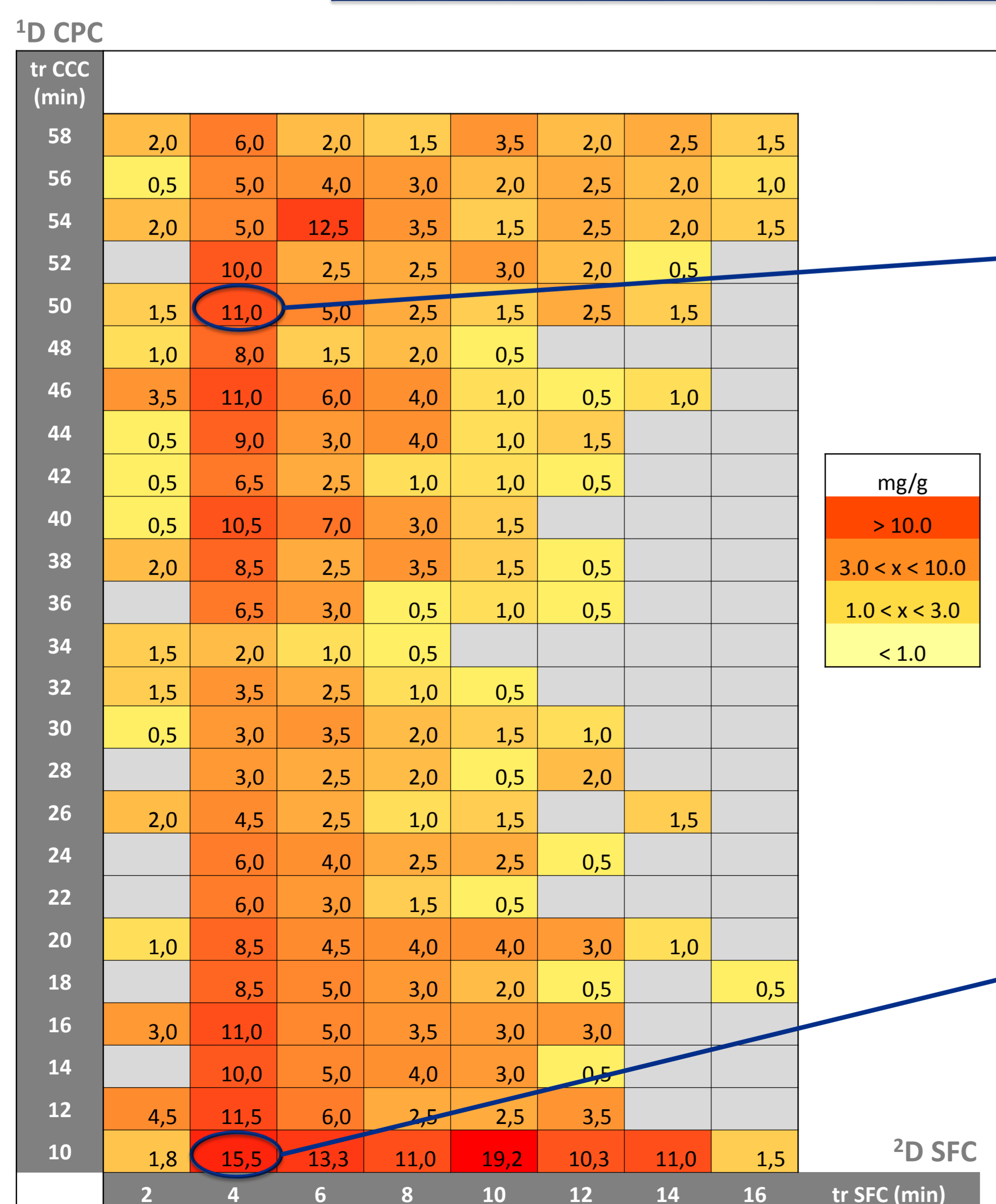
2D: SFCprep



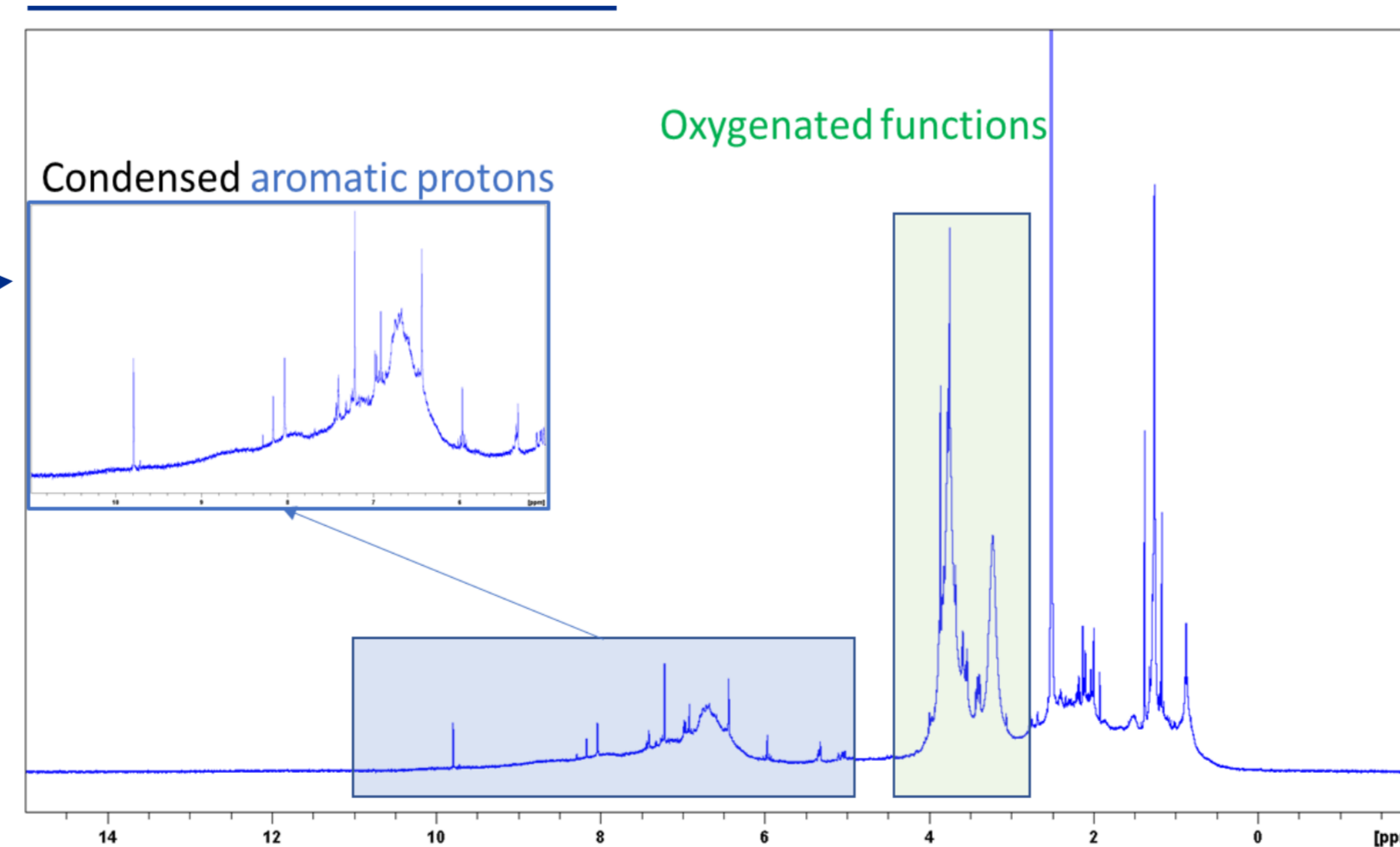
Zorbax silica column (150 x 4.6 mm, 5 μ m)
Modifier: Methanol, make-up solvent: Acetonitrile
Vinj = 100 μ L (50 mg/mL) in THF

Isolation of high molecular weight compounds by CPC x SFC

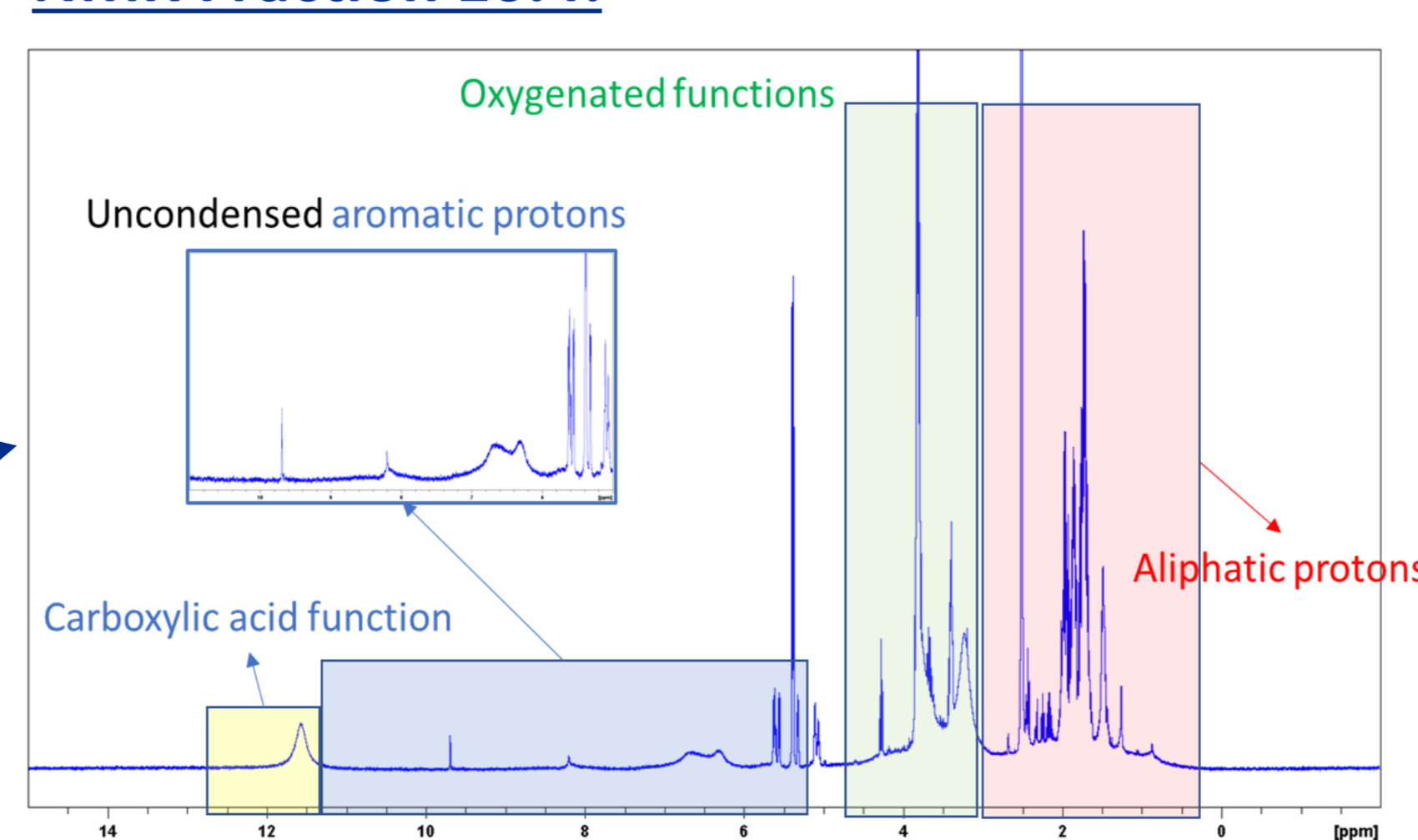
Thermal oil fractionation by CPC x SFC – mass balance



NMR Fraction 50.4:



NMR Fraction 10.4:



Conclusion

	1D: CPC retention	2D: SFC retention
Interactions	π - π	OH
Thermal Oil	-	+
Catalytic Oil	+	-

- Catalytic pyrolysis is known to improve aromatic ring condensation and deoxygenation

2D Preparative CPC x SFC

- ✓ Large 2D occupation rate (75%)
- ✓ Production of mg-scale fractions
- ✓ Easier NMR identification

- These new tools can help in the characterization of bio-oils, to improve catalytic process and hence the valorization of biomass energy.