

Cloud Point Analysis and Big Data as a Potential Alternative to SEC-data?

(How) can we compare SEC data?



SCM-11

SEC and big data

SEC at BASF

- Currently ~20 SEC systems running in parallel
- 13 Lab technicians
- Divided over 2 floors
- Several hundreds (>250) of internal clients with all different chemistries
- People work on different project and changes frequently
- Hard to keep the entire overview.....

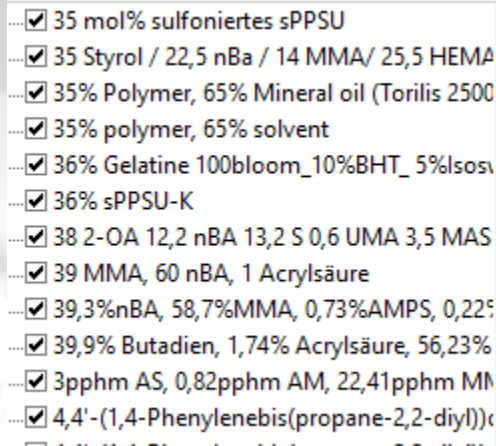
... especially if:

- Samples are only sent once per 3 months (low frequent)
- Samples are not always measured by the same person in the lab (due to holidays, etc.).
- Same (similar) samples sent by different people (from the same client)

SEC and big data

Where to run what?

- In theory, it is the chemistry (monomer composition) that (should) decide, where to measure what chemistry due to many years of experience with columns and solvents...
- In daily practice, each client have a different understanding of chemistry:



...
 35 mol% sulfoniertes sPPSU
 35 Styrol / 22,5 nBa / 14 MMA/ 25,5 HEMA
 35% Polymer, 65% Mineral oil (Torilis 2500
 35% polymer, 65% solvent
 36% Gelatine 100bloom_10%BHT_ 5%Isosv
 36% sPPSU-K
 38 2-OA 12,2 nBA 13,2 S 0,6 UMA 3,5 MAS
 39 MMA, 60 nBA, 1 Acrylsäure
 39,3%nBA, 58,7%MMA, 0,73%AMPS, 0,22%
 39,9% Butadien, 1,74% Acrylsäure, 56,23%
 3pphm AS, 0,82pphm AM, 22,41pphm MM
 4,4'-(1,4-Phenylenebis(propane-2,2-diyl))c

... especially if:

- Very complex mixtures
- Different notations for compositions
- Internal clients very hard to change their habits (“due to historical reasons, we cannot change it”)
- No unique names used (AA=acrylic acid or acrylamide)

SEC and big data

So what, why bother?

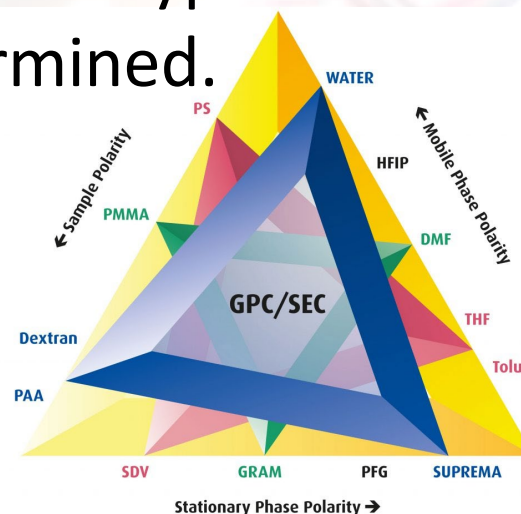
- Clients sent samples in general for data comparison with data from the past.
- No good comparable data => no happy client => remeasure, waste of time

What do you need?

- Some kind of indicator that tells me, similar chemistry runs well in e.g. THF on this column type.
- This indicator should be simple to be determined.

POLARITY

Before we continue, let's first check what SEC can compare well...



SEC and big data

How well can we compare SEC data?

Works well

- Determination of relative molar mass
- Only samples measured under identical chromatographic conditions can be compared
- Same chemistry within a SEC run
- Routine like data processing, like standard samples, with identical integration limits
- Always the same chemistry on the same columns

Is not so easy

- Determination of absolute molar masses (requires special detectors)
- Different chromatographic conditions leads to wrong interpretation
- Individual measurements with large time intervals
- Non routine samples always need special attention where integration limits are set and might depend on the order in which they are measured
- Measuring samples with different chemistry within a single run.

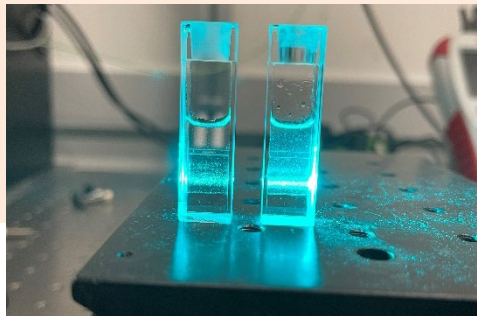
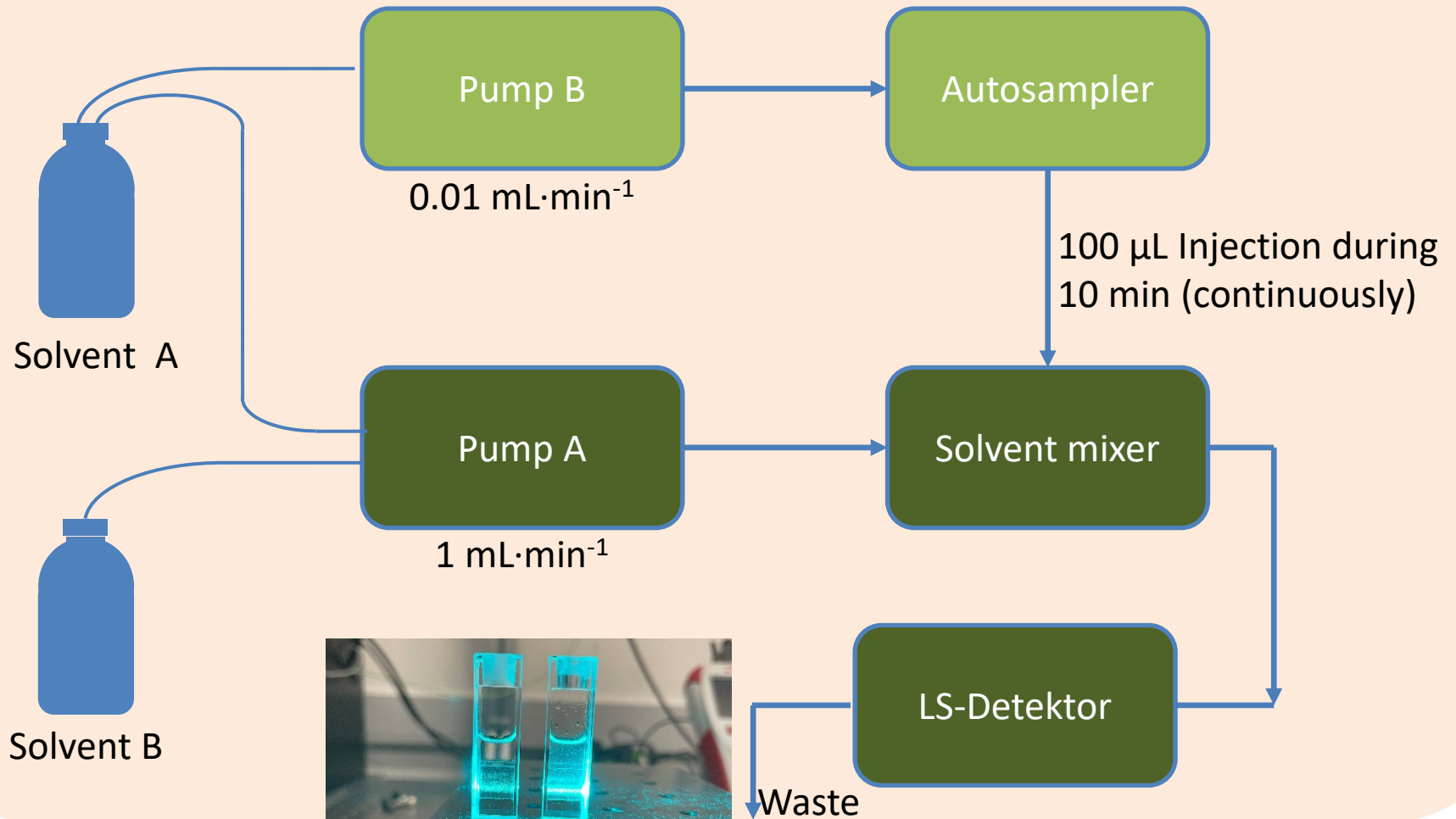
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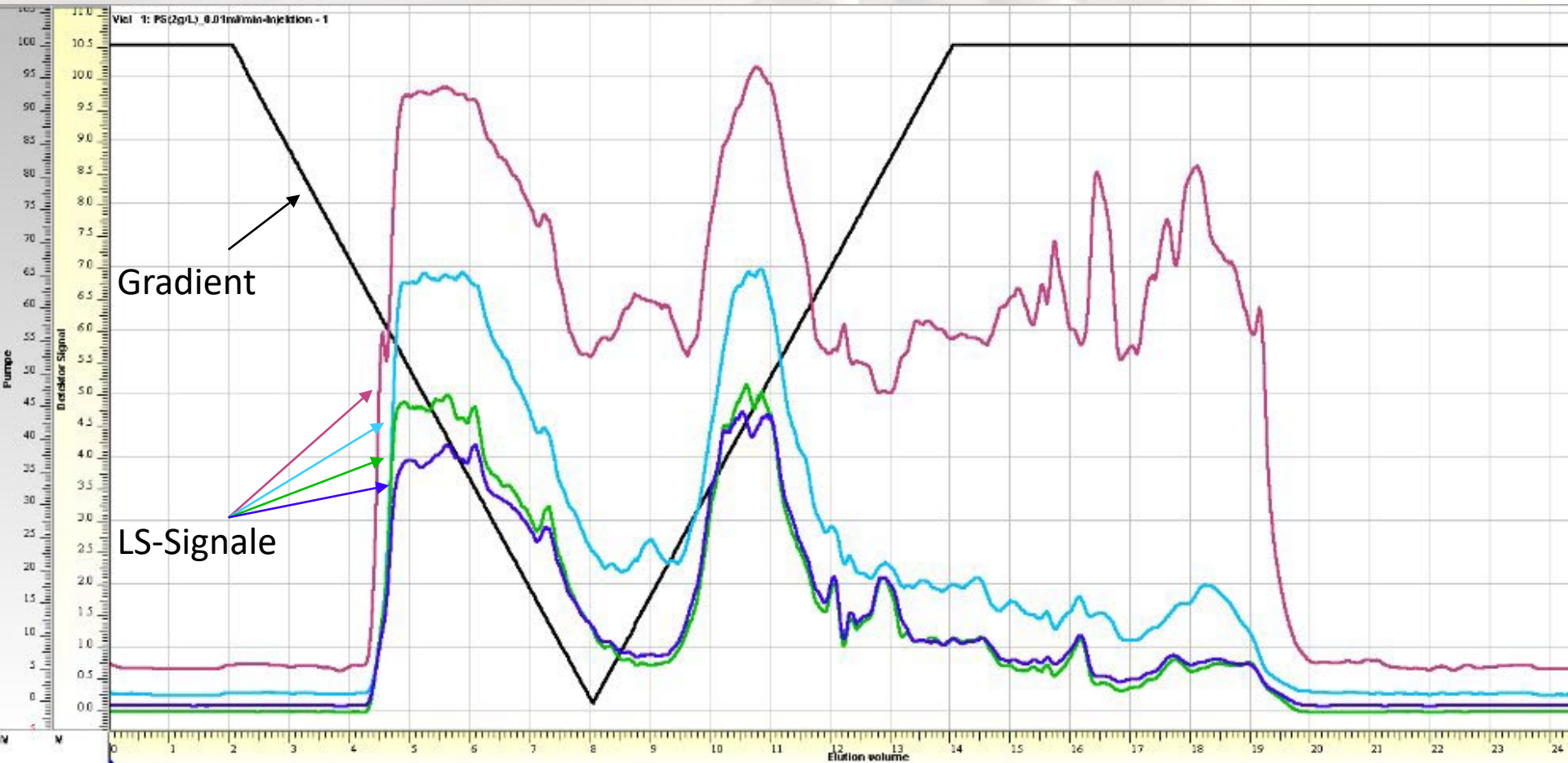
- Solution: LC without column by re-using the SEC solutions => determination of cloud points

SEC and big data

Setup



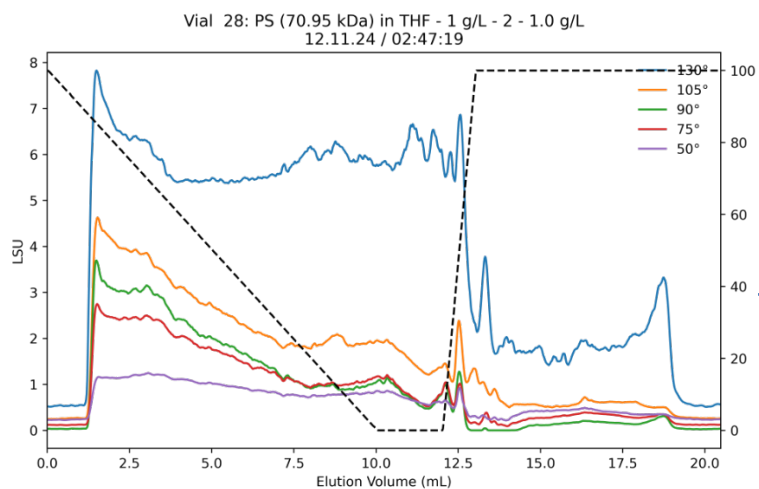
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Only a linear gradient makes sense....

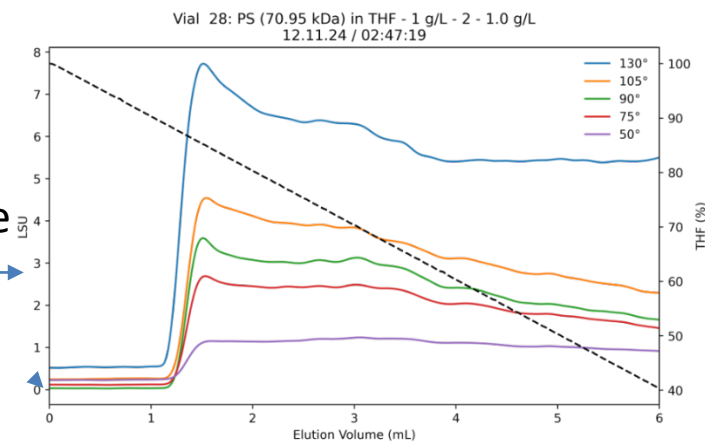
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■ Data processing

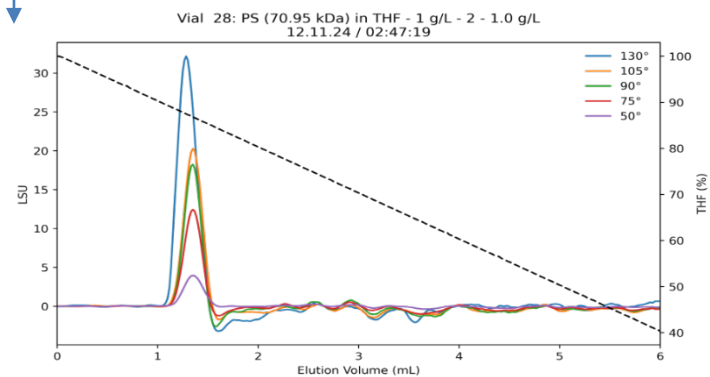


Raw data

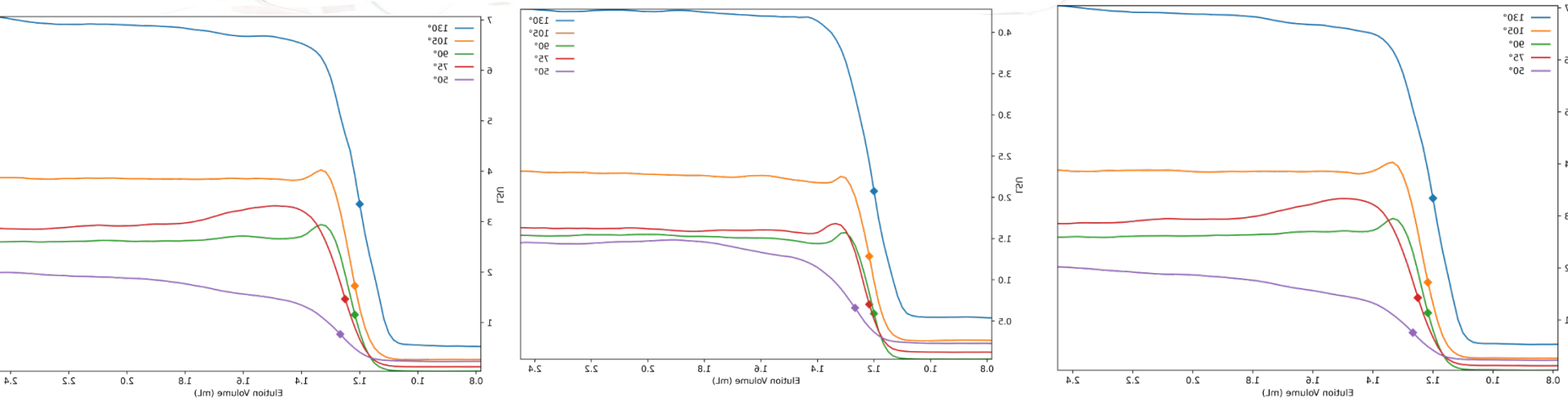
Reduce noise



1st order derivative

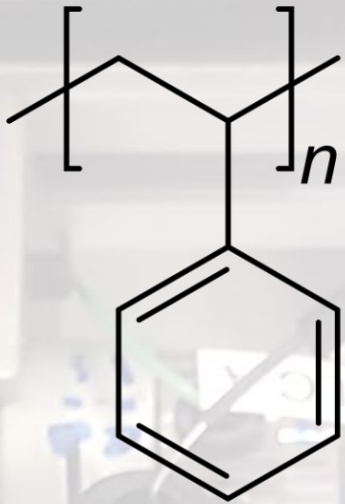


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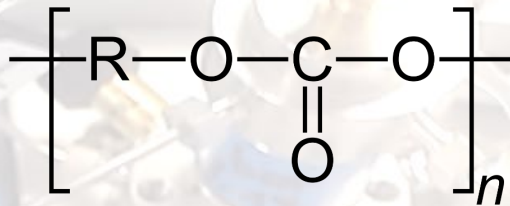


	130°	105°	90°	75°	50°
#1	88.35	88.18	88.35	88.18	87.66
#2	88.34	88.14	88.14	87.78	87.64
#3	88.38	88.16	88.16	87.82	87.67
# 4	88.34	88.52	88.52	88.34	87.47
# 5	88.35	87.83	87.83	87.67	87.67
(%THF)	88.35	88.17	88.20	87.96	87.62
s (% THF)	0.02	0.24	0.26	0.29	0.09

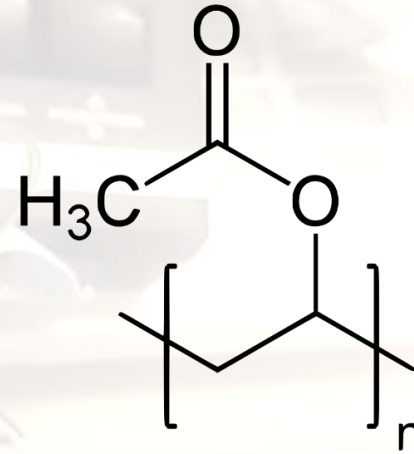
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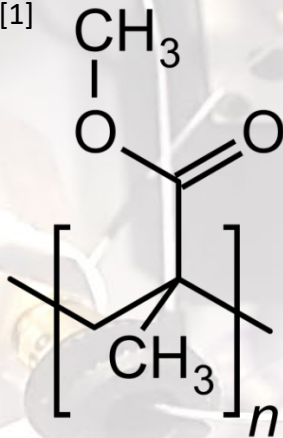
Polystyrol (PS)^[1]



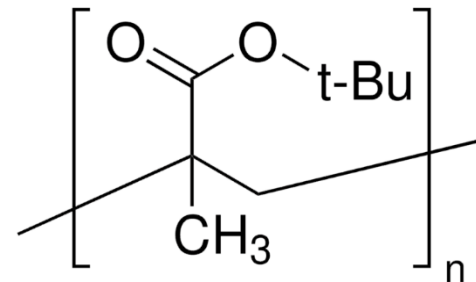
Polycarbonat (PC)^[2]



Polyvinylacetat (PVAC)^[3]



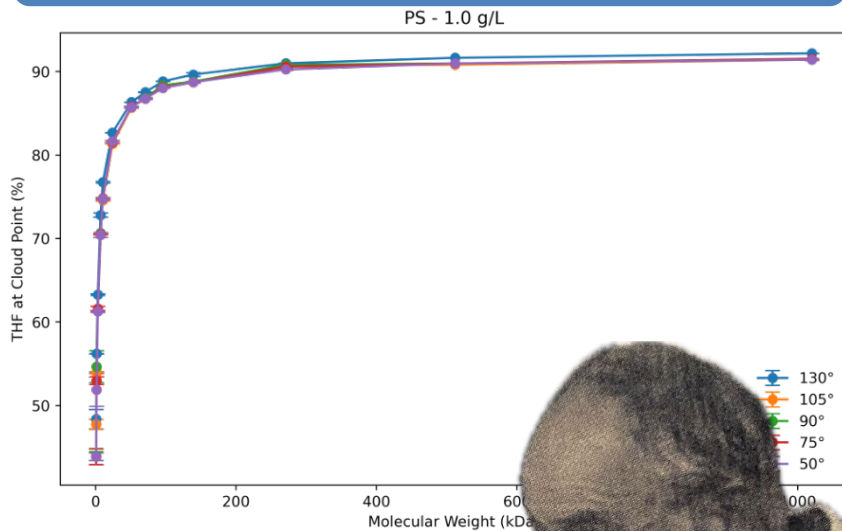
Polymethylmethacrylat (PMMA)^[4]



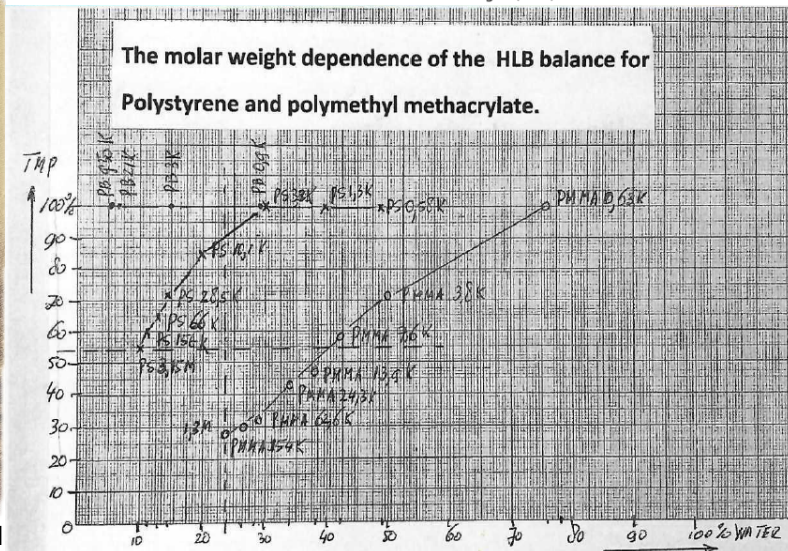
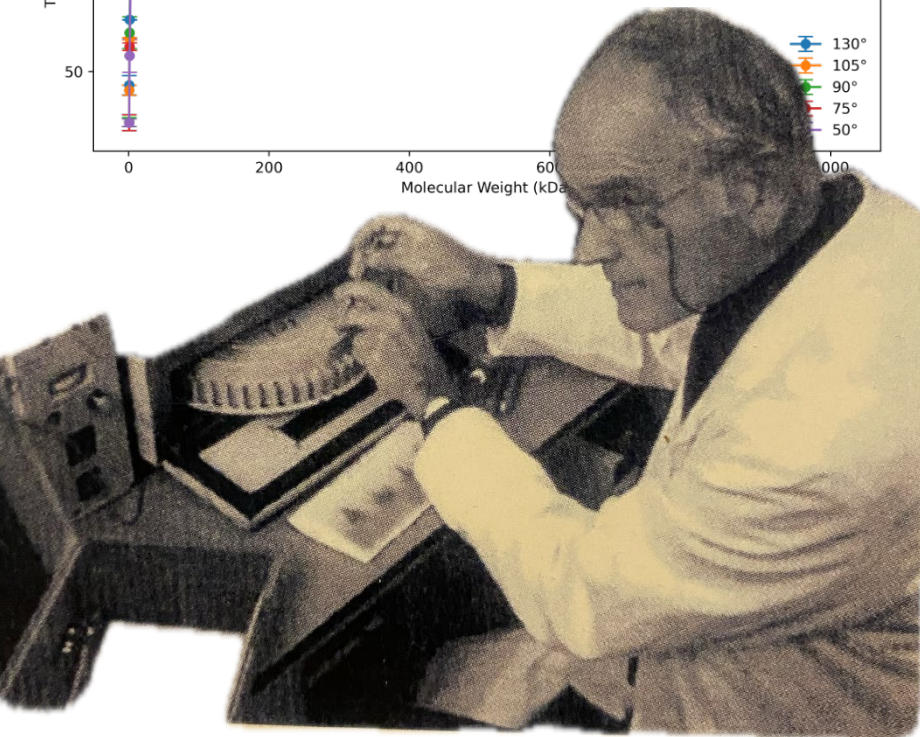
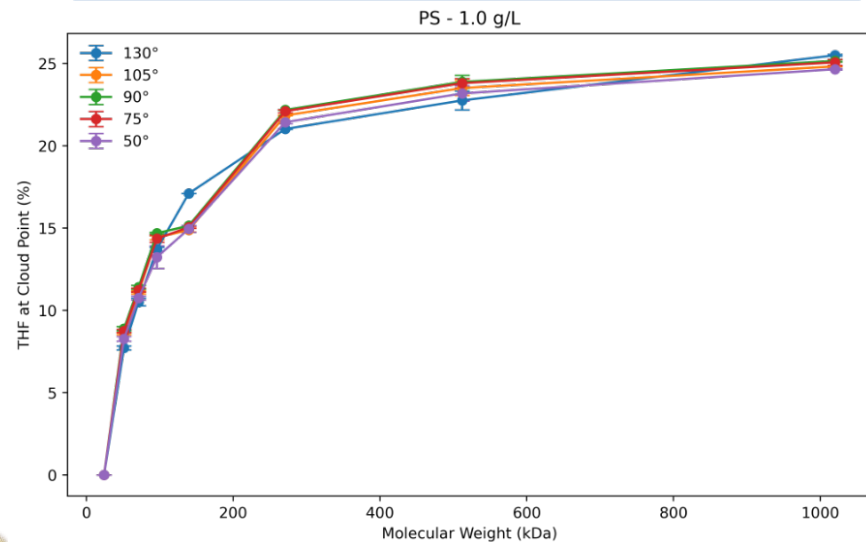
Poly(tert-butylmethacrylat) (PtBMA)^[5]

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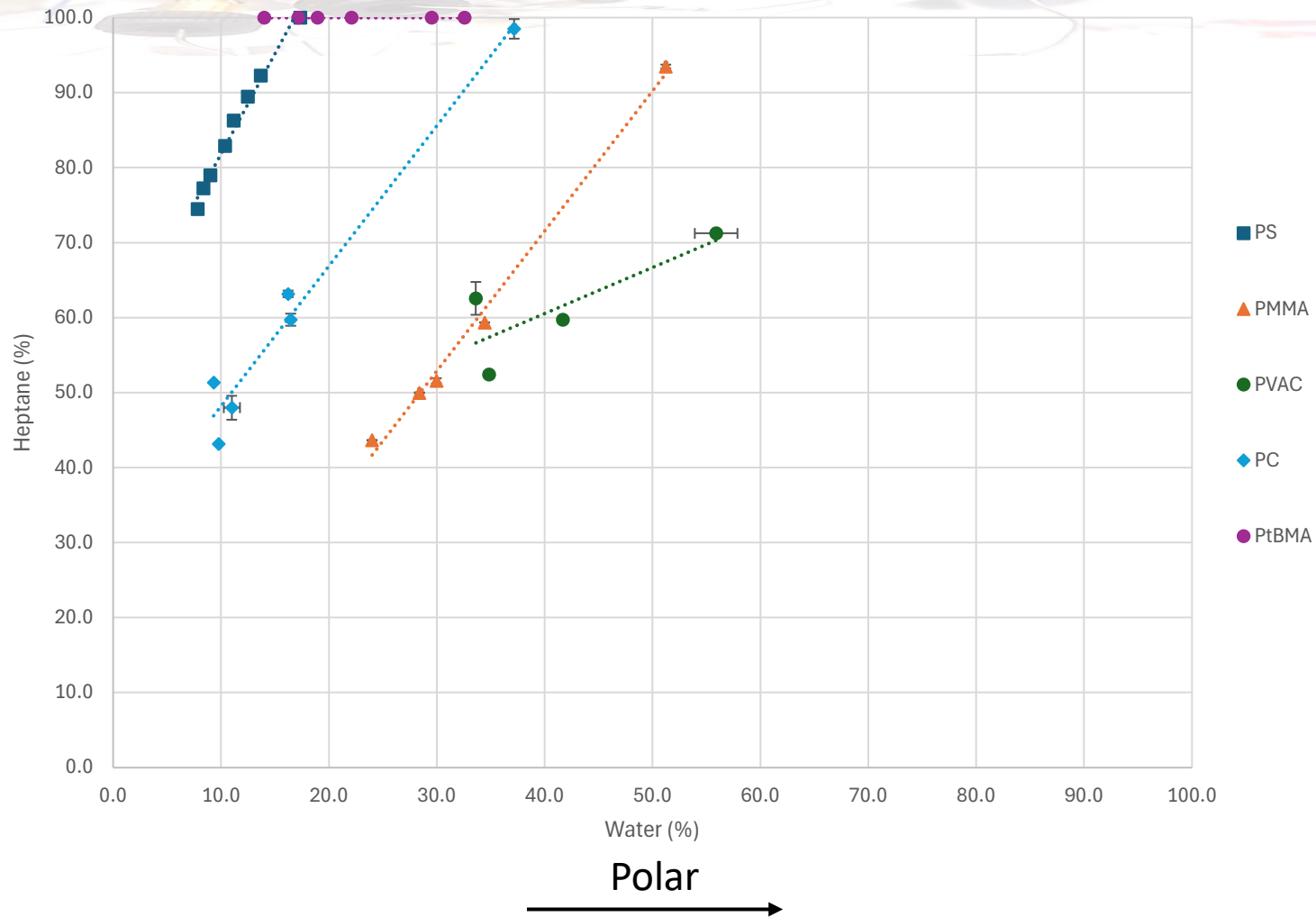
THF \rightarrow H₂O (polar)



THF \rightarrow n-Heptan (unpolar)

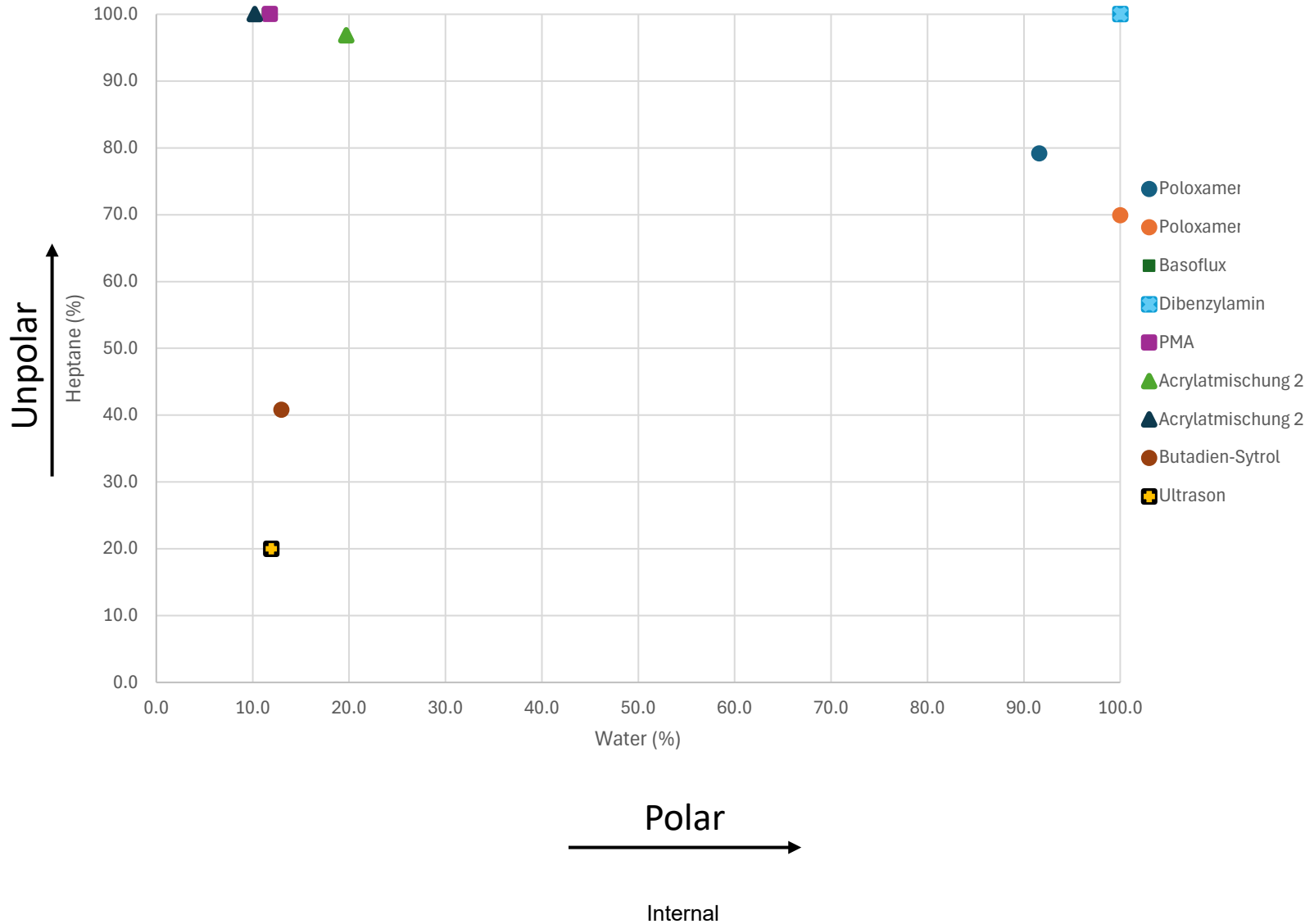


SEC and big data



Plotting the non-solvent compositions shows a linear trend?

SEC and big-small data



SEC and big data

Conclusion:

- Plotting polar vs non-polar gives a very clear indication of the “kind” of polymer we are dealing with.
- Reproducibility is very good.
- This could be a very powerful tool for clustering SEC data which need to be seen in time (current dataset is way too small).

SEC and big data

Outlook

- Device should be “attached” to an auto sampler.
- Light scattering detector currently, has a too large cell volume.
- Device should be minimized in terms of flows ($\ll 1\text{mL}/\text{min}$)



Thanks for listening!!!

There is no break like a coffee break 😊



Questions: please, try
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