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Application possibilities of preparative size exclusion chromatography in a HTE workflow

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Preparative SEC

Preparative size exclusion chromatography can be used to purify compounds or to fractionate the molecular weight distribution of a sample in a straightforward manner. Due to the larger diameter of the preparative column, large sample amounts (compared to normal SEC systems) can be injected, in order to obtain larger amounts of fractions for further analysis in a HTE workflow.
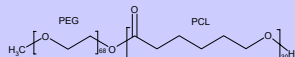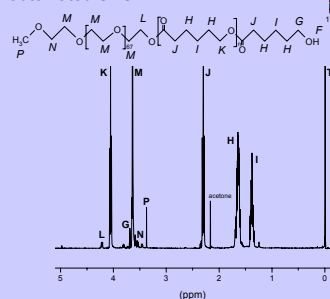
Viscometer
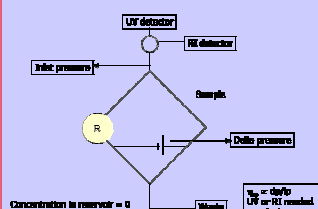
SEC with viscometry allows the determination of absolute molecular weights utilizing the universal calibration method. Two different signals are measured (inlet pressure and differential pressure) to obtain the specific viscosity. The intrinsic viscosity can subsequently be calculated using a RI or UV signal.
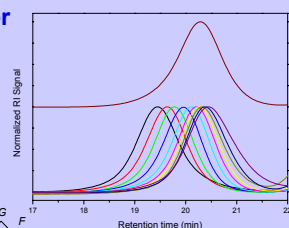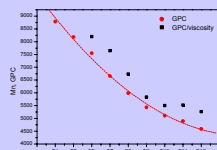
PEG-PCL block copolymer

A PEG-PCL block copolymer was fractionated and all fractions were subsequently analyzed by automated SEC.


Fraction	Ratio PEG/PCL Mol%
F1	0.84
F2	1.02
F3	1.22
F4	1.45
F5	1.89
F6	2.09
F7	2.32
F8	2.45
F9	2.51
F10	2.68

^{1}H-NMR was used to evaluate the ratio between PEG / PCL of the fractionated polymers. The Figure shows an example with assigned peaks.

SEC results obtained by a conventional PEG calibration in comparison with the results from SEC with viscometer. The viscosity measurements can be used together with the calculated ratios of PEG/PCL to obtain the absolute degree of polymerization of the two blocks.
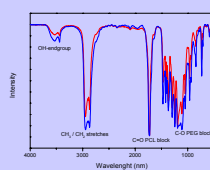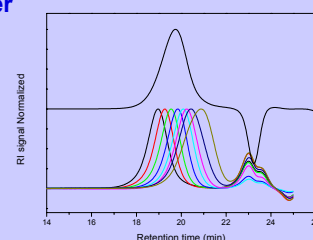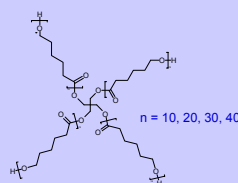

IR measurements obtained with a plate reader setup represent a very useful tool in the HTE workflow for determining the presence of certain functionalities. All samples are spotted on a multiple sample plate and automatically measured.

Star-shaped PCL polymer

n = 10, 20, 30, 40

A PCL star-shaped polymer (n=40) was fractionated in a similar way as the linear PEG-PCL block copolymer. The fractions were analyzed by SEC, MALDI and NMR. From the NMR data, using the CH_2-OH end-group signal the absolute molecular weight can be calculated.
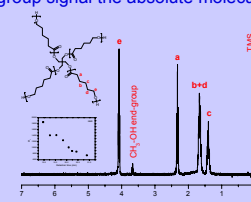
Automatic spotting of MALDI samples was used to measure MALDI-TOF-MS spectra of every fraction. The resulting M_p values from MALDI were used to make a SEC calibration curve.

Using the elution volume from SEC and the M_p value from analyzing the fractions by MALDI, a "absolute" calibration curve could be obtained. The M_n values plotted obtained from MALDI and the M_n values obtained from the "absolute" calibration are in excellent agreement.

Four star-shaped PCL polymers with different M/I ratios were characterized by conventional SEC (PEG Calibration), NMR and SEC with viscosimetry in order to compare the different analytical techniques.

Conclusions

We could show that fractionation can be used to separate block and star polymers. The fractions were analyzed automatically by SEC, NMR, MALDI and IR to show the possibilities of preparative size exclusion chromatography in a HTE workflow.
Combining the NMR results from the fractionated linear PEG-PCL block copolymer with viscosimetry the absolute amount of repeating units can be determined. For the star-shaped PCL polymer, the fractions were used to prepare a "absolute" calibration curve.
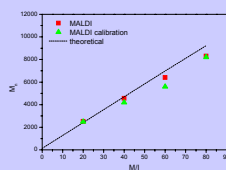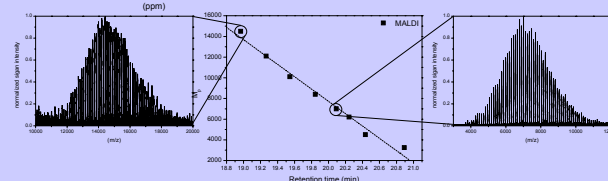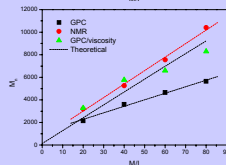
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