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Supporting Information to "Development of a Chemically Sensitive Online SEC Detector Based on FTIR Spectroscopy

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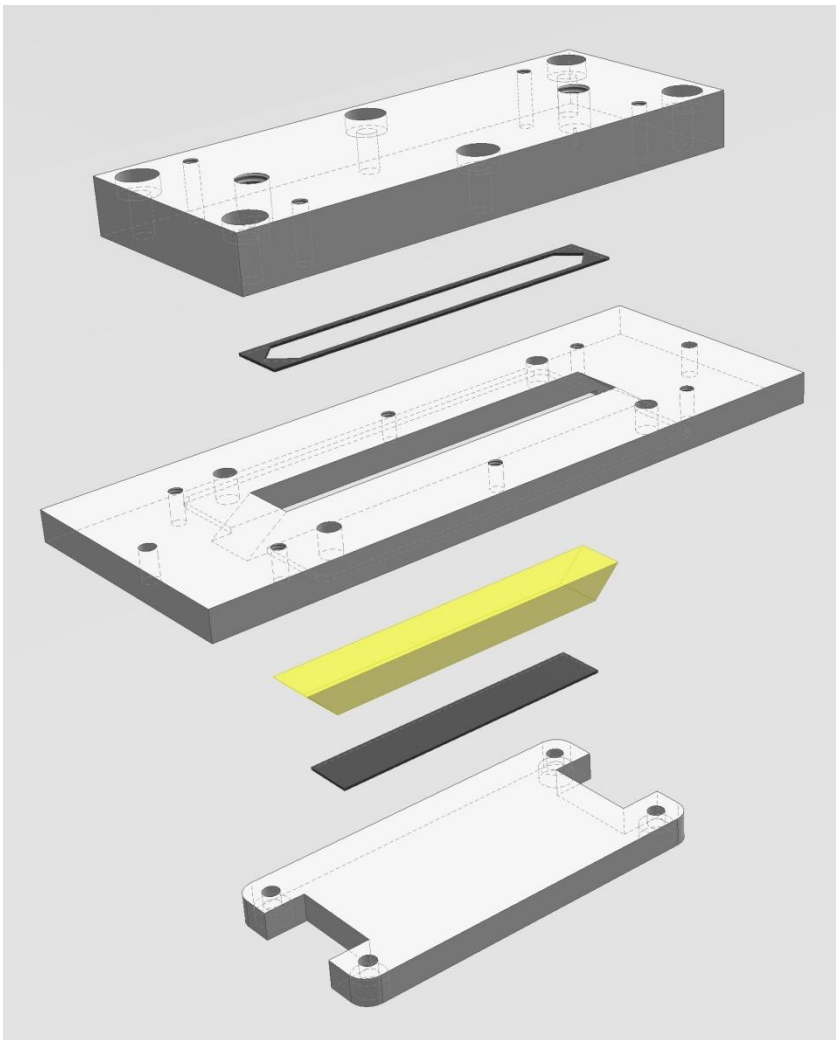
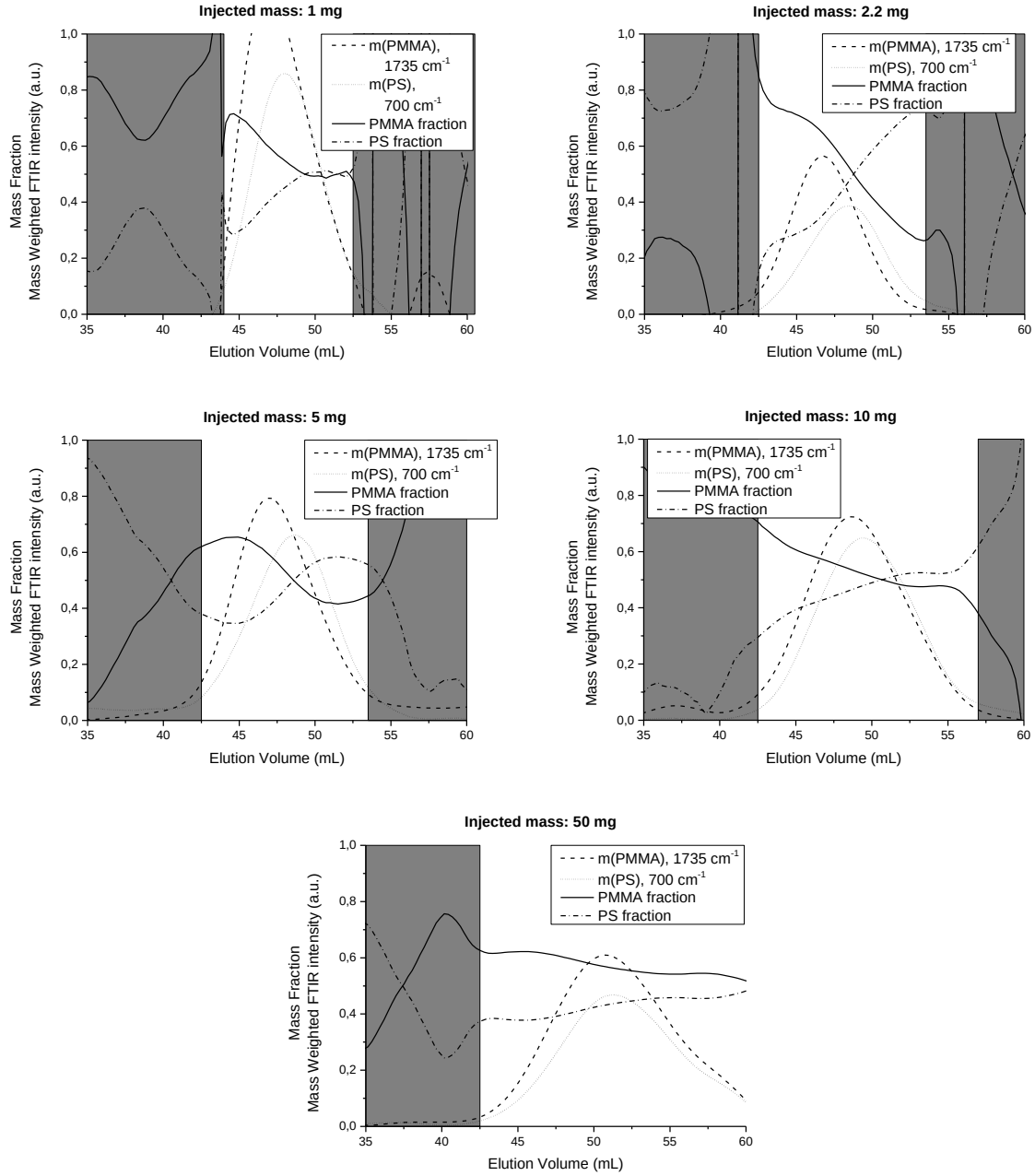
Flow cell construction



The flow cell consists of a top plate from stainless steel with ¼-28-SEC connections, which is screwed to the aluminum base plate. The counter plate at the very bottom presses all inner parts together. The ZnSe-ATR-crystal (dimensions: 7.14 cm x 1 cm x 0.6 cm) is surrounded by two pieces of perfluorinated elastomer sealing (FFKM, Kalrez®). The upper sealing defines the thickness, the inner geometry and the inner volume of the flow cell. The FFKM material is resistant to organic solvents as well as water. Therefore the cell is universally applicable to different SEC applications.

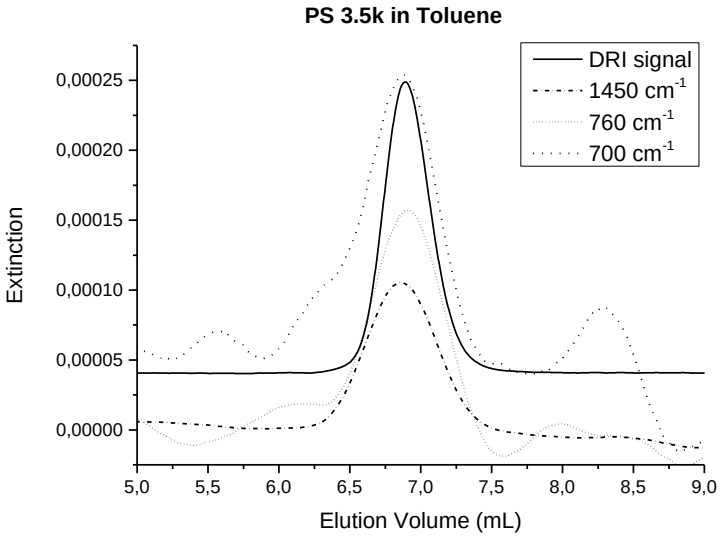
Influence of Overloading on the CCD



A PS-b-PMMA copolymer that shows changes in the chemical composition distribution (CCD) was injected several times with different volumes into a SEC system with TIMO detection to demonstrate the influence of overloading to the CCD. The eluent was THF at 1 mL/min. A semi preparative linear S SDV column was used.

Lower injected masses show a very limited S/N, which narrows the analyzable range in the elution volume. Higher injected masses and overloading broaden not only the peaks but also the CCD curves become flatter. Nevertheless, the general trend is still valid: higher molecular weight fractions are rich in PMMA, while smaller molecules show a higher PS content. A compromise between S/N and overloading effects has to be found for each analyzed sample, where the user can decide to focus on either S/N in TIMO detection or better SEC separation.

Polystyrene in Toluene



A PS sample (M_w = 3.5 kg/mol, PDI = 1.09) was injected (0.2 mg) into a analytical SDV column (8 x 300 mm, particle size: 5 µm, pore size: 50 Å) operated with toluene at 0.16 mL/min flow rate. Polystyrene and toluene have a very similar IR spectrum. Therefore many PS peaks overlap with shoulders of toluene peaks and the chromatograms showed high noise levels, e.g. the trace at 700 cm^{-1} (phenyl vibration) is too noisy for further analysis. Nevertheless the trace at 760 cm^{-1} showed to be good enough for identification and the peak at 1450 cm^{-1} can even be quantified.