

MOLAR MASSES OF BLOCK COPOLYMERS DETERMINED BY SEC AND LCCC – A LC-NMR STUDY

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ABSTRACT

The determination of molar masses of copolymers is well established. The most common used method is size exclusion chromatography (SEC) in order to obtain the number and weight average molar masses M_n and M_w , respectively. An alternative for the determination of molar masses of an individual block of copolymers is the usage of liquid chromatography at critical conditions (LCCC). In this case the critical block is invisible (molar mass independent) and the other visible block experiences preferably SEC or liquid adsorption chromatography (LAC) conditions. Nevertheless, the calculation of the molar masses for SEC or LCCC is mainly based on calibration standards, e.g. homopolymers of different molar masses having the same structure as the blocks of the copolymer. Therefore, these relative procedures can provide inaccuracies of molar masses due to different hydrodynamic volumes of copolymers and homopolymers.

The lecture will demonstrate different ways of determining molar masses with SEC and LCCC. In this case, both chromatographic separation techniques were coupled to NMR in order to get access to the individual comonomer units. NMR is one of the most powerful detectors for liquid chromatography.

In particular, the direct determination of molar masses by using intramolecular referencing as well external referencing methods will be shown. The intramolecular referencing method will be applied to SEC-NMR and LCCC-NMR. The data will be compared to the common relative methods of SEC and LCCC. Furthermore, new information to the concept of invisibility of the critical block in LCCC experiments of copolymers will be presented.