3.2 Chromatography and Separation Science (P. Schulze)

As a central facility the department provides scientific services such as isolation and analysis of compounds in chemical mixtures. A variety of modern chromatographic and electrophoretic separation methods as well as hyphenated techniques are available. Since September 2010 the group is headed by P. Schulze.

Gas chromatography (U. Häusig)

The gas chromatography laboratory provides the equipment for modern gas chromatographic techniques such as high temperature GC, GC x GC and GC-MS. Recently, a dynamic head space system has been installed enabling the pre-concentration of volatile samples. This is advantageous if complex matrices are present and small sample concentrations have to be analyzed. The MLS-GC enabled the preparative isolation of volatile compounds present in very complex mixtures. The distillation laboratory purified solvents and various substrates.

Beside routine analysis, low-cost gas bags for the analysis of gas samples have been developed (Figure 1). They were used for kinetic GC determinations of autoclave reaction gases. In contrast to commercial Tedlar® bags, the septum-tightened sample device is reusable and only the bag itself has to be replaced (€ 0.25 each). The inertness of the polymer material is somewhat lower than of commercial bags.

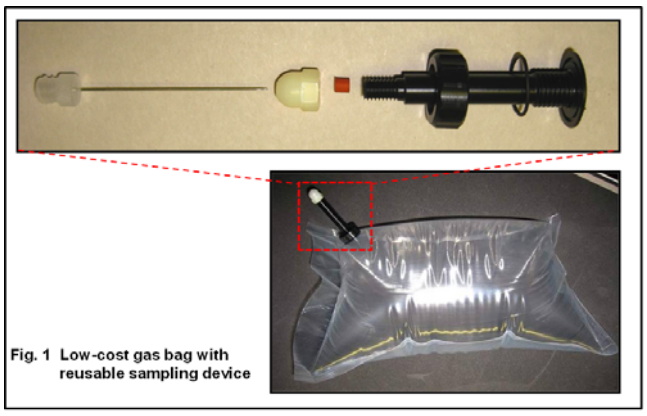

Fig. 1 Low-cost gas bag with reusable sampling device

Liquid chromatography and electrophoresis (A. Deege)

The liquid chromatography and electrophoresis laboratory applies liquid phase separations e.g. high pressure liquid chromatography and (capillary) electrophoresis. In 2008-2010 the liquid chromatography group focused on the increase of separation efficiency and the shortening of analysis time. For this reason, ultra-high-pressure systems (≥ 60 MPa) with achiral sub-2µm- and chiral 3µm-columns have been installed, resulting in higher plate numbers and significantly faster LC runs. Furthermore, a hyphenated UHPLC/MS-IT-TOF system was installed enabling the identification of unknown products or the characterization of sample contaminants. For fast chiral

determinations a standard HPLC system was upgraded to chiral supercritical fluid chromatography. The LC group was involved in the following projects:

- fast chiral separation of e.g. 150 racemic mixtures of organometallic catalysts and natural materials
- optimization of the efficiency of semi-preparative separations of synthetic materials for NMR and MS
- screening issues, for example ~ 64.500 conversion- and *ee*-determinations of epoxide hydrolases and ~ 25.500 analyses of hydroxylated steroids (Figure 2)

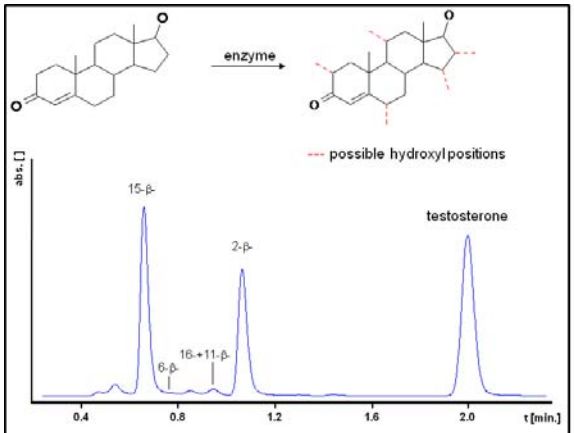

Fig. 2 Separation of hydroxylated stereoids

Two HPLC systems were equipped with valves for column switching. One is a combination of achiral 2µm columns and chiral 3µm columns enabling the separation of complex mixtures with subsequent chiral analysis of selected signals ("heart cut"). This supersedes purification steps of reaction mixtures with thin layer chromatography or solid phase extraction. The second system was utilized for simultaneous determination of saccharides and their reduction products with an improvement in analysis time by a factor of 3.

In (semi-) preparative HPLC covalently bounded stationary phases as well as chiral anion exchangers were introduced. The latter were used for separation of e.g. chiral phosphoric acids, chiral sulphonic acids and their imides.

Recently, the capillary electrophoresis section was modernized with an instrument utilizing very sensitive photodiode array detection. It was used for chiral electrophoresis of enantiomers and also for the separation of ionic liquids (Figure 3).

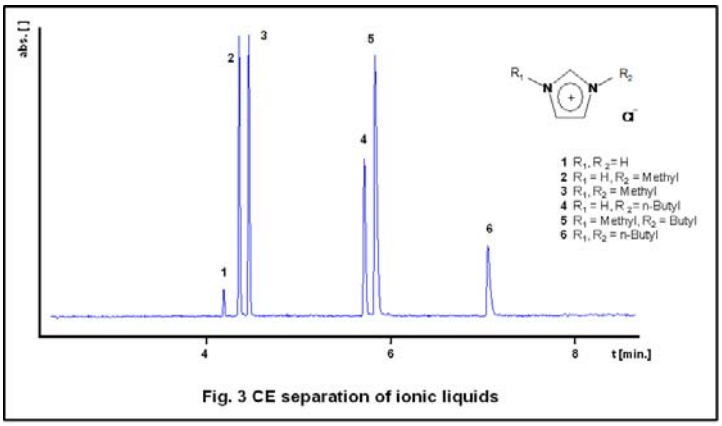

Fig. 3 CE separation of ionic liquids

Publications resulting from this research area: 411

Cooperations: O. Trapp (Heidelberg, DE)