

3.2 Chromatography and Electrophoresis (P. Schulze – 10 FTE)

The department is divided into three subgroups: the gas chromatography, the analytical high pressure liquid chromatography, and the preparative HPLC team. Each team provides analytical services for in-house scientists, such as qualitative and quantitative determinations, chiral analysis, and preparative purifications.

The gas chromatography service is executed by five employees using about 25 GC systems, e.g. split/splitless injection, (dynamic) headspace GC, high/low temperature GC, GC-MS, cooled injection systems with and without the combination of a thermal desorption unit. To gain superior chromatographic performance, mostly capillary columns and hydrogen carrier gas are used combined with different detectors like flame ionization or thermal conductivity detection. Unknown substances are identified using quadrupole mass spectrometric detection. About 10.000 GC runs are measured per year.

In the analytical HPLC laboratory a variety of instrumental liquid phase separation techniques is applied by three employees. We provide one- and two-dimensional HPLC (heart-cutting or comprehensive) and supercritical fluid chromatography with different detectors like UV diode arrays, refractive index detection, evaporative light scattering, electrochemical detection or the hyphenation to TOF- or qMS. Achiral and chiral separation columns for reversed-phase, normal-phase and ion exchange modes are available in particle sizes from sub 2 μm to 3 μm . During the reporting period, low temperature HPLC down to -20°C was introduced to increase the chromatographic efficiency and to analyze temperature-sensitive compounds. The team also provides micro-preparative HPLC purifications of small substance amounts for spectroscopic characterization. About 5800 runs are performed per year.

Three employees of the preparative liquid chromatography purify mainly reaction batches with separation columns of 10 to 50 mm inner diameter. A variety of stationary phases for reversed-phase, normal-phase or enantioselective separations are available as well as instrumentation for heart-cut HPLC and peak recycling HPLC. Semi-automatic fraction work-up is performed in rotary evaporators and a high vacuum pump. Roughly, 80 samples with a maximum substance amount between 5 and 4000 mg are processed per year. Additionally, a walk-on service is provided for scientists who would like to process urgent samples on their own.

During the reporting period the group collaborated with the University of Erlangen-Nürnberg and the University of Pavia. A third party funded ZIM-AiF project (BMW i) about online HPLC-Raman detection was conducted in cooperation with the IUTA, the University of Düsseldorf, and the companies PSS GmbH and CS-Chromatographie Service GmbH.