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High-resolution bioactivity profiling combined with hyphenated HPLC-SPE-NMR – investigation of functional food and plants with antifungal constituents

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Nature is a rich source of bioactive constituents, but traditional bioactivity-guided fractionation is a time-consuming and laborious task involving several preparative-scale chromatographic steps. In recent years, the hyphenation of analytical-scale high-performance liquid chromatography with solid-phase extraction and nuclear magnetic resonance spectroscopy, i.e., HPLC-SPE-NMR, has proven successful for full structure elucidation directly from crude extracts without any prepurification steps [1]. This even includes acquisition of direct-detected $^{13}$C NMR spectra, database-assisted NMR structure elucidation and off-line assessment of circular dichroism spectra for assignment of absolute configuration. However, the basic HPLC-SPE-NMR setup does not give any information about the bioactivity of individual constituents in the crude extract. Thus, the recent coupling of microplate-based high-resolution bioassays with HPLC-SPE-NMR, i.e., HR-bioassay/HPLC-SPE-NMR, represents one of the most promising new developments for advancing research in bioactive constituents from natural sources like food, plants and microorganisms [2]. A schematic illustration of the workflow in the HR-bioassay/HPLC-SPE-NMR analysis is given below.

In this talk, two recent studies using high-resolution bioactivity profiles for targeting subsequent HPLC-HRMS-SPE-NMR analysis towards bioactive constituents will be presented. In the first study, combined high-resolution radical scavenging and high-
resolution α-glucosidase inhibition profiles were used for targeting HPLC-HRMS-SPE-NMR analysis towards bioactive food constituents in Sea aster (*Aster tripolium* L.) and searocket (*Cakile maritima* Scop.) [3]. In the second study, high-resolution plasma membrane H⁺-ATPase inhibition profiles were used for identifying antifungal compounds chebulagic acid and tellimagrandin II from *Haplocuelum foliolosum* [4].

**References**


