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Stærk, Dan

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

Dan Staerk

Natural Products Research, Department of Drug Design and Pharmacology, Faculty of Health and Medical Sciences, University of Copenhagen, Universitetsparken 2, DK-2100 Copenhagen, Denmark

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,$^2$ plants,$^3$ 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