

**B025 Analysis of non-polar and polar compound classes of *Piper methysticum* Forst. by Coordination Ion Spray – Mass Spectrometry (CIS-MS)***Melanie Gaub*<sup>a</sup>, *Alexander v. Brocke*<sup>b</sup>, *Gudrun Roos*<sup>a</sup>, *Ernst Bayer*<sup>b</sup> and *Karl-Artur Kovar*<sup>a</sup><sup>a</sup> Department of Pharmacy, Eberhard – Karls – University, Auf der Morgenstelle 8, 72076 Tuebingen, Germany. <sup>b</sup> Research Center for Nucleic Acid and Peptide Chemistry, Eberhard – Karls – University, Auf der Morgenstelle 18, 72076 Tuebingen, Germany

For the on-line characterisation of plant extracts LC/ESI-MS is a widely used tool. However, the disadvantage of this method is the poor sensitivity of non-polar components in several plant extracts. Coordination Ion Spray – MS (CIS – MS) helps to overcome this lack of sensitivity, the characteristic weakness of electrospray.

CIS-MS is a new ionisation method in which positively and negatively charged complexes are formed by the addition of a suitable central atom to the analytes, and these complexes can be detected by mass spectrometry. Since both polar and non-polar organic compounds can form coordination compounds with an appropriate central atom, this form of ionisation is highly versatile (1-3).

For on-line LC/CIS-MS, the solution containing ions of a high complex binding affinity with analytes is added by sheath-flow technique. Various metal ions such as silver, copper, nickel, alkaline and earth alkaline metals have already been used for positive ion CIS-MS. Furthermore, central atoms with an electron deficiency such as boron can be used for the formation of negatively charged coordination compounds. CIS is not only an excellent tool in coupling HPLC and CEC with MS by a coaxial sheath-flow interface combining both applicability to a wide variety of compound classes with high sensitivity, but also offers additional structural information when used in combination with MS/MS.

These advantages are demonstrated in our extensive analysis of *Piper methysticum* Forst. (Kava-Kava; Piperaceae). We show the excellent complexation properties of the several kavapyrones leading to a more precise peak identification than LC/ESI-MS.

**References:** 1. Bayer, E. et al. (1999) *Angew. Chem. Int. Ed.* 38: 992. 2. Rentel, C. et al. (1999) *Electrophoresis* 20: 2329. 3. Rentel C. et al. (1998) *Anal. Chem.* 70: 4394.

**B026 Rapid quantification of kavapyrones and water content in Kava-Kava extracts by Near-Infrared Reflectance Spectroscopy (NIRS)***Christoph Roeseler*, *Melanie Gaub*, *Gudrun Roos* and *Karl-Artur Kovar*

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Kava-Kava (*Piper methysticum* Forst, Piperaceae) has been used for hundreds of years as an intoxicating beverage in ceremonial rites and in traditional medicine by Pacific Islanders. In Europe preparations of the plant are used for the treatment of anxiety, restlessness und uptightness. The kavapyrones are known to be the active components and are used for standardization. Several HPLC methods for the determination of the six major components (methysticin, dihydromethysticin, kavain, dihydrokavain, yangonin, demethoxyyangonin) have been published (1-3). Although these methods meet all the regulatory criteria of qualitative and quantitative determination, they are time and solvent consuming.

In our work we show that NIRS is a versatile alternative method for characterising Kava-Kava extracts. It is a rapid, non-destructive and cost-effective method allowing simultaneous determination of the components by multivariate analysis (4). A quantitative NIRS method was established for the determination of kavain, total kavapyrones and water. Reference measurements were performed by HPLC and Karl Fischer titration.

Using Partial Least Squares (PLS) regression, a multivariate calibration was done for the water content, kavain and total kavapyrones using PLS2. Satisfactory calibration statistics were obtained for kavain with a root mean square error of calibration (RMSEC) of 0.05 and a root mean square error of prediction (RMSEP) of 0.06 at a concentration range from 4.5 to 7% in the dry extracts. For total kavapyrones we obtained an RMSEC of 0.08 and an RMSEP of 0.09 at a concentration range from 28.8 to 31.3% and for water we obtained an RMSEC of 0.03 and an RMSEP of 0.03 at a concentration range from 1.5 to 4%. The study emphasizes the potential of NIRS as a rapid and highly effective alternative method to conventional quantitative analysis of plant extracts.

**References:** 1. Ganzera, M., Khan, I.A. (1999) *Chromatographia* 50: 649-653. 2. Boonen, G. et al. (1997) *J. Chromatogr. B* 702: 240-244. 3. Gracza, L. et al. (1980) *J. Chromatogr.* 193: 486-490. 4. Molt, K. et al. (1997) *Pharmazie* 52: 931-937.