The goal of the workshop held on the annual GA-meeting in Kiel was to demonstrate and to discuss the potential of NIRS in the analyses of herbals. The first speaker, Gudrun Roos (Tübingen) presented a short introduction into the method because nearly no NIRS research has been shown on GA manifestations up to now. In contrast to sharp absorption peaks occurring in the MIR region, NIR spectra show very broad bands with a weak intensity. Usually, an assignment of peaks to individual vibrations is therefore not possible. The main advantage of NIRS is high speed, which means that a lot of results can be accumulated within a short period of time. Liquids, powders and even high-viscosity substances can be measured without sample preparation. Furthermore, NIR spectra shows characteristic „fingerprints“ for individual extracts and cover all organic compounds. A herbal preparation can therefore be characterised in its completeness. Qualitative classifications and quantitative analyses are principally possible.

The practical part of the workshop focused on the problem of analysing a high number of samples to get satisfying research results. Hartwig Schulz (Quedlinburg) showed a good correlation between the results of NIRS after establishing a calibration model in comparison to HPLC-results for the analyses of echinacoside in roots of *Echinacea* species. Within a short time it is possible to select echinacoside-rich samples, which is of particular importance for in-cultivation programmes. Schulz proposed to use powdered samples to get optimal results. The calibration has to be done with a relatively high number of representative samples. Problems may occur with the prediction of minor components (content < 1 %), that do not present strong specific (N)IR absorptions. Similar results were presented by Grit Schulzki (Vestenbergsgreuth) for essential oils. NIRS is already well established in the food industry. Validated methods have been developed for the determination of essential oil in fine cut peppermint, fennel and camomile. Applying NIRS, time-consuming steam distillation of herbs could be reduced to every tenth batch, in order to validate the method and to update the calibration function. Additionally, steam distillation is rarely necessary and may only be required in case of unexpected values and to approve OOS-results. NIRS can also be useful to find adulterations: Schulz could discriminate between samples of *Harpagophytum zeyheri* and samples of *Harpagophytum
procumbens. Quantitative analyses of harpagoside and 8-O-p-coumaroyl-hargpagoside were successful as well.

Extract characterisation was another subject of discussion: The fingerprints of NIRS can not be directly compared to fingerprints of chromatographic methods, because the characteristic of a specific compound can not be assigned to single peaks. However Roos showed chemometric assignments in two dimensional graphs for different extracts of Hypericum perforatum. The samples of three extracts (50 % ethanol, 60 % ethanol and 80% methanol) could be discriminated of each other with a 2-D-PCA-Plot of 1100-2500 nm, while with TLC they showed only marginal differences. Preliminary experiments indicate the potential of NIRS as a remarkable tool for validation. However, as in cultivation programmes previously stated, a high number of samples are normally necessary for the validation of processes like powder mixing and tableting. Roos created a model to quantify a specific St. John’s wort extract (Ze117) in the tablet matrix mixture. The prediction of further mixtures showed quantitative results, which were at least as precise as results obtained with HPLC can be! Calibration of a tablet mixture is not as difficult as for a specific compound in herbal substances and could be carried out within a short time. On the base of these results even more samples could be analysed to validate the quality of a mixing process with NIRS instead of using time and solvent consuming HPLC. NIRS is meanwhile accepted as a method by the 4th edition of the European Pharmacopoeia.

Martin Diller from the German Federal Institute of Drugs and Medical Devices (BfArM) commented the new “Note for Guidance on the Use of Near Infrared Spectroscopy by the Pharmaceutical Industry and the Data Requirements for new Submissions and Variations CPMP/QWP/3309/01, Aug 2003”. Up to now, no NIRS-method has been implemented in a dossier-application of a herbal medicinal product in Germany. However, there is no reason for not accepting an NIRS-method if it is justified and validated according to the rules. The discussion showed that the practical experience with NIRS is very different from company to company. In many companies the technique is used to approve identity of inactive ingredients, but not to control quality or processes. New efforts should be focused on making NIRS a reliable tool especially for expensive validation procedures – costs could probably be reduced remarkably. Furthermore, NIRS has a high potential in routine analyses as well, especially in analysing essential oils and water in extracts or herbal drugs. Water analyses have not been discussed and could be a topic for a future workshop.
All the presentations can be studied on the GA-Homepage: www.ga-online.org

Beat Meier, Chair of the permanent committee

Speakers with e-mail and topics:

Chair: Prof. Dr. Beat Meier, Zeller AG, Romanshorn, Switzerland
Questions to discuss
beat.meier@zellerag.ch

Panelists:

Prof. Dr. H Schulz Bundesanstalt Züchtungsforschung, Quedlinburg, Germany
Can NIR replace complex quantitative methods? Example: Echinacea species
Is it possible to characterise extracts by NIRS? Example: Harpagophytum procumbens
bafz-pa@bafz.de

Dr. G. Roos University of Tübingen, Germany
Near Infrared Spectroscopy Introduction into the method
Identification of extracts
Verification of content uniformity in Hypericum perforatum powder blends
g.roos@kraeuterhaus.de

Dr. G. Schulzki PhytoLab GmBH, Vestenbergsgreuth, Germany
Determination of essential oil content in herbal drugs by NIRS
grit.schulzki@phytolab.de

Dr. M. Diller Federal Institute of Drugs and Medical Devices (BrArM), Bonn, Germany.
NIRS in Quality Control: Regulatory Aspects
diller@bfarm.de