Is it possible to characterise extracts by NIRS? 

Example: Harpagophytum procumbens

Hartwig Schulz

Federal Centre for Breeding Research on Cultivated Plants
Institute for Plant Analysis
Quedlinburg
Morphology and valuable substances of devil’s claw

Flowering shoots of *H. procumbens*

Roots and fruit of *Harpagophytum procumbens*

Molecular structure of harpagoside
Cultivation of *Harpagophytum procumbens* in Southern Africa

M. Manley, B.J. Gray, E. Joubert, H. Schulz, 11th Intern. Conference on NIRS 2003, Cordoba (Spain)
Cultivation of *Harpagophytum procumbens* in Southern Africa

M. Manley, B.J. Gray, E. Joubert, H. Schulz, 11th Intern. Conference on NIRS 2003, Cordoba (Spain)
Glucoside iridoids occurring in *Harpagophytum* species

Harpagioside

8-O-ρ-Coumaroyl harpagide

Harpagide

Procumbide
Pharmaceutical products

- antiinflammatory and analgetic effects
  (used against a range of ailments such as headaches, rheumatism and indigestion)
- adulterations of *H. procumbens* with *H. zeyheri* may occur
- South Africa exports approx. 600 tons/year to the U.S. and Europe
TLC and HPLC characterisation of devil’s claw roots

Solvent system: ethyl acetate-methanol-water (77:15:8), visualised with vanillin-sulphuric acid reagent

M. Manley, B.J. Gray, E. Joubert, H. Schulz, 11th Intern. Conference on NIRS 2003, Cordoba (Spain)
NIR reflection spectra obtained from devil's claw roots

Comparison of spectra scanned by a Perkin-Elmer FT-NIR IdentiCheck and a Foss NIRSystems 6500 system

M. Manley, B.J. Gray, E. Joubert, H. Schulz, 11th Intern. Conference on NIRS 2003, Cordoba (Spain)
NIRS calibration for the content of harpagoside in devil’s claw

Reference HPLC determination versus NIR prediction of harpagoside.
Measurements were performed on a Foss NIRSystems 6500

M. Manley, B.J. Gray, E. Joubert, H. Schulz, 11th Intern. Conference on NIRS 2003, Cordoba (Spain)
NIRS calibration for the content of 8-O-ρ-coumaroyl harpagide in devil’s claw

Reference HPLC determination versus NIR prediction of 8-O-ρ-coumaroyl harpagoside. Measurements were performed on a Foss NIRSystems 6500

M. Manley, B.J. Gray, E. Joubert, H. Schulz, 11th Intern. Conference on NIRS 2003, Cordoba (Spain)
PCA plot from FT-NIR spectra of *H. procumbens* and *H. zeyheri*

PCA plot from FT-NIR spectra of 42 dried, ground devil’s claw samples indicating the possible discrimination between the different species *H. procumbens* and *H. zeyheri*

M. Manley, B.J. Gray, E. Joubert, H. Schulz, 11th Intern. Conference on NIRS 2003, Cordoba (Spain)
NIRS calibration equation for harpagoside in an ethanolic/aqueous extract (60:40, v/v) of devil’s claw roots (mg/100 g extract)

\[ R^2 = 0.99 \]
\[ \text{SECV} = 0.855 \text{ mg/100g} \]

Is it possible to characterise extracts by NIRS?

- It is easier to analyse liquid samples than solid plant materials (with respect to homogeneity and concentration levels of the analytes).

- It must be carefully checked whether absorption bands of the solvent overlap with specific bands of the analyte.

- Generally, the influence of temperature is very high at NIRS measurements. Therefore measurement conditions must be standardised.

No general approach is possible! Feasibility studies must be performed individually for every product!