51th Annual Congress of the Society for Medicinal Plant Research

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Is it possible to characterise extracts by NIRS? Example: Harpagophytum procumbens

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Morphology and valuable substances of devil's claw



Flowering shoots of *H. procumbens*

$$\begin{array}{c|c} & & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & \\ & & \\ & & \\ & \\ & & \\ & \\ & & \\$$

Molecular structure of harpagoside



Roots and fruit of

Harpagophytum procumbens

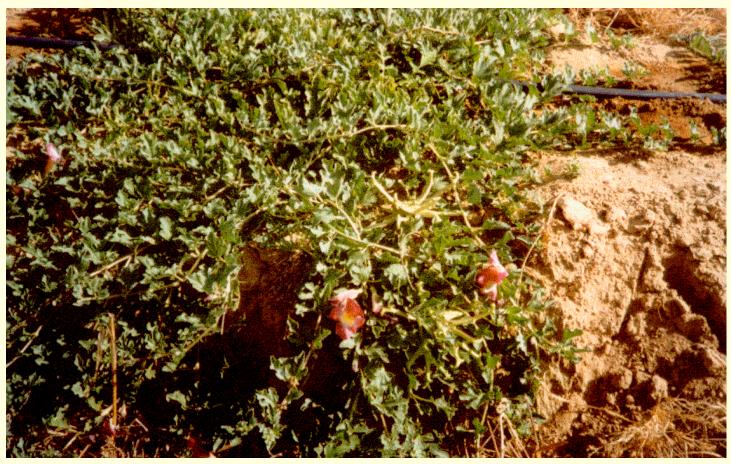
Cultivation of Harpagophytum procumbens in Southern Africa



M. Manley, B.J. Gray, E. Joubert, H. Schulz, 11th Intern. Conference on NIRS 2003, Cordoba (Spain)



Cultivation of Harpagophytum procumbens in Southern Africa



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Glucoside iridoids occurring in Harpagophytum species

Harpagoside

$$\begin{array}{c} \text{HO} \quad \text{OH} \\ \\ \text{HO} \quad \text{CH}_3 \quad \text{O-β-D-Glucoside} \end{array}$$

Harpagide

8-O-ρ-Coumaroyl harpagide

HO OH
HO
$$CH_3$$
 O- β -D-Glucoside

Procumbide



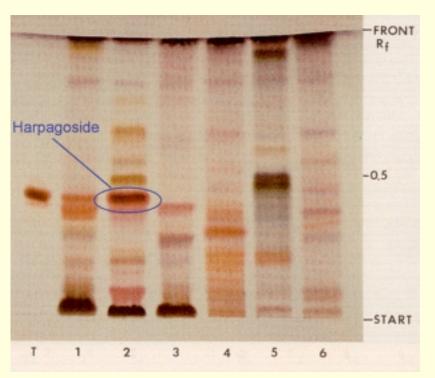
Pharmaceutical products

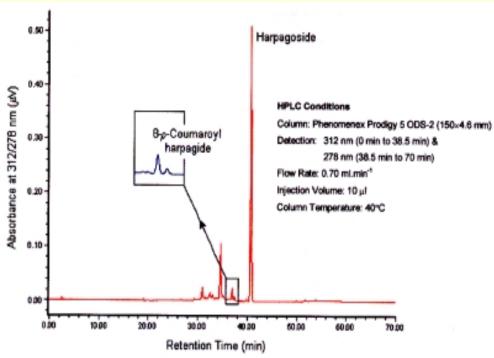
- antiinflammatory and analgetic effects
 (used against a range of ailments such as headaches, rheumatism and indigestion)
- o adulterations of *H. procumbens* with *H. zeyheri* may occur
- South Africa exports approx. 600 tons/year to the U.S. and Europe





TLC and HPLC characterisation of devil's claw roots

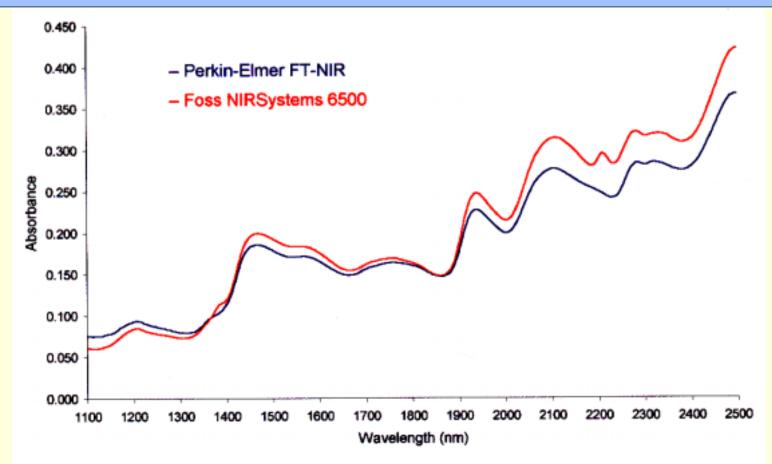




Solvent system: ethyl acetate-methanol-water (77:15:8), visualised with vanillin-sulphuric acid reagent



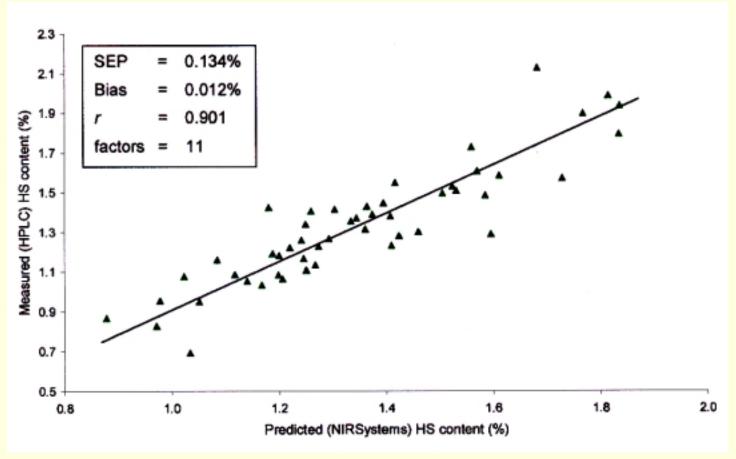
NIR reflection spectra obtained from devil's claw roots



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



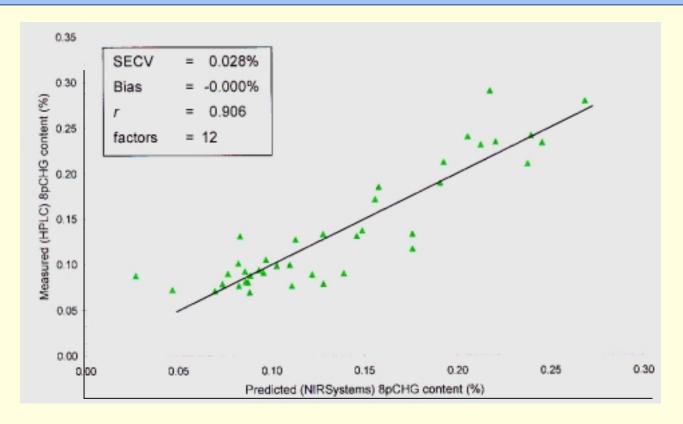
NIRS calibration for the content of harpagoside in devil's claw



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



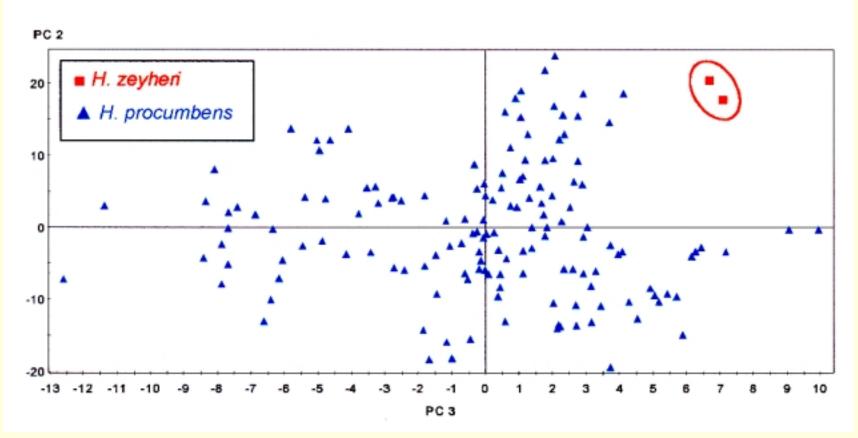
NIRS calibration for the content of 8-O-ρ-coumaroyl harpagide in devil's claw



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



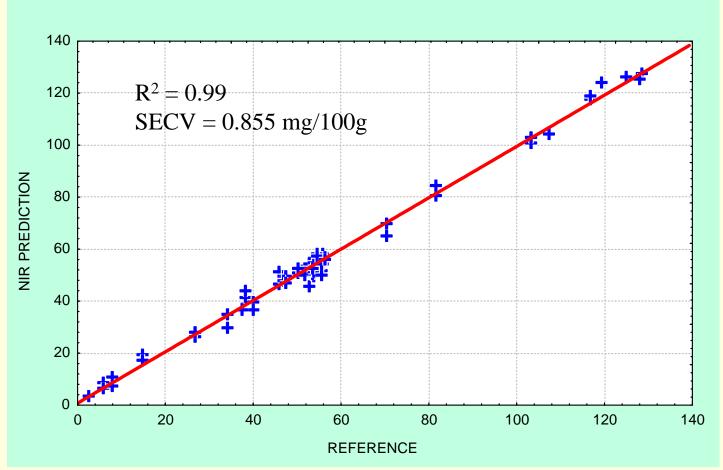
PCA plot from FT-NIR spectra of *H. procumbens* and *H. zeyheri*



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*



NIRS calibration equation for harpagoside in an ethanolic/aqueous extract (60:40, v/v) of devil's claw roots (mg/100 g extract)



H. Schulz, B. Steuer, S. Pfeffer, H. Krüger, NIR News 13, (2002), 10-11



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 analyt
- 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!

