the nature network

PhytoLab

Determination of essential oil content in herbal drugs by NIRS

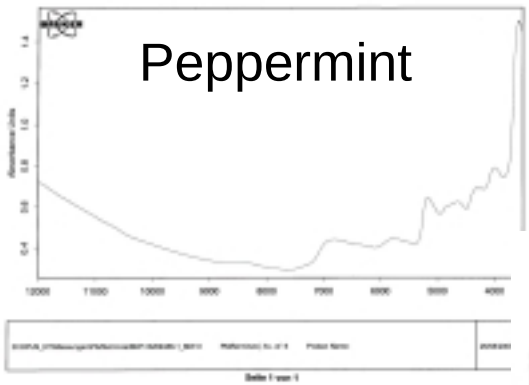
Dr. Grit Schulzki

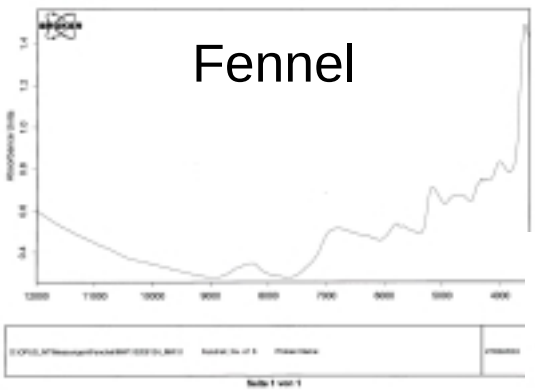
PhytoLab GmbH & CO. KG

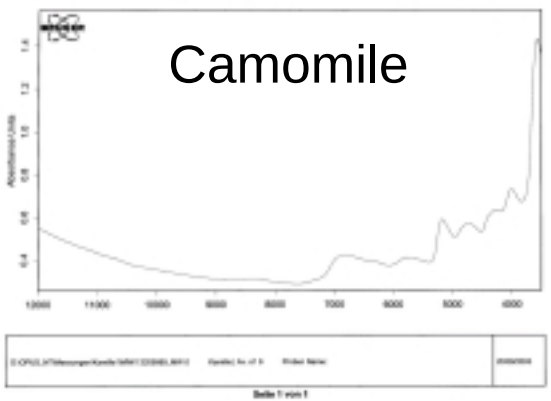
General consideration

- Development of a method for rapid analysis of essential oil with low costs
- fine cut material of peppermint, fennel and camomile
- determination by classical steam distillation is time consuming, 2 - 4,5 hours
- specific methods for each matrix
- collection of calibration data from routine samples during a period of 2 years

NIR-Spectra







Peppermint calibration data

Calibrated range:

0,9 - 1,9 % V/M essential oil, specification minimum 1 %

Number of NIR Spectra included

307 (calibration 271, test 31)

Cross Validation

R^2=69,87 RMSECV=0,100

Test Set Validation

R^2=70,40 RMSEP=0,106

Fennel calibration Data

Calibrated range:

1,9 - 7,2 % V/M essential oil, minimum specified 1 %

Number of NIR Spectra included

173 (calibration 150, test 18)

Cross Validation

R^2=92,48 RMSECV=0,263

Test Set Validation

R^2=91,97 RMSEP=0,327

Camomile calibration Data

Calibrated range:

0,17- 0,49 % V/M essential oil, minimum specified 0,2 %

Number of NIR Spectra included

213 (calibration 179, test 21)

Cross Validation

R^2=47,03 RMSECV=0,043

Test Set Validation

R^2=38,73 RMSEP=0,0437

Quality assurance - 1

Specificity

- method is specific for the defined matrix
- measurement of unknown matrices results in high M-distances
- maximum value is defined

Reproducibility

determined with peppermint matrix (n=6)

- average content: 1,52 %
- standard deviation: 0,036
- coefficient of variation: 2,4 %

Quality assurance - 2

Comparison with the reference method in case of :

- OOS-results (below the specified minimum)
- unexpected values, content out of calibration range
- M-distance out of range
- every tenth batch; (also for collection of new data for recalibration)

Measurement of control samples and collection of data in quality control charts

Comparison of NIR and distillation results

	NIR	Distillation	Comment
Fennel	3,36	3,15	
	3,38	3,15	
	3,17	4,25	calculated as peppermint, M-dist. out of range
Peppermint	1,67	1,73	
	1,17	1,15	
	1,53	1,54	
	1,50	5,71	calculated as fennel, M-dist. out of range
Camomile	0,32	0,40	
	0,33	0,40	
	0,43	0,58	matrix not included in calibration

Summary

- Valid methods could be developed for the determination of essential oil in peppermint, fennel and camomile fine cut
- the most precise method for fennel
- for camomile with the lowest oil content and the smallest calibration range sufficient accuracy in comparison to oil distillation
- NIR determination of essential oil is regarded as an equivalent method to steam distillation and could replace Ph.Eur. Method 2.8.12
- advantages: short analysis time, low costs
- disadvantage: long preparation time for data collection and method development

Thank you for your attention!