

# Purity Control of Chemical Substances using Infrared Spectroscopy

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# 1.Introduction

## Quality control

Purity and identity control is the basic and most important step for quality control in chemical and pharmaceutical industries.

## Infrared Spectroscopy

Universal workhorse for identification of organic substances. Finger print regions  
Highly specific method.



Aim:

Find parameters which are suitable for describing the difference or similarity between the spectrum of a sample and a reference

1. Parameters are based on linear regression:

$$Y = aX + b \text{ or } X = aY + b$$

Standard error of regression (describes dissimilarity)

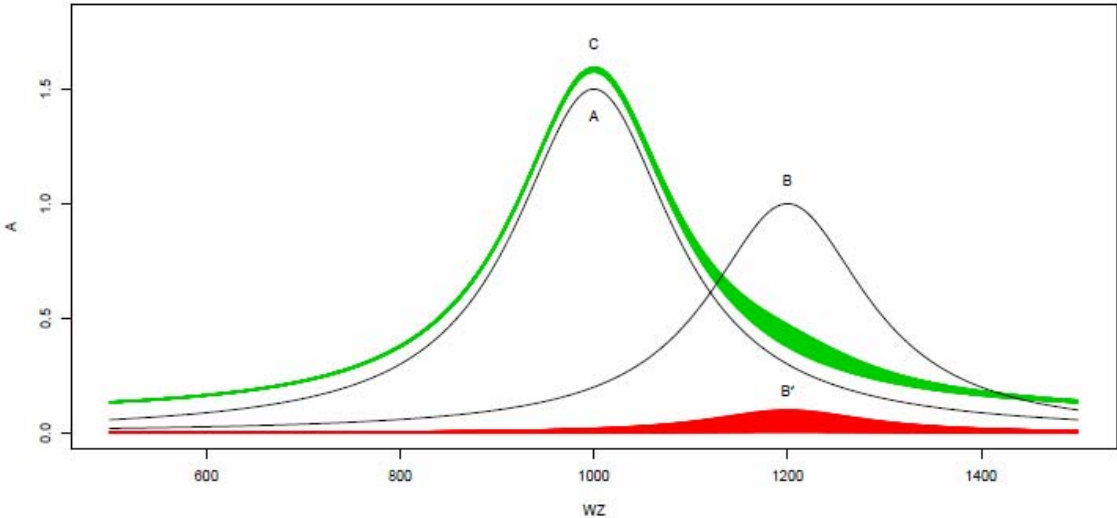
Correlation coefficient R (describes similarity).

2. Difference spectroscopy

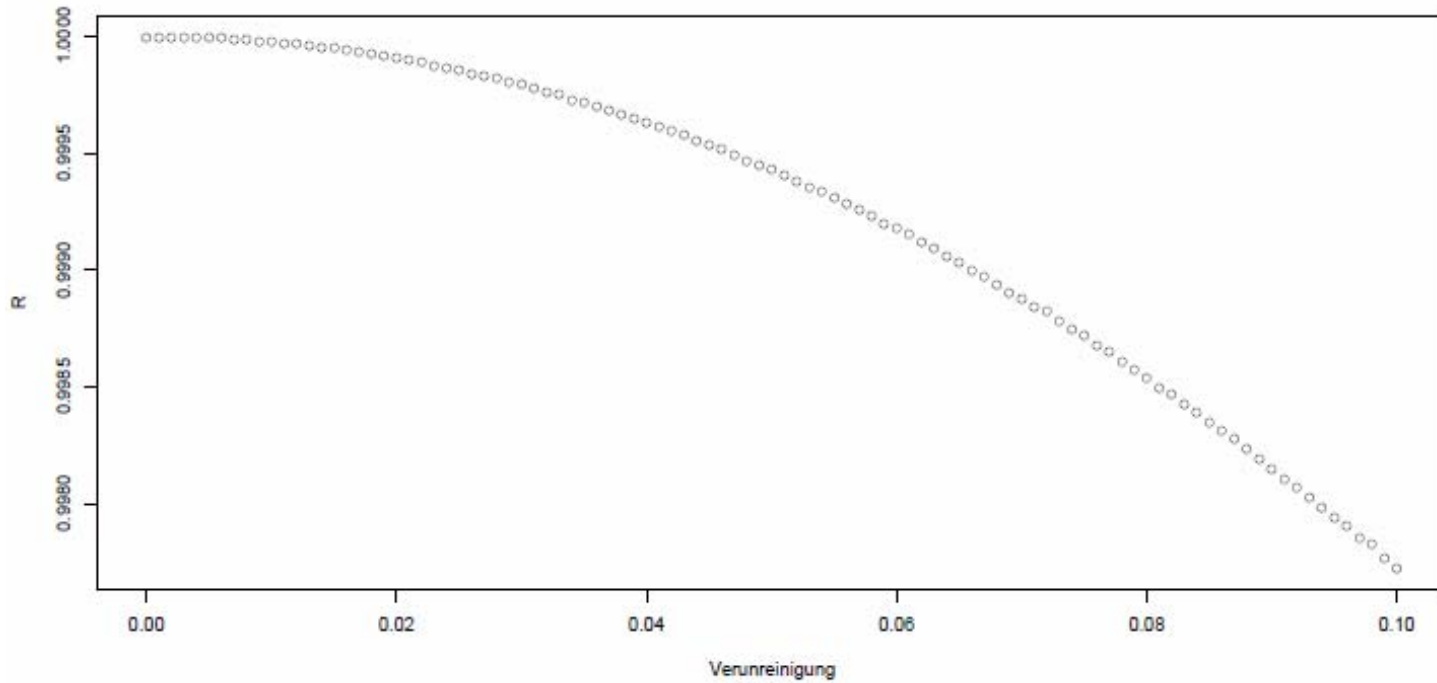
$$D = x - f \cdot y$$

## 2. Theoretical studies and simulation

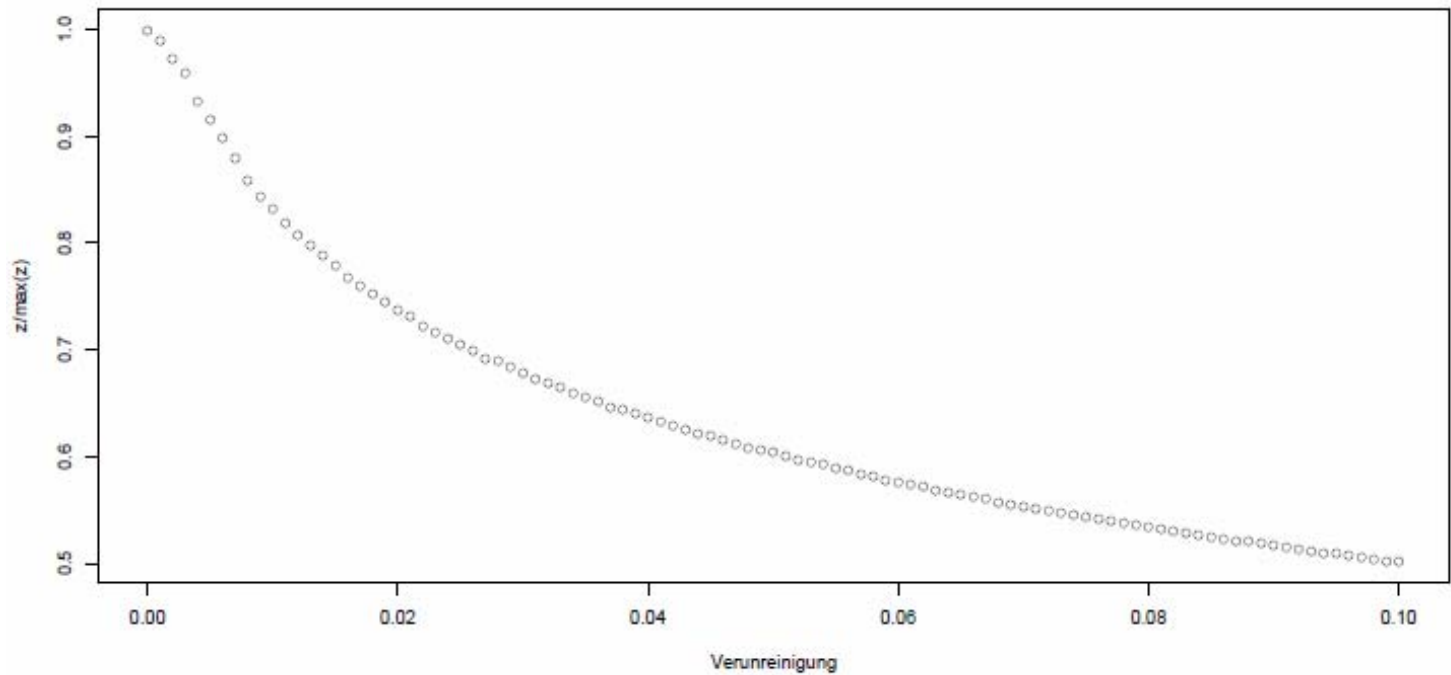
Simulation of bands of a mixture C, composed out of a main substance A plus an impurity B in different concentrations



# Correlation coefficient R



Fisher's Z coefficient:  $Z = \frac{1}{2} \times \ln \frac{1+R}{1-R}$



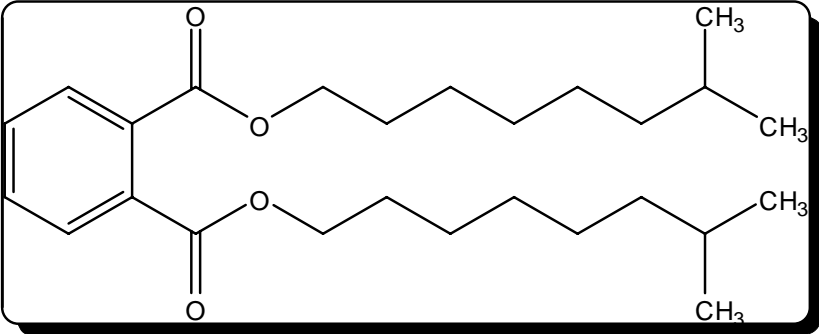
## Spectral Purity Parameters

- **$SPR_1 = 100 \times R$**
- **$SPR_2 = 100 \times Z_s / Z_0$**   
 $Z = \frac{1}{2} \times \ln (1+R) / (1-R)$   
 $Z_0 = \text{mean}(Z) - t \times \text{sd}(Z)$  (alpha=0,05)
- **$SPR_3 = 100 \times f$**   
 **$B^{\wedge} = C - f \times A$**
- **$SPR_4 = 100 \times \int B^{\wedge} / \int A$**

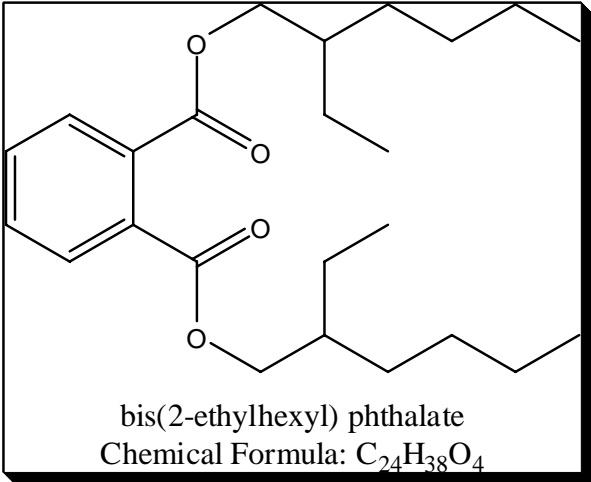
### 3. Experimental data

#### Chemicals

- Palatinol-N
- Palatinol-AH

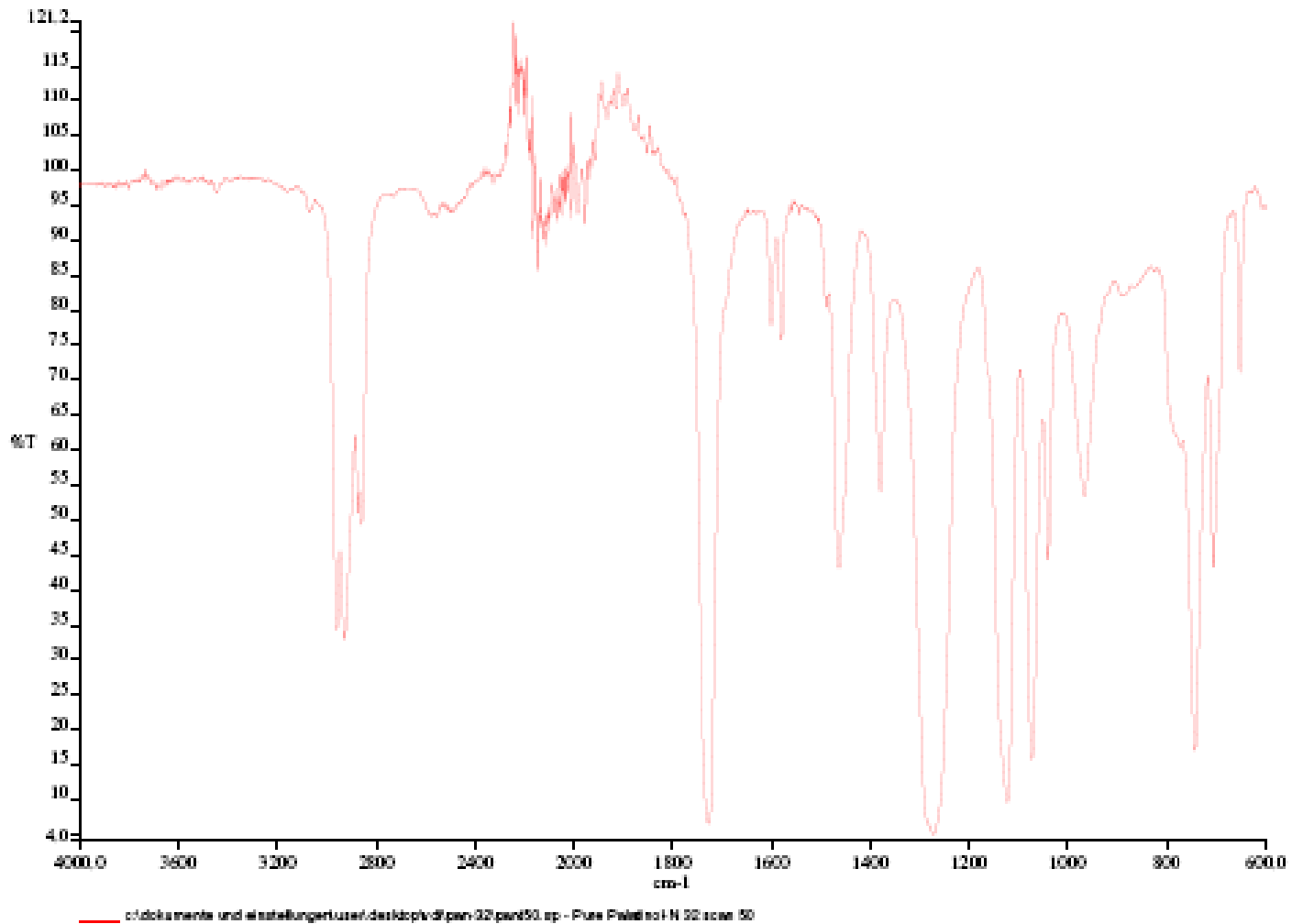


bis(7-methyloctyl) phthalate  
Chemical Formula:  $C_{26}H_{42}O_4$

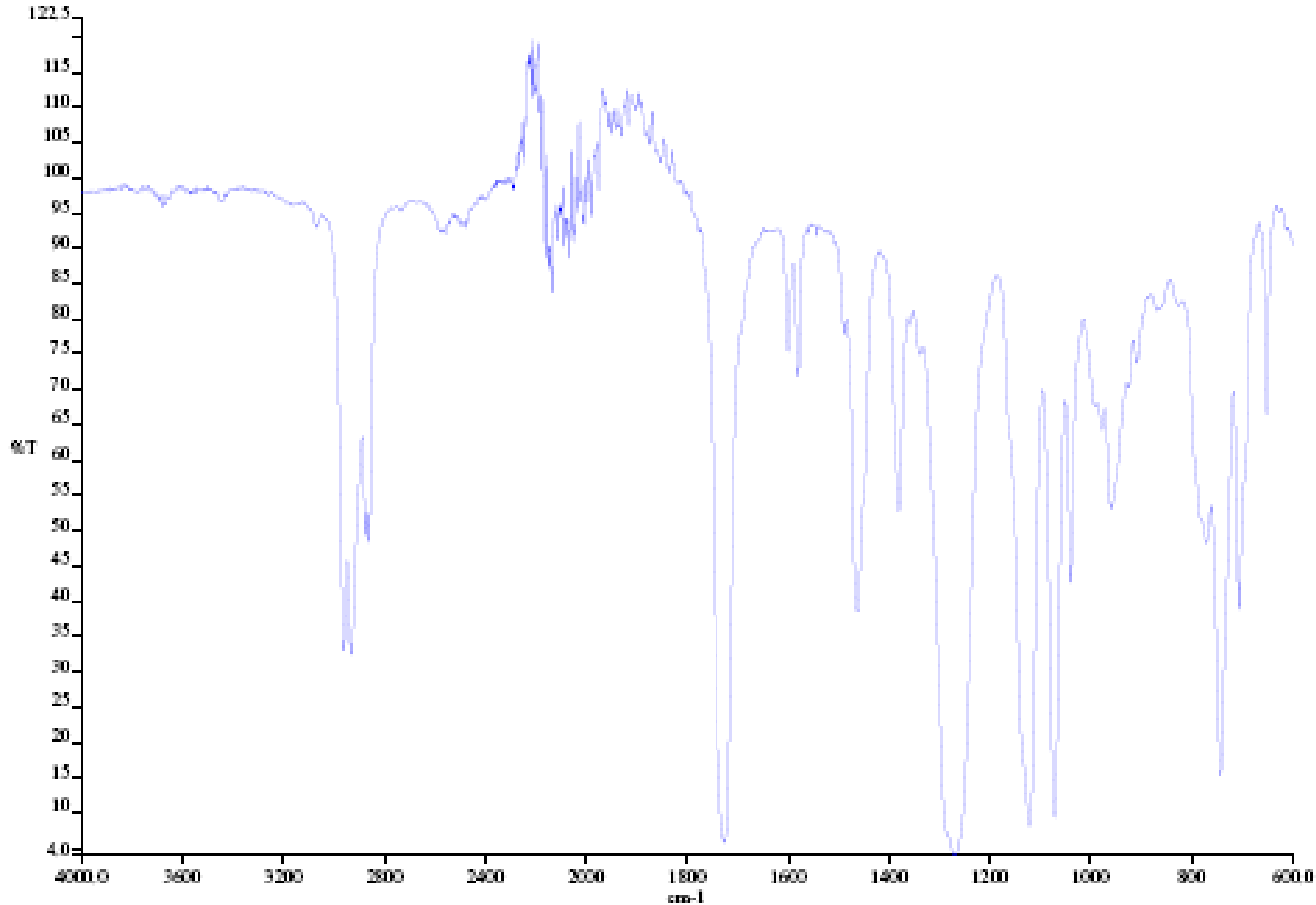


bis(2-ethylhexyl) phthalate  
Chemical Formula:  $C_{24}H_{38}O_4$

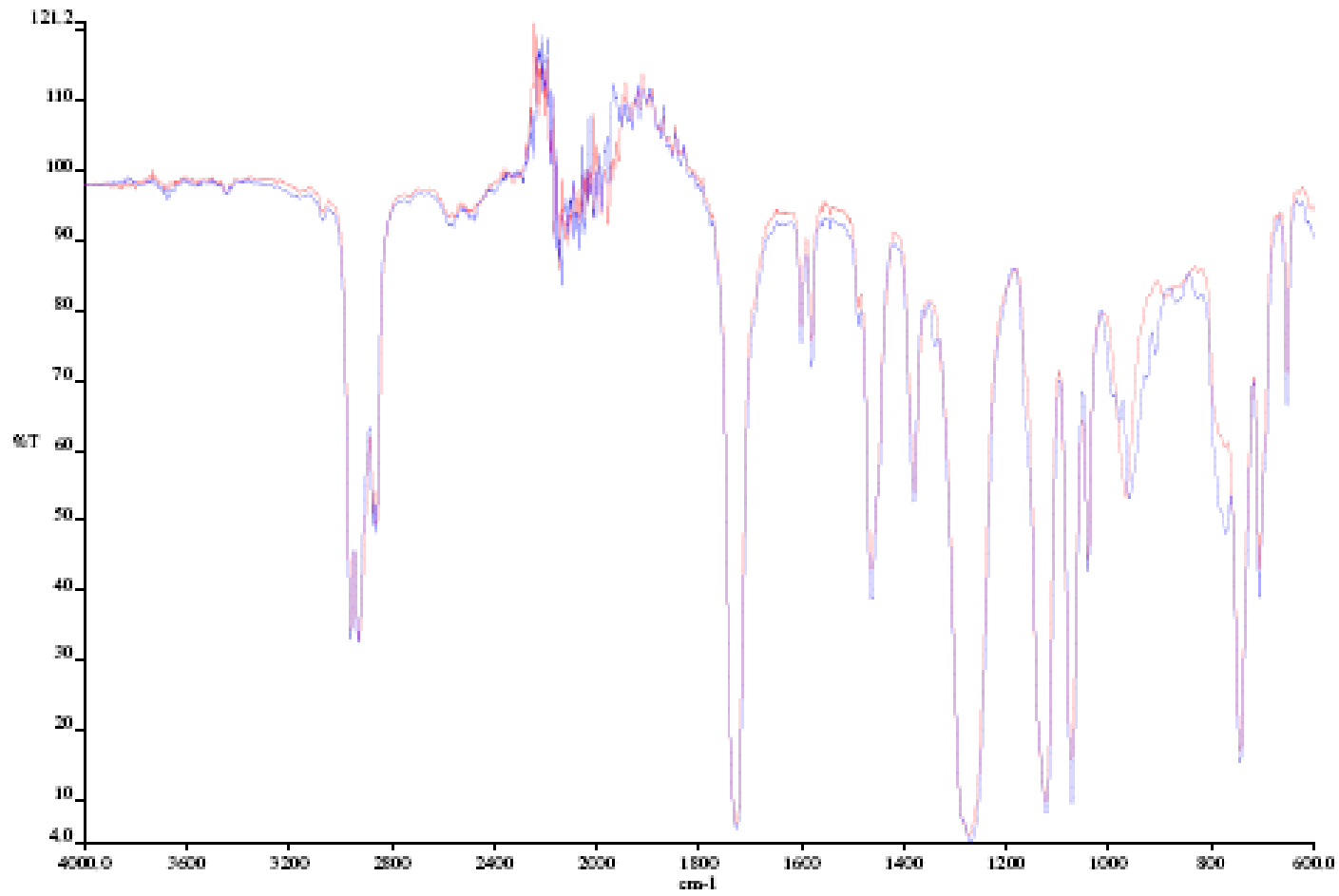
# Spectrum of Palatinol-N



# Spectrum of Palatinol-AH



# Spectra of both compounds together

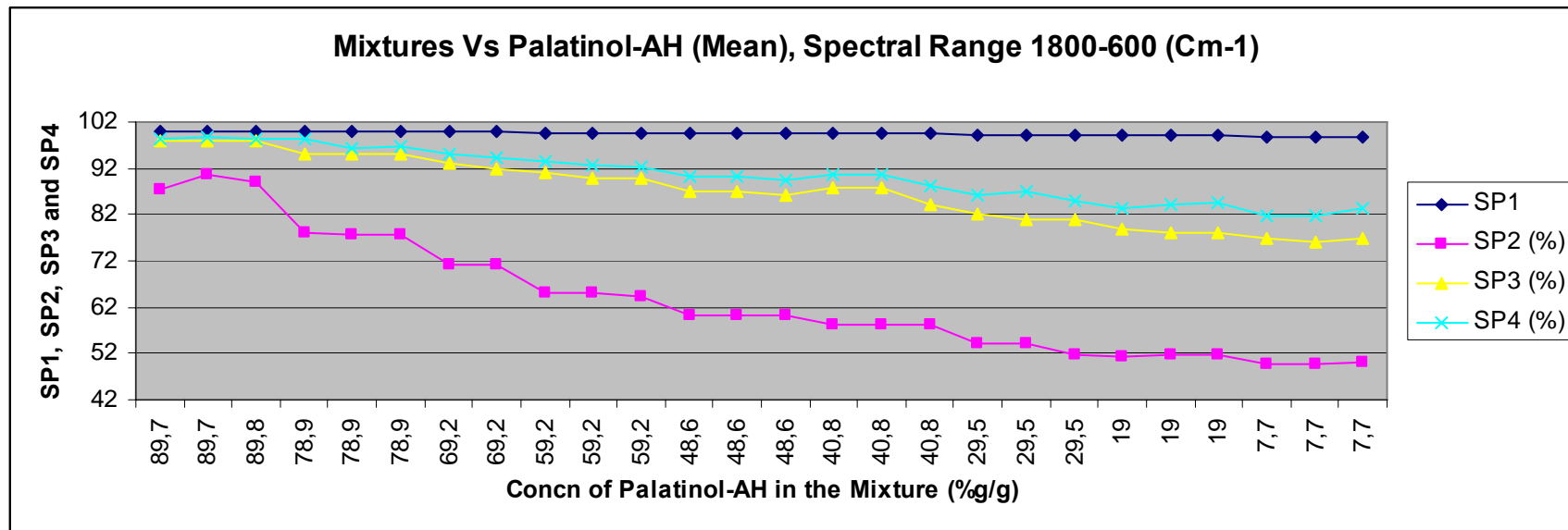


— c:\dokumente und einstellungen\user\desktop\dfpsh\02\pan650.sp - Pure Pallet to HN 02 scan 50  
— c:\dokumente und einstellungen\user\desktop\dfpsh\02\pan650.sp - Pure Pallet to IAH 02 scan 50

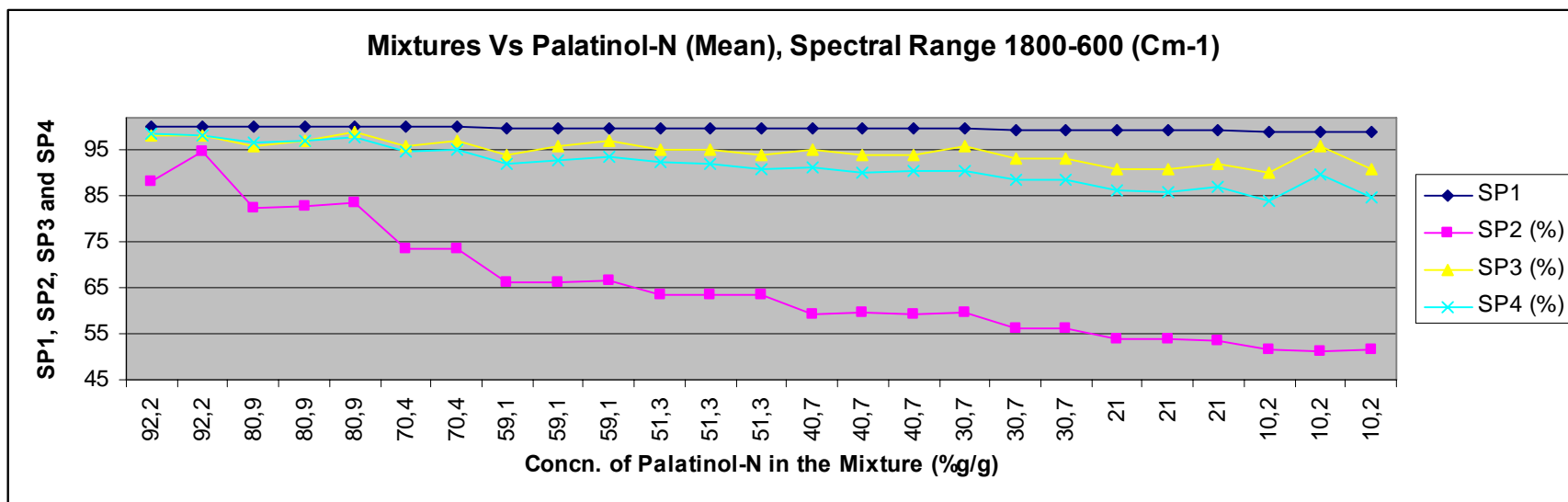
To R-Code



# Mixtures against Palatinol-AH (Mean), Spectral Range 1800-600cm<sup>-1</sup>



# Mixtures against Palatinol-N (Mean), Spectral Range 1800-600cm<sup>-1</sup>



## 4. Summary and conclusion

Investigate optimal parameters for purity control in dependence from degree of impurity

Find detections limits for impurity parameters

Develop automatic and intelligent ways of doing difference spectroscopy automatically

Try other applications like purity control of water

Danke Schön-Thank you for your attention

Frage-Questions ?