

DEVELOPMENT OF FAST STILBENE CONCENTRATION MEASUREMENT METHOD BASED ON UV-FLUORESCENCE

Pulkka, S., Antikainen, J., Venäläinen, M. & Harju, A.M.
WSE 2016

Susanna Pulkka

23.9.2016



SOUTH SAVO
REGIONAL COUNCIL

Programme for Sustainable Growth and Jobs

Leverage from
the EU
2014–2020



European Union
European Regional
Development Fund

Contents

Results from the development work of the device measuring UV excited fluorescence of stilbenes

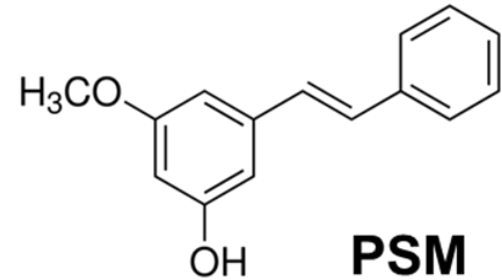
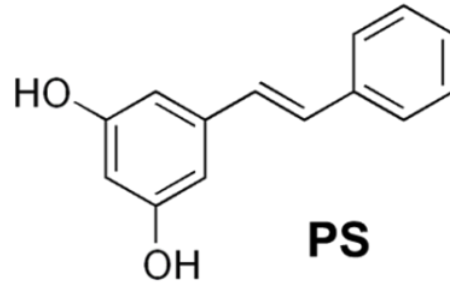
- 1) Light stability of the device was monitored
- 2) An experiment was performed, where stilbene impregnated solid sapwood specimen of Scots pine were measured
 - first with the new device and,
 - then with gas chromatography mass spectrometry (GC-MS)

Introduction

Programme for Sustainable Growth and Jobs



Introduction



Stilbenes

- phenolic compounds of Scots pine (*Pinus sylvestris* L.) heartwood → effect on natural durability

Challenge

- the wide variation on stilbene concentration among individual trees



Programme for Sustainable Growth and Jobs

Leverage from
the EU
2014–2020



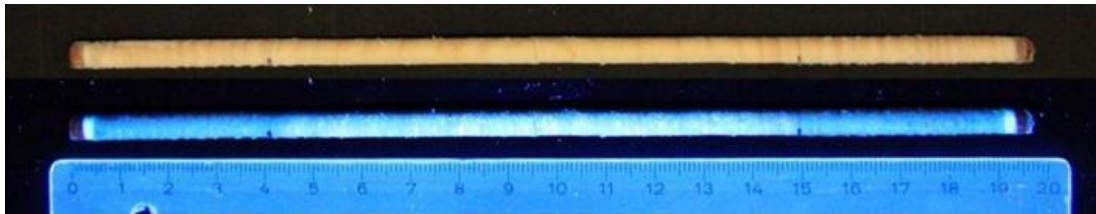
Introduction

Aim

- to develop automated fast and reliable UV fluorescence-based device to measure STB concentration on solid heartwood of Scots pine

Applications

- timber grading and
- breeding for high stilbene concentration of Scots pine heartwood



Material and Methods



Preparation of crude stilbene extract

- **Extraction** of stilbenes (STB) from freeze dried heartwood
 - Accelerated Solvent Extraction device (ASE)
 - extract containing both PS and PSM was concentrated to 20.5 g/l (GC-MS) by using a rotary evaporator
 - solution was protected from daylight.



Impregnation of sapwood samples

- preparation of impregnated sapwood
 - sapwood samples with length of 25 mm were ASE extracted and oven dried at +60 °C
- preparation of five concentrations of impregnation solution
- impregnation in stilbene solutions under vacuum at room temperature and drying in oven
- **calculated STB** concentration in wood samples
 - the amount impregnated stilbenes into wood was divided by dry mass of wood, mg/g

Measurements of stilbene concentration

- **Stilbenes** pinosylvin (PS) and pinosylvin monomethyl ether (PSM) were measured from the same impregnated sample
 - first with UV fluorescence device,
 - then with GC-MS → STB GC-MS mg/g
- **Their sum** STB was used in the analyses, since UV-fluorescence cannot separate the individual stilbenes

Principle of UV-fluorescence measurement

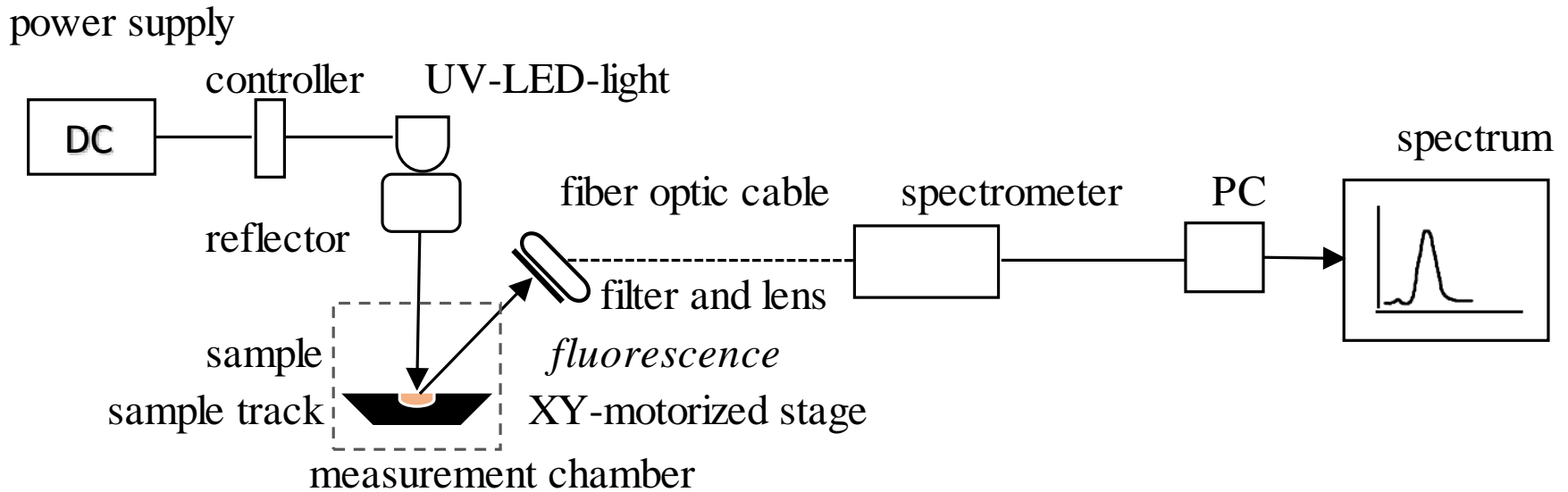


Fig. 1. Figure of the device to measure UV-fluorescence of Scots pine stilbenes.

Variable P_{rel} describing the fluorescence of STB

$$P_{rel} = \frac{I_{max}(\sum(S_{cor})/n)}{I_{max}(V_{cor})}$$

P_{rel} = **relative fluorescence peak** of solid wood sample

I_{max} = maximum fluorescence intensity

S_{cor} = background corrected spectrum of STB

n = about 21 measurement points / each sample

V_{cor} = background corrected spectrum of violet reference



Results

Programme for Sustainable Growth and Jobs



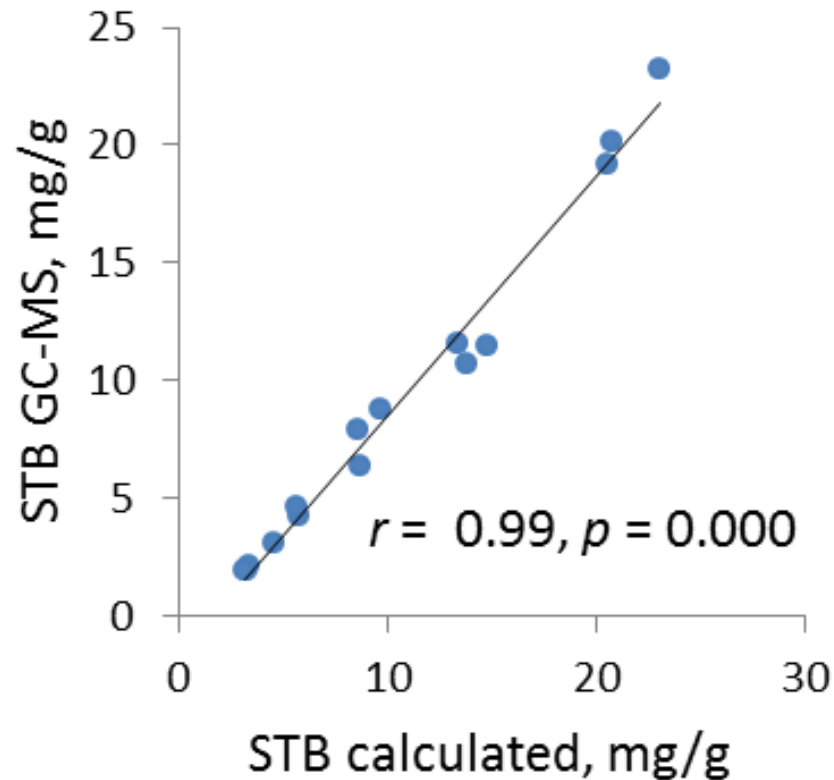
Measurement of light stability

Series	Stabilization time, h	Number of measurements	I_{max}	
			Average, au	CV, %
1	1.25	60	50974	0.34
2	5.75	15	50832	0.06
3	6.25	15	51103	0.09
4	11.5	25	50914	0.06

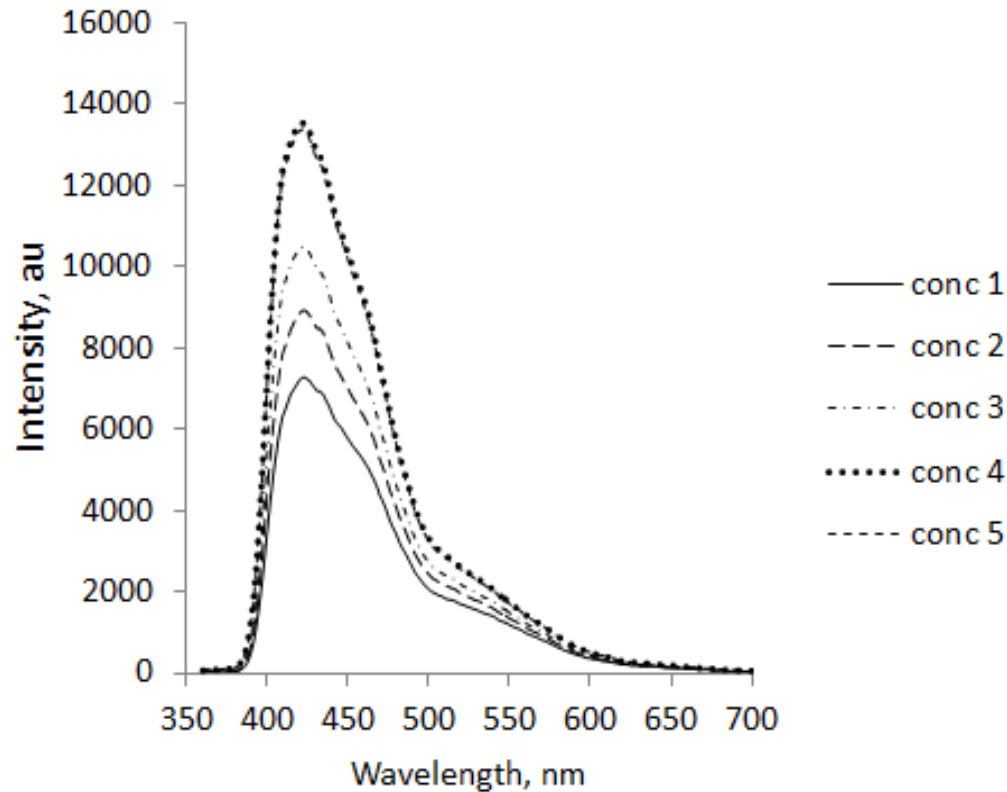
CV = coefficient of variation



Correlation between calculated and GC-MS measured STB

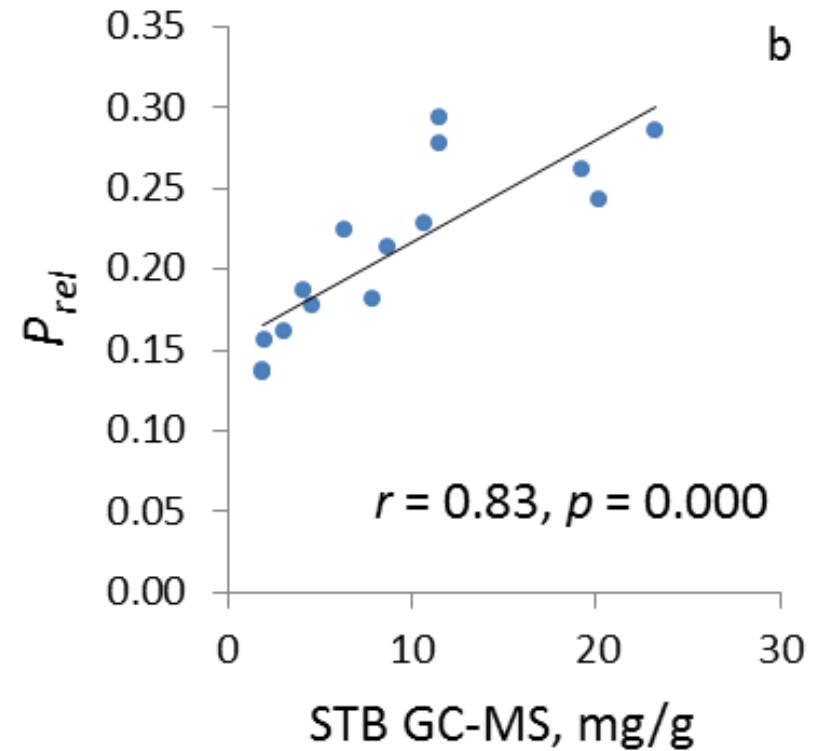
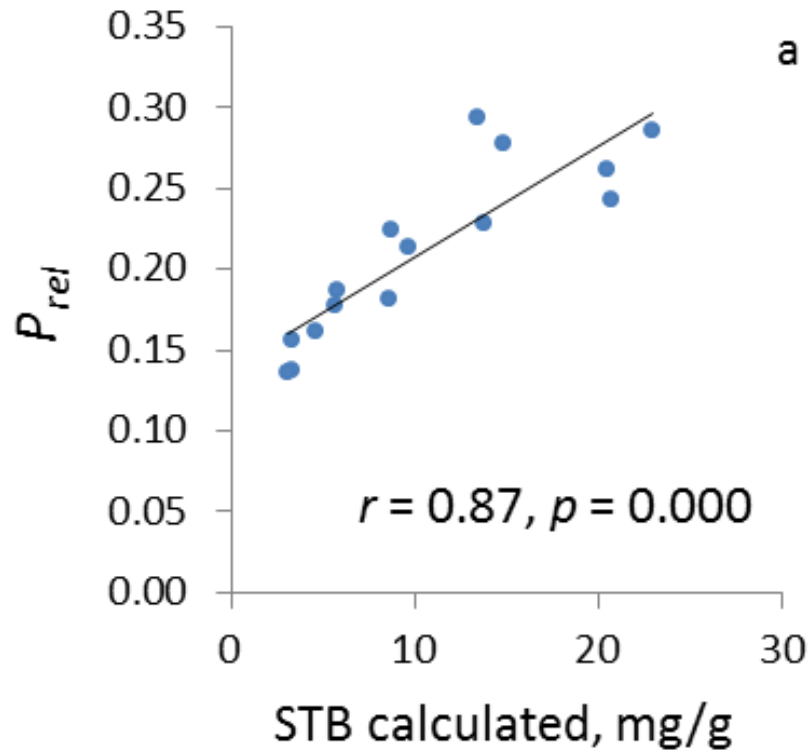


Measured spectra of impregnated sapwood samples



Measurements were averaged over three samples / concentration.

Comparison between chemical and optical measurements



Conclusions

Promising results by UV fluorescence measurement device with movable sample stage

The next is to measure series of Scots pine heartwood samples with wide natural variation in the stilbene content

For example for the grading of sawn timber, the relative differences in STB concentration could be sufficient

The challenge of the future work will be to build a calibration model that provides a prediction of the STB concentration based on the UV-fluorescence spectra

The effect of the surface properties of the wood specimen, their color, thickness and density on the predicted STB concentration needs further examination

We thank

The staff of Luke that participated in sampling, preparation, and analyses of the increment cores

Our collaborations Henri Montonen and Elmar Bernhardt from MAMK/Electronics 3K Factory are acknowledged for their skilled construction of the UV fluorescence measurement device

Financial support from Regional Council of South Savo and the City of Savonlinna are greatly acknowledged.

Thank you!