

Subject 3

Colour, Turbidity and Reflectance Measurement

Referee
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Project at the PTB (Physikalisch-Technische Bundesanstalt)

ICUMSA Method GS2-13:

Determination of the colour-type of white sugar samples

- Standard material: - Braunschweig colour-types “0” and “6” or
- ceramic tiles “0” and “6”
- Determination of the calibration values of the standard material
 - $Colour\text{-}type_{ceramic\ tiles} = -38.31 \cdot R(495)/R(620)_{ceramic\ tiles} + 37.74$
 - $Colour\text{-}type_{sugarstandards} = -41.67 \cdot R(495)/R(620)_{sugarstandards} + 41.04$

Project at the PTB

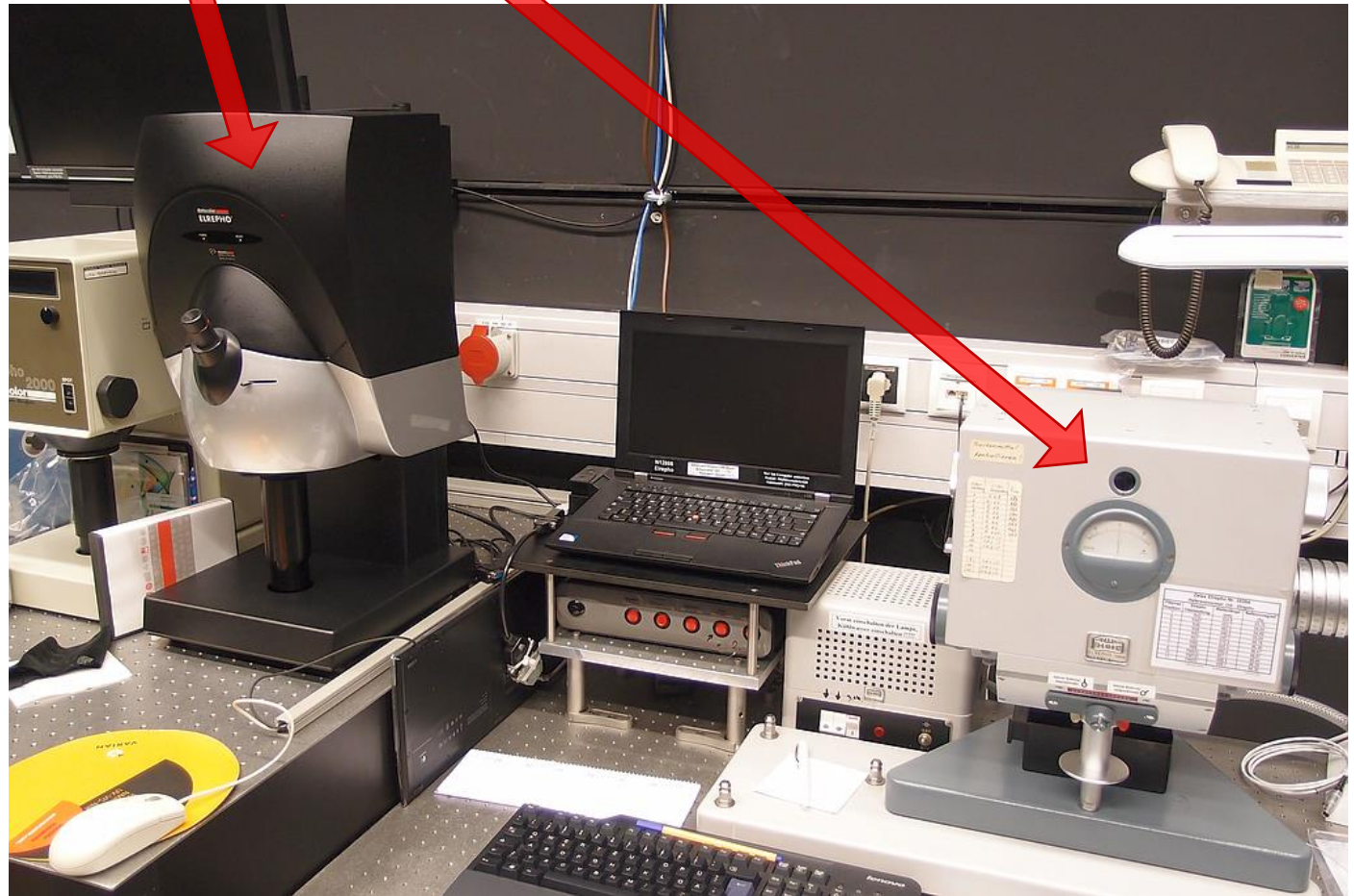
Determination of the calibration values of the standard material for ICUMSA method GS2-13 with the **Zeiss Elrepho**

- calculated from reflectance ratio $R(495)/R(620)$
- Measurement with other instruments than the Zeiss Elrepho instruments at T&I Nordzucker and the PTB will lead to deviating calibration values of the standard material

Aim of the project:

- “Transfer” of measurement procedure specified in method GS2-13 to modern commercial instrument
 - New instrument: Elrepho from company Datacolor

- The Elrephos of Datacolor and Zeiss side by side in the lab at



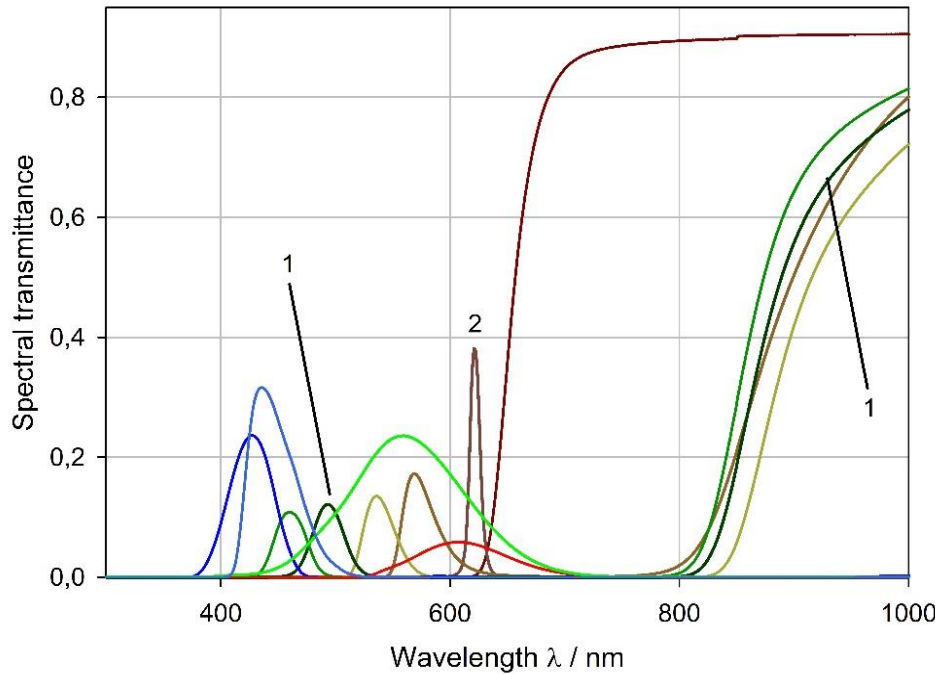
Project status

- Final report of the PTB was received in June 2018
 - Activities within the project were summarized
- Conclusion:

The “Datacolor Elrepho” can replace the traditional “Zeiss Elrepho” for calibration of sugar standards and ceramic tiles

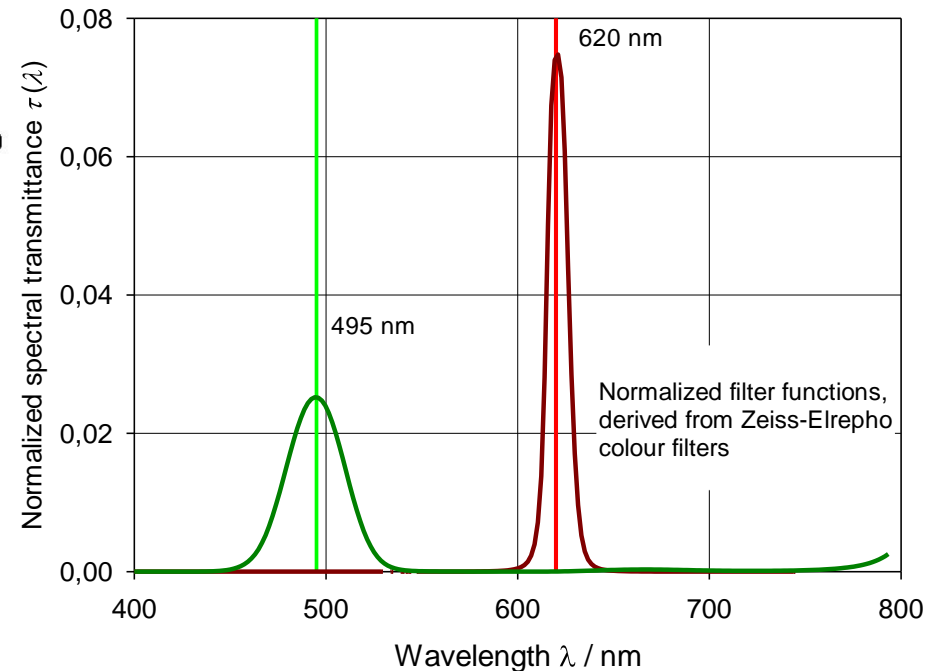
Project steps

- Verification of the filter characteristics of the Zeiss Elrephos located at T&I, Nordzucker AG and PTB with a high resolution spectrophotometer ✓
- Development of a computer program for the readout of the raw data of the diode arrays inside of the Datacolor Elrepho ✓



$$R(\lambda_n) = \frac{\int_0^{\lambda_{\max}} \rho(\lambda) \cdot V_n(\lambda) d\lambda}{\int_0^{\lambda_{\max}} \rho_w(\lambda) \cdot V_n(\lambda) d\lambda}$$

$$V_n = S_n(\lambda) \cdot \tau_n(\lambda) \cdot \varepsilon(\lambda)$$

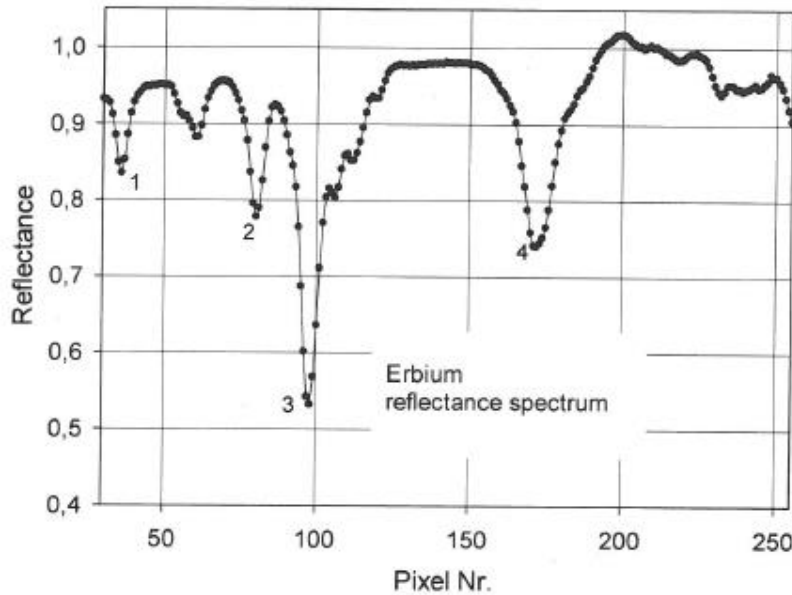


for **Carl Zeiss Elrepho:**

determination of the spectral characteristics by precision spectroscopy


Project steps

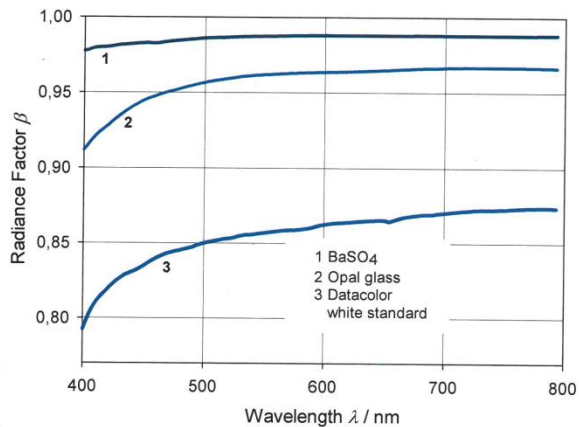
- Wavelength calibration of the Datacolor Elrepho using a Erbium reflective Erbium standard with the reference instrument at the PTB (Agilent Cary 6000i precision spectrometer) ✓



Typical Erbium reflectance spectrum as measured with the Datacolor Elrepho


Project steps

- Calibration of the reflectance scale of the Datacolor Elrepho 
 - Differences in the measured reflectance values occurred when using different white standards (barium sulphate, Datacolor white standard, opal glass)
 - A count-rate correction factor needs to be applied



Spectral variations of white standards
measured with the PTB reference set-up

Project steps

- Calculation of a factor to correct persistent discrepancies 
 - insufficient blocking of the 495 filter, unknown weight of the contribution from longer wavelengths
 - Unknown influence of an infrared-filter installed at Zeiss Elrepho which modifies the contribution of longer wavelengths
 - Different contribution of the gloss component

Persistent deviations corrected by factor c

$$r = \frac{R(\lambda_1 = 495 \text{ nm})}{R(\lambda_2 = 620 \text{ nm})} \cdot c$$

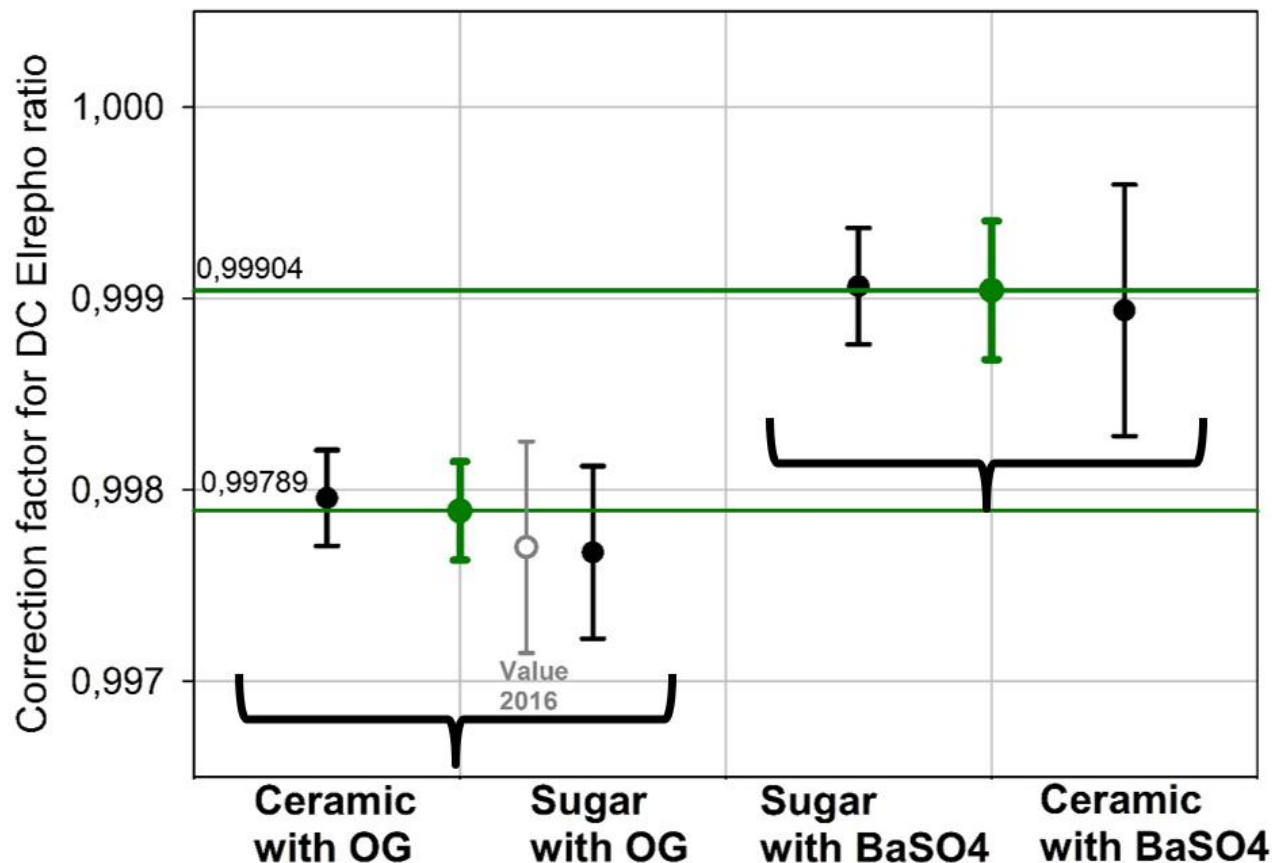
Preliminary determination of the correction factor

Type	ZE target value	computed correction for DCE ratio	
Sugar	6.11	0.99900	
Ceramic	5.57	0.99700	
Ceramic	5.50	0.99778	mean = 0.9977 ± 6,6 · 10 ⁻⁴
Sugar	5.96	0.99791	
Sugar	4.75	0.99737	
Ceramic	-0.42	0.99814	
Ceramic	-0.45	0.99697	
Sugar	0.39	0.99777	

Project steps

- Expanding the database for calculation of the correction factor ✓
 - Further measurements were carried out in 2018 comparing results for the measured colour-type of the sugar standards and ceramic tiles obtained with the 2 instruments
 - » including barium sulphate as white standard
 - » Evaluation on calibration values analysed for ceramic tiles with both instruments

FV 42029 Extended measurement campaign Spring 2018



Final results
deliver two grouped correction factors

FV 42029 Extended measurement campaign Spring 2018



Numerical results

	Ceramic samples with OG	Sugar samples with OG	Sugar samples with BaSO₄	Ceramic samples with BaSO₄
mean	0.99796	0.99767	0.99906	0.99894
# data points	58	42	42	12
<u>stddev</u>	$9.52 \cdot 10^{-4}$	$1.45 \cdot 10^{-3}$	$9.74 \cdot 10^{-4}$	$1.03 \cdot 10^{-3}$
stderr	$1.25 \cdot 10^{-4}$	$2.23 \cdot 10^{-4}$	$1.50 \cdot 10^{-4}$	$2.98 \cdot 10^{-4}$
exp. uncert. <i>U</i> (95%)	$2.50 \cdot 10^{-4}$	$4.50 \cdot 10^{-4}$	$3.03 \cdot 10^{-4}$	$6.57 \cdot 10^{-4}$
grouped result	0.99789	$2.58 \cdot 10^{-4}$	0.99904	$3.62 \cdot 10^{-4}$

Additional uncertainty ≤ 0.015 for r

$$r = \frac{R(\lambda_1 = 495 \text{ nm})}{R(\lambda_2 = 620 \text{ nm})}$$

PTB project - conclusion

- A procedure to measure the colour-type values of sugar standards (colour-type „0“ and „6“) and ceramic tiles with the „Datacolor Elrepho“ instead of the old „Zeiss Elrepho“ was established
- Comparable results are obtained if the described correction factors are used
- The project aim to provide an alternative instrument for determination of the calibration values of standard material GS2-13 was reached

Quality of MOPS buffer in method GS9/1/2/3-8

- Input to referees of ICUMSA
 - Different qualities of the MOPS reagent used to prepare the MOPS buffer might lead to differing analytical results of the colour in solution
 - Impurities in the reagent might have a masking effect to colour contributing components
 - » Need for specification of quality criteria for the MOPS reagent ?
- Study the influence of different qualities of the MOPS reagent on the measured colour in solution

Thank you for your attention !