INTERNATIONAL METROLOGY CONGRESS

CIM2025

ANALYSIS
LABORATORIJA ZA ETALONIRANJE I VALIDACIJU

ДИРЕКЦИЈА ЗА МЕРЕ И ДРАГОЦЕНЕ МЕТАЛЕ

Metrological characterization of standards – plastic films, which are used for calibration of nano volume spectrophotometers

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Abstract. *UV-VIS spectrophotometers, including nano drop (volume) spectrophotometers, provide an answer to all challenges in scientific and industrial laboratories, whether for pharmaceutical, chemical or service laboratories, all the way to more complex applications such as quantification of DNA, RNA, and protein per second, using only 1-2 µL of the sample. Certified reference materials are used to check the measurement capabilities of nanodrop spectrophotometers and other analytical instruments. Since these are solutions, which by themselves are not practical in terms of handling, stability, availability, we were thinking of using materials for their calibration, which would have more suitable properties than the solutions. We decided to use plastic films, which are thin enough not to be a problem during manipulation and measurement, which are stable in terms of determining the regular transmittance, which are optically neutral in the wider spectrum of optical radiation, and which are temperature stable and do not change their metrological characteristics during prolonged exposure to light. The paper evaluates all these influential characteristics and presents the budget of measurement uncertainty of plastic films in the UV part of the optical radiation spectrum at wavelengths of 260 nm and 280 nm (Fig.6). The term "metrologically characterized materials" represent equivalent of the materials, with precisely define regular transmittance (e.g., absorption), as well as estimated influential parameters which may appear, thus contributing to the uncertainty of the measurement.*

Calibration of nano volume spectrophotometers (Fig.3) is usually performed with some of the standard reference solutions. It is foreseen by the manufacturer of these instruments, that the mentioned solutions are applied for one-time use. After using these solutions, any excess solution is thrown away, which is basically the negative side of these SRMs. Of course, the other negative side of this method of calibration is the high price of the SRM. Our wish was to try to find a solution, namely SRM, such that the price of SRM is acceptable and that there is a possibility of using these SRM for a longer period. In this way, we would be able to calibrate Nano volume spectrophotometers with a completely different type of standard reference materials - foils. Our goal was to characterize these foils metrologically and present them as CRMs, which will enable them to be used for a long period of time, without losing, or in the best case, degrading, their metrological properties, especially in terms of long-term stability, accuracy of reproducing the standard values etc. Before the characterization itself, the mentioned standard reference materials-foils were defined in terms of their composition analysis. We used an IR spectrophotometer with Fourier transform (FT-IR) (Fig. 1) to analyse the composition of the mentioned foils in detail. The results of the analysis are presented in Fig. 2.

The influencing parameters that occur during the measurement of the spectral coefficient of transmittance, i.e. absorption, of optically neutral filters are as follows:

- homogenity, exposure, temperature and atmospheric influences, light exposure stability and the influence of dispersion.

- The influencing parameters, such as, homogenity, light exposure stability and the influence of dispersion were determined by the spectrophotometric method using the spectrophotometric system (Fig. 4), while the temperature influence parameter was determined by the method of the integration sphere (Fig. 5)

Fig.2 Spectral characteristic filter foil obtained FT-IR

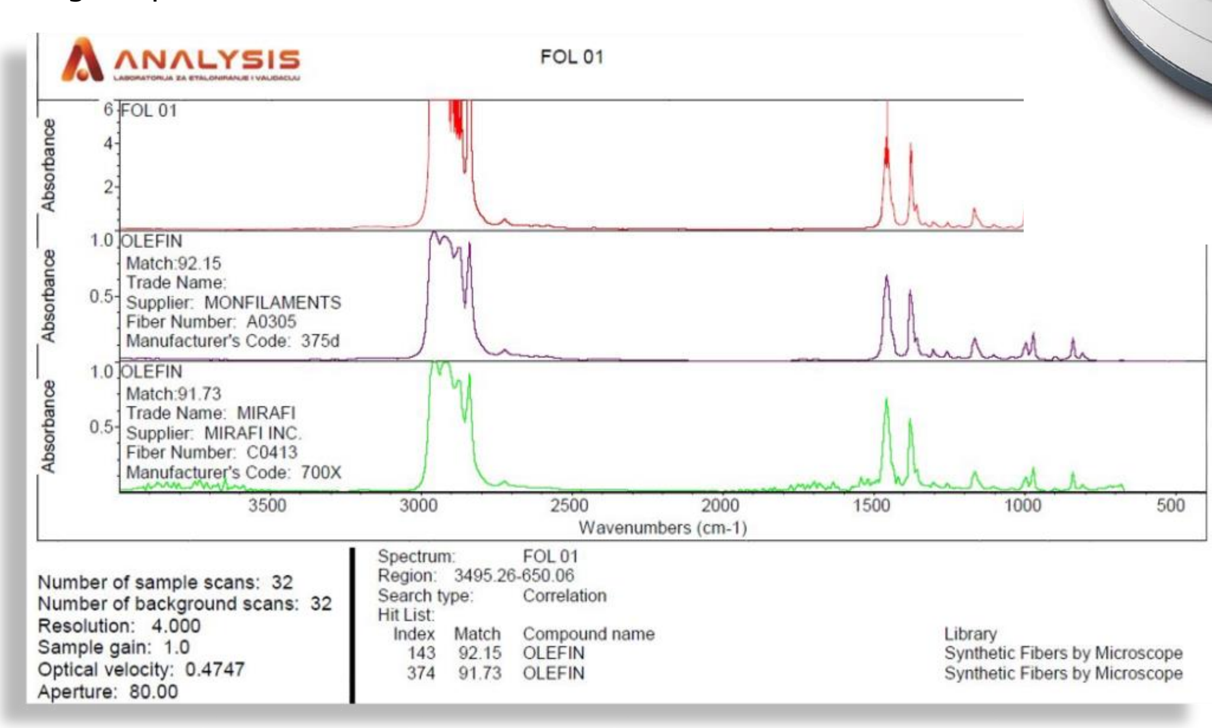

Fig.1 IR spectrophotometer with Fourier transform (FT-IR)

Fig.3 Nano drop spectrophotometer

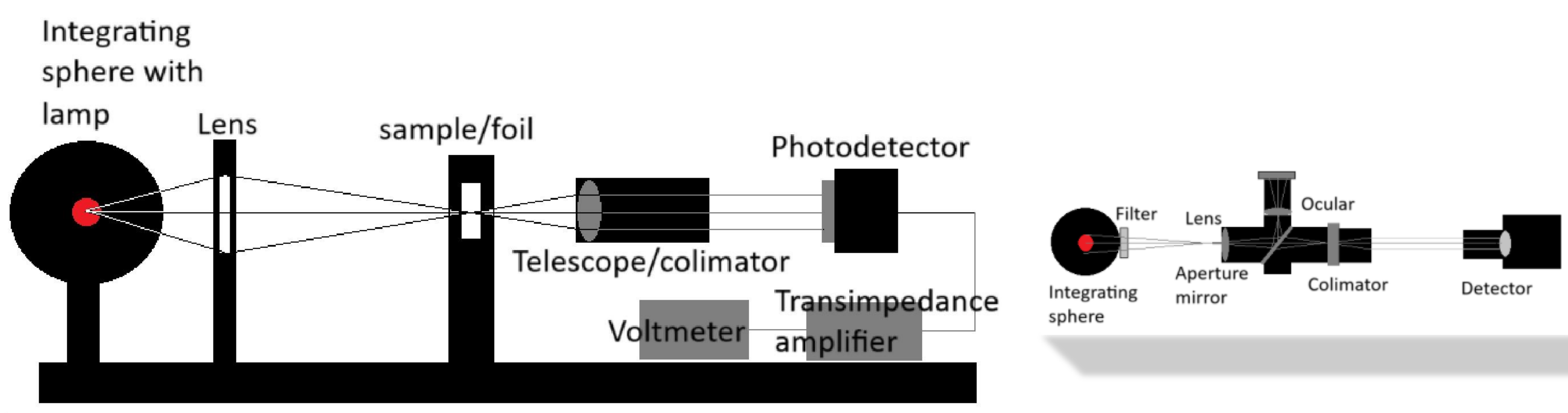

Fig. 5 Method of determination homogenity filter foils with method wich used the integration sphere

Fig.4 Optical scheme of spectrophotometric system

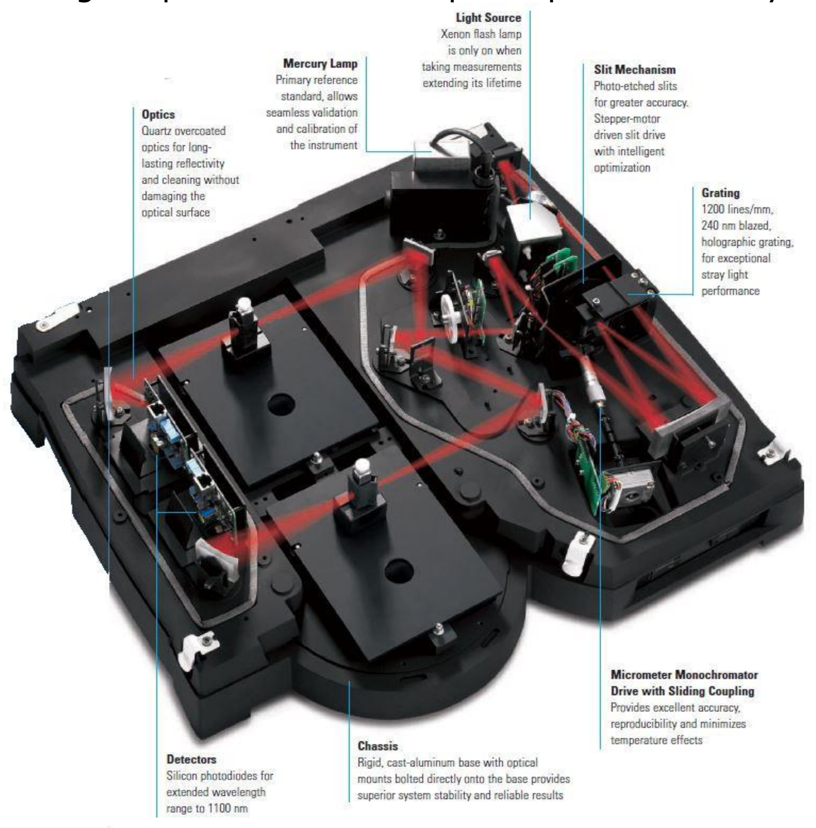

Fig. 6 Spectral characteristics standard foil filters FOL1, FOL2 and FOL3

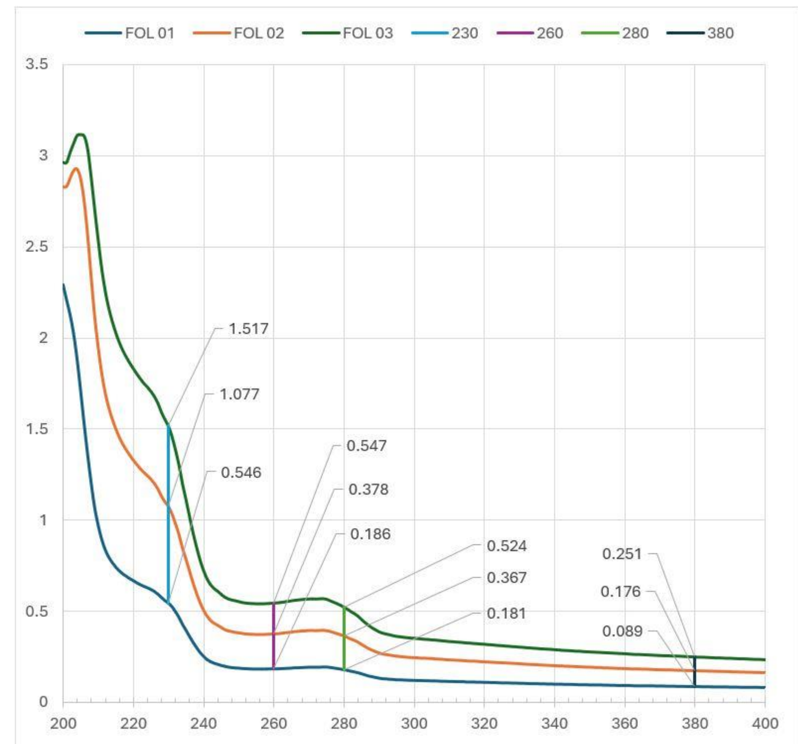

Table 1. Determined of stability during a period of one year

Measuring of the spectral coefficient of transmittance at a determined stability during a period of one year	FOL1		
	230 nm	260 nm	280 nm
240104	0,2275	0,6425	0,6323
240311	0,2244	0,6383	0,6275
240521	0,2204	0,6371	0,6226
240926	0,2277	0,6321	0,6194
241129	0,2233	0,6267	0,6137
Average τ	0,2247	0,6353	0,6231
u component of measurement uncertainty of stability	0,0070	0,0049	0,0059
u_{ave}	0,0104		

Table 2. Measurement uncertainty, resulting from the inhomogenity of the three spectrophotometric optical neutral folies

Measuring of the spectral coefficient of transmittance at a determined wavelength before light exposition	FOL1	FOL2	FOL3
1 central position	0,2268	0,6425	0,6323
2 left positions	0,2270	0,6401	0,6320
3 right positions	0,2273	0,6383	0,6275
4 up positions	0,2280	0,6371	0,6226
5 down positions	0,2272	0,6341	0,6286
τ	0,2270	0,6403	0,6306
u component of measurement uncertainty due to inhomogenity of filter surface	0,0006	0,0019	0,0025
u_{ave}	0,0032		

Table 3. Measurement uncertainty, resulting from infuence of light radiation for FOL1

Measuring of the spectral coefficient of transmittance on light exposure for FOL1	230 nm	260 nm	230 nm
1	0,2277	0,6321	0,6194
2	0,2233	0,6267	0,6137
3	0,2242	0,6393	0,6275
Average t	0,2255	0,6294	0,6166
Standard deviation	0,0024	0,0063	0,0069
Relative standard deviation	1,05%	1,00%	1,12%
u component of measurement uncertainty due to exposure to light	-0,0004	0,0028	0,0031
uave	0,0019		

Table 4. Measuring on temperature at 7,5 °C

Measuring results of spectral coefficient transmittance on 7,5 oC	FOL 1	FOL 2	FOL 3
Mean value of series at 6 measuring	0,2222	0,6322	0,6161
Standard deviation	0,00010	0,00011	0,00007
RSD %	0,00044	0,00017	0,00012
Measurement uncertainty	0,00025	0,00010	0,00007
Uav	0,00028		

Table 4. Measuring on temperature at 20,5 °C

Measuring results of spectral coefficient transmittance on 20,5 oC	FOL 1	FOL 2	FOL 3
Mean value of series at 6 measuring	0,2255	0,6288	0,6155
Standard deviation	0,00004	0,00007	0,00003
RSD %	0,00016	0,00011	0,00005
Measurement uncertainty	0,00009	0,00006	0,00003
Uav	0,00011		

Table 5. Budget of the measuring uncertainty of the measurement of the spectral coefficient of transparency FOL1

The source of measurement uncertainty	*FOL1*		
	Coefficient of sensitivity	Contribution of standard uncertainty (Type A)	Contribution of standard uncertainty (Type B)
Component of measurement uncertainty of spectrophotometric system (dispersion of results, drift, stray light, resolution, fault radiation, setting wavelength…)	1		0,00104
Long stability of the results of measurement of spectral coefficient of transmittance after one year	1	0,0104	
Component of measurement uncertainty of homogeneity of filter structure	1	0,0006	
Component of measurement uncertainty of exposure of light radiation	1	0,0004	
Component of measurement uncertainty of temperature influence	1	0,0017	
Standard uncertainty by measurement of spectral transmittance coefficient		0,0106	0,00104
Combined uncertainty of the measurements of the transmittance coefficient with k=2	0,0213		

Table 6. Budget of the measuring uncertainty of the measurement of the spectral coefficient of transparency FOL2

The source of measurement uncertainty	*FOL2*		
	Coefficient of sensitivity	Contribution of standard uncertainty (Type A)	Contribution of standard uncertainty (Type B)
Component of measurement uncertainty of spectrophotometric system (dispersion of results, drift, stray light, resolution, fault radiation, setting wavelength…)	1		0,00104
Long stability of the results of measurement of spectral coefficient of transmittance after one year	1	0,0110	
Component of measurement uncertainty of homogeneity of filter structure	1	0,0019	
Component of measurement uncertainty of exposure of light radiation	1	0,0028	
Component of measurement uncertainty of temperature influence	1	0,0002	
Standard uncertainty by measurement of spectral transmittance coefficient		0,0115	0,00104
Combined uncertainty of the measurements of the transmittance coefficient with k=2	0,0231		

Table 7. Budget of the measuring uncertainty of the measurement of the spectral coefficient of transparency FOL3

The source of measurement uncertainty	*FOL3*		
	Coefficient of sensitivity	*Contribution of standard uncertainty (Type A)*	*Contribution of standard uncertainty (Type B)*
Component of measurement uncertainty of spectrophotometric system (dispersion of results, drift, stray light, resolution, fault radiation, setting wavelength…)	*1*		*0,00104*
Long stability of the results of measurement of spectral coefficient of transmittance after one year	*1*	*0,0173*	
Component of measurement uncertainty of homogeneity of filter structure	*1*	*0,0025*	
Component of measurement uncertainty of exposure of light radiation	*1*	*0,0031*	
Component of measurement uncertainty of temperature influence	*1*	*0,0001*	
Standard uncertainty by measurement of spectral transmittance coefficient		*0,0177*	*0,00104*
Combined uncertainty of the measurements of the transmittance coefficient with k=2	*0,0356*		