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Realization of high accuracy spectrophotometric system as national standard of regular spectral transmittance coefficient

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Abstract. The national standard spectrophotometric system developed in the Laboratory for photometry and radiometry of the Bureau of Measures and Precious Metals (ZMDM) is used for realization of regular spectral transmittance scale in spectral region 360 nm to 1000 nm. Evaluation of the performances of the system shows that the measurements on neutral density filters can be performed with the uncertainties in range from 0.1 % to 2 % ($k = 2$), depending on transmittance level and wavelength. System verification was performed according to NIST SRM reference standards. The validation of the method is expected with the results of EUROMET key comparison of regular spectral transmittance, in which, the Laboratory for photometry of ZMDM was included through the - bilateral comparison of regular spectral transmittance measurements with INM (EUROMET project No 766). Realization of the system and the method used in that bilateral comparison, together with its metrological characterization and measurement uncertainty evaluation, are presented in this paper.

Introduction

With realization of primary standard spectrophotometric system and realization by definition of the value of regular spectral transmittance coefficient [1], basic conditions for achieving measurement unity in the field of spectrophotometry are accomplished.

At the beginning of the year 2004, the bilateral comparison of spectral transmittance measurements, between Laboratory for photometry of the ZMDM and Laboratory for spectrophotometry of INM, was performed. It is officially recognized as EUROMET Project No 766. This Project is included as part of the EUROMET key comparisons of regular spectral transmittance measurements.

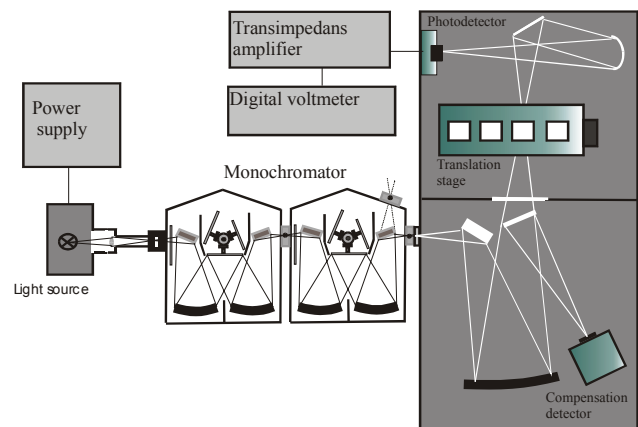
In this paper, measurement procedure, together with measurement results and uncertainty evaluation, regarding measurements performed in Laboratory for photometry as part of that project, are presented.

Design of the Spectrophotometric System

A single beam instrument with automated exchange of measured samples and compensation detector, based on double monochromator, is build up on the optical table, in the Laboratory for photometry (Figure 1).

The image of the halogen light source is projected on the entrance slit of the monochromator with quartz lens objective with $f/5.5$. In the plane of the exit slit of the

monochromator, 3 mm in diameter, circular aperture is placed, so that, in the focal plane of the optical system, the regular circular spot may be obtained. All-mirror off-axes (off-axes angle $\leq 6^\circ$) optical system consists of one plain mirror and one spherical mirror of large diameter (152 mm, $f/2$ mirror relative aperture) projecting image of circular aperture at focal plane with 1:1.1 ratio. Beam splitter is used to divert approximately 10 % of the radiant flux on to the compensation detector. Stepp motor driven translation stage has linear resolution of 10 μm . One plane and one spherical mirror have a role to refocus the spot from the Figure 1. The scheme of spectrophotometric system.



measured sample on to the detector surface with 1:1 ratio. Both detectors are silicon photodiodes Hamamatsu S1337 1010BQ. Signals from photodiodes are measured through transimpedance amplifier, with scanner and digital voltmeter. The system is fully automated [2].

In the first realization of the system, refocusing optical system was achromatic objective, what results that the system was liable to interreflections [1]. The effect was larger measurement uncertainty and limited wavelength range. In order to extend the wavelength range to UV, and to reduce the measurement uncertainty, changes in refocusing optical system (Figure 1.) was made.

Description of measuring technique

Measurement procedure is automatic exchange of measured sample in the path of the light beam. Each time, signal from main and compensation detectors are measured simultaneously, ten times each detector. Average value of those readings was taken into account. At every wavelength measurements were performed for sample and the reference (air), and before each of that, the measurement of dark current were made and correction

applied.

Reproducibility of measurements was determined up on the sample of 30 independent measurements of each sample for every wavelength. All measurements were performed during two months.

Method of characterization of the spectrophotometer

The aim of the project of realization of the national spectrophotometric system is to develop the system that can provide realization of the unit of regular spectral transmittance coefficient at uncertainty level that can fulfill requirements for accuracy in spectrophotometric measurements. This goal can be achieved only with careful interpretation of spectrophotometric data, which means the evaluation of influential parameters and correction of systematic errors wherever it is possible.

Design of the optical system defines the light beam geometry, whose influence on measurements is manifested as beam-displacement errors, interreflection errors and obliquity errors (path-length error and Fresnel-reflectance error) [3]. Uncertainties arising from these effects are evaluated, and for path-length error, the correction is applied. Interreflection error is evaluated by the method of transmittance measurements on tilted samples in polarized light [4].

Nonlinearity of the system is determined by double aperture method [5]. Wavelength accuracy [6], and stray light effects are discussed also. Detailed analysis of all these parameters is reported in this paper.

Uncertainty of measurement

Uncertainties given in table 1 are average values for wavelength range of 500 nm to 1000 nm, for 4 nm bandwidth measurements for the filter of nominal transmittance of 10 %. The numbers in table are relative values.

Table 1. Uncertainty budget.

Parameter	Type A	Type B	Uncertainty in spectral transmittance
Repeatability	0.000603		0.000603
Non linearity		0.0005	0.00045
Wavelength setting		0.1 nm	0.00003
Beam displacement		0.00004	0.00004
Inter-reflection		-	-
Obliquity effect		Corrected	0.00006
Polarization		-	-
Drift			0.0004
RMS total			0.000856

Specific uncertainties associated to each filter (nominal transmittances in range from 0.1 % to 90 %) on every wavelength are given with results of measurements, according to EUROMET's "Technical protocol for key comparison of regular spectral transmittance measurements".

In this paper, also, measurement uncertainty evaluation for the first measurement setup, with implementation of interreflection correction, is given, together with results of validation measurements made on NIST standard filters [7].

Measurement on NIST standard filters is repeated with changed (improved) system after bilateral comparison, and the results are compared with previous.

Conclusion

Validation measurements will show, that in both cases, average difference between results obtained by measurement for NIST filters and their values from the certificate, is within the estimated measurement uncertainty, which indicate that the measurement uncertainty is well determined.

Agreement between the results from measurements made on same filters with two different measurement setups, represent real test for reproducibility of the method.

EUROMET key comparison of regular spectral transmittance measurements, when finalized, should support validation of our spectrophotometric measurements. These results will be reference for some future improvements of our spectrophotometric system.

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