Update of Inmetro’s Primary Spectral Regular Transmittance Calibration System

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Abstract. We present an evaluation of the improvements made to the Laraf's Primary Spectral Regular Transmittance Calibration System (PTCS) at the Inmetro Radiometry and Photometry Laboratory (Laraf). The objective of this study was to investigate the feasibility of using the PTCS for the absolute characterization of spectral regular transmittance filters in the range of 330 nm to 1060 nm. The results obtained in this initial evaluation confirmed the reliability of the PTCS in terms of its ability to measure the spectral regular transmittance of standard filters with high accuracy in the visible range and part of the near-infrared, indicating its viability as a primary standard for traceability of spectrophotometric quantities.

1. Introduction

Spectrophotometry is a widely used technique for analyzing samples in various fields such as chemistry, physics, biology, and engineering. The basic principle of spectrophotometry is Beer-Lambert's Law, which describes the relationship between the concentration of a solution and the absorption of light in a specific wavelength range. Transmittance is a quantity related to absorption and is defined as the ratio of the intensity of light transmitted through the sample to the intensity of incident light [1].

In the characterization of optical filters, transmittance is a fundamental quantity. Optical filter is a device used to modify the optical radiation passing through it, generally by altering the spectral distribution [2]. There are different types of filters, including interference filters and absorption filters. Interference filters are formed by layers of different materials with controlled refractive indices and thicknesses. Absorption filters consist of a material that selectively absorbs a specific range of wavelengths. The characterization of optical filters can be performed using relative or absolute calibration methods [3]. In relative calibration, the transmittance of the filter is compared to the transmittance of a reference filter with a known transmittance. In absolute calibration, the transmittance of the filter is directly determined without the need for a reference filter, using a primary transmittance system.

Currently, the Brazilian National Institute of Metrology, Quality and Technology (Inmetro) offers a group of spectrophotometry calibration services based on a commercial spectrophotometer traced to other National Metrology Institutes (NMI) though sets of standard filters. The services are listed in the Key Comparison Database of the International Bureau of Weights and Measures (BIPM) [4]. Furthermore, a *primary calibration system* has been implemented at Inmetro based on a customized setup developed from the spectral responsivity facility, and validated in an intercomparison with a group of other NMI (SIM.PR-K6.2010)[5].

Here we report on the improvements of Inmetro's Primary Spectral Regular Transmittance Calibration System (PTCS). The system updates its previous version by using a customized setup based on a double monochromator arranged for additive dispersion. Three sets of standard filters were measured in the spectral range from 330 nm to 1060 nm. The results were compared to their calibration history for validation. This constitute an important step for stablishing a national traceability chain in the spectrophotometry area and provide reliable results for creating a favorable environment for development and competitiveness of the national industry.

2. Experimental Setup

A primary system for absolute regular transmittance measurements is essentially composed of a light source, a monochromator, and an optical system for light detection. Halogen, xenon and deuterium lamps are commonly used as light sources powered by a high stability current source. The monochromator is a device that selects a narrow range of radiation wavelengths, allowing only the selected range of light of the broadband lamp to reach the detector. The detector converts the incident radiation into an electrical signal, which is then amplified and measured using data acquisition devices such as amplifiers and multimeters.

The updated PTCS – see Fig. 1 – uses a double monochromator in Czerny-Turner configuration arranged for additive dispersion as the core instrument. The instrument covers the spectral range from 190 nm to 2500 nm. Radiation from the selected lamp (a 250 W quartz tungsten halogen lamp) is focused into its entrance slit. Monochromatic light is then collimated and directed to a photodetector inside a light-tight box. The sample under measurement – usually an optical filter – is positioned and then removed from the beam path. A transimpedance amplifier converts the photocurrent into a voltage value, then read with a resolution of 6.5-digit digital voltmeter.

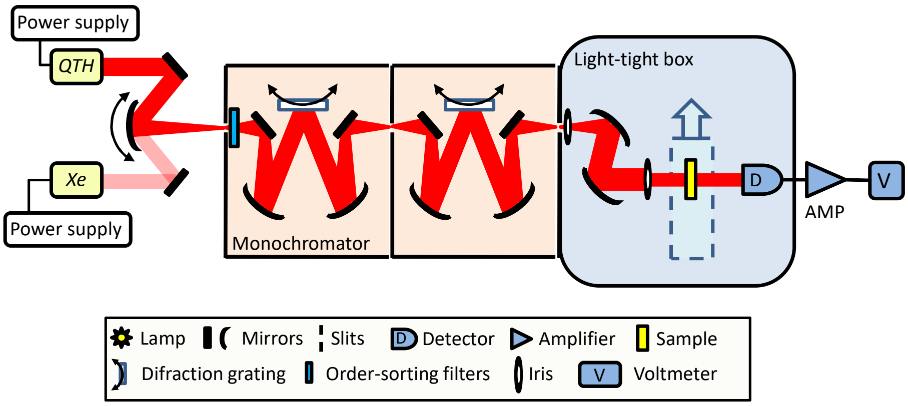

Figure 1. Experimental setup of the PTCS.

The system is automated and operated using a customized Labview software. Automation includes spectral selection, width slits, selection of the order sorting filters of the monochromator, translation of the sample in and out the beam path and data acquisition from the voltmeter. Measurement are stored in data files and then processed using a customized post-processing routine developed in Matlab, resulting in spectral regular transmittance results.

3. Metrological Standards and Measurements

Three sets of standard filters were measured and evaluated here. The first standard is the NIST SRM 2031a set [6]. It consists of standard quartz filters with metallic coating with nominal transmittances of 10%, 30%, and 90%. These filters have high precision in the range of 250 nm to 700 nm and are widely used in spectrophotometry to verify the photometric accuracy of spectrophotometers.

The second standard – NIST SRM 930e set [7] – comprises of standard glass filters with nominal transmittances of 10% and 30%. These filters are calibrated in the range from 400 nm to 700 nm and are usually used for verifying the photometric accuracy of spectrophotometers, as well as for evaluation of its stray light feature.

The third standard is a commercial set of standard glass filters with nominal transmittance of 1%, 3%, 10%, 20%, 30%, 50%, 75%, and a standard quartz filter with nominal transmittance of 90%. These filters have excellent stability in the range from 420 nm to 800 nm (from 210 nm to 800 nm for the 90% filter) and are widely used in spectrophotometer calibration.

The absolute calibration method determines the spectral regular transmittance of the sample from the ratio between measurements, taking the free space as reference. The measurement routine comprises signal and dark measurements, the latter performed by blocking the light entering the monochromator. Optical radiation is alternately measured with the sample positioned in, then out of, then in again the beam path at each set wavelength. Three measurement cycles were performed with each filter in the spectral range from 330 nm to 1060 nm, sampled at each 10 nm. Absolute characterization is relevant as it represents a primary measurement of transmittance.

Measurement uncertainty is estimated based on the Guide to the Expression of Uncertainty in Measurement (GUM) [8]. The uncertainty budget takes into account measurement repeatability and reproducibility, uncertainty due to calibration certificates of amplifier and voltmeter, uncertainty due to wavelength scale and spectral bandpass of the monochromator.

Results for each filter were compared to its calibration history over the last fifteen years and measurements performed with the spectrophotometer with Calibration and Measurement Capability (CMC) supported on KCDB.

4. Results and Discussion

Filters of the NIST SRM 2031a and NIST SRM 930e sets were measured using the PTCS. Results are validated using measurements performed by another NMI member of the CIPM Mutual Recognition Arrangement. Long-term drift of the transmittance values was computed from filter's calibration history records and considered in the uncertainty of the reference values. Figures 2 and 3 show the results of regular spectral transmittance obtained with these filter sets. The filters in first set exhibit smoother regular spectral transmittance value while filters in the second set exhibits some ripples and a drastic transmittance reduction at lower wavelengths.

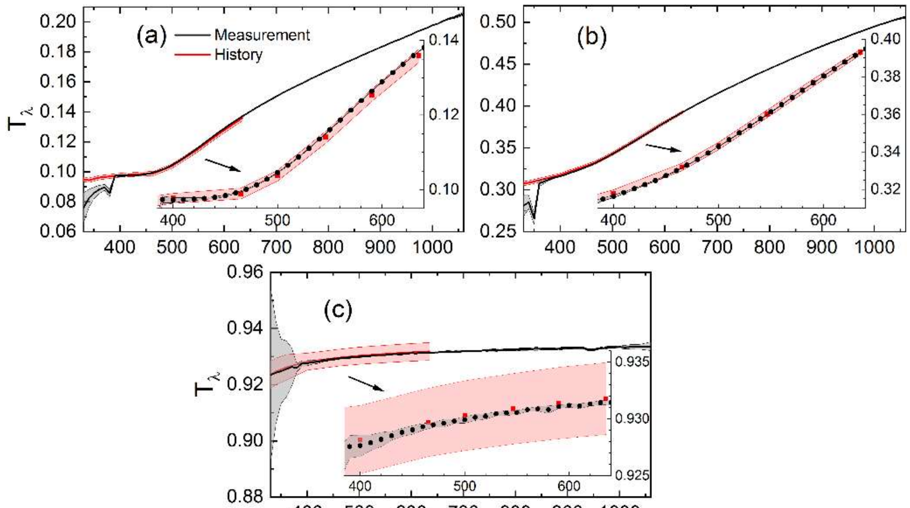

Figure 2. Regular spectral transmittance measurements of NIST SRM 2031a standard filter set and its calibration history data: (a) 10 %, (b) 30 % and (c) 90 %. Insets zoom in on the spectral range with comparable data. Color bands represent expanded uncertainty.

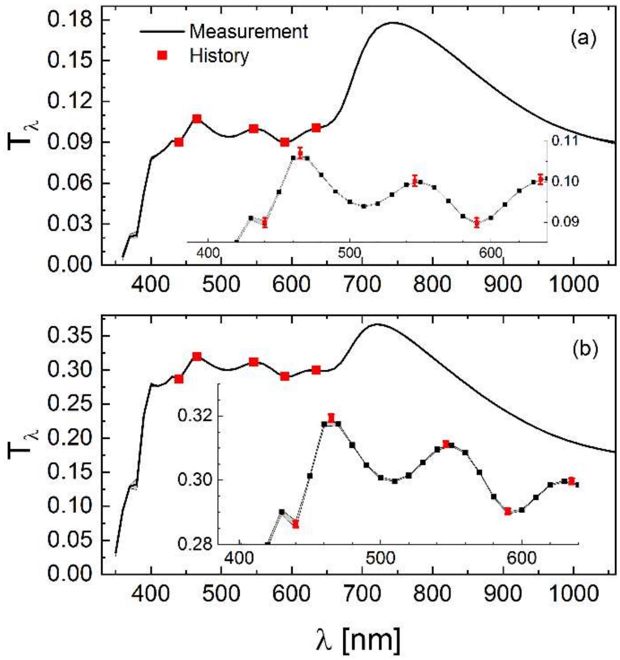

Figure 3. Regular spectral transmittance measurements of NIST SRM 930e standard filter set and its calibration history data: (a) 10 % and (b) 30 %. Insets zoom in on the spectral range with comparable data. Black color bands and red error bars represent expanded uncertainty.

Normalized error (En) methodology is employed for assessing the agreement between results. Measured data was interpolated whenever necessary for matching the history calibration data. Values of En smaller than unit indicate agreement between data within their expanded uncertainty. As shown in Figure 4, En values complies with this criterion for almost all regular spectral transmittance values, except for the filter SRM 930e: 30 % at 465 nm – possibly an outlier – and for few values below 400 nm for the SRM 2031a filter set – due to low power of the system's QTH lamp used for the measurements at the UV spectral region.

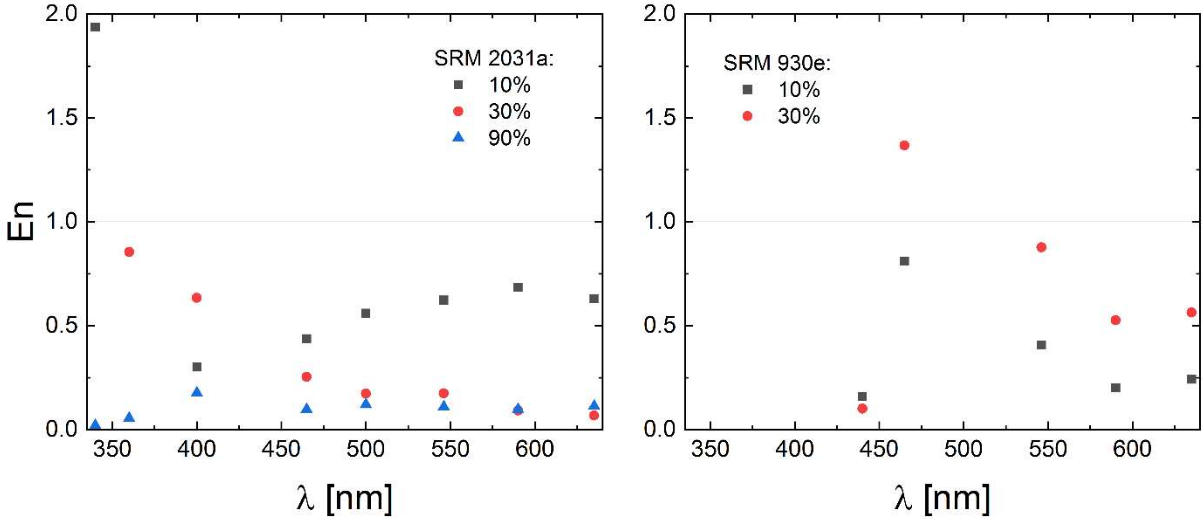

Figure 4. Normalized error of the measurement values compared to the history data as reference for validation of results for the two sets of filters.

Uncertainty values are evaluated as another metrics for the results. Figure 5 reports the expanded uncertainty (k = 2) obtained for filters in the SRM 2031a and SRM 930e standard sets.

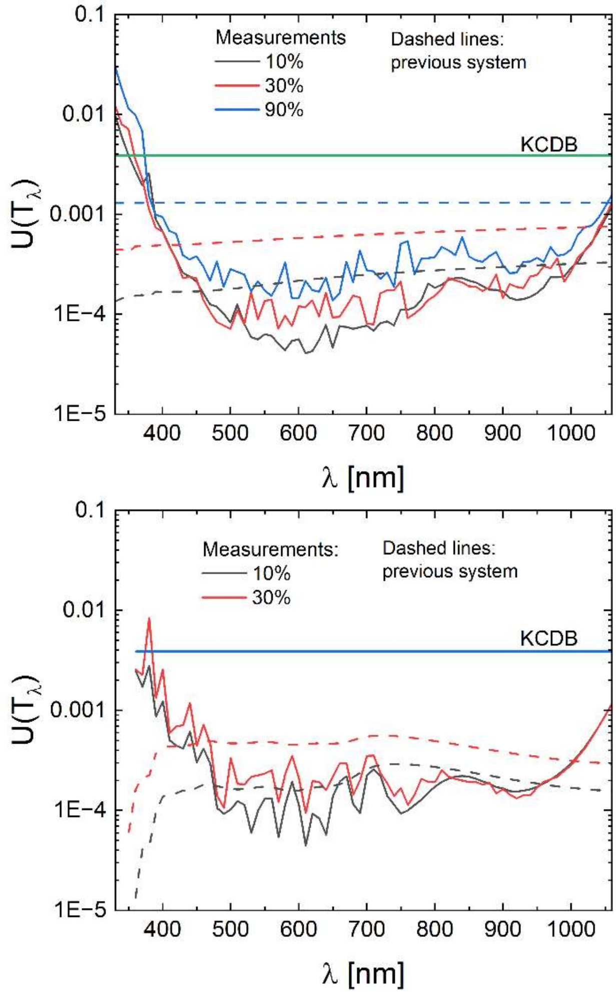

Figure 5. Uncertainty values for each filter in the (upper) SRM 2031a and (lower) SRM 930e filter sets. Dashed lines indicate the uncertainty computed in accordance to values declared in the SIM.K6.PR 2010 intercomparison. Horizontal lines represent the 0.39 % CMC limit reported by Inmetro in the KCDB [9].

Results indicate uncertainty values below the CMC limit of 0.39 % reported in the KCDB for the range from about 380 nm up to 1060 nm. Uncertainty is below 0.1 % for the range above 400 nm, where the QTH lamp employed is effectively intended to be used. Values reach down to 0.01-% level at a reduced spectral range around the central span.

Uncertainty values reported in the SIM.K6.PR 2010 intercomparison are also indicated in Figure 5. Current results are compatible and can reach up to one order of magnitude smaller values, indicating better performance of the updated system.

The last filter set was also measured using the PTCS, as shown in Figure 6. Values indicated in red are obtained from the average history calibration data using the spectrophotometer employed in calibration services (traced to other NMIs).

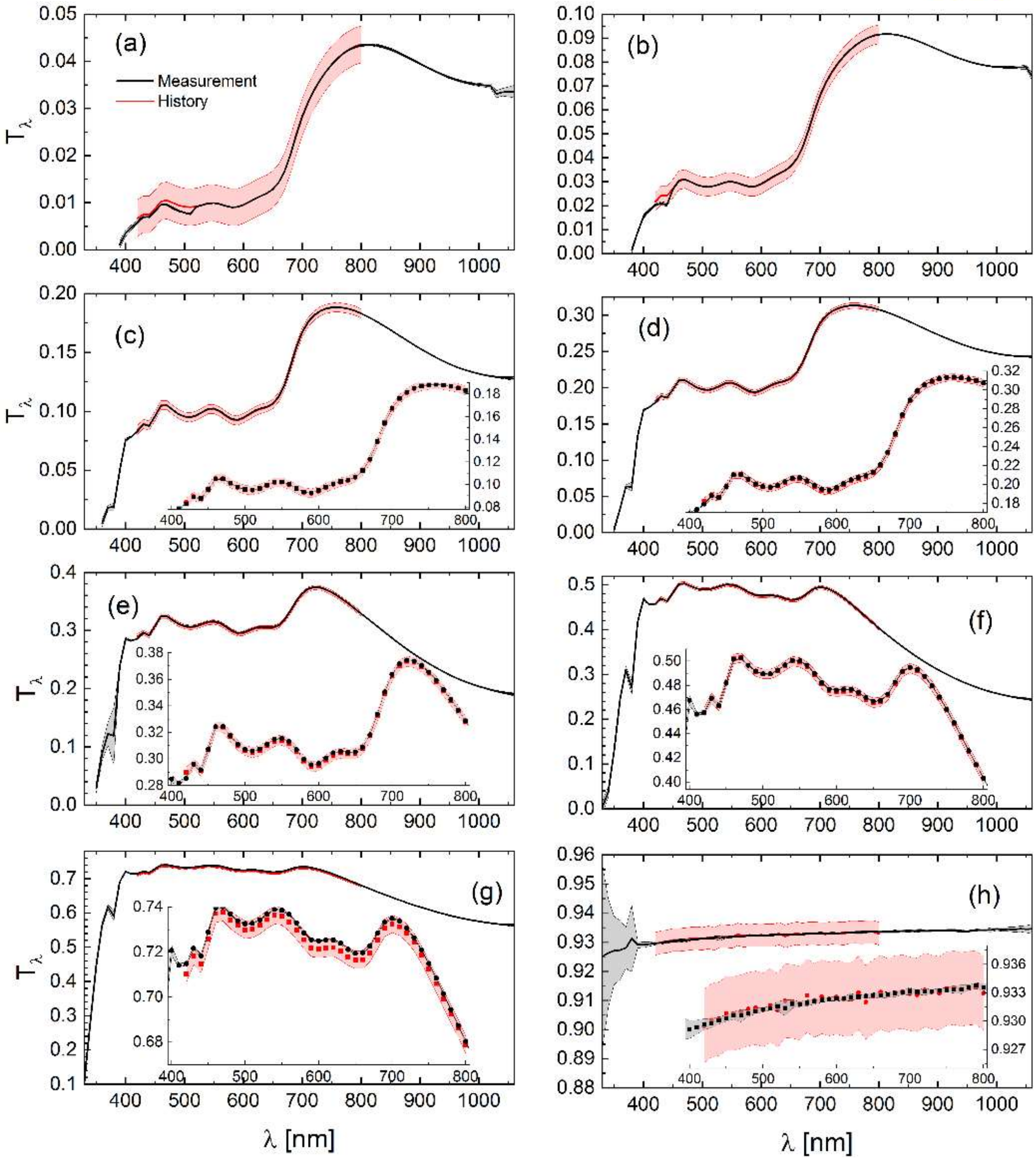

Figure 6. Regular spectral transmittance measurements of third set of filters: 1 %, 3 %, 10 %, 20 %, 30 %, 50 %, 75 % and 90 %, respectively from (a) to (h). Insets zoom in on the spectral range with comparable data. Color bands represent expanded uncertainty.

Values indicate that the transmittance values are compatible, and the uncertainty values of the measurements in the PTCS are significantly better than those of the historical data.

Figure 7 (a) shows the normalized error of the measurement values compared to the history data as reference for validation of results. Except for values near the UV region, most of the values are in agreement. In Figure 7 (b), we can see that all uncertainty values are below the value stated in the KCDB for the VIS-NIR region of the spectrum, and some of them are better than the SIM.K6.PR 2010 intercomparison.

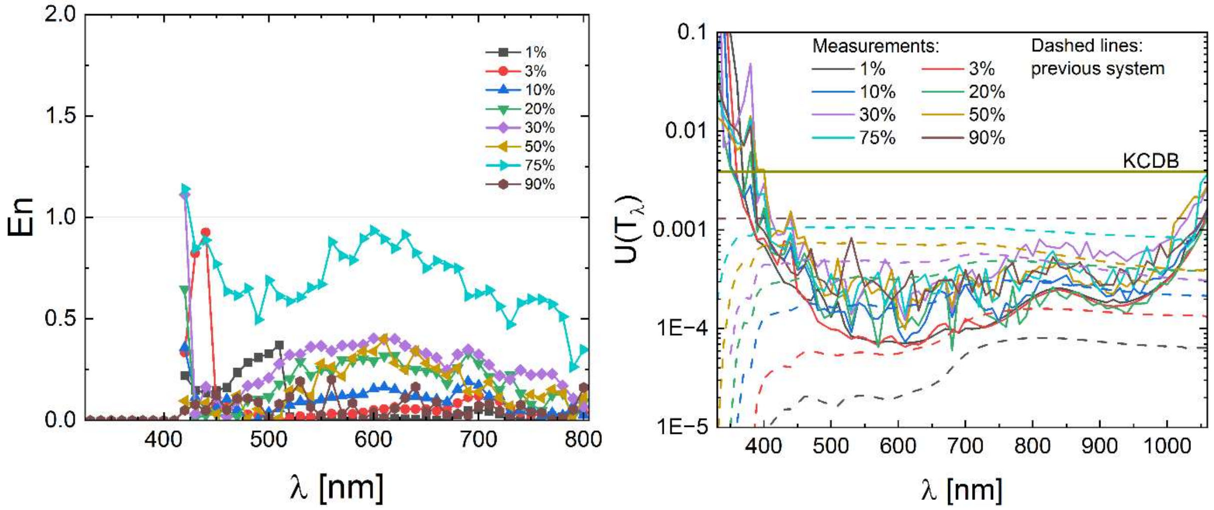

Figure 7. (a) Normalized error of the measurement values compared to the history data as reference for validation of results. (b) Uncertainty values for each filter in the third filter set. Dashed lines indicate the uncertainty computed in accordance to values declared in the SIM.K6.PR 2010 intercomparison. Horizontal lines represent the 0.39 % CMC limit reported in the KCDB.

Figure 8 shows the comparison between the spectral distribution of uncertainties for the input quantities in the 1% and 90% filters of the last filters set. Although the weight of contributions varies for both filters, both reproducibility and electronics have significant contributions.

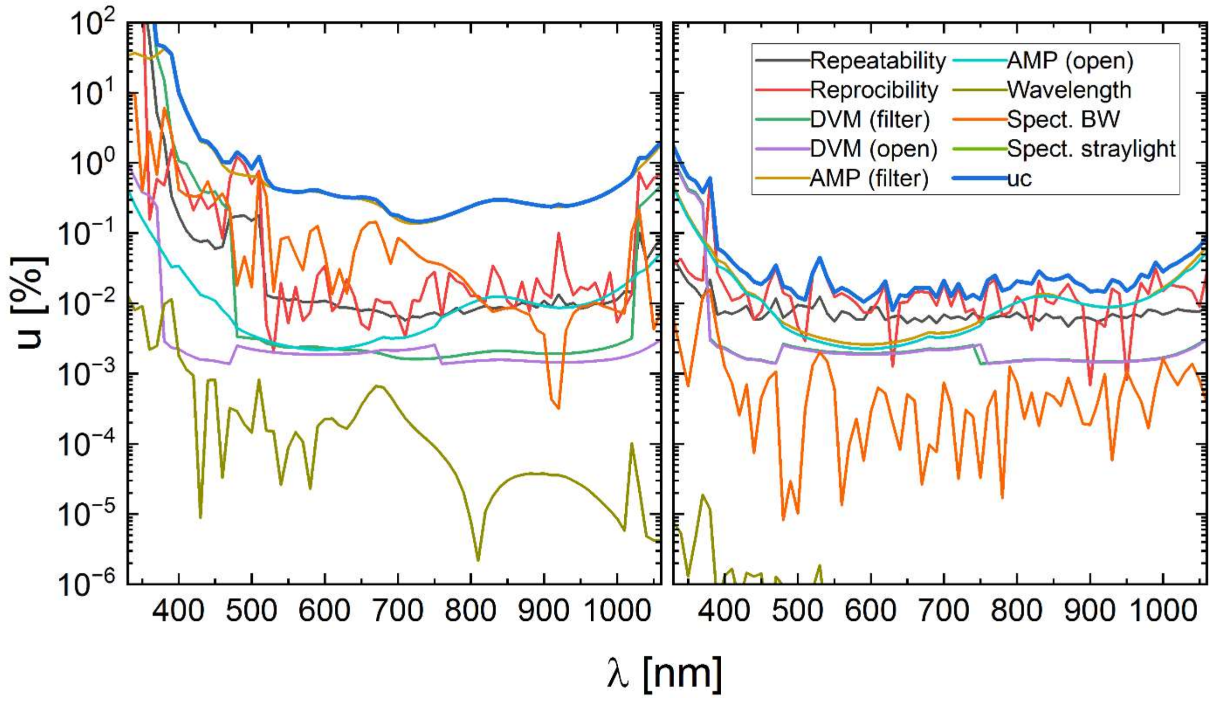

Figure 8. Comparison between the spectral distribution of uncertainties for the input quantities in the 1% and 90% filters of the last filters set. The value in blue represents the combined uncertainty.

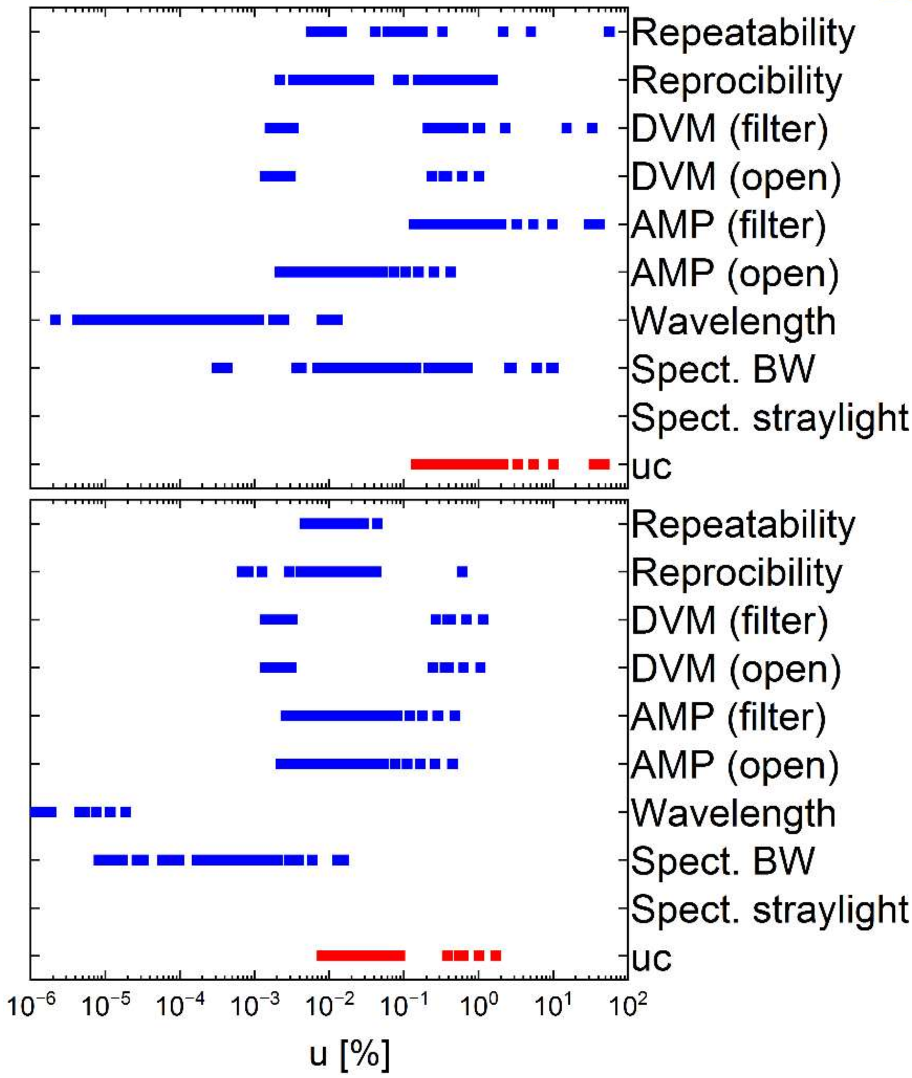

Figure 9. Order of magnitude of uncertainty contributions (in percentage values) for each input quantity for the 1% (above) and 90% (below) filters of last filters set. The value in red represents the combined uncertainty. Each horizontal line refers to a set of evaluated wavelengths.

Figures 9 shows the order of magnitude of uncertainty contributions (in percentage values) for each input quantity for the 1% (above) and 90% (below) filters of last filters set. Major contributions to the expanded uncertainty (in the UV region) come from the multimeters and amplifiers used in the measurements, as see at Figure 8. This can be attributed to the system operating close to the signal-to-noise ratio (SNR) in that region, suggesting that the SNR needs improvement for satisfactory UV region measurement in this system. One way to achieve this is by using a lamp with higher UV emission, such as a xenon lamp, for example.

However, for wavelengths above 400 nm, the uncertainty is below the CMC value in the KCDB, as observed. This indicates that for the current calibration range of this filter (from 420 nm to 800 nm), the utilized setup can be considered adequate.

Overall, the results obtained for all filters demonstrate good agreement with their calibration history, indicating the feasibility of using PTCS for the absolute characterization of optical filters in the visible and part of NIR region.

5. Conclusion

Based on the results presented in the study of the PTCS for the absolute characterization of optical filters, it can be concluded that the current setup exhibits excellent performance in the visible and part of NIR region for all evaluated filters. However, for wavelengths below 400 nm, the absolute error and expanded uncertainty exceeded the limit imposed by the uncertainty declared in the KCDB for some of the filters, indicating the need for improvements in the PTCS to cover the UV region.

Despite these deviations, the results demonstrate that the expanded uncertainty are lower than the uncertainty declared in the KCDB for most of the measurement range and the normalized error shows that the measurement are compatible with historical data, suggesting that the system can be confidently used for the calibration of optical filters in the visible and part of NIR region.

Regarding low-transmittance filters, such as the 1% filter from the commercial standard set filters, the absolute error remained below the uncertainty declared in the KCDB for most of the measurement range, suggesting that even for such low transmittance values, the uncertainty is lower than the declared value. For values above 520 nm, the expanded uncertainty were more than one order of magnitude lower than the uncertainty declared in the KCDB, indicating excellent performance of the system in that wavelength range.

Therefore, it can be concluded that the evaluation of the implemented improvement in the PTCS for the absolute characterization of optical filters has yielded promising results, indicating good performance of the system in the visible and NIR region for the calibration of optical filters. However, for wavelengths below 400 nm, some improvements are required to reduce deviations from the calibration history. Thus, future work will include the use of a xenon arc lamp for the UV region and the replacement of the amplifier/voltmeter set with a picoammeter with a measurement range of 2 nA to 20 mA and an uncertainty of a few tens of femtoamperes in the lower range. The expected result is a significant improvement in the UV region and SNR throughout the measurement range.

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