CALIBRATION OF NEUTRAL DENSITY GLASS FILTERS TO PRODUCE TRANSMITTANCE STANDARDS

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Abstract

Systematic errors in the ordinate scale of spectrophotometers are responsible for different results among different laboratories. Calibrated neutral density filters with different transmittance levels provide means for calibration to reduce the errors in the ordinate scale. The problem lies in obtaining an absolute transmittance measurement which can be used to calibrate these filters. Metrology laboratories obtain an absolute scale by determining a correction for the systematic errors using a method known in literature as the Double Aperture Method.

The paper describes the use of the Double Aperture Method in combination with neutral density glass filters in order to provide a simultaneous calibration of both the instrument ordinate scale and the glass filters. Results are discussed for filters which have been calibrated using a Perkin Elmer Lambda 900 spectrophotometer.Using the procedure described in this paper, an accuracy in the transmittance has been obtained which is better than 0.0005 in the UV/VIS/NIR wavelength region 250 nm - 2500 nm.

Key words: spectrophotometer, transmittance standard, gray filter

I. INTRODUCTION

The photometric accuracy in the ordinate scale of a spectrophotometer is limited by nonlinearity errors which in the case of measuring a transmittance T result in a systematic error $\Delta T = T_{TRUE} - T_{MEASURED}$. This error is a function of T and can be determined using reference materials (for example neutral density (ND) glass filters) with a known transmittance or using the Double Aperture (DA) method [1,2].

An ideal optical detector with a perfect linear response, which gives readings M_A and M_B for respectively radiant fluxes A and B, will give a reading $M_{AB} = M_A + M_B$ when the two fluxes A and B are incoherently added [1]. In the case $M_{AB} \neq M_A + M_B$ the detector response is nonlinear and this inequality can be used to quantify the nonlinearity. The DA method (see Fig. 1) is based on this addition principle.

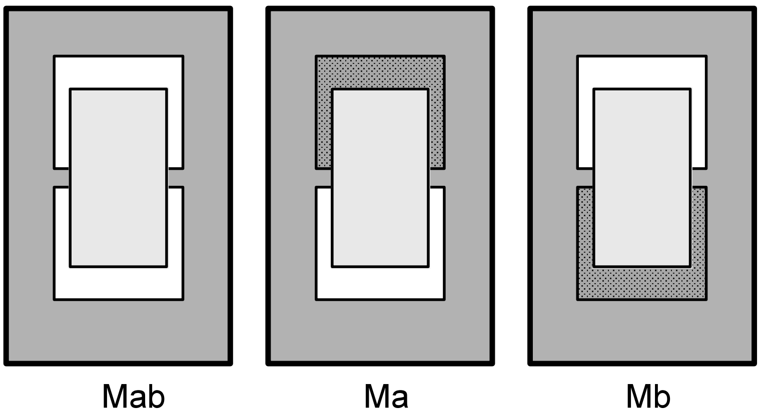

Fig. 1 Illustration of the Double Aperture method showing the three measurement modes with the cross-section of the instrument's beam projected on the two apertures.

The shape of the nonlinearity correction function $\Delta T(T)$ is usually derived from the equations describing the detector response in terms of detector gain, quantum efficiency and the parameters describing the behavior of the electronics [1,2]. In industrial spectrophotometers such a nonlinearity correction is already implemented in the hard- and software of the instrument. Nevertheless, results obtained with these instruments show a residual nonlinearity error which is typically in the order of 0.002 - 0.004 depending of the wavelength.

For the work presented in this paper, it is assumed that the transmittance of a glass sample is determined according to

$$T = \frac{M_T - < M_0 >}{< M_{100} > - < M_0 >} \quad , \tag{1}$$

in which M_T is the instrument reading on the sample with transmittance T. M_0 and M_{100} are taken before and after the sample reading. $\langle M_0 \rangle$ is the average of two 0% transmittance readings with the instrument beam blocked and $\langle M_{100} \rangle$ is the average of two 100% transmittance readings with the sample removed. A result of (1) is that by definition the nonlinearity in the transmittance scale will be zero at T = 0 and T = 1 and the residual nonlinearity in the detector response can be described by a series expansion of the form

$$\Delta T = \sum_{i=1}^{N} C_i T(1 - T^i) \quad , \tag{2}$$

where N is the minimum number of terms necessary to determine (2) sufficiently accurate. The purpose of the DA method is to find the values of the constants C_i.

A new procedure based on this method is proposed in this paper. This procedure can be used with a modern instrument like the Perkin Elmer Lambda 900 UV/VIS/NIR spectrophotometer, to calibrate simultaneously the ordinate scale of the instrument (in combination with a particular detector unit) and a set of ND filters which serve as transfer standards for the calibration of other detector units.

II. EXPERIMENTAL PROCEDURE

The DA accessory that was built at TNO consists of two square apertures separated by a 1 mm high septum (see Fig. 1) which is placed approximately at the height of the optical axis so that $M_A \approx M_B$. This septum is responsible for a transmittance loss of approximately 10% which is largely compensated by the removal of the fused silica entrance window of the sample chamber. This results in approximately the same maximum energy (M_{100}) with or without DA accessory. Each aperture can be opened or closed manually by a sliding shutter which is coated black to reduce the effect of stray radiation. The shutters are placed under an 80° angle (instead of 90°) with respect to the optical axis in order to prevent the occurrence of interreflections with the instrument's optics and with the filter.

A schematic drawing of the measurement setup in the Perkin Elmer Lambda 900 UV/VIS/NIR spectrophotometer is shown in Fig. 2. The measurements are performed in a time sequence of the following character:

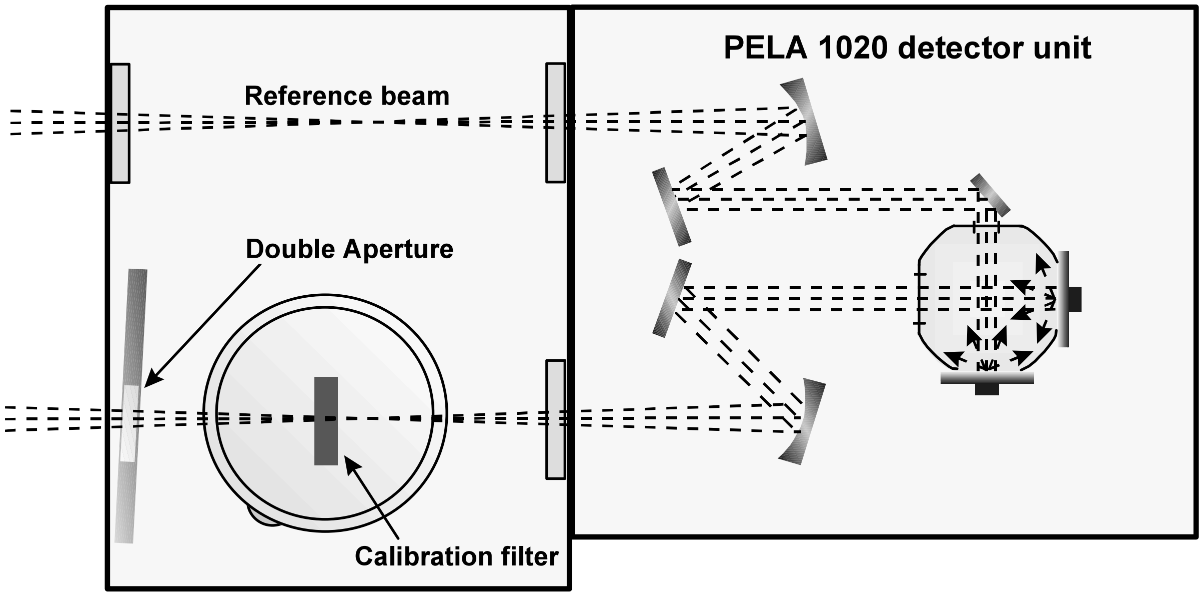

Fig. 2 Measurement geometry showing the position of the DA accessory and the ND calibration filters.

$$M_0,\ M_{100},\ M_{Ta},\ M_{Tb},\ M_{Tab},\ M_{Tb},\ M_{Ta},\ M_{100},\ M_0, \qquad (3)$$

where M_0 and M_{100} are taken with the ND filter removed and both apertures of the DA accessory respectively closed (M_0) or open (M_{100}). The readings M_{Ta}, M_{Tb} and M_{Tab} are taken with the ND filter in position and the aperture settings according to Fig. 1. The time sequence is repeated six times in order to reduce random errors. For the determination of the number of terms in (2) and the values of the constants C_i measurements where performed without filter and with 8 different filters representing a total of 9 different transmittance levels.

III. RESULTS

To give an example, measurement results obtained at 1500 nm are given in table 1. The results in this table represent averages of 6 measurements for different transmittance levels in the range 10% - 100%. The filters ICCG1 - ICCG5 are ND filters which were issued by the International Commission on Glass in the 1980's. They are intended to be used as reference standard for checking the ordinate scale of spectrophotometers but were never calibrated and certified. The filters br86 and bq86 are ND filters issued and certified by NPL [3]. The certified values have an uncertainty of 0.005. The 2 mm infrasil sample (Hereaus) was included to provide an extra transmittance level (approximately 90%).

A total number of 9 x 9 x 6 = 486 spectra were recorded: 9 spectra per sequence (3), 9 different transmittance levels, 6 measurement sequences per transmittance level). Fig. 3 shows one set of transmittance spectra determined from one measurement sequence. The nonlinearity correction (2) was determined by fitting (2) on these 486 spectra. The best results were obtained with just one term:

$$\Delta T = C\, T(1 - T) \qquad (4)$$

(equation (2) with N = 1). The values for the constant C are shown in Fig. 4.

Table 1. Results obtained with the DA accessory for a wavelength of 1500 nm.

Filter	Ta	Tb	Tab	Tab -Ta -Tb
blank *)	0.51781	0.48092	1.00000	0.00127
ICCG1	0.05675	0.05126	0.10802	0.00001
ICCG2	0.16109	0.15350	0.31451	-0.00007
ICCG3	0.25259	0.23151	0.48464	0.00054
ICCG4	0.29766	0.27852	0.57647	0.00029
ICCG5	0.39290	0.37094	0.76445	0.00061
br86	0.14523	0.13786	0.28322	0.00013
bq86	0.24330	0.22977	0.47332	0.00025
infrasil 2mm	0.48369	0.45052	0.93536	0.00114

*) no filter present.

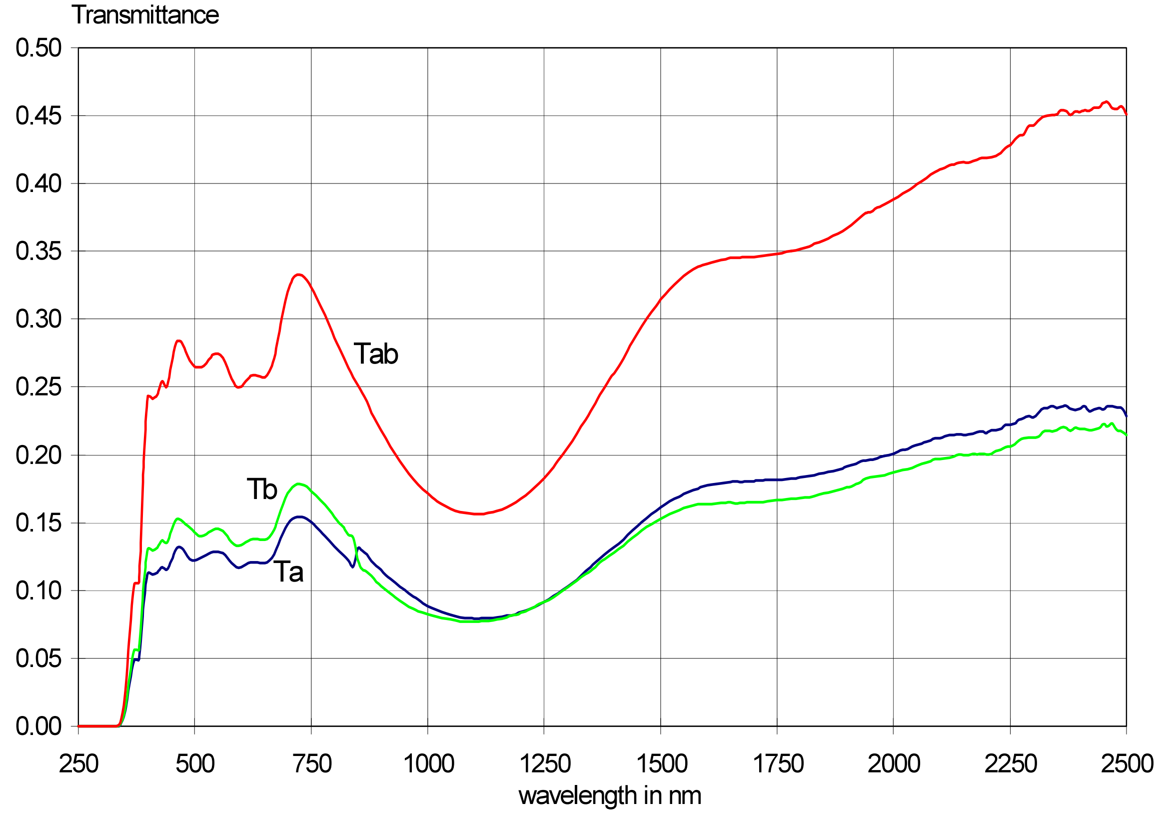

Fig. 3. One set of transmittance spectra determined for filter ICCG2

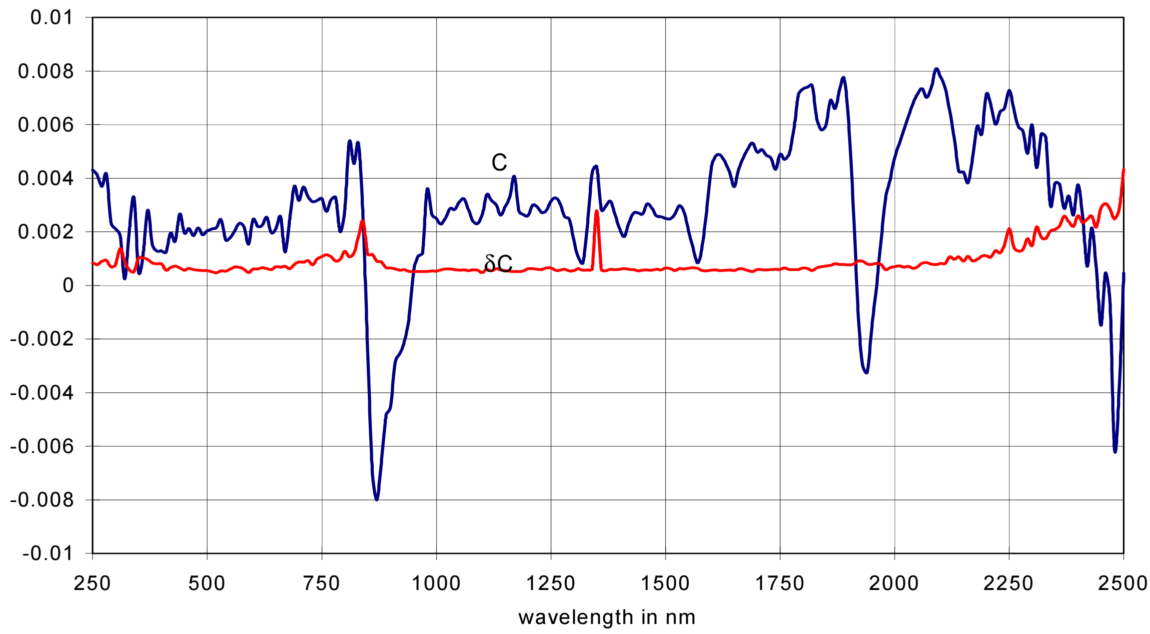

Fig. 4 Nonlinearity parameter C and uncertainty δC (95% confidence level).

The systematic error in T related to the error in C (after correction for nonlinearity) is given by

$$\delta T_s = T(1-T)\delta C \quad (5)$$

The uncertainty $\delta\lambda$ in the wavelength setting of the spectrophotometer results in a systematic uncertainty in $T(\lambda)$ given by

$$\delta T_w = \left|\frac{\partial T}{\partial \lambda}\right| \delta\lambda \quad (6)$$

The partial derivative in (6) is determined by applying the central difference approximation for each determined value of $T(\lambda_i)$ using $T(\lambda_{i-1})$ and $T(\lambda_{i+1})$ and taking a one-sided difference at the end points. Although the uncertainty in the wavelength consists of a systematic as well as a random component, the total uncertainty is assumed to be systematic with values of 0.1 nm for the UV/VIS range (λ < 860 nm) and 0.3 nm for the NIR range (λ > 860 nm).

The random error related to the reproducibility is calculated from the standard deviation S in the transmittance measurements by

$$\delta T_r = t_{m-1} \frac{S}{\sqrt{m}} \quad (7)$$

where m is the number of transmittance measurements and t is the Student t-factor [4] for m-1 degrees of freedom and a 95% confidence level.

The transmittance spectra of the five ICCG filters after correction for nonlinearity are shown in Fig. 5. The total measurement uncertainty which is also shown in Fig. 5 is given by the summation of its three components δT_s, δT_w and δT_r.

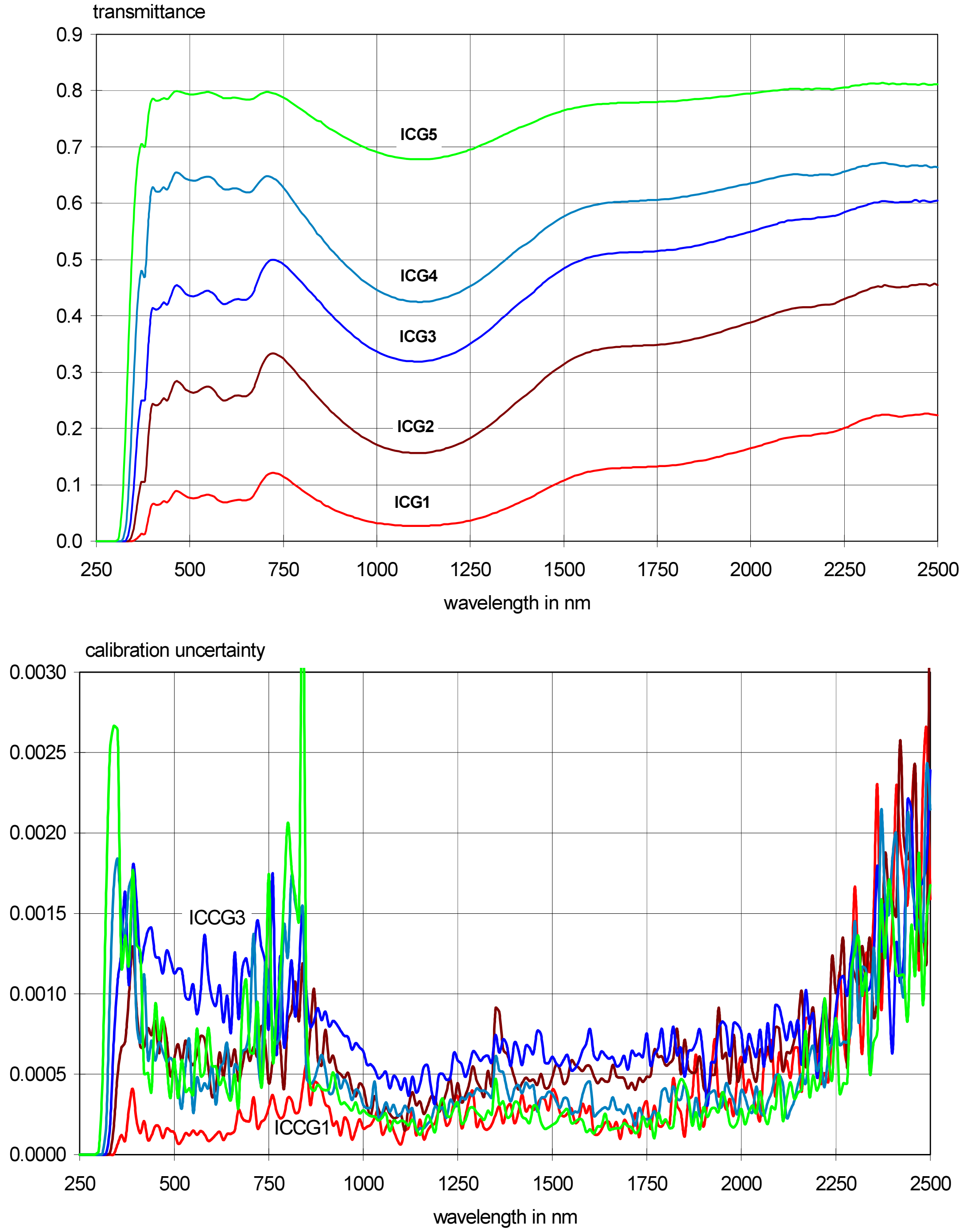

Fig. 5. Corrected spectral transmittance and corresponding calibration uncertainty of the five ICCG neutral density filters.

IV. DISCUSSION

Due to the fact that the best results were obtained with only a single term in the correction function, leading to (4), we need only one transmittance level to obtain the value of C. Future calibrations of the instrument (Lambda 900 equipped with the PELA 1020 detector unit) will therefore require only the measurements without filter. For this case the following expression is valid:

$$C = \frac{1}{1 - \frac{Tab^2 - Ta^2 - Tb^2}{Tab - Ta - Tb}} \quad . \tag{8}$$

In the case that Ta ≈ Tb ≈ 0.5, we can simplify things even more and obtain

$$C \approx 2(Tab - Ta - Tb) = 4(\Delta T)_{max} \quad . \tag{9}$$

The column Tab - Ta - Tb in table 1 clearly shows the effect of nonlinearity. As expected the effect is the largest for Ta ≈ Tb ≈ 0.5 (measurement with no filter present). The result of the nonlinearity correction obtained in the present investigation is shown for one wavelength in Fig. 6. The maximum nonlinearity error found at this wavelength is 0.00065. These results clearly demonstrates the effectiveness of the proposed method. At other wavelengths the result is similar. After the correction the values of the term Tab - Ta - Tb are much closer to zero. In fact, the total uncertainty shown in Fig. 5 is dominated by δT_r, a random component which can be reduced by simply taking more measurements.

The measurements of the ND filters may be separated from the calibration of the instrument's ordinate scale. However, the random errors occurring during the DA measurements contribute to the total error in the nonlinearity correction which should be treated as a systematic error when this correction is used to calibrate the filters. If the calibration of ordinate scale and filters is performed simultaneously they share the same random error component which means that the total calibration error for the ND filters will be less in this case.

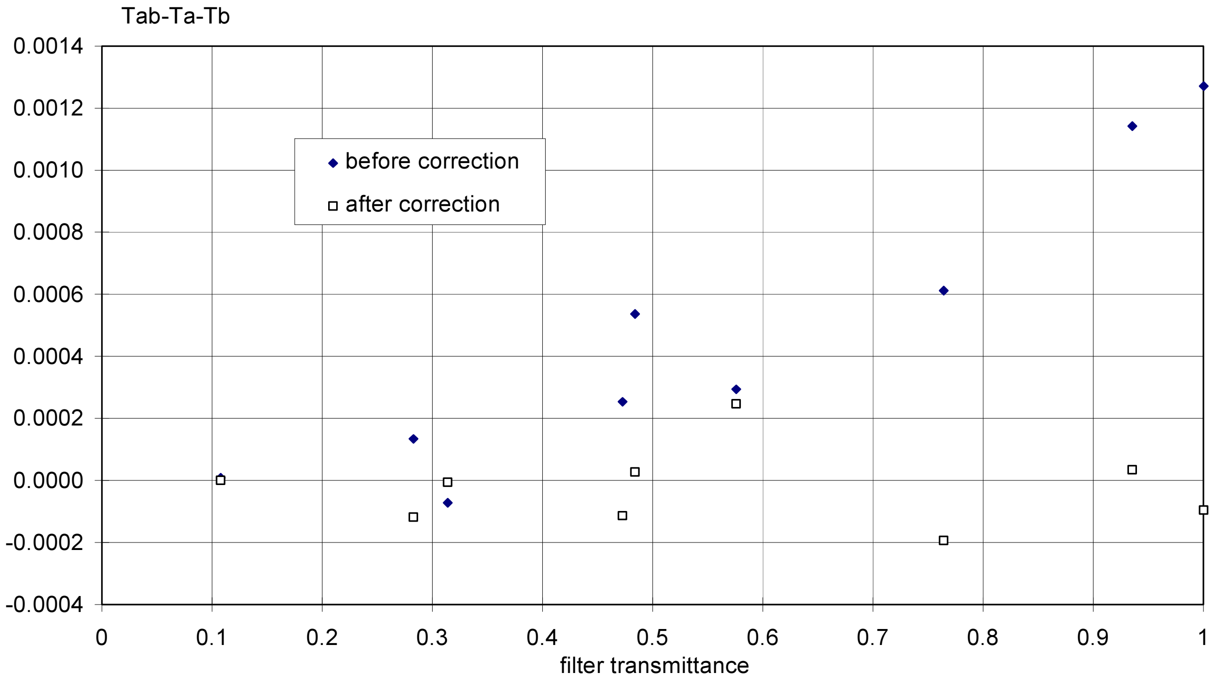

Fig. 6. Tab - Ta - Tb as function of the filter transmittance (Tab) before and after the correction according to (4)

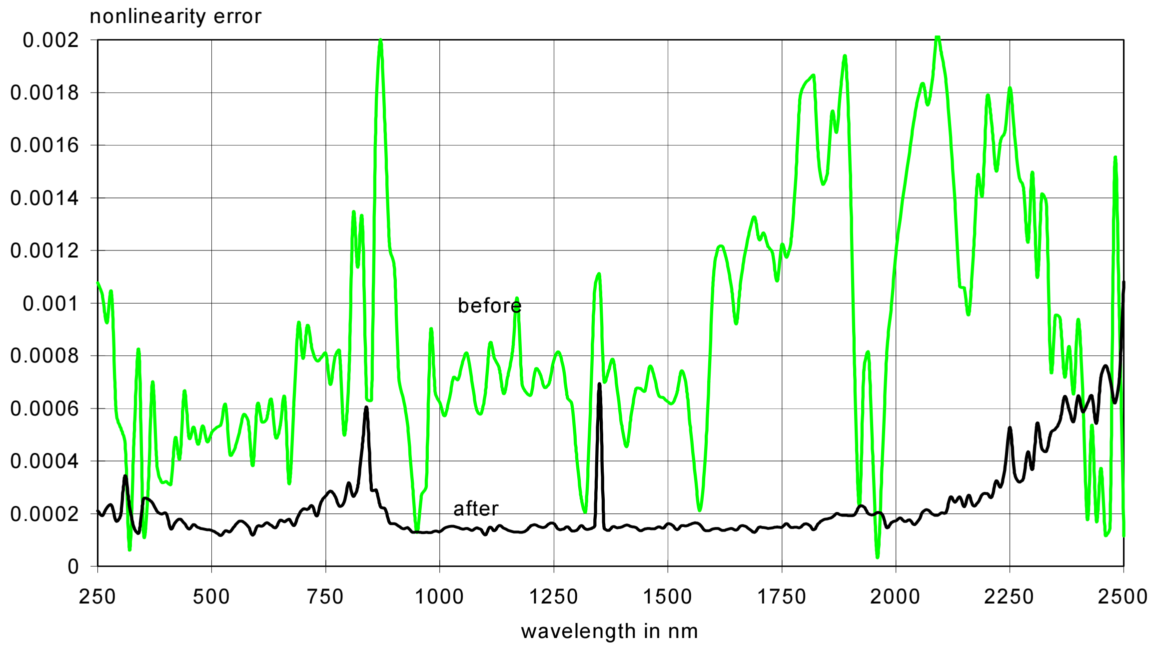

Fig. 7. Photometric accuracy of the spectrophotometer before and after correction.

V. CONCLUSION

The DA method is mostly used by metrology laboratories to obtain an absolute scale for their spectrophotometers. Industrial laboratories, by measuring the transmittance of calibrated ND filters, usually only check the nonlinearity error but they are not in the habit of using the results to make a correction and thereby obtaining better values.

A relatively simple method is proposed in this paper according to which the accuracy (systematic error) of the ordinate scale of an industrial spectrophotometer like the Lambda 900 can be enhanced to the level of 0.0002 - 0.0008 depending on the wavelength. A set of neutral density filters was calibrated, obtaining an overall total calibration uncertainty < 0.0015. The filters will be used as transmittance standards for checking and/or calibrating the ordinate scales of other spectrophotometers or the same spectrophotometer in combination with other detectors and/or accessories.

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