The Measurement of Diffuse Optical Densities. Part II: The German Standard Reference Densitometers

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This article describes the design and performance of two densitometers used for high-accuracy measurements of visual diffuse optical transmission densities. The two densitometers were developed and constructed at the Physikalisch-Technische Bundesanstalt (PTB), the German National Institute of Metrology. Both devices comply with the ISO standards 5-2 and 5-3 and are primarily used for the calibration of standard step tablets. One of these densitometers, the PTB's inverse square law densitometer, is the national standard reference densitometer capable of measuring with high accuracy optical densities up to $D = 3.3$. The other densitometer, called fiber densitometer, measures densities up to $D = 6$. For both densitometers, the expanded ISO uncertainty of density measurement (coverage factor $k = 2$) is 0.003 for densities below 1.5 and increases to 0.006 at density 6 for the fiber densitometer with spectrally neutral samples.

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Introduction

Accurate values of diffuse optical densities of scattering samples are needed for many purposes: quality assurance in medical x-ray diagnostics, industrial non-destructive x-ray testing, applications in molecular biology (DNA sequencing, for example), quality control in graphic arts, and, of course, for characterizing photographic materials. For the calibration and testing of densitometers, suitable test objects, e.g., standard step tablets, with well-known optical densities are used.

Such step tablets are calibrated by calibration services which need reference standards. For reasons of traceability, these reference standards are calibrated with very high accuracy by National Institutes of Metrology such as NIST and PTB. They develop standard measuring equipment that is capable of measuring optical densities directly, without any step tablets being needed for calibration. The PTB has developed two such densitometers for the measurement of visual diffuse transmission densities. In this article we present the measuring arrangements and principles used in these two devices, discuss the performance of these densitometers and their measurement uncertainties.

ISO Standards for the Measurement of Optical Densities

The definition of optical density and the conditions of the measuring systems are given in the International Standards ISO 5, parts one to four. We present here only a very brief survey of these standards, especially with respect to the measurement of visual diffuse transmission density.

According to ISO 5-1,[1] the optical transmission density D is defined as

$$D = -\log_{10} T \tag{1}$$

where the transmittance factor T is the ratio of the measured flux transmitted by the specimen to the flux measured when the specimen is removed from the sampling aperture of the densitometer.

The geometric measuring conditions are specified in ISO 5-2[2] and ISO/DIS 5-2[6] for transmission density and in ISO 5-4[4] for reflection density. The ISO standard diffuse transmission density is based on a method, where the sample is in "close contact" with the surface of a diffuse illuminating or receiving system. Because this surface is mostly realized by an opal glass, this method is usually called "opal glass" method. The term "close contact" means that there is a close proximity with an air gap to simulate conditions of the practice where inter-reflections between sample and opal glass are present. Because these inter-reflections influence the measurement result, ISO 5-2 specifies the reflection factor of the opal glass. ISO 5-2 also specifies the diffusion coefficient of the diffuser. In part I of this series of articles,[5] the ISO geometric conditions for determining diffuse transmission densities were discussed in detail.

The spectral conditions for the measurement of optical densities are described in ISO 5-3,[3] where both the spectral energy distribution of the incident radiant flux and the spectral product of the measuring system (i.e., the product of the influx spectrum and the spectral response of the densitometer) are specified for various applications. For transmission density measurements,

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Figure 1. Set-up of inverse square law densitometer.

Figure 2. (a) Modulation scheme applied in inverse square law densitometer. and (b) reference and sample beam, both signals have the same waveform which is out-of-phase by 180°. (c) detector output signal with the sample beam attenuated by sample. (d) and (e) chopper reference output signals.

the spectral distribution of the influx is the CIE standard illuminant A, modified by the transmittance of a typical heat-absorbing filter to protect the specimen and the optical system from heat. The functional notation of this influx spectrum is S_H according to ISO 5-3. The spectral product required for the visual transmission density is defined as the product of S_H and a spectral response of the receiver, V_T, in order that this product be equal to the product of the spectral luminous efficiency function for photopic vision, $V(\lambda)$, and the spectral distribution of CIE standard illuminant A.

PTB's Inverse Square Law Densitometer. The PTB's inverse square law densitometer is the German national standard reference densitometer capable of measuring visual diffuse transmission densities according to ISO 5-2 and ISO 5-3. It applies the "diffuse efflux mode" measuring configuration,[2] where the film sample is illuminated directionally and the transmitted flux is measured by means of a diffuse receiver.[5] The densitometer is a dual-beam device: The luminous flux is time-shared by the reference and sample path, and it is detected by applying phase-sensitive lock-in techniques. Besides the well-known advantages of the dual-beam technique, the influence of low-frequency noise contributions is reduced. The densitometer uses a null-balance measuring configuration based on the fundamental photometric inverse square law, and therefore linearity of the optical detector system is not an indispensable prerequisite.

Densitometer Set-Up and Measuring Principle. The diagram of the measuring set-up of the densitometer is shown in Fig. 1. The light of a tungsten halogen lamp is split into two beams, the reference and the sample beam. A mechanical chopper, equipped with a specially shaped chopping blade, is used to chop both light beams simultaneously. The resulting modulation waveforms are shown in Fig. 2. The two light beams show repetitive pulse groups, each of which consists of four equal square-wave light pulses. The repetition frequency of the pulse groups is f = 72 Hz, and the frequency of the single pulses is 8f. The frequencies are chosen so as to avoid any low-order harmonic of f and 8f coinciding with a low-order harmonic of the power line frequency. The modulation frequencies f and 8f are phase-locked to avoid possible low-frequency beating due to the same chopper blade being used to create these waveforms. The waveforms of the reference and the sample beam are out-of-phase by 180° with respect to each other, i.e., whenever light from the reference beam reaches the photodetector, light from the sample beam is blocked, and vice versa.

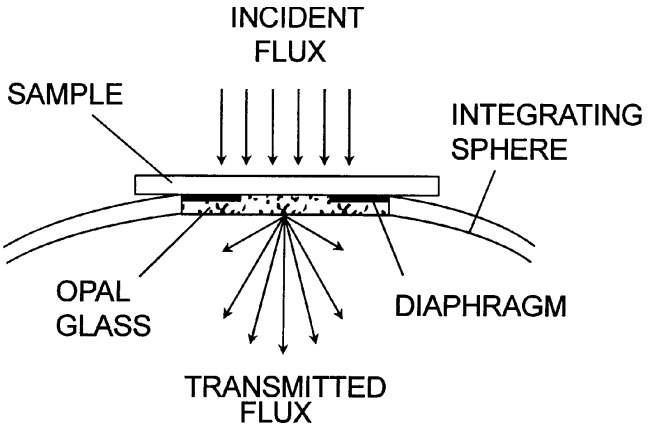

Figure 3. Diffuser used in the inverse square law densitometer.

The luminous flux of the reference beam, Φ_R, passes neutral grey filters and a variable beam aperture (for intensity adjustment). It then directly reaches an integrating sphere (sphere diameter: 11 cm; coating: barium sulphate), which is provided with a photodetector (EMI 9558 photomultiplier). An optical filter combination mounted in front of the photomultiplier is used to meet the ISO spectral conditions for measuring visual densities. The sample is in contact with an opal glass that is located in another opening of the integrating sphere, see Fig. 3. The sample beam is directed onto the sample via two mirrors mounted on a long-travel linear translation-stage (length: about 6 m). Variation of the translation stage position leads to a change in the length of the sample beam path and, as a consequence, to a corresponding change of the incident luminous flux because of the photometric inverse square law. The length of the reference beam path is not altered if the translation stage position is varied.

Because the photodetector captures both the reference beam flux Φ_R and the transmitted sample beam flux Φ_S, its output signal comprises the superposed contributions from the reference and sample beam signals. The photodetector output signal is amplified and subsequently fed to two equal digital lock-in amplifiers (Stanford SR 810). Lock-in amplifier No. 1, which is locked to the frequency f, detects the difference between reference and sample beam flux, $\Phi_S - \Phi_R$, whereas lock-in amplifier No. 2, synchronized by the 8f-reference output signal from the chopper, detects the sum of reference and sample beam flux, $\Phi_S + \Phi_R$. The digital lock-in output signals are transmitted to a computer that calculates the ratio of the sum and the difference of the two lock-in output signals, yielding Φ_S / Φ_R as the result. Density measurements are performed in two steps:

1. The first measurement is made with the sample removed. An adjustment is made to ensure that the photodetector signal contributions coming from the reference beam and the sample beam are equal, i.e., $\Phi_S / \Phi_R = 1$. To attain this balance condition, the luminous flux of the sample beam is adjusted by positioning the translation stage on the optical bench. This stage position is measured and the corresponding sample path length, $s_{100\%}$, is determined.
2. The second measurement is performed with the sample arranged in front of the opal glass. The photodetector output signal contribution resulting from detecting the sample beam flux is reduced according to the transmittance factor T of the sample (see Fig. 2c), and the quantity computed from the two lock-in output signals, Φ_S / Φ_R, then directly yields the desired transmittance factor T. However, this value of T may be affected by possible nonlinearities of the characteristic curve of the photodetector/amplifier combination. In order to neglect this contribution to the measurement uncertainty, the translation stage is moved to a new position on the optical bench (closer to the lamp) where the null-balance condition, $\Phi_S - \Phi_R = 0$ or, equivalently, $\Phi_S / \Phi_R = 1$, is fulfilled as was the case without sample. If the length of this shorter sample path is denoted as s_T, the sample transmittance factor is easily obtained by applying the inverse square law:

$$T = \left(\frac{s_T}{s_{100\%}} \right)^2 \tag{2}$$

Therefore, the measurement of the optical density is reduced to two length measurements which can be performed with sufficient accuracy. The measuring range is limited to densities of up to about 1.65 because of the maximum and minimum sample path lengths possible. However, with the help of an auxiliary sample with a density of about 1.65 (which has to be well-known) and by attenuating the reference beam flux accordingly, it is possible to achieve the balance condition once again at large sample path lengths. This procedure renders it possible to measure sample densities up to twice the value of 1.65, i.e., up to 3.3. A third follow-up measurement is not possible because of a too low signal-to-noise ratio, which is due to the relatively weak radiation emitted by the tungsten lamp.

ISO Geometric and Spectral Conditions. The densitometer complies with the geometric conditions specified in ISO 5-2, i.e., the diffusion coefficient value is larger than 0.90 and the reflectance factor of the diffuser is between 0.50 and 0.60.

Figure 3 shows in detail the arrangement of the diffuser. It consists of an opal glass combined with an integrating sphere. This arrangement was chosen because the diffusion coefficient value of the 0.42 mm thick opal glass alone is not high enough. The thickness of the opal glass was selected to achieve a reflectance factor of 0.55 of the combination (sphere and opal glass) for a wavelength of about 570 nm which is the peak wavelength of the spectral product for visual density measurements. Due to light scattering, the reflectance factor of the diffuser decreases with increasing wavelength from 0,60 for a wavelength of 420 nm to 0.50 for 710 nm.

The geometrical distribution of the diffuse receiving system is shown in Fig. 4, together with the distribution of a perfect diffuse receiver that has a cosine spatial response, and, by definition, a diffusion coefficient of 1. The diffusion coefficient of the real diffuser is 0.945 calculated according to Annex C of ISO 5-2. If the proposed new definition, given in the revised Draft ISO Standard ISO/DIS 5-2,[6] is applied, the diffusion coefficient is 0.909. Please note, that the value for the diffusion coefficient, stated in the first part of this series of papers,[5] no longer holds because the instrument was meantime modified.

The measuring aperture (size: 5 mm × 3 mm) is defined by a diaphragm that is located between sample

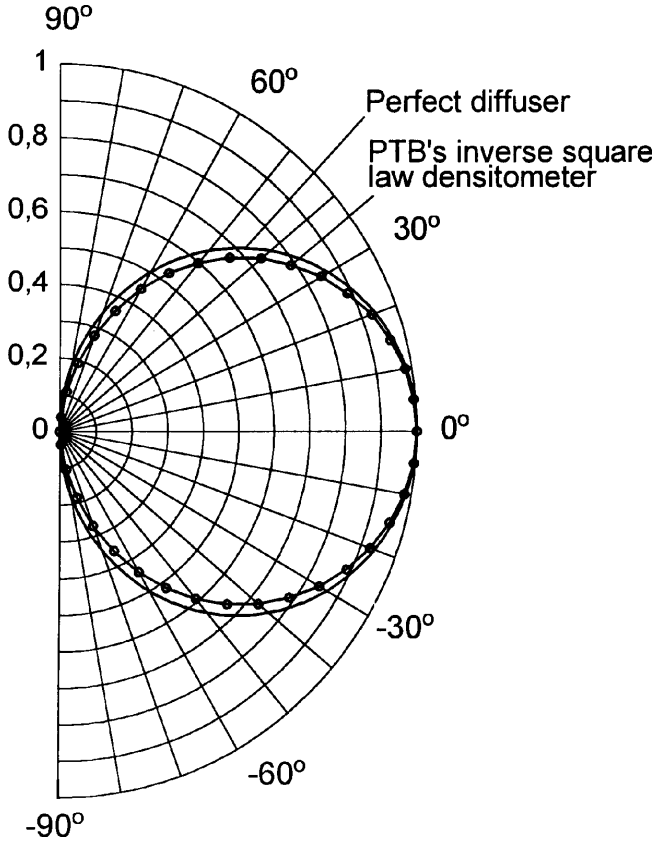

Figure 4. Geometrical distribution of the diffuse receiving system of the PTB's inverse square law densitometer.

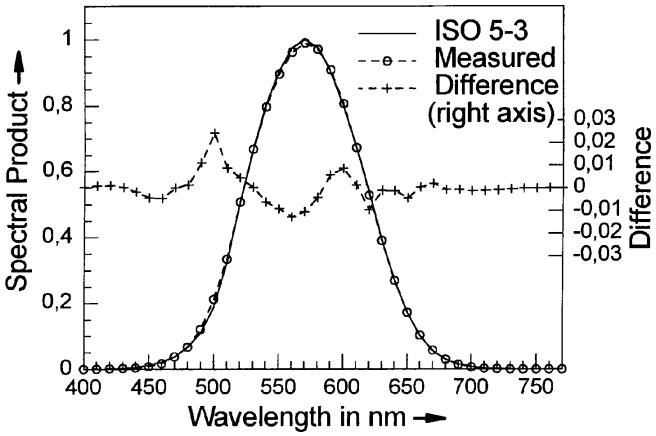

Figure 5. Spectral product as specified in ISO 5-3 and measured for the inverse square law densitometer. The difference between both curves is also shown.

and opal glass. To assure that sample and opal glass are in close contact with each other, the diaphragm is inlaid into the opal glass such that there is a smooth surface.

Figure 5 shows the spectral product for visual densities according to ISO 5-3 together with the spectral product of the densitometer, that was obtained by multiplying the measured emission spectrum of the tungsten lamp by the spectral response of the complete diffuse receiver system. If the CIE error definition used for the specification of the spectral response of photometers is applied,[7] a value of 1.4% for f'_1 is measured.

Measurement Uncertainty. To determine the combined measurement uncertainty, the ISO Guidelines for the expression of uncertainty in measurement are applied.[8] The expanded uncertainty $U(D)$ is specified, which is obtained by multiplying the combined standard uncertainty by a coverage factor k. The value of the coverage factor chosen is 2.

The repeatability of measurement (repeated measurement of the same sample under unchanged conditions) was measured; the corresponding expanded uncertainty is $U(D) < 0.001$ for $D < 1.65$ and $U(D) = 0.0025$ for $D > 1.65$.

Systematic uncertainty components originate from the uncertainty where the determination of the sample path lengths $s_{100\%}$ and s_T are affected: Here, the problem is not the measurement of the translation stage position on the optical bench, but proper knowledge of the effective distance between lamp source and detector along the optical beam path. In practice, the problem is to accurately measure an "offset" distance which has to be added to the readings of the translation stage positions. We determine this offset distance by means of the inverse square law, by extrapolation to zero distance. For this measurement, a linear photodetector is needed. The possible non-linearity of the photodetector and the extrapolation to zero distance lead to an uncertainty of the offset distance. This in turn results in a corresponding uncertainty of the density measurement which is density dependent and can be described by $U(D) = 0.0016 \cdot D$.

Another item concerns the photometric inverse square law itself. The largest dimensions of the tungsten light source and of the sampling aperture are 5 mm, which is much smaller than the nearest distance between light source and detector (about 1700 mm). Therefore, the conditions for the applicability of the inverse square law are by far fulfilled; the uncertainty contribution due to this effect is far below 0.001. However, due to additional, undesirable signal contributions (e.g., stray light from the mirrors or other sources), small deviations from the ideal inverse square law occur. Careful measurements of these deviations show a stable and systematic behavior which is taken into account in the measurement evaluation. The residual uncertainty of measurement caused by this effect is density dependent, $U(D) = 0.0007 \cdot D$.

Other sources of error are due to spatial non-uniformities of the radiation emitted by the lamp, and due to residual lock-in phase variations between the reference and the sample beam signals when the translation stage moves along the optical bench. The reason for both errors is that the solid angle of that part of the light beam that contributes to the signal, changes with the stage position. For instance, a larger distance between the sampling aperture and the lamp results in a smaller solid angle of the light beam falling onto the sampling aperture. Because the chopper blades hit the beam perpendicularly, the chopped light undergoes a phase variation. Other contributions to the uncertainty are the measurement uncertainties of the lock-in amplifiers. The total effect of all these uncertainties can be estimated to be below 0.002.

The slight deviations of the spectral product of the densitometer from the ISO spectral product will lead to corresponding systematic measurement errors if the spectral transmission of the sample depends on the

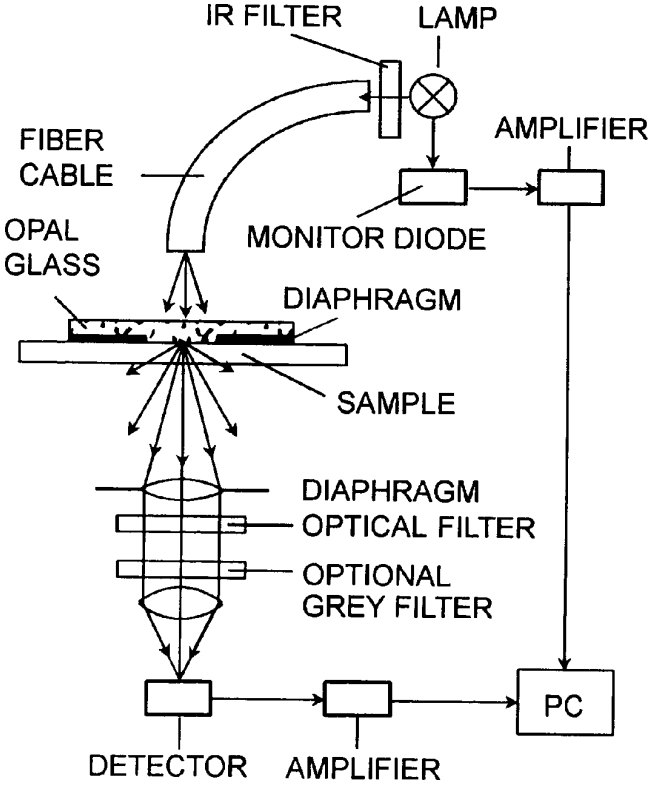

Figure 6. Set-up of fiber densitometer.

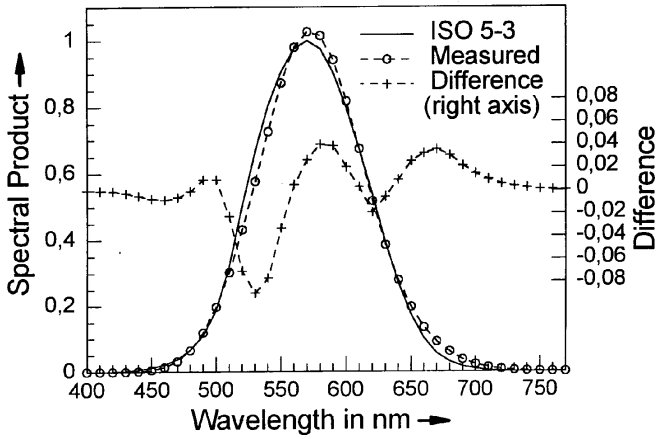

Figure 7. Spectral product as specified in ISO 5-3 and measured for the fiber densitometer. The difference between both curves is also shown.

wavelength. These systematic measurement errors can be corrected if the relative spectral transmission of the sample is known. For example, for medical x-ray films with their typical bluish base, the densitometer described here will indicate density values that deviate by about 0.001 from those of an ideal densitometer with exactly the spectral product specified in ISO 5-3.

The effects of the diffusion coefficient value on the density values measured have already been discussed in detail in part I of this series of papers especially with respect to the inverse square law densitometer.[5] This discussion need not be repeated here, and the total uncertainty values stated below do not take this aspect into account.

The resulting total measurement uncertainty for the ISO visual transmission density of spectrally neutral samples depends on density and can be expressed by $U(D) = 0.0024 \cdot D$ for densities above 1.5; for densities below 1.5 the uncertainty is $U(D) = 0.003$.

PTB's Fiber Densitometer. The fiber densitometer is specially designed for the measurement of high optical densities of neutral samples, and it thus complements the field of application of the inverse square law densitometer.[9] It is a single-beam densitometer that applies the "diffuse influx mode" configuration[2] using an opal glass as a diffuser. In this measuring mode, the sample is illuminated by diffuse light, and the transmitted luminous flux is detected with a directional receiver.

Densitometer Set-Up and Measuring Principle. A diagram of the densitometer is shown in Fig. 6. The light of a tungsten halogen lamp, of which the IR radiation is removed with a heat-absorbing filter, is coupled into a so-called randomly mixed glass fiber cable (length: 1 m, diameter: 13 mm), where the input and output positions of the single fibers do not correlate. This ensures uniform light emission over the sectional area of the fiber cable output, despite the fact that illumination of the cable input area is not quite homogeneous. In addition, this arrangement also leads to a very high luminous flux at the sample, without necessity of removing the heat produced by the lamp. The opal glass (thickness: 0.50 mm) is located about 5 mm apart from the output end of the fiber cable. The sampling aperture (6 mm × 3 mm) is defined by a diaphragm which is placed at the side of the opal glass facing the sample; as in the case of the inverse square law densitometer, the diaphragm is inlaid into the opal glass to achieve close contact of sample and opal glass. The combination of fiber cable and opal glass makes it possible to meet the ISO conditions regarding uniformity, diffusivity (diffusion coefficient: 0.952 according to ISO 5-2 Annex C, and 0.918 according to ISO/DIS 5-2) and reflectance (reflectance factor: 0.54 at 570 nm) of the diffuse illuminator.

The transmitted luminous flux is collected within a cone with a half angle of about 8° (maximum permissible half angle according to ISO 5-2: 10°). After collimation, the flux is filtered by means of an optical filter arrangement and detected with a silicon photodiode (Hamamatsu S1227-1010BQ). The spectral product of the densitometer is not as good as that of the PTB's inverse square law densitometer (see Fig. 7); the value of the f'_1 error parameter is 6.5%. Because the device is intended primarily for the measurement of spectrally neutral samples, this is actually no disadvantage.

Optional grey filters (one filter or two filters in tandem arrangement) serving as spectrally neutral attenuators can be positioned in front of the photodetector. The grey filters are glass plates coated with a durable metallic alloy. These plates ensure uniform attenuation of the radiation over a wide spectral range. The filters are used to attenuate the luminous flux obtained without a sample, which renders it possible to reduce the ratio between the signals recorded with and without a sample to values ranging between 1 and 30. This has the advantage that the same amplifier setting can be used for both signals. The attenuation provided by the grey filters must be known with high accuracy. These calibrations are performed in situ, with all densitometer

components present to include possible effects of inter-reflections.

The measurement procedure is straightforward. First, the measurement with the sample is performed using an appropriate amplifier setting. The second measurement is conducted with the sample removed and with a suitable grey filter inserted. The density value obtained corresponds to the difference between sample density and the known grey filter density. The signal-to-noise ratio is high enough to measure diffuse densities up to about $D = 6$ with sufficient accuracy. The light flux emitted by the tungsten lamp is recorded with an additional photodiode to detect possible changes of the light flux that might occur between the measurement with and without a sample.

Measurement Uncertainty

Intercomparison measurements between the inverse square law densitometer and the fiber densitometer have shown that the values indicated by both devices agree for spectrally neutral samples within the measurement uncertainty of the inverse square law densitometer. This is confirmed by the measurement uncertainty analysis. The repeatability of measurements ($k = 2$) is better than 0.002 for densities below 2, for densities between 2 and 4, $U(D) = 0.002$, and for densities above 4, $U(D) = 0.004$. The linearity of the type of photodetector used in this densitometer has been tested to be better than 0.1% within a range of two decades;[10] the contribution to uncertainty due to this effect is therefore below 0.001 in optical density. The in situ calibration of the attenuation provided by the neutral grey filters is another source of measurement uncertainty: $U(D)=0.002$ for densities below 4, and $U(D) = 0.004$ for $D > 4$. For spectrally neutral samples the total measurement uncertainty ($k = 2$) is $U(D) = 0.003$ for densities below 2, $U(D) = 0.004$ for $2 < D < 4$, and $U(D) = 0.006$ for $D > 4$.

In the case of non-neutral samples the densities measured with the fiber densitometer may differ significantly from that measured with the inverse square law densitometer; these differences might be due to the worse match of the spectral product of the fiber densitometer.

Conclusions

We have developed and set up two densitometers that fulfill the ISO geometric and spectral conditions for the measurement of visual diffuse optical densities. However, relatively great effort was necessary to meet the ISO geometric conditions; an opal glass alone serving as the diffuser was not sufficient to meet both the reflectance and the diffusivity conditions. Thin opal glasses may have the proper reflectance factor, but their diffusion coefficient is low, whereas thick opal glasses, where diffusion coefficients are large enough have reflectance factors that exceed the specified value of 0.55 by more than the permissible tolerance.

The intercomparisons between both PTB densitometers have shown that the single-beam instrument which is not a null-balance device, furnishes the same results as the sophisticated dual-beam inverse square law densitometer. It is possible to build a standard reference densitometer with the much simpler geometric and optical arrangement of a single-beam device. This device should be able to meet the customer's demands with regard to measuring range, measurement accuracy and also calibration cost.

Intercomparison measurements with NIST's newly established standard densitometer[11] have been performed. The results of these intercomparison experiments will soon be submitted for publication in this Journal.

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