

**Development and Metrological Characterization of a
Measuring System for Accurate Multidimensional
BTDF Measurements
Kumulative Arbeit**

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Cumulative Dissertation

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Abstract

This publication-based dissertation is dedicated to the accurate metrological characterization of optical diffuse transmission. Its macroscopic representation, translucency, as one of the four attributes for describing an object's optical appearance, is gaining increasing interest in fields of quality control, climate monitoring, computer graphic rendering, etc. Thus, the need for definition of the corresponding measurand, bidirectional transmittance distribution function (BTDF), and for standardization of its measurement procedures is increasing. In this work, I present the results achieved in my research period as PhD student in the Working Group 4.51 "Reflection and Transmission" at *Physikalisch-Technische Bundesanstalt* (PTB), the German national metrology institute. Major results were published as peer-reviewed scientific journal papers and are exhibited in this work, along with an extensive introduction to the theoretical background and the applied methodology, followed by additional information and discussion.

Two main aspects of accurate BTDF characterization are focused on in this work. The first one is the development of a new goniospectrophotometric facility for multidimensional BTDF measurement. This facility aims to complement the existing research and calibration capabilities of the working group in the field of optical reflection and transmission. In the future, it will provide the primary realization of quantities related to optical diffuse transmission such as BTDF in the spectral range from ultraviolet to near-infrared. Major features of the facility, such as sample- and detector-positioning, configurations of irradiation and detection systems, etc., are paid special attention to after a thorough study of other relevant goniospectrophotometers worldwide. The flexibility of switching measurement scheme between under- and over-irradiation using this new facility allows for further study on the flux-dependent transmittance, aiming at extension and generalization of the BTDF definition for strongly scattering materials.

Another focus of this work is the metrological characterization of diffusers with different scattering behaviors, exhibiting quasi-Lambertian, Gaussian, and square-shaped tophat BTDF distributions. Multiple interlaboratory comparisons were conducted between European metrology institutions, universities, and industrial partners. Measurement data were analyzed using statistical approaches, revealing current BTDF measurement capabilities and providing insights for a future Key Comparison.

Kurzfassung

Diese publikationsbasierte Dissertation ist der genauen metrologischen Charakterisierung spezieller Aspekte der optischen diffusen Transmission gewidmet. Ihre visuelle Wahrnehmung, Transluzenz, als eines der vier Attribute zur Beschreibung des optischen Erscheinungsbildes eines Objekts, gewinnt in Bereichen wie Qualitätskontrolle, Klimaüberwachung, Computergrafik-Rendering usw. zunehmend an Bedeutung. Daher steigt der Bedarf an der Definition der entsprechenden Messgröße, der bidirektionalen Transmissionsdistributionsfunktion (BTDF), und an der Standardisierung ihrer Messverfahren. In dieser Arbeit werden die Ergebnisse präsentiert, die ich während meiner Zeit als Doktorand in der Arbeitsgruppe 4.51 “Reflection und Transmission” an der Physikalisch-Technischen Bundesanstalt (PTB), dem deutschen nationalen Metrologieinstitut, erzielt habe. Die wichtigsten Ergebnisse wurden in referierten wissenschaftlichen Zeitschriften veröffentlicht und werden in dieser Arbeit dargestellt. Ferner gebe ich eine ausführliche Einführung in den theoretischen Hintergrund und die angewandte Methodik mit zusätzlichen Informationen und Diskussionen.

Die Arbeit umfasst zwei Schwerpunkte der genauen Charakterisierung von BTDF. Der erste ist die Entwicklung eines neuen goniospektrophotometrischen Messplatzes zur multidimensionalen BTDF-Messung. Dieser Messplatz soll die bestehenden Forschungs- und Kalibrierungsmöglichkeiten der Arbeitsgruppe auf dem Gebiet der optischen Reflexion und Transmission ergänzen und zukünftig die primäre Realisierung von Messgrößen im Zusammenhang mit optischer diffuser Transmission, wie z.B. BTDF, im Spektralbereich von Ultraviolett bis Nahinfrarot anbieten. Nach einer gründlichen Untersuchung anderer einschlägiger Goniospektrophotometer weltweit wird der Messplatz mit besonderem Schwerpunkt auf die wichtigsten Merkmale wie Proben- und Detektorpositionierung, Konfiguration der Bestrahlungs- und Detektionssysteme auseinandergesetzt. Die flexible Umschaltung des Messschemas zwischen Unter- und Überstrahlung ermöglicht weitere Forschungen an den strahlungsflussabhängigen Transmissionseigenschaften. Dadurch wird eine Erweiterung und Verallgemeinerung der Definition von BTDF für stark streuende Materialien möglich.

Ein weiterer Schwerpunkt dieser Arbeit ist die metrologische Charakterisierung von Diffusoren mit unterschiedlichen Streuverhalten, die z.B. quasi-Lambertsche, gaußförmige und rechteckige BTDF-Verteilungen aufweisen. Es wurden mehrere Ringvergleiche zwischen europäischen Metrologieinstituten, Universitäten und Industriepartnern durchgeführt. Die Messdaten wurden mit statistischen Verfahren analysiert, die die aktuellen BTDF-Messkapazitäten aufzeigen und Einblick in einen zukünftigen Schlüsselvergleich liefern.

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1 Summary

Light-matter interaction plays an important role in shaping the world we live in today. Here, “light” refers to a broader spectrum of electromagnetic radiation from 100 nm to 1 mm than just the visible light [1]. Distinguished by the transfer of radiant energy, light-matter interaction can be divided into three main categories: emission, absorption, and scattering. The study of light scattering dated back at least to the 17th century by Newton [2, 3]. One main way for us human beings to understand the world is through the information contained in the scattered light. Interestingly, the greatest impact on human society comes only from a very narrow part in the electromagnetic spectrum, the visible spectrum range (VIS) from approximately 380 nm to 780 nm, in which light can be directly perceived by the human visual system as the optical appearance of an object [4]. From the perceived appearance, knowledge on the object’s properties can be gained [5, 6], upon which we make judgments and decisions every day and everywhere [7–9].

In order to understand the mechanisms behind visual appearance, numerous studies have been conducted to correlate human visual perception to measurable physical quantities. Due to its complex and multidimensional nature, one approach is to use metrics of four attributes, namely color, gloss, translucency, and texture, to quantitatively describe the appearance [10, 11]. To date, some definitions of appearance itself and of its four attributes can be found in [1, 10, 12], and attempts have been made to correlate the physical phenomena to the perceptual dimensions [13, 14]. However, translucency is the only appearance attribute left behind, for which neither metrological definition nor correlation between human perception and physical properties of an object has been defined as of now [15–18].

Another approach to achieve quantitative characterization of the appearance is to define and realize measurements of physical quantities and functions that are more fundamental to describe light scattering. They include, but are not limited to, reflectance and transmittance, bidirectional reflectance distribution function (BRDF) and bidirectional transmittance distribution function (BTDF) (or together as bidirectional scattering distribution function (BSDF)), bidirectional scattering - surface

reflectance distribution function (BSSRDF), and so on¹. These quantities no longer focus on one or two attributes of the appearance. Instead, they include all radiometric information on the incident and scattered light with their geometrical, spectral, and polarization dependence.

Among them, accurate characterization of BTDF is gaining increasing interest not only in research and development but also in many industrial fields. For example, apart from being used in an optical system for beam conditioning, a transmission diffuser of known BTDF is needed for a long-term satellite-based ozone monitoring of the Earth, because measuring instruments have to be calibrated on-orbit to provide directly comparable results and prevent drift [19, 20]. The BTDF is also in demand for daylighting materials used for energy efficient buildings, since angular information of the transmitted light is given in the BTDF [21, 22]. Similar demand on the BTDF has recently triggered studies on materials for horticultural ceilings to generate diffusely transmitted light, which is found to penetrate deeper into compact crop canopies than directional light and thus enhances the photosynthesis rate [23, 24]. Moreover, the transmitting properties of materials used in clinical practice, especially in dentistry, also need to be characterized since their aesthetic features are paid equal attention to as their medical properties [25]. In the food industry, light diffusely transmitted by fruit slices is used in quality assessment because it reveals information about firmness, sweetness, and maturity of e.g. peaches or kiwifruits [26, 27]. In the field of computer graphics rendering, BTDF plays a vital role as well in vividly representing objects from reality using various models [28–31].

Despite the many application fields of BTDF, its metrological realization and traceability are not well-established compared to those of BRDF for diffuse reflection. Even some ambiguity can be observed in the definition of BTDF when it comes to very translucent and thick materials, as the incident and exiting surfaces of a sample must be separately taken into consideration, which is not the case for BRDF. Therefore, this work presents approaches to solve the problems by focusing on the accurate BTDF characterization. These approaches include the development of a multidimensional BTDF measuring facility with uncertainty on the level of a national metrology institute (NMI) and a pilot study on the metrological BTDF characterization of materials with distinctive scattering properties, especially of translucent materials with non-negligible lateral scattering of the incident radiation.

¹Please note that although BRDF, BTDF, and BSSRDF are called functions since they are dependent on several parameters and can be treated as functions, they do not fulfill the mathematic definition of a function. Therefore, in the following text they are also referred to as quantities.

In this cumulative thesis, we start with the fundamental theories needed for optical scattering in Section 1.1, where the mathematical definition of radiometric quantities and the above mentioned scattering quantities is presented, along with a brief introduction to the most widely used simulation models to describe the characteristics of different types of optical transmissive and translucent diffusers. Equipped with the necessary background information, we are able to understand the requirements which should be fulfilled in order to develop a measuring facility for accurate, traceable, and multidimensional BTDF measurement. Thus, an overview with detailed instrument parameters of major goniospectrophotometers around the world is presented at the beginning of Section 1.2 as reference for instrument comparison as well as inspiration for further development. Main results of the study of different types of BTDF samples, the development of the new primary BTDF facility at *Physikalisch-Technische Bundesanstalt* (PTB), and the validation of its measurement capabilities are published in Paper I, Paper II, and the first part of Paper IV. In the rest of this section, additional discussions on the search for reference BTDF samples and on the major components of the new BTDF facility are given. Other than that, the limitations of BTDF measurement capabilities in the European Union have been investigated and pushed forward by two multilateral interlaboratory scale comparisons, one of which is contributing largely to the metrological BTDF characterization as one of the main goals of this work. The results are reported in Paper III and more details regarding the comparison analysis are illustrated in Section 1.2.4. In Section 1.3, the general models for BTDF realization are introduced, with special attention on the difference between the under- and over-irradiation scheme. Then the measurement models specifically applied by the newly developed BTDF facility are illustrated, along with a detailed uncertainty analysis. Finally, the difficulties in the accurate measurement of thick translucent diffusers with non-negligible lateral scattering are illustrated in Section 1.3.3 and first results of the study are reported in the second part of Paper IV.

1.1 Optical Scattering

From the physical point of view, the appearance of an object is merely the light captured by the human visual system after interacting with the object depending on its physical properties [32, 33]. From the viewpoint of radiative transfer, we can use the terms “reflection” and “transmission” to describe the interaction between the electromagnetic radiation (light) and the object, depending on the change of propa-

gation direction of the radiation. Both terms can be categorized as the “scattering” process, in which the trajectory of light, consisting of multiple rays of infinitesimal size, gets deflected by the small material particles [34]. Thus, the scattering process takes place both at the air-material interface and within the bulk volume of the material. The magnitude of scattering depends on the size and distribution of the scattering centers, as well as on the wavelength and polarization of the incident light. Different physical quantities as well as simulation models accounting for the radiometric and geometric perspectives are needed to describe the scattering properties of a material. They are introduced in this section.

1.1.1 Radiometric Quantities

Radiometric quantities are needed to quantify the amount of radiation incident on and scattered by a material per unit time, area, solid angle, and spectral bandwidth [35]. They concern the different aspects of radiative transfer. Bear in mind that all the radiometric quantities are spectrally dependent, which is not explicitly expressed in the following introduction, if not specifically mentioned. A schematic of generalized geometrical description of radiometric quantities is illustrated in Figure 1.1. In

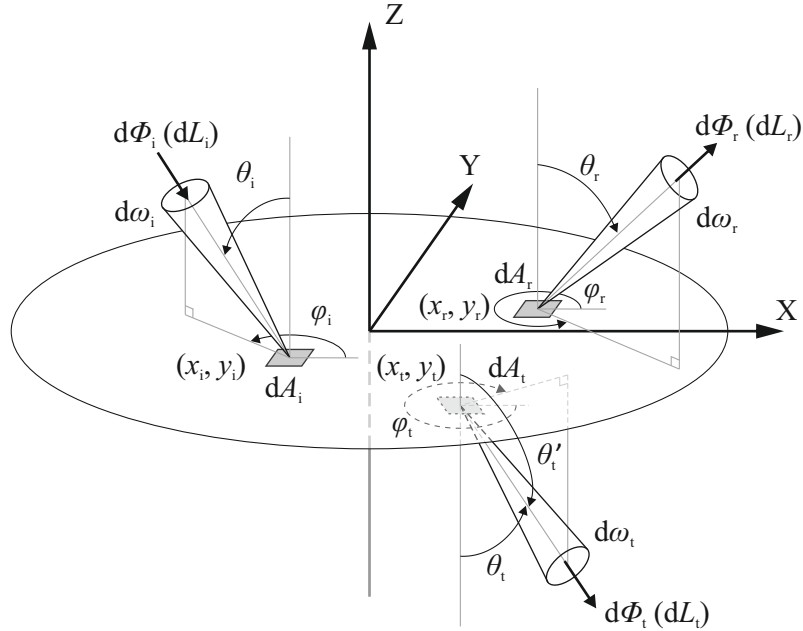


Figure 1.1: Generalized geometrical description of reflection and transmission. The subscripts “i”, “r”, and “t” indicate the radiation direction as “incidence”, “reflection”, and “transmission”, respectively.

this figure, one material surface is irradiated from the top. The center of the surface is taken as the origin of the coordinate system, with its x - and y -axis lying within

the plane of the surface and z -axis being the surface normal with direction perpendicular to the surface pointing towards the hemisphere from which the incident radiation impinges the material. Within this irradiated area, three element areas denoted dA_i , dA_r , and dA_t are chosen to represent the case of incidence, reflection, and transmission, so that the quantities for the different cases can be better distinguished. The parameters related to each case are differentiated by the subscripts “i”, “r”, and “t”, for incidence, reflection, and transmission, respectively. All three unit areas share the same coordinate system as the one for the material surface. Their spatial position on the surface is described as a pair of (x, y) coordinates. Note that dA_i and dA_r are on the upper side of the surface, while dA_t is considered to be on the lower side of the surface. The direction of radiation is given by θ , the polar angle measured from the surface normal, and φ , the azimuthal angle measured from the x -axis to the projection of the radiation direction onto the xy -plane. Thus, φ also lies within the xy -plane. Note that the transmission polar angle θ_t is referred to the angle between the negative direction of the surface normal, pointing towards the hemisphere beneath the material surface, and in the transmission direction. Although its definition does not mathematically conform with that for incidence and reflection (in that case the angle from the surface normal to the transmission direction as θ'_t in Figure 1.1), its use preserves the consistency in describing and realizing geometries for both transmission and reflection measurements.

Radiant Flux

The starting point of all radiometric quantities is the radiant flux Φ , which is the amount of energy propagating through a material surface within a unit time Δt . Thus, it is also referred to as the radiant power and has the unit [W]. This amount of radiant energy can be expressed as

$$Q = \int_{\Delta t} \Phi dt, \quad (1.1)$$

where Q is the radiant energy. Since the measurement schemes applied in the frame of this work consider only the continuous-wave (CW) radiation, the radiant flux is used in the following text rather than the radiant energy, which is normally used when referring to pulsed light.

Irradiance

The irradiance E is defined as the amount of radiant flux contained within a unit area on a surface:

$$E = \frac{d\Phi}{dA}, \quad (1.2)$$

with dA the size of the area element. It has the unit $[\text{W m}^{-2}]$. The differential notations in all radiometric quantities indicate the infinitesimal of a quantity, not the derivative in its formal sense.

Note that the irradiance is referred to “optical intensity” in many other contexts such as laser technology. In radiometry, “irradiance” and “intensity” are strictly defined and clearly distinguished to avoid confusion.

Radiance

The radiance L describes the amount of radiant flux propagating through a unit area on a surface and within a unit solid angle. The propagation direction is considered perpendicular to this unit area.

$$L = \frac{d\Phi}{dA d\Omega}, \quad (1.3)$$

with Ω the projected solid angle, i.e., the product of the solid angle ω and the polar angle θ between the propagation direction and the surface normal, onto the surface element:

$$d\Omega = \cos \theta d\omega. \quad (1.4)$$

For an area with size A and at distance l to a given point on the surface from which the radiation is emitted ($l \gg \sqrt{A}$), the solid angle subtended can be expressed as

$$\omega = \frac{A}{l^2}, \quad (1.5)$$

with the unit $[\text{sr}^{-1}]$. ω and Ω are often used interchangeably depending on the convention of use. In this thesis, we follow the recommendations made by F. E. Nicodemus in [36, 37], where the nomenclature for scattering quantities including BRDF and BSSRDF was thoroughly described for the first time. Please note that in Papers I, II, and III the solid angle is expressed as “ Ω ” and in Paper IV the notations follow the convention introduced in this thesis.

Ideally, without any reflection or absorption losses, the radiance through an optical system is a conserved quantity as a result of the étendue conservation (throughput) of the radiation.

Radiant Intensity

The radiant intensity is defined as the amount of radiant flux per unit solid angle in a given direction:

$$I = \frac{d\Phi}{d\omega}, \quad (1.6)$$

with the unit $[\text{W sr}^{-1}]$. With the help of equation 1.5, the relation between radiant intensity and irradiance can be expressed as

$$I = l^2 E, \quad (1.7)$$

with l the distance to the source.

1.1.2 Scattering Quantities

Depending on the angular distribution of radiation after interacting with the material, one distinguishes specular (regular [1]) reflection (or transmission) from diffuse reflection (or transmission). In principle, both kinds of reflection or transmission belong to the macroscopic effect of optical scattering. However, the extreme cases of fully specular or fully diffuse reflection (or transmission) only exist as mathematical description. In practical cases, the reflected (or transmitted) radiation usually consists of both specular and diffuse components and sometimes even exhibits complicated distribution [37–39]. One way to quantify the scattering is to measure the reflectance (or transmittance), defined as the ratio of reflected (or transmitted) to incident radiant flux by *Commission Internationale de l'Éclairage* (CIE) [1]. However, the obtained results only represent the measurement under given conditions such as area size of irradiation and collection, solid angle of irradiation and collection, and so on. This makes it difficult to find a generalized expression of the obtained quantities [37].

To solve this problem, Nicodemus *et al.* defined BRDF and BSSRDF as basic quantities to characterize the scattering (mainly reflecting) properties of the material in a unified approach, regardless of most of the measurement conditions and the artificial distinction between specular and diffuse reflection [37, 40]. These quantities describe the radiometric relation between the incident and scattered radiation

with arbitrary geometrical consideration and they are also referred to as spectrophotometric quantities. Later on, Bartell *et al.* extended the situation to transmission by introducing the BTDF to be another basic scattering quantity as the counterpart of BRDF [41]. Recently Ferrero *et al.* suggested to specify scattering events by the more fundamental quantities bidirectional bipositional subsurface scattering function (BBSS) and bidirectional insurface scattering function (BIS), with consideration only on the position of the light-matter interaction, i.e., subsurface and insurface scattering [42]. All the mentioned scattering quantities will be introduced in the following.

BSSRDF

We consider that the material surface in Figure 1.1 is entirely irradiated with a well-collimated beam of incident radiation. A portion of radiant flux $d\Phi_i$ confined within the infinitesimal solid angle $d\omega_i$ is incident onto the infinitesimal surface area dA_i , centered at (x_i, y_i) , from the direction (θ_i, φ_i) . The general assumption of all scattering quantities is that the scattered radiation is only the linear response to the incident radiation, i.e., only the elastic scattering process is concerned. Therefore, an infinitesimal amount of reflected radiance dL_{ssr} from the infinitesimal surface area dA_r , centered at (x_r, y_r) , into the direction (θ_r, φ_r) can be regarded as the linear response to $d\Phi_i$:

$$dL_{\text{ssr}}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r) = f(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r) \cdot d\Phi_i(\theta_i, \varphi_i, x_i, y_i), \quad (1.8)$$

where f is a function of the polar and azimuthal angles, and the spatial positions of both incident and reflected radiation. This function is called the BSSRDF, which purely describes the scattering property of the material surface. It has the unit $[\text{m}^{-2} \text{sr}^{-1}]$.

BSSRDF is one of the most general scattering quantities, because no approximation or simplification is made in its definition. Although in [37] the BSSRDF refers only to flat surfaces, it can also be applied to any kind of surface in a more generalized way [42]. From equation 1.8, it can be expressed as the ratio of the reflected radiance to the incident radiant flux, with the notation f_{ssr} :

$$f_{\text{ssr}}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r) \equiv \frac{dL_{\text{ssr}}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r)}{d\Phi_i(\theta_i, \varphi_i, x_i, y_i)}. \quad (1.9)$$

After BSSRDF was proposed and accepted, there has mostly been measurements of it performed to fulfill the needs in the computer graphics industry [43, 44]. It

was not until recently that the metrological characterization of BSSRDF was addressed. Primary facilities with different measurement methods have been developed in metrology institutes and a bilateral comparison has also been accomplished [45–47].

BRDF

Since BSSRDF is an 8-dimensional quantity which requires a huge amount of experimental effort to realize, further assumptions can be made to obtain another scattering quantity with better accessibility. We still take the situation illustrated in Figure 1.1, now with additional assumptions that the incident radiation onto the material surface is uniform and the scattering properties of the material is uniform and isotropic within the area of evaluation. This means that the incident radiance L_i is constant within the irradiated area A_i . Thus, the incident radiant flux $d\Phi_i$ can be expressed as

$$d\Phi_i = L_i \cdot \cos \theta_i \cdot d\omega_i \cdot dA_i = dE_i \cdot dA_i, \quad (1.10)$$

with dE_i being the incident irradiance within dA_i . For uniform irradiation, the contribution to the reflected radiation at (x_r, y_r) from the incident radiation can be integrated for all area elements on the surface. Additionally, the reflected radiation only depends on the distance between (x_r, y_r) and (x_i, y_i) , since the material scatters the incident radiation isotropically and uniformly. Then the reflected radiance of one infinitesimal area on the material surface can be rewritten to

$$dL_{\text{ssr}}(\theta_i, \varphi_i; \theta_r, \varphi_r; \xi) = \int_{A_i} f \cdot d\Phi_i = dE_i \cdot \int_{A_i} f \cdot dA_i, \quad (1.11)$$

with ξ being the distance between the evaluated area and the incident area. The scattering function f is now reduced to 5 dimensions, i.e., $f(\theta_i, \varphi_i; \theta_r, \varphi_r; \xi)$. The scattering properties of an uniform and isotropic material surface can be characterized by the BRDF¹ f_r , defined as

$$f_r(\theta_i, \varphi_i; \theta_r, \varphi_r) = \int_{A_i} f(\theta_i, \varphi_i; \theta_r, \varphi_r; \xi) \cdot dA_i \equiv \frac{dL_r(\theta_i, \varphi_i; \theta_r, \varphi_r)}{dE_i(\theta_i, \varphi_i)}, \quad (1.12)$$

¹This term was initially referred to as “partial reflectance” by Nicodemus *et al.* [40] when he proposed it for the first time. Later he adapted it to the current term following the use by Edwards in [48], as it is “equally concise” but “physically more descriptive”. The modifier “bidirectional” clearly denotes the two directions of incident and scattered radiation, and “distribution function” emphasizes the nature of this quantity, allowing for the utilization of existing mathematical treatments in general.

under the assumption that the evaluated area is uniformly irradiated over a large enough area on the surface. The geometrical description of BRDF can be simplified to Figure 1.2a, since it is now only a function of the polar and azimuthal angles of incident and reflected radiation. BRDF quantitatively describes the angular distribution of the reflection for a given incident direction. It is another basic scattering quantity with the unit $[\text{sr}^{-1}]$ and can take any positive values.

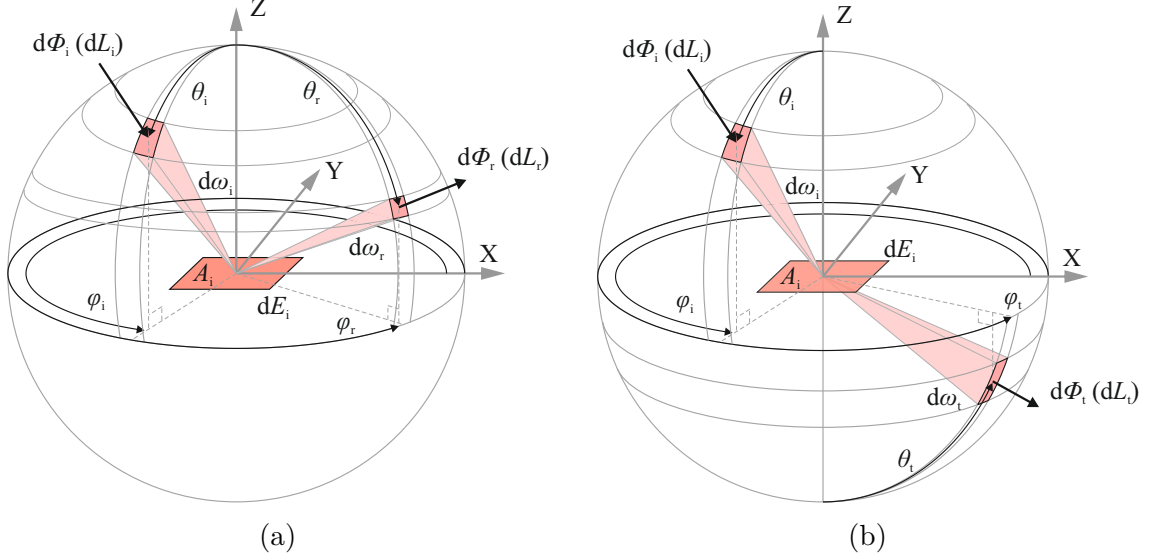


Figure 1.2: Definition of (a) BRDF and (b) BTDF.

Among all scattering quantities BRDF is the most experimentally accessible measurand and various facilities have been developed to pursue higher accuracy for the measurement of BRDF. Later in Section 1.2.1 some representative ones will be introduced. To date, the most accurate measuring facilities can obtain the BRDF value in the standard geometry, defined in [49], of a white diffuse reflectance standard, normally made of barium sulfate [50, 51] or polytetrafluorethylene (PTFE) [52, 53], with the smallest combined uncertainty of about 0.1 % [54]. More details about the different types of samples used in the measurement of optical scattering are given in Section 1.2.2.

BTDF

The BTDF is the counterpart of BRDF for characterizing the angular distribution of diffuse transmission of a material. Its basic concept was given by Bartell *et al.* [41] similar to BRDF as f_t :

$$f_t(\theta_i, \varphi_i; \theta_t, \varphi_t) \equiv \frac{dL_t(\theta_i, \varphi_i; \theta_t, \varphi_t)}{dE_i(\theta_i, \varphi_i)}, \quad (1.13)$$

with L_t being the radiance of the transmitted radiation in the other hemisphere different from the one for incident radiation, as illustrated in Figure 1.2b. BTDF and BRDF are sometimes together referred to as the BSDF.

The main difference between BRDF and BTDF lies in the choice of the reference plane, being the single material surface in their definition. For materials exhibiting primarily insurface scattering, i.e., light is scattered by the micro structures on the surface, the reference plane can be considered lying just above the highest points of those structures [37] and would be suitable for both reflection and transmission. Problems arise when the scattering consists of a significant amount of subsurface interaction. In this case, multiple scattering takes place within the material volume below the nominal reflecting (or transmitting) surface. For reflecting materials the reference plane might still be treated as the same one as in the case of insurface scattering, with appropriate geometrical corrections [55]. For transmitting materials, however, the volume scattering makes it difficult to determine the reference plane. One could still consider the incident (front) surface to be the reference plane and perform some corrections regarding the measurement geometry [56]. But one can also argue that the reference plane should be the transmitting (back) surface as it is the surface on which the transmitted radiance is evaluated. This is also the convention applied in all measurements in the frame of this work. Another option would be to choose a reference plane to be somewhere within the material, so that no correction is needed. But this would greatly depend on the sample characteristic and require accurate modeling and sample parameters to determine the reference plane. Phenomena related to volume scattering are discussed in more detail in Section 1.3.3 and in the second part of Paper IV.

BBSS and BIS

In the definition of BRDF, Nicodemus *et al.* avoided the issue caused by subsurface scattering by assuming a “large enough” irradiation area [37]. Although the subsurface scattering was discussed in this nomenclature, it did not finally answer the question: What if the irradiation area cannot be realized or regarded as “large enough”? Thus, to solve this issue, Ferrero *et al.* proposed recently a more generalized way to characterize optical scattering by defining two other fundamental quantities in [42], the bidirectional bipositional subsurface scattering function (BBSS) and the bidirectional insurface scattering function (BIS). These two quantities only differ from whether there is significant light transportation below the incident surface.

In the definition of BSSRDF, subsurface scattering has actually already been considered by the parameters of spatial dimension. However, the reflected radiance L_{ssr} can be generated either by subsurface scattering when the collected area A_r is at $(x_r, y_r) \neq (x_i, y_i)$ or by the summation of both insurface and subsurface scattering at $(x_r, y_r) = (x_i, y_i)$. This leads to a discontinuity in the definition of BSSRDF and a dependence on the irradiated area (see equation 1.17).

As suggested in the paper by Ferrero *et al.*, at $(x_r, y_r) = (x_i, y_i)$ the radiance of an infinitesimal area L_{ssr} can be expressed as

$$dL_{\text{ssr}}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r) = dL_{\text{ss}}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r) + dL_{\text{is}}(\theta_i, \varphi_i; \theta_r, \varphi_r), \quad (1.14)$$

with L_{is} and L_{ss} being the radiance generated only by insurface and subsurface scattering, respectively. L_{is} is not dependent on the spatial position of radiation because it represents the scattering at the air-matter interface and has zero value when $(x_r, y_r) \neq (x_i, y_i)$. The scattering function f_{ss} that only describes subsurface scattering, also called the BBSS, can be defined similar to BSSRDF by

$$f_{\text{ss}}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r) = \frac{dL_{\text{ss}}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r)}{d\Phi_i(\theta_i, \varphi_i, x_i, y_i)}, \quad (1.15)$$

with the unit $[\text{m}^{-2} \text{sr}^{-1}]$. And the scattering function f_{is} that only concerns insurface scattering, also called the BIS, can be defined similar to BRDF by

$$f_{\text{is}}(\theta_i, \varphi_i; \theta_r, \varphi_r) = \frac{dL_{\text{is}}(\theta_i, \varphi_i; \theta_r, \varphi_r)}{dE_i(\theta_i, \varphi_i)}, \quad (1.16)$$

with the unit $[\text{sr}^{-1}]$. Combining equations 1.9, 1.14, 1.15, and 1.16, the BSSRDF can be represented as

$$\begin{aligned} f_{\text{ssr}}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r) &= \frac{dL_{\text{ssr}}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r)}{d\Phi_i(\theta_i, \varphi_i, x_i, y_i)} \\ &= \begin{cases} f_{\text{ss}}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r) + \frac{f_{\text{is}}(\theta_i, \varphi_i; \theta_r, \varphi_r)}{dA_i}, & \text{for } (x_r, y_r) = (x_i, y_i) \\ f_{\text{ss}}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r), & \text{for } (x_r, y_r) \neq (x_i, y_i) \end{cases} \end{aligned} \quad (1.17)$$

with dA_i being the infinitesimal irradiated area. This indicates a dependence of the scattered radiance on the size of the irradiated area. For a “large enough” A_i , the second term in the first case becomes negligible compared to the other term, and the equation 1.17 in this case has the same form as in the second one. By assuming again a uniform irradiation on the material surface, the BRDF can be expressed via

integration over A_i as

$$\begin{aligned}
 f_r(\theta_i, \varphi_i; \theta_r, \varphi_r, x_r, y_r; A_i) &= \frac{dL_r(\theta_i, \varphi_i; \theta_r, \varphi_r, x_r, y_r; A_i)}{dE_i(\theta_i, \varphi_i)} \\
 &= \begin{cases} \int_{A_i} f_{ss}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r) dA_i + f_{is}(\theta_i, \varphi_i; \theta_r, \varphi_r), & \text{for } (x_r, y_r) \in A_i \\ \int_{A_i} f_{ss}(\theta_i, \varphi_i, x_i, y_i; \theta_r, \varphi_r, x_r, y_r) dA_i. & \text{for } (x_r, y_r) \notin A_i \end{cases} \quad (1.18)
 \end{aligned}$$

For transmission, the transmitted radiance L_t is generated only by subsurface scattering entering into the other hemisphere of the surface. Therefore, the BTDF can be expressed as

$$\begin{aligned}
 f_t(\theta_i, \varphi_i; \theta_t, \varphi_t, x_t, y_t; A_i) &= \frac{dL_t(\theta_i, \varphi_i; \theta_t, \varphi_t, x_t, y_t; A_i)}{dE_i(\theta_i, \varphi_i)} \\
 &= \int_{A_i} f_{ss}(\theta_i, \varphi_i, x_i, y_i; \theta_t, \varphi_t, x_t, y_t) dA_i, \quad (1.19)
 \end{aligned}$$

with now f_{ss} being a function of geometrical parameters related to incidence and transmission. Note that the definition of BRDF and BTDF is now a function of A_i and their values are dependent on the spatial position of evaluation. This dependence was observed in the measurement and reported in the second part of Paper IV. In the case where A_i cannot be treated as “large enough”, BBSS and BIS can be measured instead to derive other scattering quantities. Another advantage of introducing BBSS and BIS is that the distinction between reflection and transmission is not necessary anymore, but rather the spatial dependence of scattering is emphasized without the restriction given by that dependence [42]. It provides us with another way of quantitatively characterizing the scattering properties of material. Measurement of BBSS and BIS could also help solving the issue regarding edge losses of thick volume-scattering materials discussed in [37] and in the second part of Paper IV.

1.1.3 Simulation Models for Diffuse Scattering

As discussed in the previous sections, several fundamental quantities are defined to address different aspects of diffuse scattering at the macroscopic level. In order to study the relation between the scattering behavior and materials’ parameters as well as to achieve realistic graphic rendering, various simulation models have also been proposed. These models deal with the microscopic interaction between light and material, providing a passage for the derivation of scattering functions. Recently, a

thorough survey of important scattering models was published by Frisvad *et al.* in [57]. In this section, some relevant models are introduced briefly.

The methods used in modeling are distinguished by the physical aspects concerned and are thus applicable to different types of scattering, differentiated by the position where the interaction takes place, i.e., at the surface whose roughness is represented as an ensemble of microfacets or within the volume with an ensemble of small particles.

Geometric Models

For surface scattering, each microfacet can be regarded as a perfectly smooth interface with a certain BSDF [58–60] and for most of the cases the size of microfacets is much larger than the wavelength of light. The interaction of light with microfacets must fulfill the continuity requirement in the Maxwell’s equations, and thus, light is reflected or refracted at those microfacets following the Fresnel’s equations [61]. Therefore, the scattering properties of a material exhibiting dominant surface scattering can be simulated by considering the distribution of microfacet normals and applying models based on geometrical optics using ray tracing methods [62, 63]. For example, the model used in Paper I to explain the angle-dependent spectral transmittance of the studied samples follows the method described above. The surface roughness (standard deviation in height) is represented by microfacets obeying a normal distribution on the active surface of the sample. The substrate is considered to be transparent so that no scattering events take place inside. The situation of light rays impinging on different microfacets is schematically illustrated in Figure 1.3, with $\vec{m}_t$ pointing to the normal of each microfacet, D representing the distribution of the microfacet ensemble, θ_s being the scattering angle as a result of Fresnel transmission through the active surface, and θ_i and θ_t being the macroscopic incident and transmission angle, respectively. The simulation results reported in Paper I only consider single scattering event, i.e., light is scattered only once by each microfacet, because the calculation involving multiple scattering events would drastically increase the computation power. Furthermore, it also requires a better knowledge of the surface characteristics. Nevertheless, it is believed that for such roughened active surface, multiple in-surface scattering should be dominant and would thus enhance the effect observed in both simulation and measurement results. The influence of applying this angle-dependent spectral transmittance for a possible wavelength correction will be further elaborated in the Subsection “Wavelength Correction” in Section 1.3.2.

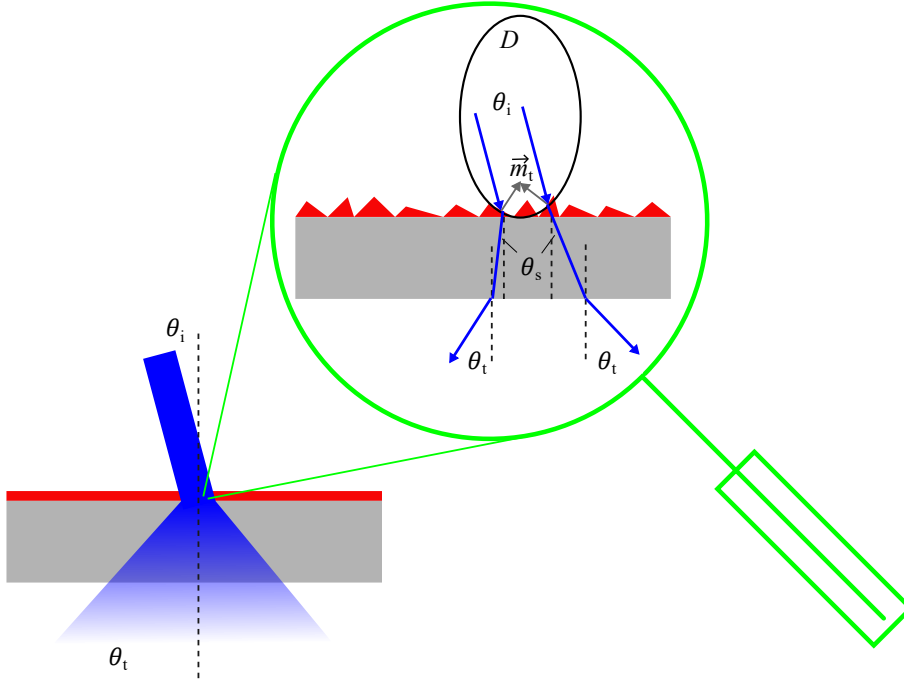


Figure 1.3: Sketch of the microfacet model used to simulate the angle-dependent spectral transmittance reported in Paper I. The macroscopic scattering effect is generated by many light rays being reflected or refracted by the microfacet structure on the material's active surface at different positions, resulting in transmission in different directions.

Radiometric Models

For volume scattering, the small material particle generates a spherical wave after interacting with a planar wave, and a directional distribution of the scattered light, also known as phase function [64–67], is given by the ensemble of particles. Radiometric models, focusing on how radiant energy is transferred and redistributed during and after the scattering process, are often used to compute the scattering characteristics of a translucent material with significant volume scattering. The most basic one, which was also the first ever scattering model, is the Kubelka-Munk model [68]. It considers the radiative transfer at one single layer of a volume scattering medium by solving a pair of coupled linear differential equations consisting of one forward going and one backward going irradiance, related by the phenomenological absorption and scattering coefficients of that medium layer. Although the original Kubelka-Munk model does not take angular dependence into consideration and is highly simplified, it can be extended and combined with other models to provide accurate simulation results [69, 70]. Another model for volume scattering materials uses the adding-doubling method, which calculates the reflection and transmission

distribution of a homogeneous layer with infinitesimal thickness and iteratively doubles the layer thickness until the scattering behavior coincides with that of the real material [71, 72]. One major advantage of adding-doubling is its high efficiency in predicting the scattering distribution of an object with known material parameters. This advantage makes it also possible to estimate the spectral scattering properties, especially the phase function, of a bulk scattering material by feeding measurement data into an inverse adding-doubling model [73, 74].

Field Models

To achieve a more realistic and physically-based simulation, the field models, describing the interaction of electromagnetic fields with material, are more accurate. The field models attempt to provide rigorous solutions to Maxwell's equations or Helmholtz equations, so they normally require high computation power and time consumption, as well as accurate parameter input. Nevertheless, the wave characteristics of light are modeled and effects such as interference and diffraction can be simulated. A rather inexpensive model among this type is based on the Lorenz-Mie theory, in which the Maxwell's equations are solved as a series expansion for the scattering of a planar wave by a sphere [75, 76]. It gives a straightforward way to simulate the scattering properties of a translucent material with randomly distributed scatterers of low concentration. But no information regarding surface scattering is contained in this model. To numerically compute the scattering for more general cases, models based on the scalar diffraction theory, calculating the Kirchhoff integral [77], are a good choice [78–80]. However, this type of models are used more often to inversely solve the scattering structure for a given scattering distribution [81, 82].

1.2 Development of PTB's primary BTDF Facility

As mentioned in Section 1.1.2, a realistic representation of an object's optical appearance requires accurate information on the complex scattering quantities. This information can only be obtained experimentally by performing radiometric measurements under laboratory conditions. Numerous goniospectrophotometric facilities have been developed around the world to measure the angular and spectral distribution of light scattered by the material, each has its own main focus of ap-

plication. However, the performance of these facilities differs from each other, normally distinguished by their measurement capabilities and measurement uncertainties. Among them, the facilities developed by a metrology institute such as an NMI or a designated institute (DI) usually can perform the most accurate measurements, i.e., they have the lowest measurement uncertainty.

At PTB, the working group 4.51 “Reflection and Transmission” developed multiple facilities for the measurement of optical scattering. The German national standard facility for optical diffuse reflection measurement is a goniospectrophotometer called *Gonioreflektometer* [54, 83]. It has been validated by multiple interlaboratory scale comparisons with other metrology institutes that the *Gonioreflektometer* is able to measure BRDF with a minimum combined expanded uncertainty of 0.2% [84–88]. Another two facilities, referred to as *das nationale Normal für gerichtete Reflexion* (NaNoRef) and *das nationale Normal für gerichtete Transmission* (NaNoTrans), are the German national standard facilities for regular reflection and transmission measurement [89, 90]. Both the *Gonioreflektometer* and NaNoRef have a goniometer as main component, so in principle they could be modified to perform angle-resolved transmittance measurement. However, the growing need for calibration service of diffuse transmission and limitations in realizing difficult measurement geometries using the existing facilities lead to the development of a new reference facility for multidimensional BTDF measurement with high accuracy.

In this section, more details regarding the development of PTB’s new primary BTDF facility, apart from those published in Paper II and IV, are introduced. First an overview of some relevant goniospectrophotometric facilities of other institutions around the globe is given with their major parameters summarized. Then some more discussions of the BTDF samples are given, followed by details of the major components of the facility. In the end of this section, the BTDF scale comparisons performed in the frame of this work are briefly introduced.

1.2.1 Overview of major goniospectrophotometers

A goniospectrophotometer normally consists of one or multiple rotation stages to realize angular positioning of the irradiation source, the sample under test, and the detection system. For the development of PTB’s new primary BTDF facility, it is worthwhile taking a look at the detailed parameters of some major goniospectrophotometers built by NMIs, DIs, and research institutions. The information is summarized as an overview in Table 1.1. Since the development state of a facility is always changing with time, specific parameters could vary due to corrections or

1. Summary

Table 1.1: Overview of parameters of relevant goniospectrophotometers

Institute Facility	Irradiation				Sample positioning	Detection				Measure-ment scheme	min. U (k = 2)					
	Type	Wavelength (nm)	Bandwidth (nm)	Spectral selection		Polarization	Size (mm)	Diver-gence (deg)	Solid angle (sr)			Detector type	Distance (mm)	Aperture angle (deg)	Solid angle (sr)	Measure-ment area (mm)
PTB Gonio-reflektometer	SR with LED	360–430	3	Double mono-chromator	unpolarized	40 (at exit)	3	2.2e–3	5-axis robot arm	Si-, InGaAs-photodiode, photo-multiplier	782.66 (sample - light source)	0.64	9.6e–5	20	N/A	0.32 %
	SR with 250 W QTH lamp	250–1700	3 (250 - 900) / 6 (900 - 1700)													0.2 %
PTB ARGon.3	SR with 250 W QTH lamp	250–1700	N/A	Mono-chromator	unpolarized	40 (at exit)	3	2.2e–3	5-axis robot arm	CCD line-scan camera	784.2 (sample - light source)	0.56	7.5e–5	20	N/A	N/A
	filtered tungsten lamp	350–850 (D65-like illuminant)	N/A													N/A
PTB modified NaNoRef	CW lasers	642 / 532 / 445 / 405	<1	N/A	linearly polarized	3.5–10.1	<0.1	<2.4e–5	3-axis linear stage mounted on rotary stage	Si-photodiode with integrating sphere	494.5	0.5–1.5	6e–5–5.4e–4	8–18.4	N/A	0.50 %
NIST STARR	150 W Xe lamp	200–400	14	Mono-chromator	polarized	17	<1	<2.4e–4	2-axis linear stage mounted on rotary stage	Si-photodiode, InAs-, Ge-photodiode with integrating sphere	669.9	2.7 / 3.8	1.8e–3 / 3.4e–3	35	N/A	0.39 %
	100 W halogen lamp	400–2500														under
NIST ROSI (STARR II)	LDLS	250–500	14	Mono-chromator	polarized	10	0.74	1.3e–4	6-axis robot arm	Si-photodiode, InGaAs-photodiode with integrating sphere	658	3.3	2.6e–3	60	N/A	0.65 %
	SC laser	500–2400														under
NIST five-axis gonio-spectrometer	Xe lamp & QTH lamp	380–780	5	Mono-chromator	unpolarized	10×12	N/A	N/A	5-axis robot arm	Si-photodiode	1250	2.7	1.8e–3	N/A	N/A	0.51 %
		300–1050	under													
NASA GSFC Scatterometer	75 W Xe lamp	250–2500	12	Mono-chromator	polarized	13	N/A	N/A	3-axis linear stage mounted on rotary stage	Si-, InGaAs-photodiode	500	1.6	6.4e–4	N/A	N/A	1.5 %
Aalto absolute reference gonio-reflectorometer	50 W QTH lamp	300–1700	5 (VIS) / 10 (NIR)	Double mono-chromator	polarized	17	0.6	8.4e–5	2 tilting plates on 1-axis linear stage with rotation stage	Si-, InGaAs-photodiode	498.15 / 472.9	2.9 / 3	2e–3 / 2.2e–3	38×38 / 19	N/A	0.4 %
	450 W Xe lamp															0.58 %
Aalto 3D-gonio-reflectorometer	SC laser	400–2400	4	Laser line tunable filter	polarized	3–20	N/A	N/A	3 rotating arms	Si-, InGaAs-photodiode	204.7	3	2.2e–3	N/A	N/A	1.1 %

Table 1.1: (...continued) Overview of parameters of relevant goniospectrophotometers

Institute Facility	Irradiation				Detection				Measure- ment scheme ($k=2$)
	Type	Wavelength (nm)	Bandwidth (nm)	Spectral selection	Polarization	Size (mm)	Diver- gence (deg)	Solid angle (sr)	
CSIC GEFE	Xe lamp	185–2000	4	Given by detector	polarized	30	1.5	5.4e–4	1 %
	incandescence lamp & LDLS	N/A	N/A			0.14	1.2	3.5e–4	1.7 %
CNAM ConDOR	200 W Xe lamp with V(λ)-filter	250–900	3	N/A	polarized	1–15	0.03	2.1e–7	5 %
CNAM primary goniospectro- photometer	QTH lamp with V(λ)-filter	VIS	1–5	Monochromator	unpolarized	3–15	0.2– 0.35	9.6e–6 –2.9e–5	1 %
CNAM μ BRDF- gonio	LDLS	170–2500	0.9	Given by detector	polarized	50 μ m	1.5	5.4e–4	6.8 %
MSL goniospectro- photometer	LDLS & SC laser	N/A	1.5	Double monochromator	polarized	4 / 8	N/A	N/A	0.2 %
CMI gonio- reflectometer	LDLS	N/A	5	Double monochromator	polarized	10	0.66	1e–4	2 %
KUL gonio- reflectometer	250 W Xe lamp	VIS	3	Given by detector	unpolarized	13	0.1	2.4e–6	2 %
NPL NRR	tungsten ribbon lamp	320–2500	N/A	Interference filter	polarized	< 20	N/A	N/A	0.2 %
NRC gonio- reflectometer	SR with 100 W QTH lamp	380–1068	5 / 20	Given by detector	unpolarized	10 (at exit)	2.9	2e–3	0.5 %
PTB primary BTDF facility	LDLS & SC laser (planned)	170–2100	N/A	Double monochromator	polarized	3 / 15 / 45	3.5° / 0.6° / 0.1°	2.9e–3 / 8.6e–5 / 2.4e–6	0.5 %
	CW lasers	642 / 445	< 1	N/A		1–50	0.1 / 0.35	2.4e–6 / 2.9e–5	N/A
	white light LED (planned)	450–700	N/A	Interference filter		N/A	N/A	N/A	N/A

growing knowledge on them. Thus, the ones shown in this table are extracted from most recent publications. “N/A” means that the information is not available or not reported in any publication.

Apart from the *Gonioreflektometer* mentioned in previous text, PTB has also developed another similar facility named “3D appearance robot-based gonioreflectometer (ARGon³)” [91]. Both facilities utilize a similar light source, an integrating sphere providing spectrally broadband and spatially homogeneous irradiation [92, 93], and in sample positioning, using the same model of five-axis robot arm to hold the sample under test. The main difference lies in the detection system. In addition to the photodiodes and photomultiplier tubes for the detection of different spectral ranges used on the *Gonioreflektometer*, ARGon³ has a line-scan and a luminance camera, providing more capabilities for the measurement of samples with non-conventional surface such as curved or anisotropic characteristics. The modified NaNoRef at PTB was a temporary setup used to perform the European-wide BTDF comparison reported in Paper III. It was an alternative solution to cope with the delayed development of PTB’s new primary BTDF facility within the European Metrology Programme for Innovation and Research (EMPIR) project BxDiff (“New quantities for the measurement of appearance”) [94]. The modified NaNoRef setup was thoroughly introduced in Paper I. Its scale of BTDF measurement was successfully transferred to the new primary BTDF facility through a comparison at a later time. Results of this internal comparison are reported in the first part of Paper IV. One might notice that the measurement uncertainty of the modified NaNoRef is lower than that of the new primary BTDF facility. The reason is that the goniometric parts of the modified NaNoRef setup have been well-calibrated through the years, whereas the primary BTDF facility is still under development and improvement. The main advantage of the new primary BTDF facility against the modified NaNoRef setup is its capability of performing BTDF measurement at any desired geometry, including those out-of-plane geometries where the directions of incidence, sample normal, and detection do not lie within one plane. This is achieved by using a six-axis robot arm for sample positioning, which will be introduced in Section 1.2.3.

The NMI of the United States, the National Institute of Standards and Technology (NIST), has also developed multiple goniospectrophotometers. One is the Spectral Tri-function Automated Reference Reflectometer (STARR), developed in the 20th century, which is the national reference instrument for spectral bidirectional reflectance measurements of spectrally neutral, non-fluorescent samples [95, 96]. It consists of different broadband light sources and photodiodes to cover the spectrum

from ultraviolet spectrum range (UV) to near-infrared spectrum range (NIR). One difference to PTB’s *Gonioreflektometer* is the applied measurement scheme. Details regarding the under- and over-irradiation schemes will be discussed in Section 1.3.1. The sample under test is held by a combination of linear and rotary stages on the STARR. Similar to the modified NaNoRef, this way of sample positioning lacks the degrees of freedom needed for out-of-plane measurements. Therefore, another facility called Robotic Optical Scattering Instrument (ROSI) (formerly known as STARR II) has been recently developed to provide more measurement capabilities using a six-axis robot arm for sample positioning and to improve signal-to-noise ratio (SNR) by applying a laser-driven light source (LDLS) and a supercontinuum (SC) laser as light sources [97–99]. There is one more facility at NIST for the measurement of optical appearance, mainly spectral reflectance, of colored samples with gonioapparent properties [100, 101]. This facility is called the five-axis goniospectrometer. Compared to STARR and ROSI, this facility provides the capability of performing spatially resolved BRDF measurements with a finer spectral resolution.

The Goddard Space Flight Center (GSFC) at National Aeronautics and Space Administration (NASA) has its own scatterometer facility to characterize the BSDF of materials for Earth’s on-orbital application [102]. The BTDF measurement capabilities of this facility were validated by a bilateral comparison of two Lambertian samples to the STARR at NIST [103, 104].

At the Measurement Research Institute of Aalto University (Aalto), which is the Finnish National Standards Laboratory, two goniospectrophotometric facilities were developed. One is the “absolute reference gonioreflectometer”, which is the national reference facility for spectral reflectance [105, 106] and another one is a newly-developed instrument called “3D-gonioreflectometer”, which provides additional capability of performing out-of-plane BRDF and BTDF measurements [107, 108]. Thus, the main difference between these two facilities lies in the realization of sample holding. Another major difference is that the 3D-gonioreflectometer can only obtain measurement results relative to a standard, calibrated by absolute measurement using Aalto’s absolute reference gonioreflectometer.

At *Consejo Superior de Investigaciones Científicas* (CSIC), the Spanish National Research Council, the goniospectrophotometer *Gonio-EspectroFotómetro Español* (GEFE) can be adapted to measure different scattering quantities [109–111]. The sample under test is held by a six-axis robot arm. BSDF can be measured using a xenon (Xe) lamp and a spectroradiometer in the over-irradiation scheme. By changing the light source to an LDLS and using a charge-coupled device (CCD) or

a complementary metal-oxide-semiconductor (CMOS) camera, absolute BSSRDF values can be obtained. The periscopic configuration of optics on this instrument also allows for retro-reflection measurements to be performed.

The goniospectrophotometric facility at *Conservatoire National des Arts et Métiers* (CNAM), the DI in France, has several lines of light sources and detection systems, making it also possible to be adapted to measure different measurands. One configuration is the primary goniospectrophotometer as the French national standard for BRDF scale in VIS [112]. This configuration can be switched to the so-called “Conoscopic Device for Optical Reflectometry (ConDOR)” mode [113–115]. Using a conoscopic design of optics, the Fourier plane of the scattered light is imaged on a CCD camera, allowing for BSDF measurements with ultra-high angular resolution. Thus, this configuration is usually used to measure specular gloss [116]. Recently, another configuration has been developed to perform BRDF measurement in the micrometer length scale (μ BRDF) [117, 118]. It has an LDLS as light source focused to a spot with diameter below 100 μ m on the sample surface. This configuration is also used to perform BSSRDF measurement.

The gonireflectometer at Measurement Standards Laboratory of New Zealand (MSL) is the national reference instrument in New Zealand. It has participated in multiple comparisons with other institutes and has a low uncertainty in BSDF measurements. Its components have been thoroughly characterized in [119–122]. Interestingly, it has been pointed out that the formerly used robot arm had larger uncertainty in sample positioning and was therefore replaced by a 3-axis rotation stage, so that the three rotation axes of the sample can be adjusted independently. It can be noticed that in Paper III and in [88], both MSL and PTB have reported BTDF or BRDF results with low uncertainties. But the measured values do not overlap within their expanded $k = 2$ uncertainties. Thus, more research is currently being conducted regarding this discrepancy.

The gonireflectometer at *Český Metrologický Institut* (CMI), NMI in Czech Republic, shares some similarities to the facilities at CSIC and MSL. It also has a periscopic configuration of the illumination optics and a detection system developed in cooperation with MSL. The gonireflectometer at *Katholieke Universiteit te Leuven* (KUL), a research university in Belgium, also has similar components to the facilities mentioned in the previous text. Details can be found in [123]. The National Reference Reflectometer (NRR) at National Physical Laboratory (NPL), NMI in the United Kingdom, was developed in the end of 20th century for absolute spectral reflectance measurement [124–127]. However, its application has been dis-

continued for some years and it is currently in the process of major remodeling. The gonireflectometer at National Research Council Canada (NRC), NMI in Canada, was inspired by the *Goniorefleksometer* of PTB [128]. The main difference lies in the detection system, as NRC’s facility utilizes array-type spectroradiometers.

1.2.2 BTDF Samples

The study involves samples with different scattering properties. Overviews of them were reported in Papers I – IV. In this section, some more discussions on transmissive and translucent samples are given, especially on the reference standard samples for BTDF measurement. Currently, there is no clear definition of “transmissive” and “translucent”, so the transition from one property to another is rather smooth. Here, we distinguish the sample to be transmissive or translucent based on whether an object, placed behind this sample, can still have a distinct image or not.

Since the absolute values of BSDF are usually difficult to measure, one easier approach is to use a set of samples as reference standards and compare the measured value of an unknown sample to them. This led to the search for a so-called perfectly reflecting diffuser (PRD). A PRD reflects the incident radiation isotropically in every direction in the hemisphere and has thus a constant BRDF of $1/\pi$ [sr⁻¹] [37]. Such material has a Lambertian surface but cannot be realized materially. As already mentioned in Section 1.1.2, several materials have been found to exhibit quasi-Lambertian properties such as barium sulfate, PTFE, ceramic [129], and opal glass [130–132]. Even if their properties deviate strongly from a Lambertian surface when irradiated at extreme incident angles, they are still commonly accepted as diffuse reflectance standards [38].

However, to date there exists no common standard for diffuse transmittance. It was found out in several studies that a material made of fused silica (SiO₂) with injected air bubbles of several micrometers in diameter could provide a more Lambertian BTDF than PTFE-based thin foil [104, 133, 134]. Note that PTFE is by far the only material which would be suited as standard for both reflection (bulk material) and transmission (thin foil) measurements. Samples made of these two materials were also involved in the round robin comparison reported in Paper III, where the authors were still convinced that thin PTFE foil would be better suited as a diffuse transmittance standard. The reason is that some test results of the fused SiO₂ sample exhibited a fluctuation in the measured BTDF values while only changing the sample’s azimuthal orientation. An assumption for this observation was that the air bubbles somehow might have formed a preferred orientation during

the manufacturing process and became thus slightly anisotropic. However, it was not experimentally confirmed due to an overall low signal level of transmitted light, so that other sources of influence could not be easily separated. What's more, as described in the Supplementary Material of Paper III, some patterns of millimeter scale could be observed when one side of the SiO_2 sample was illuminated under room light while the other surface appeared rather homogeneous. Comparison of BTDF measurements when the same sample was irradiated from the different side also resulted in slightly different values, proving the importance of specifying a reference sample surface in a comparison, even for nominally isotropic quasi-Lambertian samples.

Apart from Lambertian samples, other types of standard sample are also needed for those depicting far-off Lambertian scattering behavior. Ideally, one should compare a sample's scattering distribution to a standard with scattering characteristics as similar as possible. Because a significant difference in the angular distribution could bring large uncertainties to the relatively measured BTDF values. Therefore, for transmissive samples exhibiting e.g. a narrow Gaussian distribution such as the ground glass diffuser mentioned in Paper III, a Gaussian diffuser would be more suited as a standard sample. However, there are also other samples with very special scattering distribution as a result of e.g. an engineered surface such as the one described in Paper III. In that case, neither a Lambertian nor a Gaussian sample could be used as a proper standard to obtain accurate BTDF value. For these samples, either one sample of the same type, carefully measured, can be used as a standard, or they should be measured by applying the absolute method whenever possible.

The translucent samples exhibit strong lateral scattering of the incident radiation within the material's bulk volume. It leads to a significant size difference between the incident beam on the sample front surface and the transmitted beam on the sample back surface. This results in ambiguity in the definition of BTDF, in which the size difference of irradiated areas on the front and back sample surface is not taken into account. And this causes the measurement result to be dependent on the measurement conditions. Because in some cases the transmitted radiation can be scattered beyond the boundary of the defined collection area, resulting in the so-called "edge losses". The translucent samples investigated in this thesis are thoroughly reported in the second part of Paper IV, as well as the discussion regarding accurate characterization of them. In Section 1.3.3 some more details will be discussed.

All in all, the samples studied and characterized in this work span a large range from transmissive to translucent ones, giving a good representation of different types of light transmitting materials.

1.2.3 PTB's new primary BTDF facility

The development of PTB's new primary BTDF facility took consideration of advantages and disadvantages observed from some of the existing goniospectrophotometers. A general layout is illustrated in Figure 1.4. The facility contains three main parts to perform accurate BTDF measurement: irradiation sources, detection systems, and mechanical parts to realize different measurement geometries. These three main parts will be thoroughly introduced in this section.

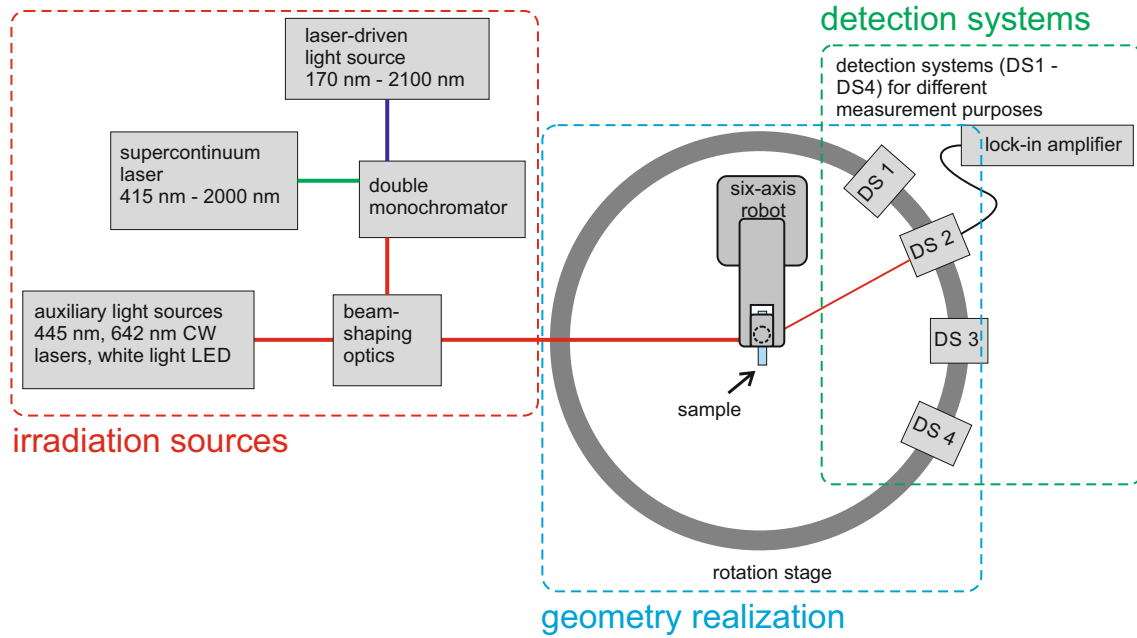


Figure 1.4: General layout of PTB's new primary BTDF facility.

The irradiation is wavelength-selected, i.e., its central wavelength and spectral bandwidth are determined before being incident on the sample. Different sources will be applied to cover the spectral range from UV over VIS to NIR. For the current setup utilizing two CW lasers as irradiation sources in combination with an imaging detection system, the flexibility in size selection of irradiation area and measurement area allows for an easy switch of the measurement scheme between under- and over-irradiation. This is discussed in more detail in Section 1.3.1 and in the second part of Paper IV. The planned extension of the irradiation spectrum also takes advantage of the high intensity provided by an LDLS and an SC laser. Moreover, the

irradiation from the broadband sources will be coupled into optical fibers and double monochromators, and then outcoupled by means of fiber collimators to generate beams with different sizes. This configuration avoids the complex optical design for the irradiation beam path and is also less influenced by ambient straylight.

To cope with the broad spectrum of interest, different detection systems need to be utilized accordingly. The current detection system, consisting of an achromatic lens, several diaphragms, a silicon (Si)-photodiode mounted on an integrating sphere, and a lock-in amplifier, will also be extended to have spectral and spatial resolution by integrating a spectroradiometer and a CMOS camera. The detection systems are mounted on a motorized rotation stage with 1.5 m diameter, whose rotation axis lies on the back side of the sample, i.e., the surface facing towards detection. The sample is held vertically by a robot arm with six moving axes. Thus, the sample's six degrees of freedom can be adjusted independently. With these two moving mechanical components, almost any desired geometry for BTDF measurement can be achieved.

When the planned further development is complete, the instrument will be enclosed in a light-proof chamber to further minimize the influence coming from straylight. More details of the facility are described in Papers II and in the first part of IV.

Irradiation Sources

Different irradiation sources are employed and planned for PTB's new primary BTDF facility. Currently in use are two narrowband CW lasers, one at 642 nm and another at 445 nm. The bandwidth of both lasers is smaller than 1 nm. They were used in the validation of BTDF scale transfer from the modified NaNoRef setup to the new primary facility. Detailed configurations of these two lasers are reported in Papers I and II. By using a scientific camera with a CMOS chip of dimensions 22.5 mm $\times$ 22.5 mm, the spatial intensity profile and divergence of the incident beam can be examined and determined.

It has been observed that the beam profile can be improved considerably by using a rotating weak diffuser, placed between the two lenses for beam conditioning (beam expander). Its position along the optical path affects both the spatial intensity distribution and the divergence of the beam. When it is exactly placed at the intermediary focus of the beam expander, the resulting beam profile becomes a superposition of the output laser beam profile and structures on the surface of the rotating diffuser. The beam divergence, on the other hand, is almost not affected

at all. By moving the diffuser slightly away from the focus, the spatial uniformity of the beam profile is improved at the cost of a slightly larger beam divergence.

This is illustrated in Figure 1.5 for the 642 nm laser, for instance. The situation without using the rotating weak diffuser can be seen in Figure 1.5a, in which the photo, taken at the position of the sample plane, of an expanded laser beam clipped with a diaphragm with diameter of 10.2 mm is shown, together with the intensity distribution passing through the beam center along the horizontal (yellow) and vertical (red) directions. The beam is well-collimated with a divergence of 0.05° (full angle, if not specifically mentioned in the following text). But the beam profile exhibits ring patterns with variation of irradiance level larger than 35 % between the bright and dark pattern (in several extreme places even more than 100 %).

By placing a rotating weak diffuser in the vicinity of the intermediary focus of the beam expander, the beam profile becomes much better, with only a 10–15 % difference of irradiance level from the center to clipping edge and no short-term variation at all. This is illustrated in Figure 1.5b, with the photo taken at the same position and the beam clipped by the same diaphragm as in the former case. The intensity distribution at the same position on the beam along the same directions is also shown. Now the beam divergence increases slightly to about 0.1° . In this configuration, the largest realizable incident beam diameter is 35 mm.

By further moving the diffuser several centimeters out of focus, the beam divergence increases to 0.35° while the beam uniformity becomes even better, as the photo shown in Figure 1.5c. The photo was taken at the same location and the beam was clipped by the same diaphragm as in the former cases, together with the intensity distribution of the same position on the beam along the same directions. Now the difference of irradiance level from center to clipping edge is still about 10–15 %, but the long-term irradiance level variation within the beam is much smaller. In this case, the largest achievable incident beam diameter is limited by the size of the 2"-iris diaphragms for straylight reduction, which is about 50 mm. The disadvantage of this configuration is the low irradiance level impinging on the sample since the beam is no longer well-collimated.

For measurements applying the under-irradiation scheme, the beam profile does not need to be perfectly uniform, since the measured result represents only the characteristics averaged within the irradiated area. As long as all the incident and transmitted radiation is fully collected by the measurement area, the result can be considered accurate. Thus, the beam profile in Figure 1.5b is suitable for this scheme for keeping a small divergence and a high irradiance at the same time. But

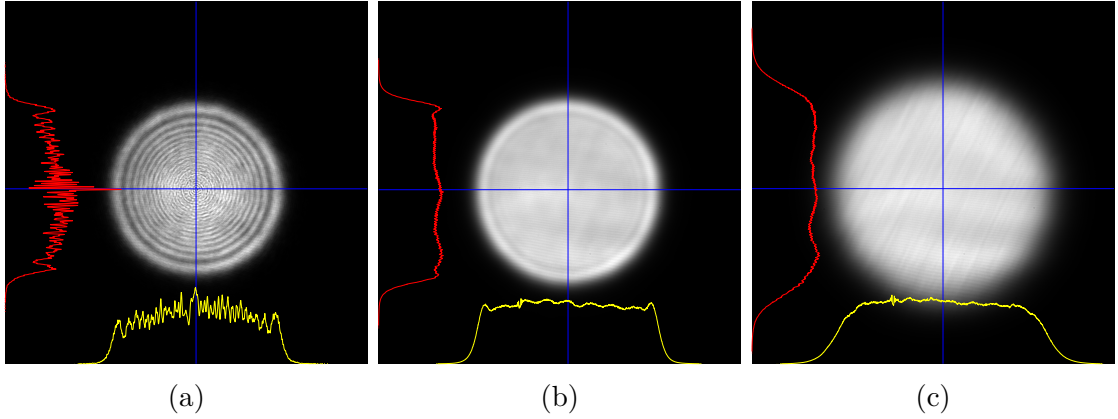


Figure 1.5: Spatial intensity profile of the incident beam clipped by a diaphragm with 10.2 mm diameter when (a) no rotating weak diffuser is used for beam homogenization or the rotating weak diffuser is placed (b) in the vicinity of and (c) several centimeters away from the intermediary focus of the beam expander.

for measurements performed in the over-irradiation scheme, the intensity variation within the beam brings additional uncertainty to the measurement results. This uncertainty source becomes non-negligible especially when translucent samples are measured. Because in this case the transmitted radiation is an average of contributions not only from the part of beam within the measurement area, but also from the neighboring parts outside of the measurement area due to lateral scattering. On the other hand, the incident radiation is only considered as the part within the measurement area. Therefore, a beam profile as uniform as possible, like the one in Figure 1.5c, would minimize the difference of transmitted radiation generated by different parts of the incident radiation. However, a compromise has to be made in the beam divergence and signal level.

In further development a white light light-emitting diode (LED) source will be added as the third irradiation line, because it can be easily used for test measurements without having too much UV and laser exposure. This source can provide radiation with high luminance from 450 nm to 700 nm through an optical fiber. Combining this LED source with interference filters, the spectrally resolved BTDF in the VIS can be measured.

The spectral range of the new primary facility will be further extended by integrating an LDLS and an SC laser into the irradiation sources. The LDLS utilizes a focused laser beam to stimulate the Xe gas into plasma state and thus generates radiation from about 170 nm to 2100 nm. It provides a high radiance especially in the UV and VIS range. The SC laser is also a broadband light source, utilizing

a pump laser to achieve spectrally broadened radiation after propagating through an optical fiber with high nonlinearity. The spectral range of this source is from about 415 nm to 2000 nm. High power is required to generate the nonlinear effect of the fiber material. Thus, the SC laser is a pulsed source with a pulse duration of 400 fs and a repetition rate of 80 MHz. The pulsed signal can be transformed to a quasi-CW behavior by sending the output radiation of the SC laser into a so-called “pulse-to-CW converter” [135, 136]. This converter consists of a bundle of 100 optical fibers with incrementally different lengths. As a result of different time delays of the pulses, the 80 MHz repetition rate can be increased to 8 GHz. In addition, each pulse is temporally broadened while propagating through the fiber, and in the end a signal with nearly constant power is generated.

These two broadband light sources will be fiber-coupled into two cascaded Czerny-Turner monochromators [137] with a focal length of 500 mm. Each monochromator consists of an interchangeable triple-grating turret and two collimating mirrors to image the radiation from the entrance to the exit slit with a 1:1 ratio. Each diffraction grating has different line density and is blazed at a specific wavelength. The spectrally selected radiation will be outcoupled using fiber collimators to achieve different sizes of the beam incident on the sample. Normally set to the same value, the width of the entrance, middle, and exit slits Δx of the double monochromator determines the spectral bandwidth $\Delta\lambda$ and central wavelength λ of radiation. However, $\Delta\lambda$ and λ usually deviate from their nominal value in real measurements. In order to lower the measurement uncertainty, this deviation needs to be taken into account in the evaluation of measurement results. For a certain grating setting of the double monochromator, a wavelength correction function describing the relation between $\Delta\lambda$, λ , and Δx can be determined with the help of gas-discharge lamps (or spectral lamps such as HgAr and HgNe), because these lamps have well-defined spectral lines as reference. By performing a wavelength scan in the working spectral range of this grating setting, the actual values of $\Delta\lambda$ can be measured at the expected λ . These measured $\Delta\lambda$ values can be fitted by a 2D-surface with respect to the reference λ and the set Δx , and their relation can be interpolated for the working spectral range of a specific grating setting. This was performed for the grating with 2400 lines/mm, which has working range 250 nm – 650 nm. The function for the 2D-fit is:

$$\begin{aligned} \Delta\lambda = & a + b \ln \lambda + c \Delta x + d(\ln \lambda)^2 + e \Delta x^2 + f \Delta x \ln \lambda + g(\ln \lambda)^3 \\ & + h \Delta x^3 + i \Delta x^2 \ln \lambda + j \Delta x (\ln \lambda)^2, \quad (1.20) \end{aligned}$$

with a to j being the fit parameters with values in Table 1.2. Similar wavelength correction functions should be performed for all gratings in future development.

Table 1.2: Fit parameters of wavelength correction function for the 2400 lines/mm grating setting.

Parameter	Value	Standard uncertainty
a	37.99	5.57
b	-19.46	2.83
c	-1.39	0.34
d	3.32	0.48
e	0.04	0.03
f	0.68	0.11
g	-0.19	0.03
h	0.01	0.00
i	-0.02	0.00
j	-0.06	0.01

Detection Systems

The detection system of the new primary BTDF facility consists of two parts. The first part collects the transmitted radiation with specific detection solid angle, subtended by interchangeable detection diaphragms, and within certain measurement area on the sample, defined by interchangeable field diaphragms. A sketch of the side view of this part is demonstrated in Figure 1.6. The size of detection diaphragm and its distance to the sample give the solid angle of the collected signal and determine the resolution of the angular scan. The field diaphragm is imaged on the sample back surface by an achromatic lens with focal length of 250 mm. In this way, both the collimated incident (red lines in Figure 1.6) and the divergent transmitted radiation (blue lines in Figure 1.6) within the same defined area on the sample surface, the measurement area, is measured. The 3.3" integrating sphere makes it possible for the collected radiation to be homogeneously and polarization-independently detected. The second part of detection system comprises photosensitive detectors and amplification devices. Currently a Si photodiode is attached to the integrating sphere to convert the optical signal into electronic current signal. Then the current signal is converted to voltage signal with an amplification factor 10^7 V A^{-1} by a pre-amplifier. The signals are detected using lock-in technique, i.e., the signal output from the source is modulated at a frequency of 12.5 Hz by an optical chopper placed directly after the source. A lock-in amplifier then measures the signal exactly at

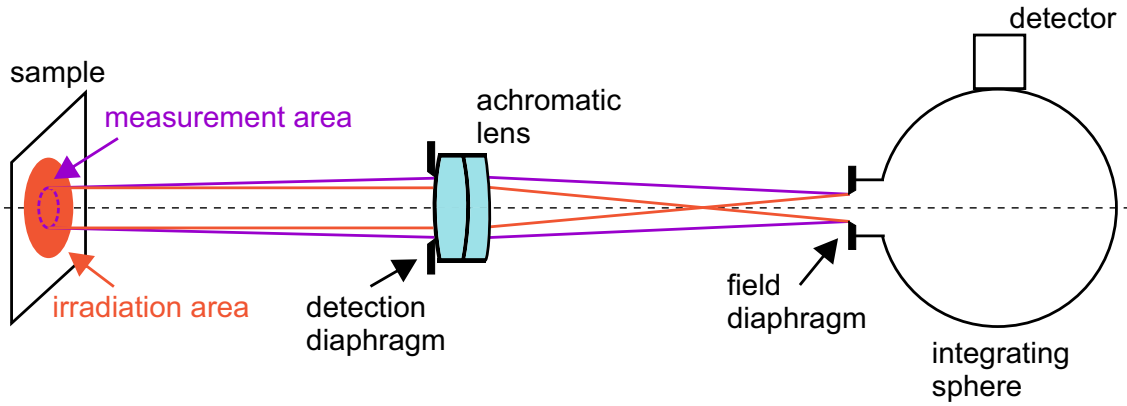


Figure 1.6: Sketch of the side view of detection optics.

this reference frequency and therefore, the straylight from other undesired sources can be suppressed.

For future development other detectors will be integrated into the detection system. A scientific CMOS camera mentioned in Section 1.2.3 can provide additional spatial resolution for the BTDF measurement. It can also reduce the number of detection angles for the angle-resolved measurements, because the spatial information captured by the camera can be converted into angular distribution. A spectroradiometer can provide spectrally resolved results for a specific spectral range with a single measurement. It would be useful when measuring with a large irradiation bandwidth such as when using the white light LED source. Different photodiodes and/or photomultiplier tubes will also be implemented to cover the whole measurement spectrum.

Different amplifiers will also be applied in future measurements. A drawback of the lock-in amplifier is the rather long “settling time”. It is the time needed for the signal to be stabilized after every value change. Two consecutive signal detections must be separated by at least this settling time to make them uncorrelated. This time is dependent on the integration time and the electronic low-pass filter applied for detection. From our test, even with the lowest order of low-pass filter the settling time should be about 20 times of the integration time. For a normal BTDF measurement with 1 s integration time, an angular scan will end up with about 2 hours for 15 detection geometries in one polarization state. Therefore, it is planned to use a ultrastable low-noise current amplifier (ULCA) [138, 139] as well as a picoamperemeter as detection amplification devices, after the facility is enclosed in a light-proof chamber. The nonlinearity of amplifiers should be characterized and later corrected for the measurement results. For example, the nonlinearity of one

prototype of the ULCA was studied using the double-aperture method introduced in [140]. It has been experimentally observed that its performance is comparable to that of the existing calibrated picoamperemeter. One advantage of ULCA is that it can be tailored to cover the typical signal range for BTDF measurements without switching electronics. Because it was found from very accurate measurement results that the switching factors between different ranges of the amplifier are a significant source of measurement uncertainty [54].

Realization of Measurement Geometry

The geometry of BTDF measurements is realized by moving the sample with the 6-axis robot arm and rotating the detection system with the rotation stage. The irradiation is static and can only be switched between different sources. The six degrees of freedom (three translational and three rotational) can be realized by the six independent joints of the robot arm. The parameters of joints are calculated from the set sample position by using the inverse kinematics of the Denavit-Hartenberg transformation [141–143]. The calculation also allows for prediction of potential collision danger and obstruction of the optical path by the robot arm itself at specific measurement geometries [144].

The repeatability of sample positioning was tested for the robot arm. To do that, a planar mirror was mounted on the flange of the robot arm and a laser beam was reflected to a screen at about 7.3 m distance. The mirror was repeatedly moved out of and into the optical path and the position of the laser spot was determined by evaluating its center of gravity of intensity distribution on the paper. With the servomotor permanently switched on and using backlash compensation to reach the set position, the repeatability of the robot arm for sample positioning is about 0.011 mrad in the rotation system.

A large rotation stage with 1500 mm diameter is responsible for realization of detection geometries, as the whole detection system is mounted on it. It has an integrated direct-drive torque motor and utilizes an absolute-encoded system for position sensing. The position is read out by two scanning heads to compensate for the possible mounting eccentricity and radial runout of the rotation stage. Similar to the robot arm, the positioning repeatability was also determined for the rotation stage. In this case, the planar mirror was mounted on the rotation stage and reflected the laser beam to a paper at about 8 m away. The mirror was repeatedly moved out of and into the optical path both in the clockwise and counterclockwise directions. The position of the laser beam on the paper was evaluated using the same method.

The positioning repeatability of the rotation stage was determined to be about 0.012 mrad in the rotation system.

With the combination of the robot arm and the rotation stage, almost all measurement geometries can be realized, including out-of-plane geometries. Controlling of these two positioning components is realized by an in-house developed LabVIEW program¹. Together with the irradiation sources and detection system mentioned in the previous subsections, fully automated BTDF measurement can be performed. After this facility was developed, its measurement capabilities have been validated, directly and indirectly, by different scale comparisons. The consistency achieved in these comparisons makes this newly developed facility suitable for the primary realization of BTDF measurement at PTB.

1.2.4 BTDF Scale Comparisons

A scale comparison of a measurand is a useful way to validate the measurement capabilities of a measuring facility or to transfer the measurement scale from one facility to another. To do so, some representative samples will be selected and measured on the participating facilities. The reported measurement results, i.e., the values of the measurand and their corresponding measurement uncertainty (normally the expanded $k = 2$ uncertainty), will be evaluated using statistical methods such as the χ^2 test to perform consistency check on the results measured by the different facilities. Consistent measurement results indicate that the involved measuring facilities can perform plausible measurement of this measurand with reasonably estimated measurement uncertainty. Within the frame of this thesis, three scale comparisons were performed for BTDF measurements. They are reported in Papers II, III, and IV. The samples involved in these comparisons are introduced in the papers and in Section 1.2.2.

The first comparison was performed between PTB's modified NaNoRef and Aalto's 3D-gonioreflectometer for two samples, one quasi-Lambertian and one narrow Gaussian. Results are reported in Paper II and summarized at the beginning of Chapter 3. In this comparison, the BTDF was compared in not only in-plane but also out-of-plane geometries.

The second scale comparison involved six metrology institutes and is reported in Paper III ("intercomparison" in the following text). The results are summarized at the beginning of Chapter 4. At PTB, the BTDF of the samples was studied and measured on the modified NaNoRef setup. This intercomparison was the first

¹This program is written by Marcel Pastuschek.

one in which the European measurement capabilities of BTDF were compared at the metrological level. It was also the first time that diffusers with distinct transmitting properties were thoroughly studied. From this comparison, insights and experience were gathered in many aspects regarding e.g. the measurement protocol, the measurement parameters, and the measurement facilities. Results of this intercomparison would serve as a solid foundation and preparation for a future interlaboratory comparison of BTDF organized by the *Bureau International des Poids et Mesures* (BIPM) to complement the currently empty Calibration and Measurement Capability (CMC) database regarding this measurand.

The measurement capabilities of PTB's new primary BTDF facility were validated by the third comparison reported in the first part of Paper IV ("internal comparison" in the following text). Results are summarized at the beginning of Chapter 5. The BTDF scale was transferred from the modified NaNoRef to the new facility. The measurement parameters, such as irradiation wavelength, incident beam size, detection solid angle, and measurement area, were kept similar, and exactly the same samples were measured. Through this internal comparison, the BTDF scale of PTB's primary facility was indirectly compared to that of Aalto's facility. This was one goal of the BxDiff project.

1.3 Measurement Methodology

In Section 1.1.2 the definition of BTDF is introduced as the differential ratio of transmitted radiance L_t to incident irradiance E_i . However, no device can be realized to measure infinitesimal quantities. In real measurements, the obtained values only represent the physical quantities averaged over a finite interval, which is also referred to as the resolution. For instance, in BTDF measurements, the incident beam must have a certain spatial extent so that at least a part of the sample under test is irradiated. The incident radiation must exhibit a certain spectral extent as well, since all irradiation sources have more or less some broadening around their central spectral line. The detector used in BTDF measurements must also have a certain spatial extent, i.e., a non-zero solid angle, so that the optical radiation received by the detector generates enough signal level to surpass the electronic noise level. Therefore, the realization of BTDF is quite different from its definition. The measurement models used to determine BTDF are introduced in detail in this section, along with a thorough description of the uncertainty analysis based on the models.

In the last part of this section, issues regarding the accurate BTDF characterization of translucent samples with non-negligible lateral diffusion are introduced.

1.3.1 Measurement Model

A schematic illustrating the parameters involved in the realization of BTDF is shown in Figure 1.7. Similar schematics are also included in Papers I, II, III, and IV.

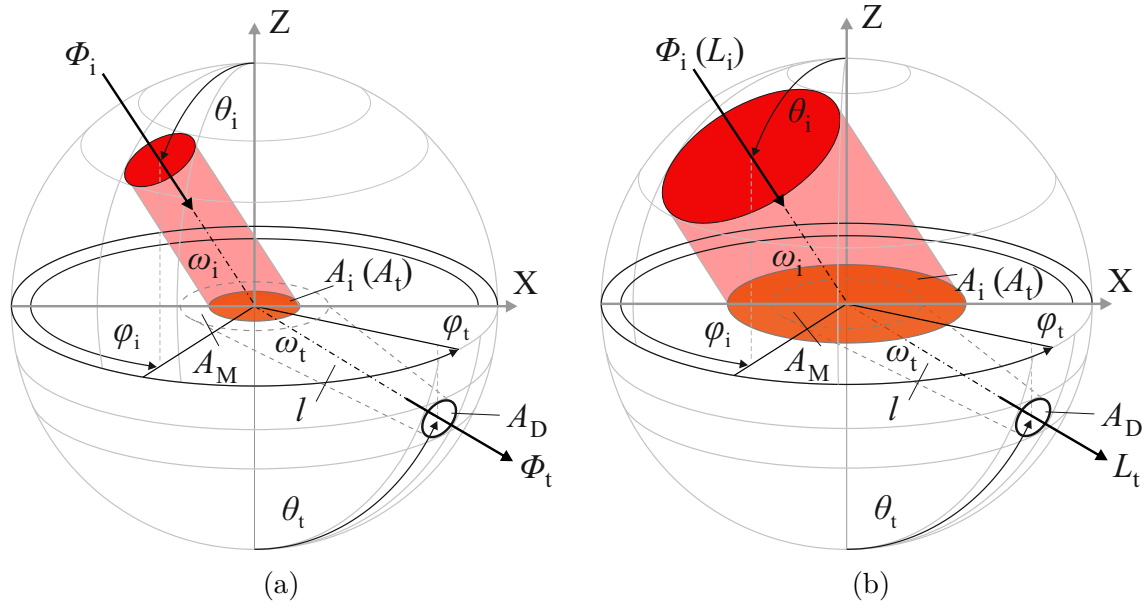


Figure 1.7: Schematic of realization BTDF measurements in (a) under-irradiated scheme and (b) over-irradiated scheme.

A collimated homogeneous beam of incident radiant flux Φ_i impinges on the front surface of the sample under test, generating a uniform irradiance E_i within an area on the sample front surface A_i . The angle between incident radiation and the sample normal is θ_i , and thus the area of the beam cross-section A_{beam} can be expressed as

$$A_{\text{beam}} = A_i \cdot \cos \theta_i, \quad (1.21)$$

relating E_i to Φ_i by

$$E_i = \frac{\Phi_i}{A_i} = \frac{\Phi_i}{A_{\text{beam}}} \cdot \cos \theta_i. \quad (1.22)$$

The incident beam is only nearly collimated under real conditions, therefore it is still weakly divergent or convergent. The incident radiance L_i on the sample front surface is thus written as

$$L_i = \frac{E_i}{\Omega_i} = \frac{\Phi_i}{A_{\text{beam}} \cdot \omega_i}, \quad (1.23)$$

with $\Omega_i = \omega_i \cdot \cos \theta_i$ being the projected solid angle of incidence. For an ideal light source, L_i should be a constant value within A_i . The area that includes all transmitted radiation on the sample back surface, i.e., the exiting surface of the radiation, is denoted as A_t . In Figure 1.7, A_t coincides with A_i since the sample thickness is assumed to be infinitesimal. Unlike L_i , the transmitted radiance L_t on the sample back surface should exhibit spatial dependence as a result of scattering. Nevertheless, the average value of L_t within a certain area can represent the characteristics of the whole sample under test, given that the sample is isotropic and homogeneous, and this area is much larger than the scale of the scattering structures or particles of the sample. This averaged L_t can be expressed as

$$L_t = \frac{\Phi_t}{A_t \cdot \Omega_t}, \quad (1.24)$$

with Φ_t being the transmitted radiant flux contained within the projected solid angle $\Omega_t = \omega_t \cdot \cos \theta_t$ of the detection system. ω_t is determined by the aperture area A_D , placed at the entrance of the detection system, and the distance l from this aperture to the sample back surface. The detector aperture restricts the amount of radiation entering the detection system and gives the detection solid angle ω_t according to equation 1.5 by

$$\omega_t = \frac{A_D}{l^2}. \quad (1.25)$$

Normally, some imaging optics are integrated in the detection system so that the measurement area A_M is well-defined on the desired sample surface by adjusting the depth of sharpness. Depending on the size relation between measurement area A_M and irradiation area A_i , the BTDF measurement scheme can be distinguished as under-irradiation and over-irradiation, as shown in Figure 1.7a and 1.7b, respectively. In the under-irradiation scheme, where $A_M > A_i$, the total radiant flux of incident and transmitted radiation is collected by the detector. Thus, combining equations 1.21, 1.22, and 1.24, the formula applied to calculate BTDF in this measurement scheme is

$$f_t = \frac{L_t}{E_i} = \frac{\Phi_t}{A_t \cdot \omega_t \cdot \cos \theta_t} \bigg/ \frac{\Phi_i}{A_i} = \frac{\Phi_t}{\Phi_i} \cdot \frac{1}{\omega_t \cdot \cos \theta_t} \cdot \frac{A_i}{A_t}. \quad (1.26)$$

For the case shown in Figure 1.7a where $A_t = A_i$, which also holds for most of the transmissive diffusers, the equation can be further reduced to

$$f_t = \frac{\Phi_t}{\Phi_i} \cdot \frac{1}{\omega_t \cdot \cos \theta_t}, \quad (1.27)$$

as seen in many literatures on BTDF measurements, including the four papers in Chapters 2–5 [81, 103, 104]. It is often employed because the under-irradiation scheme is not sensitive to slight misalignment in the measurement facility, since the area of evaluation is defined by A_i , which is always included in A_M and is not influenced by the depth of focus of the imaging detection system. Moreover, the optical power of the source is completely utilized in this scheme [41].

In the over-irradiation scheme, where $A_M < A_i$, the detector field of view (FOV) is constantly overfilled during the measurement. Thus, the radiance of incident and transmitted radiation is measured instead of the radiant flux. Using equation 1.23 the formula applied in this measurement scheme for obtaining the BTDF is

$$f_t = \frac{L_t}{E_i} = \frac{L_t}{L_i} \cdot \frac{1}{\omega_i \cdot \cos \theta_i}. \quad (1.28)$$

In this equation no consideration on the size relation between A_i and A_t is needed as in the under-irradiation case. Therefore, the over-irradiation scheme is normally applied to the measurement of thick translucent samples [35]. However, it will be introduced in Section 1.3.3 that in practice the edge loss of samples with significant subsurface scattering makes BTDF measurement in the over-irradiation scheme also dependent on the size of the irradiation area A_i .

The main difference between these two measurement schemes is illustrated in Figure 1.8. Both schematics show the back surface of a sample (square in black solid line) when perpendicularly irradiated (red filled circle). The black circle and blue ellipse in dashed line indicate the measurement area A_M projected on the sample back surface when the detection is perpendicular ($\theta_t = 0^\circ$) and at a certain angle ($0^\circ < \theta_t < 90^\circ$), respectively, assuming that the horizontal direction in this figure lies within the plane of angular scan. In the under-irradiation scheme shown in Figure 1.8a, A_M always includes the entire incident optical power and increases with detection angle θ_t , whereas A_i always has the same size. In the over-irradiation scheme as shown in Figure 1.8b, however, the size of A_i considered in the evaluation is the same as A_M and both of them change with θ_t . This difference results in the distinctive measurement models for the BTDF measurement.

On PTB's new BTDF primary facility, the optical power is converted into electrical (normally voltage) signal via a photodiode with some amplification and I/U conversion electronics in either measurement scheme. It can be expressed as

$$S = R_{\text{resp}} \cdot P, \quad (1.29)$$

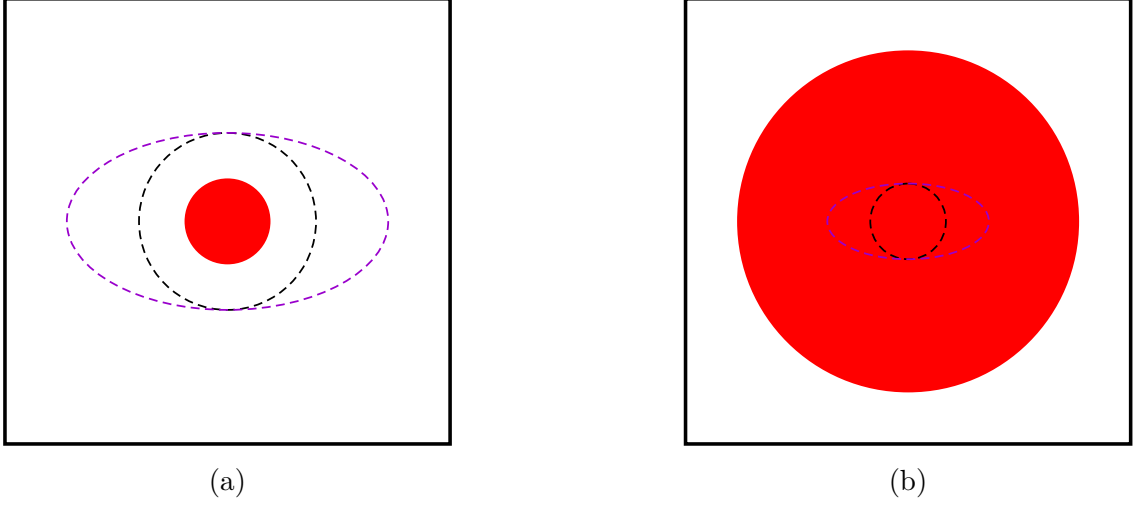


Figure 1.8: Difference between BTDF measurements in (a) under- and (b) over-irradiation scheme.

with S being the voltage reading on the detection electronic device (or current reading when using an amperemeter to measure the photocurrent directly), P being the optical power captured by the detector FOV, and R_{resp} being the responsivity of the whole detection system including, e.g., quantum efficiency and spectral responsivity of the photodiode, gain coefficient of the amplification electronics, conversion factor of the I/U converter, and so on. Note that R_{resp} is dependent on wavelength, polarization state, angle of incidence, and signal level of radiation falling onto the detector surface. In the ideal case, R_{resp} can be regarded as constant for detection of incident and transmitted signals. Thus, the model for BTDF measurements in the under-irradiation scheme using PTB's primary facility can be expressed as

$$f_t = \frac{S_t}{S_i} \cdot \frac{l^2}{A_D \cdot \cos \theta_t}, \quad (1.30)$$

since the collected optical power P is equal to the radiant flux Φ in equation 1.26.

In the over-irradiation scheme, the measured optical power is the part of radiant flux collected within A_M and must be treated differently for incidence and transmission. The incident optical power P_i is normally measured when the detector is perpendicular to the source. The L_i within A_M is dependent on θ_t and its relation to P_i is

$$L_i = \frac{P_i}{A_M \cdot \omega_i \cdot \cos \theta_t}. \quad (1.31)$$

The L_t within A_M is also dependent on θ_t and its relation to the collected transmitted optical power P_t is

$$L_t = \frac{P_t}{A_M \cdot \omega_t \cdot \cos \theta_t}. \quad (1.32)$$

Combining equations 1.28, 1.29, 1.31, and 1.32, the model for BTDF measurements in the over-irradiation scheme using PTB's primary facility is

$$f_t = \frac{S_t}{S_i} \cdot \frac{l^2}{A_D \cdot \cos \theta_i}. \quad (1.33)$$

Note that these two measurement models look rather similar, but the meaning and also the prerequisite are quite different. The BTDF of a Lambertian scattering sample measured in the under-irradiation scheme follows a cosine course while a constant signal relationship can be found for the same sample measured in the over-irradiation scheme. Thus, the measurement values of the under-irradiation scheme are said to be cosine-corrected [41]. To achieve accurate measurement, the sample (at least within A_M) must be uniformly irradiated in the over-irradiation scheme. This requires a constant value for the incident radiance L_i and a “large enough” irradiation beam, as mentioned in the definition. The under-irradiation scheme does not have these strict requirements, as long as A_i is much larger compared to the scattering structure size and in the meantime smaller than A_D and A_M to be fully collected by the detection system.

1.3.2 Uncertainty Analysis

In reality, the measurement models discussed in the previous section need to be extended with correction factors of different aspects. In this section, corrections and uncertainty sources will be discussed, with their influence on the BTDF measurement. The uncertainty analysis follows the procedures recommended in Guide to the Expression of Uncertainty in Measurement (GUM) [145].

In principle, every measurement result is an estimate of the measurand, which is derived from several physical quantities that are measured directly. Each quantity consists of a value and an uncertainty, depending on the probability distribution it follows. Some uncertainties originate from the statistical variation of multiple measurements. They are specified as Type A uncertainties representing the repeatability of the measurement and can be minimized by repeating the measurement. Other uncertainties are categorized as Type B uncertainties and are obtained from other information, such as calibrations, simulation models, and experience. All these un-

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certainties will be propagated through the measurement model to the combined uncertainty of the measurand. By choosing an appropriate coverage factor, the level of confidence that the measurand lies within an interval centering at the measurement result is determined (normally $k = 2$ for a confidence level of approximately 95 %). In this way, the accuracy of the measurement can be expressed.

For BTDF measurements performed on PTB's new primary BTDF facility, the sequence of one measurement cycle is illustrated in Table 1.3. Due to lack of signal monitoring¹, the fluctuation in irradiation power cannot be fully corrected for. But by taking average of the incident signal measured at the beginning and at the end of one cycle, at least the linear drift in the irradiation power of this cycle can be compensated². The transmitted signals are measured from 1st to the last (n^{th}) geometry (forward measurement) and subsequently from the last to 1st geometry (backward measurement) in one measurement cycle. In this way, not only the linear variation in the transmitted signal resulted from the fluctuation of irradiation power, but also the error in geometry positioning due to e.g. lack of backlash compensation can be corrected.

The applied model for the evaluation of BTDF measurements are:

$$f_t = \frac{\overline{S_t} - \overline{S_{t,\text{dark}}}}{\overline{S_i} - \overline{S_{i,\text{dark}}}} \cdot \frac{\tau_i}{\tau_t} \cdot \frac{l^2}{A_D \cos \theta_t} \cdot C_{\text{digi}} \cdot C_{\text{nl}} \cdot C_{\text{switch}} \cdot C_{\lambda} \cdot C_{\text{pol}} \cdot C_{\text{stray}} \cdot C_{\text{instr}} \cdot C_T \cdot C_{\text{wl-corr}} \cdot C_{\text{interR}} \cdot C_{\text{sam.uni}} \cdot C_{\text{irr.uni}} \cdot C_{\text{azi}}, \quad (1.34)$$

and

$$f_t = \frac{\overline{S_t} - \overline{S_{t,\text{dark}}}}{\overline{S_i} - \overline{S_{i,\text{dark}}}} \cdot \frac{\tau_i}{\tau_t} \cdot \frac{l^2}{A_D \cos \theta_i} \cdot C_{\text{digi}} \cdot C_{\text{nl}} \cdot C_{\text{switch}} \cdot C_{\lambda} \cdot C_{\text{pol}} \cdot C_{\text{stray}} \cdot C_{\text{instr}} \cdot C_T \cdot C_{\text{wl-corr}} \cdot C_{\text{interR}} \cdot C_{\text{sam.uni}} \cdot C_{\text{irr.uni}} \cdot C_{\text{azi}} \cdot C_{\text{sharp}}, \quad (1.35)$$

for the under- and over-irradiation scheme, respectively. The line over the variables for measured signal indicates that they are mean values from multiple readings. $S_{i,\text{dark}}$ and $S_{t,\text{dark}}$ are the reading of dark signals measured when the irradiation is

¹Efforts were made to set up a monitoring detector on the facility. However, it required a large number of tests to cope with the issue of synchronizing readings from the monitoring detector and the lock-in detector. This issue would be solved when a picoamperemeter is used in a future stage of development.

²Although the residual nonlinear components in power drift cannot be corrected for using this method, their influence on the measurement uncertainty was not observed from the current measurement results.

Table 1.3: Sequence of procedures in one measurement cycle of BTDF measurements performed on the modified NaNoRef and the new primary facility.

Sequence No.	Measurement procedure	Measured quantity
1	Sample moving out, detection to $\theta_t = 0^\circ$	—
2	Shutter open, incident signal reading	$S_{i,1}$
3	Shutter close, dark incident signal reading	$S_{i,dark,1}$
4	Sample and detection moving to 1 st geometry, ND-filter change	—
5	Shutter open, transmitted signal reading	$S_{t,1}$
6	Shutter close, dark transmitted signal reading	$S_{t,dark,1}$
7	Sample and detection moving to 2 nd geometry	—
8	Shutter open, transmitted signal reading	$S_{t,2}$
9	Shutter close, dark transmitted signal reading	$S_{t,dark,2}$
10	...(forward measurement until the last geometry)	...
11	Sample and detection moving to the n^{th} geometry	—
12	Shutter open, transmitted signal reading	$S_{t,n}$
13	Shutter close, dark transmitted signal reading	$S_{t,dark,n}$
14	...(backward measurement until the 1 st geometry)	...
15	Sample moving out, detection to $\theta_t = 0^\circ$, ND-filter change	—
16	Shutter open, incident signal reading	$S_{i,2}$
17	Shutter close, dark incident signal reading	$S_{i,dark,2}$

blocked by a mechanical shutter after the incident and transmitted readings, respectively. The range (amplification) of the measurement device remains unchanged for measuring the bright signal and its corresponding dark signal. τ_i and τ_t are the transmittance of ND-filters applied for measuring the incident and the transmitted signal, respectively. The rest in both equations are correction factors regarding different aspects. They are:

- C_{digi} is to correct the error introduced by the measuring device in the digitization process;
- C_{nl} is the correction factor regarding the nonlinearity of the detection system, which is dependent on both the amplifier and the detector;
- C_{switch} is the correction factor for range switching of the measuring device;
- C_λ is the correction factor concerning the deviation of applied irradiation wavelength from the wavelength for which the transmittance of ND-filters was calibrated;

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- C_{pol} is the factor related to the extinction coefficient of the polarizer;
- C_{stray} accounts for influence from straylight on both incident and transmitted signals;
- C_{instr} is the factor to correct the measured BTDF distribution regarding the instrument function;
- C_T is the factor to correct the measured BTDF value from the applied temperature to the desired temperature;
- $C_{\text{wl-corr}}$ is the factor to correct the measured BTDF value from the applied irradiation wavelength to the desired wavelength;
- C_{interR} is the correction factor for interreflection between sample and optics;
- $C_{\text{sam.uni}}$ is the correction factor for sample nonuniformity;
- $C_{\text{irr.uni}}$ is the factor to correct the influence of nonuniformity in the spatial intensity distribution of irradiation beam;
- C_{azi} is the correction factor accounting for the misalignment of azimuth when the sample's BTDF has a dependence on the azimuthal orientation;
- C_{sharp} is the correction factor related to the unsharpness of the imaging detection system. This factor only has a significant impact on measurements performed in the over-irradiation scheme.

Within the frame of this work, all correction factors are assumed to have Gaussian probability distribution and most of them possess the value one. Their influence is considered as uncorrelated uncertainties, which are quadratically added to obtain the combined standard uncertainty of the BTDF measurement results. Detailed uncertainty budgets of the BTDF results measured on the new primary facility are reported in Papers II and IV. For a better overview an exemplary uncertainty budget is summarized in Table 1.4. The determination of the directly measurable quantities and the estimation of the correction factors (or their uncertainties) are introduced in the following.

Signal Stability

Temporal fluctuation and drift in the measured signal are inevitable due to uncontrollable slight changes in the ambient condition. These can be caused by the

Table 1.4: Uncertainty budget for measurements performed on PTB's new primary BTDF facility within the frame of this work.

Source of Uncertainty	Standard uncertainty	Uncertainty in BTDF (%)
Signal stability	0.05–1.55 %	0.05–1.55
Filter transmittance	0.000074 for $\tau = 0.01$	0.74
Detection diaphragm area	0.026–0.035 %	0.026–0.035
Distance sample–detector	0.3 mm	0.09
Incidence polar angle positioning	0.05° (0.87 mrad)	not considered
Detection polar angle positioning	0.015° (0.26 mrad)	0.01–1.59
Digitization error	< 0.01 %	< 0.01
Wavelength deviation	0.2–0.3 nm	0.01–0.02
Polarization	< 0.05 %	< 0.05
Straylight	0.01–6.75 %	0.01–6.75
Instrument function	0.01–11.15 %	0.01–11.15
Temperature	0.5 K	< 0.01
Inter-reflection	< 0.01 %	< 0.01
Beam nonuniformity	15 % relative to the maximum beam intensity	0.01–0.02
Azimuthal misalignment	0.29°–0.58°	0.01–1.5
Imaging unsharpness	0.25 %	0.25
Combined standard uncertainty ($k = 1$)		0.77–11.39

temperature change in the laser cavity, the voltage change in the power supply, or the random occurrence of photons in the laser resonator. Even though the lasers applied in the BTDF measurements are specified to be extra stabilized for applications with high accuracy, variation in the laser output power still exists. The fluctuation can be roughly divided into short-term and long-term variations. The short-term fluctuation is often referred to as noise and can be minimized to a very low level by using a long enough integration time. The long-term fluctuation of a laser often exhibits a sinusoidal behavior and can be reduced by taking the average value from multiple measurements¹. Thus, fluctuation is typically categorized as Type A uncertainty. In the BTDF measurements performed within the frame of this thesis, each signal was always measured three times. This means that the value of measured signals is averaged out of six readings in one measurement cycle (forward + backward measurements). For one complete BTDF measurement three cycles are normally performed, resulting in 18 readings of the signal measured at one

¹This method is valid as long as the measurements are not taken at almost the same phase of the sinusoidal fluctuations.

geometry. In this way, the fluctuation and noise in both incident and transmitted signals can be reduced in the best case to below 0.05 % relative to the BTDF value.

The variation of laser output power also exists in an even longer temporal manner, which is called drift. As mentioned in Section 1.2.3, one measurement cycle can take several hours and the laser output power might drift away as the ambient temperature slowly changes. Drift belongs to Type B uncertainty and cannot be reduced by repeated measurement. The linear drift can be compensated by applying the measurement sequence introduced earlier in this section. However, the residual nonlinear behavior cannot be corrected as the output power is not monitored. Instead, it is estimated as the standard deviation of laser power drift observation by taking the difference between the incident signals S_i measured at the beginning and at the end of one measurement cycle as its value. This standard deviation should follow a rectangular probability distribution because the drift might take any value within the standard deviation with equal probability. Therefore, the standard uncertainty of laser power drift according to GUM is:

$$u_{\text{drift}} = \frac{\sigma_{\text{drift}}}{\sqrt{3}}, \quad (1.36)$$

with σ and u being the standard deviation and standard uncertainty of a measured physical quantity, respectively. In most of the BTDF measurements, the uncertainty of drift is smaller or comparable to the fluctuation of laser output power. In all Papers from Chapter 2 to 5, the uncertainty of fluctuation and drift is quadratically added and expressed together as the standard uncertainty of measurement signal stability. In future development of the facility, this uncertainty should be reduced by applying a monitoring detector to correct the measured signal.

Filter Transmittance

The ND-filters used in the BTDF measurements are calibrated for the VIS using the commercial setup Cary-5000 in 1 nm spectral step and at the nominal irradiation wavelength using the NaNoTrans facility. The relative uncertainty of filter transmittance is:

$$u_{\tau} = 0.5 \cdot (1 - \log_{10} \tau) \%, \quad (1.37)$$

and

$$u_{\tau} = 0.15 \cdot (1 + \sqrt{-\log_{10} \tau}) \%, \quad (1.38)$$

for Cary-5000 and NaNoTrans [146], respectively. A general principle for the selection of ND-filter is to apply the one with the lowest possible attenuation level, i.e., the highest possible transmittance, because the relative uncertainty of filters is inversely proportional to the filter transmittance according to equations 1.37 and 1.38. So far for the measurements performed on the new primary BTDF facility, the applied ND-filter has approximately 1 % transmittance, leading to the uncertainty of 0.7 % relative to the BTDF value, as reported in Papers II and IV, which is referred to the calibration on Cary-5000. Recently, the ND-filters have been calibrated on the NaNoTrans, and this uncertainty will be reduced to below 0.2 % relative to the measured BTDF value in future uncertainty analysis.

Detection Solid Angle

The uncertainty of detection solid angle stems from the uncertainty in determination of the detection diaphragm size A_D and the distance l from this diaphragm to the sample back surface. A_D is calibrated against a diaphragm standard using a confocal microscope. The relative standard uncertainty is from about 0.026 % to 0.035 %, depending on which diaphragm is applied in the measurement. The distance is first measured multiple times with an inside micrometer gauge, with a resulting standard uncertainty of 0.1 mm. Then a transmittance grating is mounted at the position of a sample and by evaluating the angular positions of the diffraction orders, the standard uncertainty of the distance is estimated to be 0.3 mm. Therefore, the standard uncertainty of distance determination is conservatively estimated as 0.3 mm, contributing 0.09 % relative uncertainty to the measured BTDF value.

Angle Positioning

Uncertainty of angle positioning is determined separately for the incidence polar angle θ_i and the detection polar angle θ_t . The former one is related to the positioning accuracy of sample under test, while the latter one is dependent on the positioning accuracy of detection diaphragm. Compared to the error introduced by positioning misalignment, the uncertainty in the repeatability of rotation stage and robot arm mentioned in Section 1.2.3 is insignificant and can be neglected. For the incidence angle positioning, a planar mirror is mounted to the robot arm using the normal sample holder and the θ_i is adjusted until the incident beam is reflected back to the irradiation source. The estimated uncertainty is 0.05° , i.e., 0.87 mrad. However, the contribution of this uncertainty to the BTDF value is generally not considered in this work since all studied samples were measured only at normal incidence.

For the detection angle positioning, its uncertainty can be estimated by the deviation from the center of a Gaussian fit on the measured instrument function (more will be introduced in the Subsection “Instrument Function” later) to the nominal $\theta_t = 0^\circ$ position. For the new primary facility, this value is estimated to be 0.015° , i.e., 0.26 mrad . On the one hand, the angle positioning uncertainty has large impact when the sample’s transmitted signal depicts strong variation with the polar angles. This is usually the case for θ_t variation. On the other hand, the influence of this uncertainty increases with θ_i for measurements performed in the over-irradiation scheme, and with θ_t for the under-irradiation scheme. An extra influence from this uncertainty source is related to the correction factor for wavelength $C_{\text{wl-corr}}$, since it is also dependent on θ_t (more will be introduced in the Subsection “Wavelength Correction” later).

Digitization Error

Digitization of signal measured by the measuring device can introduce additional uncertainty to signal reading. This uncertainty depends on the reading resolution S_{res} and the selected measuring range ζ . It is considered to possess a rectangular probability distribution and its influence can be determined from the first term in equations 1.34 and 1.35. As mentioned in the previous text, the corresponding dark signal is measured with the measuring range unchanged, thus, the uncertainty introduced by digitization resolution to the measured BTDF can be expressed as:

$$u_{\text{digi}} = \sqrt{\left(\frac{\partial f_t}{\partial S_t}\right)^2 \cdot u_{\text{res},t}^2 + \left(\frac{\partial f_t}{\partial S_{t,\text{dark}}}\right)^2 \cdot u_{\text{res},t}^2 + \left(\frac{\partial f_t}{\partial S_i}\right)^2 \cdot u_{\text{res},i}^2 + \left(\frac{\partial f_t}{\partial S_{i,\text{dark}}}\right)^2 \cdot u_{\text{res},i}^2}, \quad (1.39)$$

where $u_{\text{res},i}$ and $u_{\text{res},t}$ are the uncertainty due to reading resolution for incident and transmitted signal, respectively:

$$u_{\text{res}} = \frac{S_{\text{res}}}{2\sqrt{3}} \cdot \zeta. \quad (1.40)$$

For BTDF measurements performed in the frame of this thesis, different ranges were applied to measure incident and transmitted signals. Therefore, for the modified NaNoRef setup the relative uncertainty due to digitization error is smaller than 0.01% , even though the 12-bit measuring device can only digitize the signal to the 4th decimal place. Since the new primary facility has a 16-bit measuring device that digitizes the signal to the 5th decimal place, this uncertainty source is considered negligible.

Nonlinearity of Detector and Amplifier

The linearity issue of detector and amplifier arises when the magnitude difference of received signals is large. This issue exists for almost all photometric measuring devices. For BTDF measurements of nearly opaque materials, a difference of four to five orders of magnitude between the incident and transmitted signals can be found. Although it is possible to use ND-filters to compensate for the effect of detector nonlinearity, the use of attenuation filters also brings new sources of uncertainty to the measurement results. As mentioned in the Subsection “Filter Transmittance”, even if the attenuation filter is accurately calibrated by the national standard instrument, it already accounts for about 0.2 % relative uncertainty in the BTDF measurement when an attenuation filter with 1 % transmittance (two orders of magnitude) is applied. Compared to this, the relative uncertainty in spectrophotometric measurements due to nonlinearity would typically not exceed 0.1 % [147]. Nevertheless, the nonlinearity of the detection system should be calibrated and corrected for the measurement results. Since a calibration covering the full detection dynamic range requires large experimental effort, it was not carried out in the frame of this thesis but will be performed in the near future.

Switching Factor

As mentioned in the Subsection “Digitization Error”, the measuring ranges are switched to cope with different signal levels between the incident and transmitted signal. It was also mentioned in Section 1.2.3 that the switching factor from one measuring range to another is a dominant uncertainty source. The calibration for this can be performed in combination with linearity calibration and should be performed for the measuring device used on the primary BTDF facility in the near future.

Wavelength Deviation

The central wavelength of the irradiation has an uncertainty of about 0.2 nm for the 642 nm laser and about 0.3 nm for the 445 nm laser, because this uncertainty is dependent on the output power. This uncertainty does not largely influence the measured BTDF values in a direct way, since the transmissive and translucent materials are rather not spectrally selective. However, this uncertainty contributes to the BTDF result mainly through the resulted uncertainty in the transmittance of the applied ND-filters. As mentioned for filter transmittance, the value applied in

the measurement model is calibrated at the nominal wavelength. The deviation in the irradiation wavelength corresponds to a deviation from the actual filter transmittance to the nominal value. Therefore, its influence depends on the variation of the spectral filter transmittance. This is the reason for the large difference of influence resulted from the wavelength deviation reported in Paper II. In that paper, one measurement was performed at 405 nm and the spectral filter transmittance has a very steep variation around this wavelength, resulting in about 1.5 % uncertainty relative to the BTDF value. In contrast, for measurements performed at 642 nm, the variation of filter transmittance only results in about 0.03 % uncertainty relative to the BTDF value.

Polarization

The uncertainty regarding polarization originates from the imperfect separation of *s*- and *p*-polarization states of the incident radiation. Its influence can be estimated by the extinction coefficient of the applied polarizer together with the transmittance characteristics of the sample, applying the method introduced by Calderón et al. in [148]. For BTDF measured when the incidence direction is perpendicular to the sample surface, no dependence on polarization is expected according to the Fresnel equations [149]. Additionally, the current detection system on the new primary BTDF facility has an integrating sphere to eliminate the influence coming from polarization-sensitive detector. This can be confirmed by the insignificant difference between results measured with *s*- and *p*-polarized radiation. Thus, the contribution of polarization is neglected in the current uncertainty analysis. However, in future measurements when irradiation direction deviates from $\theta_i = 0^\circ$ as well as for materials exhibiting birefringence, the polarization influence of both incident and transmitted radiation should be carefully examined.

Straylight

Straylight is those parts of undesired radiation entering into the measuring system and being detected. The influence of straylight can be categorized as isochromatic, i.e., at the same wavelength as the irradiation, and heterochromatic, i.e., at other wavelengths than the irradiation. Since the signals are measured using lock-in technique with the chopper directly after the light source, the influence of heterochromatic straylight can be regarded as insignificant even without a light-proof compartment. The influence of isochromatic straylight was estimated by performing a normal angular scan while placing a beam blocker behind the sample. The

detected signal could not be differentiated from the background noise. Thus, the uncertainty due to straylight was considered to be at the same level as the dark signal, following a rectangular probability distribution. It turns out that straylight mainly affects the transmitted signal rather than the incident signal. Depending on the measurement geometry this influence can contribute from less than 0.01 % (e.g. at $\theta_t = 0^\circ$) to more than 5 % (e.g. at large θ_t angles where the sample's BTDF approaches zero) relative to the measurement results.

Instrument Function

The instrument function is the signature of the measuring facility, which depends on the applied A_i , A_D , A_M (only slightly), and l . It can be characterized by performing an angular scan without sample. The captured instrument function can be fitted by a modified Gaussian function as shown in equation 1.41

$$S = a + b \cdot \exp\left(-\frac{|\theta_t - c|}{d}\right)^e, \quad (1.41)$$

with parameters a being the background signal, b being the amplitude, c being the central detection angle, d indicating the full width at half maximum (FWHM) of the function, and e affecting the shape of the distribution, i.e., the width of the peak range. Different information can be extracted from such a fit, as examples shown in Figure 1.9. Four instrument functions are measured with different configurations of the three diaphragms, of which the diameters d_i , d_D , and d_M are shown in each diagram. Comparison between Figures 1.9a and 1.9b illustrates the influence of A_D , between 1.9a and 1.9c the influence of A_M , and between 1.9b and 1.9d the influence of A_i . As mentioned in the previous Subsection “Detection Angle Positioning”, its uncertainty can be estimated from the value of parameter c of the fit, which is listed in Table 1.5, along with the values of other fit parameters.

The FWHM of instrument function roughly represents the detection angular resolution. It influences the measured BTDF distribution in a convolutional way, i.e., the measured distribution is always a convolution of sample's true BTDF distribution and the instrument function. This is the influence of the finite extent that every measurement component has on the measurand, as theoretically considered in [150]. It broadens the width, flattens the slope, and decreases the peak of a true distribution. In principle, the real distribution can never be exactly obtained, but rather derived from the measured one by a deconvolution using, e.g., the method introduced in [151]. However, the existence of noise in the measurement data leads

1. Summary

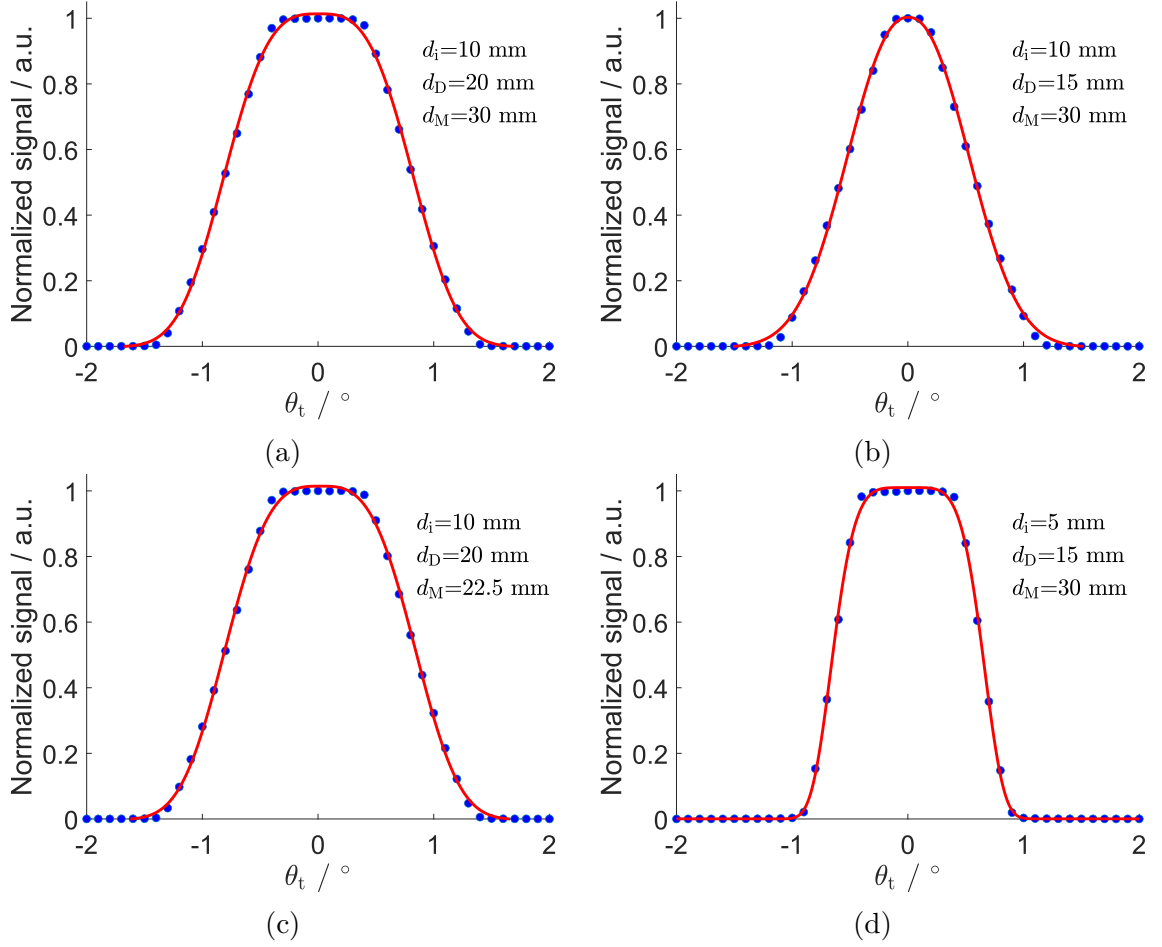


Figure 1.9: Normalized instrument functions of different configurations of the irradiated area A_i , the detection diaphragm area A_D , and the measurement area A_M . The diameters of these areas d_i , d_D , and d_M are given in each diagram.

Table 1.5: Fit parameters of instrument function measured with different configurations of the irradiated area A_i , the detection diaphragm area A_D , and the measurement area A_M .

Diaphragm configuration $d_i \mid d_D \mid d_M$ (in mm)	Fit parameters				
	a	b	c	d	e
10 20 30	-0.002	1.015	0.005	0.930	3.146
10 15 30	-0.003	1.006	0.003	0.689	2.233
10 20 22.5	-0.002	1.017	0.018	0.932	3.197
5 15 30	0.0004	1.010	-0.001	0.696	5.020

to significant difficulty in performing the deconvolution. One possible solution to this is to convolve a distribution similar to the measured one with the same instrument function and iteratively optimize the parameters of the first distribution until

the convolved one becomes identical to the measured one. Then the distribution to be convolved can be considered as the one deconvolved from the measured distribution, i.e., the true distribution. This method works well only when the measured distribution and the instrument function have similar shapes, e.g., Gaussian. And it requires quite some calculation time and is very sensitive to the parameter selection of the initially convolved distribution. Another approach introduced in [152] iteratively applies a combination of the Richardson-Lucy algorithm [153] and an early stopping rule based on an error function in a Monte Carlo ray tracing software for the deconvolution. This approach also requires quite some computation power and is limited to symmetric BTDF distribution. Since in the frame of this thesis there are also samples exhibiting far-off Gaussian characteristics, such as the engineered diffuser which has a square-shaped BTDF (sample A in the Papers), these methods cannot be applied properly.

It has been found that the deconvolution problem can be solved using a method similar to the one used to deconvolve the instrumental bandpass function out of the spectral measurement results, as introduced in CIE214:2014 [154]. In this method, the measured BTDF value is corrected by applying weighting factors, calculated from different orders of moments of the instrument function, on the neighboring measurement data points to calculate a weighted mean for that value. The calculation is straightforward and does not require large amount of integration and derivation. The method is only not easy to implement, when the angular step in the measurement data is not unified.

The influence of applied instrument function on the deviation from the real BTDF value is simulated in [150] and [155], as well as discussed regarding real measurement data in Paper III. Since the FWHM of instrument functions applied on PTB's new primary facility is relatively small compared to the characteristics of the studied samples, the influence of instrument function is considered as an uncertainty source in the analysis. For Lambertian samples this influence contributes no more than 0.01 % to the BTDF value, while for other samples, e.g., for engineered diffuser with square-shaped BTDF distribution this uncertainty ranges from 0.01 % (at the flat top) to more than 10 % (at the "bat-wings" as in Figure 5 of Paper I for the BTDF distribution of sample A measured at 642 nm at around $|\theta_t| = 8^\circ$ to 9°).

Temperature

The direct influence of ambient temperature on the BTDF measurement results is considered for the thermal expansion of the detection diaphragm, since this quantity

is included in the evaluation of BTDF. If the diaphragm is calibrated at a temperature different from the one applied in BTDF measurements, this deviation should be corrected for the results. For the measurements performed within the frame of this thesis, the applied diaphragms were calibrated under the same laboratory conditions. But still an uncertainty of 0.5 K is a reasonable estimate. Together with the thermal expansion coefficient of the diaphragm material, the additional uncertainty in A_D can be calculated. It turns out that this source contributes very limitedly to the combined uncertainty.

Wavelength Correction

For comparisons of the measured BTDF value, the wavelength has to be specified. For measurements that are not performed at this wavelength, corrections have to be made. As reported in Paper I and III, the angle-resolved spectral transmittance of the studied samples was measured using Cary 6000i, a commercial spectrophotometer, with a mini goniometer named UMA (Universal Measurement Accessory) for the determination of the wavelength correction factors. It has been observed that the spectral transmittance differs for detection polar angles. Therefore, a single correction factor is not sufficient for the BTDF measured at different θ_t . Instead, angle-dependent correction coefficients were calculated by taking the relation of spectral transmittance between two wavelengths for each θ_t . An example of these angle-dependent correction coefficients, applied to correct the BTDF of the narrow orientation of the holographic diffuser (sample D in the Papers) and of the ground glass diffuser (sample B in the Papers) from 642 nm to 633 nm, is depicted in Figures 1.10a and 1.10b, respectively.

The BTDF of these two samples exhibits Gaussian shape with similar FWHM, but the distribution of wavelength correction coefficients is quite distinctive. The correction applied to sample D will shift the measured values in different directions, i.e., lower the peak but raise the falling edge. In contrast, the correction for sample B will shift the values to the same direction with different amplitude. Nevertheless, the correction for both samples lead to a slight increase in the FWHM of the BTDF distribution, which was also observed in the results measured at different wavelengths.

Inter-reflections

Inter-reflection between optics and sample can cause the radiation to deviate from the ideal case that is used for calculating the measurand. Specifically, the inter-

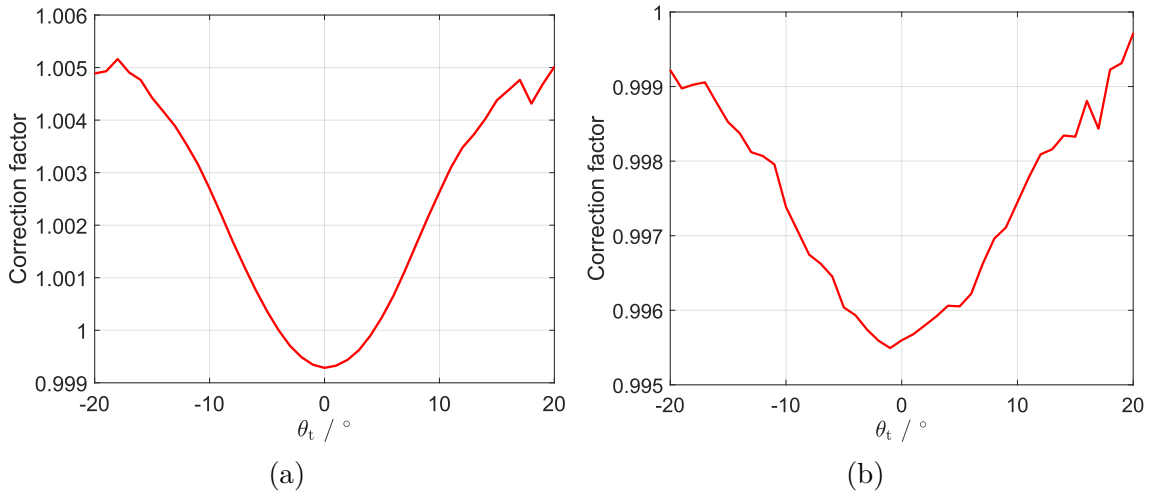


Figure 1.10: Angle-dependent coefficients applied to the wavelength correction from 642 nm to 633 nm for (a) the narrow orientation of the holographic diffuser (sample D) and (b) the ground glass diffuser (sample B).

reflections between the diaphragm for beam size A_i and the sample front surface as well as between the sample back surface and the detection diaphragm A_D need to be examined. The diaphragms used in BTDF measurements are either painted matte black or covered with black felt to minimize back reflection. Additionally, the diaphragm and sample are placed far from each other so that the back reflected radiation decreases quadratically with the propagating distance and eventually has negligible influence on the measurement result. For PTB's primary BTDF facility the distance is about 600 mm between diaphragm A_i and sample and 652 mm between sample and diaphragm A_D . This leads to a contribution smaller than 0.01 % relative to the measured BTDF, even for the case of Lambertian samples which reflect a great amount of radiation backward. Thus, this source is treated as uncertainty.

Sample Uniformity

In Paper III, the spatial nonuniformity of the sample under test was reported by one participant of the first intercomparison. However, the pilot study at PTB showed that deviation of results measured at different positions of the characterized samples could not be differentiated from the signal fluctuation. Thus, this uncertainty was generally neglected in the frame of this thesis. This should be held true for BTDF measurements under perpendicular irradiation, because the area around the sample's geometrical center is always irradiated and evaluated. But for non-perpendicular irradiation, this area on the sample front surface can vary depending on the choice

of reference plane. This means that in the under-irradiation scheme, the actual A_i may no longer be the geometrical center of the sample. This is illustrated in Figure 1.11a, when the sample back surface is chosen as reference plane. This convention also corresponds to all BTDF measurements performed in the frame of this work. Thus, for samples possessing spatial nonuniformity, change in the position of A_i will result in deviation of the measured BTDF value.

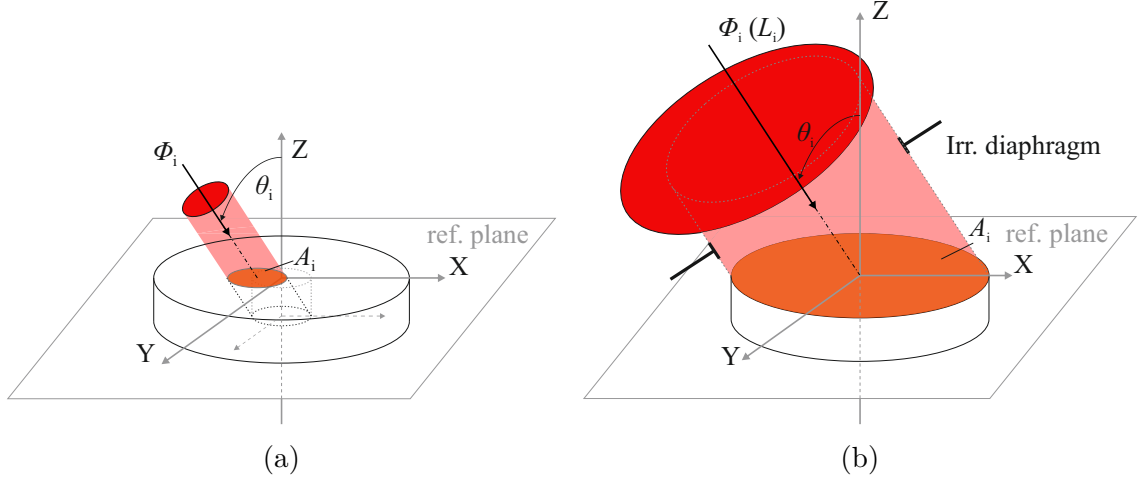


Figure 1.11: Influence of selecting sample back surface as the reference plane under non-perpendicular incidence. The sample nonuniformity should be accounted for in (a) the under-irradiation scheme due to deviation from the sample's geometrical center as incidence position. The beam nonuniformity should be taken into consideration in (b) the over-irradiation scheme since the beam part clipped by the irradiation diaphragm does not coincide with the central part of the irradiation beam.

Beam Uniformity

To estimate the influence regarding beam uniformity, the spatial intensity profile of the irradiation beam shown in Figure 1.5 can be treated as nearly flat-top with its edge at about 85 % irradiance level with respect to the center of the beam. This nonuniformity mainly influences measurements performed in the over-irradiation scheme, for which the measurement model (equation 1.33) assumes a perfectly uniform spatial intensity distribution for the irradiation beam. As shown in Table 1.3, S_i is always measured by moving the detector perpendicular to the incident beam, representing the L_i averaged over the A_M at $\theta_t = 0^\circ$. However, when evaluating the BTDF at other detection polar angles, the collected L_t is generated by L_i averaged over the corresponding A_M , which changes with θ_t as shown in Figure 1.8b. As a

result of nonuniform beam profile, the actual L_i that should be applied in the evaluation model differs from the one measured at $\theta_t = 0^\circ$ and this deviation should be corrected for.

Another influence of beam nonuniformity is related to the finite extent of applied measurement components. It introduces an additional weighting factor in the deconvolution of instrument function as discussed in the previous subsection. For nonuniform irradiance, the marginal part of beam does not contribute equally to the transmitted radiation as the central part. Using a self-written ray-tracing program, the influence of beam nonuniformity can be estimated as the difference between applying the actual beam profile and a perfectly uniform beam while also taking the BTDF of sample into consideration. In the ray-tracing method, the detection diaphragm is treated as infinitesimal since its size is much smaller compared to its distance to the sample, i.e., $d_D \ll l$. This approximation largely reduces the computation power and time needed for several dozens of measurement geometries. Compared to the correction in the previous paragraph, this influence is insignificant and treated as uncertainty.

An additional influence of beam nonuniformity has to be considered when the sample is irradiated at $\theta_i \neq 0^\circ$ and the reference plane is chosen as the back surface, as illustrated in Figure 1.11b. Unlike in the under-irradiation scheme, the irradiation area A_i can be adjusted to be centered about the geometrical center of the sample's front surface in the over-irradiation scheme by using an irradiation diaphragm. However, as indicated by the gray dotted circle within the incident beam, the clipped part does not coincide with the central part of the beam and any nonuniformity in the irradiation will contribute to the measurement uncertainty. Nevertheless, this uncertainty is easier to estimate compared to the one related to sample nonuniformity.

Azimuthal Misalignment

Two of the samples studied exhibit azimuthal dependence of their BTDF distributions. For them, uncertainty of the measurement results can be introduced by determination and realization of the sample's reference azimuth. In fact, this uncertainty source was identified as one of the three main causes for the inconsistent results in the BTDF intercomparison reported in Paper III. The influence of this uncertainty is illustrated by a simulation as shown in Figure 1.12. The reference azimuth is denoted as $\varphi_t = 0^\circ$ and for an obvious demonstration the misalignment is exaggerated to be 20° in this example. For both the holographic diffuser (sample D

in the Papers) and the engineered diffuser (sample A in the Papers), the deviation of the measured BTDF value at the misaligned azimuth is dependent on θ_t . And this deviation is estimated as the uncertainty regarding azimuthal misalignment. A 20° azimuthal misalignment would result in a maximum deviation of about 10 % for sample D and even larger deviation for sample A at the sharp falling edges of its BTDF distribution. Therefore, determination and realization of the sample's azimuthal orientation is of great importance in accurate BTDF measurements.

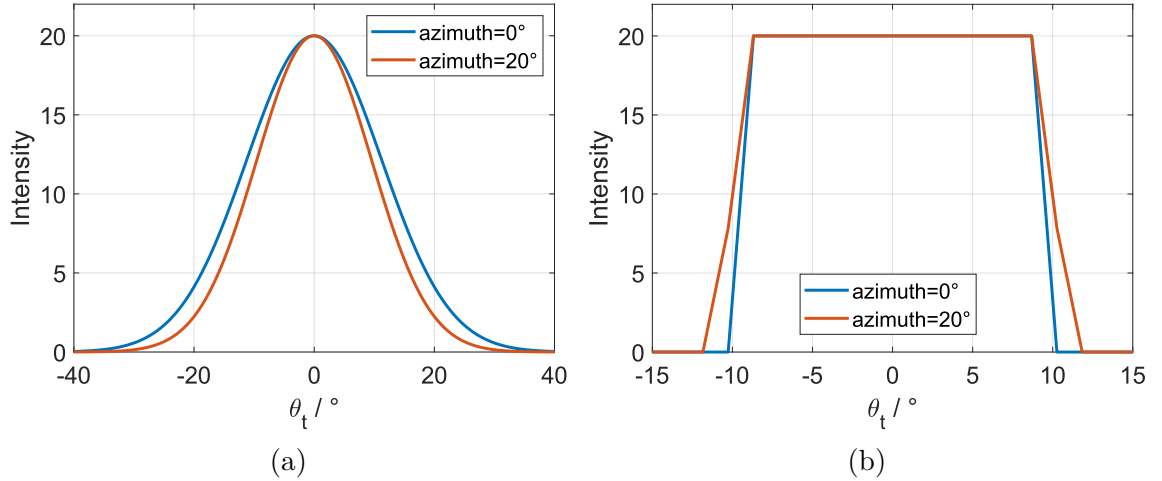


Figure 1.12: Influence of an azimuthal misalignment of $\varphi_t = 20^\circ$ on the measured BTDF for (a) the holographic diffuser (sample D) and (b) the engineered diffuser (sample A).

On PTB's primary BTDF facility, the reference azimuth can be determined using a high-resolution scientific camera placed on the rotation stage at $\theta_t = 15^\circ$ (cf. Figure 2(a) of Paper IV in Chapter 5). The camera is aligned parallel to the plane of angular scan. A target is moved 300 mm horizontally (corresponding to 1510 pixel coordinates) by the robot arm and the camera's vertical pixel coordinate remains unchanged. This indicates that the horizontal alignment of the camera is accurate to at least 0.038° . By placing a half transparent screen between sample and camera, the scattering pattern can be recorded while constantly changing the sample's azimuth (cf. Figure 3 of Paper III in Chapter 4). The methods used for determining the reference azimuth for the two involved samples are introduced in the following text.

For the holographic diffuser with an elliptical distribution, three different methods are used to determine the reference azimuth, which is the major axis of the ellipse. A schematic to explain the procedures applied in method 1 is depicted in Figure 1.13. Firstly, a Gaussian curve fit is applied to each row of the recorded scattering pattern to find the peak position and its corresponding horizontal (column) pixel coordinate.

Then, the azimuthal orientation of this scattering pattern is determined by applying a linear fit to the found peak positions of all rows. This determined orientation corresponds to a specific rotation angle of the sample holder. After that, the previous two steps are repeated for all recorded scattering patterns. At last, the relation between the determined orientations and the set sample holder rotations is fitted by a linear function to find the zero-crossing point, which indicates the set value of sample holder rotation for the reference azimuth $\varphi_t = 0^\circ$. For the example data using method 1 (Figure 1.13), the found set value of sample holder rotation is about 27.2° .

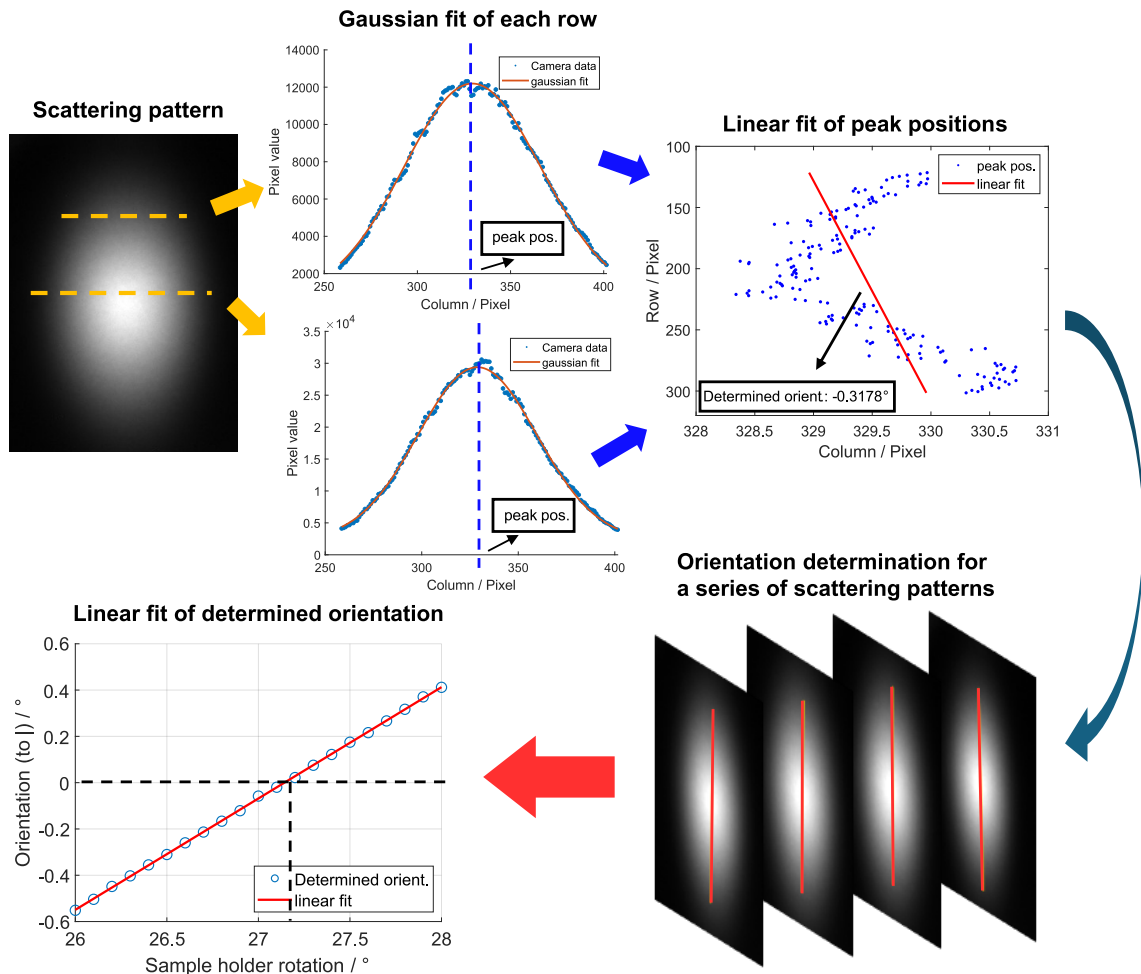


Figure 1.13: Schematic of method 1 to determine the reference azimuth of the holographic sample with elliptical scattering distribution. The photo used for determination is cropped to be slightly larger than the recorded scattering pattern.

Method 1 is time-consuming and heavily relies on the goodness of every fitting process. Thus, other methods are used as complement and validation for this method.

Method 2 utilizes least-square ellipse fitting on the boundary points of the binarized scattering pattern to determine the azimuth orientation [156]. The result using the same set of photos as in method 1 is shown in Figure 1.14. Similar to method 1, the set value of sample holder rotation for the reference azimuth is found by the zero-crossing point of the linear fit of the determined orientation for all recorded scattering patterns. For the same example data using method 2, the found set value of sample holder rotation is approximately 27.0° .

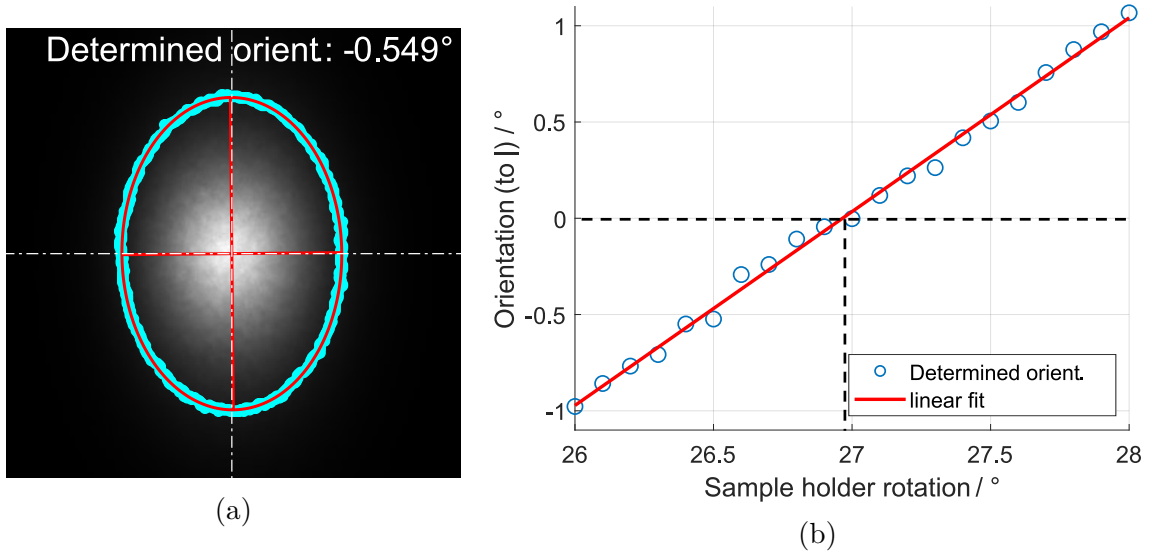


Figure 1.14: (a) Example of one recorded scattering pattern and (b) result of azimuthal orientation determination using method 2 for the holographic diffuser.

Method 3 finds the centroid as well as major and minor axes of an ellipse that has the same second central moments as the region of the binarized scattering pattern. The orientation is determined as the angle between the major axis of the found ellipse and the vertical pixel column passing through the found centroid of this ellipse. The result using the same set of photos as in methods 1 and 2 is demonstrated in Figure 1.15. Again, the set value of sample holder rotation for the reference azimuth is found by the zero-crossing point of the linear fit of the determined orientation for all recorded scattering patterns. In this example, the found set value of sample holder rotation lies between 26.9° and 27.0° .

Methods 2 and 3 are much more efficient compared to method 1, because they both treat the elliptical scattering pattern as a whole. However, the result using these two methods depends on the threshold applied to the binarization. An inappropriately chosen threshold leads to undesired neglect of boundary points of the scattering pattern. These boundary points are vital to the ellipse fitting as well as to the

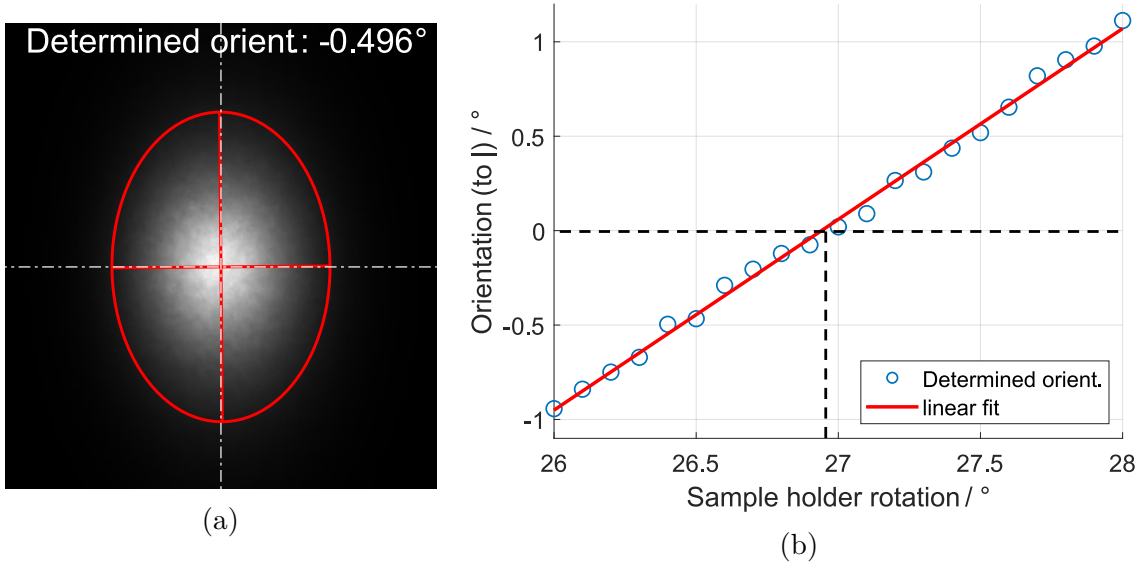


Figure 1.15: (a) Example of one recorded scattering pattern and (b) result of azimuthal orientation determination using method 3 for the holographic diffuser.

region determination. Change in the threshold results in a difference up to 0.5° in the found set value of sample holder rotation. A global image threshold using Otsu's method [157] is found to be not suitable for the scattering patterns of the holographic diffusers. Instead, the threshold is manually adjusted until no further change in the zero-crossing point is observed. Nevertheless, the result of azimuthal orientation determination using all three methods agrees well within about 0.2° .

For the engineered diffuser depicting a square-shaped distribution, the methods described above cannot be appropriately applied anymore. In fact, irregularities in the scattering pattern, such as the “bat-wings” mentioned in Paper III, the brighter central angular area and the rounded corners, make it almost impossible to mathematically determine the reference azimuthal orientation. Moreover, inter-sample-variation of these irregularities has also been observed, bringing more difficulties to the determination process. Thus, the azimuthal orientation of this kind of samples is manually determined using the method as shown in Figure 1.16. Each subfigure corresponds to the scattering pattern taken at a different azimuthal orientation: Figure 1.16b is the determined reference azimuth and Figures 1.16a and 1.16c show the case when the sample is rotated 1° from the reference azimuth counterclockwise and clockwise, respectively.

For each scattering pattern, the orientation is determined by drawing two lines parallel to the vertical edges of the square, and then measure the angle between

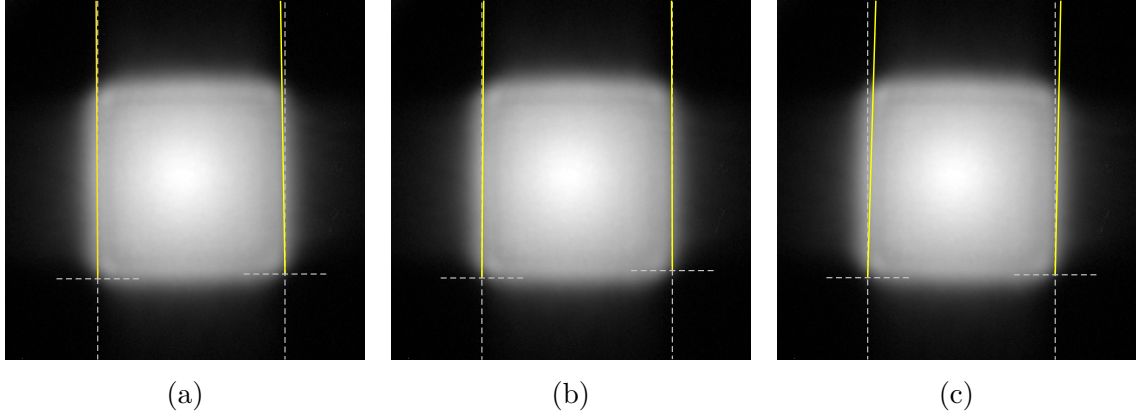


Figure 1.16: Determination of azimuthal orientation of the engineered diffuser: (a) 1° counterclockwise rotated from reference azimuth; (b) determined reference azimuth; (c) 1° clockwise rotated from reference azimuth.

the drawn lines (solid yellow) and the vertical direction (dashed light grey). The reason for using the vertical instead of horizontal direction as comparison reference is that the half transparent screen is fixed at the top with a weight at the bottom. Thus, due to the effect of gravity it is more likely that the screen is perpendicular to the optical path in the vertical than in the horizontal direction, in which possible distortion can be introduced in the photographed scattering pattern. Although this method is performed manually with less accuracy than applying a mathematical calculation, it can still be ascertained that the reference azimuth must lie between the two orientations shown in Figures 1.16a and 1.16c. Because in each of these two subfigures, the two yellow lines stay on the same side with respect to the vertical direction. Therefore, the standard deviation of the determined reference azimuth is considered as $\pm 1^\circ$ and should possess a rectangular probability distribution. This gives the standard uncertainty for the determination of $u_{\text{azi, engr.}}$ to be expressed as:

$$u_{\text{azi, engr.}} = \frac{1^\circ}{\sqrt{3}}. \quad (1.42)$$

To save experimental effort for future measurements, the reference azimuth is normally marked after being determined. In the frame of this thesis, samples are marked with a dot of about 0.5 mm diameter on the edge. For a sample with 2" diameter, the size of the dot can lead to a maximum uncertainty of about $\frac{0.5^\circ}{\sqrt{3}}$ in the realization of azimuthal orientation. For the engineered diffuser with only 1" diameter, this maximum uncertainty increases to $\frac{1^\circ}{\sqrt{3}}$. Using the simulation mentioned in the previous text of this subsection, the relative contribution of sample

azimuth misalignment to the measured BTDF value is estimated to be 0.01 % for the holographic diffuser and from 0.1 % to 1.5 % for the engineered diffuser.

Imaging Unsharpness

The uncertainty caused by unsharpness of the imaging detection system only contributes to the results measured in the over-irradiation scheme, because the area of evaluation is defined by the measurement area in this scheme. The reason for this is schematically illustrated in Figure 1.17. The sketch is adapted from Figure 1.6 and is exaggerated for a clear visualization. In both subfigures the red and purple solid lines represent the rays of incident and diffusely transmitted radiation, respectively, in the ideal case. The orange and dark blue dotted lines indicate the rays of incident and diffusely transmitted radiation, respectively, in the case of unsharp imaging.

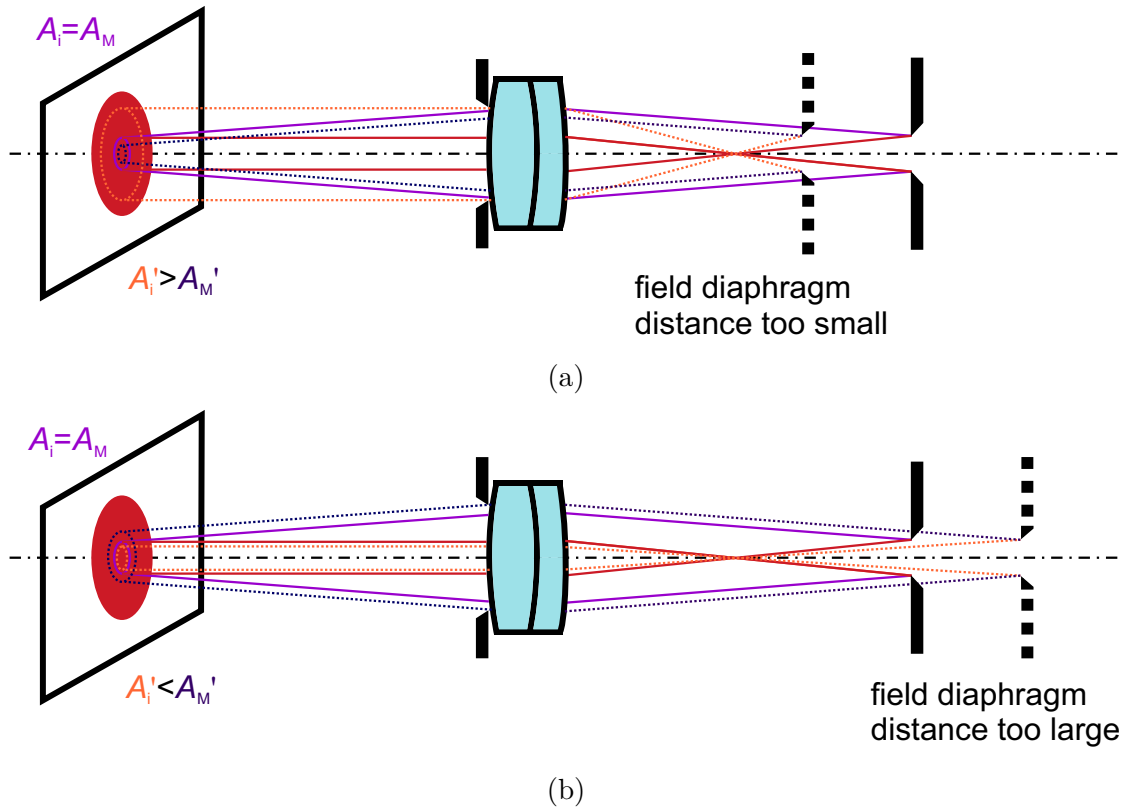


Figure 1.17: Optical path of the imaging detection system when the distance from the achromatic lens to the field diaphragm is (a) too small and (b) too large. Dotted lines indicate the optical rays in the case of unsharp imaging and solid lines the ideal case.

For the ideal case, the field diaphragm is placed at such a position, where the marginal rays of both incident and transmitted radiation coincide. Thus, the areas

for evaluating incident and transmitted radiation (A_i and A_M) have identical size, meaning that the detected transmitted radiance L_t is completely resulted from the detected incident radiance L_i . When the field diaphragm is positioned closer to the achromatic lens, as shown in Figure 1.17a, the size of A_i increases while A_M decreases. This results in under-evaluated L_t and over-evaluated L_i , i.e., a smaller BTDF value as expected. When the field diaphragm is placed farther, a large BTDF will be measured due to over-evaluated L_t and under-evaluated L_i , as shown in Figure 1.17b.

The accurate position for the field diaphragm is almost impossible to be determined only by distance measurement, since too many uncertainty sources need to be considered. One way to cope with this difficulty is to perform the alignment by means of BTDF measurement. For this purpose, the field diaphragm is mounted on a linear stage along with the integrating sphere. Using this linear stage the position of the field diaphragm can be adjusted with 0.1 mm accuracy along the optical axis. First, the BTDF of one sample at $\theta_t = 0^\circ$ is measured in the under-irradiation scheme. This value can be treated as a reference value since the under-irradiance scheme is not influenced by the imaging sharpness. Then, the same sample is measured at the same geometry in the over-irradiation scheme and the result is compared to the reference value. The longitudinal position of the field diaphragm can be gradually adjusted until the difference between the two values stays within the Type A measurement uncertainty. Finally, this set alignment can be validated by performing BTDF measurements of other samples exhibiting different scattering characteristics.

However, the position of field diaphragm is not unanimous for different irradiation wavelengths, even if an achromatic lens is used. One might estimate the maximum uncertainty regarding this issue and apply it for the entire evaluated spectral range. But in order to perform accurate measurement, the chromatic aberration requires new determination of this longitudinal position each time when the wavelength is changed. Either the procedures described in the previous paragraph should be performed for each applied wavelength, or the achromatic lens needs to be replaced by an apochromatic or a superachromatic lens or even a spherical mirror that does not introduce chromatic aberration but rather polarization-dependent issues. Since the standard Type A uncertainty for BTDF measurement using PTB's primary facility is normally smaller than 0.25 %, the uncertainty of imaging unsharpness of the detection system is also estimated to be 0.25 % relative to the BTDF value.

1.3.3 Lateral Diffusion

While measuring the BTDF of thick volume diffusers, it has been observed that the transmitted beam on the sample back surface becomes much larger than the incident beam, as a result of strong lateral scattering inside the material. This effect leads to a loss in collecting the transmitted radiance L_t using the under-irradiation measurement scheme (cf. Figure 8(a) in Paper IV). This is the reason identified in Paper III for the inconsistent comparison results of sample C, because all participating facilities performed measurements in the under-irradiation scheme, resulting in varying measurement results depending on the applied measurement parameters. This phenomenon should be treated as an extra uncertainty source in BTDF measurements, especially in scale comparisons, because most of the facilities perform BTDF measurements in the under-irradiation scheme according to Table 1.1.

However, as reported in Paper IV, further study on this type of samples reveals that even when they are measured in the over-irradiation scheme, the results still exhibit a dependence on the area size of the incident beam (cf. Figure 10 in Paper IV), i.e., L_t increases when A_i becomes larger while the irradiance of the incident spot E_i remains constant. This contradicts the definition of BTDF, in which the incident area A_i is not accounted for. A possible explanation for this is that, as A_i increases, a part of the increased incident flux Φ_i can contribute to the transmitted radiance L_t due to the strong lateral scattering, even when the measurement area A_M is laterally far away from the position of this part of incident beam (cf. Figure 11 in Paper IV). This also explains the shape of the empirical function (equation (7) in Paper IV) found to fit this dependence as shown in Figure 12 in Paper IV. The curve has a steep rise at small A_i and gradually approaches a saturation value when A_i becomes very large, because the closer the increased part of A_i to the lateral position of A_M , the more Φ_i can be laterally scattered into A_M . Therefore, an additional term relating L_t and Φ_i , which is the BBSS as introduced in Section 1.1.2, should be included in the BTDF measurement of translucent materials.

The empirical function has three free parameters which need to be associated with physical meaning. By considering the boundary conditions of this function, one can easily see that parameter a represents the saturation value at very large A_i , which can be related to the BTDF when the sample is irradiated with a “large enough” beam, as stated in the definition. On the other hand, the empirical function depicts a linear dependence on A_i with the slope $a \cdot b$ when A_i is very small. From equation 1.15 this product of parameter a and parameter b can be related to the BBSS. There remains a third parameter c , which is a dimensionless parameter and

might represent the magnitude of volume scattering of the material (e.g. in the way of “mean free path” of the scattering). However, the goodness of the fit and the uncertainty of these parameters (especially b and c) still need to be improved. Moreover, this empirical function should be generalized to other non-perpendicular incidence and transmission angles.

Thus, proposals have been made at the end of Paper IV and future work is planned. Samples with different physical thicknesses, different concentrations and sizes of scattering centers will be measured in different geometries. The aim is to provide sufficient information as well as recommendation for accurate BTDF measurements of all types of diffusers, and maybe even refine the definition of BTDF similar as the case of BRDF in [42], in which A_i must be included as an additional parameter when the irradiated area cannot be regarded as “large enough” with respect to material characteristics.

2 Paper I:

Preliminary Results of Angle-Resolved BTDF Characterization of Optical Transmissive Diffusers

Paper Summary

This paper primarily introduces the modified NaNoRef setup participated in the interlaboratory comparison, reported in Chapter 4, along with details of the investigated samples and preliminary results of the samples' BTDF distributions. The angle-dependent spectral transmittance is also discussed in Section 4 of the paper, together with its modeling, as introduced in Section 1.1.3, and its influence on a possible wavelength correction of the measured results, as introduced in the Subsection "Wavelength Correction" in Section 1.3.2. At the end of this paper, a simplified model for analyzing the uncertainty of BTDF measurements is elaborated with a preliminary uncertainty budget.

https://ceur-ws.org/Vol-3271/Paper13_CVCS2022.pdf [158]

Preliminary Results of Angle-Resolved BTDF Characterization of Optical Transmissive Diffusers

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Abstract

Optical diffusers are used in many application areas related to illumination. With the purpose of characterizing the transmission properties of optical diffusers, we present preliminary results of BTDF (Bidirectional Transmittance Distribution Function) measurement with high angular resolution at two different wavelengths. The expanded total uncertainty of the measurement is estimated to be 0.5% in the best case. In this way, the scattering distribution of optical diffusers could be quantitatively described by their BTDF values with high precision, good repeatability and reproducibility. The diffusers' angle-dependent spectral transmittances in the VIS (from 380 nm to 780 nm in the electromagnetic spectrum) are also measured using a commercially available spectrophotometer. Results are compared to simulation calculated from a simple model and a first-step explanation for the sample spectral dependency is proposed.

Keywords

Optical Diffuser, Diffuse Transmission, BTDF, Angle-resolved Detection, Wavelength Dependency

1. Introduction

When light impinges on a rough surface or propagates through a thin layer with non-uniformities in the material, it will deviate from its original trajectory and be scattered. Depending on the type of interaction, in its extremes the reflection and transmission of light are distinguished and described as either specular or diffuse. In most cases however, the situation is a mixture of these two components. Optical diffuse transmission happens almost everywhere in our daily life and people have been constantly making great use of it. In the field of satellite-based Earth remote sensing, for example, optical transmissive diffusers are used for the calibration of the on-orbit spectral radiance [1, 2]. In illumination design, diffusers are integrated in the optical system to control the distribution of transmitted light [3, 4]. In computer graphics rendering, “diffusers” are also used in different scenarios to simulate the transfer of light through thin objects in a realistic way [5, 6, 7, 8].

Characterization of diffuse transmission is of great importance, because it provides important information on the visual appearance of an object. A quantitative characterization of optical diffusers would be beneficial to both industrial and metrological aspects. Within the JRP EMPIR project *BxDiff* (New quantities for the measurement of appearance) [9], different types of measurands (BRDF, BTDF, BSSRDF) are determined to lay a foundation for better understanding the characterization of the visual appearance of objects. In this paper, preliminary results of the angle-resolved measurement on the BTDF of five different types of diffusers are presented. Measurements were carried out at PTB (Physikalisch-Technische Bundesanstalt), the national metrology institute in Germany. The combined

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total uncertainty depends on the scattering angle as well as the characteristics of the sample and is estimated to be around 0.5% in the best case. The spectral transmittance at different scattering angles is also measured on another setup and compared to the first-step simulation result. A possible explanation for the wavelength behavior is also proposed in the paper.

2. Method

2.1. Bidirectional Transmittance Distribution Function (BTDF)

To fully characterize diffusely scattered light, the BRDF (Bidirectional Reflectance Distribution Function) was first introduced by Nicodemus [10, 11]. Bartell et al. [12] then extended this concept into the field of transmission by introducing an analogous term called BTDF (Bidirectional Transmittance Distribution Function). As illustrated in Figure 1(a), a thin sample with flat surface element dA_i possesses uniform and isotropic scattering properties. Its top surface is uniformly irradiated by a differential element of irradiance dE_i from a spatial direction described by (θ_i, ϕ_i) . The BTDF is then defined as the ratio of the scattered transmitted radiance dL_t in another specific direction (θ_t, ϕ_t) , which is in the transmittance hemisphere of the sample, to the incident irradiance within dA_i :

$$\text{BTDF}(\theta_i, \phi_i; \theta_t, \phi_t) = \frac{dL_t(\theta_t, \phi_t)}{dE_i(\theta_i, \phi_i)}, \quad (1)$$

where θ is the polar angle with respect to the normal of the top surface of the sample (positive direction of the z-axis in Figure 1) and ϕ is the azimuth angle within the sample surface plane. The subscripts “i” and “t” designate incident and transmitted radiation, respectively.

By using a differential expression, the BTDF purely describes sample properties as a geometric distribution. It characterizes the transmitting properties of one point on the sample in one specific direction, with contributions from the entire incident radiant flux confined within a certain solid angle. It takes positive values with the unit per steradian (sr^{-1}) [11]. Besides its geometrical sense, the BTDF is also related to parameters such as wavelength and polarization of the incident radiation. Thus, the BTDF is typically expressed as unpolarized values at a certain wavelength.

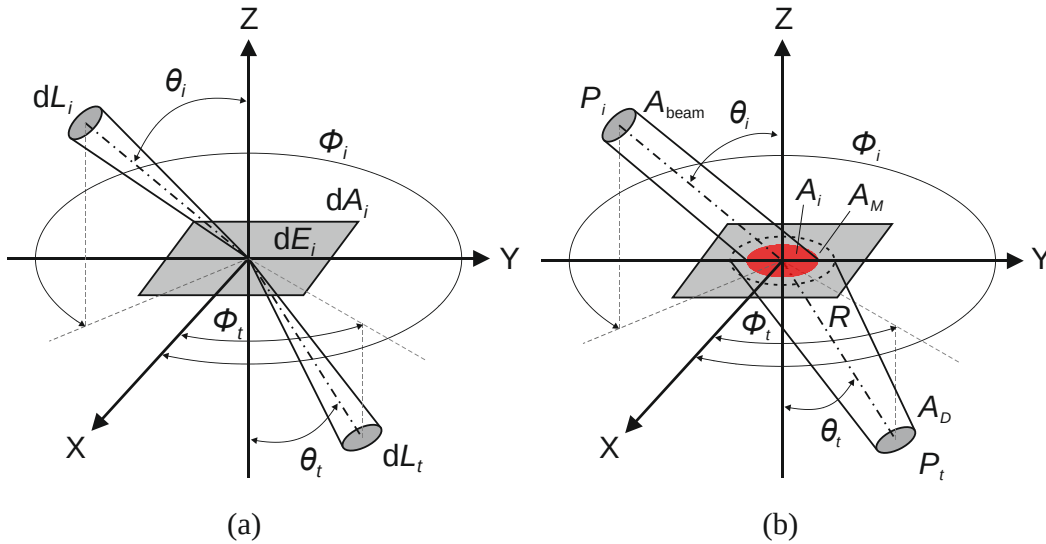


Figure 1: Geometry of BTDF: (a) general definition and (b) measurement

In practice, however, measurements cannot be realized with infinitesimal quantities. Stover [13] suggested the measurement conditions to be modified so that measurements are performed with finite resolution, which is defined by the aperture size of the detection system. It is usually larger than the irradiation spot of the incident beam and large enough for a sufficiently good signal-to-noise level, but in the meantime as small as possible to minimize the influence from the instrument itself on the measured values, which is the convolution of the real BTDF of the sample and the instrument signature. Since a real sample cannot be perfectly isotropic and uniform, measurements of BTDF should be

considered as an average over a surface area which is much larger than the size of the statistical features of the sample [12]. As depicted in Figure 1(b), a sample is irradiated by a collimated beam with finite beam size A_{beam} and its projection on the sample surface is then

$$A_i = A_{\text{beam}} / \cos \theta_i. \quad (2)$$

The average incident irradiance E_i within A_i on the sample surface takes

$$E_i = \frac{P_i}{A_i}, \quad (3)$$

with P_i the incident optical power measured when no sample is placed in the beam path. The average transmitted radiance L_t is the scattered optical power P_t detected on the other side of the sample within A_i through the detector solid angle Ω_t :

$$L_t = \frac{P_t}{A_i} \cdot \frac{1}{\Omega_t \cos \theta_t}, \quad (4)$$

where Ω_t is determined by the detector aperture size A_D and the distance R between the detector aperture and the sample back surface (front surface is the irradiated side):

$$\Omega_t = \frac{A_D}{R^2}, \quad (5)$$

and the product of Ω_t and $\cos \theta_t$ is the projected solid angle at scattering angle θ_t . Combining equations (2) to (5), we obtain the form for evaluating the BTDF from measured signals, when A_i is relatively small and the detector's field of view is constantly underfilled [13, 14]:

$$\text{BTDF} = \frac{L_t}{E_i} = \frac{P_t}{P_i} \cdot \frac{R^2}{A_D \cos \theta_t}. \quad (6)$$

2.2. Experiment Setup

We measure absolute values of BTDFs of the different scattering transmissive samples in BxDiff using the NaNoRef setup at PTB, which is originally the national reference setup for measurements of specular reflection [15]. With proper modifications to hardware and the control program, measurements of diffuse transmission could also be performed with modest uncertainty.

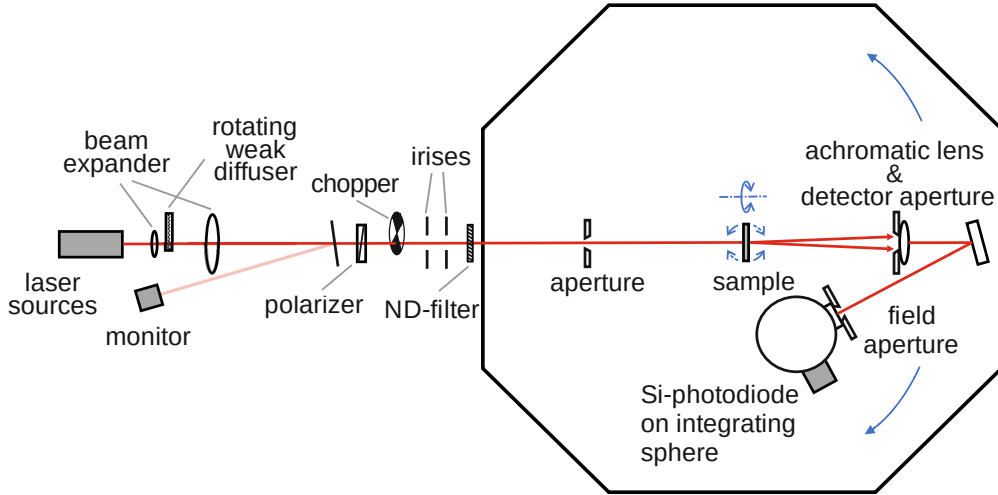


Figure 2: Sketch of BTDF setup (modified NaNoRef) at PTB, top view

As illustrated in Figure 2, the central part of the goniometric setup is an octagonal chamber, in which the sample and the detection system are enclosed to eliminate stray light from the light source and ambient light. The sample is placed at the center of this chamber and is irradiated by a collimated beam whose size is restricted by an interchangeable aperture in front of it. The sample holder is a vertical motorized rotary stage which could rotate around its azimuth (optical axis) freely, so that any desired sample azimuth could be achieved. The rotary stage is mounted on a motorized three-axes displacement unit to control the position of the irradiation spot on the sample and to adjust the longitudinal position of the sample back surface on the optical axis. The displacement unit is fixed on a horizontal rotary

table, which rotates around the central (vertical) axis of the octagon. This rotation axis is perpendicular to the optical axis, and the intersection point of these two axes lies exactly on the sample back surface. In this way the polar angle of the incident radiation onto the sample could be controlled. The detection system is mounted on another horizontal rotary arm, which is concentric to the sample rotation table. Therefore, both the table and the arm rotate within the same plane around the same axis, which is referred to as the detection axis.

In the detection system, the detector aperture is interchangeable to different diameters to realize detection with different angular resolutions and detection solid angles. Directly behind the detector aperture, an achromatic lens determines the size of the measurement area A_M (Figure 1(b)) by means of a field aperture, which is placed at the entrance port of an integrating sphere. A calibrated Si-photodiode is mounted on the detection port of it. The use of an integrating sphere homogenizes the radiation for detection and reduces speckle effects as well as polarization-related sensitivity of the detector. All these components of the detection system are fixed on the detection rotary arm and move altogether during detection. The optical path is folded with an angle as small as possible by a planar mirror due to limited space inside the chamber. By alternating the size of the field aperture, the size of the measurement area on the sample back surface could be adjusted. The parameters regarding measurement conditions are listed in Table 1.

Table 1
General conditions of BTDF measurement

Parameter	Notation	Value
Wavelength	λ	642 nm, 445 nm
Azimuth angle of incidence	ϕ_i	0° to 360°
Azimuth angle of detection	ϕ_t	0° to 360°
Polar angle of incidence	θ_i	0°
Polar angle of detection	θ_t	-25° to 80°
Irradiation beam size	A_{beam}	9.6 / 29.2 / 44.2 / 80.1 mm ²
Detector aperture area	A_D	19.6 / 81.7 / 156.1 mm ²
Distance sample to detector	R	494.5 mm
Measurement area on sample (at $\theta_t = 0^\circ$)	A_M	50.3 / 75.4 / 265.9 mm ²

Outside of the chamber are the light sources and some beam controlling optics. Two laser sources are used for the measurement, one is a fiber-coupled diode laser at 642 nm and another one is a diode-pumped solid-state laser at 445 nm. To reduce speckle effects, a rotating weak diffuser is placed in the vicinity of the intermediary focus of a beam expander. In this way, the laser beam is expanded to be able to supply varying diameters of the irradiation spot on the sample. Moreover, the coherence of the laser radiation could in this way be reduced and the beam profile is homogeneous after collimation. Polarization of the radiation is controlled by using a half-wave plate and a linear polarizer. Measurements are performed in *s*- and *p*-polarized radiation respectively, with unpolarized detection and then the unpolarized BTDF value was calculated by the mean value out of the two measurement results. The measurement is performed using the lock-in technique to suppress any unwanted optical signals that are not at the chopping frequency. As the BTDF values of different samples vary largely, neutral-density filters are used to bring the detected power to an appropriate level, reducing the influence of electronic noise and improving SNR. The two irises prevent stray light from entering the measurement chamber and filter out the undesired outer parts of the expanded beam. The monitor detector is used to correct for fluctuation in the laser output power.

For an absolute measurement, the sample was firstly moved out of the optical path. The ND-filter was set in and the incident power P_i was measured at $\theta_t = 0^\circ$. Then, the sample was moved into the optical path with its geometric center irradiated. The sample was aligned to have $\theta_i = 0^\circ$ and its azimuth ϕ_i (or ϕ_t since measurements were all in-plane) was either predefined for rotationally symmetrical samples or predetermined for azimuthally sensitive samples by evaluating the intensity distribution on a screen using a high-resolution camera, which will be introduced in more detail in the next section. A well-defined azimuthal orientation of the sample is of great importance when measured BTDF values

are compared for samples with dedicated azimuthal dependence. The angle-resolved detection of the sample scattering distribution was then carried out by rotating the detector arm to each scatter angle θ_t . The laser beam was not attenuated by the ND-filter when the transmitted scatter signal was measured.

2.3. Samples under study

Five types of samples are characterized. Two of them are quasi-Lambertian diffusers with relatively constant BTDF in a large range of scatter angles θ_t . One of them, denoted as HOD-500, is a diffuser made of high purity fused silica (HOD[®]) manufactured by Heraeus Quarzglas GmbH [16]. It has 50 mm in diameter and 2 mm in thickness and there are micro air bubbles with diameter less than 20 μm uniformly distributed in the bulk volume. Another quasi-Lambertian sample, denoted as Zenith-250, is a 0.25 mm thick foil made of Zenith Polymer[®], a high-reflecting material using sintered PTFE powder manufactured by SphereOptics GmbH [17]. It is clamped between two plates with a clear aperture of 45 mm in the center. Types of Mie scattering volume diffuser made of synthetic fused silica, which are similar to HOD-500 [18, 19, 20] and Zenith-250 [20], have been proven to be good near-to-Lambertian diffusers.

The scattering characteristics of the other three types of samples are far away from a Lambertian characteristic. The first one, denoted as DG20-220, is a rotationally symmetrical ground glass diffuser distributed by Thorlabs, Inc [21]. It has 2 inches in diameter and 2 mm in thickness with one side treated using 220 grit polish. Thus, this side of the sample is regarded as the active surface and is irradiated in the measurement. Light is scattered mainly by the surface of this type of sample, leading to a Gaussian-shaped distribution. The second type of sample, denoted as E28-14, is a holographic diffuser provided by Temicon GmbH [22]. It consists of a 125 μm thick imprinted PET foil on standard glass substrate with 2 mm thickness and it has 50 mm edge length. This type of sample has a two-dimensional Gaussian-shaped scattering characteristic, meaning that the FWHM of the scattering distribution is different when varying sample azimuth. The last type among these three, denoted as ED1-S20, is also distributed by Thorlabs, Inc. It is called an Engineered Diffuser[™] with 1 inch in diameter and 1.5 mm in thickness. It has a square-shaped scattering distribution and is thus also azimuthally sensitive. The scattering characteristic can be attributed to the individually specified microlens-units on the surface produced by direct laser writing. This type of diffuser is normally used in applications related to beam shaping [23].

For both holographic and engineered diffusers, determination of their azimuthal orientation prior to the measurement must be performed. An air-cooled CCD-camera with 1600 pixels $\times$ 1200 pixels resolution was placed towards the back surface of the sample and was horizontally aligned. A half-transparent diffuse screen was placed between the camera and the sample and adjusted to be perpendicular to the beam direction. The sample's scattering distribution falling on the screen could then be photographed by the camera from behind the screen. A series of photos was taken when the azimuth was constantly varied with an interval of 0.1° by the rotary stage holding the sample. The transmitted scattered patterns were then evaluated by different methods and comparable results with an uncertainty of 0.2° regarding the sample azimuthal orientation could be achieved. For the holographic diffuser, its cross section imaged on the screen showed an elliptical form and its $\phi_i = 0^\circ$ orientation was defined when the major axis of the ellipse was parallel to the detection plane. For the engineered diffuser, the $\phi_i = 0^\circ$ orientation was given when one edge of the square-shaped pattern was parallel to the detection plane. The so found orientations were marked by a small dot on the edge of the diffusers for later performed measurements.

3. Results

3.1. Quasi-Lambertian Diffusers

The measurement results of HOD-500 and Zenith-250 are illustrated in Figure 3(a). Due to the physical limitation on the detection range, results in the negative angle part smaller than -25° are extended by measuring the sample at $\phi_i = 180^\circ$ in the positive angle range greater than 25° , where the

same value ought to be obtained with an ideal measurement setup. This method was also used in the measurement of other samples, when the desired detection range was beyond the limitation.

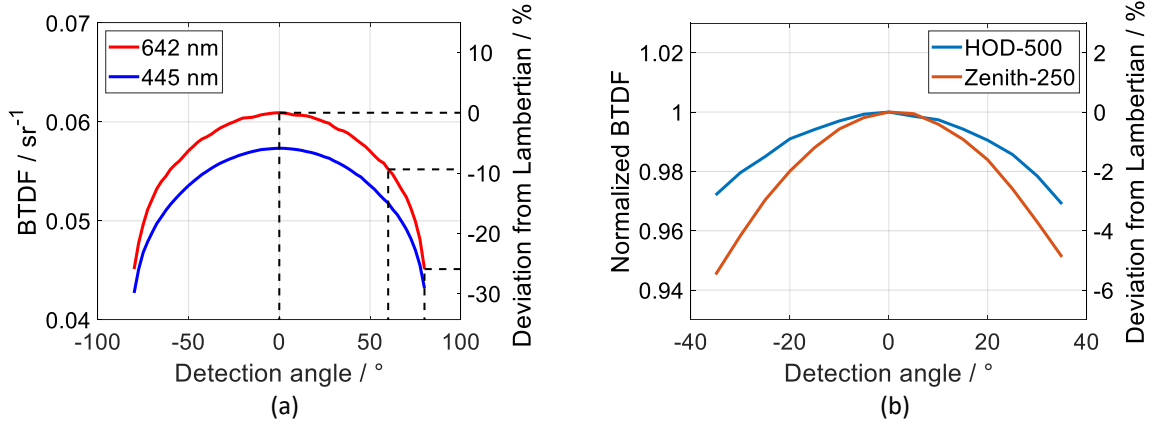


Figure 3: (a) BTDF of HOD-500 at 642 nm and 445 nm, $\theta_i = 0^\circ$, θ_t varies from -80° to 80° ; (b) Normalized BTDF of HOD-500 and Zenith-250, $\theta_i = 0^\circ$, θ_t varies from -35° to 35°

For the sake of brevity only the unpolarized BTDF values of HOD-500 are shown here. Measurement showed similar quasi-Lambertian scattering distributions at both wavelengths, with peak BTDF values of approx. 0.057 sr^{-1} and 0.061 sr^{-1} , respectively. The smaller amplitude at 445 nm could be explained by a decrease in the transmittance due to increasing scattering by a larger refractive index at shorter wavelength and a higher absorption as well. The right axis of the plot corresponds to the curve of 642 nm and shows the angle-dependent deviation from a perfect Lambertian diffuser in percentage. At 60° the BTDF drops less than 10% and at 80° approx. 25%, indicating that light is scattered uniformly by HOD-500 within a fairly large range of angles.

The normalized BTDF values of both quasi-Lambertian diffusers are plotted together in Figure 3(b) for an intuitive sample-type comparison. Within a small range of angles from -35° to 35° the discrepancy between the two diffusers already becomes significant. The BTDF of Zenith-250 decreases faster than that of HOD-500 and is thus less “Lambertian”. This could be attributed to the different types and sizes of scattering centers in the bulk volume of sample material.

3.2. Ground Glass Diffusers and Holographic Diffuser

The measured BTDF values for DG20-220 with fitted curves are shown in Figure 4(a) for 642 nm and 445 nm. Data can be fitted with a Gauss-Lorentz model. Compared to the quasi-Lambertian diffusers this ground glass diffuser has a sharp peak and the distribution is restricted in a small angle area. The FWHM (Full-Width at Half Maximum) are 15.9° at 642 nm and 16.5° at 445 nm. The slightly broader distribution at shorter wavelength might be attributed to enhanced multiple in-surface scattering, which will be discussed in more detail in Section 4.1.

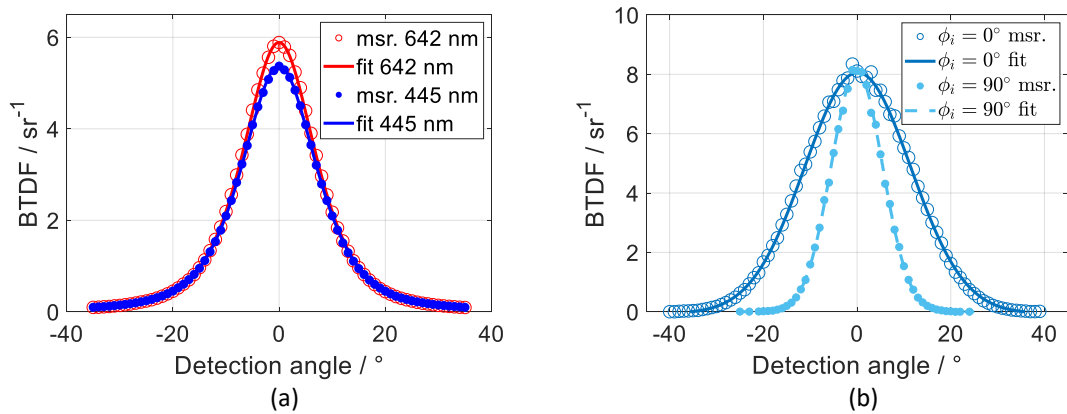


Figure 4: BTDF of (a) DG20-220 at 642 nm and 445 nm, $\theta_i = 0^\circ$, θ_t varies from -35° to 35° ; (b) E28-14 at 642 nm, ϕ_i at 0° and 90° , $\theta_i = 0^\circ$, θ_t varies from -40° to 40°

The holographic diffuser E28-14 also has a Gaussian-like scattering characteristic, but the distribution is dependent on its azimuthal orientation. In Figure 4(b), the measured BTDF values with fitted curves at 642 nm at 0° and 90° azimuths are demonstrated. The fit uses the regular Gaussian model. The FWHM values are 26.7° at $\phi_i = 0^\circ$ and 12.9° at $\phi_i = 90^\circ$, indicating that by changing the sample azimuthal orientation of 90° the width of the scatter decreases rapidly to about half of its starting value. Near the peak of the curve at 0° azimuth strong fluctuation in the BTDF values is observed, which might be the effect of laser speckle. The wavelength dependency of E28-14 is similar to that of DG20-220. The peak value is smaller and the FWHMs at both azimuths are slightly larger at 445 nm (not shown).

3.3. Engineered Diffusers

BTDF values of the engineered diffuser ED1-S20 at both wavelengths and at both azimuths are illustrated in Figure 5. Its scattering distribution is much different from the other diffusers. From about -7° to 7° the scatter remains almost unchanged, as indicated by the flat top in the curves and then drops rapidly within approx. 3° from peak value to close to zero. Although the scatter pattern is square-shaped, there are still slight differences which could be observed from the angle-resolved measurement at different azimuthal orientations. The wavelength dependency is on one hand similar to that of the surface scattering diffusers. On the other hand, at the two “horns” of the curve the scatter behaves quite distinctively at different wavelengths. This might be attributed to the special microlens structure on the sample surface, whose interaction with light changes with varying wavelength.

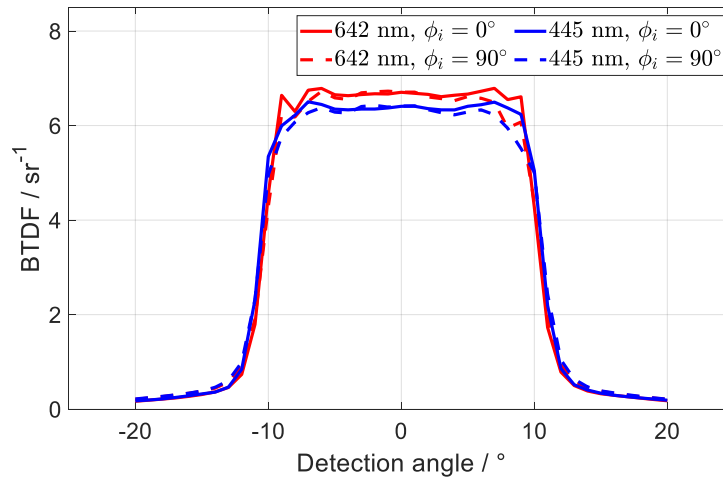


Figure 5: BTDF of ED1-S20 at 642 nm and 445 nm, ϕ_i at 0° and 90°, $\theta_i = 0^\circ$, θ_t varies from -20° to 20°

4. Discussion

4.1. Wavelength Dependency

For a good comparison between different participants within BxDiff, results are to be compared at a certain wavelength. A quasi-standard wavelength is that of the red HeNe-laser: 632.8 nm. For results that are not measured at the prescribed wavelength, a proper correction considering the wavelength-dependent sample properties should be performed. For this purpose, the angle-dependent spectral transmittance of a set of samples was measured on an Agilent Cary 6000i, a commercially available spectrophotometer with Universal Measurement Accessory (UMA) mini-goniometer.

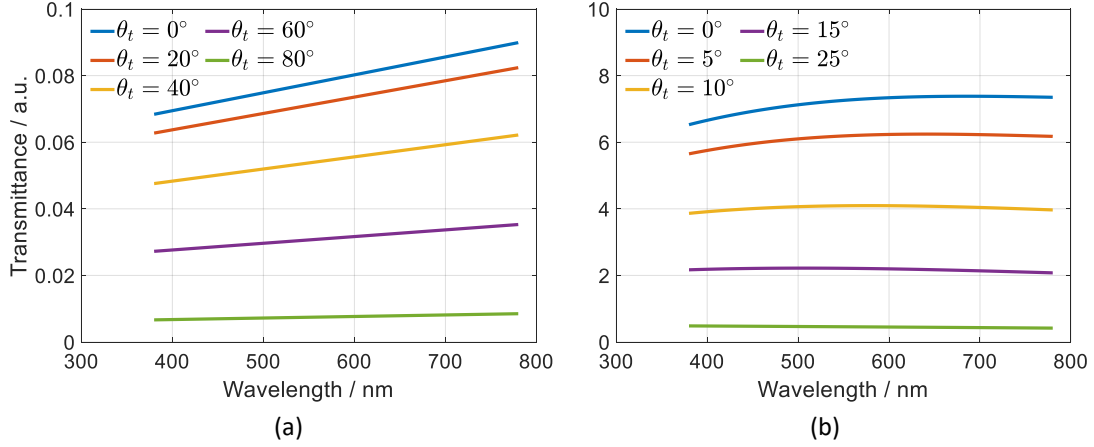


Figure 6: Spectral transmittance of (a) Zenith-250 and (b) E28-14 ($\phi_i = 0^\circ$) at different θ_t

In Figure 6(a), the diffuse transmittance into a solid angle of 0.0089 sr of Zenith-250 at different scattering angles is plotted as a function of wavelength. For bulk diffusers, the transmittance is in general fairly proportional to the wavelength. The variation rate or slope of the curve varies with different θ_t . At $\theta_t = 0^\circ$, corresponding to the central peak in the angle-dependent scattering distribution, the BTDF value changes the most with increasing wavelength, whereas at grazing angle $\theta_t = 80^\circ$ the BTDF becomes almost constant and show little spectral dependency. The wavelength dependency is different for surface scattering types. Spectral transmittance of E28-14 is given as an example in Figure 6(b). The spectral transmittance in the central peak of the angular scattering distribution rises with wavelength but flattens at around 600 nm and starts to decrease at even higher wavelengths. The wavelength of the maximum of spectral transmittance $\lambda_{\max}$ also varies with different θ_t . This is observed more clearly by the differential spectral transmittance in Figure 7(a), where the derivatives of transmittance with respect to wavelength are plotted. Interestingly, the wavelength dependency becomes completely “reversed” at larger θ_t . Instead of a positive slope, transmittance decreases constantly with increasing wavelength in the whole VIS. As a result of this wavelength dependency, not only the BTDF value but also the shape of the angle-resolved scattering distribution would be modified by the wavelength correction. For example, correction from a larger wavelength to a smaller one would cause the peak of the distribution to drop while at the falling edges the BTDF would be lifted, resulting in a larger FWHM. This also agrees with the comparison between measured curves of the surface scattering samples at different wavelengths.

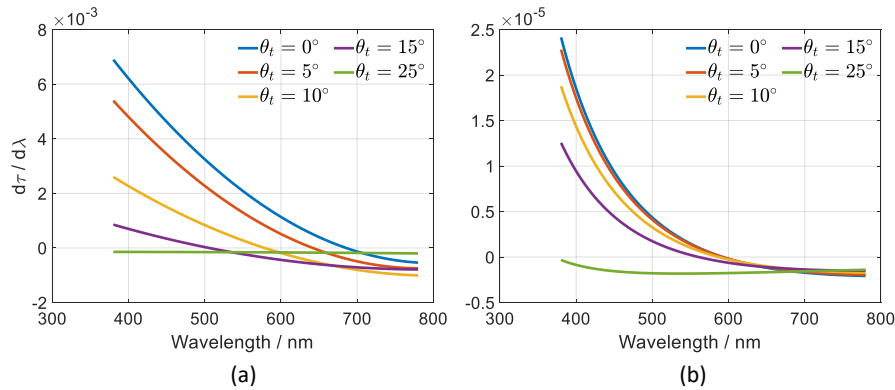


Figure 7: Differential spectral transmittance of (a) E28-14 ($\phi_i = 0^\circ$) measured at different θ_t and (b) simulation using a single scattering model

Scattering effects can likely explain this behavior. We performed a preliminary comparison to simulation results using a single scattering model where a surface scattering sample consists of an active top surface with microfacets and small scattering particles on a smooth transparent substrate. The microfacets obey a normal distribution determined by the surface roughness (standard deviation in height). The calculation is based on Rayleigh scattering where the wavelength dependence is modified

according to particle size by an exponent in $[0.0925, 1]$ (smaller exponent for larger particles). This method has been found to fit reasonably well with the Lorenz-Mie theory [24]. Even without considering multiple scattering, a similar tendency in the simulated spectral transmittance could be observed. This is indicated most clearly by the derivatives of the simulated spectral transmittance demonstrated in Figure 7(b). Compared to the measured one, the magnitude of this simulated effect is smaller, but it is expected to be enhanced by multiple scattering in the sample active surface. Further calculations are in progress and we expect that use of multiple in-surface scattering and scalar diffraction theory can bring the model closer to the observation [25, 26].

4.2. Preliminary Uncertainty Analysis

Explicit uncertainty analysis is still in progress. An estimation of the combined measurement uncertainty is around 0.5% ($k = 2$) in the best case ($\theta_t = 0^\circ$) for all sample types. Contributions to the estimated uncertainty budget of HOD-500 at 445 nm and $\theta_t = 0^\circ$, as an example, are listed in Table 2. Determination of the BTDF measurement uncertainty ΔBTDF is performed according to Stover [13]. The uncertainty mainly consists of the statistical errors from the two measured signals, ΔP_i and ΔP_t , the errors from the measurement of detection solid angle, $\Delta\Omega_t$, and from the receiver's angular position, $\Delta\theta_t$. The combined uncertainty ΔBTDF could be expressed by equation (7):

$$\frac{\Delta\text{BTDF}}{\text{BTDF}} = \sqrt{\left(\frac{\Delta P_i}{P_i}\right)^2 + \left(\frac{\Delta P_t}{P_t}\right)^2 + \left(\frac{\Delta\Omega_t}{\Omega_t}\right)^2 + \left(\frac{\Delta\theta_t \cdot \sin \theta_t}{\cos^2 \theta_t}\right)^2}. \quad (7)$$

At other angles the measurement uncertainty varies mainly with the measured signal amplitude and the cosine value of the detection angle. For both quasi-Lambertian samples, ΔBTDF remains unchanged in a large angle area and eventually increases to over 2% at grazing angles. For the other three types of samples, the signal amplitude drops quickly at the edges of the scattering distribution, leading to a sharp increase in the total uncertainty, from 0.5% to more than 3%. This estimate might hold only for slowly varying BTDF characteristics. At least for parts of the scattering distributions depicting a sudden variation, a term proportional to $\frac{\Delta\text{BTDF}}{\Delta\theta_t}$ must be added to the final uncertainty budget.

Table 2

Preliminary uncertainty budget of HOD-500, at $\lambda = 445$ nm, $\theta_t = 0^\circ$

Source	Notation	Relative uncertainty
Irradiation power	ΔP_i	0.00031
Scattered transmitted power	ΔP_t	0.00014
Detection solid angle	$\Delta\Omega_t$	0.00153
Distance sample to detector	ΔR	0.00054
Detector aperture area	ΔA_D	0.00134
ND-filter	$\Delta\tau_F$	0.00268
Receiver angle	$\Delta\theta_t$	0.00005
Estimated total uncertainty ($k = 2$)	ΔBTDF	0.005 / 0.5%

5. Conclusion

Preliminary results of the BTDF measurement involved in the EMPIR project BxDiff are presented in this paper. Five types of transmissive diffusers, two bulk scattering, one pure surface scattering, one holographic and one with engineered surface, were measured at the modified NaNoRef setup of PTB with a preliminary total uncertainty of approx. 0.5% ($k = 2$) at 0° detection angle (corresponds to the angle of specular transmission). The angle-dependent spectral transmittance of all samples was measured using a commercially available spectrophotometer to provide supporting information for a possible wavelength-dependent correction for the benefit of the comparison at a prescribed wavelength. An interesting phenomenon of the interaction between angle- and wavelength-dependency of the spectral transmittance was observed for the holographic diffuser and possible explanations are proposed

from first-step simulation results. Further study on the wavelength dependence of different sample types will be carried out to provide a more detailed quantitative explanation. Our aim is to acquire thorough information on the relation between sample microscopic structures and their scattering properties as well as a better knowledge of BTDF measurement on optical translucent materials with high precision.

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The mentioned diffusers dealt with in this paper are examples of commercially available products only and using these specific diffusers in investigations does not constitute an endorsement of these products by the authors or their institutions.

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3 Paper II:

Development of primary reference facilities and measurement comparison of standard artifacts for the bidirectional transmittance distribution function

Paper Summary

This paper thoroughly introduces the development of two primary BTDF facilities at PTB and Aalto, with details of their major components. A bilateral comparison of two BTDF samples, one quasi-Lambertian diffuser and one Gaussian diffuser, is performed at four wavelengths in the VIS. Detailed uncertainty analyses are presented for each facility and good consistency of the measurement results is achieved, indicating a successful development of both new primary facilities.

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Development of primary reference facilities and measurement comparison of standard artifacts for the bidirectional transmittance distribution function

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ABSTRACT

To determine the bidirectional transmittance distribution function (BTDF) of diffusely transmitting materials, two new primary facilities have been developed at the Physikalisch-Technische Bundesanstalt (PTB) and Aalto University (Aalto). A detailed description of both facilities and the different approaches used are presented in this paper. The performance of both facilities is compared by determining the BTDF of two different diffuser types in both in-plane and out-of-plane bidirectional geometries at four different wavelengths in the visible spectral range. Due to delayed completion of PTB's primary facility, the measured BTDF values are compared between Aalto's primary facility and another PTB setup, whose measurement scales are successfully transferred to PTB's primary facility by an internal comparison. A thorough analysis of the measurement uncertainty is presented, leading to a combined $k = 1$ standard uncertainty of 0.8%–1.2% for PTB's primary facility and 1.3%–1.7% for Aalto's primary facility. The BTDF results obtained agree well within their expanded $k = 2$ uncertainty. This indirect bilateral comparison shows that Aalto's and PTB's new facilities are suited as primary reference setups for the determination of the BTDF. These studies also reveal action points to improved measurement capabilities and for a reduction of the measurement uncertainty, depending on the type of diffuser under test.

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I. INTRODUCTION AND DEFINITION OF BTDF

The way an object looks is usually the most striking property of the object. To describe “how something looks” in a metrological way, the term visual appearance is used. Visual appearance serves as an umbrella under which different aspects are collected: color, gloss, texture, sparkle and graininess, and translucency. This paper focuses on the measurement of translucent or diffusely transmitting samples. The measurand describing this property is the bidirectional transmittance distribution function, denoted as BTDF.^{1–3} Determining BTDF values accurately is important for many

applications, such as satellite-based Earth remote sensing,^{4,5} diffusers to tailor the light distribution in optical systems and luminaires,^{6,7} computer graphics rendering,^{8,9} medical materials and prosthetics,¹⁰ or food industry.¹¹

Despite these widespread applications of BTDF, the availability of high-quality, validated, traceable BTDF measurements is still limited. To fill in this gap, one goal of the EMPIR project “JRP 18SIB03 BxDiff–New quantities for the measurement of appearance”¹² was to develop and install new facilities for the measurement of BTDF at *Physikalisch-Technische Bundesanstalt* (PTB), the national metrology institute of Germany, and at *Aalto University* (Aalto) in

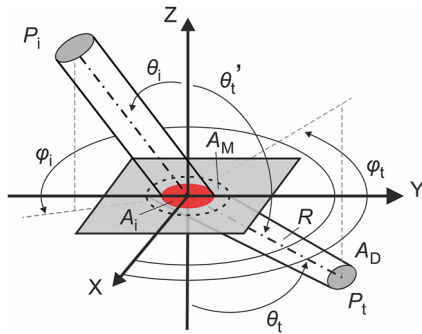


FIG. 1. Sketch of geometrical parameters for the realization of BTDF measurements.

Finland. Both facilities will be introduced in this paper, together with the results of a bilateral comparison of BTDF measurements for two different diffusers.

In analogy to the definition of the bidirectional reflectance distribution function (BRDF), BTDF is defined as

$$f_t(\theta_i, \phi_i, \theta_t, \phi_t, \lambda) = \frac{dL_t(\theta_t, \phi_t, \lambda)}{dE_i(\theta_i, \phi_i, \lambda)} [\text{sr}^{-1}], \quad (1)$$

where the measurand BTDF is referred to as f_t , E_i is the uniform irradiance on the sample generated by the incident radiant flux, L_t is the radiance of the transmitted radiant flux, $\theta_{i,t}$ and $\phi_{i,t}$ are the polar and azimuthal angles in incidence and transmission, and λ is the wavelength of the incident radiation.

Since the definition of BTDF given above is based on infinitesimal quantities, it has to be modified to be used for the realization of the measurement, e.g., in the following way. The incident beam with a finite extent irradiates a finite area A_i on the sample. The detector spans a finite solid angle $\Omega_t = A_D/R^2$ that is given by the detector aperture area A_D and its distance R to the back side of the sample. Thus, the detector collects light from a finite measurement area A_M on the sample. This leads to the often-applied equation¹³ for the realization of BTDF (when $A_M > A_i$)

$$f_t(\theta_i, \phi_i, \theta_t, \phi_t, \lambda) = \frac{P_t/\Omega_t}{P_i \cos \theta_t} [\text{sr}^{-1}], \quad (2)$$

where P_i and P_t are the incident and transmitted optical power, respectively. This is schematically shown in Fig. 1.

II. NEW PRIMARY FACILITIES

A. Primary facility for BTDF measurement at PTB

A schematic lay-out of PTB's primary facility for laser-based, small spectral bandwidth measurement of BTDF is shown in Fig. 2. The sample (S) is mounted on a six-axis robot arm (R) in the center of a large absolute-encoded rotation stage (diameter 1.5 m). The detection system is placed on two platforms on the rotation stage. One accommodates the detection optics (L3) and an integrating sphere (intS) with a silicon photodiode (Si-PD) attached to it. On the other one, a preamplifier and a lock-in amplifier (LIA) for

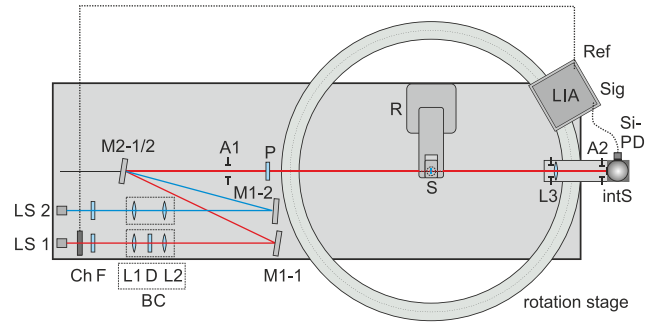


FIG. 2. Schematic drawing of PTB's new primary facility for the measurement of BTDF (explanation of components in the text).

signal acquisition are placed. The configuration of sample positioning and detection for performing transmittance measurement in the straight-on geometry, in which both incidence and transmission are perpendicular to the sample plane, is illustrated in Fig. 3. The irradiation beam path is fixed and the combination of the (R)-controlled sample orientation and the position of the detection system on the rotation stage allow almost any bidirectional geometry to be realized. These are not restricted to in-plane geometries, spanned by the sample normal and the incident beam, but also measurements out of this plane are possible. In this setup, rigid samples with a mass up to 8 kg may be measured, with their symmetry axis mainly oriented horizontally.

For the internal comparison within PTB described in Sec. III, two narrow-band cw laser light sources have been used: a diode laser (LS 1) at 642 nm and a diode-pumped solid-state laser at 445 nm (LS 2). The spatial properties of the incident beam are controlled by a beam conditioning (BC) unit. It comprises a beam expander with two lenses (L1 and L2) and a rotating weakly scattering diffuser (D) for homogenization and laser speckle reduction. The resulting beam divergence has $\sim 0.1^\circ$, expressed as full width at half maximum (FWHM). The size of the irradiation beam on the sample is determined by the aperture (A1), which is interchangeable to realize beam diameters up to 30 mm. A linear polarizer (P) defines the polarization state of the irradiation. Different attenuation filters (F) can be placed in the beam path to regulate the absolute level of the

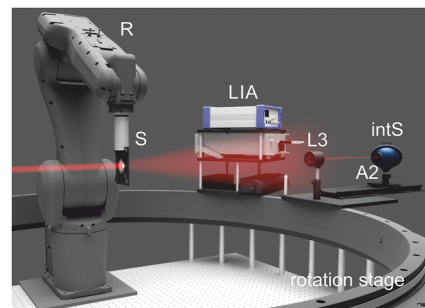


FIG. 3. Rendered image of sample positioning and detection on PTB's new BTDF primary facility. For a better visualization, some components for straylight reduction and data acquisition are not shown.

detected signal. A chopper (Ch) is used to modulate the laser outputs for lock-in detection. Individual beam conditioning lines for both lasers are set up to account for different divergence and beam profiles. The mirrors M1-1, M1-2, M2-1, and M2-2 are used to steer the beam onto the center of the sample. Mirrors M2-1 and M2-2 are exchanged depending on the laser source used. Also, the chopper, the attenuation filters, and the rotating diffuser are moved to the beam line in use.

The detection optics consists of an achromatic lens and a diaphragm placed directly in front of the lens. At a nominal distance of 652.2 mm from the sample back side to the diaphragm, the detection angular resolution can be varied from 0.6° to 3.1° by applying diaphragms with different diameters. This corresponds to detection solid angles ranging from 9.1×10^{-5} to 2.3×10^{-3} sr. This imaging detection system allows for measurements to be carried out either in under-irradiated or over-irradiated mode, depending on the size of the irradiation and the detection areas. The detection area is given by the size of the interchangeable aperture (A2) mounted at the entrance of the integrating sphere, which is imaged on the sample back surface by the achromatic lens. With a magnification of 1.4, the largest achievable detection area has a diameter of 30 mm. Controlling the hardware settings and collecting the measurement data are done using an in-house developed LabVIEW program. Once the different beams are aligned, measurements can be carried out fully automated.

B. Primary facility for BTDF measurement at Aalto

The primary BTDF facility at Aalto University is based on the 3D gonioreflectometer for spectral BRDF measurements. A detailed description of the 3D gonioreflectometer and its uncertainty budget, which can be at least partly applied on BTDF measurements, can be found elsewhere.¹⁴ The 3D gonioreflectometer is calibrated using a reference material whose BRDF is traceable to Aalto's reference gonioreflectometer.¹⁵ Figure 4 shows the 3D gonioreflectometer setup in its configuration for transmittance measurements. The Aalto approach allows also non-rigid samples to be measured. The sample is mounted on a horizontally aligned sample holder (6), while three motorized arms are used to set the bidirectional geometry [(2) for irradiation polar angle θ_i , (3) for detection or viewing azimuth angle ϕ_t , and (4) for detection polar or zenith angle θ_t]. A supercontinuum laser is used for irradiation. To select the wavelength, laser-line tunable filters are used. The irradiation optics (1) includes an SMA fiber collimator, order-sorting filter, a polarizer, beam expanding and steering optics, the monitor detector, and an adjustable iris for beam size. After impinging on the sample, the transmitted radiation is collected via an off-axis parabolic mirror (5) and fiber coupled to the detector box. Depending on the wavelength, either a Si- or InGaAs photodiode is used for detection. Transimpedance amplifiers convert the current signals for voltage measurement. Motion control of the facility and data acquisition are achieved by using a LabVIEW program run on personal computer (PC).

A standard measurement procedure of a test sample, using the 3D gonioreflectometer, includes adjustments of the sample holder height by using a linear translator micrometer. Thus, the geometric center of the instrument can be set to different heights for samples of different thicknesses. The sample surface facing the incident beam

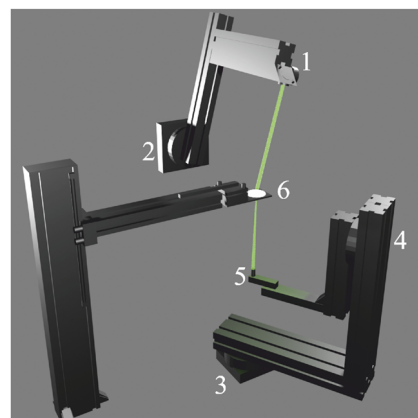


FIG. 4. Schematic drawing of Aalto's new primary BTDF facility.

is used to define the center point. To ensure accurate alignment, the reference face of the test sample is adjusted with a self-leveling multi-beam laser, enabling a repeatable alignment within 0.1 mm. Furthermore, to obtain the BTDF result of the test sample, a reference sample is measured both in the reference gonioreflectometer for its BRDF and in the 3D gonioreflectometer for its reflected signal. The ratio of both instruments' results allows for BTDF measurements of test samples in any geometry at pre-measured wavelengths.

Irradiation of the test sample and detection of the signal is performed by three motorized arms. The irradiation polar angle can be varied in a range of 0° – 90° with irradiation azimuth angle at 0° and 180° , and by using an iris, a beam size at the sample from 8 to 20 mm in diameter can be provided. During the measurements, an 8 mm beam size was used, which follows a Gaussian beam profile. The detector is equipped with a two-axis motorized arm to scan the half-sphere below the sample plane. The first arm controls the viewing polar angle, which can be adjusted within the range of 0° – 90° . The second arm sets the viewing azimuth angle, allowing variations from 0° to 180° . Both arms enable in- and out-of-plane measurements of non-rigid samples. The detection has an angular resolution of 2.89° . The angular resolution corresponds to a solid angle of ~ 0.0022 sr, determined by the off-axis parabolic mirror housing aperture diameter ($\varnothing$ 10.85 mm) and the detector aperture-to-sample distance (204.7 mm).

The irradiation is provided by a combination of a supercontinuum laser and a laser-line tunable filter. The system provides a collimated beam in the wavelength range from 400 to 2400 nm, which has a bandwidth of 4 nm. The optical power can be adjusted in the supercontinuum laser power source to optimize the irradiation according to the sample type, with an average power of 0.3 mW. Polarization of the beam is controlled by inserting polarizers, which have extinction coefficients of 1000:1, in the cage system and in the beam path. The stability of the laser power is monitored by using a beam splitter and a two-color monitor detector, with sensitivity in the wavelength range from 400 to 1700 nm, before the iris. Transmitted light is collected by using an optical fiber that directs the light to a detector housing, which includes a silicon and an InGaAs photodiode. A transimpedance amplifier provides amplification in the dynamic range from 10^2 to 10^9 that covers signal ranges

TABLE I. Measurement parameters for the BTDF comparison.

Parameter	Symbol	Value
Wavelengths	λ	642, 532, 445, 405 nm
Polarization of incident light	Pol.	Unpolarized as mean of two polarization directions s and p
Angles of incidence	(θ_i, φ_i)	$(10^\circ, 0^\circ)$ (sample B) $(0^\circ, 0^\circ)$ (sample E)
Angles of detection	(θ_t, φ_t)	$(0^\circ, 0^\circ)$ and $(14.11^\circ, 135.44^\circ)$ (sample B) $(0^\circ, 0^\circ)$ (sample E)

for Lambertian and more transparent samples, such as frosted glass samples.

III. BTDF MEASUREMENT COMPARISON

To compare the BTDF scales of the measurement facilities at Aalto and PTB, two different samples were chosen. One sample made of a 0.25 mm thick PTFE foil with a Lambertian scattering characteristic, denoted sample E, and one sample made of bulk NK7 glass with a sandblasted active surface, possessing a Gaussian scattering distribution with FWHM of 16° , denoted sample B. The BTDF for sample E has been determined in an in-plane geometry, and for sample B, both in-plane and out-of-plane geometries have been realized. The details can be found in Table I. The geometry $(0^\circ, 0^\circ)$ for both incidence and detection implies the direction perpendicular to the sample plane. The measurements were performed at four wavelengths.

Completion of PTB's primary BTDF facility was delayed due to delivery problems of some vital components. Thus, the measurements for the comparison, which had to be performed within the

schedule of the BxDiff-project, were carried out on PTB's primary facility for the measurement of regular transmission and reflection, which has been modified accordingly¹⁶ (abbr. "modified NaNoRef"). Subsequent measurements have shown that the results from both PTB facilities agree well within their specified uncertainty. An exemplary result for sample B at 642 nm, depicting the congruence, is shown in Fig. 5.

IV. MEASUREMENT UNCERTAINTY

A. Uncertainty budget for PTB's BTDF facilities

The main contributions to the uncertainty budget for the modified NaNoRef and the new primary BTDF facility at PTB are listed in Tables II and III, respectively.

Uncertainty in the measurement signal is determined by signal noise and instability in the laser power, the latter being different for both setups. To reduce the measurement noise, each measurement

TABLE II. Uncertainty budget for PTB's modified NaNoRef facility. Boldface denotes the combined $k = 1$ standard uncertainty.

Source of uncertainty	Standard uncertainty	Uncertainty in BTDF (%)
Measurement signal ^a	0.1%	0.1
Transmittance of attenuation filter	0.00015 ($\tau = 0.1$) 0.00002 ($\tau = 0.01$)	0.15–0.2
Detector aperture area ^b	0.1%	0.1
Distance sample–detector	0.1 mm	0.04
Detection polar angle positioning	0.01°	0.01–0.6
Wavelength ^c	0.1–0.65 nm	0.03–1.5
Detection angular resolution	<0.2%	<0.2
Beam spatial nonuniformity	20% relative to the maximum beam intensity	<0.01
Polarization	<0.05%	<0.05
Inter-reflection	0.001%	0.001
Digitization error	0.01%	0.01
Correction of detector nonlinearity	<0.01%	<0.01
Straylight	1/5 of dark signal	<0.05
Combined standard uncertainty ($k = 1$)		0.3–1.6

^aThis uncertainty includes the drift and fluctuation of both irradiating and transmitted optical power.

^bThis uncertainty includes the uncertainty of area measurement, possible dust contamination, and influence of temperature change.

^cThis uncertainty depends on the laser source and laser output power, as well as the transmittance characteristic of the attenuation filter.

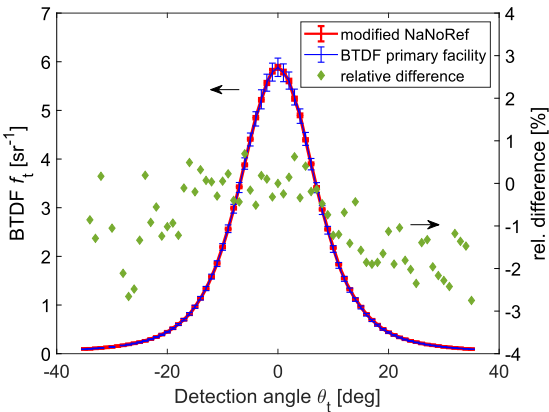


FIG. 5. Exemplary results of sample B measured at 642 nm, for the agreement between new primary BTDF facility and modified NaNoRef setup at PTB, shown for $(0^\circ, 0^\circ)/(0^\circ-35^\circ, 0^\circ)$ and $180^\circ)$ geometries (negative detection angles indicate $\varphi_t = 180^\circ$). The green dots correspond to the relative difference between the BTDF values measured on both setups.

TABLE III. Uncertainty budget for PTB's new primary BTDF facility. Boldface denotes the combined $k = 1$ standard uncertainty.

Source of uncertainty	Standard uncertainty	Uncertainty in BTDF (%)
Measurement signal ^a	0.2%	0.2
Transmittance of attenuation filter	0.00007 ($\tau = 0.01$)	0.7
Detector aperture area ^b	0.03%	0.03
Distance sample–detector	0.3 mm	0.09
Detection polar angle positioning	0.015°	0.01–0.9
Wavelength ^c	0.2 nm	0.01
Detection angular resolution	<0.2%	<0.2
Beam spatial nonuniformity	20% relative to the maximum beam intensity	<0.01
Polarization	<0.05%	<0.05
Inter-reflection	0.001%	0.001
Straylight	Similar to dark signal	<0.2
Combined standard uncertainty ($k = 1$)		0.8–1.2

^aThis uncertainty includes the drift and fluctuation of both irradiating and transmitted optical power.

^bThis uncertainty includes the uncertainty of area measurement and influence of temperature change.

^cThis uncertainty depends on the laser source and laser output power, as well as the transmittance characteristic of the attenuation filter.

was repeated nine times, and the statistics was considered as signal fluctuation. As no monitoring was performed on the laser output power, the incident signal was measured before and after the transmitted signal to eliminate any linear drift in the laser power. The residual drift was considered as the instability in the laser output power.

Neutral density (ND) filters were used to adjust the optical signal to the dynamic range of the detector when measuring the incident irradiation power. The neutral density filters used on the modified NaNoRef setup were calibrated by the national standard setup for regular transmission at PTB and have minimum relative uncertainties according to the PTB Calibration and Measurement Capability (CMC)-entry for spectral transmittance,¹⁷ whereas the filters used on the new BTDF primary setup were currently only calibrated by a commercial instrument and have a larger relative uncertainty.

The uncertainty of the detection solid angle originates from the uncertainty in the area determination of the detector diaphragm and its distance to the sample back side. The larger NaNoRef-uncertainty was due to possible dust contamination by anti-reflection coating

residual. This problem was avoided for the new BTDF facility. The instrumental distance was measured by a calibrated inside micrometer multiple times and the uncertainty was 0.1 mm for the modified NaNoRef and 0.3 mm for BTDF facility. An additional distance uncertainty is accounted for sample E due to an observed non-flatness of the foil. (PTB's measurements on sample E were corrected for this sample-specific distance error and the uncertainty was estimated to be $1./\sqrt{3}$ mm).

The uncertainty in the detection polar angle positioning is dominated by the alignment of the detector diaphragm, as the accuracy and reproducibility of the motorized detection arm/rotation stage are small in usual applications. It was estimated to be 0.01° for the modified NaNoRef setup and 0.015° for the new primary BTDF setup. Its contribution to the combined uncertainty is dependent on both sample scattering characteristics and detection polar angle setting. This component is dominant in the measurement uncertainty of sample B since, in both measurement geometries, the detection was made at the angle where the BTDF distribution exhibits large variation.

The wavelength uncertainty is dependent on the central wavelength shift of the laser spectrum from its nominal value when the lasers are operated at different output power levels (except for 532 nm). Its influence depends on the spectral characteristics of the sample, which was measured as a function of transmission angle for perpendicular incidence at PTB,¹⁶ and on the spectral transmission of the ND-filter. For sample E, the latter one contributes mainly at shorter wavelength.

The influence of the finite detection angular resolution is an important component in the uncertainty budget for sample B due to its narrow scattering distribution. It is characterized by the so-called instrument function, which describes the angular resolution ability of the measuring system and is determined by an angle-scan without sample.¹³ The measured BTDF then is the result of convolving the true sample BTDF with the instrument function. Its impact is proportional to the second derivative of the distribution with respect to the scatter angle ($\frac{\partial^2 \text{BTDF}}{\partial \theta_s^2}$). Since the angular resolution of 1° applied on both PTB setups was sufficiently small with respect to the width of sample B's distribution, the <0.2% difference between the measured and the true distribution is considered as relative uncertainty using a deconvolution method in analogy to CIE 214.¹⁸ For sample E, whose scattering distribution is quite flat, this influence is considered as an uncertainty of 0.01% to its BTDF.

The spatial intensity variation in the irradiation beam was considered as the uncertainty of beam nonuniformity. The beam profile was measured by a laser beam analyzer and showed a 20% intensity decrease at the outer part of the beam compared to the beam center. A simple ray-tracing simulation taking this edge reduction into account by a parabolic profile resulted in only small uncertainty contributions for all measurements reported here.

The uncertainty in polarization stems from the irradiation since the detection is unpolarized. It was determined by the extinction coefficient of the linear polarizer and the difference between the two polarized BTDF values. The contribution of this component is negligible for sample E, and for sample B this component still contributes less than 0.05% to the BTDF.

The inter-reflection between optical elements was estimated, mainly between the achromatic lens and the detection diaphragm

due to the small distance. This component contributes only 0.001% to the BTDF.

The digitization error accounts for the finite resolution when the measured value was digitized by the signal reading devices. On the modified NaNoRef setup, the measured data were digitized to the fourth decimal place, which results in an uncertainty of 0.01% in the BTDF. The device on the new primary BTDF setup has a higher resolution that could digitize the measured data to the sixth decimal place. Thus, this uncertainty is negligible.

The linearity of the detector used on the modified NaNoRef setup was calibrated at PTB and the measurement results were corrected. The uncertainty of the correction has an influence of no more than 0.01% on the measured BTDF. The linearity of the detector used on the new primary BTDF setup was not yet calibrated, and thus, no information is given here. We assume that this uncertainty component would influence the measurement in a similar way, as the same type of detector was used for the measurement on the primary BTDF setup.

The straylight was estimated by performing the BTDF measurement with a beam blocker placed near the back surface of the sample. The signal of the scattered light under this condition could not be differentiated from the signal when the irradiation beam was blocked. On both PTB's setups, the straylight reduction was supported by the use of the lock-in amplifier technique and the depth of sharpness of the imaging detection system. Due to an additional light-proof chamber, where the sample was placed separated from the light source, straylight reduction was superior on the modified NaNoRef setup. Therefore, the straylight on the modified NaNoRef setup was estimated to be 1/5 of the dark signal level and similar to the dark signal level on the new primary BTDF facility.

B. Uncertainty budget for Aalto's BTDF facility

The uncertainty budget for the primary BTDF facility at Aalto University is given in Table IV. The third column of Table IV shows the uncertainty in BTDF for sample B. The uncertainty budget has previously been evaluated for BRDF measurements elsewhere.¹⁴ The following paragraph describes major changes in uncertainties when measuring BTDF. The reader should note that several uncertainties in BTDF are a quadratic sum of the standard uncertainties to account for the reference and test sample measurement uncertainties.

Measurement noise represents the largest component in the 3D gonireflectometer uncertainty budget. It was evaluated by recording the scattered signal of a PTFE sample at 2-second intervals over a period of 45 min.¹⁴ This approach allows us to assess signal fluctuations beyond the normal nine measurement points used for calculating each BTDF value. Measurement points that fluctuate around the signal mean can be considered as noise. The uncertainty in measurement noise has increased by a factor of 1.4 due to the lower signal when measuring transmittance as compared to reflectance measurements. To mitigate measurement noise, multiple measurement points can be acquired, and the uncertainty can be reduced by using the standard deviation of the mean. The measurement noise uncertainty in BTDF consists of both the detector and monitor uncertainties, which have been combined quadratically.

TABLE IV. Uncertainty budget for the primary BTDF facility at Aalto University. Boldface denotes the combined $k = 1$ standard uncertainty.

Source of uncertainty	Standard uncertainty	Uncertainty in BTDF (%)
Measurement noise ^{a,b}	0.42%–0.57%	0.51–0.70
Instrument stability	0.10%	0.14
Wavelength ^c	0.15 nm	0.01–0.18
Straylight (hetero- and isochromatic)	<0.01%	0.01
Detector linearity ^b	0.05%–0.09%	0.07–0.13
Beam spatial nonuniformity ^c	0.14%	0.14
Irradiation polar angle ^c	0.09°	0.09
Detection polar angle ^c	0.09°	0.02–0.90
Detection azimuth angle ^c	1.4°	1.1
Sample surface level	0.1 mm	0.14
Polarization ^c	0.10%	0.1
Reference material	0.5%	0.50
Combined standard uncertainty ($k = 1$)		1.34–1.70

^aThis uncertainty depends on the detection polar angle.

^bThis uncertainty depends on the detector type and selected gain of amplifiers.

^cThis uncertainty depends on the sample.

The instrument stability was evaluated by measuring samples on different days, while removing and inserting the sample again. The repeatability had a relative standard deviation of 0.1%.

The standard uncertainty in the central wavelength was determined by scanning the collimated beam against a calibrated radiometer.¹⁹ The results show that the laser-line tunable filter has a standard uncertainty of 0.15 nm. The uncertainty in BTDF is determined from the slope of the BTDF as a function of wavelength, which is sample dependent.

Straylight uncertainty was evaluated for iso- and heterochromatic light.¹⁴ Straylight contributions are confined to scattering inside the instrument darkened compartment. Therefore, heterochromatic straylight from the supercontinuum laser is limited by its out-of-band rejection level. The heterochromatic straylight contribution in uncertainty was evaluated as less than 0.01%. The isochromatic straylight was evaluated by illuminating a diffuse white PTFE sample out of focus. The results showed that in the darkened compartment intra-scattering contribution was at the same level as the dark signal. As a result, the isochromatic straylight contribution to the uncertainty was less than 0.01%.

Detector linearity has been studied elsewhere.^{20,21} The measurement conditions of the test sample match that of literature, with signal currents from 10^{-11} to 10^{-8} and 10^{-8} to 10^{-5} A for the Si and InGaAs detectors, respectively. Therefore, the standard uncertainty in the Si detector linearity is 0.05% and 0.09% for the InGaAs detector. Similar to the case of measurement noise, the relative

uncertainty in BTDF due to detector linearity is determined from the combined uncertainties of the detector and monitor detector, which are added quadratically.

Beam spatial nonuniformity uncertainty was assessed by using a scanning-slit optical beam profiler.¹⁴ The beam profiler shows the beam intensity as a function of x-y coordinates. By taking slices of the Gaussian beam profile, slight asymmetries in the beam can be detected. Different slices of the Gaussian beam profile were compared, and the largest variations showed a relative difference of 0.5%. For samples with linear spatial inhomogeneities, the beam nonuniformity translates into a relative standard uncertainty of 0.14%, which follows a rectangular probability distribution. For Lambertian samples, this standard uncertainty effect on BTDF is <0.01%.

Irradiation and detection angle uncertainty depend on the goodness of the alignment procedure for intersecting the rotational axes to the geometrical center. Thus, the uncertainty in angles consists of the cumulative error in alignment residual errors. Currently, the detection azimuth angle uncertainty skews the cumulative uncertainty, and work on improving the alignment procedure for the detection azimuth angle is ongoing to lower this uncertainty component of 1.1%. The uncertainty in irradiation and viewing angles are also sample-dependent. The uncertainty can be found from multiplying the standard uncertainty in angle with the slope of the BTDF as a function of the corresponding angle. Sample B has a large slope in its BTDF as a function of θ_i due to its Gaussian transmission distribution and shows a large uncertainty in the range from 0.02% to 0.9%. The uncertainty in BTDF as a function of azimuth angle was estimated from the relative difference in BTDF when comparing values using opposite incident polar angles, mirrored around the sample reference side normal, which should ideally be as close as possible.

The sample level uncertainty affects the solid angle of the measurement. The sample surface is aligned by using a self-leveling laser that sets the geometrical center of the goniometer axes. The alignment process is repeatable within 0.1 mm, which introduces a 0.14% uncertainty in the solid angle.

The uncertainty in polarization was evaluated from the polarizer extinction coefficients. Therefore, samples that change in BTDF as a function of polarization have an uncertainty of 0.01% due to uncertainty in polarizer state.

The uncertainty in the reference sample BRDF has been evaluated in the reference gonireflectometer uncertainty budget.¹⁵ The uncertainty was increased for transmittance measurements due to a lower signal.

V. COMPARISON RESULTS AND DISCUSSION

The results of the comparison between PTB and Aalto are presented in this section. The weighted mean of the reported BTDF from both labs was calculated using the inverse of the squared standard uncertainty as weighting factor and the data consistency was checked by chi-squared (χ^2) test.

The results of sample E are shown in Fig. 6, together with the measurement geometry expressed in the form $(\theta_i, \phi_i)/(\theta_t, \phi_t)$. The BTDF measured on both facilities agree within their expanded ($k = 2$) uncertainty and the χ^2 test has passed for all wavelengths. The BTDF values given by PTB are systematically larger than those

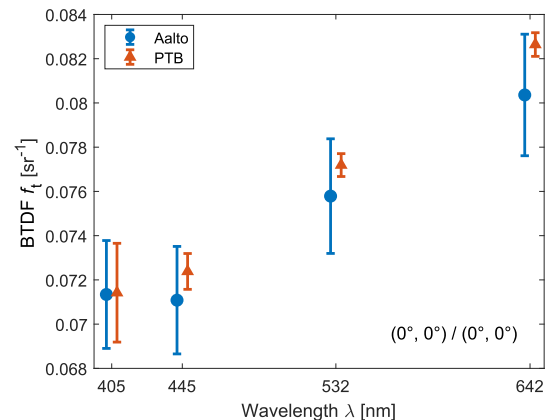


FIG. 6. Comparison results for sample E with $k = 2$ uncertainty.

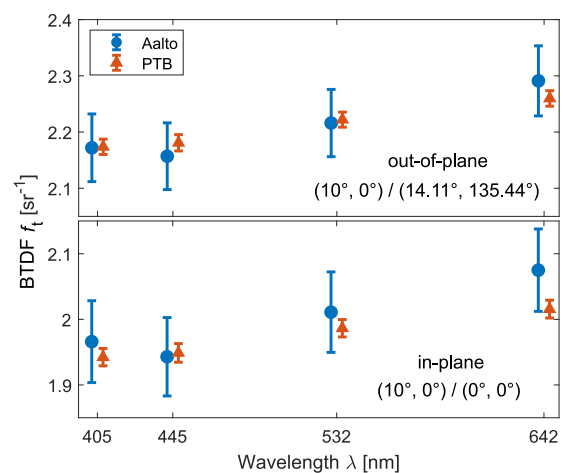


FIG. 7. Comparison results for sample B with $k = 2$ uncertainty.

by Aalto. The effect might be explained by the non-flatness of the foil as it exhibits a pre-bent form that could not be completely removed by the sample holder. In PTB's measurements, the bending was facing toward the detection, resulting in an underestimated collection solid angle, whereas in Aalto's measurements, it was in the opposite direction and the measured results were not corrected.

Figure 7 shows the BTDF results for sample B, together with the geometries in the form $(\theta_i, \phi_i)/(\theta_t, \phi_t)$ for both in- and out-of-plane measurements in the corresponding subplot. The consistency check shows that the results also agree within their expanded uncertainty for both in-plane and out-of-plane geometries. For this rigid sample, no obvious systematic deviation was found between both facilities.

VI. SUMMARY AND OUTLOOK

Two new measurement facilities have been developed and installed at PTB and Aalto University for high-quality, traceable

BTDF measurement. Their performance has been tested in a bilateral comparison for two different diffusers, one with a Lambertian and the other one with a Gaussian scattering characteristic. The results in both in-plane and out-of-plane geometries agree well within their expanded ($k = 2$) uncertainties for all four visible wavelengths applied. The presented bilateral comparison proved the consistency of the BTDF measurements carried out on the two primary facilities in PTB and Aalto. Main contributions to the uncertainty budget have been identified and will be considered in future improvement of the systems.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

J. Fu: Data curation (equal); Formal Analysis (equal); Visualization (equal); Writing—Original Draft Preparation (equal). **T. Quast:** Conceptualization (equal); Formal Analysis (equal); Visualization (equal); Writing—Original Draft Preparation (equal). **E. Velke:** Resources (equal). **M. Esslinger:** Data curation (supporting); Resources (equal). **M. Pastuszek:** Software (equal). **A. Schirmacher:** Conceptualization (equal); Formal Analysis (equal); Funding Acquisition (equal); Supervision (equal); Writing—Review & Editing (equal). **R. Aschan:** Data curation (equal); Formal Analysis (equal); Visualization (equal); Writing—Review & Editing (equal). **F. Manoocheri:** Conceptualization (equal); Funding Acquisition (equal); Supervision (equal); Writing—Review & Editing (equal). **E. Ikonen:** Funding Acquisition (equal); Supervision (equal).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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4 Paper III:

Survey of bidirectional transmittance distribution function measurement facilities by multilateral scale comparisons

Paper Summary

This paper reports on two interlaboratory comparisons of the BTDF measurement capabilities for European NMIs, DIs, research institutions, and industrial partners. First the measurand BTDF and the investigated samples are thoroughly introduced. Then the measurement protocol and comparison methodology are elaborated. Relevant parameters of the participating facilities are also summarized in an overview in the paper and with more details in the Supplementary Material. More focus is laid on the first intercomparison, with results presented in Section 3 in the paper. The overall inconsistency for almost all sample types leads to detailed discussions with respect to sample characteristics, measurement conditions and limitations, and reported uncertainty budgets. With the help of calculated degree of freedom (DoE), issues related to these aspects are identified for improved measurements in the future. Nevertheless, it is also shown that at least a subset of the participants is able to perform BTDF measurement equivalent to the state of the art. The second intercomparison shows good consistency for all sample types, successfully connecting the scale of BTDF measurement between metrology and industry. The issue related to lateral diffusion, as introduced in Section 1.3.3, is observed in both intercomparisons, leading to further investigation as described in Chapter 5.

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Survey of bidirectional transmittance distribution function measurement facilities by multilateral scale comparisons

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Abstract

In recent years, a growing demand for the capability of performing accurate measurements of the bidirectional transmittance distribution function (BTDF) has been observed in industry, research and development, and aerospace applications. However, there exists no calibration and measurement capabilities-entry for BTDF in the database of the *Bureau International des Poids et Mesures* and to date no BTDF comparison has been conducted between different national metrology institutes (NMIs) or designated institutes (DIs). As a first step to a possible future key comparison and to test the existing capabilities of determining this measurand, two interlaboratory comparisons were performed. In comparison one, five samples of three different types of optical transmissive diffusers were measured by five NMIs and one DI. By specific

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sample choice, the focus for this study lay more on orientation-dependent scatter properties. In comparison two, where one NMI, one DI, one university, and three industrial partners investigated their measurement capabilities, the dependence on the orientation was not assessed, but two additional samples of the same material and different thickness were measured. Results of the two comparisons are presented, giving a good overview of existing experimental solutions, and showing specific sample-related problems to be solved for improved future BTDF measurements.

Supplementary material for this article is available [online](#)

Keywords: BTDF, intercomparison, diffuse transmission, goniospectrophotometry, applied radiometry, transmissive diffuser

1. Introduction

Fields of application for optical diffuse transmission are manifold and range from greenhouse ceilings, architectural accessory, to beam forming in lighting, and satellite radiation calibration, resulting in a demand for the traceable characterisation and calibration of optical transmission with high accuracy. This can be accomplished by the measurement of the bidirectional transmittance distribution function (BTDF), which would allow transmittance for any irradiation and detection conditions to be obtained. However, there exists neither a calibration and measurement capabilities-entry in the *Bureau International des Poids et Mesures* (BIPM) database for the BTDF measurand [1], nor has a key comparison arranged by the Consultative Committee of Photometry and Radiometry (CCPR) of the International Committee for Weights and Measures (CIPM). Only a bilateral comparison on the BTDF scale of two quasi-Lambertian diffusers performed by the National Aeronautics and Space Administration (NASA) and the National Institute of Standards and Technology (NIST) has shown good consistency between the two participating instruments [2]. To get an insight on existing European measurement capabilities and gain experience in the characterisation of a larger variety of different diffusers, intercomparisons on the BTDF scale between partners of the EMPIR project 18SIB03 (BxDiff) [3] were performed.

This study reports on two multilateral scale comparisons of BTDF measurements. Comparison one was performed between national metrology institutes (NMIs) and designated institutes (DIs) from Germany—*Physikalisch-Technische Bundesanstalt* (PTB), Denmark—*Danish Fundamental Metrology* (DFM), France—*Conservatoire National des Arts et Métiers* (CNAM), Finland—*Aalto-yliopisto* (Aalto), Czech Republic—*Cesky Metrologický Institut* (CMI), and the non-European project partner *Measurement Standards Laboratory* (MSL) from New Zealand. It mainly aimed to lay the foundation for a future scale comparison between the national standard facilities for diffuse transmission. Unlike comparison one, comparison two also involved commercially available instruments in order to evaluate the present capabilities of these systems. The NMIs and DIs taking part in this comparison were

from Spain—*Consejo Superior de Investigaciones Científicas* (CSIC) and Sweden—*Research Institutes of Sweden* (RISE). The other participants were the *Katholieke Universiteit Leuven* (KU Leuven, Belgium) with their home-built instrument, and three industrial partners with commercial measuring systems: *Saint-Gobain Recherche* (Saint-Gobain), *Covestro Deutschland AG* (Covestro), and *Temicon GmbH* (Temicon). The latter two participants supplied different types of samples for both comparisons.

Measurements were performed for samples with various scattering characteristics in different in-plane geometries. The measured BTDF results were compared at two different wavelengths in comparison one and at one wavelength in comparison two. Since the two comparisons have different details and parameters, an overview of the structure of this paper is given in the following. Readers are invited to focus on those sections that are most relevant for their interests or application.

- Section 2 introduces the measurand, the sample details, the measurement procedures, the participated measuring instruments, and the methodology of both comparisons.
- Section 3 presents the results and discussions of comparison one, in which the metrological community dealt with accurate measurement of more difficult samples.
- Section 4 presents the results and discussions of comparison two, in which less complicated samples were studied by a broader group of participants including metrology institutes, university, and industries.
- Section 5 concludes both comparisons.

2. Methods

2.1. Description of the measurand

The measurand in both comparisons is the BTDF, which was firstly described by Bartell *et al* [4] as an expanded counterpart in the measurement of transmittance to the bidirectional reflectance distribution function (BRDF), which was introduced by Nicodemus [5, 6] as the measurand for reflection. The BTDF is defined as the derivative of the transmitted radiance L_t with respect to the uniform incident irradiance E_i :

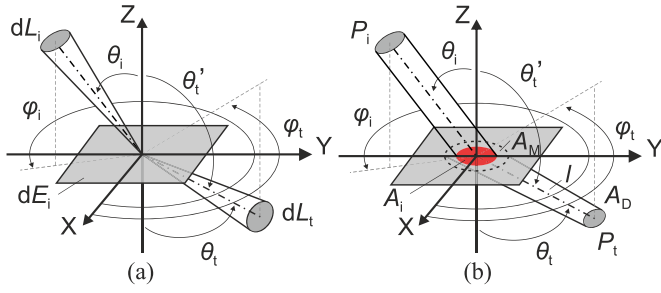


Figure 1. Geometrical variables of (a) BTDF definition and (b) BTDF measurements. Modified from figure 1 in [9] and figure 1 in Adapted from [10]. CC BY 4.0. Both licensed under a Creative Commons Attribution (CC BY) licence. Meaning of the symbols in the figure is introduced in the text.

$$f_t(\theta_i, \varphi_i; \theta_t, \varphi_t) = \frac{dL_t(\theta_t, \varphi_t)}{dE_i(\theta_i, \varphi_i)}, \quad (1)$$

with the measurand BTDF referred to as f_t . It is dependent on the polar angle θ with respect to the sample normal (z-axis), and the azimuth angle φ with respect to the x-axis, both lying within the sample plane. The subscripts ‘i’ and ‘t’ designate the direction of the incident and transmitted radiation, respectively. The measurand describes the scattering of a transmitting sample into the unit solid angle and has the unit of the inverse steradian (sr^{-1}). The BTDF also depends on the wavelength λ of the incident radiation and is typically expressed as the unpolarised value at a specific wavelength.

In the measurements, however, the infinitesimal values in the definition cannot be realised [7]. The measured BTDF is always an average over an area on the sample surface [4], and detected within a finite solid angle of the detector, as illustrated in figure 1.

The BTDF can be measured as an absolute value in two measurement schemes. In the under-irradiated scheme, where the measurement area A_M is larger than the irradiated area A_i , the BTDF is calculated as [2, 7, 8]:

$$f_t = \frac{P_t}{P_i} \cdot \frac{1}{\Omega_t \cos \theta_t}, \quad (2)$$

with P_i and P_t being the incident and transmitted optical power and Ω_t the detector solid angle determined by the detector aperture area A_D and the distance l between the detector aperture and the sample back surface:

$$\Omega_t = \frac{A_D}{l^2}, \quad (3)$$

where $\sqrt{A_D}$ should be much smaller than l . In the over-irradiated scheme (measurement area $A_M < \text{irradiated area } A_i$), where the entire sample should be uniformly irradiated, the BTDF is measured as the ratio between the transmitted radiance L_t and the incident radiance L_i , along with the geometric factor [8]:

$$f_t = \frac{L_t}{L_i} \cdot \frac{1}{\Omega_t \cos \theta_t}. \quad (4)$$

For relative measurements, the measurand can be estimated by comparison with a reference standard for diffuse reflection, as no agreement on the reference standard for diffuse transmission has yet been reached. Thus, the BTDF of the sample under test is obtained with the aid of the calibrated BRDF of the reference standard f_{ref} and the ratio between the transmitted optical power of the sample under test P_t and the reflected optical power of the reference standard P_{ref} measured under the same condition [8]:

$$f_t = f_{\text{ref}} \cdot \frac{P_t}{P_{\text{ref}}}. \quad (5)$$

2.2. Description of measurement protocols

Altogether seven different sample types were chosen for the two comparisons. They give a good representation of diffusers being present on the market and used in different fields of application. These samples can be roughly divided into three categories as listed in table 1. Different sample types were used in the two comparisons so that varying key aspects were addressed. In this paper, all samples are mentioned by their designation as presented in table 1.

Each comparison comprised five different sample types, whose details are given in table 1. Unlike comparison one, comparison two lay the emphasis on different scattering magnitudes and thicknesses, rather than azimuthal orientation dependence. Both comparisons were performed in a star-type manner as schematically shown in figure 2, providing individual, nominally identical sample sets for each participant. Measurements were conducted by two pilot labs in each comparison. Each pilot measured two sample sets and distributed them to two non-pilot participants so that each one received one set. After their measurements, the pilots repeated the measurements on the two sets to ensure that no variation was introduced in the shipping process or during measurements. Additionally, one extra set was compared between the two pilots following a similar procedure, with repeated measurements carried out by the first pilot. Because of technical difficulties, deviations from the described procedure occurred in some cases, which are considered in the evaluation.

In all measurements, the sample under test was irradiated at the geometrical centre under normal incidence, i.e. $(\theta_i, \varphi_i) = (0^\circ, 0^\circ)$. Adapted to the scattering characteristics, the detection geometries vary for each sample type. For comparison one, the BTDF was measured in a narrower angular range than in comparison two. The samples B, C, and E were measured in the detection geometries $(\theta_t, \varphi_t) = (0^\circ\text{--}35^\circ, 0^\circ \text{ \& } 180^\circ)$ with angular step of $\Delta\theta_t = 5^\circ$. For the other two orientation-dependent samples, the measurements were conducted in two perpendicular azimuthal directions. For sample A, the detection geometries were chosen as $(\theta_t, \varphi_t) = (0^\circ\text{--}20^\circ, 0^\circ \text{ \& } 180^\circ)$ and $(0^\circ\text{--}20^\circ, 90^\circ \text{ \& } 270^\circ)$. For sample D, the parameters were $(\theta_t, \varphi_t) = (0^\circ\text{--}40^\circ, 0^\circ \text{ \& } 180^\circ)$ and $(0^\circ\text{--}20^\circ, 90^\circ \text{ \& } 270^\circ)$. Both samples were measured with a smaller angular step of $\Delta\theta_t = 1^\circ$. For comparison two, since all sample types involved are nominally independent of φ_t , the

Table 1. Details of sample types used in both comparisons^a.

Category	Comparison	Name	Designation	Material	Description of scattering properties	Dimensions (mm)
Quasi-Lambertian	1, 2	Heraeus HOD®-500	C	Fused synthetic SiO ₂ with uniform air-bubbles ($\varnothing < 20 \mu\text{m}$)	Bulk scattering	$\varnothing 50 \times 2$
	1, 2	SphereOptics Zenith® Polymer	E	Sintered polymer PTFE foil, sandwiched between two 90 mm $\times$ 110 mm plates	Bulk scattering	$\varnothing 45 \times 0.25$
Dedicated angle-dependent scattering, no azimuthal dependence	1, 2	Thorlabs DG20-220	B	N-BK7 substrate, active surface roughened using 220 grit	Gaussian, FWHM 16°, surface scattering	$\varnothing 50.8 \times 2$
	2	Covestro IOL1113-2, thin	Co1	Scatter centres in polycarbonate matrix	Gaussian, FWHM 50°, bulk scattering	$50 \times 50 \times 1$
	2	Covestro IOL1113-2, thick	Co3	Scatter centres in polycarbonate matrix	Gaussian, FWHM 130°, bulk scattering	$50 \times 50 \times 3$
Dedicated angle-dependent scattering, with pronounced azimuthal dependence	1	Thorlabs ED1-S20	A	Microlens units made of Zeonor® injection moulded on N-BK7 substrate	Square-shaped, FWHM $\sim 20^\circ/20^\circ$, functional surface	$\varnothing 25.4 \times 1.5$
	1	Temicon PAN001008	D	125 μm thick imprinted PET foil on standard glass substrate	Gaussian elliptical, FWHM $26^\circ/13^\circ$, holographic diffuser	$50 \times 50 \times 2$

^a The listed samples are examples of possibly suitable products. Their choice does not constitute an endorsement by the BxDiff consortium and similar commercial products may provide equivalent results.

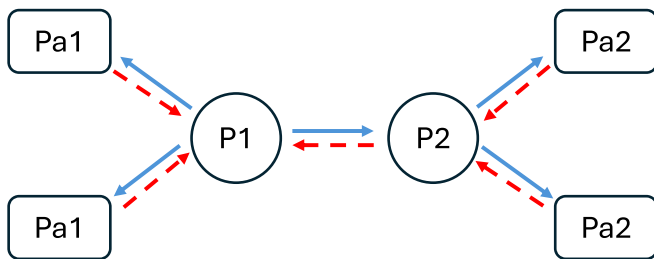


Figure 2. Schematic of the nominal star-type distribution of sample sets between pilots (Px) and non-pilot participants (Pax) in each comparison. The solid blue arrow shows the distribution of one sample set after the first measurement, and the dashed red arrow represents the return of that sample set after the second measurement, followed by another repeated measurement by the original distributor. Deviations occurred in some cases due to technical difficulties.

detection geometries were chosen as $(\theta_t, \varphi_t) = (0^\circ\text{--}80^\circ, 0^\circ)$ with angular step of $\Delta\theta_t = 5^\circ$.

To minimise orientation-dependent errors, the mentioned azimuthal directions for samples A and D were carefully determined prior to the measurements. In figure 3, scattering patterns for these two sample types are shown. They were collected on a half-transparent diffuse screen behind the sample by a camera, which was aligned to the detector scan plane beforehand. From images consecutively acquired while rotating the sample around its normal, the optimal azimuthal orientation with respect to the laboratory coordinates was

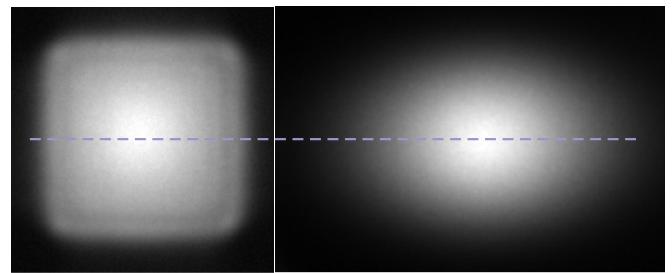


Figure 3. Scattering patterns of sample types A (left) and D (right), with the dashed line indicating the $\varphi_t = 0^\circ$ azimuth angle/detection scan plane.

determined and marked. For sample A, the zero azimuth was defined by aligning the upper and lower edges of the square-shaped pattern to the detector scan plane, as shown in the left image in figure 3, with the dashed line indicating the scan plane. For sample D, the zero azimuth was determined when the major axis of the elliptical scattering pattern was made parallel to the scan plane. To provide identical situations for the measurements to be carried out by different participants, the orientation-independent samples are also marked with a black dot on the back side of the sample for an arbitrarily chosen azimuth. In this way, the $\varphi_t = 0^\circ$ azimuth is indicated by a (virtual) line connecting the sample geometrical centre and the marking dot. For square samples the $\varphi_t = 0^\circ$ azimuth is referred to one specific sample edge.

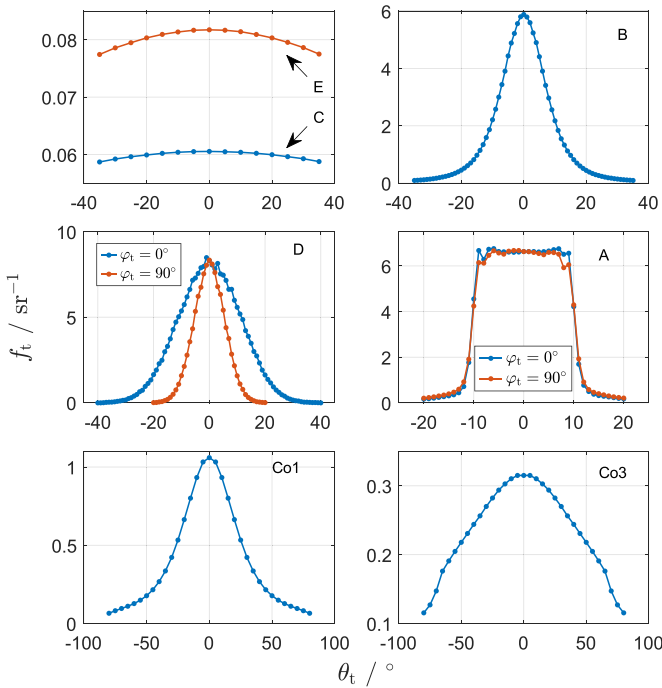


Figure 4. Angular transmission characteristics of all sample types used in the two comparisons. (Negative polar angles indicate azimuthal orientation $\varphi_t = 180^\circ$.) The azimuthal dependence of samples A and D was analysed by the different angular transmission characteristics measured at two perpendicular azimuthal directions.

The influence of wavelength was dealt with as follows. In comparison one, BTDF values were determined for nominal wavelengths of 633 nm and 445 nm. In comparison two, the measurand was defined for the wavelength 633 nm. If the required wavelength was not accessible experimentally, a correction with respect to the sample spectral dependence was performed. This will be discussed in more detail in section 2.3 and in section S3 in the supplementary material (SM).

Figure 4 exemplifies the BTDF of all sample types. The two quasi-Lambertian samples C and E exhibit a slowly varying BTDF profile with a drop of approximately 10 % at $\theta_t = 35^\circ$, but differ in amplitude and slightly in shape. The BTDF of sample B shows a much narrower angular distribution of approximately 16° FWHM with a peak at $\theta_t = 0^\circ$ and a much higher maximum BTDF value. Sample D exhibits a Gaussian shaped BTDF profile with a width varying with the azimuth angle. The FWHM of the profile is approximately 26° at $\varphi_t = 0^\circ$ and 13° at $\varphi_t = 90^\circ$. Sample A shows distinctively almost constant BTDF values in the central angular range and a steep drop from maximum to nearly zero at around $\theta_t = 10^\circ$. Samples Co1 and Co3, which only differ in their thickness (1 mm and 3 mm, respectively), present Gaussian shaped BTDF distributions with different widths of around 50° and 130° and maximum BTDF values around 1 sr^{-1} and 0.3 sr^{-1} .

The large variation in scattering characteristics indicates the need for applying different measurement conditions to properly measure the different types of samples. Especially for samples A, B, and D, a high detection angular resolution was

recommended in the protocol to determine more accurately the rapidly changing BTDF values. For the Lambertian-like distributions of samples C and E, and for the wide Gaussian shaped distributions of samples Co1 and Co3, a lower detection angular resolution is suitable, which is also beneficial to measure the overall lower BTDF level as more scattered radiation is collected by a larger solid angle.

2.3. Description of measurement instruments

A brief description of the different measurement instruments used in both comparisons is given in the following section. More details of the participants' instrumentation are provided in section S1 in the SM.

The basic parameters of the instruments involved in comparison one are presented in table 2.

Measurements at PTB were performed on the modified NaNoRef setup, which is originally the national reference standard for specular reflection. Some detail about the original facility can be taken from [11, 12] and the modified setup used for the BTDF measurement is reported in detail in [9]. In brief, the samples mounted on several motorised translational and rotary stages were irradiated by collimated laser beams at wavelengths of 642 nm and 445 nm with a spatially uniform and speckle-reduced profile. The imaging detection system allowed different sizes of the measurement area A_M to be realised. All components of the detection system were mounted on another independent rotary arm, which rotates around the centre of the sample back surface at a distance of 494.5 mm to achieve different detection polar angles for the angle-resolved BTDF measurement.

The measuring system at DFM comprised a CW laser operating at 663 nm, which was weakly focused for the irradiation on the sample. The BTDF was measured at two orthogonal polarisations, and the average was reported. The detector with a detection aperture was mounted on a rotary arm, which rotates at a distance of 355 mm around the sample. For the nominal wavelengths prescribed in comparison one, PTB's and DFM's data were corrected using the angle-dependent spectral transmittance of all sample types as taken by extra measurements at PTB [9]. Details regarding the correction can be found in section S3 in the SM.

The facilities of CNAM, CMI, and MSL performed the measurement in a similar way. The monochromatic irradiation on all instruments was achieved by spectrally selecting broadband radiation with different spectral bandwidths. At CNAM a depolariser was used in irradiation, whereas CMI and MSL performed the measurement under polarised irradiation. The samples were held by a robot arm (CNAM and CMI) or mounted on motorised stages (MSL) and the detection system was mounted on a motorised rotation ring with different diameters on all three facilities. All detection systems consisted of an imaging mirror and diaphragms of different sizes so that the detection angular resolution could be varied. The size of the measurement area A_M on the sample back surface could be defined using the imaging mirror. The distance from the sample to the detection aperture varied for these facilities. CNAM has the largest distance among all participants,

Table 2. Basic parameters of the measurement instruments and assignment of the participants' roles in comparison one.

Participant, role ^a	Irradiation source		Aperture angle (full)		Measurement area ($\theta_t = 0^\circ$) ^b	Distance sample to detector	Detection system	Measurement method	Measurement scheme
	Type & Wavelength	Bandwidth	Irradiation	Detection ^b					
PTB P1	642 nm diode laser & 445 nm solid-state laser	0.5 nm (642 nm)/ 1 nm (445 nm)	0.1°	0.5°/1°/1.5°	50.3 mm ² /75.4 mm ² / 265.9 mm ²	494.5 mm	Si-photodiode on integrating sphere	Absolute	Under-irradiated
DFM P2	663 nm CW laser	1.2 nm	N/A	0.8°	27 mm ²	355 mm	Si-photodiode	Absolute	Under-irradiated
CMI Pa1	Monochromatic 633 nm & 445 nm, from broadband source	5 nm	0.66°	2.1°	N/A	462.2 mm	Si-photodiode on integrating sphere	Absolute	Under-irradiated
MSL Pa1	Monochromatic 633 nm & 445 nm, from broadband source	1.5 nm	N/A	0.9°/1.9°	19.6 mm ² / 50.3 mm ² /63.6 mm ²	500 mm	Broadband Si-photodiode	Absolute	Under-irradiated
CNAM Pa2	Monochromatic 633 nm & 445 nm, from broadband source	2 nm ^c /5 nm	0.2°–0.35°	0.8°/1°/2°	28.3 mm ² /78.5 mm ²	1150 mm	Photodiode	Absolute	Under-irradiated
Aalto Pa2	Monochromatic 633 nm & 445 nm, from broadband source	5 nm/4 nm ^d	N/A	3°/8° ^d	N/A	472.9 mm/ 130 mm ^d	Si-photodiode	Absolute/ relative ^d	Under-irradiated

^a Px = pilot x, Pax = participant in the sub-group of pilot x.

^b For some participants, this parameter varies depending on sample scattering characteristics.

^c Only used in the measurement of sample type A at 445 nm.

^d Only used in the measurement of sample type E.

while CMI and MSL applied distances similar to PTB. More details about the involved facilities are presented in [13, 14] for CNAM and MSL, respectively.

At Aalto, the measurements of samples A, B, C, and D were performed on the absolute goniospectrophotometer. The samples were held horizontally, and the irradiation and detection systems were mounted on two independent motorised rotary stages. The samples were irradiated with a monochromatic radiation which was spectrally selected from a broadband light source, and the optical signal was detected by a Si-photodiode. Details of this setup are described in [15]. Sample E was measured on the transfer standard goniospectrophotometer applying a much larger solid angle. This measurement used the relative method by measuring against a standard calibrated on Aalto's absolute setup. Therefore, the uncertainties of sample E turned out to be larger than the uncertainties of other samples.

The basic parameters of the instruments involved in comparison two are presented in table 3.

At CSIC, BTDF was measured using the goniospectrophotometer described in [16] and [17]. It was originally conceived for BRDF measurements and was adapted for BTDF measurements. The samples were irradiated using broadband irradiation with high directionality and uniformity achieved by using a Köhler optical system. A spectroradiometer was used for detection, providing spectral information from 380 nm to 780 nm. A relative measurement was performed in the over-irradiated scheme, using a diffuse reflectance standard as reference, whose BRDF had been previously calibrated in the standard geometry $0^\circ:45^\circ$.

The measuring system at Covestro consisted of a halogen lamp, providing a band-pass filter for spectral evaluation at 632 nm. The measurement was performed in the relative mode referring to a PTFE reflection standard in the under-irradiated scheme.

At Saint-Gobain, BTDF was measured with an OMS4 goniospectrophotometer commercialised by OPTIS. The OMS4 setup and its usage for BTDF measurements is described in [18]. For this work, a broadband source (Xe-lamp) filtered at 635 nm was used for sample illumination. The signal was detected using a photomultiplier tube.

A commercial instrument was used by Temicon to perform BTDF measurements with high resolution stepped detector and collimated illumination.

A spectrally broad illumination (Xe-lamp), modified by a bandpass filter (VIS region) and collimating optics was used at KU Leuven. The detection was a CCD-based spectrograph with variable integration time, in combination with ND filters in the optical path results in a high dynamic range of 6 decades. The measurement was relative using a reference tile (primed Barium Sulphate reflection standard—PTB), and was carried out under-irradiated. This instrument is described in detail in [19].

The BTDF measurement setup at RISE used a laser-driven light source with collimating optics, creating an irradiation spot of 10 mm in diameter. The primary collector was a 4 inch integrating sphere with a silicon detector, positioned at a distance of 500 mm from the sample. The spectral BTDF was

determined using an interference filter. The measurement was performed in the under-irradiated scheme.

2.4. Methodology of the comparisons

The evaluation of both comparisons follows the instructions in [20] (procedure A) together with the Guidelines for CCPR Key Comparison Report Preparation using the cut-off-assisted Mandel-Paule method [21, 22]. The BTDF is assumed to be independently measured by all participants and is compared in consideration of inter-sample-set variations.

Starting from the BTDF values reported by each non-pilot participant $f_{k, \text{par}}$ (k is the sample set number) with their associated standard uncertainties $u(f_{k, \text{par}})$ and the BTDF values measured by pilot one, f_{k, p_1} , and pilot two, f_{k, p_2} , with their standard uncertainties $u(f_{k, p_1})$ and $u(f_{k, p_2})$, one specific sample set is chosen to be the reference set, to which the data from all participants are normalised in the way as shown in equations (6) and (7), assuming that the reference set was measured by pilot 1 (f_{ref, p_1}). Note that the slash '/' in the subscript of some variables in the following discussion indicates that the same formula can be applied to either the variable with the subscript before '/' or the variable with the subscript after '/'.

$$\begin{aligned} f_{k, \text{par}/p_1}^{\text{norm}} &= f_{k, \text{par}/p_1} \cdot \frac{f_{\text{ref}, p_1}}{f_{k, p_1}}, \text{ sample set } k \text{ measured by pilot 1} \\ f_{k, \text{par}/p_2}^{\text{norm}} &= f_{k, \text{par}/p_2} \cdot \frac{f_{\text{ref}, p_1}}{f_{k, p_2}} \cdot \frac{f_{k^*, p_2}}{f_{k^*, p_1}}, \text{ sample set } k \text{ measured by pilot 2} \end{aligned} \quad (6)$$

$f_{k^*, p_{1/2}}$ is the value of the additional pilot-comparison set k^* measured by pilot 1/2, which helps connect the measurement scales between their sub-groups. Identical normalisation is also performed for the uncertainties:

$$\begin{aligned} u(f_{k, \text{par}/p_1}^{\text{norm}}) &= u(f_{k, \text{par}/p_1}) \cdot \frac{f_{\text{ref}, p_1}}{f_{k, p_1}}, \text{ sample set } k \text{ measured by pilot 1} \\ u(f_{k, \text{par}/p_2}^{\text{norm}}) &= u(f_{k, \text{par}/p_2}) \cdot \frac{f_{\text{ref}, p_1}}{f_{k, p_2}} \cdot \frac{f_{k^*, p_2}}{f_{k^*, p_1}}, \text{ sample set } k \text{ measured by pilot 2} \end{aligned} \quad (7)$$

In comparison one, the reference set is selected as the pilot-comparison set, i.e. $f_{k^*, p_{1/2}} = f_{\text{ref}, p_{1/2}}$. In this way, the above equations for $f_{k, \text{par}/p_2}^{\text{norm}}$ and $u(f_{k, \text{par}/p_2}^{\text{norm}})$ are reduced to the first case in equations (6) and (7).

After being normalised, the data of multiple sample sets for each pilot are further reduced to one data set by means of the arithmetic mean. In the end, only one data set ($f_n^{\text{norm}}, u(f_n^{\text{norm}})$) per participant n contributes to the determination of the reference value R of the comparison. A provisional R for every sample type in every geometry is calculated as the weighted mean of all participants' data:

$$R = \frac{\sum_{n=1}^N w_n f_n^{\text{norm}}}{\sum_{n=1}^N w_n}, \quad (8)$$

Table 3. Basic parameters of the measurement instruments and assignment of the participants' roles in comparison two.

Participant, role ^(a)	Irradiation source			Aperture angle (full)		Measurement area ($\theta_i = 0^\circ$)	Distance sample to detector	Detection system	Measurement method	Measurement scheme
	Type & Wavelength	Bandwidth	Polarisation	Irradiation	Detection					
CSIC P1	Xe-lamp, broadband irradiation	N/A	Unpolarised	707 mm ²	1.5°	5°	38.5 mm ²	Spectro-radiometer	Relative	Over-irradiated
Covestro P2	632 nm Halogen light source (100 W) with band filter	5 nm	Unpolarised	12.5 mm ²	N/A	2.2°	50.2 mm ²	Integrated flux detection	Relative	Under-irradiated
Saint-Gobain Pa1	635 nm, Xe-lamp	10 nm	Unpolarised	50 mm ²	0.2°	0.2°	50 mm ²	Photomultiplier	Relative	Under-irradiated
Temicon Pa1	633 nm diode laser	N/A	<i>s</i> & <i>p</i>	50.3 mm ²	$\ll 1^\circ$	$< 0.05^\circ$	N/A	Photodiode with AD converter	Relative	Under-irradiated
KU Leuven Pa2	Xe-lamp, broadband irradiation	3 nm	Unpolarised	132.7 mm ²	0.1°	1.6°	706.9 mm ²	Spectrograph + CCD	Relative	Under-irradiated
RISE Pa2	Broadband irradiation source with bandpass filter	10 nm	Unpolarised	78.5 mm ²	N/A	2.9°	176.7 mm ²	Si-photodiode on integrating sphere	Relative	Under-irradiated

^a Px = pilot x, Pax = participant in the sub-group of pilot x.

with the weights:

$$w_n = u^{-2}(f_n^{\text{norm}}), \quad (9)$$

and the standard uncertainty of the reference value R :

$$u(R) = \sqrt{\frac{1}{\sum_{n=1}^N w_n}}. \quad (10)$$

By applying a chi-squared (χ^2) test, the overall consistency of the results can be checked:

$$\chi_{\text{obs}}^2 = \sum_{n=1}^N \frac{(f_n^{\text{norm}} - R)^2}{u^2(f_n^{\text{norm}})}. \quad (11)$$

If more than 95 % of the calculated χ_{obs}^2 are smaller than the χ^2 distribution with $N - 1$ degrees of freedom, i.e. 11.07 for $\chi_{0.05}^2(5)$ for most of the cases in both comparisons and 9.488 for $\chi_{0.05}^2(4)$ for one exceptional case in comparison one, the measured data can be regarded as consistent and the degree of equivalence (DoE) for participant n is expressed as $(d_n, U(d_n))$:

$$\begin{aligned} d_n &= f_n^{\text{norm}} - R, \\ U(d_n) &= 2\sqrt{u^2(f_n^{\text{norm}}) - u^2(R)}, \end{aligned} \quad (12)$$

which indicates the consistency of the data provided by each participating lab to the reference value R depending on the relation between $|d_n|$ and $U(d_n)$. Consistency is achieved when 95 % of the data fulfil $|d_n| < U(d_n)$. If more than 5 % of the data show $|d_n| > U(d_n)$ or $|d_n| \ll U(d_n)$, this indicates either inconsistent measurement data, or overestimated uncertainties.

If the χ^2 test fails, the participants' uncertainties $u(f_n^{\text{norm}})$ are adjusted by introducing a cut-off value $u_{\text{cut-off}}$:

$$\begin{aligned} u_{\text{cut}}(f_n^{\text{norm}}) &= u(f_n^{\text{norm}}), \text{ for } u(f_n^{\text{norm}}) \geq u_{\text{cut-off}} \\ u_{\text{cut}}(f_n^{\text{norm}}) &= u_{\text{cut-off}}, \text{ for } u(f_n^{\text{norm}}) < u_{\text{cut-off}} \end{aligned} \quad (13)$$

which is calculated as the following:

$$\begin{aligned} u_{\text{cut-off}} &= \text{mean}\{u(f_n^{\text{norm}})\}, \\ \text{for all } u(f_n^{\text{norm}}) &\leq \text{median}\{u(f_n^{\text{norm}})\}. \end{aligned} \quad (14)$$

Then, a new reference value is calculated by repeating the process from equations (8) to (11) to check whether the global consistency is achieved with slightly larger $u_{\text{cut}}(f_n^{\text{norm}})$ for some participants. If the consistency condition is still not fulfilled, the uncertainties are further adjusted by taking into account an additional interlaboratory variance s^2 [23]:

$$u_{\text{mp}}(f_n^{\text{norm}}) = \sqrt{u_{\text{cut}}^2(f_n^{\text{norm}}) + s^2}. \quad (15)$$

The Mandel-Paule adjusted uncertainties $u_{\text{mp}}(f_n^{\text{norm}})$ now replace the uncertainties $u(f_n^{\text{norm}})$ in equations (8)–(11) with iteratively increasing s until the χ^2 test passes, and the new DoE is calculated.

3. Results of comparison one

In this section, the results of comparison one are presented and discussed. Note that the measured BTDFs for the same sample type may deviate in both comparisons, this could be caused either by different measurement conditions of the instruments involved, or by inter-sample variation. The normalised reported data can be found in section S2 in the SM.

3.1. Consistency of reported data

Figure 5 illustrates the χ_{obs}^2 values of every sample type in every geometry at both wavelengths for the initially reported measurement data. The black line in the subfigures indicates the 95 % quantile of the $\chi^2(N - 1)$ distribution, resulting from the corresponding degrees of freedom. Note that there are only 5 data sets for the narrower BTDF distribution of sample D and 6 data sets for the other samples. The uncertainty components which have been taken into account by each participant are summarised in table 4. ‘+’ and ‘−’ sign indicate whether the component has been considered or not in the uncertainty budget by that participant and ‘N/A’ stands for ‘not applicable’ since some uncertainty components do not concern all participants. The main reasons for the observed inconsistencies for each of the samples are discussed in the following paragraphs.

3.1.1. Samples C and E. The measurement data are in general more consistent at the longer wavelength than at the shorter one, especially for the two quasi-Lambertian samples C and E. For the latter, global consistency is achieved in every geometry at 633 nm. At 445 nm, the data are also consistent in more than half of the geometries, even though the measurements at 445 nm are more difficult due to a lower optical transmitted signal and detector response. Observations showed that sample E, being a thin foil, exhibited some mounting issues due to non-flatness. Even better congruence could be achieved if all participants had reacted on this, either by increasing the uncertainty contribution to the detection solid angle or by applying a correction on the measured BTDF value. In light of the flatness issue, it is surprising that the data for sample E show better consistency than for the rigid bulk type sample C. Possible explanations for the lower consistency of sample C could be a spatial inhomogeneity of the sample (already described in [24] for natural SiO₂ volume diffusers, more details in section S5 of the SM) and a strong lateral diffusion of the incident beam. Both lead to an enhanced dependence of the BTDF on the measurement parameters, especially the area sizes of irradiation A_i and measurement A_M .

The stronger lateral scattering by sample C compared to sample E can be observed by evaluating the spatial intensity distribution on the sample back surface taken by a high-resolution camera, aligned perpendicularly to the incident beam path. The photos of both samples irradiated with a laser beam of 5 mm diameter are shown in figure 6, with red circles indicating the incident beam area A_i . Due to the large difference in the thickness and the material's scattering magnitude

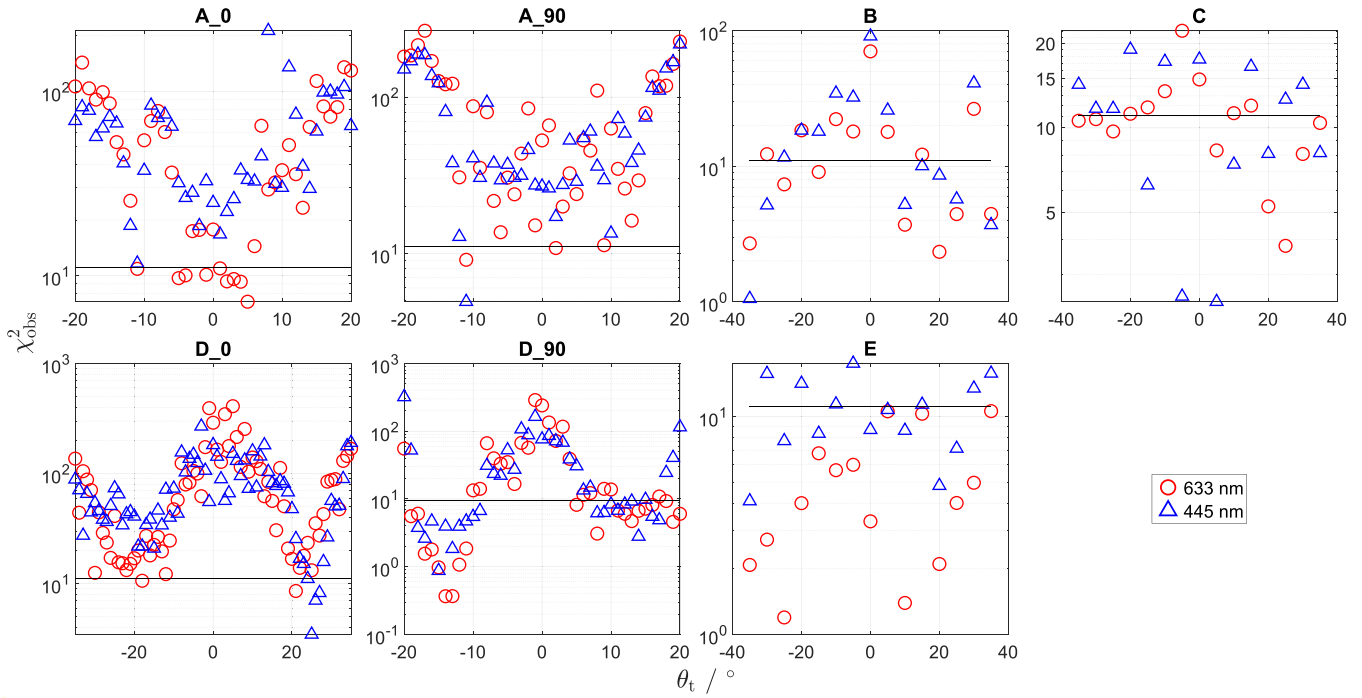


Figure 5. χ^2_{obs} values for all sample types at 633 nm and 445 nm for the initially reported results (details see text) in comparison one. The black line in each subfigure indicates the 95 % quantile of χ^2 distribution for the corresponding degrees of freedom. χ^2_{obs} values below the black line imply a global consistency for the results measured in those geometries by all participants, whereas χ^2_{obs} values above the black line indicate an inconsistency in the reported data and further procedures to reduce the inconsistency are performed as described in the text.

of these two sample types, the size of the irradiation spot increased to about 7.5 mm in diameter on the back surface of sample C, whereas on sample E the size of the spot almost does not change after being transmitted.

On the instruments of some participants, A_M is well-defined on the sample back surface by an imaging system. For measurements with an undefined A_M , the risk of detecting stray light apart from the scattered light is high. But even for a well-defined A_M , if it is only slightly larger than A_i , some loss in the measured BTDF is unavoidable as the diffusely transmitted radiation could no longer be fully collected within A_M . In this case the measurement scheme could no longer be regarded as under-irradiation, nor as over-irradiation, since the intensity distribution of the diffused spot may not be spatially homogeneous. Therefore, it is necessary to use an area A_M being sufficiently larger than A_i when measuring a thick bulk scattering sample, so that the whole diffused spot can be measured. On the other hand, A_M should not exceed the physical boundary of the sample as it increases with higher detection polar angle θ_t . This makes the selection of proper measurement parameters important. Their influence is illustrated in more detail in section S4 in the SM by a measurement on sample C with different A_M . While all participating labs in comparison one performed the measurement in the under-irradiated scheme, the relation between A_i and A_M varies for each facility. However, only PTB considered the lateral diffusion of the irradiation spot in the uncertainty budget and provided larger uncertainties for this sample than for the others.

3.1.2. Sample B. The χ^2 test for sample B shows consistency in only half of the geometries. At both wavelengths the inconsistency increases at smaller θ_t angles and becomes the largest at the peak of the BTDF distribution. The latter observation can be explained by the different detection angular resolution used on different instruments (see table 2). In this geometry, participants applying a high angular resolution tend to measure higher BTDF values, which more closely reproduce the true BTDF value, whereas a low resolution results in smaller values. This convolution effect can be regarded in analogy to the influence of the instrumental bandpass function in spectral measurements, as described in [25]. For the measurement facilities involved in comparison one, the detection angular resolution can almost be represented by the FWHM of the instrument function, which is determined by the size and divergence of the irradiation beam and the detector solid angle. It can be measured as the angular spread of the incident beam without the presence of a sample. The measured BTDF distribution is always the true BTDF distribution convolved with this instrument function [7]. For peak-shaped distributions (such as sample types B and D), the instrument function lowers the peak and broadens the width. Its impact is proportional to $\frac{\partial^2 f_t}{\partial \theta_t^2}$. If experimentally feasible, the instrument function should be adapted to the expected angular width of the true distribution of the sample under test. For Gaussian-shaped distributions, the FWHM of the instrument function can be recommended to be about 1/20 of the FWHM of the sample's true BTDF distribution, so that the relative difference in the peak value can be under 0.2 %, as long as the sensitivity of

Table 4. Uncertainty components considered by each participant in comparison one. ‘+’ means ‘considered’ and ‘–’ means ‘not considered’, ‘N/A’ means ‘not applicable’.

Participant	Irradiation		Detection		Signal acquisition			Lateral diffusion of irradiation beam ^c								
	Wavelength	Beam spatial nonuniformity	Angle positioning	Solid angle	Angular resolution	Signal readings ^a	Detector linearity	Digitisation error	Polarisation	Attenuation filter ^b	Sample inhomogeneity	Inter-reflection	Stray light	Temperature	Wavelength correction ^d	Sample azimuth ^e
PTB	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+	+
DFM	–	–	+	+	+	+	+	–	–	+	–	–	+	–	+	–
CMI	+	–	+	+	–	+	+	–	+	+	–	–	+	–	N/A	–
MSL	+	+	+	+	+	+	+	–	+	+	+	+	+	+	N/A	+
CNAM	–	–	+	+	^f	+	+	–	N/A	N/A	–	–	–	–	N/A	–
Aalto	+	–	+	+	–	+	+	–	+	–	+	–	+	–	N/A	–

^a Including fluctuation, drift, and noise for incident, transmitted, and dark signals.
^b For CMI and MSL, the gain ratio of the amplifier for signal acquisition.
^c Only observed on sample type C due to thick bulk volume.
^d Only concerns PTB and DFM, other participants measured at the nominal wavelengths.
^e Only concerns sample types A and D.
^f Only applied for sample type A.

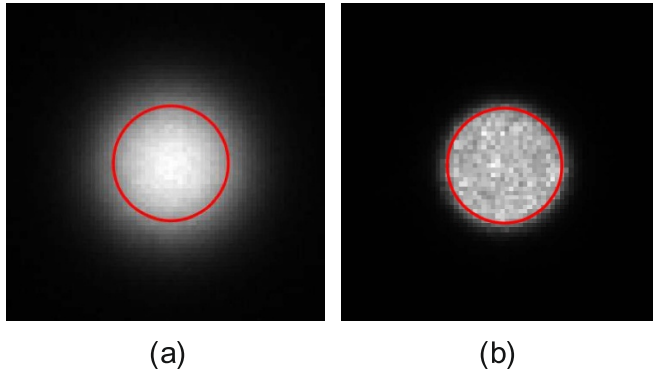


Figure 6. Photos of the back side of (a) sample C and (b) sample E irradiated with laser beam of 5 mm in diameter (indicated by a red circle). It can be seen that the irradiation beam was transmitted with strong lateral scattering due to the thick bulk volume of sample C, resulting in an obvious size difference between the spot on the front and the back side of the sample.

the instrument is high enough for an acceptable signal-to-noise ratio [26].

In comparison one, sample B was measured with detection angular resolutions ranging from 0.8° to 3° . The FWHM of the sample's true BTDF distribution is approximately 16° , corresponding to a recommended instrument function of approximately 0.8° FWHM for a moderate measurement uncertainty. If the angular resolution is too low, a correction on the measured distribution or increased measurement uncertainties would be necessary for a proper comparison. The impact of doing so is exemplified in figure 7 by performing such a correction on the measured BTDF distribution of sample B with an instrument function of 1° and 3° FWHM, respectively. Compared to the originally measured BTDF distribution, the deconvolved BTDF shows about 2 % difference at $\theta_t = 0^\circ$ and $\theta_t = 18^\circ$ when measured with the lower angular resolution. On the other hand, if the measurements are made with the higher resolution, the difference stays reasonably small for every θ_t angle. This highlights the importance of taking the instrument function into account when evaluating the measurement uncertainty. However, not all participants included the influence of the instrument function in their uncertainty budgets or corrected their measurement results accordingly, which contributed to the large deviations observed for the smaller θ_t angles for sample B.

3.1.3. Sample D. For sample D, the instrument function influences the measurement in a similar way. In the wide orientation ($\varphi_t = 0^\circ$) the impact is less pronounced than in the narrow orientation ($\varphi_t = 90^\circ$). This is discussed in detail in the SM (section S6). Another aspect regarding the applied angular resolution, which was observed in all PTB's measurements at the wide orientation, is the spiky behaviour in the central angular area of the BTDF distribution (see figures S12 and S13 in the SM). It is assumed that the scattering by structures on the holographic layer, causing this spiky behaviour, was resolved only by PTB's highest applied detection angular resolution

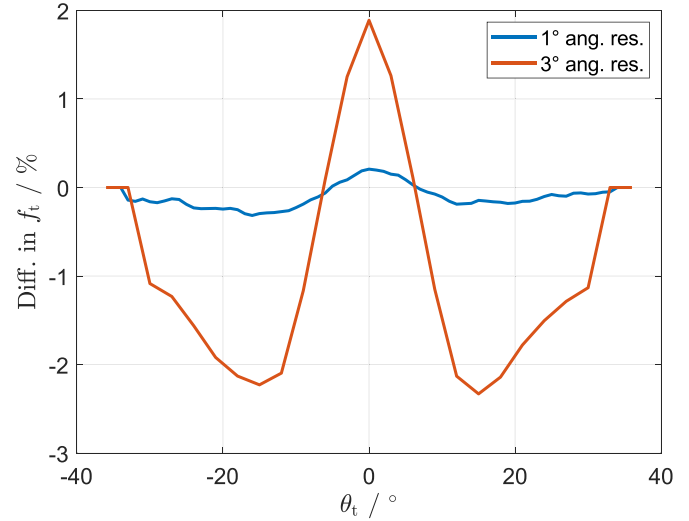


Figure 7. Difference between the measured and the corrected BTDF distribution of sample B regarding the applied instrument function of 1° (blue) and 3° (red) FWHM. This highlights the importance of taking into account the influence of the applied instrument function for a proper comparison.

of 0.5° . This again emphasises the necessity of having well-defined measurement parameters for a future key comparison.

Apart from the instrument function, a limited accuracy in realising the azimuthal orientation can also strongly contribute to the measurement uncertainty of sample D. As outlined in section 2.2, the orientation of samples was pre-determined and marked. If the uncertainty in the sample azimuth alignment is approximately 1° , the associated uncertainty due to this misalignment can be up to 0.5 % to the BTDF. However, this uncertainty source was only considered by PTB and MSL in the uncertainty analysis. This might explain why the inconsistency of sample D is larger than that for sample B, because sample D is much more sensitive to uncertainties regarding the azimuthal angle.

3.1.4. Sample A. Orientation-related uncertainties might also explain the data inconsistency observed with sample A. It is supposed that the BTDF of sample A, which exhibits a square-shaped top-hat distribution, should be independent of φ_t for θ_t angles close to 0° . On the other hand, the width of the top-hat part of the BTDF distribution would be larger for φ_t deviating from the correct value. Thus, the inconsistency is large at the falling edges ($9^\circ < |\theta_t| < 11^\circ$). In the central angle range, the reported data are only consistent for $\varphi_t = 0^\circ$ at 633 nm, but not for $\varphi_t = 90^\circ$. This could be attributed to the micro-lens units on the sample's functional surface, which generate an imperfect top-hat BTDF distribution with slight variation in the central angular range. Therefore, the measured BTDFs in this range do depend on φ_t . Again, the uncertainty regarding the sample azimuth was only considered by two participants, so this likely contributed to the inconsistency that was observed for $\varphi_t = 90^\circ$. More detail about this is given in section S7 of the SM.

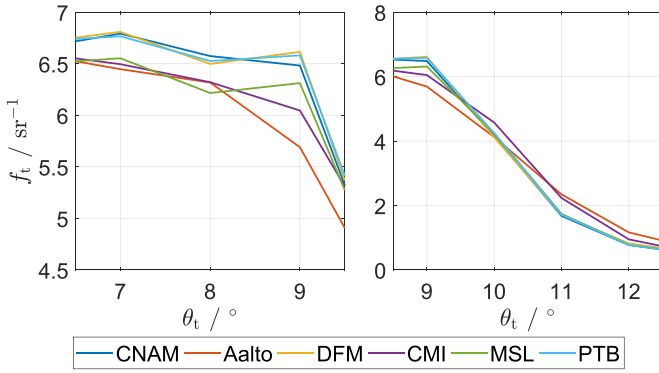


Figure 8. Different angular ranges of normalised BTDF distribution of sample A for $\varphi_t = 0^\circ$ at 633 nm measured by all participants in comparison one. It can be observed that participants who applied a high detection angular resolution could measure the sharp variation in the BTDF distribution of sample A, indicated by the similar shape of the DFM, MSL and PTB curves in both angular ranges.

The instrument function influences the measurement of sample A in the way that the sharp variation ranging from $\theta_t = 7^\circ$ to 9° ('batwing') and from 9° to 12° (falling edge) in the BTDF distribution is modified (figure 8). The variations in the batwings were resolved by CNAM, DFM, MSL, and PTB, who all applied similarly high angular resolutions. CMI and Aalto used a lower angular resolution. Thus, their measured BTDF distributions do not exhibit these structures. The observations for the falling edge of sample A's BTDF distribution also underline this issue, as the curves measured by CMI and Aalto exhibit a flatter slope than the other four participants. Therefore, it is not surprising that the global consistency is not achieved in these angular ranges.

3.2. Degrees of equivalence

Since the χ^2_{obs} calculated from the reported data does not fulfil the requirement $\chi^2_{\text{obs}} \leq \chi^2_{0.05}(N-1)$ for almost all of the sample types (except for sample E at 633 nm), the normalised uncertainties of all participants are first adjusted using the cut-off. After applying the cut-off, the data becomes consistent for sample C at both wavelengths, but for other samples the inconsistency is only slightly reduced. This implies a general underestimation of uncertainties, for which some reasons were given in the previous section. Therefore, the uncertainties were further increased using the Mandel-Paule method as described in section 2.4, although the additional interlaboratory-variance only accounts for shortcomings in the determination of uncertainty in an unspecified way. The procedure is performed until the χ^2 test passes for each geometry. For sample D at $\varphi_t = 0^\circ$ the data points at $|\theta_t| > 35^\circ$ are excluded in the Mandel-Paule adjustment since the BTDF in these geometries is almost zero. The development of each participant's uncertainties regarding the cut-off and the Mandel-Paule adjustment is plotted for each sample in section S8 in the SM.

The DoEs are then calculated using the metric $d_n/U(d_n)$ (simplified as d/U in the following text) for each participant after the uncertainty adjustment, and results are presented in

figures 9 and 10 for the comparison at 633 nm and 445 nm, respectively. For measurement data with reasonable uncertainties, the d/U metric of each sample should be scattered between -1 and 1 . For some participants, their d/U metric shows this behaviour after the uncertainty adjustment, while the d/U of other participants still indicates either a poor measurement, an under- or overestimated uncertainty, or a wrong basic assumption, for example, when distributions measured with considerably different detection angular resolution are compared.

In general, we can see that the d/U values for DFM are close to 0 for all samples at both wavelengths, suggesting that DFM overestimated all of their measurement uncertainties. In the following paragraphs, we consider the d/U metrics for each sample in turn.

3.2.1. Sample C. For sample C at 633 nm, the d/U metric of Aalto's data splits from the other participants, with the Aalto values being lower than the others. Since Aalto submitted low uncertainties for this sample, they would have had a large weight in the calculation of the reference value, dragging the reference value closer to their measurements. The deviation between Aalto and the other participants suggests that it is likely that Aalto underestimated their uncertainty.

3.2.2. Sample B. For sample B at 633 nm, the d/U values of all participants are scattered between -1 and 1 , with only a few lying outside this range. This suggests that the adjusted uncertainties are adequate to describe the deviations observed between the participants. The inequivalence of CNAM's data in the central angular range, with the d/U values above 1, might suggest a measurement problem, as the d/U metric for CNAM's measurements at other θ_t angles stays close to -1 . At 445 nm, PTB's data show inequivalence for negative θ_t angles but are equivalent for $\theta_t \geq 10^\circ$. This shows some asymmetry in the measurement, together with possibly overlooked uncertainty components. The influence of detection angular resolution is observed by Aalto's generally close to -1 d/U values at both wavelengths, due to Aalto's measured values being lower than other participants.

3.2.3. Sample D. For sample D, the equivalence is only achieved for DFM, CNAM, and CMI at the wide orientation even after the uncertainty adjustment. PTB tended to measure higher BTDF values in the central angular range, whereas MSL measured lower values. This may be explained by the difference in the detection angular resolution applied on different instruments. However, the uncertainties provided by both participants could not cover the difference between their data, leading to the poor consistency. Similar to PTB's data of sample B at 445 nm, the difference in the d/U metric of Aalto's data between positive and negative θ_t angles might imply some asymmetry in their measurement setup, along with generally understated uncertainties.

For the data of the narrow sample D orientation, inequivalence in PTB's data is observed in the central detection

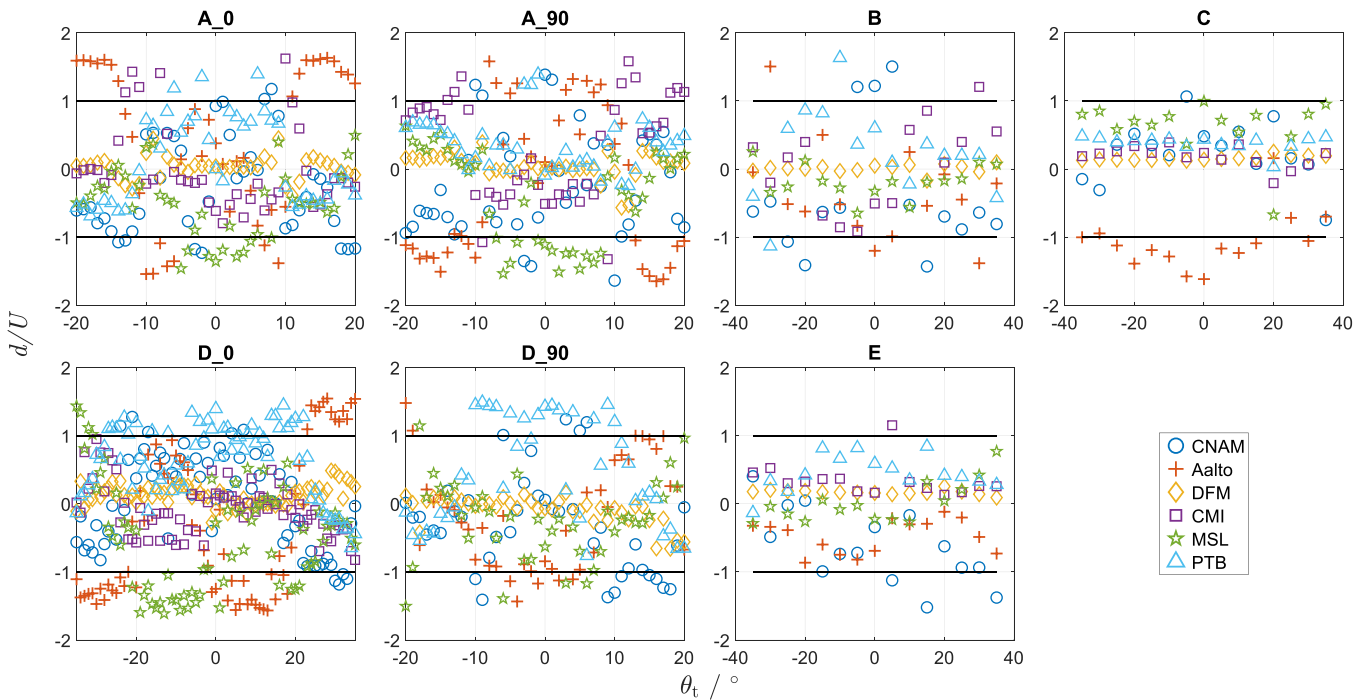


Figure 9. d/U metric for all participants in comparison one at 633 nm, after uncertainty adjustment using cut-off and Mandel-Paule method. Points randomly scattered within the -1 to 1 interval indicate equivalent measurements of the sample with adequate uncertainty analysis.

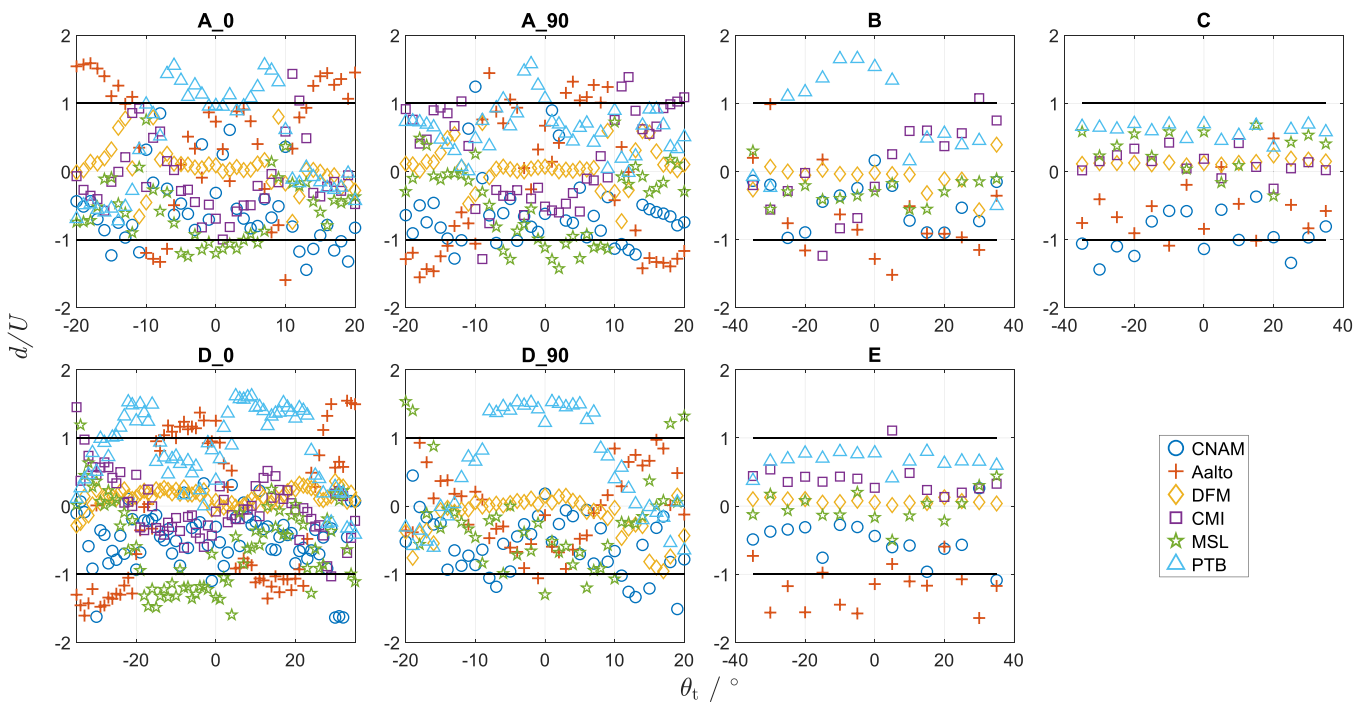


Figure 10. d/U metric for all participants in comparison one at 445 nm, after uncertainty adjustment using cut-off and Mandel-Paule method. Points randomly scattered within the -1 to 1 interval indicate equivalent measurements of the sample with adequate uncertainty analysis.

range, as PTB reported the highest BTDF values in this measurement. Apart from the influence of the instrument function as discussed in the previous section, another reason is that the measurements of the inter-pilot sample D failed to connect the lab scales between the two pilots and thus, the two

sub-groups of the comparison. Only for this specific sample, the BTDF value reported by DFM deviates from other samples of the same type and is much lower than the value reported by PTB, whereas PTB's measurements of different sample D did not reveal large inter-set variation. As a result, the

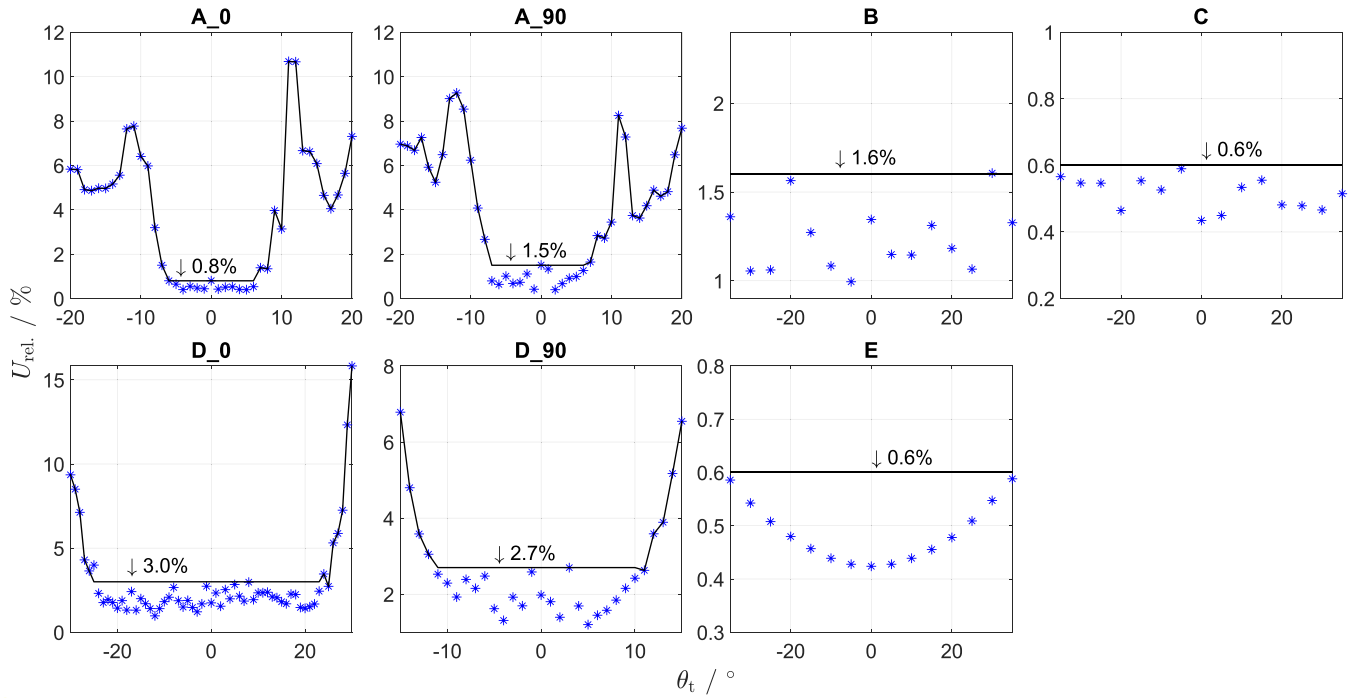


Figure 11. Relative standard ($k = 1$) uncertainty of every sample type for a fictive mean participant after applying cut-off and Mandel-Paule method. The fictive mean participant is taken on that number n of participants, who possess the smallest uncertainty for a specific sample type. In order of occurrence $n = 3, 3, 3, 2, 3, 4, 2$. In this way, the participants' potential to perform high quality BTDF measurements could be assessed despite that the agreement in comparison one is not sufficiently good.

normalisation incorrectly lowered the data from those participants in DFM's sub-group. In PTB's sub-group, on the other hand, CMI did not supply data for sample D at narrow orientation and MSL reported lower BTDF values. Therefore, only the data from PTB are left much higher while calculating the DoE.

3.2.4. Sample A. For sample A in both principal azimuths, measured at 633 nm, the equivalence of PTB's and CNAM's data is generally good. The inequivalence in CMI's and Aalto's data again indicates the problem of applying low detection angular resolutions to measure the sharp variations in the distribution. On the contrary, MSL's data show inequivalence at angles where the BTDF only exhibits slight variation. The d/U metric below -1 indicates generally low BTDF values. The tendency that MSL measured lower values whereas PTB did the opposite can only be understood as a systematic difference between their measurement scales. The type of irradiation source may be identified as a possible influence factor. For BTDF measurements, narrowband CW lasers were used at PTB, while a pulsed supercontinuum light source was used for irradiation in the longer wavelength range at MSL. With the latter source, ps-pulsed irradiation with high peak power is applied. However, possible influence on, for example, filter transmittance or other optical components was not studied.

For sample A at 445 nm, inequivalence is observed in PTB's data while CMI's data become equivalent. A possible explanation would be a problematic normalisation process caused by the absence of DFM measurements for this

wavelength. This is described in more detail in section S3 of the SM.

3.3. Discussion

In the previous sections (and in the SM), the measurement results and related uncertainty contributions are analysed sample-, instrument-, and geometry-specifically. The main effects that led to the observed inconsistencies between the involved participants are differences in the irradiation and measurement area sizes A_i and A_M , differences in the applied detection angular resolution, and misalignment regarding the samples' azimuthal orientation. These identified effects can be considered by participants in the future to perform improved BTDF measurements and to set up more reliable uncertainty budgets.

Even though the agreement in comparison one is not sufficiently good, it is possible to use the results from this comparison to assess the participants' potential to perform high quality BTDF measurements. This can be done by using only a subset of data to generate a so-called 'fictive mean participant', as shown in figure 11. The relative standard uncertainty of this fictive mean participant was calculated by taking the mean of the uncertainties of the labs with the smallest uncertainty values after applying the processes described in section 2.4. The sample-specific relative uncertainties for the fictive mean participant plotted in the subfigures range from 0.6 % to 3 %.

The lowest uncertainties can be achieved for samples C and E. The uncertainties for these samples are close to the

uncertainties reported in [2], indicating that at least a subset of participants, applying adequate experimental settings, is capable of performing equivalent measurements.

Another aspect for future improvement concerns the procedure of the comparison. Although some work was invested in setting up technical protocols, some measurement parameters were only specified as a recommendation, so as to not exclude project partners with less flexible setups from participating in this comparison. This has led, in some cases, to measurement results which are not comparable by principle, if resulting effects are not corrected. A prominent example is the detection angular resolution, which must be stated precisely in future comparisons.

Despite the observed shortcomings, the results from this comparison allow us to identify potential reference samples for BTDF measurements. A thin PTFE foil like sample E, being highly insensitive to varying experimental settings, may serve as a good candidate for a Lambertian laboratory standard. Azimuthally independent samples like sample B are capable of serving as narrow-angle references. Mie-scatter samples like sample C find frequent use in aerospace measurements due to their good physical properties and stability [27, 28]. However, the mentioned lateral diffusion effect should be taken into account when referring to the true BTDF value of this sample type or measurements must be performed under similar conditions.

Not surprisingly, the largest inconsistencies were observed for the samples with high azimuthal dependence. Besides the mentioned instrument function issue, the results imply that the involved participants must step up their efforts in defining and aligning to the principle-axes of the samples for future comparisons.

4. Results of comparison two

In this section the results of comparison two are presented and discussed. In order to distinguish between the errors introduced by angular evaluation and those from the absolute value assigned to the measurement, all the reported measurement results were further adjusted for having exactly the same reference value calculated for $\theta_t = 0^\circ$ geometry. This way, the deviations observed in other geometries are independent of the absolute calibration, but only include the other typical sources of uncertainties in goniospectrophotometric measurements. The absolute value of the measurements, required for transferring the scale, was provided by using a reflectance standard as reference by most of the participants. The method used here avoids including the deviations between standards. In addition, it excludes the issue with the different size of irradiated area that is explained later in this section.

The results of the BTDF measurements, after adjustments explained above and the evaluation procedure described in section 2.4, can be found in section S2 in the SM. In general, although there are large differences among the participants, it can be stated that the relative uncertainties are generally higher at steeper slopes in the BTDF distribution. The DoEs for the measurement of each participant are given in figure 12.

Measurements are consistent for smaller θ_t , and they become more inconsistent as θ_t increases. It is hard to draw a conclusion on the impact of the angular distribution of the BTDF. For instance, there is no significant difference between samples B (narrow distribution) and C (wide distribution). In general, we might say that the reported uncertainties are well estimated. The exception might be the measurements from Temicon. The large inconsistency might be due to an underestimation of the impact of the straylight in their measuring system. It is also noticeable that the data from Covestro and Saint-Gobain are extremely consistent for sample C, and slightly less for other samples. It could point to an overestimation of the reported uncertainty. But it might also be that the reported uncertainty at any detection angle is almost completely dominated by the uncertainty from the scale transfer at 0° . Note that Saint-Gobain and Covestro generally reported the largest uncertainties (see figure S15 in the SM).

As explained above, the error committed by transferring the scale at a known geometry was excluded in the previous analysis. This partially avoids considering the differences due to the different irradiated areas in the comparison. Its impact is explained and shown in the following lines.

According to the considerations in [29], the radiance of a translucent object, with high contribution of scattering within the bulk, is proportional to the incident radiant flux. When measuring the BTDF, the evaluated radiance L_t per unit of irradiance E_i increases for larger irradiated areas A_i because more radiant flux is entering the material. As a consequence, the BTDF is higher for larger A_i , and this effect is more significant when the contribution of the scattering in the bulk with respect to the scattering at the surface is larger.

Since each participant in this comparison kept a constant A_i for all measurements, and, in addition, each participant used a different value for this parameter, it is possible to study the dependence of the BTDF on it. This is shown in figure 13, where, for the sake of a clear visual comparison, the ratio of the measured BTDFs at $\theta_t = 0^\circ$ with respect to the BTDF measured with the largest A_i (at CSIC) is shown versus the irradiated area size used on the participating measuring systems (excluding Temicon, who did not report absolute BTDF values), for all samples involved in comparison two. It is observed that the ratio decreases consistently towards lower irradiated areas, as expected. In addition, the results seem coherent with the hypothesis that this effect is larger for samples possessing stronger scattering in the bulk. The lowest variation is observed for sample B, which presents mainly surface scattering and seems rather transparent. Samples E and Co1 show a larger and similar variation (around 15 %), as they are both volume scattering samples with small thicknesses (0.25 mm and 1 mm, respectively). The largest variation (almost 30 %) is observed for sample Co3, which has the same material as Co1 but with a larger thickness (3 mm), and also for sample C, which also has a thick bulk scattering volume (2 mm). These results show the importance of an adequate irradiated area for having a well-defined BTDF measurand. For over-irradiated measurements, the irradiated area has to be large enough to avoid variation in the measurement caused by different sizes of A_i . For under-irradiated measurements, the measurement area A_M needs to

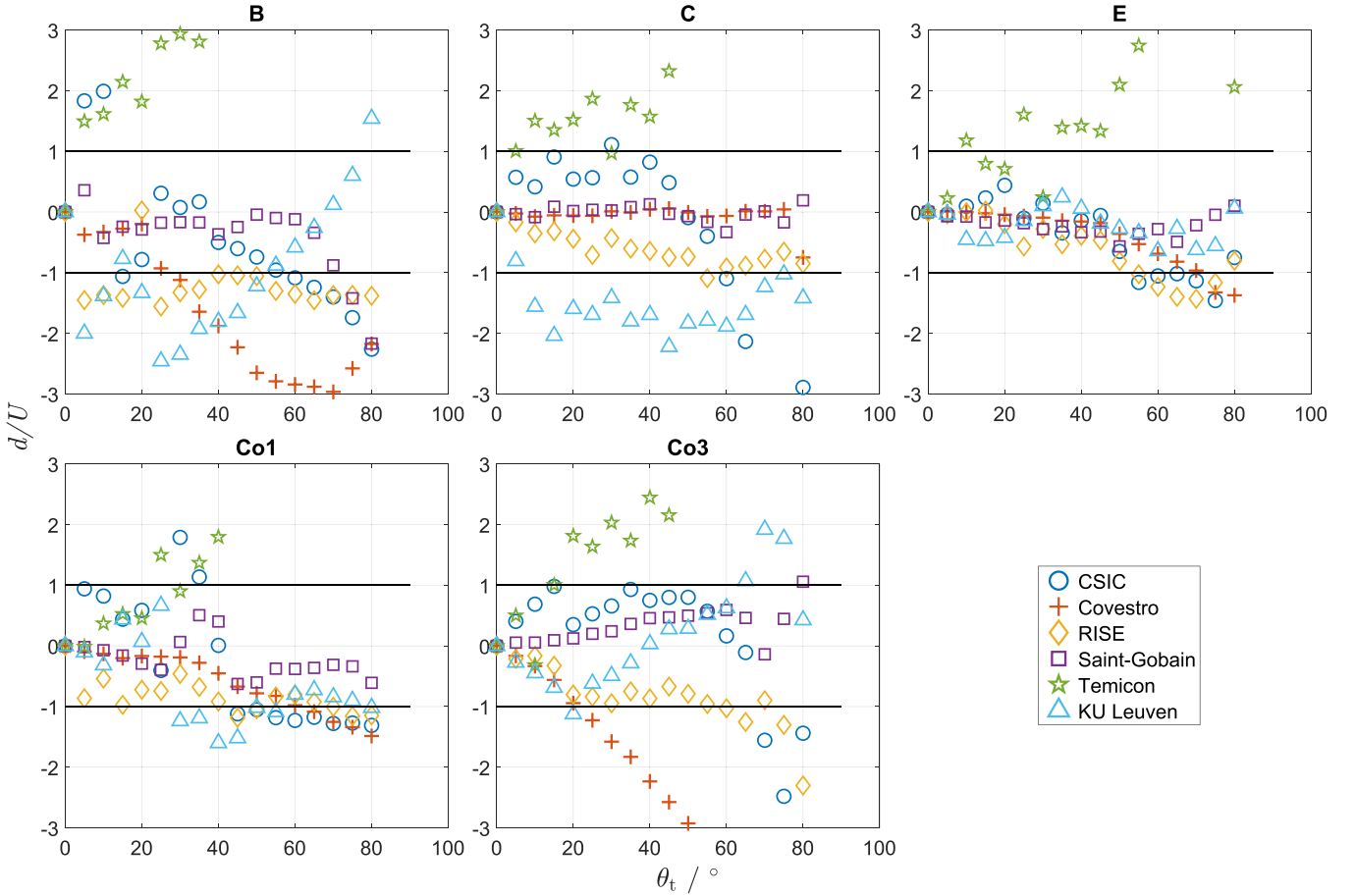


Figure 12. d/U metric for participants in comparison two. Points randomly scattered within the -1 to 1 interval indicate equivalent measurements of the sample with adequate uncertainty analysis.

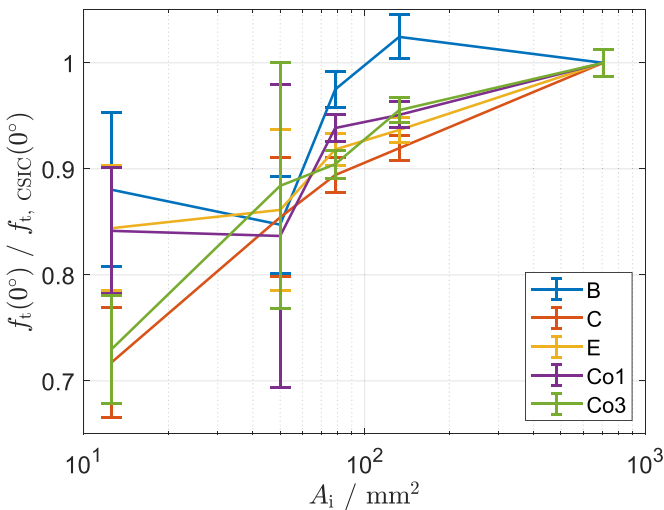


Figure 13. Dependence of the measured BTDF ($\theta_t = 0^\circ$) on the applied irradiated area size A_i in comparison two. For a clear visual comparison, all BTDF values are shown relative to the ones measured by CSIC, who applied the largest A_i in all measurements.

be large enough, as discussed in section 3.1. The inconvenience of this second measurement scheme is that for thick and translucent samples A_M should be very large, and the measured

BTDF is only an average within A_M , which might exhibit high spatial variation. It makes the results measured from different measurement schemes not completely comparable.

5. Conclusion

In this paper, the capability of performing accurate BTDF measurements on the facilities of six NMIs, two DIs, one university, and three industrial partners was thoroughly studied by means of two multilateral scale intercomparisons. In comparison one, the NMIs and the DI could perform consistent measurements on the quasi-Lambertian samples regardless of the different measurement conditions of each facility. However, for samples possessing a narrow peak or rapid change in their BTDF distribution, inconsistency was observed between the participants, mainly due to the difference in the applied detection angular resolution. The results of samples with dedicated azimuthal dependence implied several aspects that require more attention for improved future measurements. One important aspect was a better determination and realisation of the sample azimuth angle. Despite some inconsistency in the results, the potential to perform high-quality BTDF measurement equivalent to the state of the art was demonstrated by a subset of the participants.

In comparison two, the generally consistent measurement results successfully connected the scale between metrology institutes and industrial partners. There was one common issue observed in both comparisons when measuring highly translucent samples with large contribution of scattering coming from the bulk volume. The lateral diffusion of the irradiation spot made the measurement performed in the under-irradiated scheme, as most of the participants did in both comparisons, strongly dependent on the measurement conditions. However, even in the over-irradiated scheme the measured BTDF still exhibited dependence on the size of the irradiated area. This behaviour needs further investigation for more consistent results.

For future comparisons, a more precise definition of measurement parameters is crucial and a thorough study on the sample characteristics would allow a better congruence in the participants' results.

Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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Supplementary Material

Survey of bidirectional transmittance distribution function measurement facilities by multilateral scale comparisons

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S1. Detailed introduction to participants’ instrumentation

The sketch of the instrument used for the BTDF measurements at PTB is presented in figure S1. Two lasers were used as irradiation sources for the measurement. One is a diode laser at 642 nm, fibre-coupled into a collimator, another one is a diode-pumped solid-state laser at 445 nm. The laser beams were expanded by two lenses. In the vicinity of the intermediary focus of the beam expander, a rotating weak diffuser was positioned to reduce the coherence of the laser radiation and thus reduce the speckle effect. It also improved the spatial homogeneity of the beam profile. The polarisation state of the laser radiation was set by a polariser before impinging on the sample. An optical chopper was used to achieve detection with lock-in technique. Neutral-density (ND) filters of different attenuation levels were set into the beam path when measuring the direct laser radiation, so that the signal received by the detector could be kept within its linear response range. Diaphragms placed in front of the sample determined the irradiation spot size on the sample. The sample under test was mounted on a three-axis motorised translational stage, which itself was mounted on a rotary stage to realise different incident polar angles. The diffusely transmitted radiation was collected by a given solid angle, which was defined by the size of the detector aperture and its distance to the sample back surface. Positioned directly behind the detector aperture, an achromatic lens imaged the sample back side at the entrance port of an integrating sphere, where a field stop with varying diameter determined the size of the measurement area on the sample. A silicon photodiode was mounted on the exit port of the sphere. The whole imaging detection system, indicated by the dashed ellipse in figure S1, was mounted on another rotary arm, which could rotate around the sample back surface to realise different detection polar angles.

DFM’s setup for measuring the BTDF is shown in figure S2. It employs a CW laser operating at 663 nm for illumination of the sample, and a moving silicon photodetector for collecting the scattered light in the horizontal plane. Lock-in detection was employed, and ND filters in the incoming beam were used to extend the intensity range of the measurement. For the

measurements presented in this paper, the illuminated area on the sample was 27 mm^2 . The detector was positioned at a distance of 355 mm from the illumination spot, and an aperture of area 21 mm^2 was placed in front of the detector. The aperture in front of the detector spans a planar angle of 0.83° in the scattering plane relative to the illumination spot. This detector configuration accepts a wide range of incoming angles. Thus, the measurement was performed in under-illumination mode, where the illumination defines the measurement area. The measurement did not employ a reflection standard for reference, instead the BTDF is calculated from the ratio between the transmitted intensity and the intensity of the illumination beam measured without a sample.

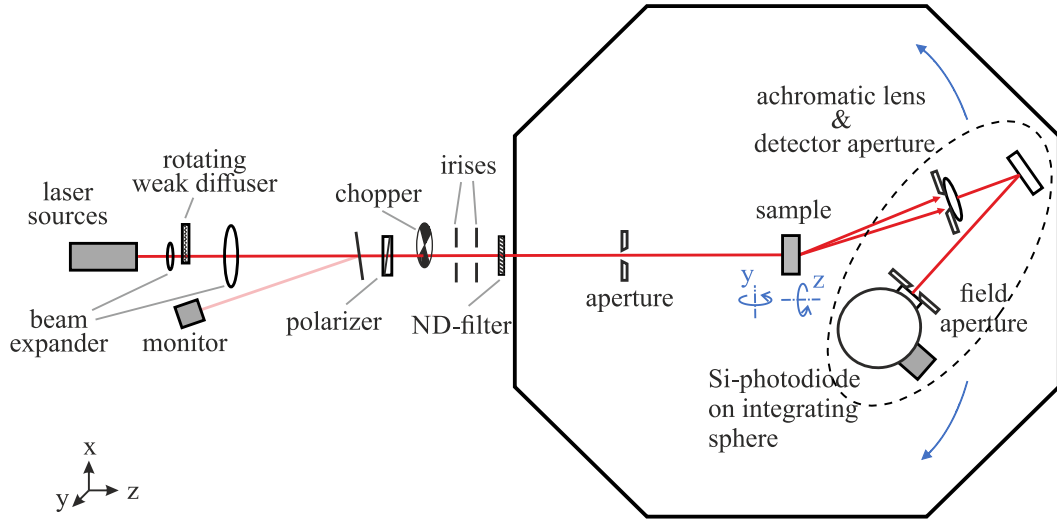


Figure S1. Sketch of PTB's BTDF measurement setup.

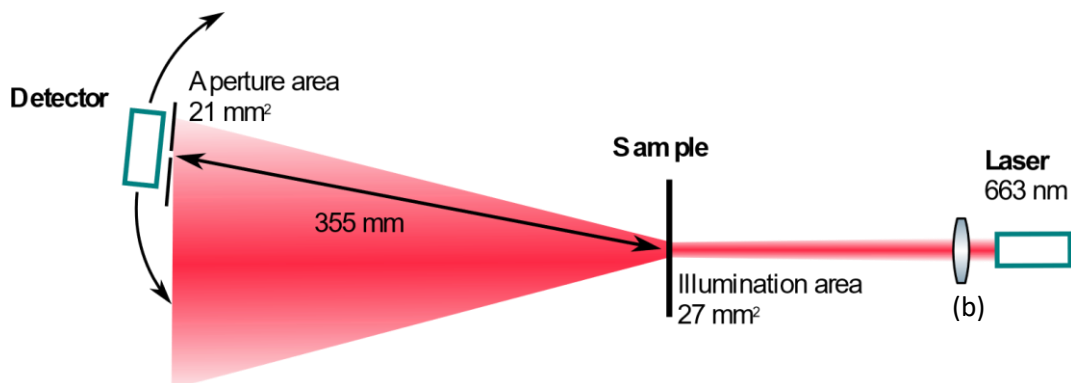
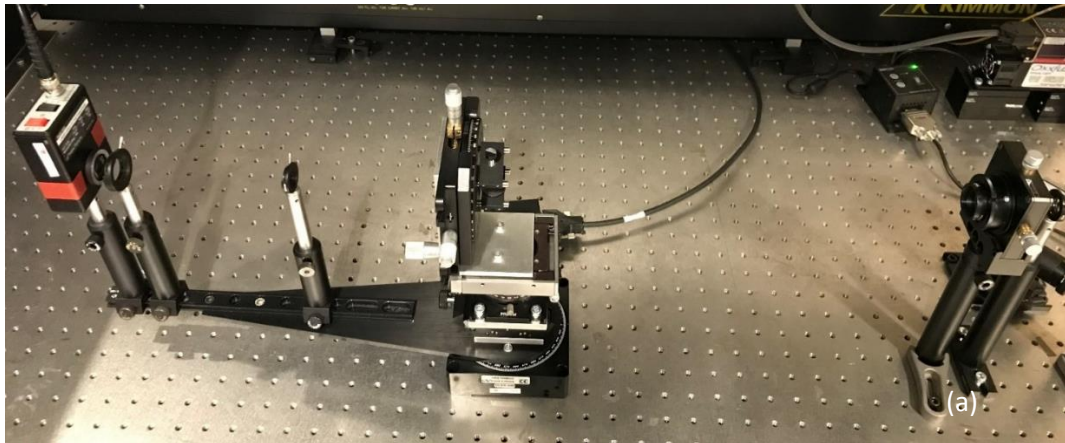


Figure S2. (a) Photo and (b) sketch of DFM's BTDF measurement setup.

As illustrated in figure S3, the instrument at CMI comprises a six-axis robot arm to hold the sample and a motorised ring to rotate the detection system. The robot is set to hold the sample vertically so that its 6th joint rotation axis is coaxial with the rotation axis of the detector ring. In this way, the robot arm does not obstruct the transmitted radiant flux from the sample. The monochromatic illumination is provided by a broadband high intensity laser-driven light source (LDLS) coupled with a monochromator with a spectral bandwidth of 5 nm. The optical output from the monochromator is then collimated to illuminate the sample by an off-axis parabolic mirror (OPM) and set to the proper alignment with the sample by a beam steerer. The size of the illumination spot is determined by the fixed aperture in front of the detector ring. The illumination polarisation is controlled by a linear film polariser mounted on a motorised rotation stage. The polarisation-independent detection system, developed in cooperation with MSL, is composed of an aperture, a mirror and a low noise silicon photodiode mounted on a small integrating sphere. The light passing through the aperture of 17 mm diameter is collected by the mirror and reimaged at the input port of the integrating sphere. The distance between the detector aperture and the sample is 462.2 mm, resulting in an angular resolution of the detection system of 2.1° . The photodiode's photocurrent is transformed into a voltage signal by a switched integrator amplifier developed by CMI with a dynamic range up to 5 orders of magnitude.

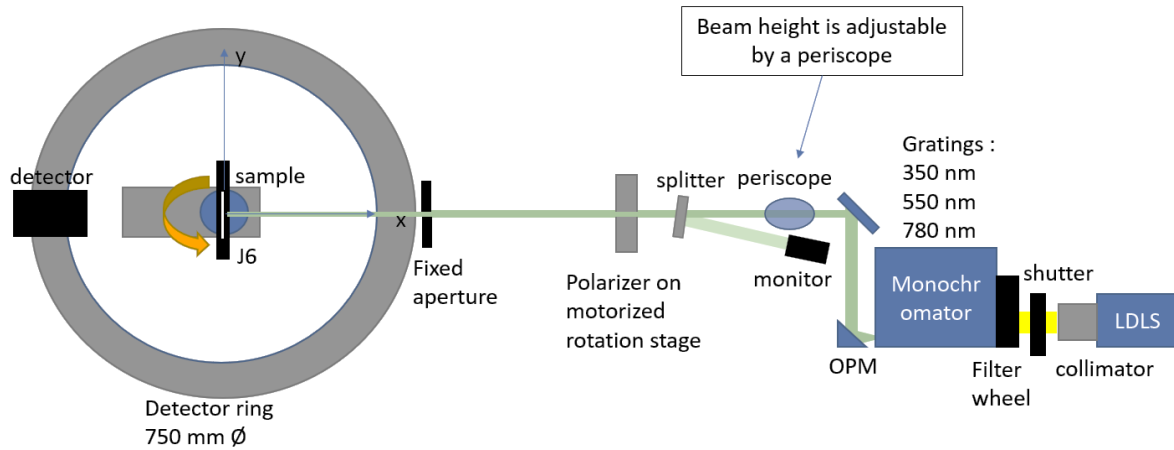


Figure S3. Sketch of CMI's BTDF measurement setup.

The measuring system used at MSL is presented in figure S4. Light from the LDLS or supercontinuum source is directed into a monochromator. The monochromatic light is passed through a fibre to the collimating optics. A fibre shaker is attached to this fibre, which despeckles the beam. The collimating optics have been designed to use Köhler illumination to collimate the beam. There is a filter wheel positioned in the beam path that contains ND filters of various optical densities, allowing the intensity of the incident light to be adjusted. The beam is then polarised and a beam splitter directs 8 % of the light into the monitoring detector. The rest of the light is directed onto the sample, which is mounted on a three-axis rotation stage. The three-axis rotation stage is positioned in the centre of a slew ring, on which the detector is mounted.

The instrument used in the BTDF measurements at CNAM is shown in figure S5. BTDF is measured in an 'underfilled' configuration. The illumination system consists of a QTH (quartz tungsten halogen) source with monochromator and optical elements (lenses and diaphragm). The bandwidth of the source can be adjusted from 1 nm to 5 nm. Light is depolarised using a depolariser. The illumination spot on the sample can be adjusted from 3 mm to 15 mm in diameter. The aperture of the illumination beam can vary from 0.2° to 0.35° . The sample is held vertically by a six-axis robot arm at the centre of the goniometer and adjusted perpendicular to the source using a mirror. The detection is placed on a rotating breadboard (using a motorised rotation ring). It can measure at detection angles varying between $\pm 90^\circ$. The detection system consists of a silicon photodiode with optical elements (mirror and diaphragm). The collection solid angle of the detection is defined by the diaphragm. The diaphragm is located at about 1.15 m from the sample plane. The detection's aperture can vary from 0.8° to 2° . The light source is modulated and the photocurrent produced by the photodiode is measured using a lock-in amplifier.

The absolute goniophotometer used for the BTDF measurement at Aalto is presented in figure S6. Monochromatic illumination can be achieved by using broadband light sources and monochromators. The spectral bandwidth of illumination is controlled by the width of the input and middle slits of the double monochromator. Adjusted by an aperture in front of the sample, the illumination beam size has 8 mm or 12 mm in diameter in the BTDF measurements. The sample is placed at the centre of a turntable and can be moved out of the beam by motorised translational units. At a distance of 472.9 mm, the scattered transmitted light is collected by a precision aperture with a diameter of 25 mm. The resulted detection angular

resolution is approximately 3° . The photosensitive area of a Si-photodiode is imaged on the sample surface and the resulted collection field of view is larger than the incident beam.

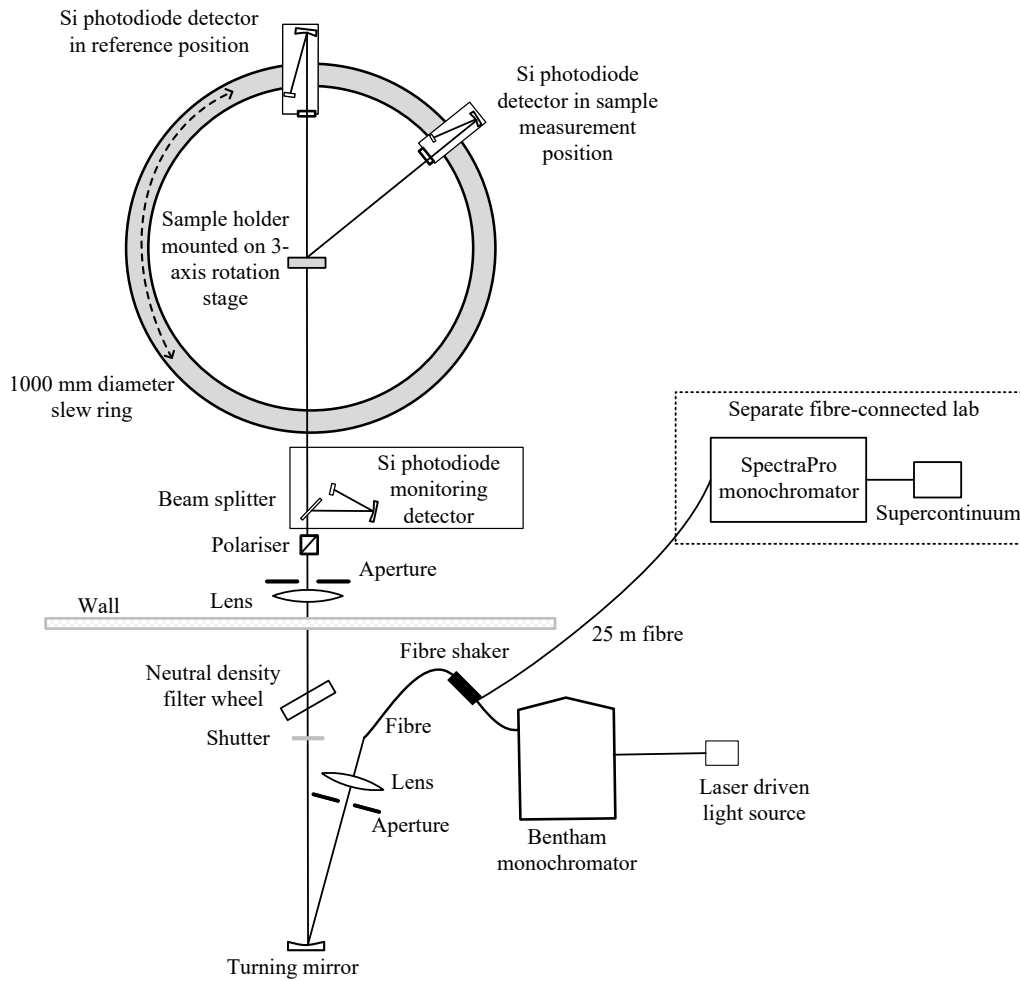


Figure S4. Sketch of MSL's BTDF measurement setup.

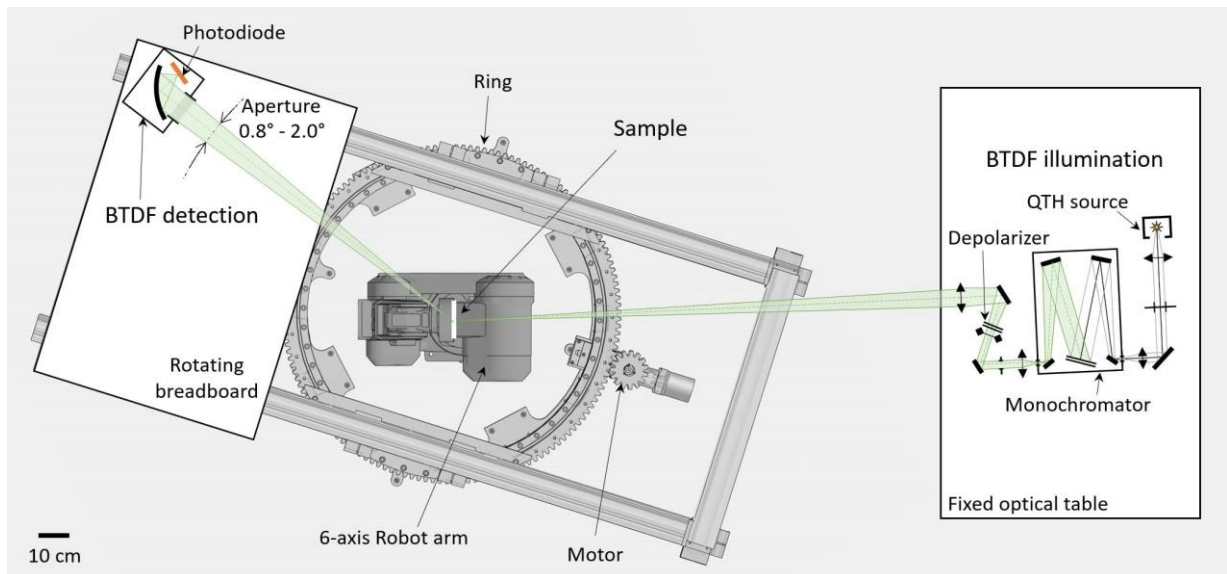


Figure S5. Sketch of CNAM's BTDF measurement setup.

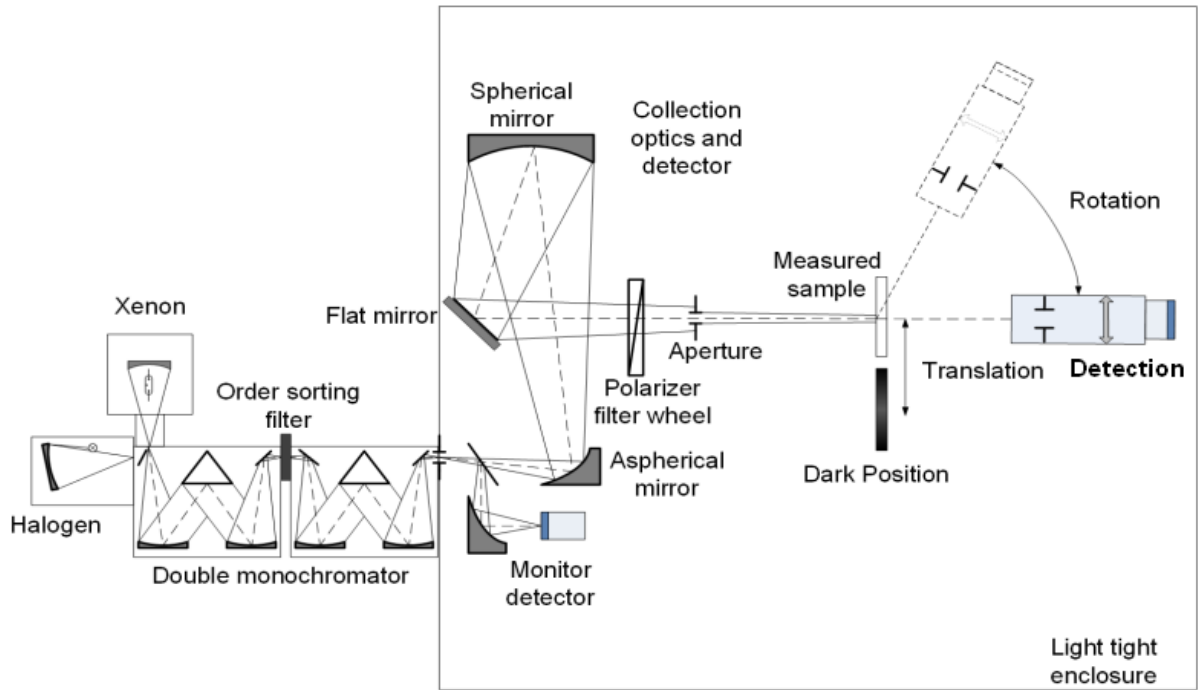


Figure S6. Sketch of Aalto's BTDF measurement setup.

The measurement setup used at CSIC is shown in figure S7. The uniform illumination on the sample is achieved by a broadband Xenon (Xe)-lamp in combination with a Köhler system. The size of the illumination beam is given by the aperture (P1) after the first lens (L1) and has a diameter of 30 mm. Held by a six-axis robot arm the sample can be positioned in the desired irradiation angles. A spectroradiometer is mounted on a cogwheel, allowing the collection angle to be chosen regardless of the irradiation, at a distance of 350 mm to the sample. With an angular resolution of 5° the transmitted signal in the spectral range from 380 nm to 780 nm can be obtained. The measurement area on the sample has 7 mm in diameter.

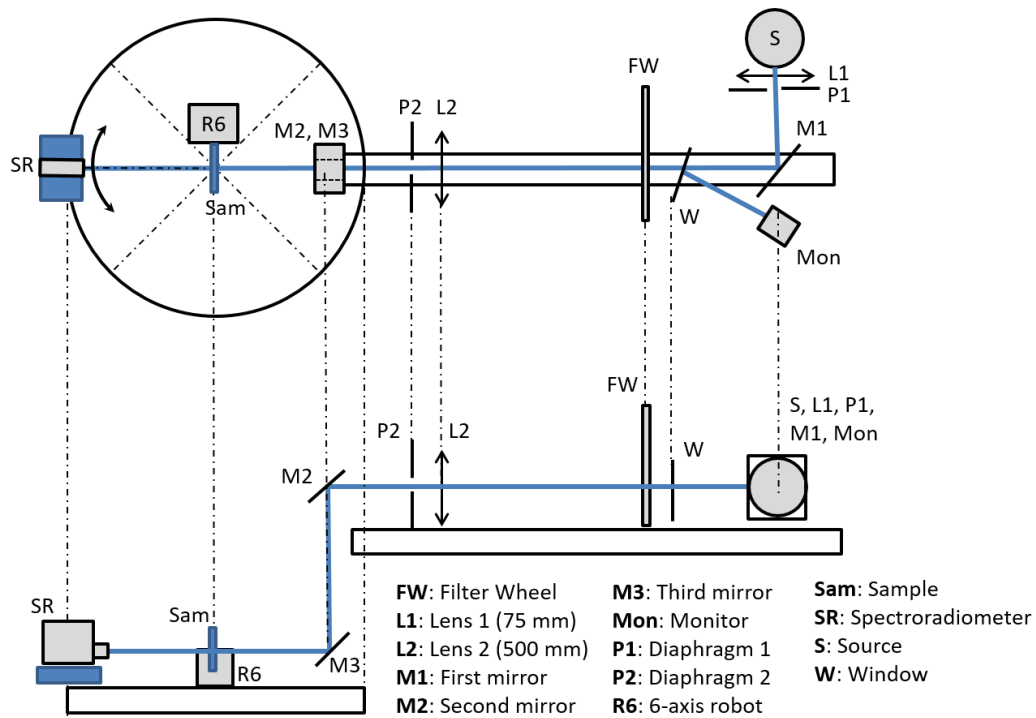


Figure S7. Sketch of CSIC's setup used for BTDF measurements, with above the top view and below the side view.

The instrument used at Covestro is illustrated in figure S8. The illumination is realised by spectrally selecting the radiation from a 100 W halogen light source with a band filter at 632 ± 5 nm. The spot size on the sample is 4 mm in diameter. The sample under test is mounted on a motorised rotating axis with positioning accuracy of 0.01° . The detection system is placed at a distance of 170 mm from the sample. The transmitted optical power within an area of 8 mm diameter on the sample surface is collected by a full acceptance angle of 2.2° and detected unpolarised. The relative BTDF measurement is calibrated by a PTFE Standard measured at the specular reflection polar angle of 30° while illuminating the sample at an incident polar angle of 30° .

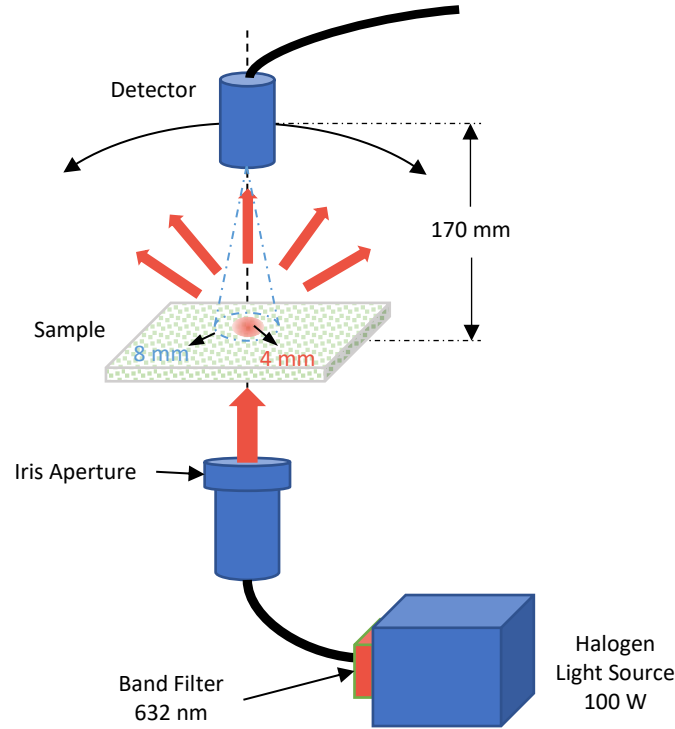


Figure S8. Photo of Covestro's BTDF measurement setup.

The measuring system at Saint-Gobain is a commercially available goniospectrophotometer OMS4 provided by OPTIS (in Ref. [18]). A Xe-lamp filtered at 635 nm was used as illumination source and it generates an illumination beam with divergence of 0.2° and a beam spot of around 8 mm in diameter on the sample. A photomultiplier tube is used as the detector and is placed at a distance of 1000 mm away from the sample. In this way, the BTDF of the sample can be measured with an angular resolution of 0.2° .

The facility used for BTDF measurements at Temicon is presented in figure S9. A 633 nm diode laser is used as the illumination source and its power can be adjusted between 0.1 mW and 100 mW. The laser beam is expanded and collimated to realise an illumination spot of approximately 8 mm in diameter on the sample. The sensor has a size of approximately $0.5 \text{ mm} \times 2 \text{ mm}$ and is mounted on a motorised rotation stage with 750 mm radius. The angular resolution can be adjusted depending on the scattering characteristic of the sample under test.

The sketch of the BTDF instrument at KU Leuven is illustrated in figure S10. The illumination is realised by using a broadband Xe-lamp (1) and band filters (7) with a divergence of 0.1° and a spot size of 13 mm diameter on the sample (6). The sample is mounted on a manual rotation stage. The detection (5) is performed by a CCD-based spectrograph with a bandwidth of 3 nm at 632.8 nm wavelength. High dynamic range of six magnitudes of the detector is possible due to variable integration time of CCD and three ND filters of 10 %, 1 %, and 0.1 %. The angular resolution of the detection is about 1.6° and almost full spherical coverage can be achieved with an angular step size of 0.1° through the use of two automated rotary stages mounted perpendicular to each other. The relative BTDF measurements were performed with respect to a BRDF standard, which was calibrated by PTB.

The measurement setup at RISE is shown in figure S11. It uses an LDLS (Energetic EQ-99) with collimating optics. The irradiation spot can be adjusted between about 3 mm to 20 mm in diameter. The primary collector is a 4-inch integrating sphere with a silicon detector which can be positioned at an arbitrary distance up to 1000 mm from the sample. The angular resolution of the system is 2.9° , which is determined by the set distance, the spot area, and the aperture size of the collector.

The measurements are normally performed in-plane at arbitrary detection angles between $\pm 90^\circ$. Spectral BTDF is determined by using an interference filter with an integrating sphere with a Si-photodiode.

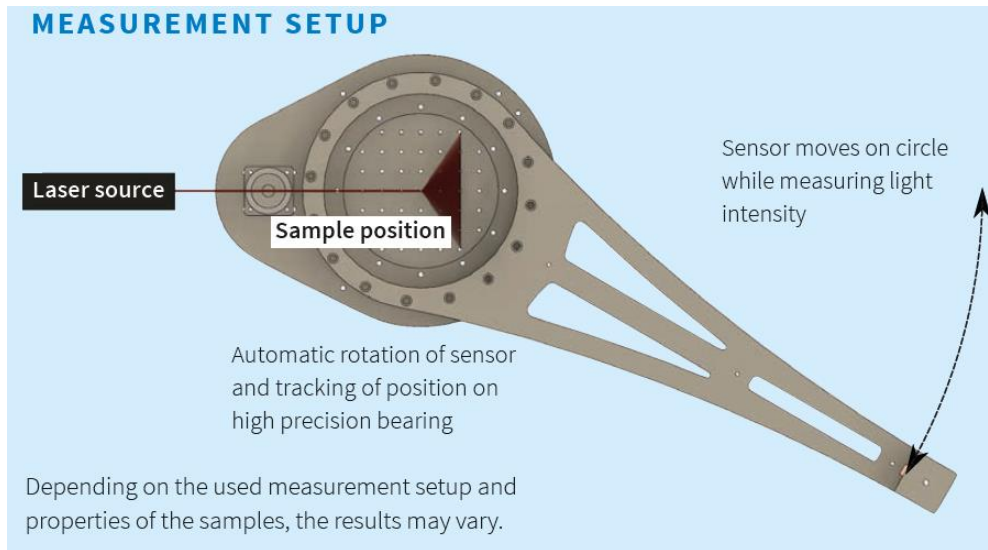


Figure S9. Sketch and description of the BTDF measurement setup at Temicon.

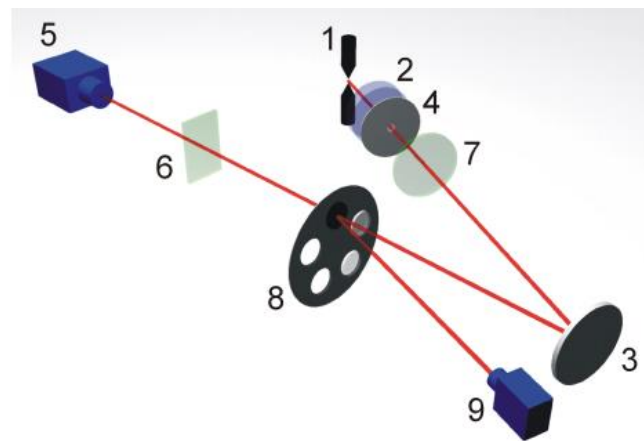


Figure S10. Sketch of the BTDF measurement setup at KU Leuven: (1) Xe-lamp (source), (2) condenser lens, (3) concave mirror, (4) iris diaphragm, (5) detector head, (6) sample holder, (7) long-wave pass filter, (8) filter wheel, and (9) reference detector.

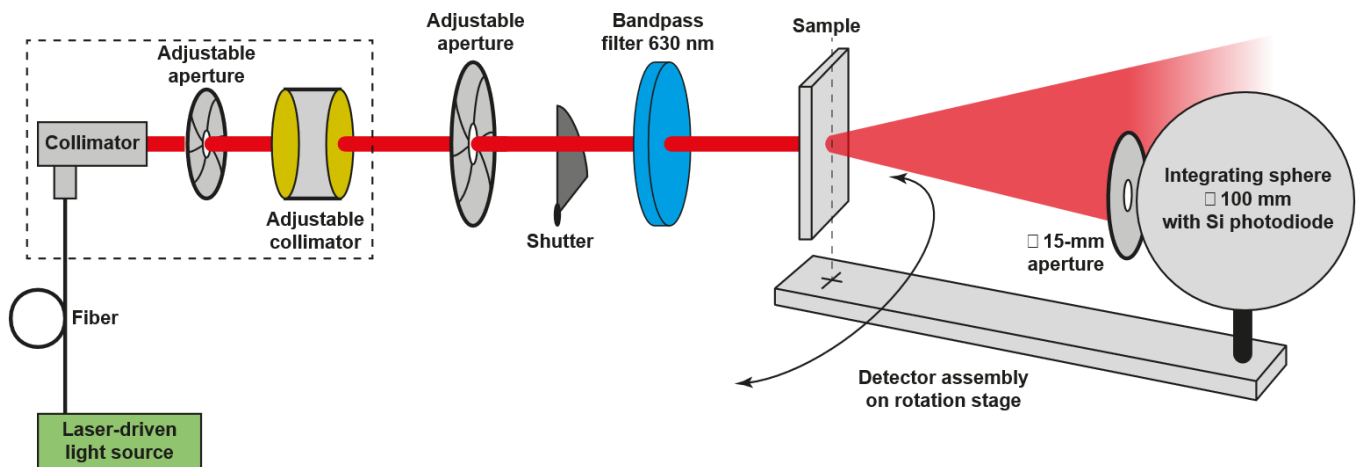


Figure S11. Sketch of the BTDF measurement setup at RISE.

S2. Participants' measurement results

In order to reduce the influence from the inter-sample-set variations on the result of comparison one, the BTDF values measured by the participants were normalised to those measured by the pilots, using the method described in subsection 2.4 in the main text. Thus, the normalised measurement results of all participants and pilots in comparison one are demonstrated in figures S12 and S13 for 633 nm and 445 nm, respectively.

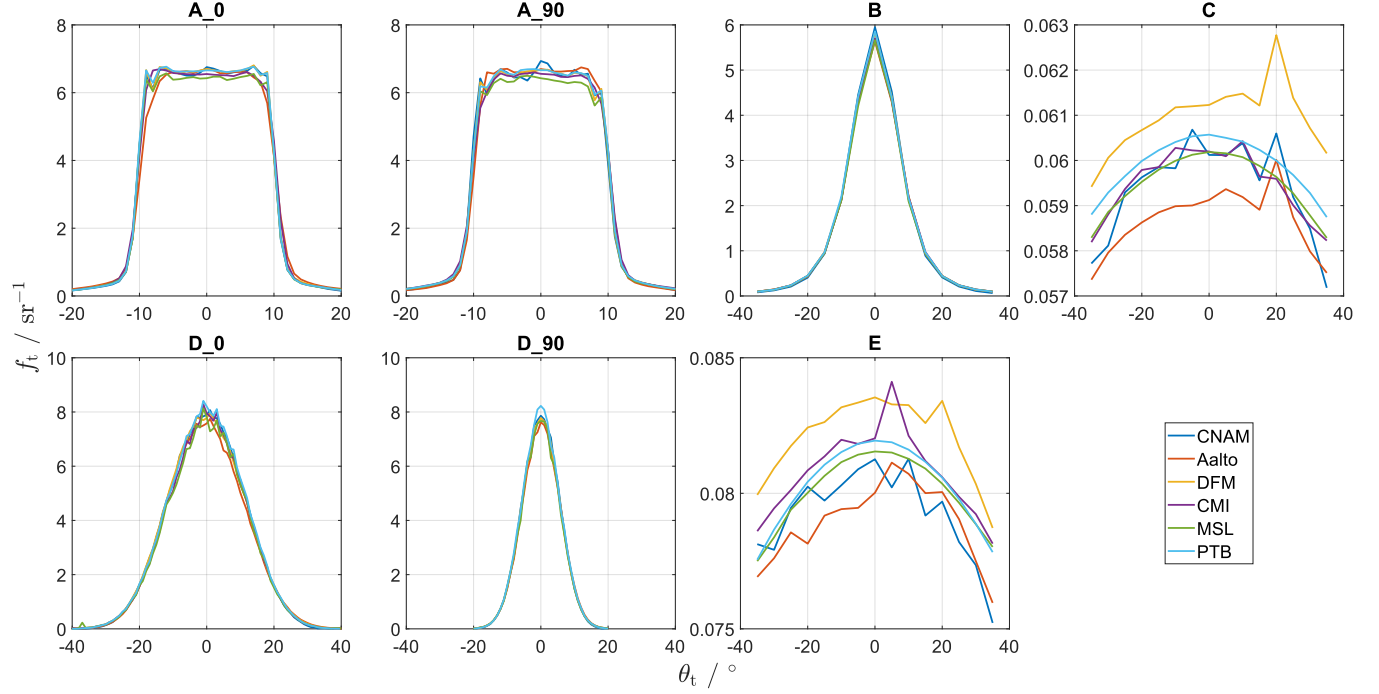


Figure S12. Normalised participants' data for 633 nm in comparison one.

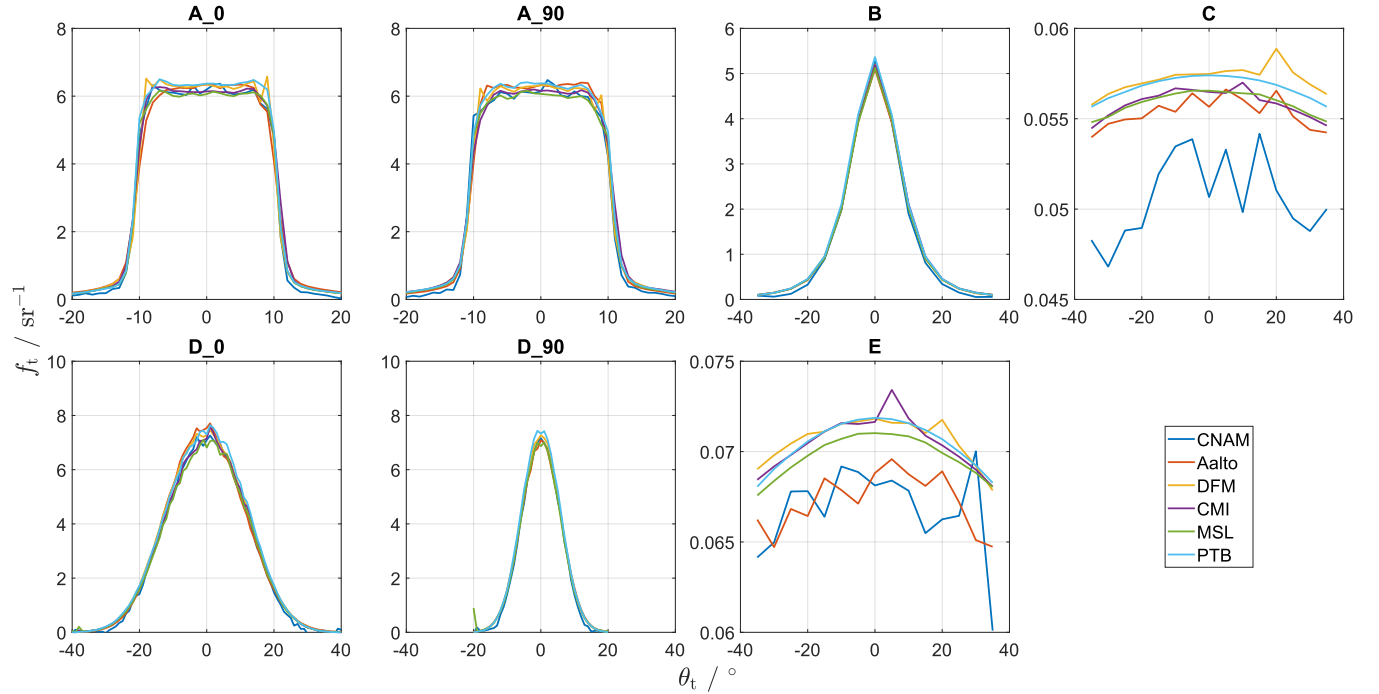


Figure S13. Normalised participants' data for 445 nm in comparison one.

The measurement results of all participants in comparison two, normalised and adjusted as described in the main text (subsection 2.4 and section 4, respectively), are shown in figure S14 for each sample. The reported expanded ($k = 2$) relative uncertainties are shown in figure S15.

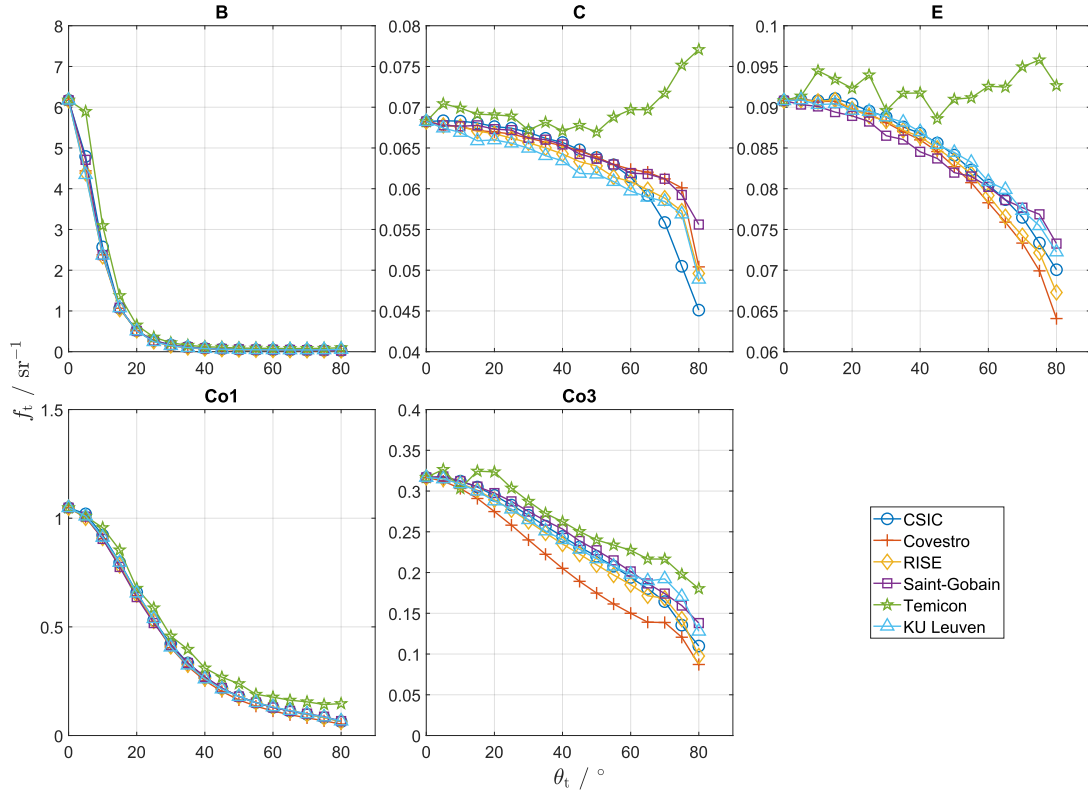


Figure S14. Normalised and adjusted BTDF results for all participants in comparison two.

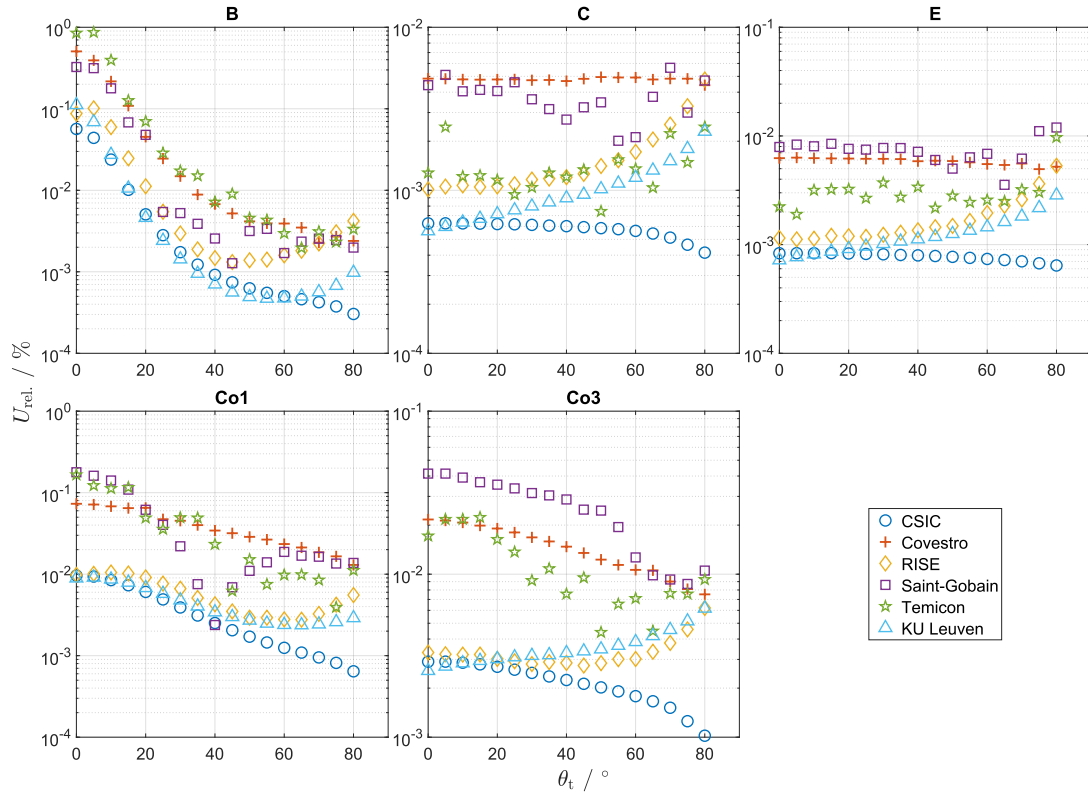


Figure S15. Relative expanded ($k = 2$) uncertainty reported by participants in comparison two.

S3. Wavelength correction

The measurement results of PTB and DFM were corrected for samples' spectral dependence, as both participants were not able to perform the measurements at the prescribed wavelengths. The spectral transmittance from 380 nm to 780 nm of all samples involved in the first comparison was measured at different detection polar angles θ_t under normal incidence using a commercial instrument (Varian UMA accessory on a Cary 6000i spectrophotometer) at PTB. It was observed that the sample's spectral transmittance also depends on the detection polar angle. Therefore, the correction was performed for every θ_t angle by applying an angle-dependent correction factor $\gamma_{\text{corr}}(\theta_t)$ as in eq. (1). The standard uncertainty for this wavelength correction was assumed to be 20 % of the correction factor, i.e.,

$$\begin{aligned} f_{t,\text{corr}} &= f_t \cdot (1 + \gamma_{\text{corr}}(\theta_t)), \\ u(f_{t,\text{corr}}) &= 0.2 \cdot |\gamma_{\text{corr}}(\theta_t)|. \end{aligned} \quad (1)$$

For 445 nm, the data measured by PTB was slightly corrected regarding the real output wavelength of the blue laser. As no measurement was possible at DFM for this wavelength, and in order not to lose measurement results by the participants in the sub-group of DFM, namely CNAM and Aalto, the DFM results for 663 nm were also used for the comparison at 445 nm, by directly correcting them to this wavelength. The standard uncertainty of this correction was again estimated to be 20 % of $\gamma_{\text{corr}}(\theta_t)$. For sample types C and E, the correction worked quite well, as both samples exhibited an approximately linear spectral dependence with varying slopes at different θ_t angles. For sample types B and D, although their nonlinear spectral dependence introduced larger uncertainties in the wavelength correction, its impact on the comparison results was still insignificant, as other uncertainty sources such as the applied instrument function dominated in the total uncertainty for these two samples. However, for sample type A, the wavelength correction did not work well. This is explained in figure S16, where the BTDF distributions (measured by PTB) at 633 nm and 445 nm of sample A are compared. For a better visual comparison, the distribution measured at 445 nm is upscaled with respect to the 633 nm BTDF value at $\theta_t = 0^\circ$.

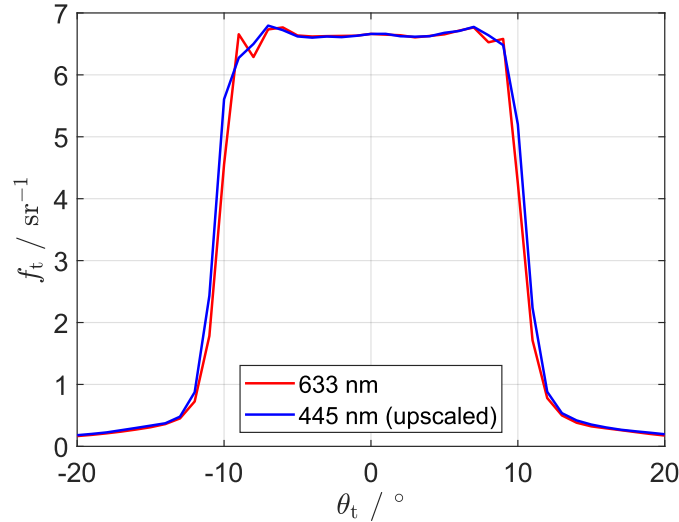


Figure S16. PTB's BTDF results for sample A ($\varphi_t = 0^\circ$) measured at different wavelengths with a detection angular resolution of 1° . The measured BTDF distribution at 445 nm is upscaled to 633 nm for a better visual comparison. The obvious shape difference of the BTDF distribution measured at different wavelengths explains the unsuccessful wavelength correction applied on the DFM's data, and this eventually leads to the inconsistency in the comparison of this sample type.

While the upscaled 445 nm BTDF values in the top-hat angular range lie close to the 633 nm values, the different structures in the scattering distributions could not be accounted for by simply applying the wavelength correction, as the interaction of irradiation with the functional surface varies with wavelength. The 'batwings' at around $|\theta_t| = 9^\circ$, observed at 633 nm, are not present in the 445 nm distribution. As a result, the measurement results of CNAM and Aalto might be incorrectly normalised by the obvious discrepancy for these variations.

S4. The lateral diffusion of irradiation spot on sample C

As discussed in the main text, the lateral diffusion effect was very obvious on sample C, which made the measured BTDF strongly dependent on the measuring parameters, i.e., the irradiated area A_i and the measurement area A_M . The influence on

the BTDF ($\theta_t = 0^\circ$) measured with different A_M while keeping A_i constant is illustrated in figure S17. As expected, larger BTDF was obtained with a larger A_M , since more part of the laterally diffused irradiation spot was detected. The increase in the BTDF can be fitted with a simple inverse exponential equation, from which a saturation value can be extrapolated, which would correspond to the BTDF value measured with an infinitely large measurement area. For future comparisons of such highly translucent samples, either the measurement parameters should be clearly specified to the participants, or all participants should perform multiple measurements with varying parameters to obtain the true BTDF.

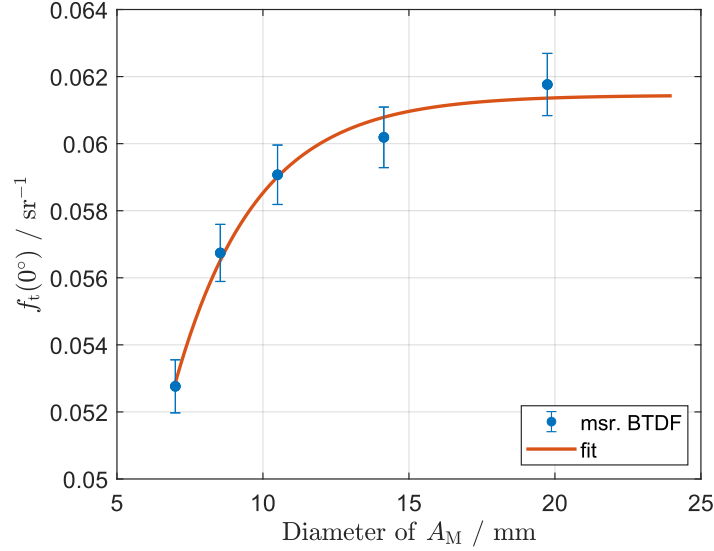


Figure S17. BTDF of sample C ($\theta_t = 0^\circ$) measured with different sizes of measurement area A_M , while keeping the diameter of the irradiated area constant at $d_i = 2.5$ mm. The error bars depict the standard ($k = 1$) uncertainty of the measured BTDF values.

S5. Spatial inhomogeneity of different sample types

Apart from the main factors for the inconsistency in comparison one, as discussed in the main text, the spatial inhomogeneity of some sample types might also contribute to the inconsistency.

The spatial inhomogeneity of sample C was observed by illuminating the sample under normal room light, as shown in figure S18. The back surface of the sample (facing towards the detection in the measurement) appears to be more homogeneous compared to the front surface. The size of the spatial patterns on the front surface is several millimetres, whereas on the back surface they are not obvious. The formation of these patterns might be caused by the manufacturing process. Since the irradiation beam size of different instruments varied largely, the resulting BTDF values may depend on this parameter.

Sample B was also observed to have spatial inhomogeneity in its active surface. It was reported by MSL that a relative deviation of approximately 3 % was observed in the measured BTDF while moving the irradiation beam over a distance of 5 mm.

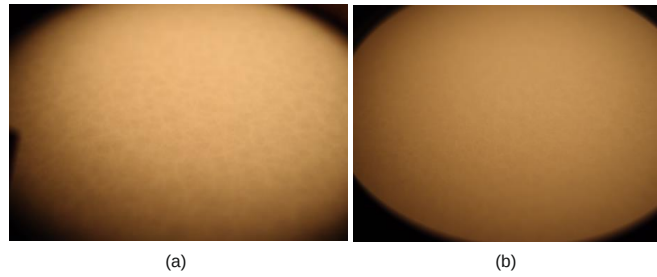


Figure S18. (a) The front surface and (b) the back surface of sample type C, photographed under room light illumination. Some inhomogeneous patterns of several millimetres can be observed on the front surface (facing towards the irradiation in the measurement). This might contribute to the inconsistency in the comparison of sample C.

For sample A, the spatial inhomogeneity in its functional surface was also observed by MSL, but with less significant influence on the measured BTDF as for sample B. This inhomogeneity might stem from the random distribution of the micro-lens units on the functional surface, but evidence by measurement is missing.

For other sample types involved in comparison one and two, no obvious spatial inhomogeneity was observed.

S6. Influence of instrument function on sample type D, in the narrow azimuthal orientation

The influence of the applied instrument function, i.e., angular resolution, can be observed most clearly in the narrow azimuthal orientation ($\varphi_t = 90^\circ$) of sample type D. Among all participants, PTB used the highest angular resolution 0.5° , followed by CNAM and DFM with 0.8° and MSL with 0.9° . CMI only performed the measurement at azimuth $\varphi_t = 0^\circ$ with 2.1° angular resolution and Aalto measured both orientations with 3° angular resolution. The expectation can be substantiated by taking a closer look at the peak area of the normalised (to eliminate the influence from inter-sample-set variations) BTDF distributions for $\varphi_t = 90^\circ$, as shown in figure S19. As a result, PTB obtained the largest peak value, followed by CNAM and DFM, who measured a larger value than MSL and Aalto. Thus, the χ_{obs}^2 value in the central angular area for this orientation shows a similar shape to sample B, but with even larger inconsistency.

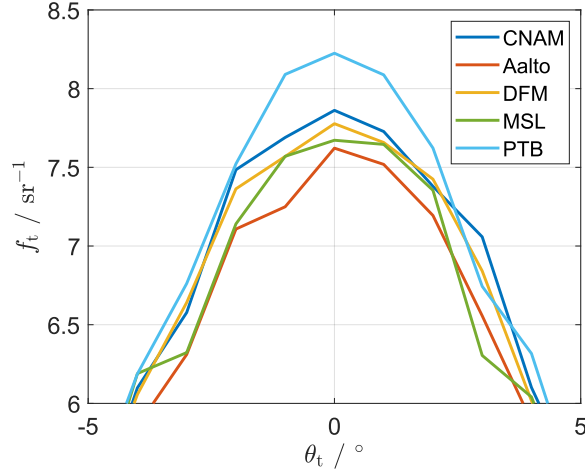


Figure S19. Normalised BTDF distributions of sample D for $\varphi_t = 90^\circ$ at 633 nm measured by participants in comparison one. Only the central angular area is shown for a clear visualisation. The difference in the BTDF values measured by PTB and by the other participants might contribute to the inconsistency in the comparison of this sample.

S7. Inconsistency of sample type A regarding the azimuth angle

The BTDF of central angular area of sample type A also depends on the sample azimuth angle, as the top-hat profile generated by the functional surface of this sample is not ideal. This can be verified by comparing the normalised (to eliminate the influence from inter-sample-set variations) BTDF distribution of all participants in comparison one at 633 nm in this range, as illustrated in figure S20. Both pilots (DFM and PTB) obtained similar BTDF distributions for both azimuth angles, but a clear difference in the shape of the curves can be observed for different φ_t . Some participants' data has similar shape to the pilots' data after normalisation, while the others show a different behaviour. Furthermore, the difference between all participants appears larger for the $\varphi_t = 90^\circ$ azimuth orientation than for $\varphi_t = 0^\circ$. This might indicate a limited accuracy in realising the azimuth angle for those participants, and also an underestimation of this component in their uncertainty analysis.

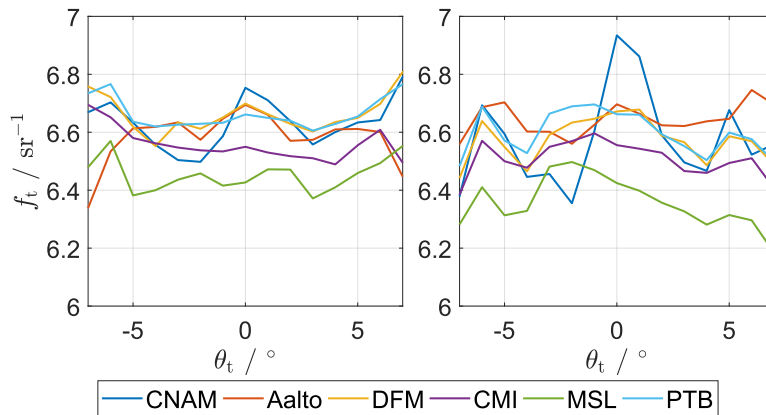


Figure S20. Normalised BTDF distribution of sample A for $\varphi_t = 0^\circ$ (left) and $\varphi_t = 90^\circ$ (right) at 633 nm measured by the participants in comparison one. Only the central angular area is shown for a clear visualisation. The larger difference between all participants' BTDF values at $\varphi_t = 90^\circ$ might indicate a limited accuracy in realising the azimuth angle on some participants' measuring facilities.

S8. Uncertainty adjustment of participants in comparison one

The evolution of the uncertainty adjustment for each participant comparison one is presented in the following figures in this section, for both wavelengths at 633 nm and 445 nm. The black solid symbols represent the originally reported relative uncertainties and the hollow symbols show the adjusted relative uncertainties, with the blue ones for cut-off adjustment only and the red ones for further adjustment using the Mandel-Paule method.

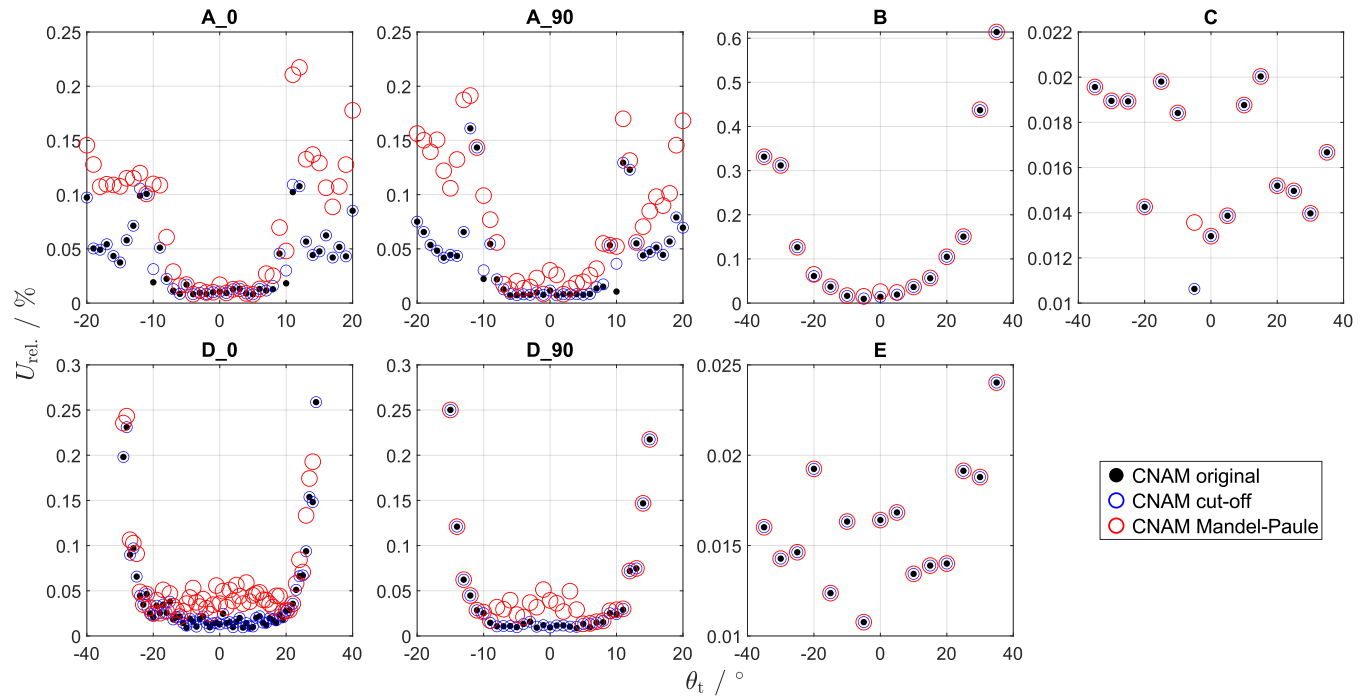


Figure S21. Development of CNAM's uncertainties (relative) at 633 nm.

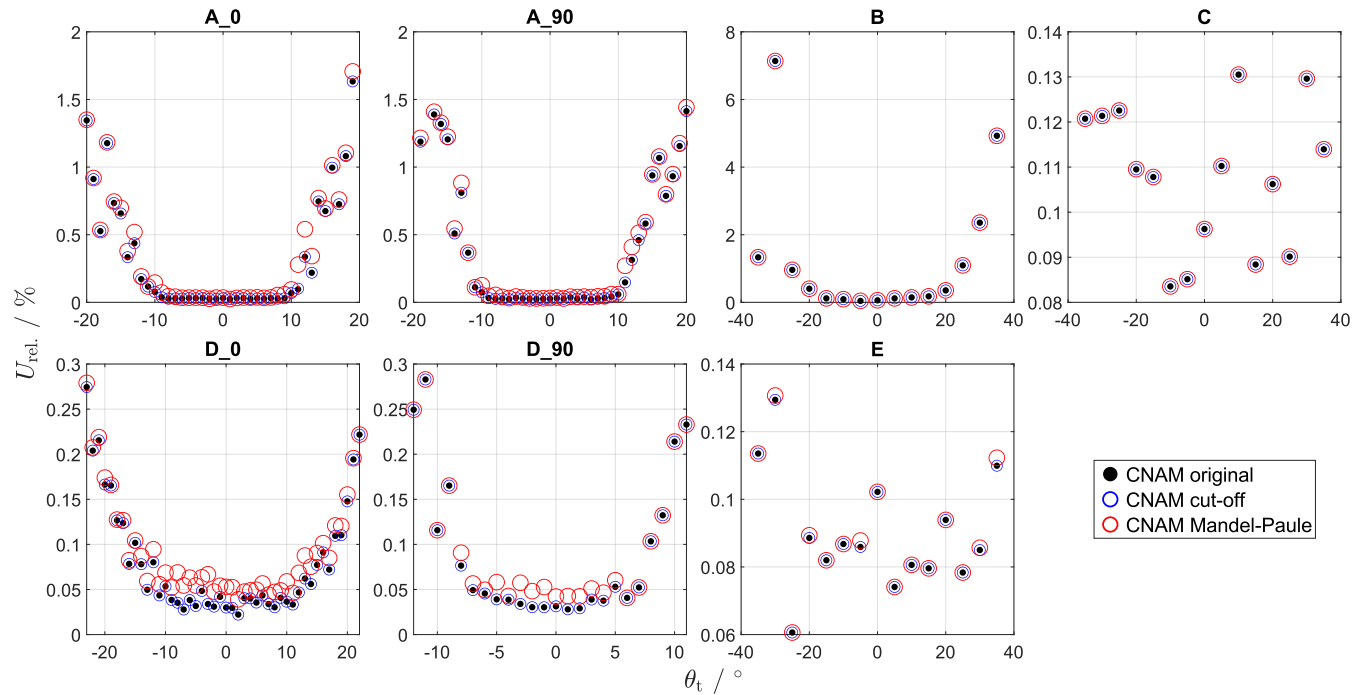


Figure S22. Development of CNAM's uncertainties (relative) at 445 nm.

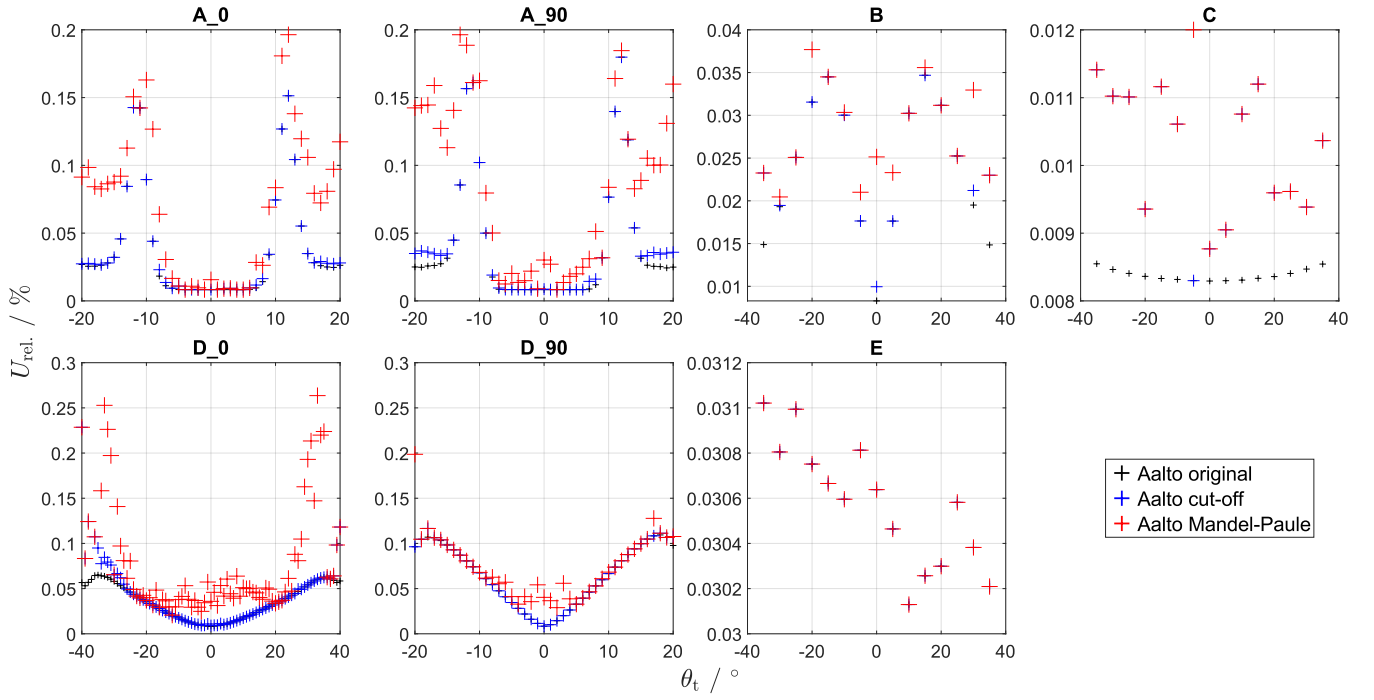


Figure S23. Development of Aalto's uncertainties (relative) at 633 nm.

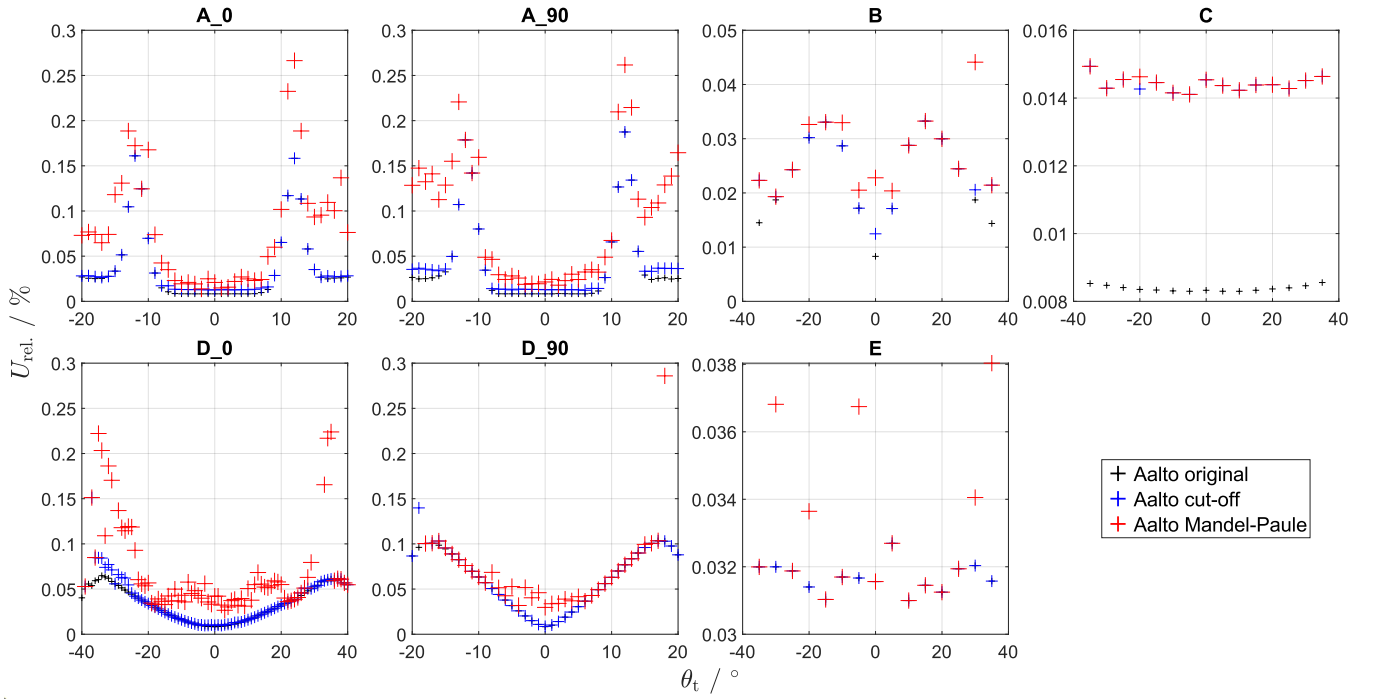


Figure S24. Development of Aalto's uncertainties (relative) at 445 nm.

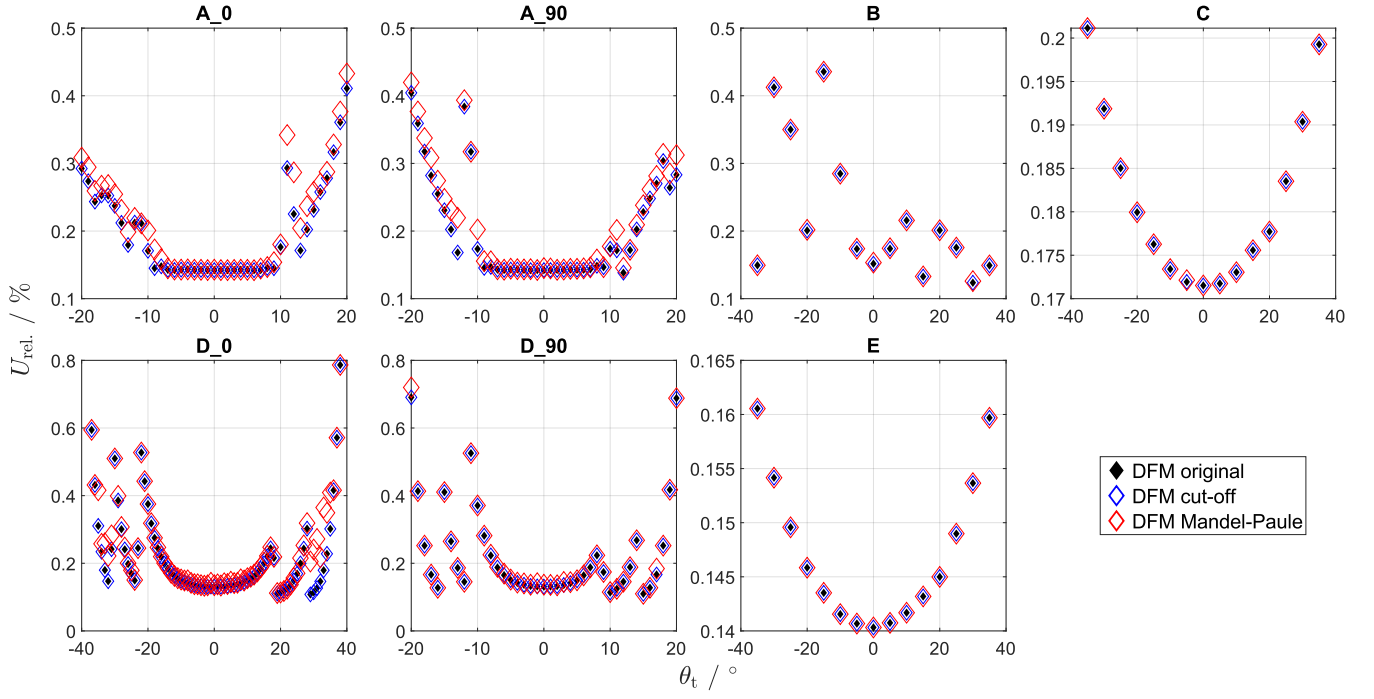


Figure S25. Development of DFM's uncertainties (relative) at 633 nm.

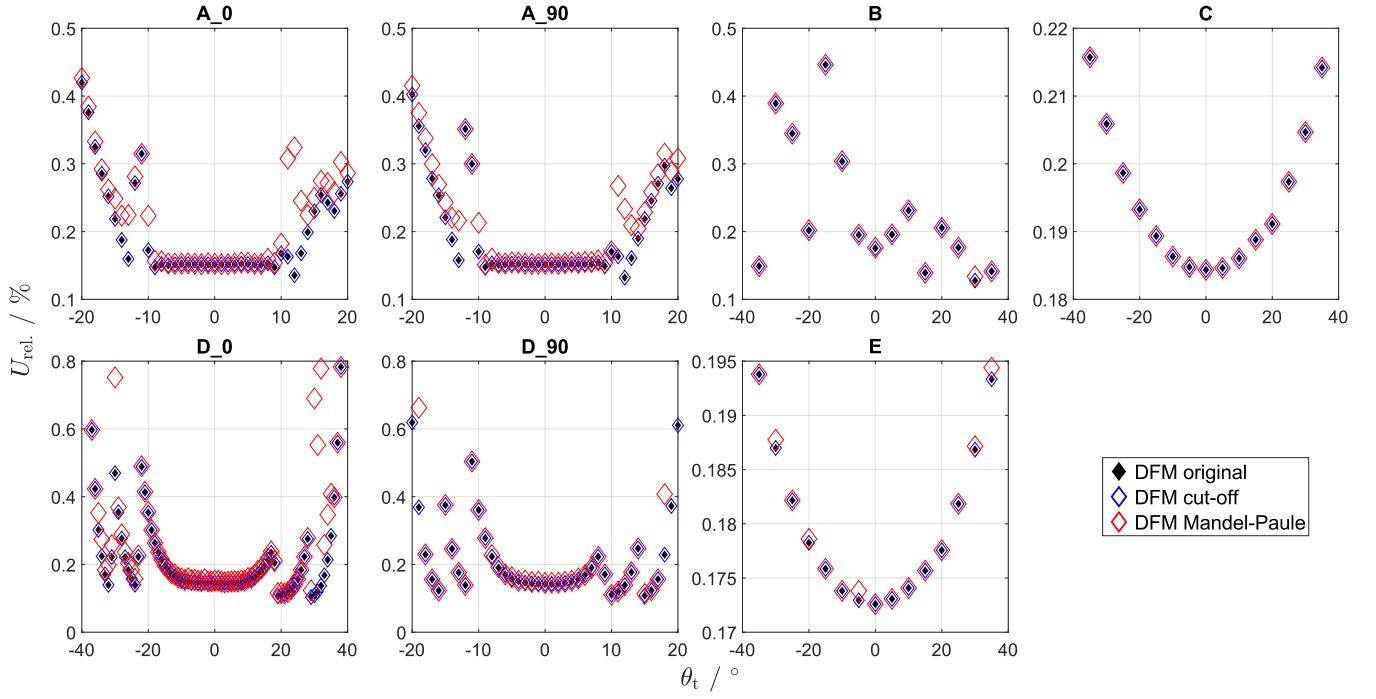


Figure S26. Development of DFM's uncertainties (relative) at 445 nm.

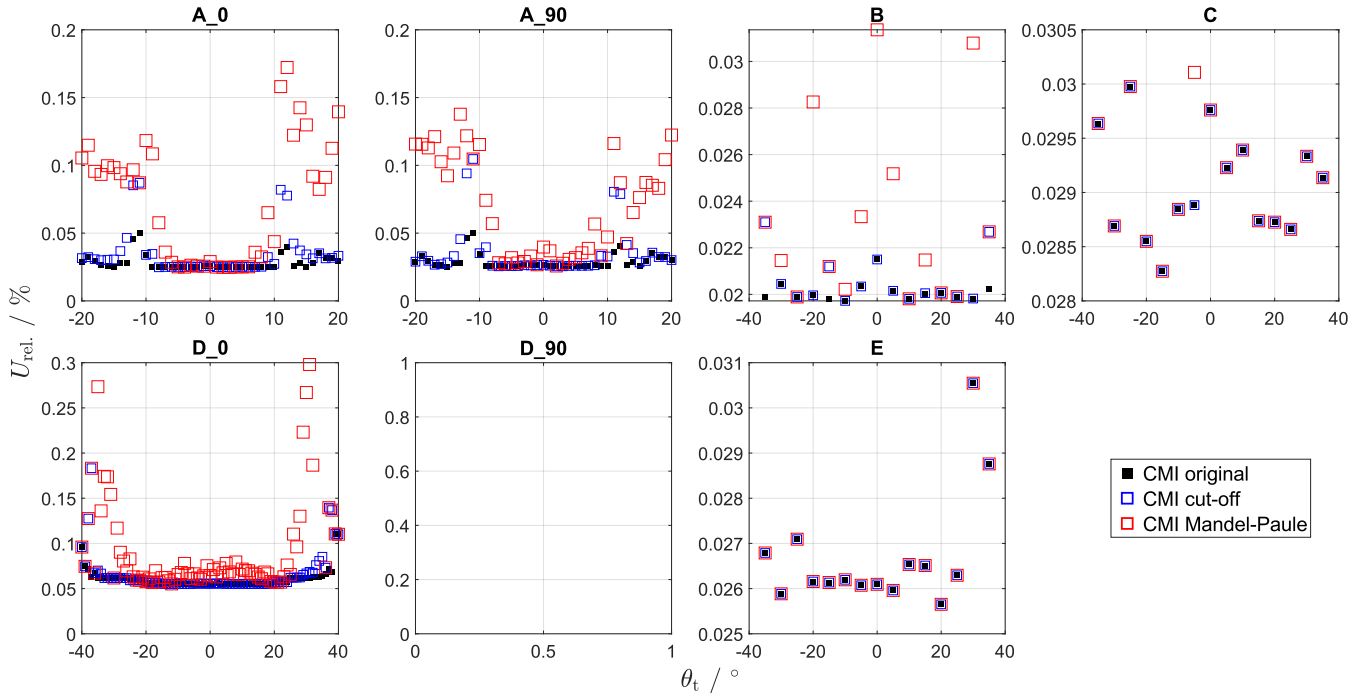


Figure S27. Development of CMI's uncertainties (relative) at 633 nm.

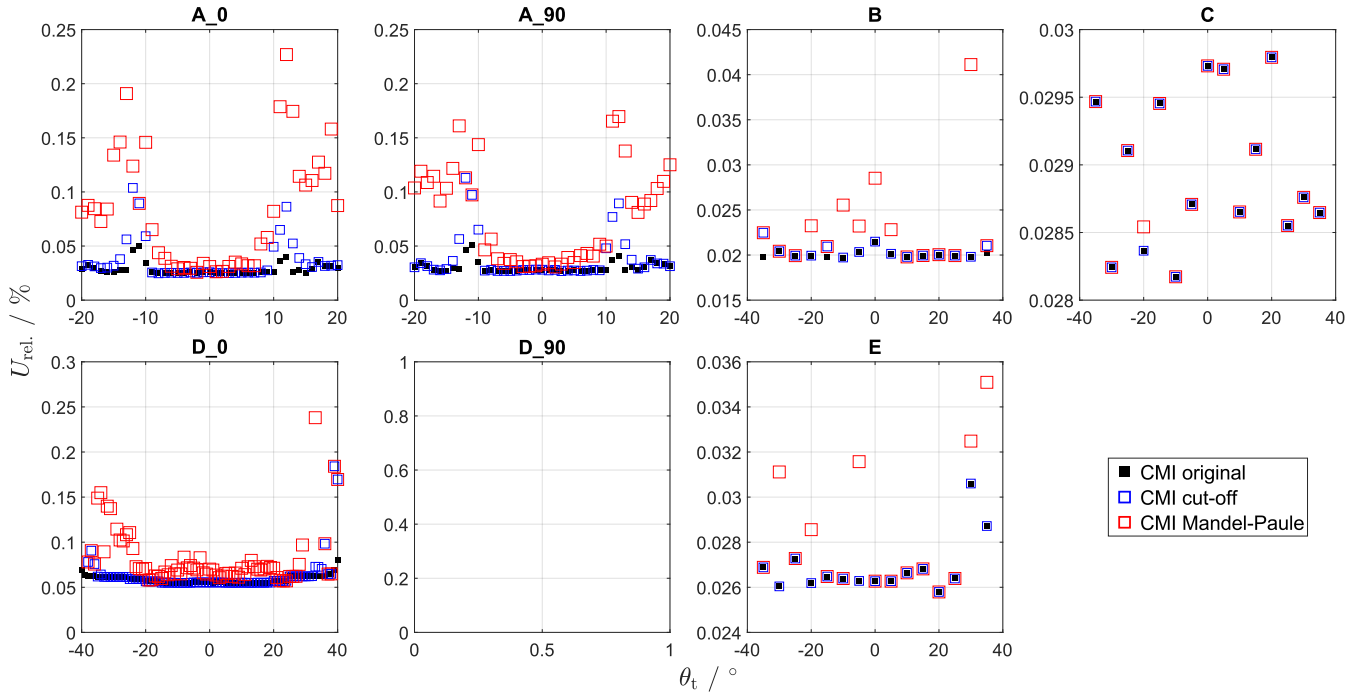


Figure S28. Development of CMI's uncertainties (relative) at 445 nm.

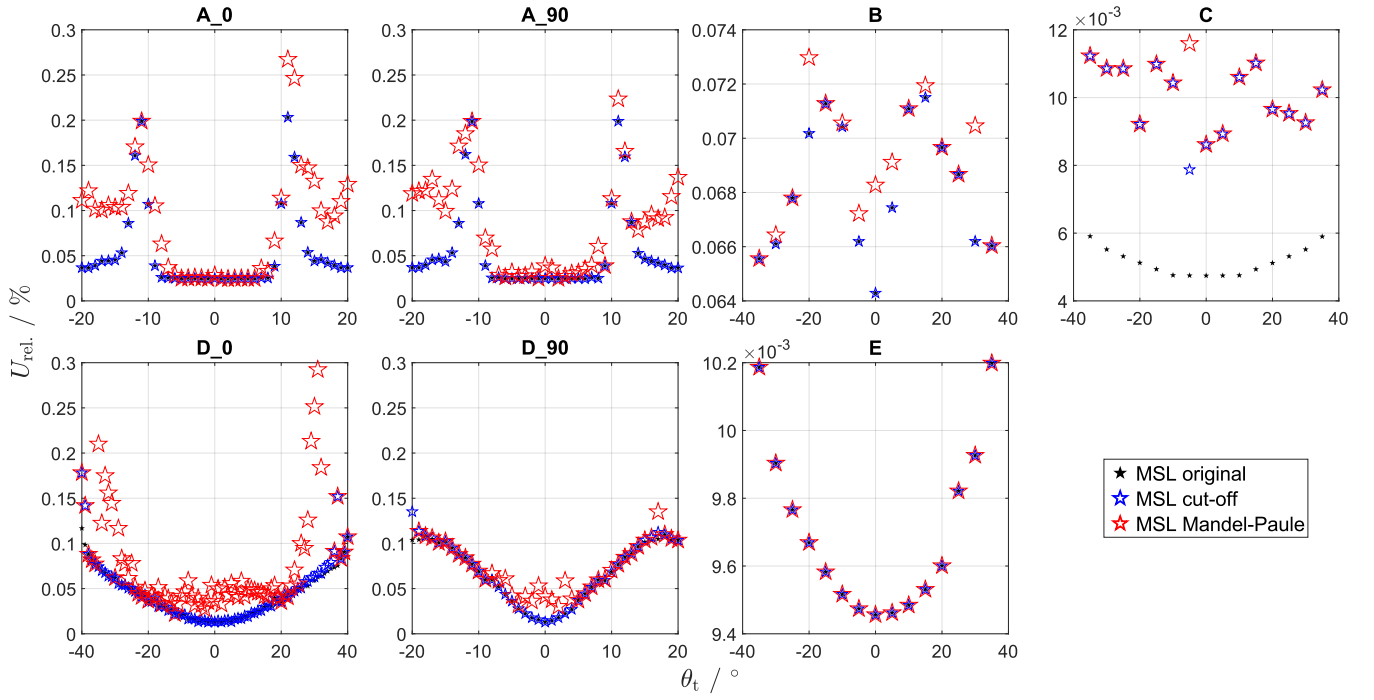


Figure S29. Development of MSL's uncertainties (relative) at 633 nm.

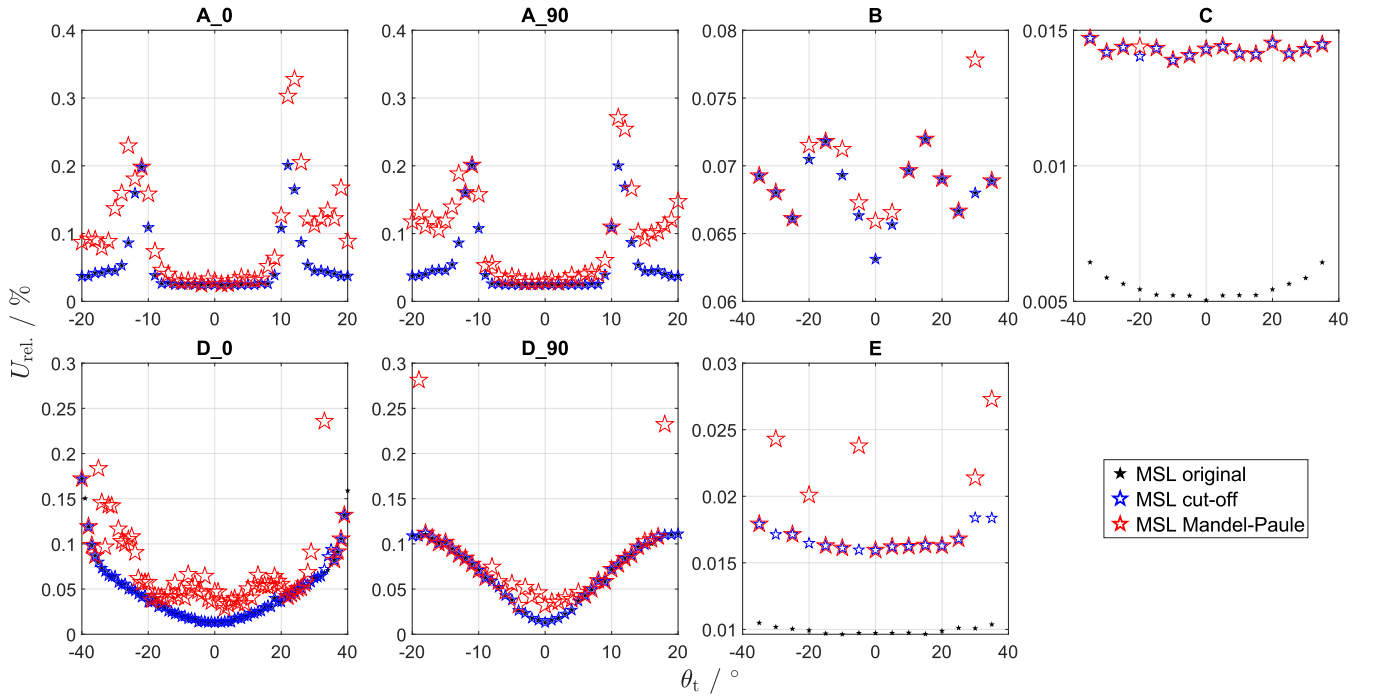


Figure S30. Development of MSL's uncertainties (relative) at 445 nm.

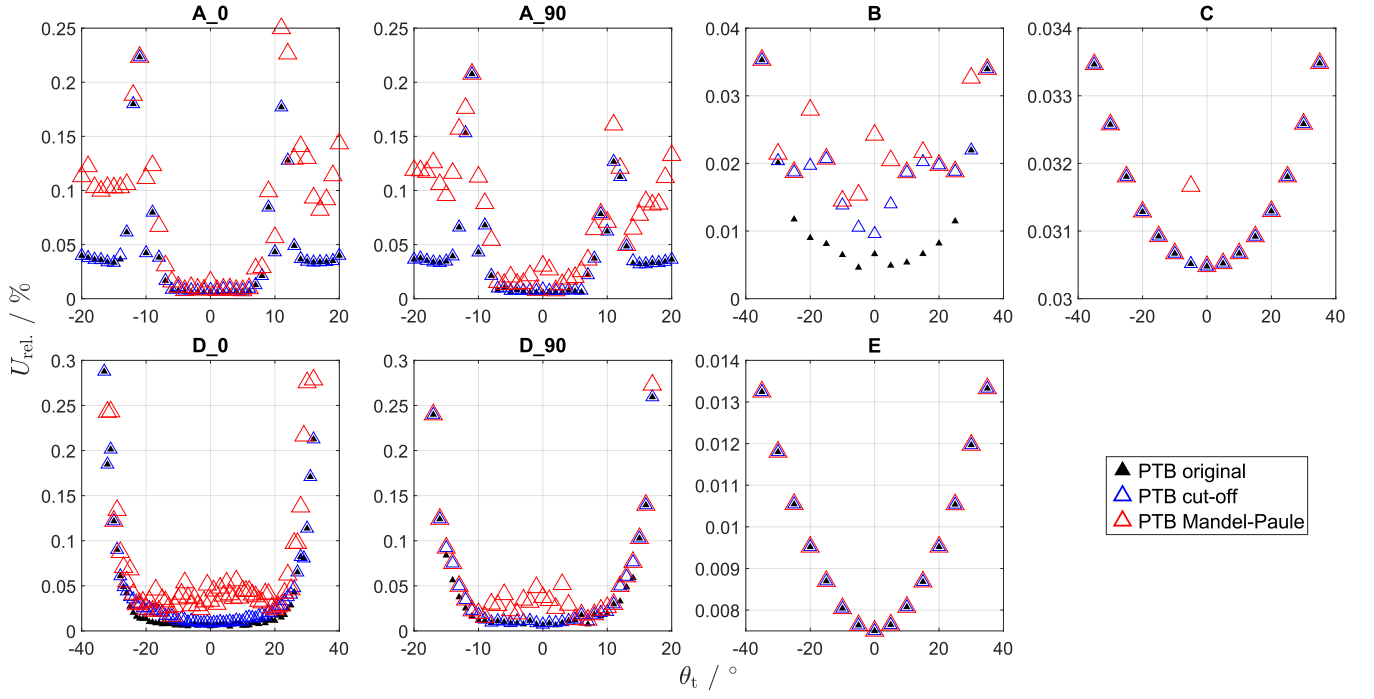


Figure S31. Development of PTB's uncertainties (relative) at 633 nm.

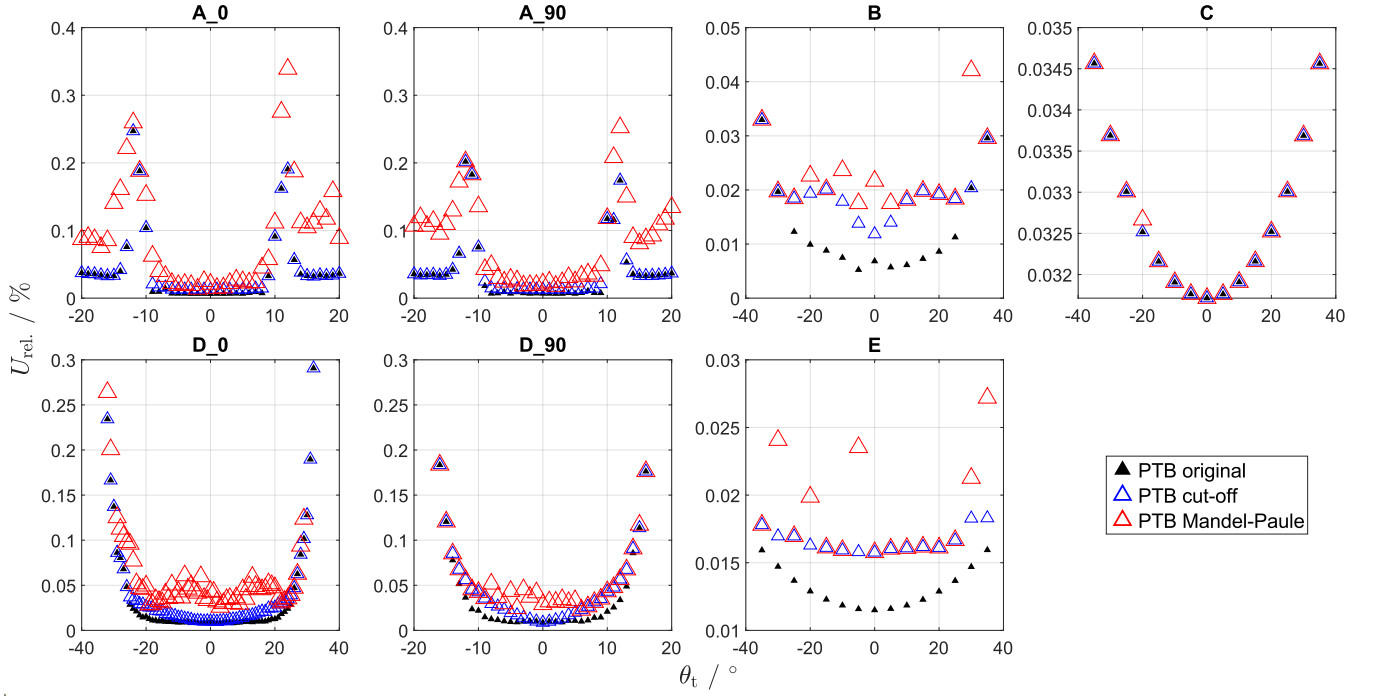


Figure S32. Development of PTB's uncertainties at (relative) 445 nm.

5 Paper IV:

Accurate characterisation of optical diffuse transmission — primary facility and extension to translucent samples

Paper Summary

In the first part of this paper, the internal BTDF comparison between the modified NaNoRef setup and the newly developed primary BTDF facility at PTB is introduced. Consistency is achieved for all samples at most of the measurement geometries, indicating a successful scale transfer. The complete uncertainty budget of the new primary facility is presented for each sample, with more details given in Section 1.3.2. In the second part, the lateral diffusion of incident radiation when performing BTDF measurements of optically thick translucent samples is thoroughly introduced and discussed. The two measurement schemes, as introduced in Section 1.3.1, are compared and analyzed. A dependence of the measured BTDF results on the incident radiant flux, which is not included in the BTDF definition, is observed from first measurement results. Discussion on this behavior is given and a phenomenological model is found to fit the results well, leading to further research proposals of the physical meaning of fit parameters and generalization of the fit model.

<https://doi.org/10.1088/1681-7575/ad615c> [160]

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Accurate characterisation of optical diffuse transmission—primary facility and extension to translucent samples

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Abstract

To perform accurate, high-quality and traceable measurement of optical diffuse transmission, which is quantified using the bidirectional transmittance distribution function (BTDF), a new primary facility has been developed at the Physikalisch–Technische Bundesanstalt (PTB), the German metrology institute. This newly developed reference facility will complement the available calibration services and research facilities at PTB for regular reflectance and transmittance and diffuse reflectance. The performance of the new BTDF setup has been investigated in an internal comparison, where consistency of the results was achieved. A thorough uncertainty analysis results in a minimum combined standard ($k = 1$) uncertainty of about 0.8 %. Research on the accurate characterisation of the BTDF of samples with non-negligible lateral scattering is being carried out on this new setup. Some first results have shown that the measured BTDF value depends on the applied irradiation area size. An empirical model has been found to describe the diffuse transmission of such translucent samples with different scattering parameters.

Keywords: diffuse transmission, BTDF, primary facility, comparison, lateral scattering, translucent material

1. Introduction

In recent years, an increase in the demand for accurate, high-quality, and traceable measurements of the optical diffuse transmission has been seen in many fields of industry and research and development, such as medical materials and prosthetics [1], food quality evaluation [2], computer graphic rendering [3, 4], and satellite-based Earth remote sensing [5,

6]. The measurand for the quantitative characterisation of optical diffuse transmission is the bidirectional transmittance distribution function (BTDF) [7–9]. It is a geometrical description of the transmitting properties of a material, defined as equation (1):

$$f_t(\theta_i, \varphi_i; \theta_t, \varphi_t) = \frac{dL_t(\theta_i, \varphi_i; \theta_t, \varphi_t)}{dE_i(\theta_i, \varphi_i)}, \quad (1)$$

where the BTDF is denoted as f_t , L_t is the radiance of the transmitted radiation and E_i is the uniform irradiance on the material surface generated by the incident radiation. The polar and azimuth angles θ and φ describe the direction of the incident and transmitted radiation, indicated by the subscripts ‘i’ and ‘t’, respectively. The BTDF of a material is also dependent

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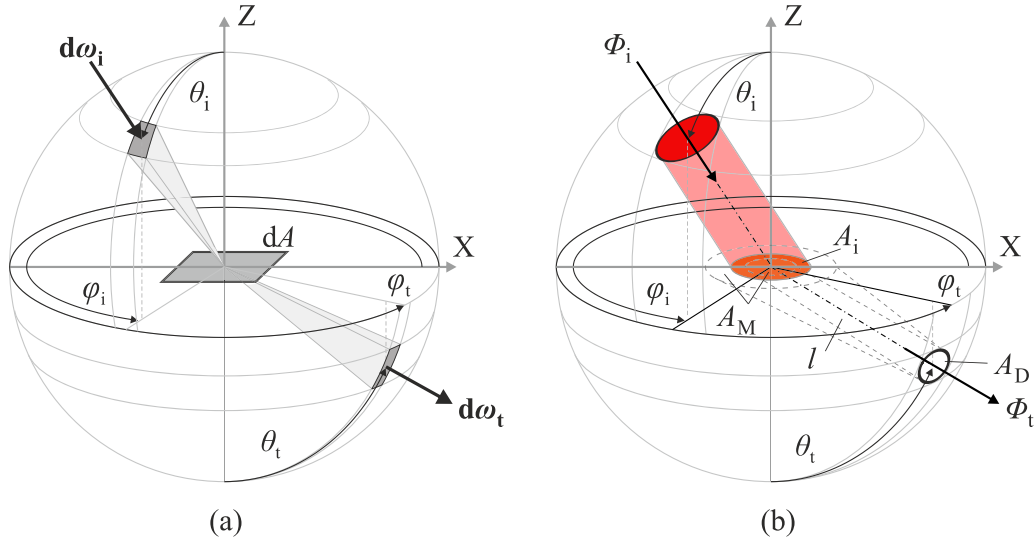


Figure 1. Definition (a) and realisation (b) of BTDF. The polar and azimuth angles of the incident and transmitted radiation are designated as θ and φ , with subscripts ‘i’ and ‘t’ indicating the incidence and transmission, respectively. The unit solid angle, in which the radiant flux Φ are accounted for in the definition, is denoted as ω . Different area sizes are indicated by the symbol A with corresponding subscripts. A_i and A_M stand for the irradiation and measurement area in the realisation, respectively, and A_D represents the size of the detector aperture area, which determines the detection solid angle together with its distance l to the sample back surface as $\omega_t = \frac{A_D}{l^2}$.

on the wavelength and polarisation state of the incident radiation and has a positive value with unit sr^{-1} . The definition and realisation of BTDF are illustrated in figure 1, with Φ the radiant flux, ω the solid angle, l the distance from sample to detector and A the area size (A_i for irradiation area, A_M for measurement area and A_D for detector aperture area).

To realise traceable BTDF measurement with high accuracy, a new reference setup (abbr. ‘BTDF primary facility’) has been developed at the Physikalisch–Technische Bundesanstalt (PTB) [10]. In this paper, vital components of the setup are introduced in detail, together with first results, an internal comparison with another BTDF setup at PTB and a thorough uncertainty analysis, showing a minimum combined uncertainty of about 0.8 % ($k = 1$). The performance of the new BTDF primary facility has been validated by the consistency of the measurement results achieved in this internal comparison as well as an indirect bilateral comparison to the primary BTDF facility of Aalto University in Finland [10]. This newly developed BTDF primary facility will complement the available calibration services and research facilities at PTB for regular reflectance and transmittance [11, 12] and diffuse reflectance [13, 14].

The flexibility in the selection of the irradiation and measurement area sizes using the BTDF primary facility allows for accurately characterising diffuse transmission of materials with non-negligible lateral scattering (referred to as ‘thick’ or ‘translucent’ in the following text). The strong lateral diffusion of the irradiating beam might result in a notable dependence of the measured BTDF value on the applied irradiation area sizes [15]. By applying an empirical relation, we aim to describe the transmittance of thick volume diffusers in a more generalised way.

2. New primary BTDF facility at PTB

The development of PTB’s new primary BTDF facility has been carried out in the framework of the EMPIR project ‘New quantities for the measurement of appearance’ (‘BxDiff’, JRP 18SIB03) [16]. This reference facility uses a robot-based goniometric measurement setup to realise the absolute determination of BTDF in arbitrary bidirectional geometries [13]. A schematic layout of the current state of PTB’s new reference setup is shown in figure 2(a) and a rendered image depicting the situation where a sample is measured when both irradiation and detection directions are perpendicular to the sample plane is shown in figure 2(b) [10].

The major components of the facility have been introduced in detail in [10]. In short, the sample under test (S), held by a six-axis robot arm (R), is irradiated by linearly polarised (P) narrow band CW lasers (LS 1 and LS 2) after beam conditioning to reduce speckle effect and generate spatially homogeneous illumination. The detection system is mounted on a rotation stage with 1.5 m diameter, whose rotation axis lies on the sample back surface facing towards the detection system. Being collected by an integrating sphere (IntS), the transmitted signal is detected unpolarised by a silicon photodiode (Si-PD) using lock-in technique (LIA) with the help of an optical chopper (Ch). Attenuation filters (F) are used to keep the signal within the dynamic range of the LIA. Interchangeable apertures (A1 and A3) of different sizes determine the irradiation area size on the sample front surface (A_i in figure 1(b), can be varied from 1 mm to 30 mm in diameter) and the measurement area size imaged by L3 on the sample back surface (A_M in figure 1(b), can be varied from 1.5 mm to 30 mm in diameter), respectively. Depending on the applied combination

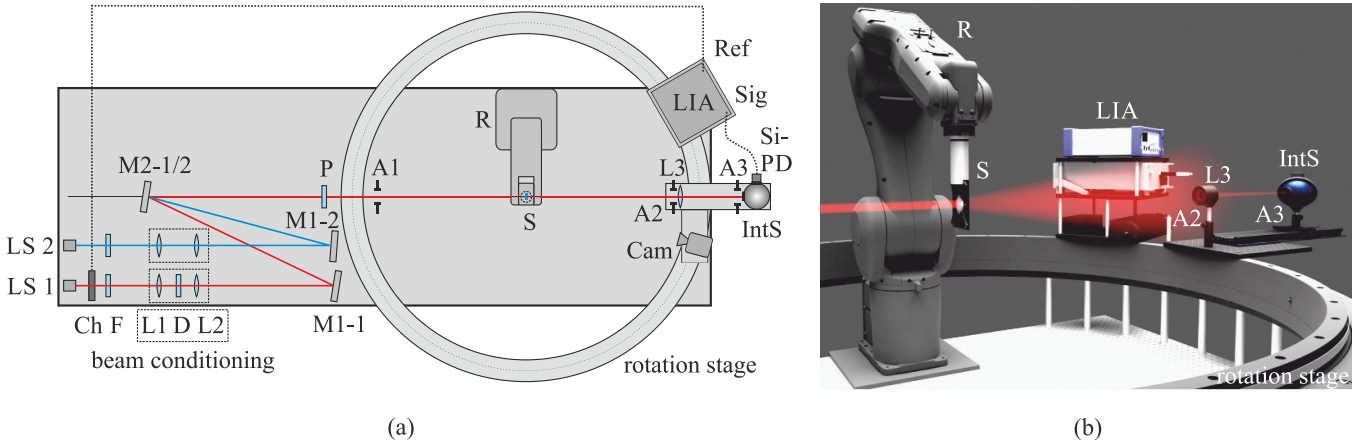


Figure 2. (a) Schematic layout of the current state of PTB's new primary BTDF facility. Abbreviations of the major components are explained in detail in the main text. (b) Rendered image of sample positioning and detection on PTB's new BTDF primary facility. For a clear visualisation, some components are not shown in this image. Adapted from [10]. CC BY 4.0.

of A_1 and A_3 , measurements can be performed in either under- ($A_i < A_M$) or over-irradiation ($A_i > A_M$) scheme. With a nominal distance of approximately 652 mm from the sample back surface, the interchangeable detector aperture A_2 (A_D in figure 1(b), can be varied from 7 mm to 35 mm in diameter) achieves different resolutions of angle-scan ranging from 0.6° to 3.1° in the full width at half-maximum (FWHM).

A low-noise camera (Cam) with 1600×1200 -pixel resolution is also mounted on the rotation stage. The plane of the sample back surface is focused using an objective attached to the Cam. It is aligned parallel to the plane of the angle-scan, which helps determine the major axis of the sample's scattering distribution or align the sample marker to the detection plane. The size of A_i can also be determined using the camera by mounting a screen with dimensional scale at the sample position. Once the setup is aligned and the sample is mounted, the measurement can be performed fully automated using an in-house developed LabVIEW programme.

3. The internal comparison for scale transfer

Another provisional setup, which is based on the modified facility for regular reflectance (abbr. 'modified NaNoRef'), has been used to perform BTDF measurement when the development of the new BTDF primary facility was in progress [17]. After the completion of the new reference setup, the measurement scale has been successfully transferred from the modified NaNoRef to the BTDF primary facility by an internal comparison, which is introduced in detail in this section. The scale transfer helped restore the calibration capacity of the original reflectance facility and provided a solid foundation for further BTDF measurement in the more difficult out-of-plane geometries.

3.1. Samples for the internal comparison

The samples studied in the internal comparison were selected in the framework of BxDiff project as a set of representative

artefacts for optical diffuse transmission studies. Their details and the BTDF measurement results of these samples are reported in [17]. For a better overview, the information of these samples is summarised in table 1.

The listed samples were used to perform the internal comparison. Samples C and E are bulk scattering samples of different material and different thickness, showing a quasi-Lambertian scattering distribution. The scattering of the other three samples is realised by a thin surface. Sample B is a ground glass diffuser, possessing a Gaussian-shaped scattering distribution with approximately 16° FWHM. Sample D is a holographic diffuser, which scatters the incident radiation with different magnitude in the two orthogonal axes, resulting in FWHMs of 26° and 13° in the two major axes of the elliptical Gaussian scattering profile. Sample A has an engineered surface consisting of microlens-units. Its functional surface generates a square-shaped scattering distribution, with a width of approximately 20° and very steep falling edges.

3.2. Results of the internal comparison

All samples were measured under similar measurement conditions on the two setups, i.e. size of the irradiation area A_i and measurement area A_M , wavelength of the incident radiation, detection angular resolution as well as incident and detection angles. The detailed parameters applied on the modified NaNoRef setup are reported in [17]. Measurement results are plotted in figures 3–7, together with the expanded ($k = 2$) uncertainty bars for each setup. Note that all components such as diaphragms, attenuation filters, sample holders, and detectors, etc. are specific for each facility and no components were calibrated by any one of the two facilities. Therefore, the influence of any possible correlations between the two facilities could be regarded as insignificant and thus was not considered in the uncertainty analysis. The relative difference between the BTDF values $\Delta f_{i,rel.}$ measured by the two setups (green diamonds, referred to the right axis) is also plotted for a better visual comparison. It is calculated using equation (2):

Table 1. Details of transmissive diffusers for the internal comparison^a.

Designation	Name	Material	Scattering distribution	Dimensions (mm)
A	Thorlabs ED1-S20	Microlens-units made of Zeonor© injection moulded on N-BK7 substrate	Square, FWHM $\sim 20^\circ/20^\circ$, functional surface	$\varnothing 25.4 \times 1.5$
B	Thorlabs DG20-220	N-BK7 substrate, active surface roughened using 220 grit	Gaussian, FWHM 16° , surface scattering	$\varnothing 50.8 \times 2$
C	Heraeus HOD®-500	Fused synthetic SiO ₂ with uniform air-bubbles ($\varnothing < 20 \mu\text{m}$)	Bulk scattering	$\varnothing 50 \times 2$
D	Temicon PAN001008	125 μm thick imprinted PET foil on standard glass substrate	Gaussian elliptical, FWHM $26^\circ/13^\circ$, holographic diffuser	$50 \times 50 \times 2$
E	SphereOptics Zenith® Polymer	Sintered polymer PTFE foil	Bulk scattering	$\varnothing 45 \times 0.25$

^a The listed samples are examples of possibly suitable products. Their choice does not constitute an endorsement by the BxDiff consortium and similar commercial products may provide equivalent results.

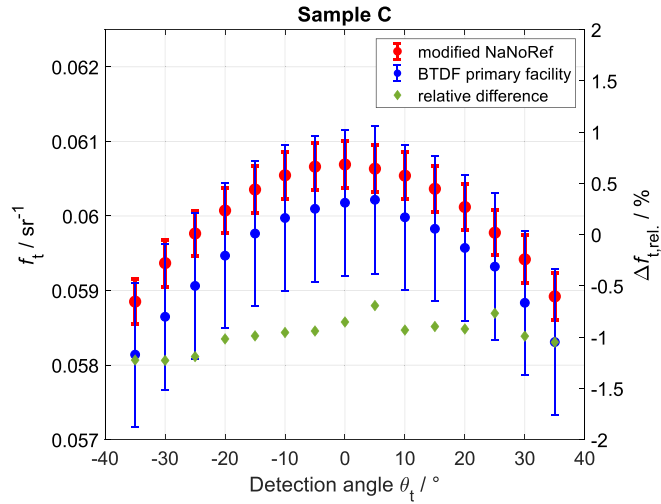


Figure 3. BTDF values f_t and the expanded ($k = 2$) uncertainties of sample C measured on the modified NaNoRef setup (red) and the BTDF primary facility (blue), together with the relative difference $\Delta f_{t,rel.}$ between the two setups (green).

$$\Delta f_{t,rel.} (\%) = \left(\frac{f_{t,pri.BTDF} - f_{t,mod.NaNoRef}}{f_{t,pri.BTDF}} \right) \cdot 100, \quad (2)$$

with $f_{t,pri.BTDF}$ being the values measured on the primary BTDF facility and $f_{t,mod.NaNoRef}$ being the values measured on the modified NaNoRef setup. Readers should note that the negative polar angles indicate $\varphi_t = 180^\circ$, and at $\theta_t = -31^\circ$ and -29° no BTDF value could be acquired on the primary BTDF facility since the incident beam is blocked by optical components on the rotation stage at these angles.

In figures 3 and 4 the results for the two Lambertian samples C and E are shown, respectively. The BTDF values measured on both facilities agree within their expanded ($k = 2$) uncertainty. Sample C has a slightly larger relative uncertainty in its BTDF due to a slightly lower transmitted signal level, indicated by the lower BTDF values, in the measurement.

For sample B, the consistency is also achieved at all detection angles, as demonstrated in figure 5. The relative difference

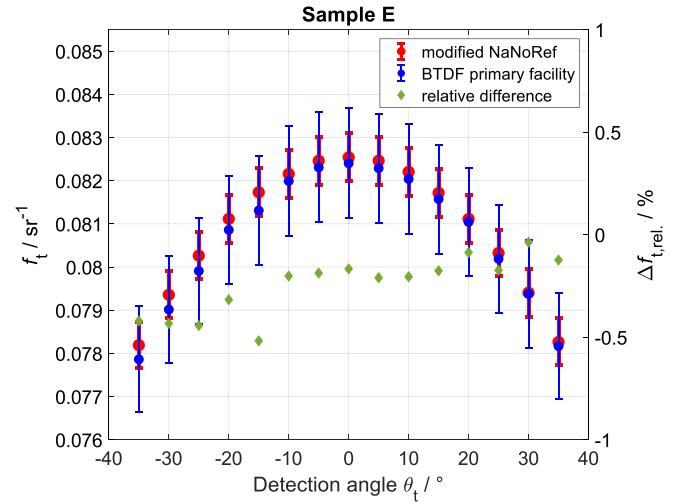


Figure 4. BTDF values f_t and the expanded ($k = 2$) uncertainties of sample E measured on the modified NaNoRef (red) setup and the BTDF primary facility (blue), together with the relative difference $\Delta f_{t,rel.}$ between the two setups (green).

in the peak angular range lies below 1 % and slightly increases to be still smaller than 3 % at larger θ_t where the transmitted signal is low.

For sample D, only the BTDF at the reference azimuth ($\varphi_t = 0^\circ$) was compared, as shown in figure 6. On both setups similar structures in the BTDF curves are observed, especially near the $\theta_t = 0^\circ$ range. In [17] this phenomenon was considered to be the effect of laser speckle, whereas from this internal comparison these variations could be concluded as sample holographic characteristics, which were resolved by applying a high resolution in the angle-scan. For this sample, consistency is achieved at most of θ_t . As will be discussed in section 3.3, the uncertainty regarding the sample characteristics was not taken into consideration for this sample. Therefore, the consistency fails at some measurement points where large variation in the BTDF distribution appears.

For sample A, only the BTDF at the reference azimuth ($\varphi_t = 0^\circ$) was compared, as illustrated in figure 7. Good

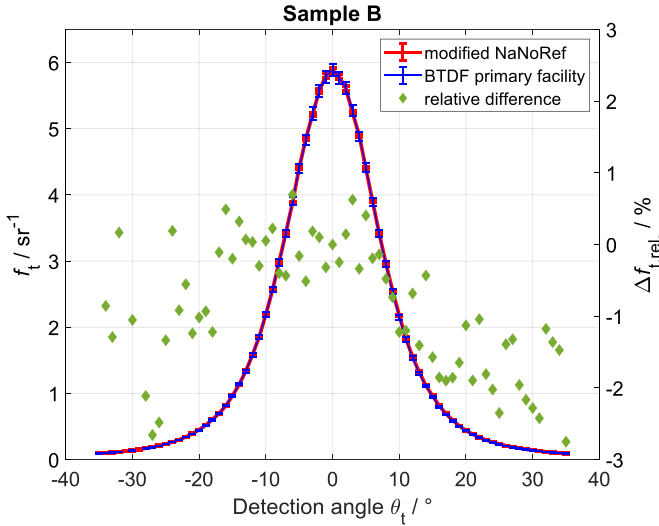


Figure 5. BTDF values f_t and the expanded ($k = 2$) uncertainties of sample B measured on the modified NaNoRef setup (red) and the BTDF primary facility (blue), together with the relative difference $\Delta f_{t,rel.}$ between the two setups (green). Adapted from [10]. CC BY 4.0.

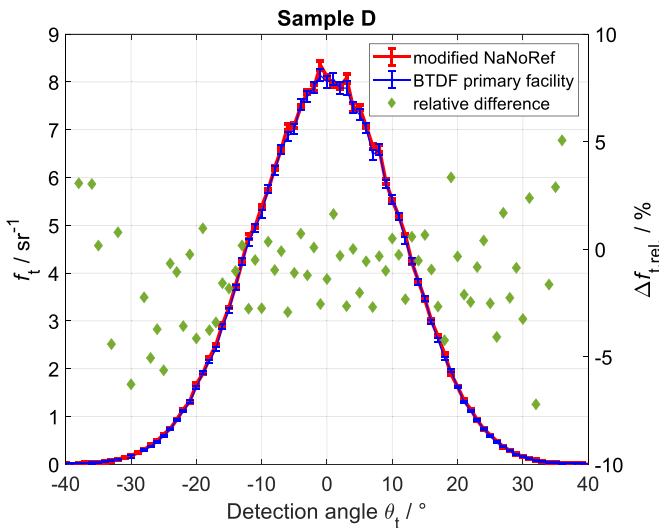


Figure 6. BTDF values f_t and the expanded ($k = 2$) uncertainties of sample D (wide azimuth orientation) measured on the modified NaNoRef (red) setup and the BTDF primary facility (blue), together with the relative difference $\Delta f_{t,rel.}$ between the two setups (green).

consistency was achieved at all θ_t between the values measured by the two setups. In the central angular range (around $\theta_t = 0^\circ$) the BTDF values are very close. The relative difference lies between zero and -1% for most of the points in this range, indicating a similar systematic deviation between the two setups as observed for all other samples as well. Large deviation appears when θ_t is at the falling edges of the BTDF distribution (approximately 8° – 12°), where the gradient of the variation in BTDF is high. As will be discussed in section 3.3, the uncertainty in this range is also estimated to be large and

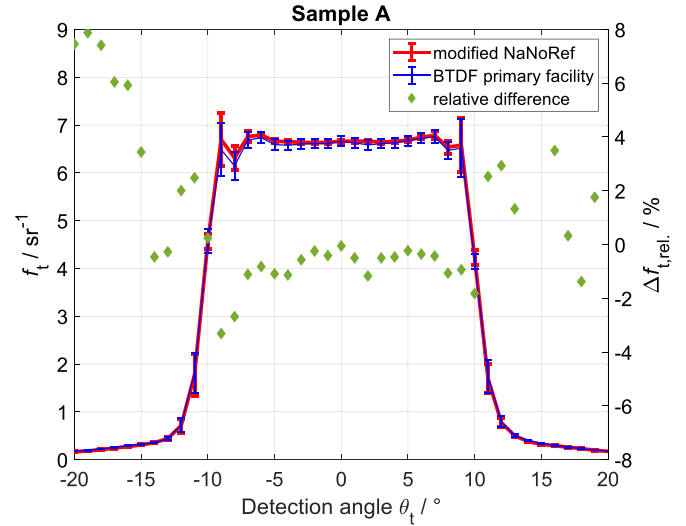


Figure 7. BTDF values f_t and the expanded ($k = 2$) uncertainties of sample A at reference azimuth ($\varphi_t = 0^\circ$) measured on the modified NaNoRef setup (red) and the BTDF primary facility (blue), together with the relative difference $\Delta f_{t,rel.}$ between the two setups (green).

therefore, the results are still consistent. In the ‘foot’ range of the curve ($\theta_t > 15^\circ$), where the BTDF values are small, the results are consistent as well. However, unlike in the central angular range, the results measured on the primary BTDF facility become slightly larger than those measured on the modified NaNoRef at approximately $\theta_t > 10^\circ$, as indicated by the positive values of the relative difference. This contradicts the systematic deviation observed in all other cases. Possible explanation for this effect will be discussed in section 3.3.

In summary, the performance of the new BTDF primary facility at PTB was validated by the internal comparison, as an overall good consistency between the two BTDF setups was achieved on the measurement of five different transmissive diffusers, leading to a successful scale transfer of BTDF measurement.

3.3. Uncertainty analysis of the new primary BTDF facility

The main components of the uncertainty budget of PTB’s new primary facility have been investigated thoroughly in [10]. Here, we present the sample-specific uncertainty budget in table 2 for the measurements presented in this paper. Note that for sample D the uncertainty was considered only for detection angle $\theta_t < 33^\circ$, as the BTDF values at larger θ_t approach zero and the uncertainty is dominated by fluctuation in measurement signal and straylight. The uncertainty regarding polarisation is generally not considered, since at perpendicular incidence no significant difference was observed when irradiating the sample with *s*- or *p*-polarised beam and no polarisation-selective scattering was observed.

Uncertainty in the transmittance of the applied attenuation filter is one of the most dominant components for all samples, because the filters were only calibrated by a commercial spectrophotometer in the current state. In the near future they will

Table 2. Uncertainty budget for the new BTDF primary facility.

Source of uncertainty	Uncertainty in BTDF (%) for each sample				
	A	B	C	D ^a	E
Measurement signal ^b	0.15–0.27	0.05–0.65	0.21–0.26	0.11–1.55	0.08–0.13
Transmittance of attenuation filter	0.74	0.74	0.74	0.74	0.74
Detector aperture area	0.03	0.03	0.03	0.04	0.03
Distance sample–detector	0.09	0.09	0.09	0.09	0.09
Detection polar angle positioning	0.01–1.59	0.01–0.25	< 0.01	0.01–0.52	< 0.01
Wavelength	0.01–0.02	0.01–0.02	0.01	0.01–0.02	0.01
Detection angular resolution	0.01–11.15	0.01–0.36	< 0.01	0.01–0.43	< 0.01
Beam spatial nonuniformity	0.01–0.02	0.01	< 0.01	< 0.01	< 0.01
Inter-reflection	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Straylight ^b	0.01–0.40	0.01–0.19	0.23–0.29	0.06–6.75	0.17–0.22
Sample azimuth misalignment	0.10–1.50	—	—	0.01	—
Combined ($k = 1$)	0.77–11.39	0.77–1.12	0.81–0.84	0.76–6.99	0.77–0.79

^a The uncertainty of sample D is only considered for $\theta_t < 33^\circ$ as the BTDF value at larger detection angle approaches zero.

^b This uncertainty includes the influence of both irradiating and transmitted signals.

be calibrated at the primary facility for regular transmittance at PTB [11, 12] and the relative uncertainty ($k = 1$) of this component can be reduced to be below 0.2 %.

For the two quasi-Lambertian diffusers, the combined standard ($k = 1$) uncertainty is kept below 1 % at all detection angles since the transmitted signal is quite constant within the detection angle range, and therefore, the measured BTDF is not sensitive to possible misalignment in detection angle position and uncertainty in angular resolution. For the other samples this is different, as indicated by the large variation range in the uncertainty components regarding measurement signal, straylight, detection angle position and angular resolution. For sample A, the uncertainty of the detection angular resolution becomes extremely large at the sharp falling edges, as any slight misalignment in θ_t would lead to great change in the measured BTDF value. The detection angular resolution dominates the uncertainty budget of sample A in this angular range, since the finite extent of the applied detection solid angle flattens the slope of the falling edge. This also holds for sample B, as the uncertainty regarding detection angular resolution is large at the peak and the flank (at around $|\theta_t| = 15^\circ$) of the BTDF curve, where the variation in the slope ($\frac{\partial^2 f_t}{\partial \theta_t^2}$) is large.

For samples A and D, an additional component regarding the azimuth misalignment was considered in the uncertainty budget. This component could explain the phenomenon regarding the reversed systematic deviation of sample A between the two setups. In the central angular range of a square-shaped distribution, the BTDF is insensitive to azimuth change. Thus, a similar systematic deviation between the two setups is found as for the other samples. At the falling edges and in the ‘foot’ range of the distribution, the BTDF changes drastically when the azimuth is slightly different. Since the reference azimuth of this sample was marked by a black dot with finite size on its back surface and was aligned using the camera, it is highly possible that the measurements on different setups were performed at slightly different sample

azimuths. Therefore, this error in realising $\varphi_t = 0^\circ$ leads to the inconsistency in the inter-setup relative difference observed from the results of sample A. Nevertheless, this component also proves that the uncertainty was correctly analysed as consistency in the measurement results was generally achieved for this sample.

3.4. Future improvements of the primary BTDF facility

In the near future, the extension of the available irradiation wavelength will be carried out by integrating two broadband light sources, a supercontinuum laser covering the visible (VIS) and near infrared (NIR) range and a laser-driven light source for the ultraviolet (UV) to VIS range, to the current setup. Both light sources will be coupled into a double monochromator through fibre optics and spectrally selected irradiation will also be outcoupled using fibre collimators to realise different irradiation area sizes. It is expected that spectral information of the sample under test can be obtained in the range from approximately 250 nm to 2100 nm.

To measure the broadband spectral information, upgrade for the current detection system is also planned. For example, different detectors will be needed to collect the radiation in different wavelength ranges. A camera-based detection system such as a spectroradiometer or a 2D camera would also help increase the measurement efficiency and gather spatial information, allowing for BTDF measurement of samples possessing anisotropic, structured, or even curved surfaces.

4. Transmission of diffusers with non-negligible lateral scattering

As described in section 2, the diameter of the irradiation area A_i can be varied between 1 mm and 30 mm and the diameter of the measurement area A_M can be varied between 1.5 mm and 30 mm on the new BTDF primary facility. By applying

a smaller A_i than A_M , the measurement can be performed in the under-irradiation scheme, whereas a larger A_i than A_M switches the measurement scheme into the over-irradiation mode. In this section the difference of applying these schemes to the BTDF measurement of samples with strong lateral scattering is discussed. For these translucent samples, the lateral scattering of the incident radiation can be regarded as the result of multiple subsurface scattering during transmission through the material. It can be observed by the size difference between the irradiated area on the front and back surface of the sample. Results of this type of samples measured with different measurement parameters are presented. To describe the observed dependence of the measured BTDF on the irradiation area size A_i , an empirical function was found and the relation of the free parameters to the fundamental scattering quantities [15], i.e. BTDF and the bidirectional bipositional subsurface scattering function (BBSS) is discussed.

4.1. Under- and over-irradiation schemes for measuring translucent diffusers

The under-irradiation measurement scheme is more generally applied than the over-irradiation scheme due to its simple and insensitive alignment and efficiency in signal power exploitation [9, 18]. In this scheme the BTDF is determined as the average value of radiant fluxes [15]:

$$f_t = \frac{\Phi_t}{\Phi_i} \cdot \frac{1}{\omega_i \cos \theta_t} \cdot \frac{A_i}{A_t}, \quad (3)$$

where the meaning of parameters can be taken from figure 1(b). Here, the different sample surfaces are distinguished by using A_i for the irradiated area on the front surface (facing towards irradiation source) and A_t for the area on the back surface (facing towards detection) in which all transmitted radiant flux is included.

For transmissive diffusers with negligible lateral scattering, the size difference between A_t and A_i is insignificant and equation (3) can be simplified to:

$$f_t = \frac{\Phi_t}{\Phi_i} \cdot \frac{1}{\omega_i \cos \theta_t}, \quad (4)$$

which is the equation applied in [10, 17–19] to evaluate BTDF measurements. Thus, by applying the under-irradiation scheme the BTDF of this type of samples can be appropriately measured.

However, for translucent samples with strong lateral scattering, measurements using the under-irradiation scheme cannot be evaluated using the simplified equation (4) any more. Since the beam size A_t on the sample back surface evolved greatly due to lateral scattering, if the measurement area A_M is not large enough to include the whole A_t , the measured BTDF will be smaller as a result of loss in collecting the transmitted radiant flux Φ_t . This situation is illustrated in figure 8(a). The measured BTDF value would then depend on the applied A_M , leading to incompatible measurement results. Note that uncertainty regarding this effect was not considered in the uncertainty budget for all samples in the internal

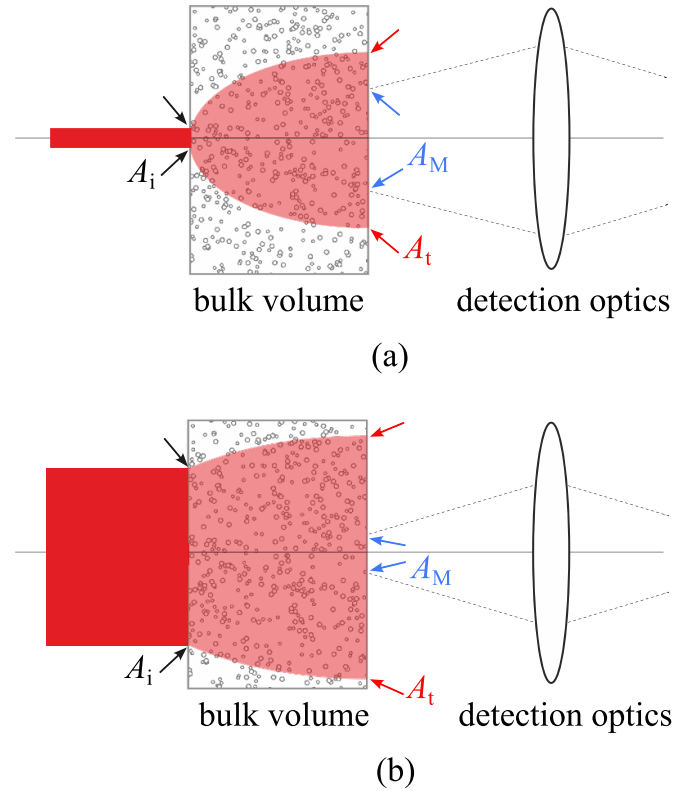


Figure 8. Schematic drawing of BTDF measurement of samples with non-negligible lateral scattering in (a) under- ($A_i < A_M$) and (b) over-irradiation ($A_i > A_M$) scheme. As strongly scattered by the thick bulk volume in the lateral direction (difference in A_i and A_t), the transmitted radiation is measured more accurately using the over-irradiation scheme.

comparison mentioned in section 3, since the applied measurement parameters on both facilities were similar.

In the under-irradiation scheme, in order to obtain the correct BTDF value, an A_M as large as possible should be applied. However, the projection of A_M also increases as the detection angle θ_t becomes larger and would eventually be limited by the physical size of the sample under test. Either a very large A_M or a very large sample would be difficult to realise. In addition, for translucent samples with strong lateral scattering, the spatial radiance distribution of the transmitted beam on the sample back surface is not uniform anymore, and the measured BTDF would not represent an arbitrary position on the sample, but the average across the measurement area. Therefore, following the discussions in [8, 15, 20] of BRDF measurement of highly diffuse materials or samples with significant subsurface scattering, it is also recommended to perform BTDF measurement of translucent samples by applying the over-irradiation scheme, as shown in figure 8(b). In this scheme, the evaluation of BTDF should follow equation (5):

$$f_t = \frac{L_t}{L_i} \cdot \frac{1}{\omega_i \cos \theta_t}, \quad (5)$$

where ω_i is the solid angle of the incident beam. Despite the challenges of applying the over-irradiation scheme as

mentioned in [9, 20], it provides the accessibility of varying only the incident radiant flux Φ_i while keeping the incident irradiance E_i and the measurement area A_M constant. This allows for a systematic study of the flux-dependent behaviour of the diffuse transmission of translucent materials.

4.2. Flux-dependent diffuse transmission

It was proposed in [15] that the transmitting properties of translucent samples can be accurately characterised by measuring another fundamental quantity, which describes the significant contribution coming from subsurface scattering. This quantity is the BBSS, with its definition shown in equation (6):

$$f_{\text{BBSS}}(\mathbf{x}_i, \mathbf{r}_i; \mathbf{x}_t, \mathbf{r}_t) = \frac{dL_t(\mathbf{x}_i, \mathbf{r}_i; \mathbf{x}_t, \mathbf{r}_t)}{d\Phi_i(\mathbf{x}_i, \mathbf{r}_i)}, \quad (6)$$

where $\mathbf{x}$ represents the spatial position of the radiation on the sample surface and $\mathbf{r}$ is the direction of the radiation, given by θ and φ . However, such measurement is very complex as the BBSS has eight geometrical dimensions. Instead, we propose that using results of BTDF measurement performed in the over-irradiation scheme for varying incident flux Φ_i , the diffuse transmission of such samples could also be accurately described by applying a phenomenological model. In this scheme, the radiance of the transmitted radiation L_t of the translucent samples will depend not only on E_i , as stated in the BTDF definition in equation (1), but also on Φ_i . This dependence of L_t on Φ_i is strictly described by the BBSS, which is related to the volume scattering, sample thickness, and even detection angle. In these BTDF measurements, we assume that the incident beam generates a uniform irradiance E_i on the sample front surface. Thus, the dependence of L_t on Φ_i can be represented by the dependence of L_t on the irradiated area A_i .

This means that by increasing the irradiation beam size A_i while maintaining E_i , i.e. increasing Φ_i , the radiance of the transmitted radiation L_t should also increase. This effect has been observed by performing BTDF measurements on translucent volume diffusers with 6.4 mm thickness using the new primary BTDF facility in the over-irradiation scheme. A photo of the investigated samples is shown in figure 9, illustrating the visual effect provided by the different scattering parameters, such as size and mass concentration of the scattering particles, detailed in table 3.

In order to compare the influence of different scattering parameters on the flux-dependent diffuse transmission, the factors L_t/E_i , which would correspond with the BTDF values introduced in the previous text, are plotted, normalised to the value measured with the largest applied A_i , in figure 10 as a function of A_i for the samples mentioned above. In the measurement, A_M was kept constant with 5 mm diameter while A_i varied with diameters ranging from 6.1 mm to 30 mm. The incidence and detection directions were both perpendicular to the sample plane.

For samples with less pronounced volume scattering, e.g. S1, the L_t/E_i shows no significant increase with larger A_i ,

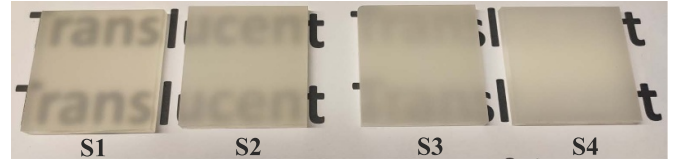


Figure 9. Photo of translucent volume diffusers with non-negligible lateral scattering characteristics, investigated using the primary BTDF facility in the over-irradiation scheme. The magnitude of lateral scattering increases from left to right, as indicated by the visual effect of translucency provided by the different scattering parameters of the sample.

Table 3. Details of samples with non-negligible lateral scattering reported in this work.

Designation	Particle size (mean diameter)	Mass concentration	Index of Refraction (589.3 nm)
S1	8–10 μm	1.5 %	1.535
S2	8–10 μm	3.0 %	1.535
S3	2.2 μm	0.3 %	1.42
S4	0.8–1 μm	1.25 %	1.49

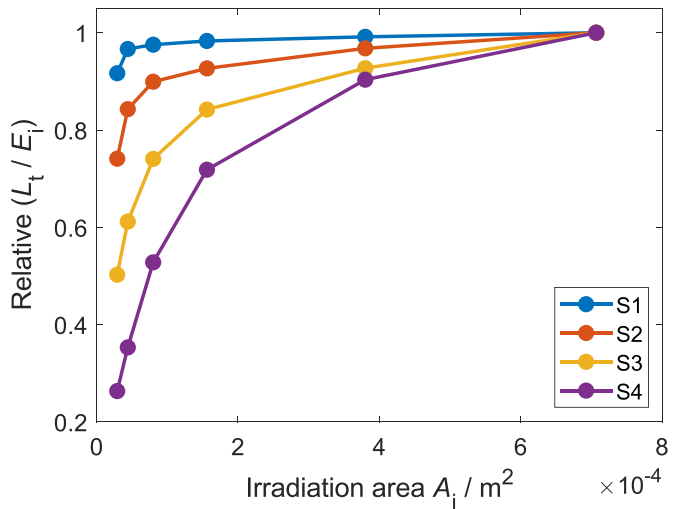


Figure 10. Relative values of L_t/E_i as function of A_i , with diameters ranging from 6.1 mm to 30 mm, measured at 642 nm under perpendicular incidence and detection, for four 6.4 mm thick polycarbonate volume-scattering diffusers with different scattering properties. A_M was kept constant at a diameter of 5 mm. Results are normalised to the value measured with the largest applied A_i for each sample.

whereas for very translucent samples, e.g. S4, the dependence of diffuse transmission on A_i or the incident radiant flux Φ_i is obvious. This phenomenon is elaborated in the schematic drawing in figure 11. For thick samples, an increased portion of the incident radiant flux $d\Phi_i$ could be scattered multiple times by the bulk volume and enter into A_M , causing an increase in the collected L_t . In contrast, the insignificant lateral scattering of thin samples would not result in any change in the collected L_t , as $d\Phi_i$ cannot be scattered

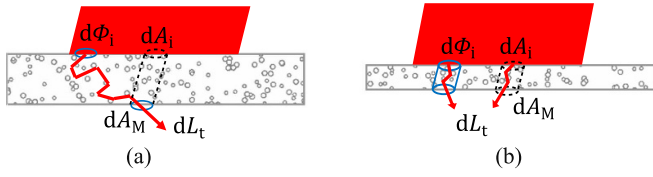


Figure 11. Schematic drawing to illustrate the contribution of incident radiant flux Φ_i to the collected transmitted radiance L_t for (a) thick and (b) thin samples. As scattered by the thick bulk volume, the lateral displacement of the transmitted radiation cannot be neglected and could result in an increase in the collected L_t within the measurement area A_M .

with significant lateral displacement to its original incident position.

4.3. Diffuse transmission of arbitrary diffusers

As discussed in the previous section, it is desired to have a generalised way of quantitatively characterising the diffuse transmission of any kind of diffusers measured under different conditions. It has been observed that L_t increases with A_i for all the measured samples, showing an asymptotic behaviour for very large A_i . This asymptotic behaviour depends on the scattering properties of the sample. On the other hand, for a very small A_i it is believed that the measured L_t should be predominantly generated by volume scattering, and thus related to the BBSS of the sample.

Currently, an empirical function has been found to fit the described dependence of the measured L_t/E_i value on A_i for all samples, as written in equation (7):

$$\frac{L_t}{E_i} = a \left(1 - \left(1 + \frac{b}{c} A_i \right)^{-c} \right), \quad (7)$$

with a , b and c as free parameters and A_i as the independent variable. It is assumed that the fit parameters could be related to the fundamental scattering quantities. For a very large A_i , the ratio approaches a , which coincides with the BTDF of the sample for an infinite value of A_i . This BTDF, analogously to the BRDF definition for translucent samples [8], is experimentally obtained when using large enough irradiation areas to keep the BTDF at larger areas invariable. For a very small A_i , the proposed empirical function depicts a linear dependence of $\frac{L_t}{E_i}$ on A_i with slope $a \cdot b$, which is expected from the theory described in [15] and therefore might be related to the BBSS of the sample. Parameter c might be regarded as a metric related to the magnitude of the volume scattering, depending on the scattering characteristics of the sample.

As an example, the L_t/E_i values of sample S3 measured with different A_i at perpendicular incidence and detection are plotted in figure 12, together with the fit using the proposed function in equation (7). The grey band in the figure depicts the 95 % confidence interval of the fit. All measurement points with their $k = 2$ uncertainty bars lie within this band, indicating a good agreement between the fit and the results.

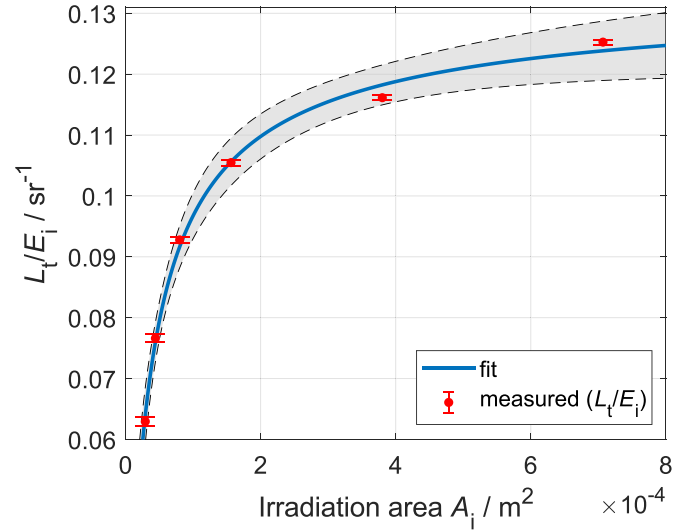


Figure 12. L_t/E_i values of sample S3 (red dots with bars showing their expanded $k = 2$ type A uncertainty only) as function of A_i , with diameters ranging from 6.1 mm to 30 mm, measured at 642 nm under perpendicular incidence and detection. A_M was kept constant at a diameter of 5 mm. Results are fitted with the proposed empirical function (blue line), with the grey band between the two black dashed lines depicting the 95 % confidence interval of the fit.

The values of the fit parameters and their standard uncertainty are listed for the four measured translucent samples in table 4. The parameter a was determined with small uncertainty compared to the other two parameters from the curve fitting process. The relative uncertainty of parameters b and c decreases as the lateral scattering becomes stronger. These results indicate that for completely characterising the transmittance of samples with non-negligible lateral scattering, the three parameters a , b , and c must be determined, using the strategy consisting of measuring the factor L_t/E_i at different A_i in the over-irradiation scheme. However, more measurement points would be needed to improve the goodness of the fit and lower the uncertainty of the fit parameters. A broader selection of thick translucent samples with varying scattering parameters will help deepening the understanding of the physical meaning of the free parameters in this empirical function. Measurements in different geometries other than the perpendicular incidence and detection would also need to be studied, and the applicability of the proposed empirical function should be tested on the results in those geometries.

5. Conclusion

In this work, a newly developed facility for BTDF measurements at PTB has been introduced. Its performance has been validated by an internal comparison on five different transmissive diffusers. Results of the comparison have been reported in detail, with a thorough sample-specific uncertainty analysis demonstrating an overall good consistency and indicating improvement potentials and future action points as well. After the capability of this measurement setup has been proven to

Table 4. Fit parameters and their standard uncertainty for the investigated samples.

Fit parameter	S1	S2	S3	S4
a / sr^{-1}	0.3271 ± 0.0019	0.1709 ± 0.0049	0.1349 ± 0.0067	0.1118 ± 0.0035
$b / \times 10^3 \text{ m}^{-2}$	151 ± 63	104 ± 37	36.5 ± 3.4	10.7 ± 0.3
$c / \text{unitless}$	2.71 ± 1.95	1.02 ± 0.47	0.68 ± 0.19	1.29 ± 0.27
$a \cdot b / \times 10^3 \text{ sr}^{-1} \text{ m}^{-2}$	50 ± 21	17.7 ± 6.3	4.93 ± 0.52	1.193 ± 0.048

be suited as a primary calibration and research facility, BTDF measurements of diffusers with non-negligible lateral scattering have been performed in the over-irradiation scheme. A dependence of the measured diffuse transmittance values on the incident beam size has been observed and could be fitted with an empirical function. Further studies are required for a physical understanding of the fit parameters and for a quantitative description of arbitrary diffusers under arbitrary measurement conditions by obtaining results from BTDF and BBSS measurements.

Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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6 Conclusion & Outlook

In this dissertation, I presented my contributions toward accurate BTDF characterization of transmissive and translucent materials from my time as Ph.D. candidate in the Working Group 4.51 “Reflection and Transmission” at PTB. Focuses were laid on the development of a multidimensional BTDF measuring facility with high-accuracy as well as on the metrological BTDF characterization of materials with distinctive scattering properties.

The main findings and achievements can be summarized as follows:

- An overview of the best capabilities worldwide of performing angle-resolved measurement of optical scattering is provided with detailed parameters as reference for development of new measuring facilities.
- A new primary facility was successfully developed at PTB for BTDF measurements. In the current state, the BTDF of samples up to 8 kg (incl. holder) can be measured with angular resolution ranging from 0.6° to 3° at wavelengths 642 nm and 445 nm with spectral bandwidth smaller than 1 nm. The BTDF measurement capabilities of this new primary facility were validated by a successful internal scale transfer as well as a bilateral comparison with another BTDF primary facility at Aalto, the Finnish NMI.
- In the bilateral comparison with Aalto, the out-of-plane BTDF measurement of a sample with Gaussian distribution was metrologically studied for the first time. Consistency was achieved at wavelengths in VIS.
- The measurement models applied to obtain BTDF values using the new primary facility are thoroughly introduced. The uncertainty analysis took as many sources as possible into consideration based on current knowledge and resulted in a minimum combined standard uncertainty of about 0.8 % relative to the measurement result in the best case.
- The measurement schemes for BTDF characterization were discussed in detail. The under-irradiation scheme is normally applied to measure transmissive sur-

face scattering samples, often exhibiting a narrow BTDF, while for bulk scattering samples depicting a quasi-Lambertian distribution the over-irradiation scheme achieves better accuracy. In order to perform accurate BTDF measurements for all types of materials, efforts were intentionally made for the realization of a flexible alternation between these two schemes.

- The non-negligible lateral scattering of incident radiation by translucent materials was identified to be one main reason for the discrepancy observed in the interlaboratory BTDF comparison, because it introduces a radiant-flux-dependent transmittance in the measured BTDF. The flexibility of switching measurement schemes provided an experimentally feasible way to further study accurate BTDF characterization of translucent materials. An empirical model was found to describe the dependence well.
- Possible sources contributing to the discrepancy in the interlaboratory comparison other than lateral scattering were thoroughly analyzed, revealing that the instrument function was of the greatest importance in accurate BTDF measurement.
- The two reported intercomparisons were the first metrological studies regarding BTDF characterization of samples with not only quasi-Lambertian but also far-off-Lambertian scattering distributions. They provided valuable information and laid solid foundation for a future Key Comparison of this measurand.

Along with the highlighted findings, main components for irradiation, sample-positioning, and detection of the new primary BTDF facility were introduced with detailed parameters. It was discussed that requirements on irradiation beam divergence and spatial intensity uniformity are different for the two schemes and tradeoffs have to be made. Means to adjust them depending on sample characteristics were presented. The importance of a well-defined measurement area on the sample back surface achieved by using an imaging detection system was also illustrated. Methods to determine sample's reference azimuthal orientation were thoroughly introduced and compared, showing consistent results within the uncertainty of realization.

Other than that, the temporary modified NaNoRef setup involved in the BTDF intercomparison was also introduced. This setup helped gather experience in accurate BTDF measurements and provided vital knowledge on the investigated samples, which were presented with details of their materials, geometrical sizes, and scattering behaviors. Furthermore, the angle-resolved spectral transmittance in VIS of

the investigated samples was measured using a commercial device. It was observed that the spectral dependence varies with the transmission angle and was the most obvious for samples depicting surface scattering properties. In collaboration with *Danmarks Tekniske Universitet* (DTU), this observation was successfully explained by simulation using a microfacet model.

All the above mentioned findings, most of which were published in the Papers presented in this work, constitute the scientific core of this work, accompanied by an extensive introduction to the theoretical background of BTDF and other relevant goniospectrophotometric quantities.

Future perspectives: The spectral range of the new primary BTDF facility will be extended to cover from about 200 nm to about 2000 nm by using two broadband irradiation sources, an LDLS and an SC. Fiber optics will be used to achieve collimated irradiation beam with different sizes. Correspondingly, different detectors will be integrated into the detection system to cope with the broad spectral range. Additionally, a camera-based detection system will also be used to obtain spatially resolved BTDF results.

The uncertainty analysis revealed places in which most of the improvement effort should be invested. To reduce the uncertainty regarding laser power drift, a monitoring detector is needed to measure a fraction of the irradiation signal simultaneously to the detection system. Calibration of the attenuation filters will bring the contribution from this most dominant uncertainty source down to about 0.2 %. In the far future, light-proof compartments will enclose the facility to reduce the influence of straylight.

Further investigation will be made to relate the free parameters in the found empirical model for BTDF characterization of translucent materials to scattering quantities and physical properties of the material. To do so, a broader selection of samples with different physical thicknesses, scattering center concentrations, and scattering particle sizes will be characterized with different sizes of irradiation and measurement areas. The obtained results will be fed into different scattering models to extract scattering parameters to validate whether the model is able to accurately predict the complete scattering behavior of a specific material from existing measurement data.

The ultimate goal of developing this new facility is to be able to provide primary calibration service for the measurement of BTDF. For that, this facility needs to participate in a future interlaboratory comparison organized by the Consultative

Committee for Photometry and Radiometry (CCPR) so that new CMC-entries for BTDF measurement can be added to the BIPM database. Up to the time of writing this thesis, the CMC-entry for BTDF still remains absent in the BIPM database.

This facility would also be able to perform BRDF measurement to a limited extent. Since it is possible to flexibly switch between measurement schemes of under- or over-irradiation, this facility will complement the measurement capability of the *Gonioreflektometer* at PTB. Moreover, the contribution of this new BTDF primary facility can be extended to future research project in fields related to e.g. horticultural material, medical phantoms, and so on.

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List of Acronyms

SiO₂	silica
Aalto	Aalto University
Ar	argon
ARGon³	3D appearance robot-based gonireflectometer
ASTM	American Society for Testing and Materials
BBSS	bidirectional bipositional subsurface scattering function
BIPM	<i>Bureau International des Poids et Mesures</i>
BIS	bidirectional insurface scattering function
BRDF	bidirectional reflectance distribution function
BSDF	bidirectional scattering distribution function
BSSRDF	bidirectional scattering - surface reflectance distribution function
BTDF	bidirectional transmittance distribution function
CCD	charge-coupled device
CCPR	Consultative Committee for Photometry and Radiometry
CIE	<i>Commission Internationale de l'Éclairage</i>
CMC	Calibration and Measurement Capability
CMI	<i>Český Metrologický Institut</i>
CMOS	complementary metal-oxide-semiconductor
CNAM	<i>Conservatoire National des Arts et Métiers</i>
ConDOR	Conoscopic Device for Optical Reflectometry
CSIC	<i>Consejo Superior de Investigaciones Científicas</i>
CW	continuous-wave
DI	designated institute
DIN	Deutsches Institut für Normung
DoE	degree of freedom
DTU	<i>Danmarks Tekniske Universitet</i>
EMPIR	European Metrology Programme for Innovation and Research
FOV	field of view
FWHM	full width at half maximum
GEFE	<i>Gonio-EspectroFotómetro Español</i>
GSFC	Goddard Space Flight Center
GUM	Guide to the Expression of Uncertainty in Measurement
Hg	mercury
ILV	International Lighting Vocabulary
KUL	<i>Katholieke Universiteit te Leuven</i>

LDLS	laser-driven light source
LED	light-emitting diode
MSL	Measurement Standards Laboratory of New Zealand
NaNoRef	<i>das nationale Normal für gerichtete Reflexion</i>
NaNoTrans	<i>das nationale Normal für gerichtete Transmission</i>
NASA	National Aeronautics and Space Administration
ND	neutral density
Ne	neon
NIR	near-infrared spectrum range
NIST	National Institute of Standards and Technology
NMI	national metrology institute
NPL	National Physical Laboratory
NRC	National Research Council Canada
NRR	National Reference Reflectometer
PRD	perfectly reflecting diffuser
PTB	<i>Physikalisch-Technische Bundesanstalt</i>
PTFE	polytetrafluorethylene
ROSI	Robotic Optical Scattering Instrument
SC	supercontinuum
Si	silicon
SNR	signal-to-noise ratio
STARR	Spectral Tri-function Automated Reference Reflectometer
ULCA	ultrastable low-noise current amplifier
UV	ultraviolet spectrum range
VIS	visible spectrum range
Xe	xenon

List of Symbols

Symbol	Unit	Description
A_D	m^2	size of diaphragm area of the detection system
A_M	m^2	size of measurement area on sample back surface
A_{beam}	m^2	size of incident radiation beam area
A_i	m^2	size of irradiated area on sample front surface resulted from incident radiation
A_r	m^2	size of irradiated area on sample front surface resulted from reflected radiation
A_t	m^2	size of irradiated area on sample back surface resulted from transmitted radiation
A	m^2	size of an area
P_i	W	incident optical power collected by detection system
P_t	W	transmitted optical power collected by detection system
P	W	optical power collected by detection system
R_{resp}	V W ⁻¹ or A W ⁻¹	responsivity of detection system
S_{res}	V or A	resolution of signal reading
$S_{i,1}$	V or A	detector reading of incident optical signal in forward measurement
$S_{i,2}$	V or A	detector reading of incident optical signal in backward measurement
$S_{i,\text{dark},1}$	V or A	detector reading of dark incident signal in forward measurement
$S_{i,\text{dark},2}$	V or A	detector reading of dark incident signal in backward measurement
$S_{i,\text{dark}}$	V or A	detector reading of dark incident signal
S_i	V or A	detector reading of incident optical signal
$S_{t,1}$	V or A	detector reading of transmitted optical signal in 1 st measurement geometry
$S_{t,2}$	V or A	detector reading of transmitted optical signal in 2 nd measurement geometry
$S_{t,\text{dark},1}$	V or A	detector reading of dark transmitted signal in 1 st measurement geometry
$S_{t,\text{dark},2}$	V or A	detector reading of dark transmitted signal in 2 nd measurement geometry

List of Symbols

Symbol	Unit	Description
$S_{t,\text{dark},n}$	V or A	detector reading of dark transmitted signal in n^{th} measurement geometry
$S_{t,\text{dark}}$	V or A	detector reading of dark transmitted signal
$S_{t,n}$	V or A	detector reading of transmitted optical signal in n^{th} measurement geometry
S_t	V or A	detector reading of transmitted optical signal
S	V or A	detector reading of measured signal
d_D	m	diameter of detector diaphragm
d_M	m	diameter of measurement area on sample back surface
d_i	m	diameter of irradiated area on sample front surface resulted from incident radiation
$\Delta\lambda$	m	spectral bandwidth
Δx	m	slit width
Q	J	radiant energy
f_{is}	sr^{-1}	BIS
f_r	sr^{-1}	BRDF
f_{ssr}	$\text{m}^{-2} \text{sr}^{-1}$	BSSRDF
f_{ss}	$\text{m}^{-2} \text{sr}^{-1}$	BBSS
f_t	sr^{-1}	BTDF
Φ_i	W	radiant flux of incident radiation
Φ_t	W	radiant flux of transmitted radiation
Φ	W	radiant flux
I	W sr^{-1}	radiant intensity
E_i	W m^{-2}	irradiance of incident radiation
E	W m^{-2}	irradiance
C_T		correction factor for temperature
C_{azi}		correction factor for misalignment of sample azimuthal orientation
C_{digi}		correction factor for digitization error of measuring device
C_{instr}		correction factor for instrument function
C_{interR}		correction factor for interreflection
$C_{\text{irr.uni}}$		correction factor for spatial uniformity of irradiation beam
C_{nl}		correction factor for detector nonlinearity
C_{pol}		correction factor for polarization
$C_{\text{sam.uni}}$		correction factor for spatial uniformity of sample under test

Symbol	Unit	Description
C_{sharp}		correction factor for sharpness of imaging detection system
C_{stray}		correction factor for straylight
C_{switch}		correction factor for range switching of measuring device
$C_{\text{wl-corr}}$		correction factor for wavelength correction
C_{λ}		correction factor for irradiation wavelength
k		coverage factor for expanded uncertainty
λ	m	wavelength of radiation
l	m	distance from sample back surface to detector diaphragm
ω_i	sr	solid angle of incident radiation
ω_t	sr	solid angle of transmitted radiation
Ω_i	sr	projected solid angle of incident radiation on sample front surface
Ω_t	sr	projected solid angle of transmitted radiation on sample back surface
Ω	sr	projected solid angle
ω	sr	solid angle
φ_i	°	azimuthal angle of incident radiation
φ_r	°	azimuthal angle of reflected radiation
φ_t	°	azimuthal angle of transmitted radiation
φ	°	azimuthal angle
L_{is}	$\text{W m}^{-2} \text{sr}^{-1}$	radiance from insurface scattering
L_i	$\text{W m}^{-2} \text{sr}^{-1}$	radiance of incident radiation
L_r	$\text{W m}^{-2} \text{sr}^{-1}$	radiance of reflected radiation from a surface
L_{ssr}	$\text{W m}^{-2} \text{sr}^{-1}$	radiance of reflected radiation from an infinitesimal surface element
L_{ss}	$\text{W m}^{-2} \text{sr}^{-1}$	radiance from subsurface scattering
L_t	$\text{W m}^{-2} \text{sr}^{-1}$	radiance of transmitted radiation
L	$\text{W m}^{-2} \text{sr}^{-1}$	radiance
ζ		range of measuring device
σ_{drift}	W	standard deviation of laser output power drift
σ		standard deviation of the measurement of a physical quantity
τ_i		transmittance of ND-filter applied to measured incident signal
τ_t		transmittance of ND-filter applied to measured transmitted signal

List of Symbols

Symbol	Unit	Description
τ		transmittance of ND-filter
θ_i	$^\circ$	polar angle of incident radiation
θ_r	$^\circ$	polar angle of reflected radiation
θ_t	$^\circ$	polar angle of transmitted radiation
θ	$^\circ$	polar angle
t	s	time
$u_{\text{azi, engr.}}$	$^\circ$	standard uncertainty of azimuthal orientation determination for the engineered diffuser
u_{digi}		standard uncertainty of digitization error
u_{drift}	W	standard uncertainty of laser output power drift
$u_{\text{res, i}}$	V A ⁻¹	standard uncertainty of signal reading resolution for incident signal
$u_{\text{res, t}}$	V A ⁻¹	standard uncertainty of signal reading resolution for transmitted signal
u_{res}		standard uncertainty of digitization error
u_τ		standard uncertainty of transmittance of ND-filters
Δt	s	unit time
u		standard uncertainty of a physical quantity

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