New Realization of Spectral Diffuse Reflectance Standard at NMIJ

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ABSTRACT

Recently, the national standard of spectral diffuse reflectance in the visible region has been successfully realized at NMIJ using a new integrating sphere-based technique (modified Sharp-Little method) for an absolute reflectance measurement. An NMIJ integrating sphere using Spectralon^{TM}, optimized for reflectance measurements, has been employed for this method. The relative expanded uncertainty (k = 2) of the standard is 0.22 to 0.34 %, depending on the wavelength. In this paper, the detailed methodology and instrumentation of the modified Sharp-Little method with NMIJ integrating sphere is presented together with the outline of experimental results including uncertainty estimation and some calibration results.

1. INTRODUCTION

Reflectance is one of the fundamental optical quantities inherent to materials and being utilized for various purposes in many fields. In particular, spectral diffuse reflectance in the visible region is closely related to reflectance colorimetry. One of the significant requirements in recent colorimetry is quality control in measurement. With the rapid progress of technology, the range of application in colorimetry has been expanding and more accurate colorimetry has been being required worldwide. Strict quality control of color measurement devices according to an appropriate quality system such as ISO-9000s or ISO-17025 is indispensable to meet such requirements. The quality systems usually require reliable traceability of calibration and measurement, which definitely means fundamental measurement quantities should have the calibration system directly traceable to national standards. In addition, global-MRA (Mutual Recognition Arrangement) was signed up in 1999 for aiming at wider agreements in international trades and other affairs related to calibration and measurement. The degree of equivalence among national standards has been becoming more important to ensure the mutual recognition of calibration certificates issued by each National Metrology Institute. For these reasons, there have been strong demands for the calibration systems of spectral diffuse reflectance directly traceable to the national standard in Japan. Nevertheless, there were neither national standards nor calibration services for spectral diffuse reflectance in Japan.

In response to the industrial demand in Japan, National Metrology Institute of Japan (NMIJ) has been undertaking a new research project about accurate spectral diffuse reflectance measurements. After the five years research, the national standard of spectral diffuse reflectance in the visible region, based on absolute reflectance measurement, has been successfully realized at NMIJ. In this paper, new integrating sphere-based technique and instrumentation for realizing the national standard are described in detail. This paper also introduces the outline of experimental results including uncertainty estimation and some calibration results performed at NMIJ.

2. ABSOLUTE REFLECTANCE MEASUREMENT

The method for absolute diffuse reflectance measurement is roughly classified into two types. One is the method using an integrating sphere, and the other is the method using goniospectrophotometric technique. An integrating sphere was developed at the end of 19th century for the necessity of photometry, and it has been widely used for diffuse reflectance measurement afterwards. Integrating spheres have been widely used for the absolute diffuse reflectance measurement because of its simplicity in measurement as compared with the goniospectrophotometry. In the goniospectrophotometry, it usually takes extremely long time to detect all the reflected flux from a sample to be measured in each spatial position and spatially integrate it, which would often cause serious problem due to a long-term drift of a light source and ageing of the sample.

Up to now, various methods using the integrating sphere for the measurement of absolute diffuse reflectance have been proposed.[1] The basic concept of the new absolute diffuse reflectance measurement performed at NMIJ originates in the Sharp-Little method, which is one of the well-known integrating sphere-based methods proposed in 1920 by Sharp and Little,[2] and later improved by Budde.[3] In the original Sharp-Little method, an external light source irradiates a part of the integrating sphere wall. Then the radiances of a sample put onto the sphere wall and of a different part of the sphere wall are measured by switching the position of a detector. A baffle is fixed inside the sphere to prevent the first reflection component from reaching the sample. Assuming the whole integrating sphere system behaves ideally, the ratio of the two measured radiances corresponds to the radiance factor of the sample in d/0 geometry. This method has been used in many institutes as absolute method for spectral diffuse reflectance measurements. But this original method needs a lot of precondition to calibrate the sample correctly, which is usually difficult to satisfy. Considering the problem of the original method in detail, NMIJ developed the new absolute measurement technique with high accuracy, modified Sharp-Little method, by making some theoretical, experimental and instrumental modifications in the original method.

The features of the modified Sharp-Little method are; 1) movable baffle assembly, 2) geometric correction and 3) removable port plugs. Fig.1 shows the schematic diagram of the modified Sharp-Little method. At first, monochromatic radiant flux $\Phi_0(\lambda)$ from an external light source irradiates a part of the integrating sphere wall *B*, same as the original method. A position of the baffle assembly *X* is switchable inside the sphere, and the radiances ($L_1(\lambda)$ and $L_2(\lambda)$) of another part of the sphere wall *C* are measured in two different geometries with respect to baffle positions. One of the geometries (in-baffle condition), the baffle prevents the radiant flux due to the first reflection from reaching the target *C*, and only the radiant flux due to multiple reflections irradiates there. In the other geometries (off-baffle condition), the radiant flux due to both the first and the multiple reflections irradiates the target *C*. As can be seen easily, the main difference from the original method is the position where radiance is measured in two different geometries; the measurement target is fixed in our method, whereas two positions on the sphere wall are measured in the original method. Fixing the target during the radiance measurement is quite effective to eliminate the error due to reflectance non-uniformity of the sphere wall and reproducibility of the detector position. Then, making some corrections for the imperfection of the integrating sphere, the ratio of the two measured radiances $L_1(\lambda)/L_2(\lambda)$ corresponds to the average reflectance of the entire sphere wall in d/d geometry. After that, some corrections regarding the measurement geometry are made and the average reflectance in d/0 or 0/d geometry is determined. In addition, the integrating sphere used for this method has removable port plugs with the same finish as the rest part of the sphere wall. Considering the reflectance distribution over the sphere wall and making some corrections, spectral diffuse reflectance of each port plug can be determined from the average reflectance. These port plugs are used as the reference standards for other calibration using spectrophotometers in NMIJ.

Another feature in our new approach for realizing the national standard, which is less often

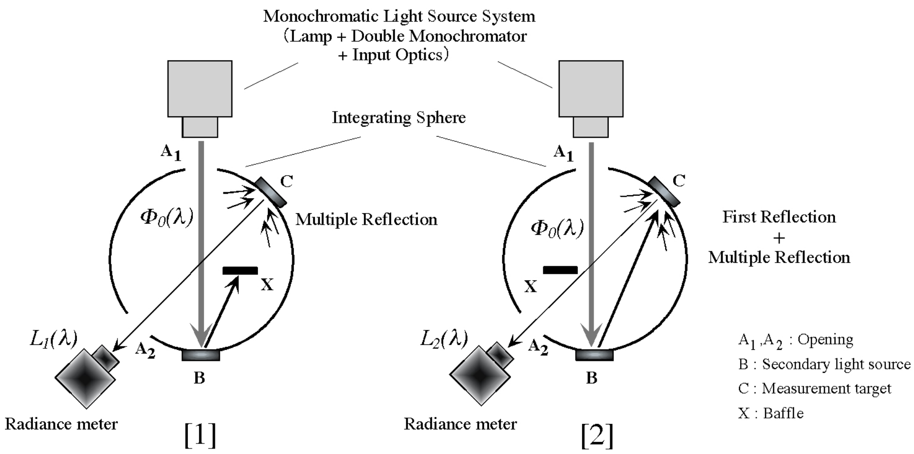

Figure 1: Schematic Diagram of the modified Sharp-Little method. The geometry [1] shows the in-baffle condition, and the geometry [2] shows the off-baffle condition.

noticed but surely more significant, is the employment of an integrating sphere with uniform reflectance. In the most integrating sphere-based method for absolute reflectance measurements, the sphere wall has been assumed to have approximately identical reflectance on the whole. But a series of our study revealed that most integrating sphere had relatively large non-uniformity. For example, integrating spheres with $BaSO_4$ coating usually have more than 1.5% reflectance non-uniformity on the average in the visible region. Several studies reported that reflectance non-uniformity would cause the disagreement of calibration results among the different geometries. Furthermore, it should be noted that what we can obtain in the absolute measurement using integrating sphere-based methods is averaged reflectance of the sphere wall. So it is obvious that such reflectance non-uniformity would directly and greatly affect the uncertainty in the integrating sphere-based measurements under the conventional assumption. Considering these problems, prior to this study, the new integrating sphere optimized for diffuse reflectance measurements (NMIJ integrating sphere) was developed with emphasis on the reflectance uniformity. Spectralon^{TM} is used as the material for the sphere wall. Spectralon^{TM} is the trade name for the PTFE-based resin and one of the most well known white diffusing materials for various excellent reflection properties.[4] The reflectance non-uniformity of the sphere wall in the visible region is about 0.16 % on the average. The uncertainty due to the non-uniformity is estimated to be almost the same level, which is the great improvement as compared with the measurement using a conventional integrating sphere with $BaSO_4$ coating.[5]

3. EXPERIMENTAL SETUP

The experimental set-up for the absolute reflectance measurement based on the modified Sharp-Little method consists of a monochromatic light source system, NMIJ integrating sphere and radiance meter. A double grating monochromator (f = 250 mm) is employed as the main part of the monochromatic light source system to reduce stray-light. The monochromatic light source system is also equipped with an illuminator, order-sorting filters, shutter and a collimating optics. The illuminator has two sources, a 300W quartz-tungsten-halogen lamp and a 300W xenon arc lamp, and they are switched over according to the wavelength range. The radiant flux exiting from is made almost collimated with about 10 mm diameters. The bandwidth of the incident radiation was set to be about 1.5nm. The inside diameter of the NMIJ integrating sphere has 50.8 cm. It is equipped with six portholes and port plugs of 5.08 cm diameter each, one baffle and rod to sustain the baffle to optimize for the experimental set-up based on the modified Sharp-Little method shown in FIg.1. The detailed designing and specifications of the NMIJ integrating sphere are described in ref.5. The radiance meter used in this study consists of an achromatic lens with a focal length of 90 mm, aperture mirror, filter wheel, shutter, stray-light reducing baffles, viewing optics and a photo detector. A head-on type electrically cooled photomultiplier tube is used as the photo detector. The diameter of the target for radiance measurement is about 5.0 mm.

Measurements were performed in the wavelength range from 360 nm to 830 nm with 10 nm intervals. The measurement at each wavelength was repeated to 20 times and their average was used to calculate the absolute spectral diffuse reflectance.

4. RESULTS

Fig.2 shows the absolute spectral diffuse reflectance in 0/d geometry obtained in this study. In the modified Sharp-Little method, at first, the absolute spectral diffuse reflectance in d/d geometry can be obtained as an average reflectance over the sphere wall, which corresponds to the ratio of the radiance in the two different geometries with respect to the baffle position as shown in Fig.1. As corrections for the imperfection of the integrating sphere, absorption of radiation in the integrating sphere due to the baffle assembly and gaps between the sphere wall, loss of the radiation due to the openings and non-Lambertian property of the sphere wall were evaluated, respectively. In addition, to obtain the absolute spectral diffuse reflectance in 0/d and d/0 geometry using the reflectance in d/d geometry, radiance distribution inside the integrating sphere and angle dependence against the reflectance were also evaluated. The correction factor for the imperfection of the integrating sphere is 0.0062 to 0.0068, and that for geometric conversion is –0.0044 to –0.0054 in 0/d and –0.0035 to –0.0046 in d/0, respectively. All the correction factors are wavelength dependent. The absolute spectral diffuse reflectance shown in Fig.2 is relatively in accordance with the previously published reflectance data of Spectralon^{TM} and PTFE-related materials. The main components of uncertainty

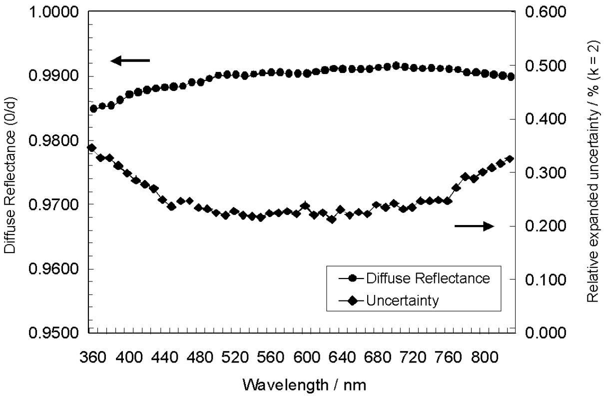

Figure 2: Absolute spectral diffuse reflectance in 0/d geometry as an average reflectance over the sphere wall obtained by the modified Sharp-Little method, and relative expanded uncertainty (k = 2) of the absolute measurement.

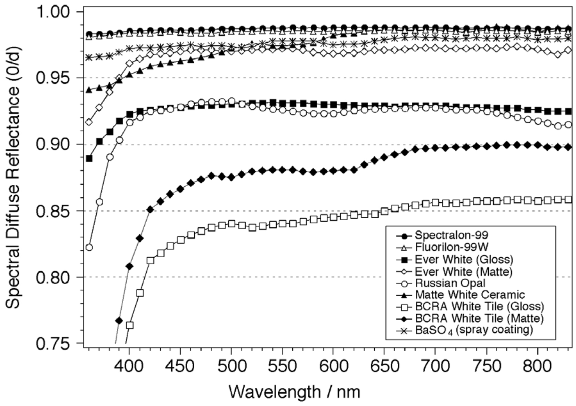

Figure 3: Example of calibration results for typical white materials in 0/d geometry.

associated with the modified Sharp-Little method are repeatability of the measurement, non-uniformity of the sphere wall, stray-light, wavelength accuracy, temperature, imperfection of correction factor and aging of the sphere wall. As shown in Fig.2, the relative expanded uncertainty (k = 2) of the absolute measurement is 0.22 to 0.34 %, depending on the wavelength.

5. CONCLUSIONS

The national standard of spectral diffuse reflectance in the visible region was successfully realized using the combination of the modified Sharp-Little method and the NMIJ integrating sphere. The modified Sharp-Little method was developed by making some theoretical, experimental and instrumental modifications in the original Sharp-Little method, which would improved the uncertainty mainly due to measurement geometries and imperfections of integrating spheres such as reflectance non-uniformity. The NMIJ integrating sphere showed the great help to perform the modified Sharp-Little method with ideal condition as much as possible. The combination of them has the great possibility for realizing the spectral diffuse reflectance standard with the lowest possible uncertainty.

In addition to the realization of the national standard, a reference spectrophotometer was also developed and started its operation.[6] NMIJ has just completed the international comparison (CCPR-K5) and started to provide new calibration services for the spectral diffuse reflectance in the visible region directly traceable to the national standard. The calibration will be carried out for white diffusing materials in the visible region (from 360 nm to 830 nm) with 10 nm intervals as shown in Fig.3. Two geometries (0/d and d/0) are available in the calibration. The estimated total uncertainty of the calibration is 0.35 % (k = 2) on the average in the visible region. Further research works to establish more accurate absolute reflectance measurement and to extend wavelength range for the national standard to UV and NIR region are now on progress.

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