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Comparison of different measurement methods for transmittance haze

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Abstract

Transmittance haze is increasingly important to the LCD and solar cell industry. Most commercial haze measurement instruments are designed according to the method recommended in the documentary standards like ASTM D 1003 (ASTM 2003 *Standard Test Method for Haze and Luminous Transmittance of Transparent Plastics*), JIS K 7361 (JIS 1997 *Plastics—Determination of the Total Luminous Transmittance of Transparent Materials—Part 1: Single Beam Instrument*) and ISO 14782 (ISO 1997 *Plastics—Determination of Haze of Transparent Materials*). To improve the measurement accuracy of the current standards, a new apparatus was designed by the Center for Measurement Standards (Yu *et al* 2006 *Meas. Sci. Technol.* **17** N29–36). Besides the methods mentioned above, a double-beam method is used in the design of some instruments. There are discrepancies between the various methods. But no matter which method is used, a white standard is always needed. This paper compares the measurement results from different methods, presents the effect of the white standard, and analyses the measurement uncertainty.

1. Introduction

The definition of transmittance haze is the ratio of the diffuse transmittance (DT) to the total transmittance (TT). Transmittance haze is usually applied to characterize the light scattering caused by the particles in transparent or translucent materials such as plastic, glass and liquids. Although haze is often used to describe the transparency of plastic and glass, high accuracy of haze measurement is not necessary for their associated traditional industries. The importance of haze measurement has increased recently due to the development of liquid crystal displays and optical thin films such as thin film solar cells. The intense competition in these developing industries makes quality control for materials more and more important. Haze and luminous transmittance data are especially useful for quality control as they can be used to identify internal defects of the material. Therefore, much attention has been paid to haze measurement accuracy and traceability in recent years in Taiwan.

Most national laboratories established their haze measurement system according to the method recommended by the earliest documentary standard ASTM D 1003 [1] in which an integrating sphere and a reference white standard are employed for the measurements. The drawback of ASTM D

1003 is its measurement precision since the error caused by the non-equivalence of the effective reflectance inside the sphere cannot be eliminated. To improve on this, a compensative port is added on the integrating sphere in the later documentary standards like JIS K 7361 [2] and ISO 14782 [3]. Many industrial haze meters in use today are designed not only according to the test method specified by ASTM D 1003, but also by JIS K 7361 and ISO 14782. From the theoretical analysis, it is clear that total transmittance (TT), diffuse transmittance (DT) and haze cannot all be measured precisely using only one of the above methods. In response to this, a new apparatus was designed by the Center for Measurement Standards (CMS) of Industrial Technology Research Institute (ITRI) in Taiwan [4]. Besides the methods mentioned above, the double-beam method is another choice which can also get more accurate results than ASTM D 1003, and its results are similar to those obtained by the CMS's apparatus. Since there are discrepancies between the measurement results of different methods, to offer the calibration services for all methods, CMS established a haze measurement system which can meet the requirements for all of the above methods by designing the integrating sphere and the path of the incident light beam. The experimental results show that no matter which method is used, the reflectance of the reference white standard has a significant

effect on the haze values, and this phenomenon matches the theoretical analysis in [4]. Therefore, attention must be paid to maintaining the reflectance of the white standard as close to that of the integrating sphere as possible. The comparison between different methods and the effect of the reflectance characteristic of the white standard on the haze measurement results are presented in the following sections. The structure and uncertainty analysis of the measurement system are also described.

2. Principle

Since the principle of each method is similar, let us use ASTM D 1003 in figure 1 as the example. In addition to the detector port (not shown in figure 1) there are two other ports on the sphere. The detector output signals are equal to I_1 to I_4 for procedures (1) to (4), respectively. I_1 is proportional to the total flux of the incident beam and I_2 is proportional to the total flux transmitted through the test specimen. The ratio of these two signals I_2/I_1 is equal to the TT of the test specimen. I_3 is proportional to the light scattered by the system. I_4 is proportional to the light scattered by the system plus the diffusion part of the beam transmitted through the test specimen. Therefore, the DT of the test specimen is equal to the diffusion light $I_4 - I_3 \times I_2/I_1$ divided by the total incident light I_1 or $(I_4 - I_3 \times I_2/I_1)/I_1$. The reason for multiplying the factor I_2/I_1 by I_3 is that the ratio of the light incident on the integrating sphere in procedure (4) and procedure (3) is I_2/I_1 . Finally, according to the definition of haze and dividing the DT by the TT, haze is obtained as $I_4/I_2 - I_3/I_1$. I_1 to I_4 depend not only on the flux of the incident light beam, system scattering and reflectance characteristic of the test specimen but also on the effective reflectance inside the sphere. Different positionings of components on the integrating sphere cause different effective reflectances inside the sphere. The effective reflectance can be expressed by the sphere multiplier M, where

$$M = \frac{\rho_{\rm o}}{1 - \rho_{\rm s} \left(1 - \sum_{i=0}^{n} f_i\right) - \sum_{i=0}^{n} \rho_i f_i}.$$

 ρ_0 is the initial reflectance for incident flux, which is equal to the reflectance of the white standard for procedures (1) and (2) or equal to the reflectance of the light trap for procedures (3) and (4) in figure 1; ρ_s is the reflectance of the sphere wall; ρ_i is the reflectance of port opening *i*; f_i is the port area of port opening *i* divided by the sphere area. From figure 1, it is obvious that no two procedures in D 1003 share the same structure. The components' positions on the sphere are different for each procedure. Therefore, measured deviation caused by the reflectance difference between (1) and (4) cannot be eliminated by dividing their signal mutually, and thus none of the accurate values for TT, DT or haze can be obtained. This shows that the precision of D 1003 is not good enough.

To improve on this, a compensative port is added in JIS K 7361 and in ISO 14782, as shown in figures 2 and 3.

JIS K 7361 has the same structure as ISO 13468 for the TT measurement. The compensative port gives JIS K 7361



Figure 1. The test method specified in ASTM D 1003. TT is equal to I_2/I_1 , DT is equal to $[I_4 - I_3(I_2/I_1)]/I_1$ and haze is equal to $I_4/I_2 - I_3/I_1$.



Figure 2. The test method specified in JIS K 7361. The TT is equal to I_2/I_1 and haze is equal to I_3/I_1 .



procedure (3), signal= I_3 procedure (4), signal= I_3

Figure 3. The test method specified in ISO 14782.

the same structure for procedures (1) and (2) and thus the accurate value of TT is yielded. But the precision of the degree of haze is still not good enough for JIS K 7361. The compensative port in ISO 14782 makes it possible to obtain the accurate value of haze by dividing I_4 by I_2 and I_3 by I_1 since the differences of structure between (1) and (3) as well as between (2) and (4) are eliminated even though its TT and DT both deviate from their accurate values. To obtain the accurate values of TT, DT and haze simultaneously, an additional port is added on the sphere of CMS's apparatus. As figure 4 shows, the sphere for each procedure now has an open port, a light



Figure 4. New apparatus designed by CMS for TT, DT and haze measurement.



Figure 5. Double-beam method.

trap, a reflectance standard and a test specimen. Therefore, whether for I_2/I_1 , I_3/I_1 , I_4/I_1 or I_4/I_2 , the difference between structure characteristics can always be cancelled out, allowing the accurate values of TT, DT and haze to be obtained.

Figure 5 depicts the measurement technique of the doublebeam method. In figure 5, signals $I_{x,ref}$ and $I_{x,probe}$ are obtained by blocking the probe beam and the reference beam, respectively, where x represents the signal for procedure (x). For the double-beam method, signal I_x for procedure (x) is replaced by $I_{x,probe}/I_{x,ref}$; other calculations for TT, DT and haze are the same as ASTM D 1003. The distinguishing feature of the double-beam method is using $I_{x,probe}/I_{x,ref}$ to eliminate the non-equivalence of the effective reflectance between each procedure since here I_x is independent of the effective reflectance. Therefore, accurate results can also probably be achieved by using the double-beam method.

In fact, accuracy depends not only on the measurement methods but also on the system design, especially the instrument scattering and the reflectance of the white standard. For the optimum design, the light scattered by the instrument is nil and the reflectance of the white standard is the same as the reflectance of the sphere. For most actual light trap designs the instrument scattering is approximately 0.1% and the reflectance of the white standard may become different from the reflectance of the sphere after a period of time or



Figure 6. System schematic diagram. (This figure is in colour only in the electronic version)

improper operation. According to the theoretical analysis in [4], the higher the reflectance of the white standard or the higher the instrument scattering, the lower the haze measurement result. In this paper only the effect of the white standard is discussed, since from the theoretical analysis the effect of the 0.1% instrument scattering on the haze value is not the dominant item of the uncertainty budget.

3. System design

Figure 6 shows the schematic diagram of the haze measurement system designed by CMS/ITRI.

The diameter of the integrating sphere is 15 cm. According to the measurement technique outlined in ASTM D 1003 and also in ISO 14782, for a 15 cm sphere the diameter of the entrance and exit ports on the integrating sphere is calculated to be 2.1 cm and the diameter of the incident beam is 1.4 cm. To make the system meet the geometric requirements of different methods, except for the detector port which is connected with the fibre and locates on the north pole of the sphere, there are four other ports on the equator of the sphere. Port I is the entrance port for ASTM D 1003, JIS K 7361, ISO 14782 and CMS apparatus, or for the probe beam (path I) of the double-beam method. Port II is the compensative port for ISO 14782, JIS K 7361 and CMS. Port III is the second compensative port for CMS apparatus, or the entrance port for the reference beam (path II) of the double-beam method. Port IV is the exit port. The integrating sphere is fixed on a stage which can adjust the tile angle to make the incident beam concentric within the entrance port and exit port when the beam is unobstructed by a specimen. The light beam is generated by a QTH lamp, optical components and an aperture and the beam is approximately circular and sharply defined. Using mirror 1 the light beam can be switched to path I or path II. The unnecessary ports are covered by white plates. For example, when applying the system for ASTM D 1003, port II and port III are unnecessary. The light signal is transmitted by a bundle of fibre to the CCD-type spectrometer to measure the spectrum of the light from 360 nm to 810 nm with a step of 5 nm. With the spectra, the TT, DT and haze can be calculated

Table 1. The haze values measured by different methods.

	Haze plate				
Method	H1	H5	H10	H20	H30
ASTM D 1003 JIS K 7361 ISO 14782 CMS Double-beam	1.00 1.10 1.10 1.07 1.09	4.87 4.98 5.25 5.26 5.29	8.18 8.32 8.84 8.83 8.87	17.63 17.82 19.02 19.03 19.14	28.34 28.50 30.70 30.61 30.73

 Table 2. The discrepancies of haze between ISO 14782 and other methods.

		Haze plate				
Method	H1	H5	H10	H20	H30	
ASTM D1003	-0.1	-0.38	-0.66	-1.39	-2.36	
JIS K 7361	0.0	-0.27	-0.52	-1.20	-2.20	
CMS	-0.03	0.01	-0.01	0.01	-0.09	
Double-beam	-0.01	0.04	0.04	0.12	0.03	

for different illuminants. Thereby, the mismatch between the light source and the CIE standard illuminants (D65, C...) and also the mismatch between the spectral responsivity of the photodetector and the spectral luminous efficiency function $V(\lambda)$ of the CIE (1987) can be eliminated. The light trap is made of two black mirrors and its ability to absorb is about 99.9%. Except the spectrometer, the whole system is covered by a black box to block the environmental lighting and reduce the measurement uncertainty.

4. Comparison of different methods

A total of 14 haze plates were used as test specimens; five of them were from BYK-Gardner (Germany), four from Nippon Denshoku (Japan) and another five from NIM (China). The measurements were done by ASTM D 1003, JIS K 7361, ISO 14782, CMS apparatus and double-beam methods, and used different white plates with different reflectance as the white standard. The experimental results show that every haze plate has a similar trend for different measurement methods and white standard. Therefore we use BYK plates, H1, H5, H10, H20 and H30 as the example to explain the results. The results of BYK plates for five different methods are shown in table 1. The haze values in table 1 are calculated under the illumination of C illuminant. As described in section 2, with a well designed, ISO 14782, CMS apparatus and doublebeam method a more accurate haze value can be obtained than ASTM D 1003 and JIS K 7361. Therefore, we use the values from ISO 14782 as the standard haze values and compare the values from the other methods with ISO 14782; the discrepancies are in table 2.

Table 3 lists the total transmittance measured by each method. As mentioned in section 2, theoretically JIS K 7361 (ISO 13468) can get accurate TT, therefore the values from JIS K 7361 are used as the standards and compared with other methods; the differences of TT are shown in table 4.

From tables 1 to 4, ASTM D 1003 has the lowest values of haze. That is because in D 1003 there is a different effective

Table 3.	The TT (%) measured by	different methods.
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	Haze plate				
Method	H1	Н5	H10	H20	H30
ASTM D1003 JIS K 7361 ISO 14782 CMS Double-beam	92.78 92.29 92.93 92.09 92.24	92.89 92.29 92.78 92.39 92.37	91.24 90.52 91.11 90.55 90.59	89.05 88.16 88.76 88.15 88.14	87.39 86.79 87.36 86.58 86.76

 Table 4. The discrepancies of TT (%) between ISO 13468 and other methods.

		Haze plate			
Method	H1	H5	H10	H20	H30
ASTM D1003	0.49	0.60	0.72	0.89	0.60
ISO 14782	0.64	0.50	0.59	0.60	0.57
CMS	-0.20	0.10	0.03	-0.01	-0.21
Double-beam	-0.05	0.08	0.07	-0.02	-0.03

reflectance for each procedure, making the TT slightly higher, but the low DT leads to a low haze value. JIS K 7361 uses a compensative port to correct the TT. But the effect caused by the non-equivalence of the effective reflectance still cannot be eliminated for haze measurement, thus a lower haze value is obtained. However, the results of JIS K 7361 are better than those of ASTM D 1003. ISO 14782, CMS and double-beam methods have similar haze values. Just as in the theoretical analysis as described in section 2 and [4], for good design that is, reducing the instrument scattering and making the reflectance of the reflectance standard as close to the reflectance of the sphere wall as possible-ISO 14782, CMS and doublebeam methods can get the measurement results close to the theoretical values of haze. Nevertheless, the non-equivalence of the effective reflectance in procedures 1 and 2 of ISO 14782 cannot be eliminated, and this gives ISO 14782 a higher error in the TT than CMS and the double-beam method. Therefore only the methods of CMS and double-beam can get the accurate values of haze, TT and DT simultaneously.

5. Uncertainty analysis

The error sources of the haze measurement system include the reflectance of the white standard, stability of the light source, diameter of the incident beam, system scattering, covering of extra port with different white plate, linearity, wavelength accuracy and measurement repeatability. Since the incident beam is nearly collimated and the incident angle is close to zero, the polarization effect is neglected here. To understand the effect of the reflectance characteristic of the white standard on the haze measurement results, a task was performed by using five white plates with different reflectances, 98.61%, 96.65%, 93.65%, 89.70% and 88.21%, as the white standard. The one with the reflectance 93.65% had a similar characteristic to the integrating sphere interior wall. Figure 7 shows the experimental results of BYK H30 under different measurement methods and white standards. Figure 7 shows that the value of haze increases approximately linearly



Figure 7. The change in the BYK H30 haze value versus the change in the white standard reflectance.

Table 5. Uncertainty budget of H30 under the ASTM D 1003method.

Source	1σ uncertainty/%
Reflectance of the white standard (within $\pm 2\%$ deviation from the reflectance inside the sphere)	0.4/√3
Stability of the light source	0.0144
Diameter of incident beam (12 mm to 15 mm)	0.0115
System scattering (0.1% to 0.4%)	0.0289
Extra port covered by different	
white plate (85% to 96%)	0.08
Linearity	0.0465
Wavelength accuracy (0.1 nm)	0.004
Measurement repeatability	0.04
Combined uncertainty Expanded uncertainty	0.25 0.5 (k = 2)

with the decrease in the reflectance of the white standard no matter which method is used. The slope of the curves is about -0.2, meaning that the deviation of 1% of the reflectance of the white standard will induce 0.2% error of the haze value in this case. Therefore, to get an accurate measurement result, it is important that the white standard and the integrating sphere have the same reflectance characteristic.

The uncertainty budget of the haze measurement system for H30 under the ASTM D 1003 method is listed in table 5.

To estimate the uncertainty from beam size, we adjusted the aperture to change the diameter of the incident beam from 12 mm to 15 mm and found that the standard uncertainty was within 0.0115% for the five test specimens. To evaluate the uncertainty from system scattering, we used the formula in [4] and changed the y value in the formula from 0.1% to 0.4%.

Table 6. Uncertainty for different haze range (k = 2).

H1	H5	H10	H20	H30
0.1	0.2	0.2	0.4	0.5

The error caused by 0.1% to 0.4% system scattering is 0.0289% for the test specimen H30 under the ASTM D 1003 method. From table 5, the key quantity is the reflectance of the white standard.

Using the uncertainty analysis method as described above, the uncertainties for different methods and different test specimens can be obtained. The final results are summarized in table 6.

6. Conclusions

This paper compares the methods of ASTM D 1003, JIS K 7361 and ISO 14782, a new apparatus developed by CMS and the double-beam method. The experimental results show that the lowest haze values are from ASTM D1003 and then from JIS K 7361. The haze values of ISO 14782, the CMS method and the double-beam method are similar but only the CMS and double-beam method can obtain accurate values of TT, DT and haze simultaneously.

The effects of the white standard and the uncertainty analysis are also discussed in the paper. No matter by which method haze measurement was performed, higher white standard reflectance results in a lower haze value. The error caused by the improper white standard reflectance is significant and is the crucial parameter of the uncertainty budget. Therefore, a good measurement result depends on more than just a good system design; attention must also be paid to maintaining the reflectance of the white standard as close to that of the integrating sphere as possible.

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