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TECHNICAL REPORT

INTERCOMPARISON ON MEASUREMENT OF (TOTAL) SPECTRAL RADIANCE FACTOR OF LUMINESCENT SPECIMENTS

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Standardization of colour measurement
Luminescence colours

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SUMMARY

This Technical Report describes an intercomparison of measurements of the (total) spectral radiance factor (SRF) of luminescent materials. Seven luminescent specimens were provided as painted panels: two were white containing FWAs and five were chromatic (green, yellow, two orange, red). The SRFs were to be measured with direct specimen illumination by a source simulating CIE D₆₅. The SRFs that would have been obtained under D₆₅ illumination were to be calculated by a method from among those previously studied by the committee. Data sets from four laboratories (one supplying two sets) gave useful results. The mean CIELAB color difference from the mean of the five data sets was 2.6 units averaged over all data sets and specimens, with a range among specimens from 1.9 to 3.4 units and a range among data sets from 1.5 to 4.2 units. The mean color difference from the results of an arbitrary reference laboratory was 3.6 units averaged over all data sets and specimens, with a range among specimens from 3.0 to 4.2 units and a range among data sets from 2.1 to 5.3 units. Maximum values of the SRFs varied considerably among the data sets, over ranges varying from 11.4% to 33.6% among the samples. It is the committee's feeling that these results are representative of the present state of the art for this measurement, despite the relatively poor performance seen.

RÉSUMÉ

Ce rapport technique décrit une comparaison de mesures du facteur de luminance énergétique spectral (total) (SRF) pour des matériaux luminescents. Sept échantillons luminescents furent fournis sous forme de plaquettes peintes: deux étaient blanches contenant un agent fluorescent d'azurage et cinq étaient colorées (une verte, une jaune, deux oranges et une rouge). Le SRF devait être mesuré avec un éclairage direct des échantillons par une source réalisant l'illuminant D_{65} de la CIE. Le SRF qui aurait été obtenu sous éclairage par D_{65} devait être calculé par une méthode choisie parmi celles étudiées précédemment par le comité. Les résultats de quatre laboratoires (un des laboratoires a fourni deux séries de résultats) fournissent des informations utiles. La différence de couleur CIELAB moyenne, calculée sur l'ensemble des résultats des laboratoires et des échantillons, par rapport aux moyens des cinq groupes de résultats était de 2,6 unités, avec un écart pour les échantillons compris entre 1,9 et 3,4 unités et un écart pour les résultats des laboratoires compris entre 1,5 et 4,2 unités. La différence de couleur moyenne, calculée sur l'ensemble des résultats des laboratoires et des échantillons, par rapport aux résultats d'un des laboratoires choisi arbitrairement comme référence était de 3,6 unités avec un écart pour les échantillons compris entre 3,0 et 4,2 unités et un écart pour les résultats des laboratoires compris entre 2,1 et 5,3 unités. Les valeurs maximales du SRF varient considérablement d'un laboratoire à l'autre avec des écarts variant de 11,4 % à 33,6 % suivant les échantillons. De l'avis du comité, ces résultats sont représentatifs de la situation actuelle pour ce type de mesures, en dépit des résultats relativement imparfaits obtenus.

ZUSAMMENFASSUNG

Dieser Technische Bericht enthält Ergebnisse einer Vergleichsmessung des (Totalen) spektralen Strahldichtefaktors (SRF) an lumineszierenden Materialien. Es wurden sieben lumineszierenden Proben in Form beschichteter Tafeln bereitgestellt: zwei Proben waren weiß und enthielten fluoreszierenden Aufheller, fünf Proben waren farbig (grün, gelb, 2 x orange, rot). Die SRFs sollten bei direkter Probenbestrahlung unter Verwendung einer Strahlungsquelle gemessen werden, die die Verteilung nach CIE D₆₅ simuliert. Die für D₆₅-Bestrahlung gültigen SRFs sollten nach einer der von diesem Komitee zuvor untersuchten Methoden berechnet werden. Die von vier Laboratorien angegebenen Datenreihen (ein Laboratorium ist mit zwei Datenreihen vertreten) ergaben brauchbare Ergebnisse. Der mittlere CIELAB Farbabstand vom Mittelwert aus der Mittelung über alle fünf Datenreihen und Proben betrug 2,6 Einheiten. Hierbei lag der Streubereich der Proben zwischen 1,9 und 3,4 Einheiten und der Streubereich der Datenreihen zwischen 1,5 und 4,2 Einheiten. Der mittlere Farbabstand bezogen auf die Ergebnisse eines willkürlichen Referenzlaboratoriums betrug nach Mittelung über sämtliche Datenreihen und sämtlicher Proben 3,6 Einheiten mit einer Streubereich von 3,0 bis 4,2 Einheiten zwischen den Proben und einem Streubereich von 2,1 bis 5,3 Einheiten zwischen den Datenreihen. Die maximalen Werte der SRFs zwischen den Datenreihen variierten beträchtlich: zwischen 11,4 % und 33,6 % für die Proben. Trotz der relativ schlechten Ergebnisse vertraten die Mitglieder des Komitees die Meinung, daß diese Ergebnisse für den gegenwärtigen Stand dieser Art von Messungen bezeichnend sind.

1 INTRODUCTION

During the quadrennium 1975-1979, a Task Force on Colorimetric Aspects of Luminescence, chaired by Fred W. Billmeyer, Jr., within the Luminescence Subcommittee of CIE Technical Committee 2.3, Materials, evaluated methods for predicting the (total) spectral radiance factors (SRFs) of luminescent specimens under standard illuminants from measurements of the specimens made using sources simulating those illuminants. Emphasis was placed on methods using one-monochromator, integrating-sphere spectrophotometers because of their widespread availability. Accuracy of the prediction was found to depend primarily upon the closeness of match between the simulation source and the standard illuminant and on the accuracy with which the spectral power distribution irradiating the specimen was known. Choice among several calculation methods was of secondary importance. The results were drafted as a CIE Technical Report, but by decision of the CIE Action Committee, this report was not issued. Instead, the results were reported in the literature [1].

Between 1979 and 1983, the same group of participants, operating as the Subcommittee on Luminescence of TC 2.3, conducted an international intercomparison on the measurement of the SRFs of luminescent specimens. Six laboratories in three countries participated, but the results of two laboratories were so far away from the mean of the others that they were not included in the final results. The measurements were completed in 1981 (except

for remeasurement in one laboratory in 1983) but the calculations required were not completed until after the 1983 CIE Session. Thus, with the revision of the structure of the CIE, it was appropriate to form Technical Committee 2-08 under the same chairmanship to complete the calculations and prepare the present Technical Report.

2 HISTORICAL REVIEW

2.1 Nature of the Problem

There are two kinds of luminescent materials widely used in industry, differing only in the locations of their excitation and emission wavelength regions. The first, which is mainly considered in this report, is the chromatic luminescent materials used for safety and signal applications. These materials are generally excited by absorption of radiation in the ultraviolet and short-wavelength visible regions, and emit in the longer-wavelength visible region. The second type, containing fluorescent whitening agents (FWAs), absorbs in the ultraviolet and emits in the short-wavelength visible region; otherwise the two types are very similar.

The most widely used type of instrumentation for measuring specimens of these luminescent materials is the one-monochromator spectrophotometer, with the monochromator in the viewing beam. The large majority of these instruments in the field have integrating-sphere geometry. It is this type of instrument that has been studied most intensively by the committee.

It might be hoped that with the present state of the art, measurements with various of these instruments would lead to nearly identical results, but a study [2] showed that this is far from the case. For measurements of a single specimen with seven different instruments, all said by their manufacturers to utilize simulators of CIE standard illuminant D₆₅, values of the maximum SRF covered a range of

130 to 260 units (relative to the perfect reflecting diffuser at 100 units). Values of luminescent emission were estimated to cover a 4-to-1 range.

Since it is well known that the SRF of a luminescent material depends on the irradiance on the specimen, and this in turn depends on the nature of the instrument source, it is clear that the differences reported resulted from differences in the the spectral power distributions of their D_{45} simulators. These varied among the instruments, including continuous-operation xenon lamps, filtered-xenon flashtubes, mosaic-filtered incandescent lamps intended to simulate D_{45} in both the ultraviolet and visible regions, and filtered incandescent lamps intended to simulate D_{45} in the visible region, but deficient in ultraviolet power in comparison.

This state of affairs stimulated the committee's study of methods for calculating the SRFs of luminescent materials under standard illuminants from their measurements under instrument sources. Before reviewing this study, we consider instruments further.

2.2 Instrument Considerations

2.2.1 One-Monochromator Instruments

Use of a one-monochromator instrument with the monochromator in the viewing beam is the most direct way to simulate the appearance of a luminescent specimen. The eye sees the total radiance from the specimen, the sum of the reflected and fluoresced radiances. To measure the corresponding quantity, the (total) radiance factor, it is

only necessary that the irradiance on the specimen be the same as that used in the visual observation, or that specified in the method to be followed, usually D_{45} . This irradiance is determined by the instrument source, the filtering that makes the source a D_{45} simulator, and any other instrument components, such as an integrating sphere, that comprise the irradiating system. In the usual double-beam instrument, the radiance from the specimen is compared to that from a fixed reference, and by means of suitable calibration, the instrument measures the radiance factor. The monochromator in the viewing beam allows wavelength-by-wavelength analysis to provide the (total) spectral radiance factor.

If, as in some older instruments, the monochromator is in the illuminating beam, the specimen is irradiated by monochromatic light. This does not correspond to the usual viewing situation, but provides a useful quantity called here the conventional reflectometer value [3]. The quantity depends upon the spectral responsivity of the instrument measuring system.

It is well known [4,5] that the irradiance on the specimen is altered by the integrating sphere, if used, and that the resulting irradiance depends on the spectral nature of the specimen. This results from the dependence of the efficiency of the sphere on the sizes and reflectances of its wall and ports, including the specimen port [6,7].

The use of bidirectional geometry (45/0 or the (usually) equivalent 0/45) eliminates this effect and

greatly simplifies the measurement of luminescent materials under precisely specified conditions. Only recently, however, have bidirectional spectrophotometers become widely available, and the vast majority of one-monochromator instruments in the field utilize integrating spheres.

2.2.2 Two-Monochromator Instruments

Reflectance spectrophotometers with two monochromators, one each in the illuminating and viewing beams, can provide detailed information on the excitation, emission, and reflectance spectra of luminescent materials. Such instruments can be considered modifications of the chemist's spectrofluorimeters, in which the geometry of illumination and viewing has been altered, usually to bidirectional. Research instruments of this type have been described [8-13], of varying degrees of sophistication, and their performance has been reviewed [14].

These instruments do not provide directly the SRF, but Donaldson [9], in his pioneering work, said that to obtain the fluorescent radiance factor for heterochromatic radiation, one simply adds up the emitted fluxes for each excitation wavelength, weighted according to the corrected excitation for the desired illuminant. This is true for a single fluorophor under normal conditions, but the question of what happens when there are two or more fluorophors and there is energy transfer between them, as may be the case for the chromatic luminescent materials considered in this report [15], has not been studied.

Despite this caveat, the two-monochromator method is usually considered the referee method for the determination of the pertinent luminescent quantities. The accuracy of the data determined with a one-monochromator spectrophotometer is likely to be poorer than that from a two-monochromator instrument, though the difference may be small.

2.3 Calculation of SRFs under Standard Illuminants

The calculation of the SRF of a luminescent specimen that would be obtained if it were irradiated by a standard source, from measurements made by the use of a simulator of that source, would go far to remove the variability among instruments, to insure correlation with visual observations, and to provide adequate tests of conformance to colorimetric specifications. It was for these reasons that the committee studied calculation methods between 1975 and 1979. The results of these studies are provided in the literature [1,2,16], and are summarized briefly here.

The basis for the calculation methods is the following.

1. The fluorescent and reflected radiance factors are first separated, by methods to be described.
2. The irradiance on the specimen is determined.
3. The fluorescent radiance factor is modified, again by methods to be described, to correspond to that which would have been obtained if the specimen were irradiated by the standard source.
4. The reflected radiance factor is added to the new fluorescent radiance factor.

2.3.1 Separation of Fluorescent and Reflected Radiance Factors

Four methods were studied [17] for carrying out this separation:

1. The "Two-Mode Method" [18] uses measurements of the (total) SRF at wavelengths shorter than the lower limit of emission, at which it is equal to the reflected radiance factor, and measurements of the conventional reflectometer value at wavelengths longer than the upper limit of excitation, at which where it is equal to the reflected radiance factor. But there is an "overlap" region that may be as wide as 40-120 nm between these two for chromatic luminescent paints, plastics, and textiles, in which both excitation and emission take place. In this region the measured quantities are larger than the true reflected radiance factor, often by substantial amounts. This method has larger errors than the alternatives, and is not recommended [17].

2. In the "Filter-Reduction" method [19], the SRF is measured with a source successively altered by a series of sharp short-wavelength cutoff filters. Within the overlap region, the amount of excitation is reduced but not eliminated, and the reflected radiance factor predicted is still too high, but the errors are not serious for relatively weak fluorescers.

3. In the "Fluorescence-Weakening" method [20,21], the specimen is irradiated successively by two different sources, for which the ratio of the irradiances is known.

With the aid of a single calibration measurement, the reflected radiance factor can be calculated. This method requires a rather large amount of luminescence for accuracy, but it is the method of choice for highly luminescent specimens in the absence of two-monochromator measurements.

4. The "Two-Monochromator" method is the most accurate for separating the reflected radiance factor from the total SRF.

2.3.2 Measurement of the Irradiance on the Specimen

Difficulties in measuring the spectral irradiance at the specimen plane are not trivial, and require the use of an accurate spectroradiometer, equipment not found in most industrial laboratories. If bidirectional geometry is used, the measurements are sensitive to small changes in geometry and to specimen surface irregularities. In any case, the accurate calibration of the spectroradiometer, especially at short wavelengths, is difficult.

If integrating-sphere geometry is used, the measurement problem is alleviated by the averaging properties of the sphere, but the measurement must be repeated for each specimen, or, alternatively, the spectral efficiency of the sphere may be calculated each time from the measured reflectances of the sphere wall and the ports [4,7].

2.3.3 Calculation of the Modified Fluorescent Radiance Factor

Several methods have been proposed and studied [1,2,16] for the desired calculation. Each utilizes the SRF $\beta_{T,1}(\lambda)$

of the specimen irradiated by source 1 of spectral irradiance $S_1(\lambda)$, the relative number N_1 of quanta absorbed and converted to luminescence under source 1, and the relative number N_2 of quanta similarly absorbed and converted by the specimen under source 2, with spectral irradiance $S_2(\lambda)$, to calculate $\beta_{T,2}(\lambda)$ by the following equation [19]:

$$\beta_{T,2}(\lambda) = \beta_s(\lambda) + \beta_{L,1}(\lambda) [S_1(\lambda) N_2 / S_2(\lambda) N_1]$$

where $\beta_s(\lambda)$ is the reflected radiance factor and $\beta_{L,1}(\lambda)$ is the fluorescent radiance factor under source 1. Values of N are calculated from the equation

$$N = \int_{\lambda'} X_{\lambda'}(\lambda) S_q(\lambda') d\lambda' = \int_{\lambda'} X_{\lambda'}(\lambda) S_e(\lambda') \lambda' d\lambda'$$

where X is the (corrected) excitation spectrum, adjusted to correspond to equal photon incidence at all excitation wavelengths. Here the subscript q is used to designate quantum units, and the subscript e to designate power units.

The various calculation methods differ only in the means of evaluating N by different approximations. They are the following:

1. Method of Eitle and Ganz [19], in which $N(\lambda)$ is calculated by

$$N = \int_{\lambda'} [1 - \beta_s(\lambda')] A(\lambda') S_q(\lambda') d\lambda'$$

where the term in brackets is the absorptance and $A(\lambda')$ is a factor determined by means of a Kubelka-Munk analysis that is not always applicable and requires several assumptions [22,23]. For these and other reasons this method was not

considered further in the committee's study.

2. Method using X from a two-monochromator instrument in place of the product of the absorptance and $A(\lambda')$. This information is likely to be the most accurate available, and its use should provide the best approximation to the true values of N .

3. Method of Alman and Billmeyer [22], in which X is calculated from measurements of the SRF and the conventional reflectometer value obtained by use of a one-monochromator instrument.

4. Method of Allen and Sanders [23,24], in which the absorptance is retained but $A(\lambda')$ is set equal to a constant over the excitation wavelength range.

5. Method of Grum and Costa [25], in which it is assumed that the product of the absorptance and $A(\lambda')$ is constant over the excitation wavelength range. This is the simplest of the approximate calculation methods.

Finally, after the modified fluorescent radiance factor is calculated by one of these methods, the desired SRF is obtained by adding to it the reflected radiance factor.

Much of the material in Section 2 up to this point was taken from ref. 26.

2.4 Other Developments

The following publications are of interest in relation to the work of the committee. The list is not implied to be complete.

Publication CIE No. 51 [27] provides a method for evaluating the suitability of a test source as a simulator

of CIE standard illuminant D_{65} , D_{45} , or D_{75} . For each of these three standard illuminants, SRF data are supplied for five pairs of nonfluorescent samples that are metameric matches. The colorimetric differences are computed for the test illuminant; the average of these differences is taken as the Visible Range Metamerism Index, and this is used as a measure of the quality of the test illuminant as a simulator for nonfluorescent samples. For fluorescent samples, the quality is further assessed in terms of an Ultraviolet Range Metamerism Index; this consists of the average of the colorimetric differences computed with the test illuminant for three further pairs of samples, each pair consisting of a fluorescent and a nonfluorescent sample that are a metameric match under the standard illuminant.

Chong and Billmeyer [16,28] studied by visual methods the accuracy of several methods of assessing the performance of daylight simulators, including that used in the above CIE report. Four methods based on colorimetric weighting gave significantly better correlations than one based on radiometric weighting, with little choice among them; the CIELAB color-difference equation was preferred to CIELUV; and the choice of 2° or 10° standard observer had no effect.

Grum [29] discussed fluorescent standards, describing the requirements on materials that should be met, the reasons why such standards are needed and by whom, and the optical materials considered by the CIE and the ISCC.

Clarke and McKinnon [30] described a proposed new interior daylight illuminant representing the type of

daylight most commonly used in the critical assessment of colors and that can be more easily simulated than D_{65} , and showed that it can be used validly for the measurement of fluorescent materials.

Billmeyer and Hepfinger [15] studied energy transfer between fluorophors in chromatic luminescent materials of the type used in the intercomparison described in this report, showing that such energy transfer does take place among the fluorophors, but not between them and titanium dioxide. A shift in wavelength of the emission peak with change in specimen thickness was attributed to reabsorption and reemission within the overlap region.

Minato, Nanjo, and Nayatani [31] described errors caused by instrument polarization in the two-monochromator measurement of luminescent specimens. In general these errors can be expected to be less than 1 CIELUV unit.

Chen and Billmeyer [32] described and tested an adjustment method for measuring luminescent specimens, originally proposed by Blaise [33]. Special instrument requirements and the complexity and length of the procedure are serious disadvantages.

Chen and Billmeyer [34] proposed a method for measuring luminescent specimens in an integrating-sphere spectrophotometer using a compensation filter designed to provide the desired spectral irradiance on the specimen. In a preliminary test they showed that even a very rough approximation to the desired filter gave encouraging results.

Minato, Nanjo, and Nayatani [14] presented an in-depth study of colorimetric measurement of luminescent materials and its accuracy by using the two-monochromator method.

Grum [35] wrote the chapter "Colorimetry of Fluorescent Materials" in Vol. 2, *Color Measurement*, of his series on *Optical Radiation Measurements*. Much of the material in the unissued CIE Technical Report prepared by the committee was quoted at length, but without attribution.

Mielenz [36] edited Vol. 3, *Measurement of Photo-luminescence*, in the series *Optical Radiation Measurements*. Much of the book concerns measurement of luminescent solutions rather than reflecting specimens.

In a paper in Japanese, Minato, Nanjo, and Nayatani studied the accuracy of "the CIE proposed method for colorimetry in fluorescent materials" [37], referring to the work of this committee. They derived methods for estimating the accuracy of the one-monochromator method relative to the two-monochromator method, for the case of 45/0 geometry, and showed that the one-monochromator method was capable of estimating total SRFs to within 1%, corresponding to less than 1 CIELUV unit. But in the overlap region, errors could be as high as 2-4%, and the error of the fluorescent radiant efficiency was also significant.

The same authors also published [38] (in Japanese) a study of relative spectral integrating-sphere efficiency for the measurement of fluorescent materials, comparing the quantity calculated by integrating-sphere theory to that determined by measurement of the irradiance at the specimen

port. Color differences were less than 1.6 CIELUV unit.

The American Society for Testing and Materials (ASTM) issued a *Standard Practice for Color Measurement of Fluorescent Specimens*, ASTM Designation E 991-84 [39]. This short document is based on the methods outlined in this report. It requires that the measurement be made with a 45/0 (or 0/45) spectrophotometer in which the specimen is directly illuminated by a D_{65} simulator meeting the source conformance factor requirement of this report; this is stated to be equivalent to a rating not worse than BC (CIELAB) relative to D_{65} as determined by Publication CIE No. 51 [27]. If these criteria are not met, the user is referred to the literature for correction methods. The origin of the source conformance factor is given [40].

A more extensive *Recommended Practice for Method of Measurement for Color of Fluorescent Objects* [41] has been prepared by the Color Science Association of Japan. The basic or principal method involves spectrophotometric measurement with an instrument utilizing 45/0 or 0/45 geometry and direct illumination by a Japanese Industrial Standard Z 8902 xenon source to obtain the spectral total radiance factor. Subsequently one of three (subordinate) methods can be used to obtain the spectral reflected radiance factor and apply the necessary corrections for the desired results: They are the two-mode, the fluorescence-weakening, and the two-monochromator methods.

A Drafting Committee of Tentative Method of Colorimetry for Fluorescent Objects of the Color Science Association of

Japan, chaired by L. Mori, carried out an intercomparison very similar to that described in this report [42]. Almost identical results were obtained; they are discussed below.

Finally, three papers dealing with fluorescence were presented at the 1985 AIC Congress held in Monaco, June, 1985 [43-45].