## INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

### PHYSICAL CHEMISTRY DIVISION

COMMISSION ON PHYSICOCHEMICAL MEASUREMENTS AND STANDARDS

## SUBCOMMITTEE ON CALIBRATION AND TEST MATERIALS

# RECOMMENDED REFERENCE MATERIALS FOR REALIZATION OF PHYSICOCHEMICAL PROPERTIES

Recommendations (1976)

EDITOR: E. F. G. HERINGTON

## SECTION: REFLECTANCE

COLLATORS: H. FEUERBERG and H. TERSTIEGE

PERGAMON PRESS OXFORD · NEW YORK · PARIS · FRANKFURT PHYSICAL CHEMISTRY DIVISION COMMISSION ON PHYSICOCHEMICAL MEASUREMENTS AND STANDARDS SUBCOMMITTEE ON CALIBRATION AND TEST MATERIALS

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#### INTRODUCTION

If a material is exposed to optical radiation, some portion of the incident radiation is reflected, another portion absorbed and often a third portion transmitted (Ref. 1). These recommendations deal with the process of reflection and characterization of the quantity *reflectance*. Reflection is a return of radiation by a specimen without change of the wavelength. The reflected radiation can be regular, diffuse or mixed.

Regular (specular) reflection is reflection without diffusion in accordance with the laws of optical reflection as by a mirror.

Diffuse reflection is reflection in which, on the macroscopic scale, there is no regular reflection.

Reflectance,  $\rho$ , is the ratio of the reflected radiant (luminous) flux to the incident flux.

Where mixed reflection occurs, the reflectance may be divided into two parts, corresponding to the two modes of reflection (and then the reflectance is often termed the total reflectance) to distinguish it from whichever of the component parts is the dominant one.

Regular (specular) reflectance,  $\rho_r$ , is the ratio of the regularly reflected radiant (luminous) flux to the incident flux.

Diffuse reflectance,  $\rho_d$ , is the ratio of the diffusely reflected radiant (luminous) flux to the incident flux.

Radiance (luminance) factor  $\beta$ , is the ratio of the radiance (luminance) of the sample to that of the perfect reflecting diffuser identically irradiated (illuminated).

The conditions of irradiation and view of the specimen must be specified in every instance; the CIE recommended geometries have been published (Ref. 2).

The basis of reflectance measurements and applications of reflectance are described in detail by Kortum (Ref. 3). There has been an increased interest in reflectance measurements recently and indeed the reflection properties of paper, of textiles, and of building materials have now gained much importance. Reflectance measurements are also used in the aerospace industry, where reflectance values are required for radiant heat transfer analysis. Absolute measurements of regular reflectance and of diffuse reflectance are frequently very difficult. The results of measurements of reflectance depend very much on the measuring conditions, especially on the spectral composition of the radiation, the state of polarization of the radiation, the angle of incidence, the angle of viewing, the angular extent of the incident radiation and the viewing beam, the thickness of the sample, the temperature and the state of the surface. Measurements of reflectance can be made easier and measurements by various people can be made comparable by the use of reliable reference materials.

Reflectance reference materials are recommended by the CIE (Commission Internationale d'Eclairage) and by several Technical Committees of ISO (International Organization for Standardization), e.g. by ISO/TC 6 for brightness and reflectance measurements of paper, boards and pulps, but these organizations have not published catalogues of reference materials. Therefore it is hoped that the present recommendations will be of assistance to people making reflectance measurements.

Reflectance is usually measured by comparing the reflecting properties of a sample with that of a reference material. Reflectance reference materials previously used for this purpose were freshly smoked magnesium or pressed magnesium oxide but some other reference material was required because the reflectance values of magnesium oxide were found to be inadequately reproducible partly because this material ages relatively rapidly. Magnesium oxide has therefore not been included in the following list of recommended materials. Barium sulphate has been proved to have better reproducibility; its absolute reflectance varies only slightly from sample to sample and it is most eminently suitable as a reference material for reflectance. For these reasons barium sulphate is recommended by the CIE Committee TC-2·3 in (Ref.) and by ISO Committee TC/6 and by the German Standard Specification (Ref.4). Barium sulphate is available with spectral reflectance (radiance) factors given for six wavelengths from 350 to 700 nm.

Plates of Pyroceram porcelain, opalescent glass, enameled iron discs and ceramic tiles with certified reflectances are convenient to use on a routine basis because they are easy to clean and are permanent in their optical behaviour. All these materials have to be calibrated by a barium sulphate sample. Regular reflectance values of suitable samples of black glass with a high refractive index can be calculated from the refractive index values. The reflectance of these materials should always be related to the perfect reflecting diffuser and corrections made for the deviations of the reflecting reference material from the behaviour of a perfect reflecting diffuser. Eight reference materials are described on the following pages; some information on coloured tiles and on black glasses is available in another publication from this Commission (Ref. 5).

The following provisos apply to the information on Reference Materials: (a) the recommended materials have not been checked independently by IUPAC, (b) the quality of materials may change with time, (c) the quoted sources of supply may not be exclusive sources because no attempt has been made to seek out all possible alternative sources, (d) the IUPAC does not guarantee any material that is recommended.

#### REFERENCES

- 1. Radiometric and Photometric Characteristics of Materials and Their Measurement. Publication CIE, No. 38 (TC-2·3) (1977).
- 2. Colorimetry, Publication CIE, No. 15 (E-1.3.1) (1971)
- 3. G. Kortüm, Reflexionsspektroskopie, Springer, Berlin (1969).
- Colour Measurement. Reflectance Standards for Colorimetry and Photometry, DIN 5033, Part 9 (1970).
- Physicochemical Measurements: Catalogue of Reference Materials from National Laboratories. Pure Appl. Chem. 48, 503 (1976).

1

Physical property: Regular spectral reflectance Unit: Dimensionless Recommended reference material: Aluminium on glass Range of variables: 0.2537-30µm Physical state within the range: solid Class: Certified Reference Material Contributor: J. P. Cali

Intended usage: This reference material is used for the calibration of measurement equipment and for the evaluation of the thermal radiation properties of materials.

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Sources of supply and/or methods of preparation: Samples (Ref.1) are available from supplier (E) under the SRM Numbers 2001 - 2004. The mirrors are available in four sizes:

SRM No.	Size of blank (cm)	Coated area (cm)
2001	7.6 x 10.2 x 1.9	5.1 x 7.6
2002	3.8 x 3.8 x 1.3	2.5 x 2.5
2003	Disk: 2.9 diameter x 1.0 thick	Entire surface
2004	Disk: 2.4 diameter x 0.6 thick	Entire surface

Pertinent physicochemical data: Each mirror is certified for near-normal  $(9^{\circ})$  regular reflectance at wavelengths ranging from 0.2537 to 30 micrometers and at corresponding resolved bandwidths from 1.0 to 1800 nanometers. The standard deviation of the mean of six replicate measurements is given.

#### REFERENCE

2

1. National Bureau of Standards Special Publication 260-38, Washington, D.C. (1972).

Physical property: Regular spectral reflectance Unit: Dimensionless Recommended reference material: Gold on glass Range of variables: 0.2537-30µm Physical state within the range: solid Class: Certified Reference Material Contributor: J. P. Cali

Intended usage: This reference material is used for the calibration of measurement equipment and for the evaluation of the thermal radiation properties of materials.

Sources of supply and/or methods of preparation: Samples (Ref. 1) are available from supplier (E) under the SRM Numbers 2005-2008. The mirrors are available in four sizes:

SRM No.	Size of blank (cm)	Coated area (cm)
2005	7.6 x 10.2 x 1.9	5.1 x 7.6
2006	3.8 x 3.8 x 1.3	2.5 x 2.5
2007	Disk: 2.9 diameter x 1.0 thick	Entire surface
2008	Disk: 2.4 diameter x 0.6 thick	Entire surface

Pertinent physicochemical data: Each mirror is certified for near-normal regular reflectance at wavelengths ranging from 0.2537 to 30 micrometers and at corresponding resolved bandwidths from 1.0 to 1800 nanometers. The standard deviation of the mean of six replicate measurements is given.

#### REFERENCE

1. National Bureau of Standards Special Publication 260-38, Washington, D.C. (1972).

3

Physical property: Regular reflectance Unit: Dimensionless Recommended reference material: Polished glass with a specified refractive index Range of variables: 380-780nm Physical state within the range: solid Class: Calibration and Test Material Contributors: H. Feuerberg and H. Terstiege

Intended usage: This reference material is used for the calibration of reflectometers employed for gloss assessment of plane surfaces (Refs. 1,2) and it can also be used with goniophotometers.

Sources of supply and/or methods of preparation: Samples are available from supplier (A).

Pertinent physicochemical data: Black glass with a fictive refractive index of 1.567 gives a reflectometer value of 100 for  $20^{\circ}$ ,  $60^{\circ}$  and  $85^{\circ}$  geometries. Black glasses with refractive indices lower than 1.567 which exhibit reflectometer values lower than 100 are suitable as reference materials (Ref. 3). The specular reflection for various angles of incident radiation are calculated from values of the refractive index by means of the Fresnel equation for unpolarized light.

#### REFERENCES

German Standard Specification DIN 67 530 (1972).
 ASM D523 - 67.

3. W. Czepluch, Farbe und Lack 78, 619 (1972).

4

Physical property: Radiance factor, reflectance Unit: Dimensionless Recommended reference material: Barium sulphate Range of variables: 350-700nm Physical state within the range: solid Class: Certified Reference Material Contributors: H. Feuerberg and H. Terstiege

Intended usage: This reference material is used for the calibration of spectral reflectance photometers.

Sources of supply and/or methods of preparation: Samples are available from suppliers (B), (C) and (D).

Tablets of 25, 45 or 60mm diameter are pressed and can be produced from barium sulphate powder by means of a mechanical powder press: the tablets must be protected from dust anu moisture and can be used for several weeks (Ref. 1). The powder can be pressed into tablets only once. Further details concerning the technique have been given (Ref. 2).

Pertinent physicochemical data: Each batch of this reference material issued by supplier (D) is certified (Ref. 3) by supplier (I). Six values of the radiance factor,  $\beta$ , are given (Ref. 4) for d/O and 45/O geometries for the range 350-700nm.

#### REFERENCES

1. H. Terstiege, Lichttechnik 26, 277 (1974).

- 2. Radiometric and Photometric Characteristics of Materials and Their Measurement.
- Publication CIE, No.38 (TC-2.3) (1977).
- 3. H. Korte and M. Schmidt, Lichttechnik 19, 135 (1967).
- 4. German Standard Specification DIN 5033, Part 9 (1972).

5

Physical property: Reflectance Unit: Dimensionless Recommended reference material: Pyroceram porcelain Range of variables: 380-780nm Physical state within the range: solid Class: Calibration and Test Material Contributors: H. Feuerberg and H. Terstiege

Intended usage: This reference material can be used for the calibration of spectral reflectance photometers.

Sources of supply and/or methods of preparation: Samples are available from supplier (K) with directions for use.

Pertinent physicochemical data: The reflectance factors are measured with high accuracy by the use of barium sulphate as reference material.

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Physical property: Reflectance Unit: Dimensionless Recommended reference material: Opalescent glass Range of variables: 320-800nm Physical state within the range: solid Class: Calibration and Test Material Contributors: F. J. J. Clarke, H. Feuerberg and H. Terstiege

Intended usage: Opalescent glass is used for the calibration of spectral reflectance photometers, reflectometers or colorimeters.

Sources of supply and/or methods of preparation: Specimens are available from suppliers (G), (H) and (K).

Pertinent physicochemical data: Supplier (G) provides the 0/45 radiance factor, the diffuse part of the 0/d reflectance and the total 8/d reflectance determined with respect to barium sulphate. For each of the three geometries the CIE colorimetric quantities (x, y, Y) are computed from the spectral values for CIE Standard Illuminants A, C and D65 (Refs. 1,2).

Supplier (H) supplies a rental service (period 2 or 3 weeks) of calibrated specimens which can be supplied with reflectance values for d/0 or 0/d geometries.

Supplier (K) provides a calibration certificate for spectral reflectance factors with each specimen but specimens have to be recalibrated after two or three months usage. Calibration can be done by the user with barium sulphate as a reference material or calibration can be done by supplier (A).

#### REFERENCES

- F. J. J. Clarke, F. A. Garforth and D. Parry, National Physical Laboratory Report, MOM 13, National Physical Laboratory, Teddington (1975).
- 2. W. Budde, Calibration of Reflectance Standards, J. Res. Nat. Bur. Std. 80A, 585 (1976).

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Physical property: Reflectance Unit: Dimensionless Recommended reference material: Enameled iron discs Range of variables: 380-760nm Physical state within the range: solid Class: Calibration and Test Material Contributor: E. Juhász

Intended usage: These discs can be used for the calibration of spectral reflectance photometers.

Sources of supply and/or methods of preparation: Supplier (F) provides a set of 16 discs: one white, one grey, four red, four yellow, three green and three blue all different.

Pertinent physicochemical data: The spectral reflectance factor has been determined with high accuracy relative to barium sulphate for the wavelength range 380-760nm in 10nm steps. Tristimulus values and chromaticity coordinates for the two illuminants A and C and geometries 0/d, d/0, 45/0 and 0/45 are calculated (Ref. 1).

#### REFERENCE

 Gy. Dézsi and L. Fillinger, Mérésügyi Közlemémyek. Journal of the National Office of Measures, Hungary 9, 34 (1968).

8

Physical property: Radiance factor, diffuse reflectance and total reflectance Units: Dimensionless Recommended reference material: Ceramic tiles Range of variables: 300-750nm Physical state within the range: solid Class: Certified Reference Material Contributor: F. J. J. Clarke Intended usage: These tiles can be used to calibrate reflection spectrophotometers, reflectometers and colorimeters (Refs. 1-4).

Sources of supply and/or methods of preparation: Tiles in sets of 12 measuring 100mm x 100mm are available from supplier (G). In each set three are spectrally neutral greys for the investigation of errors of linearity or geometrical errors, and nine are spectrally selective colours for the investigation of waveband or wavelength calibration, scanning or recording mechanism errors or spectral response errors. Infrared measurements to 2µm are also available.

Pertinent physicochemical data: The 0/45 radiance factor, the diffuse part of the 0/d reflectance and the total 8/d reflectance of the tiles have been determined with barium sulphate as the reference material. For each of three geometries the CIE colorimetric quantities (x,y,Y) have been computed from the spectral values for CIE Standard Illuminants A, C and D65.

#### REFERENCES

- 1. F. J. J. Clarke and P. R. Samways, National Physical Laboratory Report MC 2, National Physical Laboratory, Teddington (1968).
- 2. F. J. J. Clarke, Printing Technol. 13, 101 (1969).
- 3. F. J. J. Clarke, Die Farbe 20, 299 (1971).
- 4. F. J. J. Clarke, Colour 73, Froc. Inv. volour Assoc. p.346. Hilger, London (1973).

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- (A) Bundesanstalt für Materialprüfung, Unter den Eichen 87,
   D-1000 Berlin 45 (Federal Republic of Germany)
- (B) Eastman Kodak Company, Rochester, New York 14650 (USA)
- (C) Hopkins and Williams Ltd., Freshwater Road, Chadwell Heath, Essex (UK)
- (D) E. Merck, Postfach 4119.
   D-6100 Darmstadt 2 (Federal Republic of Germany)
- (E) National Bureau of Standards, U.S. Department of Commerce, Washington, DC 20234 (USA)

- (F) National Office of Measures, Németvölgyi Út 37, H-1124, Budapest XII (Hungary)
- (G) National Physical Laboratory, Teddington, Middlesex TW11 OLW (UK)
- (H) National Research Council, Ottawa, Ontario KIA OR6 (Canada)
- Physikalisch-Technische Bundesanstalt, Bundesallee 100, Braunschweig D-3300 \* Merck (Federal Republic of Germany)
- (K) Carl Zeiss A.G., D-7082 Oberkochen/Württ (Federal Republic of Germany)