International Standard 7724/2

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Paints and varnishes — Colorimetry — Part 2 : Colour measurement

Peintures et vernis — Colorimétrie — Partie 2 : Mesurage de la couleur

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Contents

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Paints and varnishes — Colorimetry — Part 2 : Colour measurement

0 Introduction

This International Standard, ISO 7724, comprises the following parts :

Part 1 : Principles.

Part 2 : Colour measurement.

Part 3 : Calculation of colour differences.

Parts 1, 2 and 3 of ISO 7724 describe methods for the instrumental determination of the colour co-ordinates and colour differences of paint films, as required for such purposes as

a) the objective description of colour differences between a test specimen (a test panel coated with a paint, or a specimen from a painted article) and a reference specimen;

b) determining colour deviations in the production of painted articles so that the results may be used for the control or regulation of the process;

c) the objective description of changes in colour caused by weathering and other chemical or physical influences;

d) the objective supervision of colour reference standards.

NOTE — Colour reference standards are subject to ageing, which may lead in the course of time to pronounced changes in colour. High accuracy colorimetry is required for the timely detection of these changes. This is of particular importance when ordering against such reference standards.

1 Scope and field of application

This part of ISO 7724 describes the method for determining the colour co-ordinates of paint films. The method is only applicable to paint films that appear to be uniformly of one colour, i.e. monochromatic, when examined with normal vision. Paint films that do not completely hide a non-transparent substrate represent an opaque system and may be measured by using the procedure described in this part of ISO 7724.

Luminescent paint films, transparent paint films and translucent paint films (for example for displays or lamp glass), retroreflecting paint films (for example for traffic signs), and metallic paint films are outside the scope of this part of ISO 7724.

2 References

ISO 1512, *Paints and varnishes — Sampling.*

ISO 1513, *Paints and varnishes — Examination and preparation of samples for testing.*

ISO 1514, *Paints and varnishes — Standard panels for testing.*

ISO 2808, *Paints and varnishes — Determination of film thickness.*

ISO 3534, *Statistics — Vocabulary and symbols.*

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

ISO 7724/1, *Paints and varnishes — Colorimetry — Part 1 : Principles.*

ISO 7724/3, *Paints and varnishes — Colorimetry — Part 3 : Calculation of colour differences.*

CIE Publication No. 15, Supplement No. 1, *Special metamerism index : Change in illuminant.*

3 Principle

Measurement of the tristimulus values X_{10}, Y_{10}, Z_{10} by one of the following methods

— measurement of the spectral photometric characteristics (spectral reflectances or spectral reflectance factors) of the paint film with a spectrophotometer and calculation of the tristimulus values;

— measurement of the tristimulus values with a tristimulus colorimeter.

Calculation of the chromaticity co-ordinates x_{10}, y_{10} or the colour co-ordinates in the approximately uniform CIE 1976 ($L^*a^*b^*$) colour space from the tristimulus values, as described in ISO 7724/1.

4 Choice of illumination and viewing conditions

One of the measuring conditions for illumination and viewing specified in ISO 7724/1 should be chosen taking into account the surface texture, the reflection properties of the paint film tested and the information that is to be obtained by the measurement.

4.1 Smooth non-textured paint films

All the measuring conditions specified in ISO 7724/1 are suitable for determining the colour co-ordinates of smooth non-textured paint films.

With high gloss specimens, results obtained under all measuring conditions are comparable if the tristimulus values determined with an integrating sphere without gloss trap are corrected for the surface reflection.

NOTE — The surface reflection is the part of the light falling on a specimen that is reflected (diffusely and specularly) at the surface; another part is back-scattered by the pigments (volume reflection). The surface reflectance (Fresnel reflectance) for illumination at incidence between 0° and 8° on paint films is approximately 0,04.

For all other specimens the choice of measuring conditions depends on whether the specular reflection is to be included in or excluded from the measurement (see ISO 7724/3).

4.1.1 Measurement including the specular reflection

Use the measuring condition 8/d or d/8 (*without* gloss trap for both).

NOTE — If the gloss changes without a visually perceptible change in colour, for example after weathering, the tristimulus values measured including the specular reflection will generally not be influenced.

4.1.2 Measurement excluding the specular reflection

Use the measuring condition 8/d or d/8 (*with* gloss trap for both) or 45/0 or 0/45.

NOTE — If the gloss changes, the diffuse part of the surface reflection will change and consequently the tristimulus values measured excluding the specular reflection change too.

4.2 Paint films with surface texture

4.2.1 Measurement including the specular reflection

For the spectral characterization of paint films with surface texture (for example textured finishes) use the measuring condition 8/d or d/8 (*without* gloss trap for both).

4.2.2 Measurement excluding the specular reflection

For matt or low gloss specimens use the measuring conditions 8/d or d/8 (*with* gloss trap for both). Alternatively, use the measuring condition 45/0 or 0/45 if the specimen is turned during the measurement, or the measuring condition 45/0 if the specimen is illuminated toroidally or by two beams at 90° to each other.

NOTE — For paint films with surfaces that have high gloss and are textured, the measuring condition 8/d or d/8 (with gloss trap) and 45/0 or 0/45 should not be used because random specularly reflected light may reach the detector.

5 Choice of standard colorimetric observer and standard illuminant

The CIE 1964 supplementary standard colorimetric observer and standard illuminant D65 should preferably be used.

For the colorimetric determination of a special metamerism index (change in colour difference between two specimens caused by substituting a test illuminant for the reference illuminant, preferably D65[1]) standard illuminant A should be used as the test illuminant.

Tristimulus colorimeters of early design are provided with filters that are only suitable for determining tristimulus values with the CIE 1931 standard colorimetric system for standard illuminant C. If the colorimetric determination is based on the tristimulus method, the use of this combination of standard colorimetric observer and standard illuminant should be allowed, but its use should be reported.

6 Apparatus

6.1 Spectrophotometer

For high precision colorimetry, a single- or preferably a double-beam reflection spectrophotometer equipped with a prism or grating monochromator and a photometer head that satisfies the illumination and viewing conditions chosen as described in clause 4 shall be used.

Using these instruments, the repeatability with which spectral reflectances or spectral reflectance factors can be measured should be better than the larger of the following two values : 0,2 % of the reading or 0,001 (absolute).

The repeatability over long time intervals should not exceed these values by a factor of more than 3.

NOTE — For the purpose of this part of ISO 7724, the repeatability of the measuring method is the value below which the absolute difference between two single test results obtained for the same paint film under the same conditions (same operator, same spectrophotometer, at a short time interval) may be expected to lie with a 95 % probability. The repeatability is obtained by multiplying the standard deviation of the measuring method by the appropriate factor (see ISO 5725).

1) see CIE Publication No. 15, Supplement No. 1.

The accuracy should be better than the larger of the following two values : 0,5 % of the reading or 0,002 (absolute).

NOTE — For the purpose of this part of ISO 7724, accuracy is the closeness of agreement between the true value and the mean result which would be obtained by applying the same method of colorimetry a very large number of times (see ISO 3534).

If the colorimetric measurements are for the objective supervision of colour reference standards [clause 0, d)], a spectrophotometer of the type described above should be used in preference to the abridged type (6.2) or a tristimulus colorimeter (6.4).

6.2 Abridged spectrophotometer

For the applications described in clause 0, a) to c), an abridged spectrophotometer equipped with a photometer head that satisfies the illumination and viewing conditions chosen as described in clause 4 is sufficient, except when highly chromatic paint films with steep spectral reflectance curves are to be measured. Such an instrument is a photometer containing at least 16 interference filters distributed uniformly over the wavelength range 400 to 700 nm and having a half-bandwidth of 20 nm or less.

For the short- and long-term repeatability, the same conditions as those defined for spectrophotometers in 6.1 apply.

The accuracy should be better than the larger of the following two values : 1 % of the reading or 0,004 (absolute).

6.3 Integrator

For weighting the measured spectral photometric characteristics with the relative spectral power distribution of the standard illuminant S_λ and the colour-matching functions $\bar{x}_{10}(\lambda)$, $\bar{y}_{10}(\lambda)$, $\bar{z}_{10}(\lambda)$ and the summation as described in ISO 7724/1, a computer facility with adequate storage may be used.

Abridged spectrophotometers and some automatic spectrophotometers embody electronic or mechanical integrators.

6.4 Tristimulus colorimeter

The integration can also be performed optically with three tristimulus filters which shall be constituted so that the measurements appear in a simple linear relationship with the tristimulus values. Instruments of this category, which are termed tristimulus colorimeters, have to be equipped with a photometer head that satisfies the illumination and viewing conditions chosen as described in clause 4.

The three filters have to be fitted with respect to their spectral transmittance $\tau_x(\lambda)$, $\tau_y(\lambda)$, $\tau_z(\lambda)$ to the colour-matching functions, to the relative spectral power distribution of the standard illuminant and of the light source of the instrument, and to the sensitivities of the photoelectric detectors. With few exceptions the manufacturers of tristimulus colorimeters make no provision for close fitting. The τ_x filter completely absorbs light in the wavelength range below 500 nm. The tristimulus value X_{10} is therefore formed by weighting the reflectance factors measured with τ_x and τ_z filters with various constants followed by addition.

Because of the difficulties in fitting filters to the required parameters, tristimulus colorimeters are generally unsuitable for measuring colour *per se* and should be restricted to measuring colour difference. Even in the latter use, however, there may be difficulties in establishing compliance with a reference colour if the standard and the specimen are metameric. Consequently, tristimulus colorimeters are best restricted to applications b) and c) described in clause 0.

The repeatability with which tristimulus values can be measured should be better than the higher of the following two values : 0,2 % of the reading or 0,001 (absolute).

The accuracy can be worse than 1 %, depending on the lightness, and the shape of the reflectance curve.

7 Sampling and preparation of test specimens

For the applications described in clause 0 a) and c) take a representative sample of the paint to be tested as described in ISO 1512.

Examine and prepare the sample for testing as described in ISO 1513.

Prepare the test panels in accordance with ISO 1514. Apply the coating, dry and, if necessary, condition the paint film as directed in the manufacturer's instructions or as otherwise agreed.

NOTE — If the paint film is of reversible temperature-dependent colour, the temperature of the test specimen should be maintained at 23 ± 2 °C by appropriate measures (for example by the use of a thermostatically controlled specimen holder). Paint films of irreversible temperature-dependent colour should be postconditioned, until the colour does not change any more. In the case of paint films that do not completely hide the substrate, the colour depends on the colour of the substrate and the film thickness, which should be determined by one of the procedures described in ISO 2808.

From painted articles [application described in clause 0, b)] take test specimens that are representative of the surfaces to be measured. If necessary, the number of specimens should be agreed upon between the parties.

The paint film specimen shall be plane and clean. The dimensions of the test specimen should be adequate to permit measurement with the size dimensions of the sample port of the instrument in use with the sample port being not less than 10 mm in diameter.

8 Reflectance standards

8.1 Primary standard

The primary standard for measuring the reflectance or reflectance factor of the specimen is the perfect reflecting diffuser as

recommended by CIE and defined as an ideal uniform diffuser with a spectral reflectance equal to unity for all wavelengths.

8.2 Secondary standard

Because the primary standard cannot be realized in practice, a secondary standard is needed with known spectral reflectances that are as close as possible to those of the primary standard.

8.2.1 Source

Compressed barium sulfate powder in tablet form is normally used as the secondary standard. The manufacturer of the barium sulfate powder used to prepare the tablets should state the spectral reflectances $\varrho_{8/d}(\lambda)$ or spectral reflectance factors $R_{45/0}(\lambda)$ of the powder for various wavelengths. These wavelengths shall be chosen so as to allow interpolation of reflectances or reflectance factors to within an accuracy of $\pm$ 0,001. The stated values shall refer to barium sulfate tablets prepared by the described method and measured using an absolute measuring technique, i.e. related indirectly to the primary standard (see clause 11).

The powder shall be free of contaminants and shall be suitable for compressing.

NOTE — The spectral reflectance $\varrho_{8/d}(\lambda)$ and spectral reflectance factor $R_{d/8}(\lambda)$ of tablets made of barium sulfate powder almost coincide. The extent to which the spectral reflectance factors $R_{45/0}(\lambda)$ and $R_{0/45}(\lambda)$ of tablets made of barium sulfate powder agree has yet to be determined.

8.2.2 Preparation and checking

With a mechanical powder press used solely for this purpose, mould constant amounts of barium sulfate powder into tablets having a minimum thickness of 5 mm and a density of 1,6 to 1,7 g/cm³. The tablets shall exhibit a textureless, plane and matt surface. Clean the powder press carefully after each pressing operation as traces of residual powder may degrade the surface quality of tablets. Tablets shall not be reprepared from the same powder sample.

The repeatability of the reflectance of the secondary standard prepared according to this part of ISO 7724 shall be better than 0,2 % with a given batch of barium sulfate powder.

Reflectance standards prepared in this way, if carefully handled and stored in a desiccator, can be used for 1 week. Care should be taken to ensure that they are not exposed to ultraviolet radiation (λ < 270 nm) because this could induce a change in the spectral reflectances in the wavelength range below 450 nm (see clause 11).

The following apparatus and procedure have proved suitable for preparing barium sulfate standards. A schematic representation of the powder press is shown in the figure.

A metal ring which will later serve as a frame for the tablets is first closed at the bottom by a clean matt-ground glass plate [see the note and the figure a)]. The ring and glass plate are held together by a clamp (not shown). A weighed amount of powder (1,65 g/cm³ tablet volume) is poured into the cylinder inserted in the ring and the two sections of the powder press are united so that the plunger enters the cylinder. Both sections are now screwed together. To increase the density of the tablets, the top section should be tapped gently several times with a rubber hammer. The top section can then be screwed in further. Tapping and screwing should be repeated two or three times until the powder fills only the conical portion of the ring. The top section with the plunger and cylinder should then be removed and replaced by a lid. The press should now be turned over [figure b)] and the clamp opened to allow the removal of the bottom section with the glass plate. The exposed surface of the tablet should come level with the front side of the ring, which abuts with the port of the photometer. The powder has now been compressed so tightly that the surface of the tablet can even be used in the perpendicular position or suspended without losing any grains of powder.

NOTE — The surface of the glass plate that faces the powder is ground matt with abrasive (grain size about 0,3 µm) and afterwards etched with dilute hydrofluoric acid.

The glass plate should be thoroughly cleaned in chromosulfuric acid followed by rinsing first in distilled water and then in ethanol and allowed to dry. For interim cleaning a wad of cotton wool impregnated with ethanol is sufficient.

8.3 Working standards

For routine measurements with an individual instrument it is possible to use working standards with spectral reflectances that remain stable over a long period. These working standards need not be uniform diffusers but shall be calibrated with the aid of the barium sulfate standard and the instrument with which they are used. They should be made from stable and durable materials such as opal glasses, vitrolite or ceramic tiles. The surface shall be polished to ease removal of surface contamination and for ease in cleaning.

Neutral grey working standards should be used to check the linearity of the photometric scale and to extend the photometric range of the instrument, so that dark specimens may be measured more reliably than would otherwise be possible.

Checking the repeatability and the accuracy of the test method can be carried out by using a number of spectrally selective working standards.

9 Procedure

9.1 Method using a spectrophotometer

The instrument shall be operated in accordance with the manufacturer's instructions.

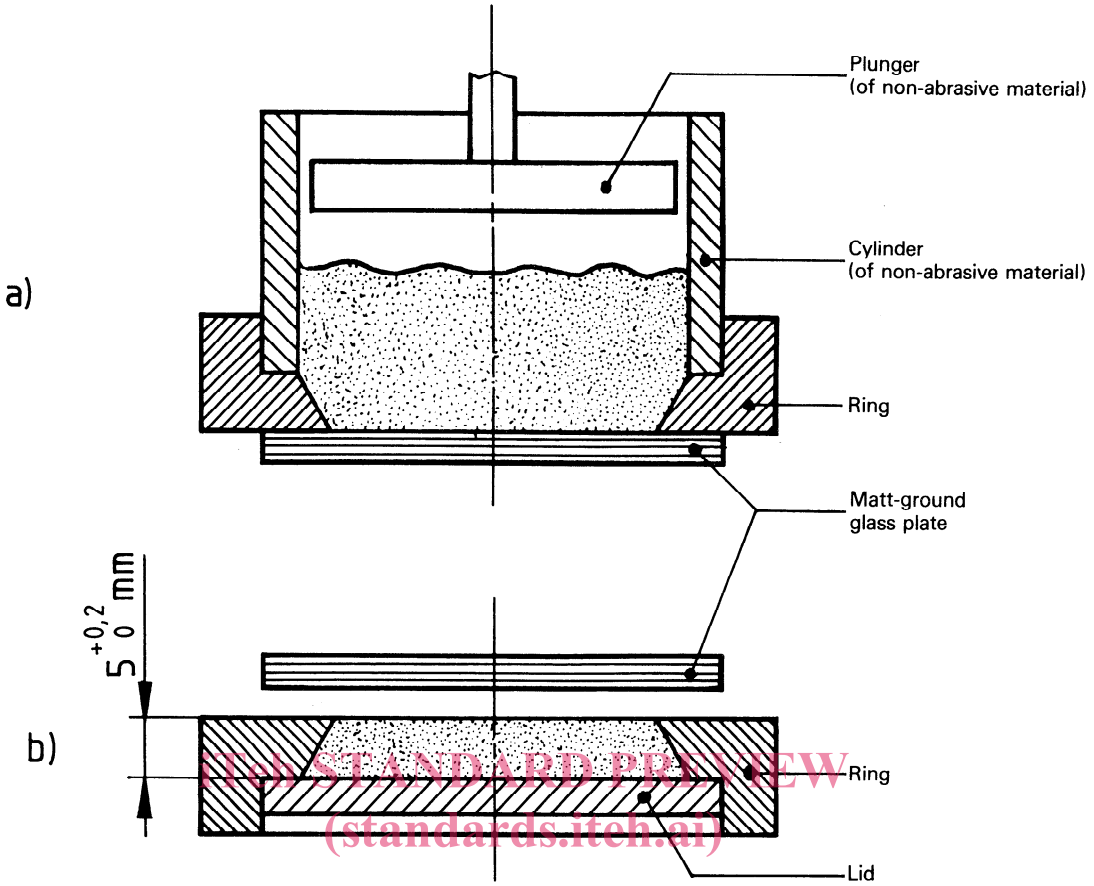

Figure — Powder press for preparing barium sulfate ($BaSO_4$) reflectance standards

Adjust the photometric scale or the intensity measuring unit and the integrator, if provided. Set the zero point by use of a highly efficient light trap at the sample port. Do not set the zero point by blocking the light beam. Set the upper end-point of the scale by an appropriate reflectance standard over the entire visible wavelength range.

Check the linearity of the photometric scale by using grey working standards with known spectral reflectances.

Calibrate the wavelength scale using cadmium or mercury discharge lamps with sharp emission lines.

NOTE — Further instrumental errors can be avoided by the exclusion of diffused light (for example by using the appropriate stray light filter) and by cleaning contaminated optical surfaces.

Check whether the repeatability corresponds to the value given by the manufacturer of the instrument by using suitable spectrally selective working standards.

Periodically check again the linearity of the photometer scale, the calibration of the wavelength scale, and repeatability, dependent on the time and intensity of use of the instrument.

Adjust the slit width or the slit-width programme to a desirable balance between spectral resolution and sensitivity with respect to the chosen wavelength interval (see next paragraph) and the lightness of the paint film that is to be measured. Select an appropriate recording speed.

Measure in wavelength intervals $\Delta\lambda = 10$ nm over the spectral range 380 to 770 nm using monochromatic light with a half-intensity width of less than 10 nm. In special cases, such as highly chromatic paint films with steep spectral reflectance curves, measure in 5 nm wavelength intervals using monochromatic light with a half-intensity width of less than 5 nm.

Dependent on the chosen measuring conditions, determine the spectral reflectances or the spectral reflectance factors, or when using an integrator, the tristimulus values and chromaticity co-ordinates of the specimen in relation to the reflectance standard.

To detect the introduction of an error into any measurement, repeat each measurement. In the case of discrepancies larger than the characteristic repeatability given by the instrument's manufacturer, repeat the measurement several times and, if necessary, eliminate the cause of the discrepancies.