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Standard Reference Materials:

USE OF STANDARD LIGHT-SENSITIVE PAPER FOR CALIBRATING CARBON ARCS USED IN TESTING TEXTILES FOR COLORFASTNESS TO LIGHT

U.S.
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Standard Reference Materials:

Use of Standard Light-Sensitive Paper For Calibrating Carbon Arcs Used in Testing Textiles For Colorfastness to Light

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Inquiries concerning the technical content of this paper should be directed to the author(s). Other questions concerned with the availability, delivery, price, and so forth will receive prompt attention from:

Office of Standard Reference Materials
National Bureau of Standards
Washington, D.C. 20234

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Office of Standard Reference Materials

CONTENTS

	PAGE
1. INTRODUCTION	1
2. NBS MASTER LAMP	3
3. LIGHT-SENSITIVE PAPER	4
4. STANDARD FADING HOURS (SFH)	6
5. EFFECT OF DEVIATIONS FROM STANDARD CONDITIONS	7
5.1 BLACK-PANEL TEMPERATURE	7
5.2 HUMIDITY	8
5.3 INTERMITTENT EXPOSURE	9
6. PROCEDURE FOR EXPOSURE	9
7. RATING OF THE PAPERS AFTER EXPOSURE10
7.1 VISUAL ESTIMATION10
7.2 REFLECTANCE MEASUREMENTS11
7.3 CALIBRATION BY OTHER PROPERTIES13
8. FREQUENCY OF CALIBRATION13
9. PROCEDURE FOR ORDERING PAPER AND BOOKLETS15
10. REFERENCES17

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USE OF STANDARD LIGHT-SENSITIVE PAPER FOR CALIBRATING
CARBON ARCS USED IN TESTING TEXTILES FOR
COLORFASTNESS TO LIGHT

by

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The use of NBS Light-Sensitive Paper and NBS Booklets of Standard Faded Strips in the standardization of carbon arcs is described. These lamps are used to determine the fading characteristics and degradation of textiles and other materials when subjected to visible and ultraviolet radiation. The light-sensitive paper is useful in calibrating the arcs in terms of NBS Standard Fading Hours (SFH) or equivalent exposure in the NBS Master Carbon-Arc Lamp.

Key words: Carbon-arcs, standardization of; color-fastness, testing of; fading tests, standardization; light-sensitive paper; paper, light-sensitive; textiles, fading of.

1. INTRODUCTION

Carbon-arc lamps are widely used for testing colorfastness and stability of materials to visible and ultraviolet radiation. The variability of the output of a given lamp and the differences between lamps have led to the extensive use of standard reference materials that undergo measurable changes when exposed to this radiation.

The American Association of Textile Chemists and Colorists, following work begun in 1931 or earlier, developed a set of 8 blue wool standards covering various ranges up to 640 hours of exposure. The steps are logarithmic, each standard requiring approximately twice the exposure of the previous one to obtain equivalent fading. These are in extensive use in testing the fading of textiles. One of these, denoted as L-4, has been the subject of a detailed study

[1] which showed that for carbon-arc exposures near 20 hours the standard deviation of a single instrumental measurement of color difference corresponded to about 3 Standard Fading Hours (SFH). A group of 9 observations near 20 SFH would then be expected to range from 15.5 to 24.5 SFH, since statistical theory predicts that the range is approximately the product of the standard deviation by the square root of the number of observations. This precision is adequate for its intended purpose of placing an unknown specimen in the proper step of the 8-step logarithmic scale of exposures, but is not good enough to serve as a basis for calibrating and adjusting the arcs.

Consequently the National Bureau of Standards, following research work begun about 1940 [2], developed and issued standard light sensitive-paper, for which during a given exposure the change in spectral reflectance (at various wavelengths) is from 10-20 times that of the wool [1] and the change in luminous reflectance, or tristimulus \underline{Y} value, is 15-20 times that of the wool. The precision of determination of exposure is found to be about 4-fold better than that with the wool. A group of 9 observations on the paper near 20 SFH is expected to range from about 18.8 to 21.2 SFH. The period of 20-24 hours is particularly important because it corresponds approximately to the life of one set of carbons.

In calibrating a given carbon arc the paper is exposed and then compared with six strips of the paper which have been exposed for different known periods of time in the NBS Master Carbon-Arc Lamp. The calibration procedure outlined below is the same as that currently specified in Appendix B of AATCC Test Method 16A-1971 (Technical Manual, American Association of Textile Chemists and Colorists, P.O. Box 12215, Research Triangle Park, North Carolina 27709).

2. NBS MASTER LAMP

The present NBS Master Lamp is an Atlas Electric Device type SMC-R enclosed carbon-arc Fade-Ometer operated on alternating current.* The arc voltage is 120-145 V and the current 15-17 A (ranges specified by the manufacturer). The construction of the unit is almost identical with that of the Fade-Ometer described in detail by Boor and Trucker [3]. It uses No. 70 solid carbons and No. 20 cored carbons. The solid carbon is placed in the upper holder and the cored carbon in the lower holder for one run, and these positions are reversed for the next run. The arc is enclosed in a special Pyrex glass globe (No. 9200PX).

The specimens are exposed (without backing) in a rack 20 in. (50.8 cm) in diameter rotating at 3 rpm.

*Footnote:

Manufactured by Atlas Electric Devices Co., 4114 N. Ravenswood Avenue, Chicago, Illinois 60613.

Certain commercial equipment and instruments are identified here and elsewhere in this publication in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the equipment or instruments identified are necessarily the best available for the purpose.

A black panel of enameled metal is placed in one of the specimen positions in the rack. The black-panel temperature is held at $150 \pm 5^{\circ}\text{F}$ ($65.6 \pm 2^{\circ}\text{C}$) by dampers which automatically control the ratio of exit air to recirculated air. The control element for the dampers is in the stream of humidified inlet air and is manually set to maintain the black-panel temperature. It should be noted that the American Association of Textile Chemists and Colorists specifies a black-panel temperature of $145 \pm 5^{\circ}\text{F}$ ($63 \pm 3^{\circ}\text{C}$) in AATCC Test Method 16A-1971.

The variation during a single run seldom exceeds $\pm 4^{\circ}\text{F}$. The ambient temperature (77°F , 25°C) and humidity (45% RH, Dew Point 54°F , 12°C) and size of our present laboratory are such that under these conditions the temperature of the exit air is usually $118 \pm 2^{\circ}\text{F}$ ($47.8 \pm 1^{\circ}\text{C}$).

Moisture is added to the circulating air by water sprayed from a rotating disk to raise the relative humidity of the exit air to 30% at the dry-bulb temperature of 118°F . A bimetallic thermostat responding to wet-bulb temperature starts and stops the rotating disk. The humidity is controlled by automatically maintaining the wet-bulb temperature at a desired value (88°F , 31°C in the present instance).

The dew point of the exit air under these conditions is 79°F (26°C). It would be quite inconvenient to maintain a higher humidity, since a dew point higher than room temperature gives rise to condensation difficulties when the air is allowed to cool from 118°F to room temperature.

3. LIGHT-SENSITIVE PAPER

Successive lots of light-sensitive paper have been issued by the National Bureau of Standards since about 1945 [2]. They are now sold as items in the NBS Standard Reference Materials program and carry the designations SRM 700 for the unexposed paper and SRM 701 for the booklets

of six strips which have been exposed for stated periods of time in the NBS Master Lamp. The particular lot is designated by a letter following the SRM number. Thus, SRM 700a and SRM 701a were issued in June 1965, and were followed by SRM 700b and SRM 701b in June 1967. The most recent lot is SRM 700c and SRM 701c, issued in November 1971. Lots differ slightly both in the reflectance of the unexposed paper and in the rate of fading. Consequently the paper from a given lot can be used only with the booklet from the same lot. Each order of booklet and package of paper is accompanied by a Certificate of Calibration including the numerical values characteristic of the particular lot.

The paper, made in the National Bureau of Standards paper mill, contains the direct azo dye Benzo Azurine G, Colour Index Direct Blue [eight] No. 24140. The unexposed paper is supplied in pieces $2\frac{5}{8}$ by $3\frac{1}{4}$ inches. Each booklet contains 6 paper strips (about $1\frac{1}{2}$ by 3 inches) which have been faded by exposure for different times in the NBS Master Carbon Arc described in the preceding section. To facilitate production the booklets are assembled in four different series with the following nominal values:

Series A - 6, 8, 10, 14, 18, and 22SFH;
Series B - 7, 9, 11, 15, 19, and 23SFH;
Series C - 8, 10, 12, 16, 20, and 24SFH;

and

Series D - 9, 11, 13, 17, 21, and 25SFH.

The different series have the same-sized intervals, and the booklets of each series are fully equivalent to each other.

4. STANDARD FADING HOURS (SFH)

The quantitative basis for establishing a unit standard fading hour (SFH) at the National Bureau of Standards before 1950 has been discussed in detail in a previous publication [1]. A "Fade-Ometer Hour" had already been used for some years in the textile industry, and 20 SFH was defined as being equivalent to 20 clock hours (CH) of exposure in the Atlas Type FDA-R Carbon-Arc Fade-Ometer, the type in most general use at the time. Since February 1949, every effort has been made by repeated comparisons involving light-sensitive paper to keep constant the dosage of carbon arc radiation represented by 20 SFH.

In order to attain this aim, over the years repeated observations have been made of the color changes found in a given lot of paper for 20 hours of operation of the NBS Master Carbon-Arc Lamp. No significant differences have been found. Each new lot of paper has been compared carefully with one or more of the preceding lots at the time it has been introduced. The exposures of the new lot have been expressed in terms of the SFH defined by the preceding lots.

Over the years the conditions of operation of the master carbon-arc lamp have been controlled and specified with progressively increasing detail and some changes in the arc have been made. As a result, it is found that a 20-hour exposure in the present NBS Master Lamp under the conditions of operation given above causes fading of the light-sensitive paper by an amount equivalent to about 18 SFH.

Defining a severity factor F as the ratio of standard fading hours SFH to clock hours CH , one finds that F for the NBS Master Lamp is about 0.9 at 20 CH. It is possible to vary the severity factor for the carbon arc somewhat by changing the arc voltage by using a different transformer

tap, but only a limited range of adjustment is possible. The severity factor can be altered markedly by deviations from the standard conditions of specimen distance, humidity, and black-panel temperatures. It should be kept in the range 0.9 ± 0.1 at 20 CH. The severity factor generally decreases with decreasing exposure time. The severity is obviously lower than that employed prior to 1950 (when $F = 1.0$). The reduction is probably due chiefly to the lowering of the recommended effective black-panel temperature from its early value of about 165°F (73.9°C) to 150°F (65.6°C).

5. EFFECT OF DEVIATIONS FROM STANDARD CONDITIONS

In calibrations at the National Bureau of Standards all work is done under the standard conditions already described. It is of interest to examine the effects of several deviations from these standard conditions.

5.1 BLACK-PANEL TEMPERATURE

A recent unpublished NBS study of the fading of the paper at lower black-panel temperatures (while maintaining 30% RH for the exit air) shows a reduction in the number of SFH at the rate of about 0.3 SFH for each Fahrenheit degree. Thus if a 20-hour exposure gives 18 SFH at 150F, about 16.5 SFH will be found at 145°F and about 12.9 SFH at 133°F. The corresponding severity factors are 0.9, 0.83, and 0.65, respectively. The last temperature (133°F, 56.1°C) is the lowest black-panel temperature attainable without increasing the cooling facilities in the room where our arc is located.

Similar observations regarding the effect of temperature on the fading of NBS Light-Sensitive Paper Lot 1554 were reported by Boor and Trucker [3].

5.2 HUMIDITY

In the NBS Master Lamp the relative humidity is maintained at 30% at the temperature of the exit air, usually about 118°F. This is accomplished by adding moisture to the air passing over the specimens. It has been pointed out that the dew point is an important parameter in these considerations [4]. The dew point under these conditions is about 79°F. Reported values of dew point and relative humidity appreciably higher than those just mentioned are questionable, because measurement inaccuracies almost always lead to spuriously high values of humidity. Furthermore, if no condensation is observed as the exit air cools to room temperature, the dew point must be below room temperature. On the other hand, if condensation is observed, the presence of liquid water droplets can cause serious experimental difficulties.

If the lamp does not have provision for adding moisture, the dew point of the air around the specimens will be approximately the same as that of the laboratory. Unless the laboratory humidity is controlled, the dew point may vary from a maximum of perhaps 79°F on a very humid summer day to 10°F or less during the winter. The corresponding range of relative humidity when the air is heated to 118°F without the addition of moisture is from 30% to about 2%.

The fading of the paper, as well as that of most textiles, is considerably reduced as the humidity is lowered below the standard condition of 30% at exit-air temperature. Some indication of the magnitude of this effect can be noted from the results of Boor and Trucker [3] on paper from Lot 1554. The reduction in fading is particularly great when the relative humidity falls below 20%.

5.3 INTERMITTENT EXPOSURE

The light-sensitive paper, as well as many textiles, fades more in a given time if exposed intermittently than if exposed continuously, probably because of an increase in relative humidity of the air and moisture content of the paper while the lamp is shut off, with more rapid fading when it is first turned on again. It is therefore necessary to begin tests with a fresh set of carbons in the lamp and to avoid shut-downs.

6. PROCEDURE FOR EXPOSURE

Mount a piece of unexposed light-sensitive paper in a specimen holder of the lamp without backing and place it in the lamp at the time it is started. The side of the paper facing the arc should be the one containing no printing. Furthermore, the paper should be placed so that the printed matter is not immediately back of the portion of the strip which is being exposed. Vacant positions in the specimen holder should be filled with dummy specimens to reduce variations in reflections and air currents.

At the end of the run remove the strip and allow it to stand in the dark at room temperature for at least 2 hours in order for it to cool and to regain its normal moisture from the air. If the fading is to be judged only visually, trim off and discard the unexposed edges of the paper and of the strips in the booklets as they may affect the judgment of the fading. For instrumental measurements the edges should remain attached for enhanced ease of handling during the measurements.

7. RATING OF THE PAPERS AFTER EXPOSURE

7.1 Visual Estimation

Trim off and discard the unexposed edges of the paper, as already mentioned, so that the exposed paper may be placed immediately adjacent to the standard faded strips in the booklet. To make a visual comparison, hold the booklet in one hand, allow the pages to flip open one after another, rear cover first, and slip the exposed paper under one standard faded strip after another, being careful to have the standard strip superimposed closely upon the exposed paper with the long dimension of the two papers in the same direction. Make the comparison in the light from a daylight fluorescent lamp, or equivalent source, with illumination of 50 foot candles or more on the papers. The plane of the incident and reflected rays should be perpendicular to the long edge (machine direction) of the paper and booklet. The incident light should strike the paper at an angle of 45° , and the angle of viewing should be perpendicular to the surface of the paper. Avoid touching the surfaces of the exposed paper and the standard strips with the fingers, as the paper is sensitive to moisture and is easily soiled.

From the comparison, estimate the exposure (to the nearest Standard Fading Hour) that would duplicate the fading of the test piece. For example, if the color of the test piece was considered to be mid-way between those of the standard strips labelled 16 and 20 SFH in the booklet, the estimated value would be 18 SFH. If the test piece had been exposed for 20 hours, the severity factor SFH/CH would then be $18/20 = 0.9$, the value recommended for normal operation of a carbon arc. The exposure in SFH at other times would then be taken as 0.9 of the time in clock hours.

The severity factor for the carbon arc should normally be kept between 0.8 and 1.0 at 20 CH. If it is found to be

outside this range, change of the transformer tap or other adjustments should be made until this condition is attained.

In making preliminary comparisons of faded papers and standard faded strips, it is permissible to breathe on the paper momentarily to increase its moisture content. Final comparisons, however, should be made after the paper has been conditioned overnight or longer in the dark in air at 50% relative humidity at room temperature.

7.2 REFLECTANCE MEASUREMENTS

Although the paper and booklets were originally designed for simple visual estimation of the fading of test strips without the use of any accessories other than the eye, greater precision can be obtained by the use of an instrument measuring a property which changes with exposure. The property used at the National Bureau of Standards in evaluating the standard faded strips in the booklets is the $45^\circ, 0^\circ$ luminous-directional reflectance factor R_d as measured with a green filter with a Hunter Color and Color Difference Meter (Gardner type) [5]. This is equal to the tristimulus value \underline{Y} . As with the visual estimation, the plane of the incident and reflected rays should be perpendicular to the long edge of the paper. Likewise the incident light should strike the paper at an angle of 45° , as the reflectance normal to the surface is measured. The individual numerical value found at the time of preparation of each strip in the booklet is written in ink on the strip itself. The corresponding exposure (to the nearest 0.1 SFH) may be obtained from this value by interpolation in the table or graph furnished with the Certificate of Calibration for the particular lot of paper. In this manner the exposure of the standard faded strips can be fixed much more accurately. Their position in the booklet indicates the exposure only to the nearest unit SFH.

The papers may change in reflectance on further exposure to light, heat, humidity or by mishandling or soiling.

Reflectance measurements on different instruments often show appreciable differences, even when measuring the same piece of exposed paper. Differences in reflectance readings can arise from differences in geometry of incident and viewing light beams, linearity of photometric scales, spectral sensitivity of the photocell, spectral transmittance of filters, and similar causes.

Reflectance measurements are made with the paper against a black background with the machine direction of the paper perpendicular to the plane of the incident and reflected rays. The machine direction (grain) of the paper lies along the longer side of both the unexposed paper and the standard strips in the booklet.

Most reflectometers yielding tristimulus \underline{Y} values can be adjusted, if necessary, to make their readings agree with those shown in ink on the standard faded strips. For repeated standardizations a porcelain plaque of about the same spectral reflectance as the exposed paper is recommended in order to avoid repeated handling of the paper.

Some reflectometers have a measurement precision that permits readings to four significant figures, as shown in the graph and table contained in the Certificate of Calibration. However, a comparison of reflectance measurements, on single strips given the same exposure shows a standard deviation of about 0.002. This corresponds to about 0.8 SFH near 20 SFH and about 0.7 SFH near 6 SFH.

If the user has available an instrument measuring reflectance and if the values observed with the six standard faded strips agree with or can be adjusted to agree with the values of \underline{Y} written in ink on each strip, the instrument is in agreement with the one at the National Bureau of Standards. Consequently the graph and table

given in the Certificate of Calibration can be utilized to obtain SFH for any test strip exposures.

7.3 CALIBRATION BY OTHER PROPERTIES

If the agreement just mentioned can not be obtained, or if the available instrument measures some property of the paper other than the tristimulus value \underline{Y} , the standard faded strips may still be used to calibrate this instrument. The property, presumably an optical property, can be any one which changes quantitatively with exposure. In this case a measurement is made on each of the six standard faded strips. The values found with the user's instrument are plotted as a function of the exposure of the standard strips in SFH (obtained from the reflectance marked on each strip) to obtain a six-point calibration curve for the property measured. When the user's instrument is calibrated in this manner, the same number of SFH should be obtained from a newly-exposed test strip of paper as would have been obtained if the calibration had been made in terms of any other property, including specifically the tristimulus \underline{Y} value used at the National Bureau of Standards.

8. FREQUENCY OF CALIBRATION

Many laboratories always expose one or more strips of paper along with the textile specimens under test, thus obtaining a calibration every time the arc is operated. This is desirable since the fading rate of a carbon arc lamp may change from day to day or even during a test.

If the paper is not used during every run, a definite schedule for calibration at fixed intervals, should be developed by each user in accordance with his own requirements. Certainly the arc should be recalibrated whenever a new lot of carbons is put into use or any other

significant change is made. The more frequently checks are made, the greater will be the assurance that the lamp is performing as desired.

For obtaining exposures near to a desired number of SFH, duplicate strips of paper may be used, so that a preliminary value of the severity factor may be obtained from one strip before the estimated end of the run. The following instructions illustrate the procedure for obtaining an exposure of 20 SFH.

Place the textile specimen and two (or more if desired) pieces of the paper in the lamp at the same time, side by side. Remove one of the papers from the lamp about 4 hours before the estimated end of the test, noting the time. Allow this paper to stand in the dark at room temperature for 2 hours; compare the fading with that of the standard faded strips; and obtain the severity factor for converting clock hours to SFH as already outlined. Use this factor to calculate the time in clock hours to give the desired number of SFH at the end of the test.

The piece of paper left in the lamp with the textile will have received the same radiant-energy exposure as the textile at the end of the test. The actual exposure in SFH at the end of the test will then be obtained by rating the fading of this paper with the standard faded strips in the booklet. The result can be used as evidence that the test was as desired or sufficiently near to it, or that the test must be repeated.

For long exposures, a succession of papers will have to be used and the number of Standard Fading Hours shown by them added together to obtain the total exposure. In making such tests, the paper should be changed with each change of carbons.

9. PROCEDURE FOR ORDERING PAPER AND BOOKLETS

Different issues of the light-sensitive paper have different fading rates. Consequently, the booklets of standard faded strips and the fading curve supplied in the Certificate of Calibration for any one issue of unexposed paper apply to that issue only; therefore booklets and unexposed paper of the same issue should be used together. The lower case letter at the end of the standard sample number indicates the issue of the standard sample. The booklet of standard fading strips No. 701c should be used with unexposed paper No. 700c. It cannot be used with No. 700b or any earlier issue of the paper. However, when the supply of unexposed paper No. 700c at NBS becomes exhausted, a new issue will be supplied as Standard Reference Material 700d. It will then become necessary for the customer to order a new booklet of standard faded strips No. 701d to be used with the new issue of paper. This is unavoidable because it is not possible at present to control the manufacture of the paper so that all of the issues of paper are exactly the same with regard to fading rate.

The unexposed paper cannot be used satisfactorily without the booklet of standard faded strips because of differences in measuring instruments, as outlined earlier. This publication, explaining the use of the paper and booklet, and a Certificate of Calibration accompany each shipment of a sample of the unexposed paper or booklet of standard faded strips. They will be sent separately, free, on request.

Order by Standard Reference Material Number (e.g., 700c for the unexposed paper; 701c for the booklet of standard faded strips). Address orders to:

Office of Standard Reference Materials
National Bureau of Standards
Washington, D. C. 20234
Attention: Standard Reference Materials Unit

Prices and terms of purchase are given in NBS Miscellaneous Publication 260, "Catalog and Price List of Standard Reference Materials Issued by the National Bureau of Standards".

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