

# Certificate of Calibration

## Standard Reference Material 702

### Light-Sensitive Plastic Chip

### 0.124 Inch

Two tentative curves for the fading of light-sensitive plastic (chips) are presented. These plates are made of poly (methyl methacrylate) containing the dye, Solvent Yellow 33, C. I. 47,000, and their thickness is nominally  $\frac{1}{8}$  inch. They are to be used in the calibration of artificial fading and weathering apparatus. When used in such equipment, the radiation causes fading of the dye. The calibration of these plates was carried out in a carbon arc Fade-Ometer and is not directly transferable to other types of equipment.

The calibration of the plates was carried out against NBS Standard Reference Light Sensitive Paper No. 700a. This paper provided the definition of the Standard Fading Hour Unit (SFH). The calibration was carried out in the NBS master lamp, which is an Atlas Fading Device SMCR Type Carbon Arc Fade-Ometer. The diameter of the rack is 20 inches, and the samples to be exposed are mounted so that they are at the level of the arc. The lamp was operated so that the black panel temperature, as defined in AATCC Test Method 16A-1964, Appendix A, was  $150^{\circ} \pm 5^{\circ} \text{F}$ , and so that the relative humidity, as measured by wet- and dry-bulb thermometers at the air exit of the lamp was  $30 \pm 5$  percent.

The method of calibration was as follows: Exposures of plates were carried out for nominally 8, 16, 32, 64, 128, and 256 SFH of exposure. For each of these times, nine plates were chosen at random, using a table of random numbers, from the lot of plates. The plate was broken in two, one-half saved for subsequent measurement of transmission and the other half mounted at random in nine of the nineteen positions of the NBS Standard Lamp. The holder for the plates was an Atlas CD-LSR holder modified to hold the plates. Its designation by Atlas Fading Devices Company is F-1376. The plates were exposed without backing in the lower position of the holder. The other ten positions of the lamp were occupied by Standard Fading Paper No. 700a. Since the fading curve of the paper is not defined beyond 25 SFH, in those runs which necessitated longer exposures than this, the lamp was stopped at appropriate times, the faded papers removed and measured, the plastic plates moved to other locations chosen at random and the empty positions filled with fresh papers. This was continued until the requisite number of SFH of exposure was achieved.

The thickness of each half chip was measured at  $73.5^{\circ} \text{F}$  with a micrometer capable of estimation to  $10^{-4}$  inch. The average of all the measurements was 0.1243 inch with a total spread of 0.0017 inch.

At the end of the exposure time the transmittance of the plastic plates was measured by the NBS Photometry and Colorimetry Section on the GE Recording Spectrophotometer according to ASTM Tentative Method D1920-64T. The slit width was approximately 10 nm (nanometers,  $10^{-9}$  meter) of spectral width. Both unexposed and exposed halves of each plate were measured. To reduce polarization effects, two measurements were made on each plate. The orientation of the plate for these two measurements differed by a rotation of the plate by 90 degrees about the axis normal to the plane of the plate. The results of the measurements at these orientations were averaged.

The results for the averages of the nine unexposed and exposed halves for each exposure time are given in table 1. Transmittance is given both at the minimum which occurs nominally at 418 nm, and at 420 nm as recommended in ASTM Tentative Method D1920-64T. It is strongly recommended that the transmittance at the minimum be used since accurate calibration of the wavelength scale is then obviated.

All the results were subjected to statistical analysis. It was found empirically that the transmittance of the plates followed the equation.

$$\log T = a + b (\text{SFH})^{1/2}. \quad (1)$$

The data may also be represented on a plot of  $\log T$  versus  $\log SFH$ . However, to obtain as adequate a fit on such a plot as obtained from eq 1, a cubic equation  $\log T = a + b \log SFH + c (\log SFH)^2 + d (\log SFH)^3$  is required. Since eq 1 is much easier to use, it was adopted. The values of the constants for eq 1 and their standard errors are given in table 2 for calibration both by transmission measured at the minimum at 418 nm and at 420 nm. The table also gives the standard error of fit and the coefficient of variation computed from it.

Analysis was also carried out for the transmittance of each exposed plate divided by the transmittance of the unexposed half of the plate. These results showed a greater standard error of fit and it is concluded that there is no correlation between the transmittance of the exposed plate and the unexposed plate. The transmittance of the unexposed plate was measured to be 0.1519 at the 418 nm minimum and 0.1552 at 420 nm. The standard deviations were 0.0035 and 0.0036 respectively.

The results are plotted in figures 1 and 2 for transmittance at the minimum and at 420 nm against SFH respectively. The curves in these graphs are calculated from eq 1 and these are the calibration curves for this Standard Reference Material. The experimental points are also shown on these graphs.

#### Recommended Method of Use

It is anticipated that these plates will be used according to ASTM Method D1920-64T. However, certain factors should be borne in mind:

(a) If a spectrophotometer with different slit width from the GE Recording Spectrophotometer is used, different results from those shown in the figures will be obtained. The unexposed plate should have a transmittance of 0.152 at the minimum at 418 nm and 0.155 at 420 nm.

(b) The calibration was carried out at a black panel temperature of  $150^\circ \pm 5^\circ F$ , measured as in AATCC Test Method 16A-1964, and at a relative humidity in the lamp of 30  $\pm$  5 percent as measured by wet and dry bulb thermometers at the air exit from the test chamber of the lamp. While it is expected that relative humidity will not affect the fading rate of these plates appreciably, temperature almost certainly will and these calibration curves are therefore not certified for other temperatures.

These plates were manufactured by the Stimsonite Division, Elastic Stop Nut Corporation of America, Chicago, Illinois. The calibration was carried out at the National Bureau of Standards Institute for Materials Research by Paul J. Shouse and E. Passaglia of the Polymer Physics Section, E. Passaglia, Chief. This certificate was prepared by E. Passaglia.

TABLE 1

SFH of Exposure	Clock Hours of Exposure	Measurements at 418 nm minimum		Measurements at 420 nm	
		Transmittance of exposed plate	$\sigma^a$	Transmittance of exposed plate	$\sigma^a$
8.8	11.1	0.2319	0.0023	0.2358	0.0025
16.9	18.0	.2456	.0022	.2490	.0028
32.8	37.0	.2692	.0022	.2734	.0025
70.9	80.0	.3121	.0018	.3167	.0019
134.1	148.9	.3733	.0031	.3761	.0032
262.8	300.0	.4764	.0055	.4800	.0052

<sup>a</sup> Standard deviation of an individual measurement.

Transmittance of unexposed plates.

at minimum at 418 nm:	0.1519	$\sigma = 0.0035$
at 420 nm:	0.1552	$\sigma = 0.0036$

TABLE 2

Fit to Equation:  $\text{Log } T = a + b (\text{SFH})^{1/2}$

*For transmittance measured at minimum at 418 nm*

$\text{Log } T = 0.70492 + .02369 (\text{SFH})^{1/2}$

Standard errors:

of $a$ :	0.00116
of $b$ :	0.000124
of an individual point:	0.00420
Coefficient of variation:	0.97%

*For transmittance at 420 nm*

$\text{Log } T = -0.69690 + 0.02338 (\text{SFH})^{1/2}$

Standard errors:

of $a$ :	0.00120
of $b$ :	0.000129
of an individual point:	0.00434
Coefficient of variation:	1.00%