U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards

NBS Special Publication 692

Transmittance MAP Service

Kenneth L. Eckerle, Jack J. Hsia, and Victor R. Weidner

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March 1985

U.S. Department of Commerce
Malcolm Baldrige, Secretary

National Bureau of Standards
Ernest Ambler, Director

Library of Congress
Catalog Card Number: 85-600513
National Bureau of Standards
Special Publication 692
Natl. Bur. Stand. (U.S.),
Spec. Publ. 692
50 pages (Mar. 1985)
CODEN: XNBSAV

U.S. Government Printing Office
Washington: 1985

For sale by the Superintendent
of Documents,
U.S. Government Printing Office,
Washington, DC 20402

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TRANSMITTANCE MAP SERVICE

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An introduction to the Transmittance Measurement Assurance Program (MAP) service is given. Documentation for the service is provided through a comprehensive list of references. The results of a pilot run for the MAP service are given in a sample calibration report.

Key Words: didymium filters; bandpass; glass filters; measurement assurance program; neutral density filters; reference spectrophotometer; transmittance; transmittance MAP service; uncertainty estimation; wavelength calibration

I. Introduction

The transmittance Measurement Assurance Program (MAP) [1]* provides a means for a laboratory to assess the accuracy of its spectral transmittance measurement capabilities. A laboratory which participates in this program will be sent a package of transmittance filters which have been measured at NBS. These are to be measured by the laboratory on its spectrophotometer(s) and returned, together with the measurement results, to NBS. NBS will then remeasure the filters and send a final analysis of the result to the participating laboratory. The range of filter measurements provided in the MAP package permits an evaluation of the accuracy of a laboratory's spectral transmission measurements and will often reveal the cause of systematic errors if any exist. This publication describes the contents and use of this MAP package.

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*Figures in brackets indicate the literature references on page 16.

II. MAP Package

The contents of the MAP package are itemized in Table 1 [2]. They include seven neutral density filters with nominal transmittances ranging from 0.92 to 0.001. Schott glass had previously been shown to be useful [2,3], and the filters were all constructed of this glass except the clear filter which is made of NBS crown glass. The filters are available in the three sizes given in Table 1. A participating laboratory should specify the size desired. These filters are calibrated by NBS using the method described in reference [4] (Included in Appendix A) at 548.5 nm for spectral passbands from 1.5 to 10.5 nm. The long term stability of a master set calibrated over 2 years showed that the transmittance of the clear glass filter decreased slightly while the other filters in creased slightly. The relative changes over two years for the seven filters in the master set were -0.12, 0.13, 0.15, 0.21, 0.36, 0.64, and 0.99 percent. In addition, each package contains one didymium glass filter which is to be used for wavelength scale calibration: several wavelengths of transmittance minima and points of inflection have been measured by NBS for these filters.[5] These wavelengths have been shown [6] to be stable over long periods of time; therefore these didymium filters are not normally remeasured by NBS with every use of the MAP package.

III. Instructions for the Care of the MAP Standards

Upon receipt of the MAP package the participating laboratory should open the shipping container and the flat metal box containing the filters, saving all the containers and packing material for use when returning the package to NBS. The flat metal box is opened by removing the six knob screws and should be used for storing the filters when they are not in use. Each neutral density filter is mounted in an aluminum frame with a Teflon cover. The filters must not be removed from these frames nor should they be cleaned except to remove surface dust or lint by light stroking with a clean camel-hair brush. In the unlikely event that a filter becomes contaminated or damaged NBS should be notified by telephone so that a procedure can be developed jointly which will minimize the loss of time and data.

IV. Instructions for Data Acquisition

Illustration 1 shows a form for reporting the participating laboratory's measurements of the neutral density filters. The filters should be measured as near their centers as possible (See appendix A and Section VII below for additional information on uniformity) and at a wavelength of 548.5 nm. This wavelength setting, as well as the wavelengths reported in the didymium glass filter measurements described below should be based upon the normal wavelength scale associated with the spectrophotometer being measured. Usually this will simply be the reading of a counter on the instrument but if such readings are normally corrected by an error correction curve then these corrections should be applied in these measurements too. The final report from NBS will, of course, refer any wavelength errors to this same scale. At least four transmittance measurements should be made of each neutral density filter (preferably on two successive mornings and afternoons) and the arithmetic mean and a standard deviation for the four measurements be computed.

Illustration 2 shows a form for reporting the measurements of the didymium glass filter. This filter is to be used in two experiments to test the accuracy of the wavelength scale of the instrument. For certain instruments a third experiment may be performed to diagnose causes of any anomalies observed in experiments 1 and 2.

Experiment 1. The transmittance minima are determined directly from the instrument or a graph by inspection using transmittance versus wavelength curves. (See ref. [5], pp. 4-9 and Appendix B).

Experiment 2. In addition to the transmittance minima, points of inflection are determined. Included in the MAP package will be a table of the nominal calibration wavelengths and corresponding inflection point transmittance values required. The instrument wavelength must be adjusted until the tabulated transmittance is observed, and the corresponding wavelength is recorded. Since this is sometimes cumbersome, it is satisfactory to take measurements of the transmittance on either side of the tabulated transmittance and to perform a linear interpolation to find the wavelength corresponding to the given transmittance. (See ref. [5] p. 23 and Appendix B.)

Experiment 3. This experiment applies only to single beam instruments or to double beam instruments which can be configured like a single beam instrument. Most modern spectrophotometers are double beam instruments which automatically measure simultaneously both a 100% transmitting reference standard and the unknown sample, and this experiment may not be feasible for such an instrument. A single beam spectrophotometer is one which requires the operator to insert the 100% transmitting reference into the beam alternately with the sample to be measured. Usually the instrument gain is adjusted to read 100% while the reference is in the beam and then when the sample is in the beam the instrument reads directly the sample transmittance. For such a single beam instrument a further improvement in the wavelength scale accuracy can be obtained by consideration of the instrument sensitivity function (sometimes called the responsivity factor). Therefore for this experiment a graph is required of the signal versus wavelength, obtained simply by scanning the instrument over the visible spectrum with no sample in the instrument. In spectral regions where this signal changes rapidly with wavelength, the didymium glass spectral features will appear shifted. Although a calibration with the didymium glass filter calibrates the instrument completely with respect to the centroid of the passband (including the effects of the instrument sensitivity function), data from this experiment could increase the accuracy of a wavelength correction curve by correcting or identifying outliers in the wavelength measurements. The instrument sensitivity function could help NBS to distinguish between valid variations due to the instrument sensitivity function itself or other causes such as offset in the wavelength due to the sine bar or lead screw.

V. Pilot Run

After the MAP package and results of the participating laboratory's measurements have been received by NBS, the neutral density filters will be remeasured and a report will be prepared and returned to the laboratory. An example of such a report stemming from a pilot test of this MAP procedure is shown in Appendix B. In this case the laboratory possessed six spectrophotometers with varying passbands as indicated in the report. The report is self-explanatory. In particular, it gives the NBS before-and-after average values of the filter transmittances and the discrepancy between the laboratory's measurements and these values. It also gives an upper bound to the uncertainty in the NBS values. This uncertainty is based upon the arithmetic sum of a systematic error [7], three times the standard error based on four measurements, and the possible change in the filter between the 'before' and 'after' measurements. Finally it presents a table of wavelength errors based upon the didymium glass filter measurements and a graphical presentation of this data. In the example in Appendix B the results show that this laboratory's instrument #1 requires a wavelength adjustment.

VI. Recommendation for Participation

It is strongly recommended that any laboratory which participates in this MAP program acquire a set of filters as check standards which are similar to those supplied in this MAP package and that these be measured along with the MAP package filters. These filters will then serve as an inhouse reference which should be remeasured periodically and whenever any change in equipment, technique, or operator has taken place. A plot of such measurements against time can be used to establish a control chart in order to verify the continuing accuracy of a measurement process. The user should strive to obtain at least 15 such data points. More detail on control charts can be found in ref. [1] and examples in ref. [3].

VII. NBS Research Results.

The MAP package filters are measured using a reference spectrophotometer developed at NBS.[7,8,9] A statistical analysis showed measurement at 5 wavelengths [4] to be sufficient to specify the transmittance at bandpasses between 1.5 and 10.5 nm. Scattering, polarization, and uniformity were also checked. The linearity of the spectrophotometer was checked using the double aperture method [10]. Other systematic errors are discussed in reference [11], and their effect is published in reference [7]. These transmittance filters are calibrated for specular or "regular" transmittance with no inter-reflections between sample and optics or sample and detector. The uncertainty of the neutral filters varies with the transmittance. The NBS uncertainty of transmittance values (U) is estimated to be the arithmetic sum of a systematic error of approximately 0.0001 to 0.000003 transmittance units for transmittances from 0.92 to 0.001, respectively [7] plus 3 times the standard error based on four measurements. Since these measurements are for specular (or regular) transmittance, the effect of scattering by the filters on transmittance is negligible [4]. Polarization was also found to be statistically insignificant [4]. Non-uniformity of the filters was found to be statistically significant, so NBS would prefer the transmittance measurements to be made near the center of the filters. However, this is not always possible. Further, non-uniformity of small cuvette size filters can be large due to a possible wedge effect [12]. Consequently, the uniformity of one set of cuvette sized MAP filters were carefully measured for uniformity. The results are shown in Tables 2 through 8. Based on these data, NBS requests participants to specify spot size and location of the beam on the filter. The NBS uncertainty for the wavelength calibration using the didymium glass is approximately 0.3 nm or less [5].

VIII. Summary

The MAP service provides a means of checking linearity and wavelength of spectrophotometers with an accuracy which was not previously available. A user guide for participating in the MAP service is presented along with references documenting the method.

Acknowledgements. This project is supported by the Office of Physical Measurement Services (W. P. Reed, Acting Chief). We would like to thank R. K. Kirby, formerly with the Office of Physical Measurement Services. The authors wish to acknowledge J. B. Shumaker for his critical review and constructive suggestions. Thanks are due S. T. Chang for his help.

The Calibration Advisory Group subcommittee on the Transmittance MAP Service was composed of T. Vorburger, M. C. Croarkin, and R. W. Burke.

TABLE 1

Transmittance MAP Package

1. Neutral Density Filters: Calibrated at 548.5 nm only

Filter No.	Nominal Transmittance (%)	Remarks	
1	92	Borosilicate crown glass	
2	70	Schott	NG-11
3	50		NG-11
4	25		NG-4
5	10		NG-4
6	1		NG-9
7	0.1		NG-9

Size	Filter Holder	Filter Aperture
Large	51 x 51 mm	38 mm dia
Medium	51 x 38 mm	25 mm dia
Small	Cuvette	30 x 8 mm

2. Didymium Glass Filters for Wavelength:

Calibrated between approximately 400 to 750 nm for wavelength for triangular bandwidths between 1.5 and 10.5 nm.

Size		Remarks
Large	51 x 51 mm	Corning
Small	Cuvette	Corning

TABLE 2

Map of Uniformity of Filter 1 of a set of cuvette sized filters. The spotsize illuminating the filters is 2 by 6 mm with the long dimension vertical. The values in parentheses are the coordinates in mm of the center of the area on the filter with respect to the center of the filter which is illuminated when facing the detector. The transmittances, τ, are listed along with plus/minus three times the standard error based on four measurements. The values in square brackets are the deviations in transmittance units from the value of transmittance at the center position.

(-1,6)	(0,+6)	(+1,+6)
τ = 0.91688±0.00010	τ = 0.91722±0.00039	τ = 0.91731±0.00039
[0.00022]	[0.00056]	[0.00065]
(-1,0)	(0,0)	(+1,0)
τ = 0.91675±0.00033	τ = 0.91666±0.00017	τ = 0.91603±0.00027
[0.00009]	[0]	[-0.00063]
(-1,-6)	(0,-6)	(+1,-6)
τ = 0.91662±0.00024	τ = 0.91632±0.00021	τ = 0.91624±0.00018
[-0.00004]	[-0.00034]	[-0.00042]

TABLE 3

Map of Uniformity of Filter 2 of a set of cuvette sized filters. The spotsize illuminating the filters is 2 by 6 mm with the long dimension vertical. The values in parentheses are the coordinates in mm of the center of the area on the filter with respect to the center of the filter which is illuminated when facing the detector. The transmittances, τ, are listed along with plus/minus three times the standard error based on four measurements. The values in square brackets are the deviations in transmittance units from the value of transmittance at the center position.

(-1,6)	(0,+6)	(+1,+6)
τ = 0.69053±0.00015	τ = 0.68983±0.00030	τ = 0.69006±0.00018
[-0.00144]	[-0.00214]	[-0.00191]
(-1,0)	(0,0)	(+1,0)
τ = 0.69148±0.00021	τ = 0.69197±0.00020	τ = 0.69190±0.00021
[-0.00049]	[0]	[-0.00007]
(-1,-6)	(0,-6)	(+1,-6)
τ = 0.69133±0.00016	τ = 0.69131±0.00018	τ = 0.69150±0.00016
[-0.00064]	[-0.00066]	[-0.00047]

TABLE 4

Map of Uniformity of Filter 3 of a set of cuvette sized filters. The spotsize illuminating the filters is 2 by 6 mm with the long dimension vertical. The values in parentheses are the coordinates in mm of the center of the area on the filter with respect to the center of the filter which is illuminated when facing the detector. The transmittances, τ, are listed along with plus/minus three times the standard error based on four measurements. The values in square brackets are the deviations in transmittance units from the value of transmittance at the center position.

(-1,6)	(0,+6)	(+1,+6)
τ = 0.52054±0.00010	τ = 0.52038±0.00031	τ = 0.52053±0.00015
[0.00015]	[-0.00001]	[0.00014]
(-1,0)	(0,0)	(+1,0)
τ = 0.52049±0.00014	τ = 0.52039±0.00015	τ = 0.52029±0.00017
[0.00010]	[0]	[-0.00010]
(-1,-6)	(0,-6)	(+1,-6)
τ = 0.52049±0.00020	τ = 0.52020±0.00020	τ = 0.52028±0.00015
[0.00010]	[-0.00019]	[-0.00011]

TABLE 5

Map of Uniformity of Filter 4 of a set of cuvette sized filters. The spotsize illuminating the filters is 2 by 6 mm with the long dimension vertical. The values in parentheses are the coordinates in mm of the center of the area on the filter with respect to the center of the filter which is illuminated when facing the detector. The transmittances, τ, are listed along with plus/minus three times the standard error based on four measurements. The values in square brackets are the deviations in transmittance units from the value of transmittance at the center position.

(-1,+6)	(0,+6)	(+1,+6)
τ = 0.23625±0.00007	τ = 0.23603±0.00012	τ = 0.23595±0.00006
[0.00022]	[0]	[-0.00008]
(-1,0)	(0,0)	(+1,0)
τ = 0.23623±0.00008	τ = 0.23603±0.00012	τ = 0.23588±0.00007
[0.00020]	[0]	[-0.00015]
(-1,-6)	(0,-6)	(+1,-6)
τ = 0.23638±0.00013	τ = 0.23623±0.00011	τ = 0.23585±0.00008
[0.00035]	[0.00020]	[-0.00018]

TABLE 6

Map of Uniformity of Filter 5 of a set of cuvette sized filters. The spotsize illuminating the filters is 2 by 6 mm with the long dimension vertical. The values in parentheses are the coordinates in mm of the center of the area on the filter with respect to the center of the filter which is illuminated when facing the detector. The transmittances, τ, are listed along with plus/minus three times the standard error based on four measurements. The values in square brackets are the deviations in transmittance units from the value of transmittance at the center position.

(-1,+6)	(0,+6)	(+1,+6)
τ = 0.096705±0.000093	τ = 0.096746±0.000047	τ = 0.096532±0.000089
[-0.000103]	[-0.000062]	[-0.000276]
(-1,0)	(0,0)	(+1,0)
τ = 0.096726±0.000079	τ = 0.096808±0.000039	τ = 0.096538±0.000031
[-0.000082]	[0]	[-0.000270]
(-1,-6)	(0,-6)	(+1,-6)
τ = 0.096629±0.000041	τ = 0.096661±0.000038	τ = 0.096709±0.000078
[-0.000179]	[-0.000147]	[-0.000099]

TABLE 7

Map of Uniformity of Filter 6 of a set of cuvette sized filters. The spotsize illuminating the filters is 2 by 6 mm with the long dimension vertical. The values in parentheses are the coordinates in mm of the center of the area on the filter with respect to the center of the filter which is illuminated when facing the detector. The transmittances, τ, are listed along with plus/minus three times the standard error based on four measurements. The values in square brackets are the deviations in transmittance units from the value of transmittance at the center position.

(-1,+6)	(0,+6)	(+1,+6)
τ = 0.0091033±0.0000265 [-0.0000343]	τ = 0.0091233±0.000189 [-0.0000143]	τ = 0.0091324±0.0000188 [-0.0000052]
(-1,0)	**(0,0)**	**(+1,0)**
τ = 0.0091043±0.0000158 [-0.0000333]	τ = 0.0091376±0.0000100 [0]	τ = 0.0091332±0.0000128 [-0.0000044]
(-1,-6)	**(0,-6)**	**(+1,-6)**
τ = 0.0091103±0.0000076 [-0.0000273]	τ = 0.0091242±0.0000157 [-0.0000134]	τ = 0.0091483±0.0000207 [+0.0000107]

TABLE 8

Map of Uniformity of Filter 7 of a set of cuvette sized filters. The spotsize illuminating the filters is 2 by 6 mm with the long dimension vertical. The values in parentheses are the coordinates in mm of the center of the area on the filter with respect to the center of the filter which is illuminated when facing the detector. The transmittances, τ, are listed along with plus/minus three times the standard error based on four measurements. The values in square brackets are the deviations in transmittance units from the value of transmittance at the center position.

(-1,+6)	(0,+6)	(+1,+6)
τ = 0.0009277±0.0000054 [-0.0000020]	τ = 0.0009236±0.0000030 [-0.0000061]	τ = 0.0009248±0.0000032 [-0.0000049]
(-1,0)	(0,0)	(+1,0)
τ = 0.0009280±0.0000065 [-0.0000017]	τ = 0.0009297±0.0000042 [0]	τ = 0.0009260±0.0000040 [-0.0000037]
(-1,-6)	(0,-6)	(+1,-6)
τ = 0.0009305±0.0000044 [+0.0000008]	τ = 0.0009309±0.0000020 [+0.0000010]	τ = 0.0009304±0.0000044 [+0.0000007]

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ILLUSTRATION 1

Regular Transmittance, T

(Measured or interpolated at 548.5 nm)

Date of Measurement:

Filter Set No.:

Type of Instrument:

HALF HEIGHT BANDWIDTH:

Sample Beam (Collimated or Convergent):

Detector System (with or without averaging sphere):

Other Information:

Filter No.	T	Standard Deviation	No. of Measurements
-1			
-2			
-3			
-4			
-5			
-6			
-7			

ILLUSTRATION 2

WAVELENGTHS AT MINIMUM TRANSMITTANCE AND INFLECTION

(λ_{MIN}, λ_{INF})

Date of Measurement:

TYPE OF INSTRUMENT:

HALF HEIGHT BANDWIDTH:

Didymium glass filter:

EXPERIMENT 1. (FINDING TRANSMISSION MINIMA)

No.	Nominal λ_{MIN} (nm)	Measured λ_{MIN} (nm)
1	440	
2	478	
3	529	
4	585	
5	684	
6	740	

EXPERIMENT 2. (FINDING INFLECTION POINTS)

Nominal λ_{INF}(nm)	Trans- mittance, T	Measured λ_{INF}(nm)	Nominal λ_{INF}(nm)	Trans- mittance, T	Measured λ_{INF}(nm)
406			568		
429			600		
450			733		
485			756		
537					

EXPERIMENT 3. (Include curve of instrument sensitivity function.)

JOURNAL OF RESEARCH of the National Bureau of Standards
Vol. 88, No. 1, January-February 1983

Measurement Assurance Program Transmittance Standards for Spectrophotometric Linearity Testing:* Preparation and Calibration

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August 26, 1982

A Measurement Assurance Program for spectrophotometry is being established in order to assist laboratories involved in spectrophotometric calibrations. This paper deals with the preparation and calibration of neutral density glass filters for checking the linearity of photometric response, as applied to spectral transmittance measurements. Several sets of filters were prepared from suitable neutral glass to provide nominal transmittances of 92, 70, 50, 25, 10, 1, and 0.1% at a wavelength of 548.5 nm. These filter sets will be available in three sizes: these are, 38 mm diameter aperture in 51 x 51 mm holder, 25 mm diameter aperture in 51 x 28 mm holder, and 30 x 8 mm aperture in a cuvette holder. The filters were calibrated for spectral transmittance on the NBS Reference Spectrophotometer for high accuracy transmittance measurements. Measurements were made with a 1.5 nm passband collimated sample beam. The filters were checked for uniformity and measurements were made to determine the effects of sample beam polarization. The transmittance data for the wavelength range of interest were analyzed by statistical methods to determine the effects of passband for a range of 1.5 nm to 10.5 nm passband. The results of these measurements are presented in tabular and graphical detail for the master filter set.

Key words: experimental design; filter uniformity; linearity testing; measurement assurance program; neutral density glass; passband effects; polarization effects; polynomial fitting; spectrophotometry; statistics; transmittance standards.

1. Introduction

This paper deals with the preparation and calibration of neutral density glass filters for checking the linearity of photometric response, as applied to spectral transmittance measurements.

The application of absolute techniques, such as the double-aperture method [1],[1] for checking the linearity of a spectrophotometer's response, is difficult to realize on many instruments because of problems involving sample and reference beam geometry or detector configurations. The other approach to checking linearity is through the use of a set of transmittance standards. Several sets of glass filters have been prepared and calibrated for this purpose. A Measurement Assurance Program (MAP) is being established through which these sets of transmittance standards will be used for the purpose of improving the accuracy of spectrophotometer measurements in laboratories participating in the program.

*This project is supported by the Office of Measurement Services (Dr. B. C. Belanger, Chief) and coordinated by Dr. L. J. Kieffer.

**Center for Radiation Research, National Measurement Laboratory.

†Center for Applied Mathematics, National Engineering Laboratory.

[1]Figures in brackets indicate literature references at the end of this paper.

2. Selection and Preparation of the Standards

Some of the factors to be considered in selecting suitable filters for transmittance standards are: available range of transmittances, light scattering properties, uniformity, stability, passband sensitivity, and wavelength sensitivity. Schott[2] neutral density glass types NG-4, NG-9, and NG-11 were selected on the basis of these characteristics and properties as well as previous experiences with Schott glass by NBS in the preparation of Standard Reference Material (SRM) 930 [2].[3] Two levels of transmittance were prepared from each of the glass types as follows:

[2]Certain commercial materials are identified in this paper in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Bureau of Standards nor does it imply that the materials identified are necessarily the best available for the purpose.

[3]SRM 930 consists of three filters with nominal transmittances of 10%, 20%, and 30% calibrated at several wavelengths. Originally these SRM filters were designed for use by clinical chemists and are sold by the set. Our goal with the present filters was to provide a larger dynamic range so that the filters could be used to measure linearity in applications when other methods are difficult if not impossible. Also, these filters are not for sale, but are to be retained by NBS and issued only temporarily as part of a MAP measurement service.

Glass Type	Nominal Transmittance	Equivalent Transmission Density
NG-11	70%	0.15
NG-11	50%	0.3
NG-4	25%	0.6
NG-4	10%	1.0
NG-9	1%	2.0
NG-9	0.1%	3.0

An additional filter of borosilicate crown glass having a nominal transmittance of 92% (transmission density 0.036) is included in the set.

The filter sets were prepared in three sizes in order to accommodate different instruments. The largest filters are mounted in 51 × 51 mm holders with a filter aperture of 38 mm diameter. The intermediate size filters are mounted in 51 × 38 mm holders with a filter aperture of 25 mm. The smallest filters are mounted in cuvette holders and provide a filter aperture of 30 × 8 mm.

The preparation of the filters from the stock glass was done in the NBS Optical Shop. The tolerances on parallelism of the two faces of the filters was maintained at approximately 0.01°. They were polished to a flatness of 3 fringes or better.

Calculations of the required thicknesses were made from internal transmittance data supplied by the manufacturer. The internal transmittance of 1 mm path length for the three glass types is as follows:

Glass Type	1 mm path
NG-4	0.32
NG-9	0.035
NG-11	0.78

The values of transmittance for a wavelength of 500 nm were computed from these internal transmittance values. These transmittance values were converted to equivalent transmission density. Since the transmission density is linear with thickness, it is possible to determine, by graphical techniques, the required thickness for a desired transmittance value. Figure 1 shows the relationship of transmission density to thickness for the glass types NG-4, NG-9, and NG-11. The degree of accuracy in determining the required thickness for a given transmittance value by this graphical technique is dependent on the initial accuracy of the internal transmittance values used to determine the transmittance of 1 mm and 2 mm thicknesses. The actual transmittance values achieved by this technique closely approximated the desired nominal transmittance values for the filter set.

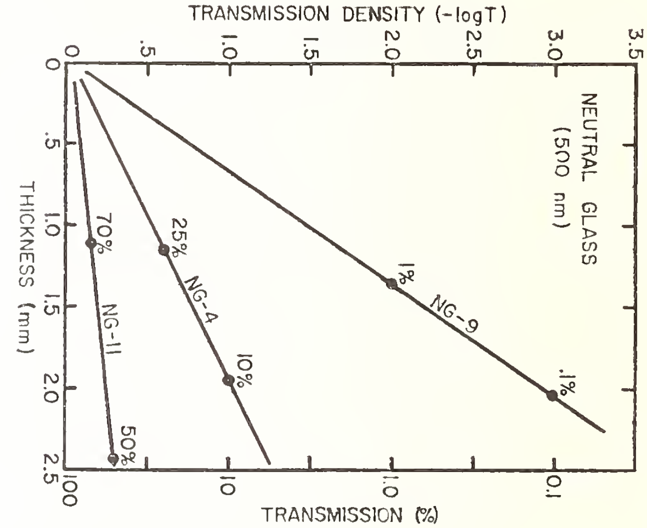

FIGURE 1. Transmission density versus thickness of Schott NG-4, NG-9, and NG-11 neutral glasses as derived from internal transmission data for 1 mm and 2 mm thicknesses.

3. Transmission Analysis

3.1 Light Scattering

Scattering of a collimated sample beam transmitted through the neutral filters was quantitatively assessed by the ASTM recommended method [3] for measuring haze percentage. The measurements were made on the reference hazemeter [4] using the ASTM recommended geometry and methods. This instrument has a well collimated circular incident beam having a color temperature of approximately 6800 K. A visual response filter at the detector modifies its response to give the hazemeter a peak sensitivity at approximately 550 nm. The instrument measures total forward scattering relative to the total transmission within the definition of this response and for the ASTM recommended geometry. The haze percentage determined for a set of filters was found to be as follows:

ERRATA

"Measurement Assurance Program Transmittance Standards for Spectrophotometric Linearity Testing: Preparation and Calibration"

by Kenneth L. Eckerle, Victor R. Weidner, Jack J. Hsia, and Karen Kafador

PAGE	COLUMN	LINE	NOW READS IN PART	SHOULD READ
20	2	Fig. 1	Rotated 90° clockwise	Should rotate 90° counterclockwise
24	1	Table 2	2,p 2,p 3,p 3,p	2,p 2,s 3,p 3,s
24	1 & 2	Table 3 Line 8 in Table 3	.9709	.09709
27	2	22	$\tau_w(\lambda_o) = \int_{\lambda_o-\omega}^{\lambda_o+\omega} h_w(\lambda) f(\lambda-\lambda_o) d\lambda / \int_{\lambda_o-\omega}^{\lambda_o-\omega} h_w(\lambda) d\lambda$	$\tau_w(\lambda_o) = \int_{\lambda_o-w}^{\lambda_o+w} h_w(\lambda) f(\lambda-\lambda_o) d\lambda / \int_{\lambda_o-w}^{\lambda_o+w} h_w(\lambda) d\lambda$
27	2	25	$\int_{\lambda_o-\omega}^{\lambda_o+\omega} h_w(\lambda) d\lambda = 1$	$\int_{\lambda_o-w}^{\lambda_o+w} h_w(\lambda) d\lambda = 1$
27	2	27	$\tau_w(\lambda_o) = \int_{\lambda_o-\omega}^{\lambda_o+\omega} h_w(\lambda) f(\lambda-\lambda_o) d\lambda$	$\tau_w(\lambda_o) = \int_{\lambda_o-w}^{\lambda_o+w} h_w(\lambda) f(\lambda-\lambda_o) d\lambda$
28	1	14	equal distances $\pm 2\delta$	equal distances $\pm\delta$, and at approximately $\pm 2\delta$
28	2	17	$\|\lambda-x_1\|\lambda-x_2\|$	$\|\lambda-x_1\| \cdot \|\lambda-x_2\|$
28	2	19	$1_o=$	$\lambda_o=$
29	2	6	$h_w(w+\lambda_o)$	$h_w(x+\lambda_o)$
29	1	13	$\hat{\Theta}'$	$\hat{\Theta}$

Filter	Haze Percentage	Nominal Transmittance
1-1	0.05%	92%
1-2	0.02%	70%
1-3	0.05%	50%
1-4	0.06%	25%
1-5	0.04%	10%
1-6	—*	1%
1-7	—*	0.1%

* The 1 and 0.1% transmitting filters were too low in transmittance for analysis by the hazemeter. It was concluded from these results that the filters scattered less than 0.1% of the transmitted sample beam.

3.2 Survey Transmittance Measurements

The purpose of a survey study of the neutral filter transmittance was to assess their over-all spectral properties in the visible spectrum and select a specific wavelength for detailed analysis and calibration. Since the filters are not perfectly neutral, it was desirable that a wavelength be selected for calibration, at which the transmittance values of the filters would be least sensitive to variation in passband and wavelength scale errors associated with various spectrophotometers.

With the exception of the borosilicate crown glass filter, the filters in the set are of a similar type glass with a common dye in three different concentrations for the NG-4, NG-9, and NG-11 glass types. The spectral transmission density curves of these filters in the spectral range between approximately 440 nm and 620 nm shows that the absorptions appear at the same wavelengths for the different concentrations associated with these glass types. Figures 2 through 8 illustrate the results of a 1-nm interval survey of the spectral transmittance of the filters between 440 and 620 nm. These measurements were made with a commercial spectrophotometer. The slit-width was maintained at approximately 0.1 mm (0.37 nm passband). Further measurements were made of the 10% filter with slit-widths of 1.0, 2.0, and 3.0 mm. These slit-widths correspond to passbands of 3.7, 7.4, and 11.1 nm, respectively.

There are four wavelengths within the wavelength range 440 to 620 nm corresponding to peaks and valleys that could be used for the purpose of establishing a calibrated set of photometric scale standards. However, only one wavelength is needed and the absorption peak at approximately 548.5 nm was selected as being the most suitable. The spectral peak at approximately 464 nm and the absorption valleys at approximately 510 and 591 nm do not show a significant change in transmittance for the range of passbands used in this survey. A photometric resolution of approximately ±0.01% is required in order to detect the effect of passband on the spectral transmittance measured at these wavelengths.

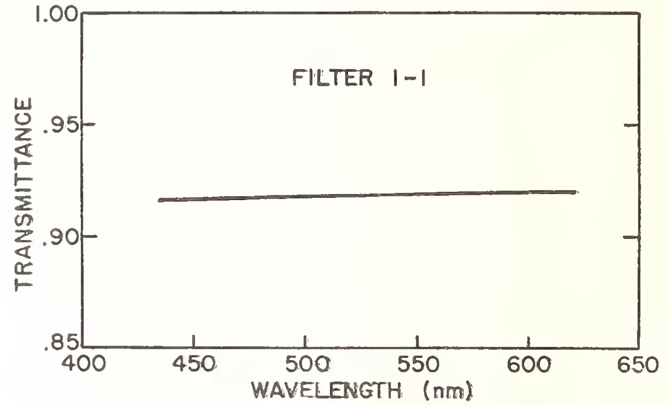

FIGURE 2. Spectral transmittance of Master Filter 1-1.

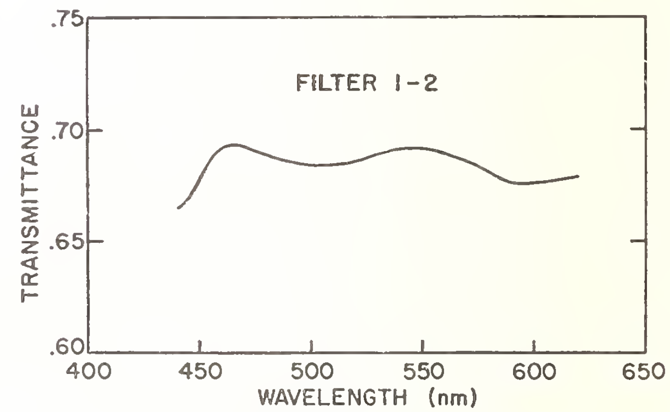

FIGURE 3. Spectral transmittance of Master Filter 1-2.

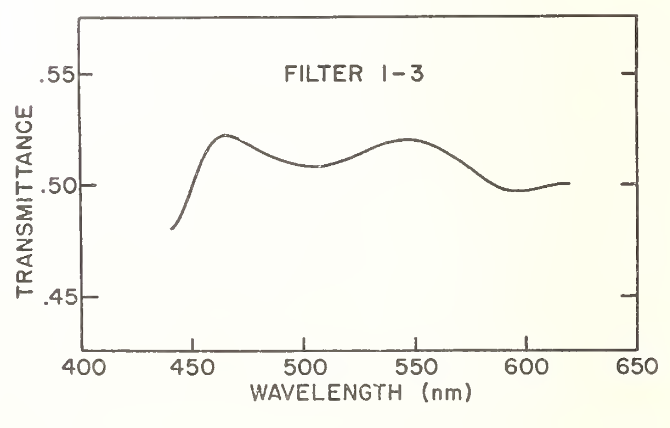

FIGURE 4. Spectral transmittance of Master Filter 1-3.

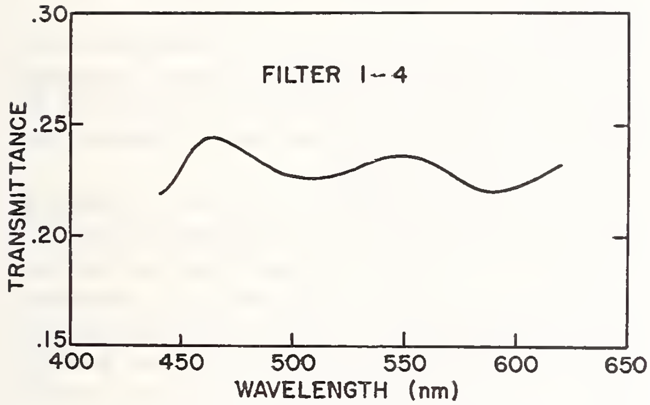

FIGURE 5. Spectral transmittance of Master Filter 1-4.

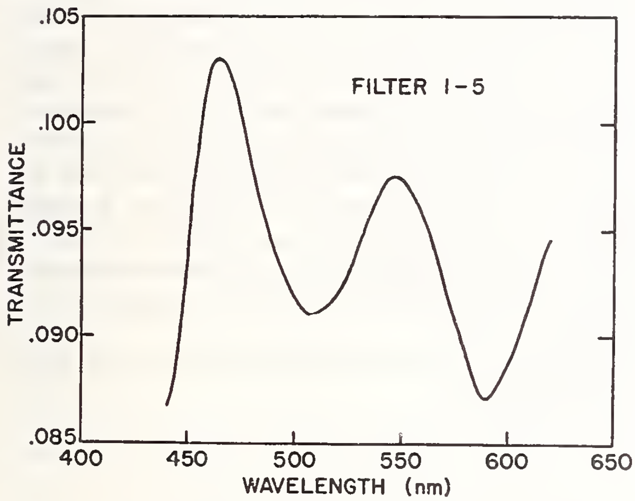

FIGURE 6. Spectral transmittance of Master Filter 1-5.

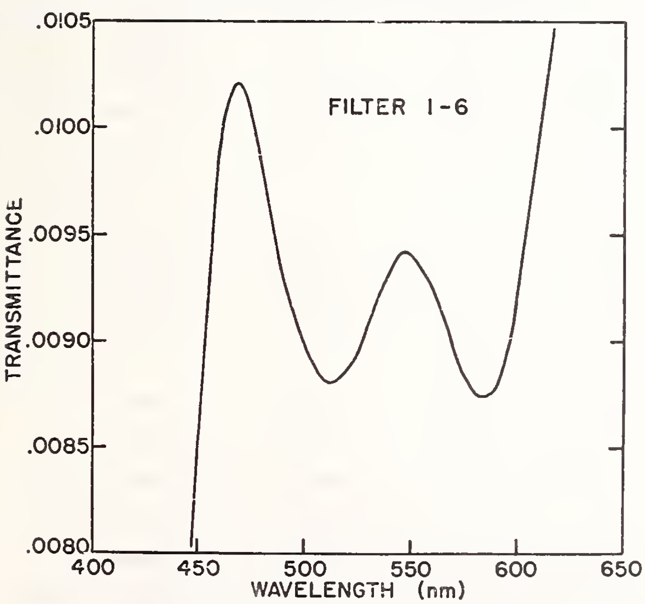

FIGURE 7. Spectral transmittance of Master Filter 1-6.

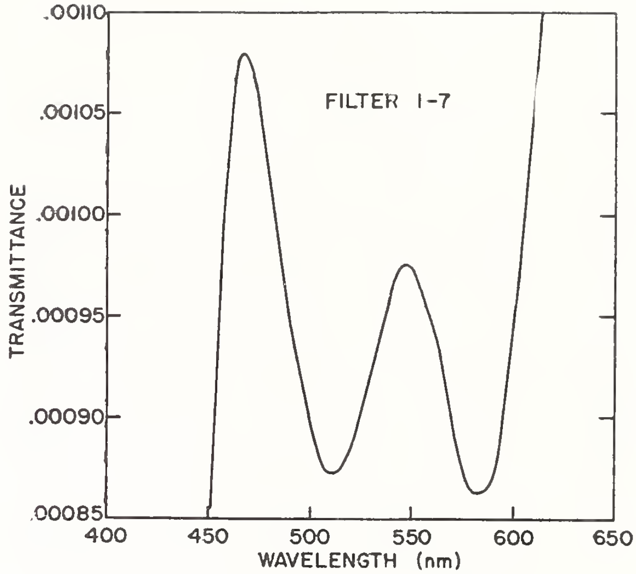

FIGURE 8. Spectral Transmittance of Master Filter 1-7.

3.3 High Accuracy Transmittance Measurements

Calibration of the spectral transmittance of the master set of filters and three other sets was done on the NBS Reference Spectrophotometer [5,6,7] for measuring high accuracy transmittance. The measurements were made with a passband of 1.5 nm and a beam diameter of 10 mm. Measurements were made for perpendicularly (s) and horizontally (p) polarized incident sample beams at 1.5 nm intervals from 545.5 to 557.5 nm. Each filter was scanned for uniformity at 547 nm. The uniformity measurements were made at three locations; center, 2 mm horizontally from center, and 2 mm below center. Transmittances for master set #1 filters are listed in table 1. The results of the uniformity scan at 547 nm are listed in table 2. The transmittance values listed in table 1 are an average of the two polarizations. The values for both polarizations are listed for the uniformity measurements made at 547 nm. The instrumental uncertainties in the values listed in tables 1 and 2 are estimated to be ±0.04% for filter 1-1 to ±0.0005% for filter 1-7. A complete description of the measurement sequence and data reduction for the high accuracy measurements and an analysis of the errors associated with such measurements is given in refs. [5-7].

4. Statistical Analysis

A statistical analysis of the data obtained on the spectral transmittance of the neutral filters was used to determine the magnitude of change in transmittance values due to changes in passband and also the magnitude of

TABLE 1. *Spectral Transmittance of Master Set No. 1 for a Passband of 1.5 nm.*

Wave-length (nm)	Filter 1-1	Filter 1-2	Filter 1-3	Filter 1-4	Filter 1-5	Filter 1-6	Filter 1-7
536.5	0.9164	0.6902	0.5177	0.2339	0.0955	0.00919	0.000915
538.0	.9165	.6905	.5181	.2345	.0959	.00922	.000922
539.5	.9165	.6908	.5185	.2350	.0962	.00926	.000925
541.0	.9165	.6910	.5187	.2353	.0964	.00928	.000929
542.5	.9166	.6912	.5190	.2357	.0966	.00931	.000933
544.0	.9166	.6913	.5192	.2360	.0969	.00932	.000935
545.5	.9166	.6913	.5192	.2362	.0970	.00933	.000936
547.0	.9166	.6914	.5194	.2363	.0971	.00934	.000938
548.5	.9168	.6913	.5193	.2363	.0971	.00934	.000938
550.0	.9167	.6912	.5191	.2363	.0971	.00934	.000940
551.5	.9167	.6912	.5190	.2362	.0971	.00933	.000937
553.0	.9167	.6911	.5190	.2361	.0970	.00931	.000934
554.5	.9166	.6908	.5187	.2358	.0968	.00930	.000934
556.0	.9168	.6908	.5184	.2355	.0966	.00928	.000929
557.5	.9168	.6904	.5180	.2351	.0964	.00924	.000923

TABLE 2. *Transmittance Uniformity of Master Set No. 1 at 547 nm.*

Location, Polarization	Filter 1-1	Filter 1-2	Filter 1-3	Filter 1-4	Filter 1-5	Filter 1-6	Filter 1-7
1, p	0.9166	0.6913	0.5193	0.2362	0.0971	0.00934	0.000939
1, s	.9166	.6913	.5193	.2362	.0971	.00935	.000940
2, p	.9166	.6914	.5193	.2361	.0971	.00934	.000937
2, p	.9167	.6914	.5193	.2362	.0971	.00934	.000938
3, p	.9164	.6913	.5192	.2362	.0971	.00934	.000938
3, p	.9165	.6913	.5192	.2362	.0971	.00933	.000939

change due to location of the sample beam (filter uniformity).

Preliminary data from the commercial instrument indicated that the central wavelength of interest was at approximately 547 nm. However, a further analysis of the data from the high accuracy instrument indicated that 548.5 nm was a better choice.

Data are presented in table 3 to illustrate the effect of passband on the transmittance at the central wavelength for the master set #1 filters. These values are based on calculation for a triangular passband, using continuous integration. Figures 9 through 15 show these results plotted with the standard error limits. The data listed in table 4 illustrate the magnitude of change due to position location for the master set of filters.

Remeasurement of the spectral transmittances of the neutral filters was made one year after initial calibrations. The results of this second calibration for master set #1 are compared with the original calibration and listed in table 5 for wavelength 548.5 nm. Differences in spectral transmittances of the filters shown in table 5 are too small to be clearly interpreted as changes with the possible exception of filter 1-1. Here the apparent change in transmittance was −0.0009. Further measurements at longer time intervals will be required to confirm any real changes in spectral transmittance due to aging.

Additional details of the statistical analysis are given in appendix A.

TABLE 3. *Transmittance of Master Set No. 1 at 548.5 nm as a Function of Passband for a Triangular Passband.*

Passband (nm)	Filter 1-1	Filter 1-2	Filter 1-3	Filter 1-4	Filter 1-5	Filter 1-6	Filter 1-7
1.5	0.91665	0.69133	0.51928	0.23632	0.09713	0.009342	0.000939
	(3)	(2)	(2)	(2)	(1)	(2)	(4)
3.0	.91665	.69132	.51927	.23632	.09711	.009341	.000939
	(3)	(2)	(2)	(2)	(1)	(2)	(4)
4.5	.91665	.69130	.51924	.23628	.9709	.009338	.000938
	(3)	(2)	(2)	(2)	(1)	(2)	(4)
6.0	.91665	.69128	.51921	.23624	.09706	.009335	.000938
	(3)	(2)	(2)	(2)	(1)	(2)	(4)
7.5	.91665	.69125	.51916	.23618	.09703	.009332	.000937
	(3)	(2)	(2)	(2)	(1)	(2)	(4)
9.0	.91665	.69121	.51911	.23616	.09698	.009327	.000937
	(3)	(2)	(2)	(2)	(1)	(2)	(4)
10.5	.91665	.69117	.51905	.23604	.09693	.009321	.000936
	(3)	(2)	(2)	(2)	(1)	(2)	(4)

Note: uncertainties (one standard deviation) are in parentheses

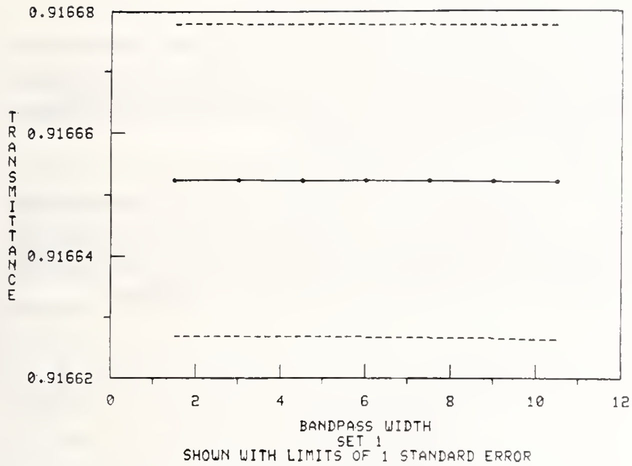

FIGURE 9. Spectral transmittance at 548.5 nm versus bandpass for Master Filter 1-1.

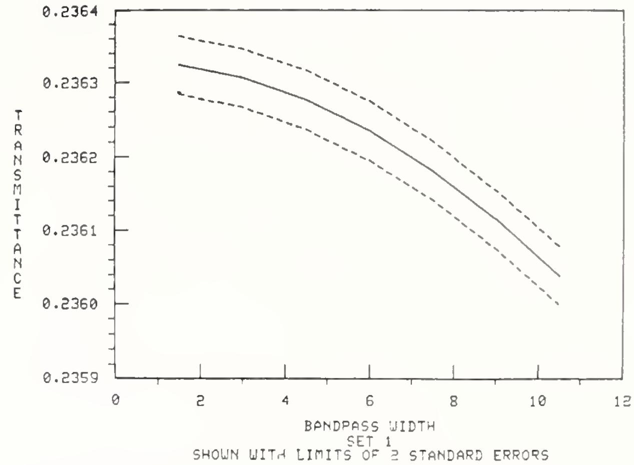

FIGURE 12. Spectral transmittance at 548.5 nm versus bandpass for Master Filter 1-4.

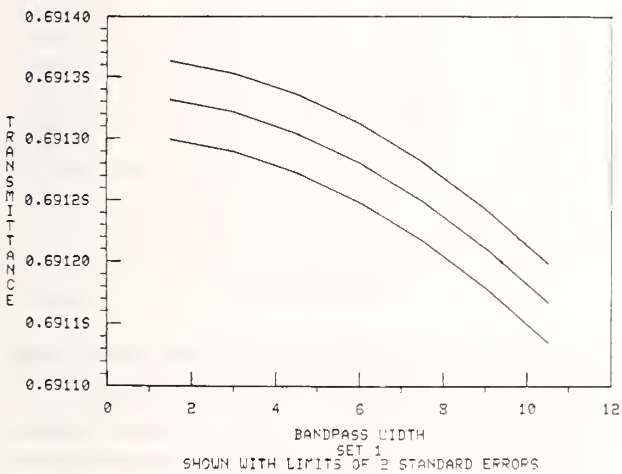

FIGURE 10. Spectral transmittance at 548.5 nm versus bandpass for Master Filter 1-2.

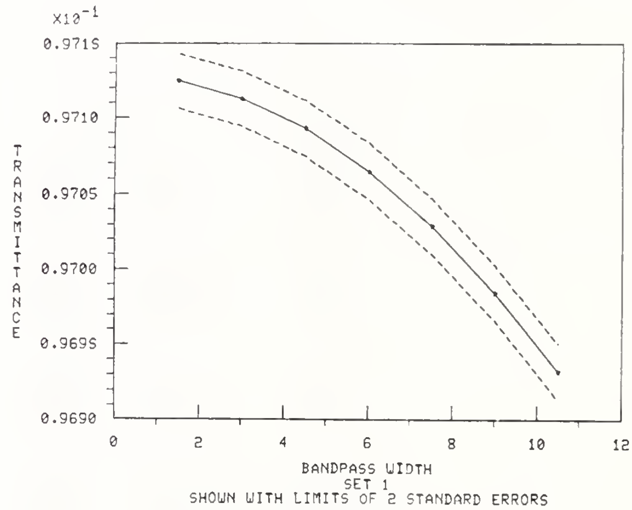

FIGURE 13. Spectral transmittance at 548.5 nm versus bandpass for Master Filter 1-5.

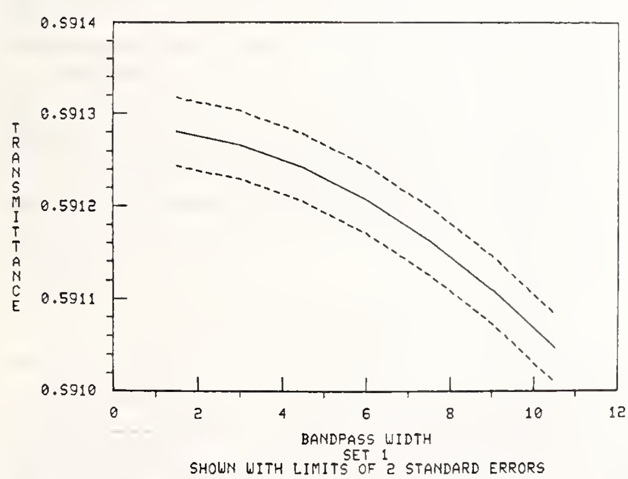

FIGURE 11. Spectral transmittance at 548.5 nm versus bandpass for Master Filter 1-3.

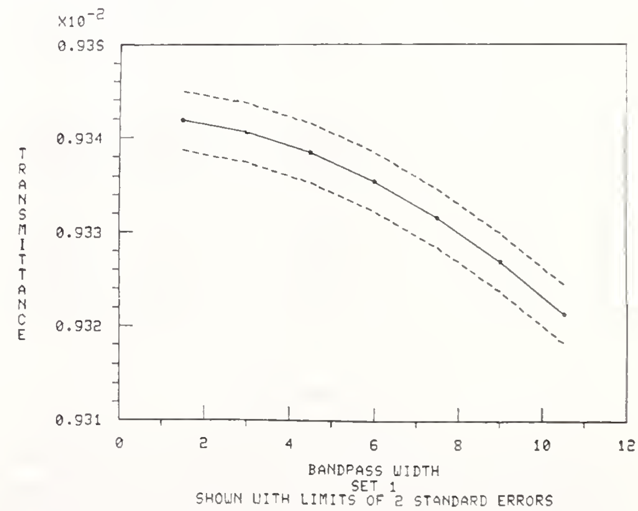

FIGURE 14. Spectral transmittance at 548.5 nm versus bandpass for Master Filter 1-6.

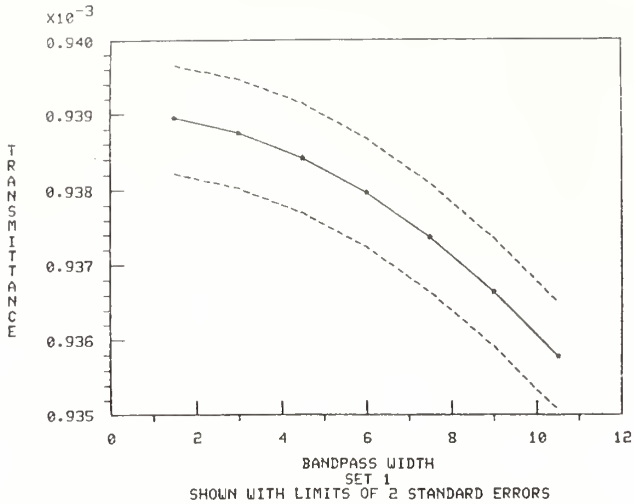

FIGURE 15. Spectral transmittance at 548.5 nm versus bandpass for Master Filter 1-7.

TABLE 4. *Magnitude of Filter Nonuniformity*

Filter No.	Location	Transmittance at 548.5 nm	Location Difference
1-1	1	0.91658	(1)-(2) = -0.00008
1-1	2	.91666	(2)-(3) = .00018
1-1	3	.91648	(1)-(3) = .00010
1-2	1	.69132	(1)-(2) = - .00006
1-2	2	.69138	(2)-(3) = .00007
1-2	3	.69131	(1)-(3) = .00001
1-3	1	.51926	(1)-(2) = - .00002
1-3	2	.51928	(2)-(3) = .00009
1-3	3	.51919	(1)-(3) = .00007
1-4	1	.23624	(1)-(2) = .00009
1-4	2	.23615	(2)-(3) = - .00004
1-4	3	.23619	(1)-(3) = .00005
1-5	1	.09709	(1)-(2) = .00001
1-5	2	.09708	(2)-(3) = .00000
1-5	3	.09708	(1)-(3) = .00001
1-6	1	.009341	(1)-(2) = - .000001
1-6	2	.009342	(2)-(3) = .000011
1-6	3	.009331	(1)-(3) = .000010
1-7	1	.000939	(1)-(2) = .000001
1-7	2	.000938	(2)-(3) = .000001
1-7	3	.000937	(1)-(3) = .000002

5. Summary

One master set and three working sets of neutral density glass filters have been calibrated for use as spectral transmittance standards for checking the photometric scale linearity of spectrophotometers. Each set consists of seven filters ranging in transmittance from 0.1 to 92%. These filters will be used in a Measurement Assurance Program (MAP). The purpose of this program will be to assist laboratories wishing to maintain a high level of confidence in the accuracy of their spectrophotometric measurements.

Data are presented that show the spectral transmittances of these filters at 548.5 nm to be relatively insensitive to variations in passband and slight errors in instrument wavelength setting. The filters are of sufficient uniformity for use as transmittance standards.

TABLE 5. *Spectral Transmittance of Master Set No. 1 for a Passband of 1.5 nm at 548.5 nm.*

Filter No.	(Apparent changes in transmittance) First Calibration (Oct. 1980)	Second Calibration (Oct. 1981)	Apparent change
1-1	0.9168	0.9159	-0.0009
1-2	.6913	.6914	.0001
1-3	.5193	.5196	.0003
1-4	.2363	.2365	.0002
1-5	.09712	.09722	.00010
1-6	.009337	.009350	.000013
1-7	.0009376	.0009395	.0000019

6. References

[1] Mielenz, K. D. and Eckerle, K. L. Spectrophotometer Linearity Testing Using the Double-Aperture Method, Appl. Opt. **11** (10), 2294 (Oct. 1972).

[2] Mavrodineanu, R. and Baldwin, J. R. Glass Filters as a Standard Reference Material for Spectrophotometer—Selection, Preparation, Certification, Use SRM 930. NBS Special Publication 260-51 (November 1975).

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[4] Weidner, V. R. and Hsia, J. J. NBS reference hazemeter: its development and testing. Appl. Opt. **18** (10) 1619 (May 15, 1979).

[5] Mielenz, K. D. and Eckerle, K. L. Design, Construction, and Testing of a New High Accuracy Spectrophotometer. Natl. Bur. Stand. (U.S.) Tech. Note 729; June 1972. 55 p.

[6] Eckerle, K. L. Modification of an NBS Reference Spectrophotometer. Natl. Bur. Stand. (U.S.) Tech. Note 913; July 1976, 39 p.

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Appendix

Statistical Design and Analysis for Calibrating the Transmittance of Filters for Spectrophotometer Linearity Testing

For four sets of filters, each set containing seven filter types of various transmittances, an assigned transmittance value needed to be determined when a triangular passband of given width is superimposed. Additionally, the polarization and location of the spot size on the filter had an unknown influence on the transmittance value. Although a commercial instrument can scan the entire spectrum at little cost, the measurements are an order of magnitude less accurate than those obtained using the high accuracy instrument. For this reason, a design was developed to determine:

(1) The values to be measured for purposes of calibration, and
(2) The existence of a possible effect due to polarity or the effect of location on the filter.

The motivation behind the choice of design is discussed in section A below. In the event of (2), the calibration must be reconsidered. On the basis of these extremely precise measurements, an effect due to location was statistically significant. This suggests that either:

(1) The calibrated values can be used only for the central location on each filter; or
(2) Additional measurements will be required to determine the magnitude of changes in transmittance due to spot location.

Since the observed differences are small (<0.2%; see table 4), the location effect may not interfere with practical usage of these filters. However, measurement as near to the central location on each filter as possible is recommended. The conclusions regarding location and polarity effects are presented in section B, and the estimation of the transmittance curve leading to the calibration values is shown in sections C and D. The method of extension for general passbands is given in section E.

A. The Design

Most organizations using these filters will have triangular passbands on their measuring equipment. Therefore, adequate determination of the transmittance using this passband is essential. However, other passband shapes may be used, and for these, a more general approach is taken. The problem then becomes one of estimating the transmittance curve with high precision. Since the measurements on the high accuracy instrument are time-consuming, a design was chosen so as to permit adequate curve estimation with relatively few points.

1. Preliminary analysis: Choice of Wavelength.

The choice for a wavelength to be calibrated required consideration of:

(i) Varying passband widths, and
(ii) The potential for imprecise wavelength specification.

We consider neighborhoods of 21 nanometers, since the widest passband that is most commonly used weights frequencies within a range of this length. The chosen wavelength is:

$$\lambda_0 = 548.5 \text{ nm}.$$

This subsection explains this choice in light of considerations (i) and (ii).

For a passband having width w and functional form $h_w(\lambda)$, the transmittance at wavelength λ_0 is given by the convolution integral

$$\tau_w(\lambda_0) = \int_{\lambda_0-\omega}^{\lambda_0+\omega} h_w(\lambda) f(\lambda-\lambda_0) d\lambda \Big/ \int_{\lambda_0-\omega}^{\lambda_0+\omega} h_w(\lambda) d\lambda,$$

where $f(\lambda)$ is the spectrum (transmittance curve). If the passband is normalized so that

$$\int_{\lambda_0-\omega}^{\lambda_0+\omega} h_w(\lambda) d\lambda = 1,$$

then this simplifies to

$$\tau_w(\lambda_0) = \int_{\lambda_0-\omega}^{\lambda_0+\omega} h_w(\lambda) f(\lambda-\lambda_0) d\lambda . \qquad (1)$$

In all that follows, we will assume that the passband h_w has been normalized in this way.

In order for this average value to be relatively insensitive to wavelength specification, $f(\lambda)$ needs to be a smooth, slowly-varying function. In mathematical terms, we search for a neighborhood where the first two derivatives of the spectrum are fairly small. This suggests a region where $f(\lambda)$ is nearly constant, or, at worst, a quadratic having very small degree of curvature. A locally linear spectrum with a noticeably nonlinear slope would yield a seriously biased estimate, whereas a locally quadratic spectrum, centered at a peak or trough, would have a negligible linear term and therefore a bias which depends primarily on the second-order term (hopefully small). Preliminary readouts on a commercial instrument suggested a central wavelength of 547.0 nm for most filters. On the basis of the first set of measurements on the high accuracy instrument, however, this was later amended to $\lambda_o = 548.5$ nm.

2. Number of Points

Assuming that the spectrum is locally quadratic and has a maximum around $\lambda_o = 548.5$ nm, our goal is to estimate the three parameters α, β, γ in the approximation:

$$f(\lambda) = \alpha + \beta\lambda + \gamma\lambda^2. \tag{2}$$

In order to estimate α, β, and γ, by $\hat{\alpha}$, $\hat{\beta}$, $\hat{\gamma}$, the proposed design specifies a total of *five* points, one at the central wavelength, and one each at equal distances $\pm 2\delta$ from the central wavelength.

Let $x = \lambda - 548.5$, so as to center the relation at the origin. A minimum of three points is required to specify the parameters. This leaves no room for the assessment of error. With more points, the variance of (1) will be a linear combination of the variances and covariances of the parameters, and will be dominated primarily by the variance of the constant term. If our chosen points are symmetrically placed about 0, it can be shown that:

$$\text{Var}\,(\hat{\alpha}) = \sigma^2/(n - c^2/e), \tag{3}$$

where

$$c = \Sigma x_i^2 = 2\sum_{x_i \geq 0} x_i^2$$

$$e = \Sigma x_i^4 = 2\sum_{x_i \geq 0} x_i^4.$$

As $(\Sigma x_i^4) \leqslant (\Sigma x_i^2)^2$ $(x_i \geqslant 0)$, the smallest value of c^2/e is 2 (all points at the origin except for the two at ± 1). A lower bound on (3) is therefore:

$$\text{Var}\,(\hat{\alpha}) = \sigma^2/(n-2). \tag{3$'$}$$

If we concern ourselves with only the first term, then, relative to σ^2, eq (3$'$) gives the reduction in the overall variance that we may hope to gain in our variance of the transmittance value. With $n = 3$, there is no reduction; with $n = 5$, the variance is already reduced to 33% of its value. An extra two points provides only an extra 13% improvement. Thus, the design specifies a minimum of two points on either side of the central wavelength.

3. Location of Points

We consider two criteria in selecting the four wavelengths (two symmetrically placed on either side of the central wavelength):

(i) Small error in quadratic interpolation of the transmittance curve;
(ii) Small variance in the transmittance estimate given by (1).

On the basis of these two criteria, the proposed design specifies measurements to be taken at:

$$\lambda_o, \lambda_o \pm 6 \text{ nm}, \lambda_o \pm 9 \text{ nm}.$$

The reasoning behind this choice is explained by considering (i) and (ii) separately.

(i) Error is quadratic interpolation. A bound for the estimate of error in polynominal interpolation of degree n at the points $a \leqslant x_1, x_2, \ldots x_{n+1} \leqslant b$ *is*

$$|\varepsilon(\lambda)| \leqslant \max_{a \leqslant \lambda \leqslant b} |f^{(n+1)}(\lambda)| \cdot |\lambda - x_1|\, \lambda - x_2| \cdots |\lambda - x_{n+1}|$$

(see, e.g., [8]). The first part of this error depends upon the transmittance curve and led to the choice of $l_0 = 548.5$ in subsection 1 above. Now we concentrate on selecting $x_1, \ldots, x_{n+1}$ so that the error is as small as possible. The answer to this problem is given by the zeroes of the Chebyshev polynomials, namely

$$x_k = (10.5\text{nm}) \cdot \cos\,[(2k-1)/(2n+2)],\; k = 1, \ldots, n+1. \tag{4}$$

For the problem at hand, $n+1 = 5$; thus:

$$x_1 = -x_5 = 9.986 \text{ nm from } \lambda_o$$
$$x_2 = -x_4 = 6.172 \text{ nm from } \lambda_o$$
$$x_3 = 0 \text{ nm from } \lambda_o\,.$$

As most passbands are in multiples of 1.5 nm, the closest multiples to these points are ± 6 nm and either ± 9 nm or ± 10.5 nm. However, the nature of the triangular passband which is frequently used in practice would assign zero weight to the values ± 10.5 nm. Since a primary goal is the estimation of the transmittance using this passband, we choose ± 9 nm.

(ii) Minimum variance of transmittance estimate. If we estimate the transmittance curve using a quadratic function, viz.

$$\hat{f}(x) = \hat{\alpha} + \hat{\beta}x + \hat{\gamma}x^2, \tag{5}$$

then the transmittance using passband of width w is

$$\hat{\tau}_w(\lambda_o) = \int_{-w}^{w} h_w(w+\lambda_o)\,(\hat{\alpha} + \hat{\beta}x + \hat{\gamma}x^2)dx.$$

Let

$$c_j = \int_{-w}^{w} x^j\, h_w(x)\, dx$$

Then

$$\hat{\tau}_w(\lambda_o) = \hat{\alpha}c_o + \hat{\beta}c_1 + \hat{\gamma}c_2 = \mathbf{c}'\hat{\Theta}$$

where

$$\mathbf{c}' = (c_o, c_1, c_2)$$

$$\hat{\Theta}' = (\hat{\alpha}, \hat{\beta}, \hat{\gamma}).$$

Then minimizing the variance of $\mathbf{c}'\hat{\Theta}$ is equivalent to

$$\min \mathrm{Var}(\mathbf{c}'\hat{\Theta}) = \min \mathbf{c}'\ \mathrm{Var}(\hat{\Theta})\ \mathbf{c}.$$

A design which minimizes the variance-covariance matrix $\mathrm{Var}(\hat{\Theta})$ of the parameter estimates is given in [9]. The design would place the two observations each at $\lambda_o + 10.5$ nm in addition to the one at λ_o. If our function is truly quadratic, such a design would be optimal.

For many reasons, however, we modify the optimal design which permits more flexibility in our choice of model. Such a design is suggested in [10]. The design recommends two different values rather than repeating them at the endpoints.

Closely related to this design is the one which minimizes the maximum variance of the best linear unbiased estimate of the function *f(x)* given by eq (5). Reference [11] shows that the five points should be placed at the zeros of the polynomial

$$10.5\,(1 - x^2)\,P_4{}'(x)\ ,$$

where $P_4{}'$ is the derivative of the fourth degree Legendre polynomial

$$P_4{}'(x) = 17.5x^3 - 7.5x\ .$$

Hence, the five points are 0, ± 7.1, ± 10.5 nm from λ_o. Again, since our passband applies decreasing weight to *f(x)* as x is further from the origin, we choose to make these points in towards 0, to ± 6 and ± 9 nm.

4. Tests for Polarity and Spot Location

Additional measurements at the central wavelength are needed to provide tests for difference in polarization and spot location. In addition to the measurement at the central wavelength with polarity 1 (point 1 at location (0,0)), measurements were taken at

Point 2: Spot location (2,0), Polarity 1
Point 3: Spot location (0,2), Polarity 2
Point 4: Spot location (0,2), Polarity 1.

This permits a check for a difference due to polarity by comparing the third and fourth points, and check for location differences by comparing points 1 and 4 (or 1 and 2 or 2 and 4). A more extensive check based on a full 2×3 factorial combination was made on the master set #1.

B. Results: Polarity and Location

The reported results are based on measurements taken on four sets of filters. A complete series of 15 measurements across the 21-nm range at 1.5 nm spacing was taken on the master set, as a check for the adequacy of the five-point design used on the other three sets. In addition, polarity and location were tested on each filter in all sets. The results of these tests are reported in this section; transmittance calculations are reported in section C.

For set 1, six measurements were taken at $\lambda = 547.0$, at both polarity 1 and 2 at the center of the filter (0,0) and at placements of two units to the right (2,0) and above (0,2) the center. This permits a 3×2 factorial analysis for detecting differences due to location and polarity.

For sets 2, 3, and 4, *t*-tests on location and polarity were calculated. The test on location was deemed significant if the comparison of either (0,0) and (0,2) or (0,2) and (2,0) indicated a chance of less than 1% under the hypothesis of no effect.

None of the tests of polarity was significant at the 0.01 level. (One filter from one set gave significance of 0.025. Out of 28 such tests, the chance of obtaining one or more spurious significances is more than 50%, so this is hardly surprising.) For location, however, the following sets showed a significant difference (level given in parentheses):

Filter	Set (level of significance)
2	3 (0.0004)
3	2 (0.013), 3 (0.002), 4(<0.0001)
4	2 (0.0013), 3 (0.0003), 4 (0.01)
5	3 (0.01), 4 (0.0003)
6	1 (0.006), 4 (0.0013) [3 (0.02)]
7	1 (0.0002), 4 (0.0025)

Out of 28 tests of significance, the chance of 13 or more coming up significant at the 0.01 level when in fact location is irrelevant is

$$\sum_{k=13}^{28} \binom{28}{k} (.01)^k (.99)^{n-k} \cong 0 \quad ,$$

and the chance that at least 2 of the 4 sets would show significance on a given filter type is

$$\sum_{k=2}^{4} \binom{4}{k} (0.01)^k (0.99)^{n-k} = 0.0006.$$

For 7 types of filters, the overall level of significance is approximately 0.004.

These tests of significance suggest that it is highly unlikely that location on the filter in measuring transmittance is irrelevant. However, all tests were based on the internal standard errors of the four measurements made within the twenty-minute measurement period. This standard error reflects only the error of the four internal readings but does not reflect the measurement-to-measurement variation caused by apparatus set-up, filter placement in the wheel or polarity switches. As such, the actual error in taking successive measurements may in fact be larger than the reported standard error. However, measurements on these filters as near to the central location as possible is recommended. The transmittance values using triangular passbands have been calculated assuming location is irrelevant (thereby using *all* data in the estimation of the transmittance curve) and assuming location has a significant effect on the transmittance value (using only the data at the center (0,0) spot). Only the latter are given for the master Set #1 in table 3 of the main report.

C. Estimation of Transmittance Curve

As there are four sets which contain all filter types, it will be helpful to use all filters having the same nominal transmittance in estimating the transmittance curve. We fit quadratic functions of the form in eq (5).

Consider one filter type from each set having a nominal transmittance (say, 69%). The average value of transmittance, given by the parameter α, is likely to be specific to each filter in the different sets. However, the curvature parameters β and γ are likely to be common to all 4 filters having a given nominal transmittance. Thus we use all these observations to fit a relation of the form

$$f(x_{ij}) = \alpha_j + \beta \cdot x_{ij} + \gamma \cdot x_{ij}^2 \, , \tag{7}$$

$i = 1, \ldots, n_j$ (= # of measurements in jth set)
$j = 1, \ldots, 4$ (= # of sets).

Note that

$$n_1 = 21,$$
$$\{x_{i1}\} = \{-12., -10.5, \ldots, 7.5, 9, -1.5, \ldots, -1.5\} \, ,$$

since set 1 has the full 15 measurements and 6 additional measurements for polarity and location, and

$$n_j = 8, \{x_{ij}\} = \{-9, -6, 0, 6, 9, 0, 0, 0\}$$

for sets 2, 3 and 4. Note also that the average level, α_j, is specific to the filter in the set, as the level may reflect the amount of dye that is contained in the filter. However, the parameters β and γ are likely to be influenced by the properties of the dye contained in this glass and are thus common to the filters with the same nominal transmittance from all four sets. This joint estimation permits the more accurate calculation of β and γ in sets 2, 3, and 4, where the number of observations is only eight.

Since transmittance may depend on location (section B), we also estimate α, β, γ, for each of the 7 filter types, using only those observations at location (0,0). In this case,

$$n_1 = 17, \{x_{i1}\} = \{-12, -10.5 \ldots, 7.5, 9, -1.5, -1.5\}$$
$$n_j = 5, \{x_{ij}\} = \{-9, -6, 0, 6, 9\}, j = 2, 3, 4.$$

These parameters were not found to differ from those using all the data by more than two standard errors. In light of potential location differences, however, table A1 provides these estimates of the parameters α, β and γ for the master set #1 filters.

D. Estimation of Transmittance Values Using Triangular Passband

As indicated in eq (1), when the transmittance curve is superimposed with a normalized passband centered at λ_o, the observed transmittance can be expressed as a convolution integral. Using a triangular passband of width

w, symmetric about λ_o, the result is a weighted average of all transmittances around the central wavelength. This weighted average may be computed either as a discrete sum:

$$\hat{\tau}_w(\lambda_o) = \Sigma\, h_w(\lambda_i)y_i \qquad y_i = \text{transmittance at wavelength } \lambda_i;$$
$$= \Sigma\, h_w(x_i+\lambda_o)y_i, \quad x_i = \lambda_i - \lambda_o, \tag{8}$$

where

$$h_w(x) = w^{-1}[1 - \text{sign}(x)\cdot x/w], \; -w \leqslant x \leqslant w, \tag{9}$$

or as an integral as in eq (1). The first approach, based on eq (8), is a discrete computation based on the observed values y_i. The second approach, eq (1), uses the values y_i to estimate $f(x)$ (eq (5)) and integrates directly. For sets 2, 3, and 4 where data inside ±6 nm exists only at the central wavelength, eq (1) is clearly the method of choice. Furthermore, the estimation of $f(x)$ uses all of the data in its estimation (section C), and is therefore likely to remove much of the variability in the values y_i.

The standard error of $\hat{\tau}_w$ can be computed using the reported standard errors (s_i) associated with each measurement:

$$SE\,(\hat{\tau}_w) = \Sigma[h_w(x_i)]^2\, s_i^2.$$

Using the triangular passband of width w (eq (9)) and the fitted relation (7), we have that the transmittance of a filter from the jth set is

$$\hat{\tau}_w(\lambda_o) = \int_{\lambda_o-w}^{\lambda_o+w} h_w(\lambda)\cdot[\hat{\alpha}_j + \hat{\beta}(\lambda-\lambda_o) + \hat{\gamma}(\lambda-\lambda_o)]^2\, d\lambda$$
$$= \hat{\alpha}_j + (w^2/6)\cdot\hat{\gamma} \tag{10}$$

for which

$$SE(\hat{\tau}_w) = [\text{Var}(\hat{\alpha}_j) + (w^4/36)\text{Var}(\hat{\gamma}) + (w^2/3)\text{Cov}(\hat{\alpha}_j,\hat{\gamma})]^{1/2}$$
$$\cong SE\,(\hat{\alpha}_j);$$

since the variance of the curvature parameter $\hat{\gamma}$ and the covariance term are typically two orders of magnitude smaller than the variance of the fitted constant term.

TABLE A1. *Coefficients for Estimation of Transmittance Curve for Set #1 (includes data from center location only).*

$f(x) = \hat{\alpha} + \hat{\beta}x + \hat{\gamma}x^2 \quad x = \text{wavelength} - 548.5$

Table gives constant term; all standard errors given in parentheses

	$\hat{\alpha}$	$\hat{\beta} \times 10^5$	$\hat{\gamma} \times 10^5$
Filter 1	.9166525	1.6493	−0.002562
	(200)	(.1877)	(.032669)
2	.6913352	−2.0067	−0.91372
	(117)	(1.5030)	(.02616)
3	.5192856	−2.7205	−1.2973
	(135)	(.1151)	(.0200)
4	.2363304	+1.1966	−1.5858
	(147)	(.0923)	(.0161)
5	.0971290	+0.8657	−1.0699
	(068)	(.0736)	(.0128)
6	.00934226	−.05959	−0.11355
	(115)	(.00979)	(.00170)
7	.000939014	−0.008245	−0.017568
	(266)	(.002893)	(.000504)

E. Other Passbands

Table 3 of the main report gives transmittances for the master set #1 filters for a triangular passband. For passbands other than those listed in table 3, the transmittance may be calculated directly via eq (10), using the estimated coefficients $\hat{\alpha}_1$, $\hat{\beta}$, and $\hat{\gamma}$ listed in table A1.

FORM NBS-443
(REV. 12-65)

APPENDIX B

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

~~WASHINGTON, D.C. 20234~~

Gaithersburg, MD 20899

REPORT OF CALIBRATION

for
Pilot Transmittance MAP
with Participant:
Laboratory A.

(Please see your purchase order number xyz dated July 23, 1984.)

1. Purpose

The purpose of this test is to determine the spectrophotometric accuracy of Participant's instrumentation via a MAP package.

2. Material

The MAP package consists of a set of seven neutral filters (Set 4) and a didymium filter (D-41) of type SRM 2010.

3. Measurements

The measurements on the neutral filters were made by NBS, then by Participant, and again by NBS. The didymium wavelength values are known not to change and were only measured by Participant. The instrument used by NBS was a high accuracy reference spectrophotometer. The instruments used by Participant were 6 commercial instruments identified as Instruments 1 through 6.

4. Results

A. Transmittance

The results for the MAP procedure are listed in Tables 1 through 4 for the indicated instruments. The symbols and quantities in these tables are defined as follows:

τ_x^B - Transmittance measured by NBS before (B) the participating laboratory at bandpass denoted by x.

τ_x^p Transmittance measured by the participating laboratory at the bandpass x.

τ_x^A Transmittance measured by NBS after (A) the participating laboratory at bandpass denoted by x.

REPORT OF CALIBRATION
Pilot Transmittance MAP
with Participant: Laboratory A

$\tau_x^{AVE} = (\tau_x^B + \tau_x^A)/2$ The average of the before and after measurement.

$(\tau_x^B - \tau_x^A)/2$ Denotes the error due to a possible change in the filter between the before and after measurements at bandpass x.

U The uncertainty in the average value τ^{AVE} accounting for systematic and random errors.

$|(\tau_x^B - \tau_x^A)/2|+U$ An upper bound combining the NBS uncertainty and possible change in the filter.

$\tau_x^{AVE} - \tau_x^p$ The difference between the average NBS value and the value of Participant at bandpass denoted by x.

If $|\tau^{AVE} - \tau^p| > |(\tau_x^B - \tau_x^A)/2|+U$, then there is a bias in the measurements due to a systematic error since the Participant stated the random error as zero. In comparing these two quantities for the different instruments and for each filter, we do not see any serious discrepancies. However, in most cases, there exists a bias. The method for obtaining the standard values for NBS are described in one of the enclosed publications.

B. Wavelength Calibration

Table 5 contains the measured deviations from the standard values for the transmittance minima and the points of inflection for SRM type 2010. (A sample certificate is enclosed.) The wavelengths of the minimum transmittances of the absorption bands are designated λ_{MIN}, and the wavelengths of the inflection points as λ_{INF}. A curve may be fitted to these data and an additive correction obtained. However, we attempted to go one step further, and accounted for systematic errors in bandwidth and transmittance using the method described in Section 2.3.3 of Special Publication 260-66 (A copy is enclosed.). Some of the experimental data have a relatively large random component as evidenced by the large standard deviations of the coefficients. The corrected data are shown in Figures B1 through B5 for the indicated instruments. The final figures B1 through B5 are not plots of the data shown in Appendix B - Table 5. The plots are derived from the data evaluation according to Equation 8 page 30 in reference [5] using the error terms and the corresponding bandwidth and transmittance corrections. We had some difficulty in estimating systematic errors for Instrument 1 supporting the view of Participant that this instrument needs recalibration. Also, there is one outlier which we cannot explain. The additive corrections indicated in the figures are the "best values" based on the information we have.

REPORT OF CALIBRATION
Pilot Transmittance MAP
with Participant: Laboratory A

5. Comments

A water mark was detected on filter 4-1. It is unknown when this spot, which decreased the transmittance, was acquired.

For the Director,

Radiometric Physics Division
Center for Radiation Research

Enclosures:

1 Report of Test
6 Tables
5 Figures

APPENDIX B
Table 1

Transmittance Results for the Indicated Instruments

Filter	$\tau_{10.0}^{B}$ (NBS)	τ^{P} Instrument 1	τ^{P} Instrument 2	$\tau_{10.0}^{A}$ (NBS)	τ^{AVE}
4-1	0.91615	0.9179	0.919	0.91474	0.91545
4-2	0.69058	0.6907	0.691	0.69014	0.69036
4-3	0.51947	0.5191	0.520	0.51889	0.51918
4-4	0.23528	0.2356	0.236	0.23559	0.23544
4-5	0.096580	0.0967	0.097	0.096404	0.096492
4-6	0.0092677	0.0095	0.0095	0.0092576	0.0092627
4-7	0.0009405	0.0011	0.0015	0.0009353	0.0009379

	$(\tau_{10}^{B}-\tau_{10}^{A})/2$	U	$\left|(\tau_{10}^{B}-\tau_{10}^{A})/2\right|+U$	$\tau^{AVE}-\tau^{P}$ Instrument 1	$\tau^{AVE}-\tau^{P}$ Instrument 2
4-1	0.00071	0.00062	0.00133	-0.00246	-0.00356
4-2	0.00022	0.00043	0.00065	-0.00034	-0.00064
4-3	0.00029	0.00036	0.00065	0.00008	-0.00082
4-4	-0.00016	0.00016	0.00032	-0.00017	-0.00057
4-5	0.000088	0.000135	0.000223	-0.000208	-0.000510
4-6	0.0000051	0.0000282	0.0000333	-0.0002374	-0.0002400
4-7	0.0000026	0.0000127	0.0000154	-0.0001620	-0.0005600

APPENDIX B
Table 2

Transmittance Results for the Indicated Instruments

Filter	$\tau^{B}_{8.0}$ (NBS)	τ^{p} Instrument 3	τ^{p} Instrument 4	$\tau^{A}_{8.0}$ (NBS)	τ^{AVE}
4-1	0.91615	0.920	0.919	0.91474	0.91545
4-2	0.69064	0.692	0.693	0.69020	0.69042
4-3	0.51955	0.522	0.522	0.51897	0.51926
4-4	0.23538	0.239	0.236	0.23569	0.23554
4-5	0.096632	0.097	0.096	0.096456	0.096544
4-6	0.0092747	0.0089	0.0088	0.0092646	0.009270
4-7	0.0009416	0.0009	0.0008	0.0009364	0.000939

Filter	$(\tau^{B}_{8.0}-\tau^{A}_{8.0})/2$	U	$\left\|(\tau^{B}_{8.0}-\tau^{A}_{8.0})/2\right\|+U$	$\tau^{AVE}-\tau^{p}$ Instrument 3	$\tau^{AVE}-\tau^{p}$ Instrument 4
4-1	0.00071	0.00062	0.00133	-0.00456	-0.00356
4-2	0.00022	0.00043	0.00065	-0.00158	-0.00258
4-3	0.00029	0.00036	0.00065	-0.00274	-0.00274
4-4	-0.00016	0.00016	0.00032	-0.00346	-0.00047
4-5	0.000088	0.000135	0.000223	-0.004560	0.000544
4-6	0.0000051	0.0000282	0.00003327	0.00036960	0.00047000
4-7	0.0000026	0.0000128	0.00001538	0.00003900	0.00013900

APPENDIX B
Table 3

Transmittance Results for the Indicated Instrument

Filter	$\tau^{B}_{2.0}$ (NBS)	τ^{P} Instrument 5	$\tau^{A}_{2.0}$ (NBS)	τ^{AVE}
4-1	0.91615	0.919	0.91474	0.91545
4-2	0.69073	0.690	0.69029	0.69051
4-3	0.51969	0.515	0.51911	0.51940
4-4	0.23554	0.236	0.23585	0.23570
4-5	0.096734	0.096	0.096558	0.096646
4-6	0.0092867	0.0088	0.0092766	0.0092816
4-7	0.0009436	0.0006	0.0009382	0.0009409

	$(\tau^{B}_{2.0} - \tau^{A}_{2.0})/2$	U	$\left\|(\tau^{B}_{2.0} - \tau^{A}_{2.0})/2\right\| + U$	$\tau^{AVE} - \tau^{P}$ Instrument 5
4-1	0.00071	0.00062	0.00133	-0.00356
4-2	0.00022	0.00043	0.00065	0.00051
4-3	0.00029	0.00036	0.00065	0.00440
4-4	-0.00016	0.00016	0.00032	-0.00031
4-5	0.000088	0.00014	0.00023	-0.00065
4-6	0.0000051	0.000028	0.0000333	0.0004810
4-7	0.0000027	0.000013	0.0000155	0.0003410

APPENDIX B

Table 4

Transmittance Results for the Indicated Instrument

Filter	$\tau^{B}_{0.1}$ (NBS)	$\tau^{p}_{\text{Instrument } 6}$	$\tau^{A}_{0.1}$ (NBS)	τ^{AVE}	$(\tau^{B}_{0.1}-\tau^{A}_{0.1})/2$
4-1	0.91615	0.919	0.91474	0.91545	0.00071
4-2	0.69073	0.694	0.69030	0.690525	0.00022
4-3	0.51970	0.524	0.51913	0.51942	0.00029
4-4	0.23556	0.239	0.23587	0.23572	-0.00016
4-5	0.096737	0.096	0.096561	0.096649	0.000088

Filter	$\tau^{B}_{2.0}$ (NBS)	$\tau^{p}_{\text{Instrument } 6}$	$\tau^{A}_{2.0}$ (NBS)	τ^{AVE}	$(\tau^{B}_{2.0}-\tau^{A}_{2.0})/2$
4-6	0.0092867	0.0093	0.0092766	0.0092817	0.0000051
4-7	0.0009436	0.0008	0.0009382	0.0009409	0.0000027

Filter	U	$\left\lvert\frac{\tau^{B}_{0.1}-\tau^{A}_{0.1}}{2}\right\rvert+U$	$\tau^{AVE}-\tau^{p}_{\text{Instrument } 6}$
4-1	0.00062	0.00133	-0.00356
4-2	0.00043	0.00064	-0.00349
4-3	0.00036	0.00065	-0.00459
4-4	0.00016	0.00032	-0.00329
4-5	0.000135	0.000223	0.000649

Filter	U	$\left\lvert\frac{\tau^{B}_{2.0}-\tau^{A}_{2.0}}{2}\right\rvert+U$	$\tau^{AVE}-\tau^{p}_{\text{Instrument } 6}$
4-6	0.0000282	0.0000333	-0.0000184
4-7	0.0000128	0.0000155	0.0001409

APPENDIX B

Table 5

Measurements by participant of λ_{MIN} and λ_{INF} with difference between NBS and the participating laboratory, $\Delta\lambda = \lambda_{NBS} - \lambda_{PARTICIPANT}$. All are expressed in nanometers.

Nominal	Instrument 1		Instrument 2		Instrument 3		Instrument 4		Instrument 5	
λ_{MIN}	λ_{MIN}	$\Delta\lambda_{MIN}$	λ_{MIN}	$\Delta\lambda_{MIN}$	λ_{MIN}	$\Delta\lambda_{MIN}$	λ_{MIN}	$\Delta\lambda_{MIN}$	λ_{MIN}	$\Delta\lambda_{MIN}$
440	439.9	1.65	441.1	0.45	441.5	0.77	441.8	0.47	445.7	-0.26
478	474.7	1.23	475.7	0.23	476.3	0.77	476.6	0.47	478.9	0.14
529	527.5	1.46	528.6	0.36	529.1	0.12	529.1	0.12	529.6	0.13
585	583.5	1.23	584.9	-0.17	585.3	0.48	586.0	-0.22	585.6	-0.19
684	685.2	-0.59	684.5	0.11	684.0	0.69	684.0	0.69	684.8	-0.13
740	--	--	742.9	0.52	--	--	--	--	740.1	-0.21
Nominal										
λ_{INF}	λ_{INF}	$\Delta\lambda_{INF}$	λ_{INF}	$\Delta\lambda_{INF}$	λ_{INF}	$\Delta\lambda_{INF}$	λ_{INF}	$\Delta\lambda_{INF}$	λ_{INF}	$\Delta\lambda_{INF}$
406	404.4	2.04	--	--	405.6	0.84	405.6	0.84	--	--
429	427.6	1.83	--	--	429.3	0.13	429.3	0.13	--	--
450	448.6	0.89	--	--	449.1	0.39	449.5	-0.01	--	--
485	483.1	1.74	--	--	484.9	-0.06	484.9	-0.06	--	--
537	534.7	1.80	--	--	536.3	0.20	536.8	-0.30	--	--
568	566.0	2.15	--	--	567.6	0.55	568.2	-0.05	--	--
600	597.1	1.95	--	--	598.7	0.35	599.6	-0.55	--	--
733	--	--	--	--	--	--	--	--	--	--
756	--	--	--	--	--	--	--	--	--	--

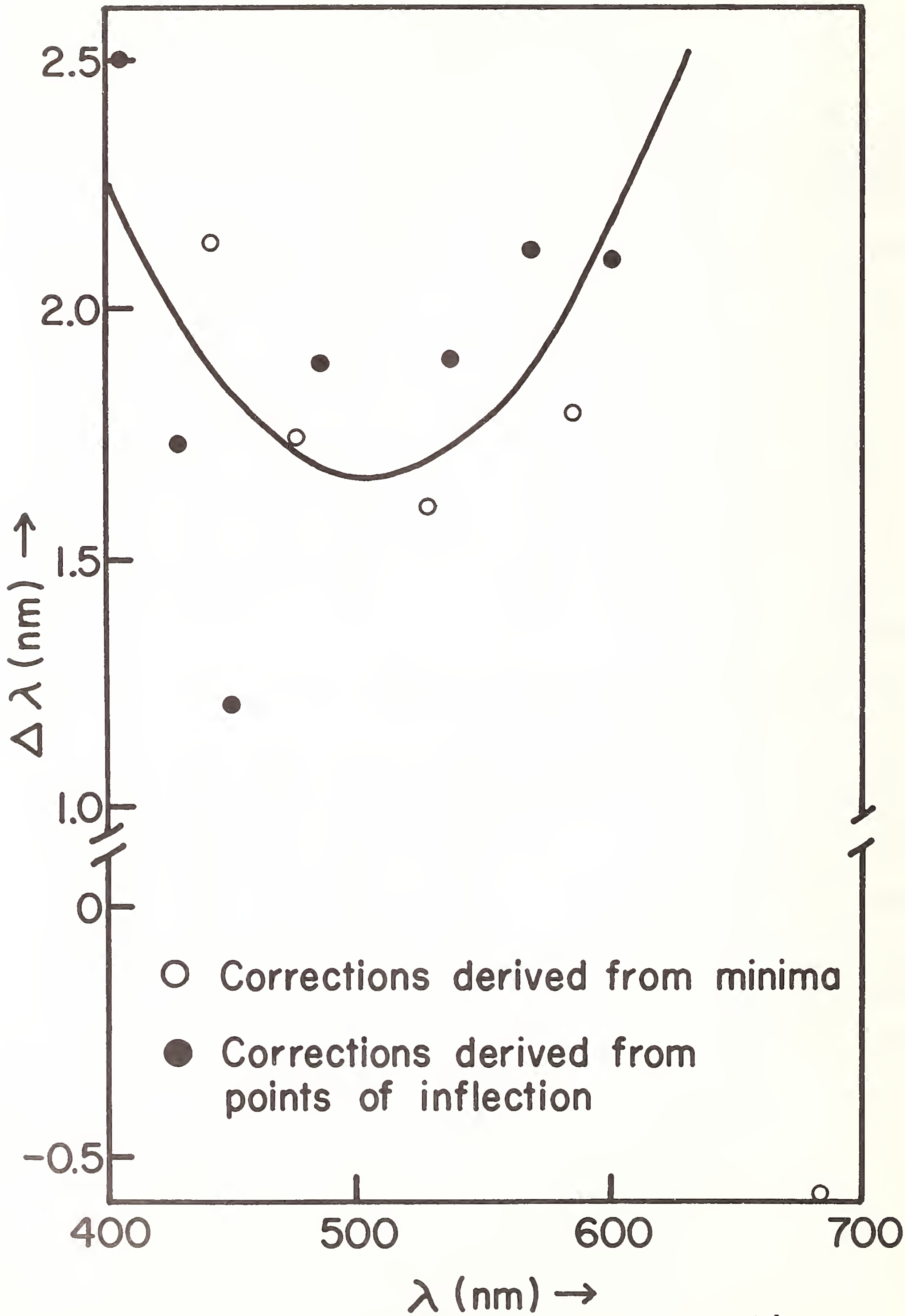

Figure B1. Wavelength calibration curve for Instrument 1

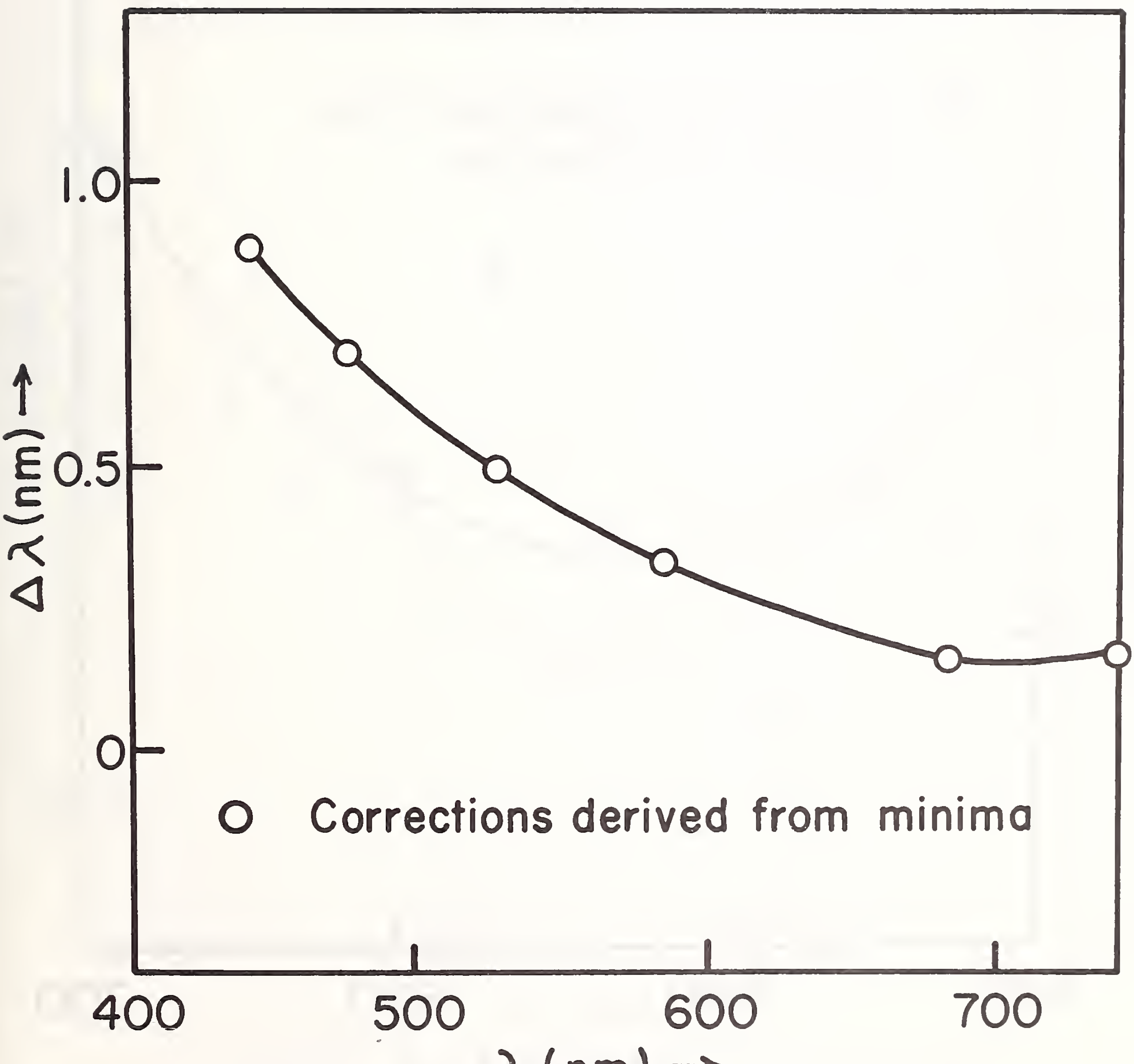

Figure B2. Wavelength calibration curve for Instrument 2

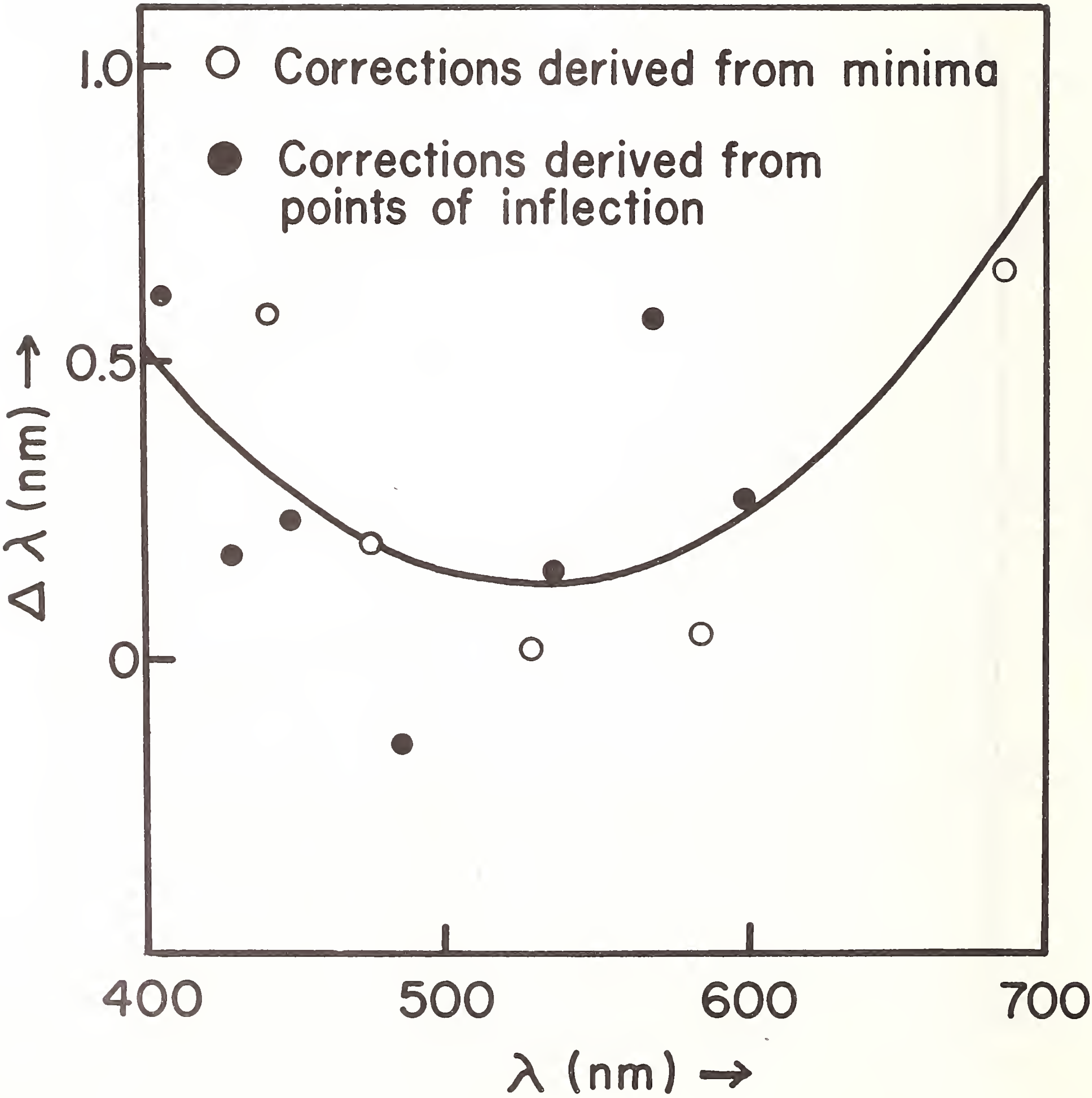

Figure B3. Wavelength calibration curve for Instrument 3

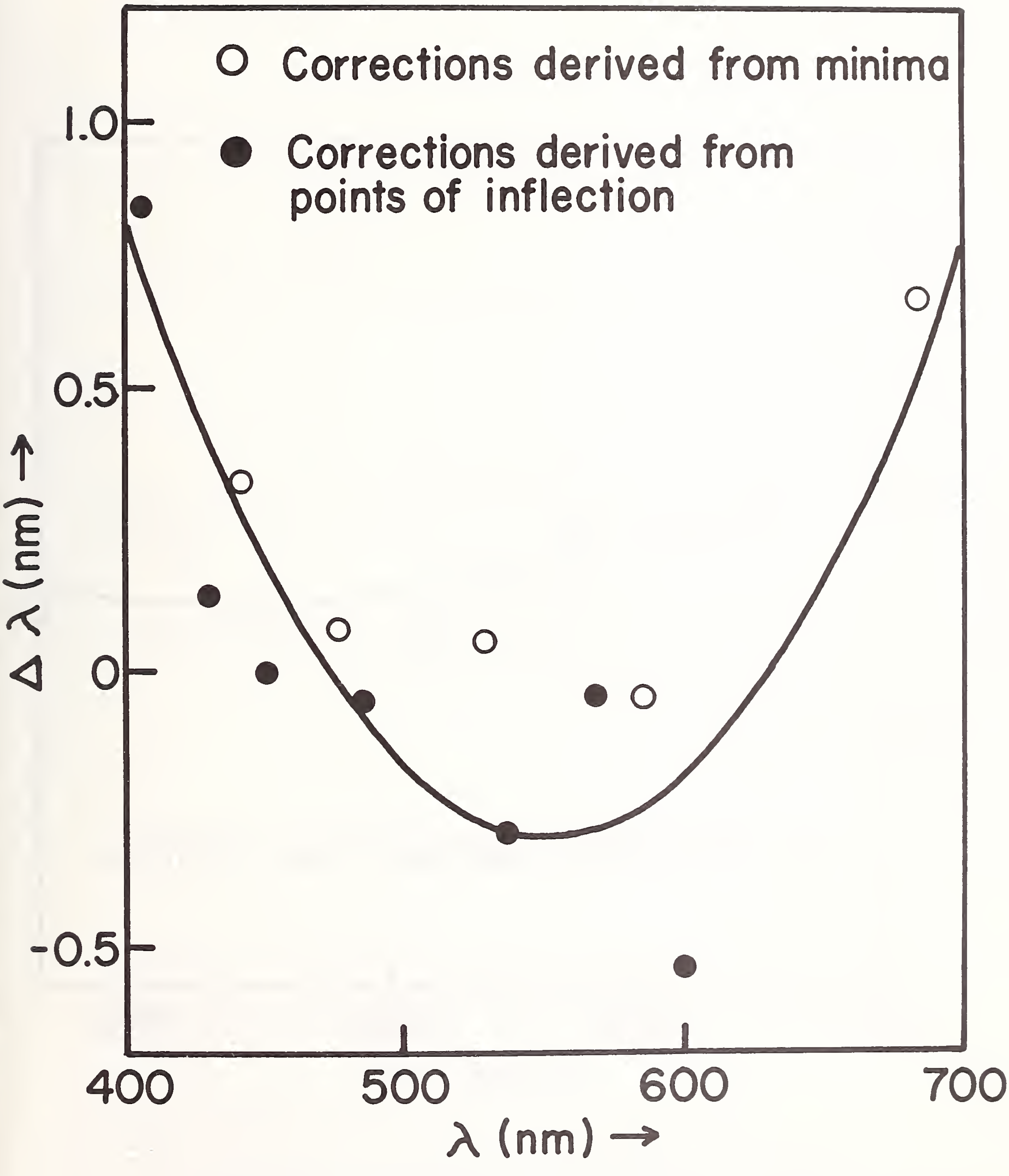

Figure B4. Wavelength calibration curve for Instrument 4

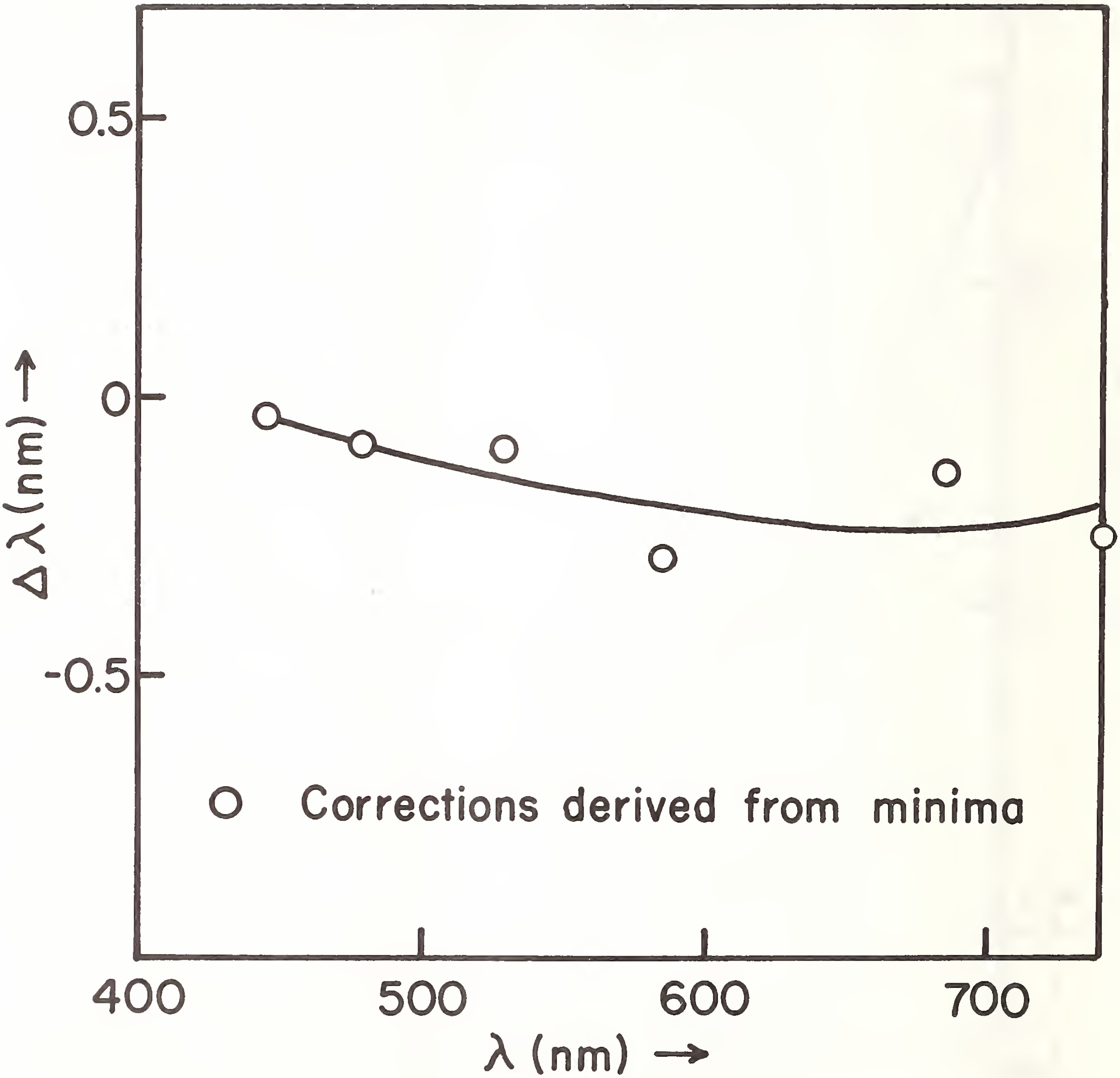

Figure B5. Wavelength calibration curve for Instrument 5

NBS-114A (REV. 2-80)

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET (See instructions)	1. PUBLICATION OR REPORT NO. NBS/SP-692	2. Performing Organ. Report No.	3. Publication Date March 1985

4. TITLE AND SUBTITLE

Transmittance MAP Service

5. AUTHOR(S)

Kenneth L. Eckerle, Jack J. Hsia, and Victor R. Weidner

6. PERFORMING ORGANIZATION *(If joint or other than NBS, see instructions)*

National Bureau of Standards
Department of Commerce
Gaithersburg, MD 20899

7. Contract/Grant No.

8. Type of Report & Period Covered

Final

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS *(Street, City, State, ZIP)*

Same as item 6.

10. SUPPLEMENTARY NOTES

Library of Congress Catalog Card Number: 85-600513

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

An introduction to the Transmittance Measurement Assurance Program (MAP) service is given. Documentation for the service is provided through a comprehensive list of references. The results of a pilot run for the MAP service are given in a sample calibration report.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

bandpass;didymium filters; glass filters; measurement assurance program; neutral density filters; reference spectrophotometer; transmittance; transmittance MAP service; uncertainty estimation; wavelength calibration

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14. NO. OF PRINTED PAGES

50

15. Price

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