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U.S. DEPARTMENT OF COMMERCE/National Bureau of Standards

Standard Reference Materials:

Holmium Oxide Solution Wavelength Standard From 240 to 640 nm — SRM 2034

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Holmium Oxide Solution Wavelength Standard From 240 to 640 nm — SRM 2034

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The work describes the methods and procedures used to determine the wavelengths of minimum transmittance of holmium oxide in perchloric acid solution. Measurements of spectral transmittance of the solutions were made by means of a high precision spectrophotometer over the wavelength range 200 nm to 680 nm. The wavelength scale accuracy of this instrument was verified by extensive measurements of mercury and deuterium emission lines. The measurements of spectral transmittance of the holmium oxide solutions were made as a function of temperature, purity, concentration, and spectral bandwidth. Analysis of the uncertainties associated with these parameters and the uncertainties associated with the calibration of the instrument wavelength scale and the data analysis have resulted in an estimated uncertainty of ±0.1 nm for the determination of the wavelengths of minimum transmittance of the holmium oxide solution.

Key words: holmium oxide; spectral bandwidth; spectrophotometer calibration; spectral transmittance; wavelength calibration; wavelength standard.

1. Introduction

This work describes the methods and procedures used to determine the wavelengths of minimum spectral transmittance of holmium oxide (Ho_2O_3) in perchloric acid ($HClO_4$) solution in the spectral region 200 to 680 nm. The object of this activity was to develop a standard for verifying the wavelength scale of uv/visible spectrophotometers, and to provide assistance toward improving the accuracy of measurements in the fields of molecular absorption spectrometry or spectrophotometry [1][1].

Holmium oxide in a glass matrix has been issued by the National Bureau of Standards (NBS) as a wavelength standard for the ultraviolet and visible spectrum since 1961 [2]. Didymium glass wavelength standards have been issued since 1945. The didymium glass wavelength standards are available from NBS as Standard Reference Material 2009; 2010; 2013; and 2014 [3].

About the Authors: Victor R. Weidner is with NBS' Radiometric Physics Division, headed by Klaus D. Mielenz and served also by Kenneth L. Eckerle; Radu Mavrodineanu is associated with the Bureau's Inorganic Analytical Research Division as a reemployed annuitant; Rance A. Velapoldi is deputy director of the NBS Center for Analytical Chemistry; and Bradley Adams served the Bureau as a student worker.

2. Experimental

2.1 Instrumentation

Measurements of spectral transmittance of the holmium oxide solutions were made by means of a Varian Cary Model 2390 recording spectrophotometer[2].

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[1] Numbers in brackets indicate literature references.

[2] Certain commercial equipment or products are mentioned in this paper in order to adequately document the work. In no case does this imply that the equipment or product is being endorsed by NBS or that it is necessarily the best equipment or product for the application.

The optical system of this double beam instrument consists of a filter predisperser and a double-pass double-sided grating monochromator. A deuterium lamp is used over the wavelength range 185 to 340 nm and a tungsten-halogen lamp is used over the wavelength range 340 to 3150 nm as a source. The spectral bandwidths are selectable from 0.07 nm to 3.6 nm. Data can be recorded by a digital readout system with thermal printer and an analog chart display.

2.2 Materials

2.2.1 Holmium Oxide

The holmium oxide solutions were prepared by dissolving the powder in a 10% perchloric acid in distilled water. The purity of the holmium oxide specimens used in the preparation of these solutions was indicated by the manufacturer [4] to be 99.99% (Lot No. Ho-0-4-007) and 99.999% (Lot No. Ho-0-5-007). Solutions were prepared with 2%, 4%, and 6% holmium oxide. These solutions were placed in 10 mm pathlength nonfluorescent fused silica cuvettes.

The actual form in which Ho is formed when Ho_2O_3 is dissolved in $HClO_4$ is that of an aquo ion of the general formula $Ho(H_2O)_n^3+$. The term "holmium oxide" and the spectral transmittances reported in the manuscript refer to this chemical species. Aqueous solutions of perchloric acid are used in this study to dissolve Ho_2O_3 since the resulting aquo ion is least likely to form complexes when subjected to changes in temperature and concentration [5].

The holmium oxide powder is stated by the manufacturer to have an average particle size of about 2 micrometers. The perchloric acid was a nominal 70-72% reagent grade (considered here as 100%). The distilled water was produced by thermal distillation. The aqueous solutions of holmium oxide in 10% perchloric acid were prepared by weighing 2, 4, or 6 g of the oxide and adding 10 mL of distilled water and 10 mL of perchloric acid. The holmium oxide was dissolved by heating at about 80 °C for one hour. The clear solution was transferred quantitatively to a 100 mL volumetric flask and was brought to volume with distilled water at room temperature.

2.2.2 Cells

The cells used for the transmission measurements of the holmium oxide solutions were conventional nonfluorescent, fused silica, cuvettes with a nominal pathlength of 10 mm. These cuvettes were provided with graded quartz-to-pyrex tubes with rubber caps.

2.3 Measurement Techniques

2.3.1 Calibration of the Spectrophotometer Wavelength Scale

The spectophotometer wavelength scale error was evaluated by measuring the emission spectrum of the instrument's deuterium lamp and the emission spectrum of a mercury pen lamp [6,7,8].

The wavelength calibration was performed at the beginning of the holmium oxide solution measurements and again at the completion of the measurements. A number of emission lines of mercury and two emission lines of deuterium were used over the wavelength range 230 nm to 690 nm (see fig. 1). The wavelength scale errors were determined for spectral bandwidths of 0.1 nm, 1 nm, 2 nm, and 3 nm.

Each emission line was scanned at a rate of 0.01 nm per second and recorded on a scale of 0.2 nm per centi-

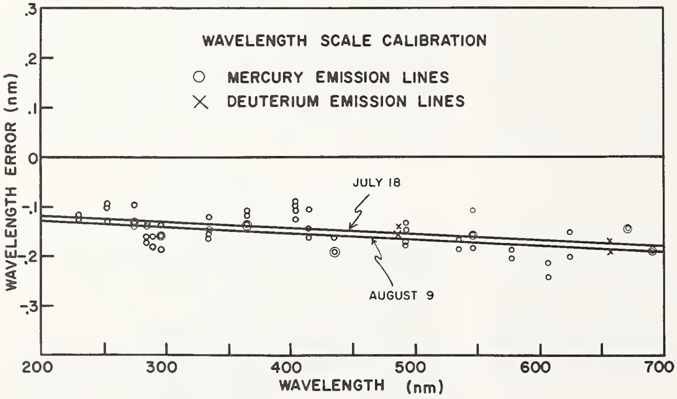


Figure 1–Wavelength scale calibration of the spectrophotometer.

meter. The recorded emission peaks were bisected, using a proportional divider, to determine the wavelengths at the center of the recorded triangular lines. Using this technique, the wavelength scale was read to the nearest 0.01 nm with a precision of ±0.005 nm. The differences between the measured wavelengths of mercury emission lines and the true wavelengths are plotted in figure 1. A linear least squares fit of these wavelength differences for the two dates July 18 and August 9 (also shown in fig. 1) was used to correct the measured values of the holmium oxide transmission minima for errors in the wavelength scale of the instrument.

The wavelength error of the instrument was essentially the same for spectral bandwidths less than 1 nm. However, at spectral bandwidths of 2 nm and 3 nm, the wavelength error was slightly different and a different correction was required.

The deuterium lamp emission lines were measured at two wavelengths each day during the holmium oxide solution characterization to verify the accuracy of the wavelength scale. These measurements showed that the calibration of the wavelength scale is constant to ±0.02 nm after a nominal warm-up of one hour. These results, as well as the repeatability of the calibration with the mercury line source, indicate that the overall stability of the instrument wavelength scale was better than ±0.05 nm during the period of time required to complete the wavelength characterization of the holmium oxide solution.

The mercury pen lamp is mounted in the lamp positioning device that ordinarily holds the deuterium lamp of the spectrophotometer. The arc tube of the mercury pen was aligned parallel to the entrance slit of the monochromator. The lamp holder is equipped with screw adjustments for vertical and horizontal alignment of the source. The entrance slit cannot be directly observed. Therefore, the source was adjusted with instrument operating in the single-beam mode until a maximum signal is observed. To test the effect of the positioning of the mercury line source on the observed emission line maxima, the pen lamp was moved horizontally across the field of view of the entrance slit to the monochromator in approximately 0.22 mm steps for a total of 12 steps or a 2.64 mm distance. The results of this experiment showed that the recorded emission maxima varied by less than ±0.01 nm for lamp positions within ±1 mm of the center position. The center position corresponded to the position of maximum signal.

2.3.2 Confirmation of Spectral Bandwidths

The mercury line source was used to confirm the spectral bandwidth settings of the spectrophotometer. The mercury emission line at 435.8 nm was scanned for spectral bandwidths of 0.1 nm, 0.25 nm, 0.5 nm, 1 nm, 2 nm and 3 nm. The emission peak was normalized to 100% on the chart recorder by adjusting the instrument gain. The bandwidth at half peak height is approximately equal to the spectral bandwidth. The natural bandwidth of the emission line is much less than the instrument bandwidth. The recorded curve has a triangular symmetry for all settings of the monochromator slitwidths. For the above-mentioned nominal spectral bandwidth settings of the spectrophotometer, the measured spectral bandwidths were 0.092 nm, 0.228 nm, 0.468 nm, 1.04 nm, 2.18 nm, and 3.20 nm, respectively. This technique for determining spectral bandwidths has some uncertainties due to assumptions made. However, it serves to confirm that the desired spectral bandwidths are closely approximated when the instrument is programmed to provide those settings; hence the effect on the transmittance minima is negligible.

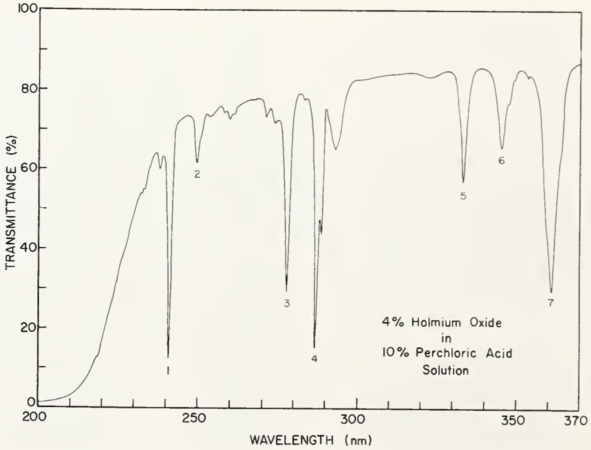
2.3.3 Determination of the Wavelengths of Minimum Transmittance

The spectral transmittance of holmium oxide in an aqueous solution of perchloric acid exhibits many absorption bands in the ultraviolet and visible spectrum. The spectrum is shown in figure 2 for a 0.1 nm spectral bandwidth scan. The total number of observed absorption bands varies as a function of the spectral bandwidth used during the recording of the spectrum. There are approximately 14 major absorption features or bands between 200 and 650 nm that can be observed clearly for a wide range of spectral bandwidth settings. Most of the major absorption bands have lesser bands between them or in close association. These smaller bands are better resolved at bandwidths less than 1 nm. Only the major absorption bands that could be of use as possible wavelength standards were selected for detailed study. The various parameters affecting the measured wavelengths of minimum transmittance of the holmium oxide solution are discussed in section 3. These include such parameters as temperature, purity, concentration, and spectral bandwidth.

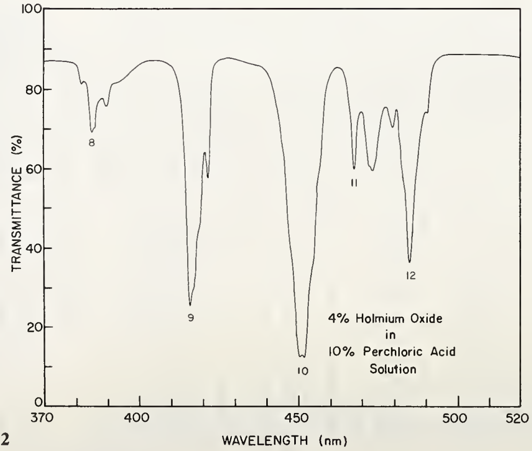
The spectral transmittance of the holmium oxide solution was digitally recorded on a thermal printer at 0.1 nm intervals with the monochromator scanning at a rate of 0.05 nm/s. The transmittance was simultaneously recorded on a chart with a wavelength display of 0.5 nm/cm.

The holmium oxide solution was contained in a 10 mm pathlength fused silica cuvette. The transmittance of the solution in this cuvette was measured relative to an air-only path in the reference beam.

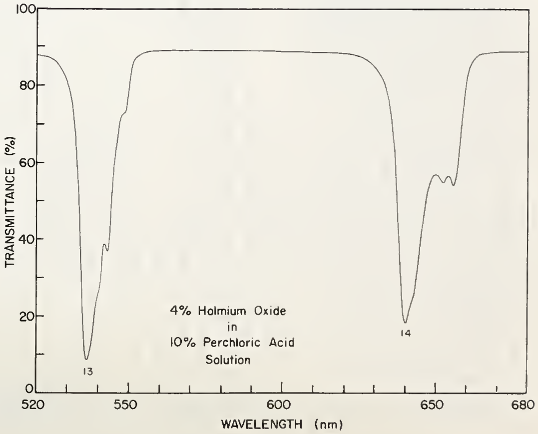
The determination of the wavelengths of the transmittance minimum of the holmium oxide solution was derived from an analysis of the recorded digital output. The 0.1 nm interval transmittance data were plotted on



2–1



2–2



2–3

Figure 2–Spectral transmittance of a 4% solution of holmium oxide in 10% perchloric acid in water, beginning at right.

graph paper on a scale of 0.1 nm per cm with subdivisions of 0.01 nm per mm so that the wavelength interval of this plotted digital data could be read to the nearest 0.01 nm between the measured data points. The location of the wavelength of minimum transmittance for a given holmium oxide band was determined graphically (fig. 3) by drawing a curve through the data points and bisecting the horizontal grid lines between the two slopes of the curve representing the absorption feature. Several of these bisection points locate the line between the two slopes that intersects the minimum transmittance point, (usually at the lowest point of the curve). The wavelength at this point of intersection was taken as the measured wavelength of minimum transmittance for the absorption feature. The true wavelength of the minimum was determined by applying a wavelength correction to the instrumental wavelength scale, as determined in section 2.3.1.

2.3.4 Accuracy of the Wavelengths of Minimum Transmittance

The overall uncertainty in the location of the wavelengths of minimum transmittance is believed to be no greater than ±0.1 nm at the 95% confidence limit. This conclusion is based on the reproducibility of the following calibration procedures:

- The uncertainty of the calibration of the instrument wavelength scale using the mercury and deuterium lamps, and the long-term and day-to-day instabilities of the instrument wavelength scale (less than ±0.05 nm).
- The imprecision of the graphical technique for deriving the measured wavelengths of minimum transmittance. (±0.02 nm).
- The dependence of the measured wavelengths of minimum transmittance on variations in temperature or concentration of the solution. (±0.02 nm).

These uncertainties have been discussed in section 2.3.1. The imprecision of the graphical technique is illustrated in table 1, where data are shown for three absorption features. Ten sets of digital results were produced by the instrument for each of these features. The digital data were plotted and the wavelengths of minimum transmittance for each absorption feature were determined for the 10 sets of data by the graphical technique. The standard deviation and standard error for the 10 determinations is also given in table 1.

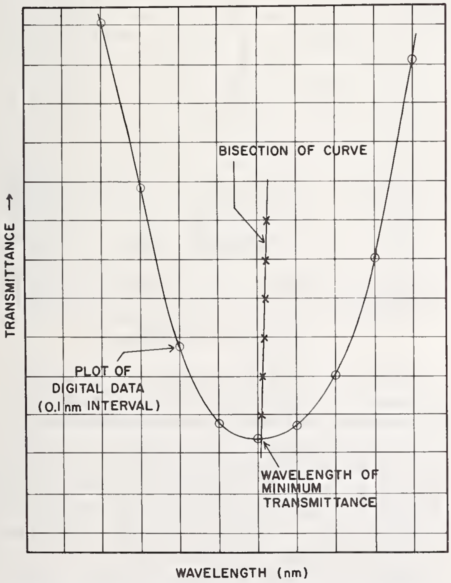


Figure 3–Graphical technique used to determine the transmittance minima from digital spectral transmittance data.

3. Measurements

3.1 Influence of the Blank Cuvette, Solvent, and Water

The spectral transmittances of an empty fused silica cuvette, a cuvette filled with distilled water, and a

Table 1. Ten cycle repetitive measurements of three holmium oxide transmittance minima. Listed minima are obtained by graphical techniques, using the digitally recorded 0.1 nm interval transmittance measurement.

	(spectral bandwidth=0.1 nm)		
Cycle	Minima No. 1	Minima No. 12	Minima No. 14
1	240.88 nm	485.08 nm	640.28 nm
2	240.87	485.10	640.30
3	240.86	485.11	640.30
4	240.84	485.11	640.30
5	240.85	485.10	640.29
6	240.84	485.11	640.30
7	240.85	485.11	640.29
8	240.85	485.11	640.30
9	240.84	485.10	640.30
10	240.85	485.11	640.29
Average:	240.853	485.104	640.295
Standard Deviation:	0.0134	0.0097	0.0071
Standard Error:	.0042	.0031	.0022

Note: The data shown in this table have not been corrected for the wavelength scale error of the spectrophotometer.

cuvette filled with the diluted perchloric acid (without holmium oxide) are illustrated in figure 4. These measurements are relative to an air path in the reference beam.

Measurements of the wavelength of minimum transmittance for the band at 241 nm were made with the holmium oxide in perchloric acid solution versus a cuvette containing only the perchloric acid in the reference beam. The wavelength of minimum transmittance of this band was found to be the same when the holmium oxide solution was measured relative to air in the reference beam and when it was measured relative to a cuvette containing the perchloric acid solution. Since the 241 nm band is within the spectral range showing a slope in the transmittance of the cuvette–perchloric acid spectra, (see fig. 4) it was considered to be the band most likely to be influenced by this slope. However, no measurable influence was detected in the location of the wavelength of minimum transmittance of this band due to these spectral features associated with the solvent or cuvette.

3.2 Influence of Temperature

The wavelengths of minimum transmittance of the holmium oxide solution were determined at 20 °C, 25 °C, and 30 °C for spectral bandwidths of 0.1 nm and 1 nm. If there is a temperature-related influence on the location of the wavelengths of minimum transmittance, it was not detected within these temperature ranges. The measured differences were attributed to random uncertainties.

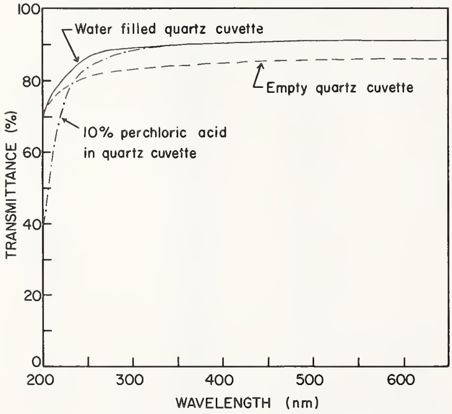


Figure 4–Spectral transmittances of an empty fused silica cuvette, a cuvette filled with water, and a cuvette filled with a solution of 10% perchloric acid in water.

3.3 Influence of Purity

Complete spectral scans of solutions made with the 99.99% and 99.999% purity holmium oxides showed no spectral differences except in the extreme ultraviolet cut-off at wavelengths less than 230 nm. The differences are illustrated in figure 5 for the wavelength range 200 to 300 nm. The wavelengths of the minimum transmittances for the 14 selected absorption bands were found to be the same for solutions prepared from these two lots of holmium oxide.

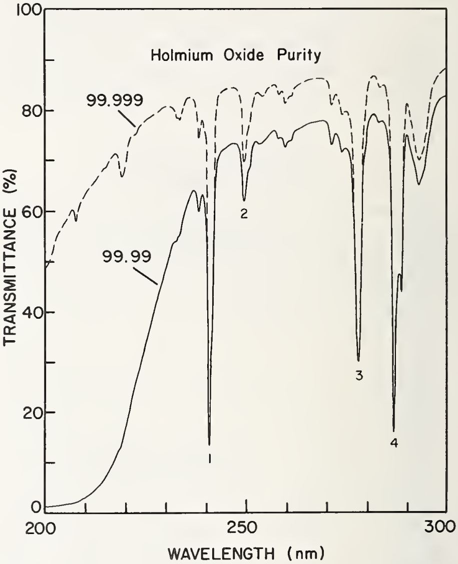


Figure 5–Spectral differences of solutions prepared from holmium oxide specimens of 99.99% purity and 99.999% purity.

3.4 Influence of Concentration

The location of the wavelengths of minimum transmittance of the holmium oxide solution as a function of concentration was tested with concentrations of 2%, 4%, and 6% in the perchloric acid solution. The general spectral scan indicated that the changes in concentration affected the measured transmittance as would be expected but did not influence the location of the transmittance minima. To verify this further, three of the bands were evaluated by the graphical technique described in section 2.3.1. The results of these concentration measurements for these bands are shown in table 2.

Table 2. Influence of holmium oxide concentration on the wavelengths of minimum transmittance.

	(spectral bandwidth = 0.1 nm)		
Holmium oxide concentration	Minima No. 1	Minima No. 12	Minima No. 14
2 %	240.84 nm	485.11 nm	640.30 nm
4 %	240.86	485.11	640.32
6 %	240.84	485.12	640.31

Note: The data shown in this table have not been corrected for the wavelength scale error of the spectrophotometer.

3.5 Influence of Spectral Bandwidth

The wavelengths of minimum transmittance of the holmium oxide solution were determined for spectral bandwidths of 0.1 nm, 0.25 nm, 0.5 nm, 1 nm, 2 nm, and 3 nm. Measurement of the instrumental spectral bandwidths is discussed in section 2.3.2. The influence of spectral bandwidth was by far the most important parameter affecting the location of the measured wavelengths of minimum transmittance. The results of this study are shown in figures 6 through 19 for the 14 minima identified in figures 2–1, 2–2, and 2–3. The data for bandwidth effects are also listed in tables 3 and 4. The measurements indicate that for most of the holmium oxide bands, the location of the wavelengths of minimum transmittance does not change significantly for spectral bandwidths of less than 1 nm. From these data it can be seen that a 0.1 nm spectral bandwidth is adequate to define the wavelengths of minimum transmittance within the stated uncertainties. For some bands the location of the minimum transmittance shifts only slightly for spectral bandwidths greater than 1 nm. However, many do show large shifts for larger bandwidth settings. These results indicate that for instruments with spectral bandwidth settings of less than 1 nm, the holmium oxide solution can serve as an excellent wavelength standard. For instruments having bandwidth settings between 1 nm and 3 nm the standard can still be of use if the instrument bandwidth is known.

4. Results

4.1 Transmittance of Holmium Oxide Solution

The general spectral signature of the holmium oxide solution is illustrated in figures 2–1, 2–2, and 2–3 for a spectral bandwidth of 0.1 nm. Some of the finer spectral features shown in these figures will be absent when the spectrum is recorded at bandwidths greater than 1 nm. The major transmittance minima selected for this study are indicated by numbers 1 through 14 as shown in these figures. These band numbers are used throughout the manuscript as a key to associate the data in the tables with the spectral features illustrated in the figures.

4.2 Selection of Useful Wavelengths of Minimum Transmittance

The selection of holmium oxide bands that are considered useful for calibration purposes was based on the influence of spectral bandwidth on the location of these minima. The transmittance minima of small side bands associated with major absorption features usually shift in wavelength with bandwidth setting or are not resolved over the normal instrumental bandwidth range. The 14 major absorption bands are listed in tables 3 and 4. The absorption bands not listed in the tables are not considered useful for wavelength calibration purposes.

4.3 Numerical Data

The wavelengths of minimum transmittance of the 4% holmium oxide solution are listed in tables 3 and 4 for six spectral bandwidths. The results of the measurements as a function of temperature are also listed for 0.1 nm and 1 nm spectral bandwidths at temperature settings of 20 °C, 25 °C, and 30 °C.

The shift in wavelengths of minimum transmittance as a function of spectral bandwidth is shown in figures 6 through 19 for the 14 selected minima. The recommended values of minimum transmittance are listed in table 3 and 4 for the measurements made at 25 °C.

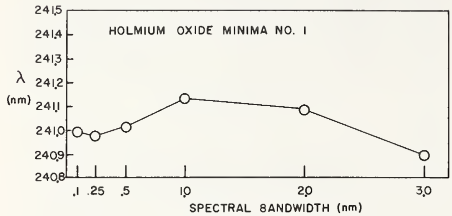
4.4 Uncertainty of Measured Wavelengths

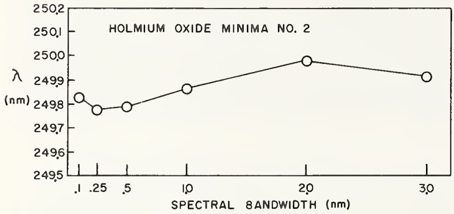
The uncertainties associated with the data listed in tables 3 and 4 have been discussed in section 2.3.4. The uncertainty in the determination of the wavelengths of minimum transmittance for the holmium oxide solution is believed to be no greater than ±0.1 nm at the 95% confidence limit using the instrumentation and techniques described in this paper.

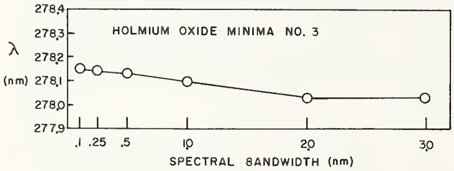
4.5 Comparison of Results with Measurements Made on the NBS Reference Spectrofluorimeter

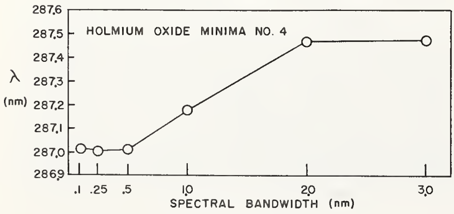
The wavelengths of minimum transmittance of several absorption bands of holmium oxide solution were determined by analysis of spectral data obtained with the NBS Reference Spectrofluorimeter [9]. This reference instrument is primarily designed to be used as a research tool for high accuracy spectral analysis of fluorescent materials and in the development of standards for use in this area of research. The versatile design of the instrument allows for its use as a high accu-

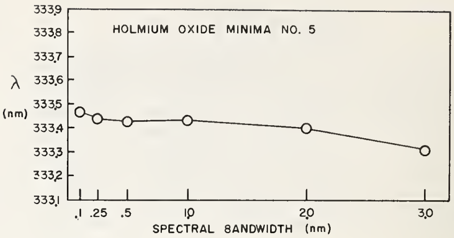
Figures 6-19—Fourteen holmium oxide transmittance minima as a function of spectral bandwidth.

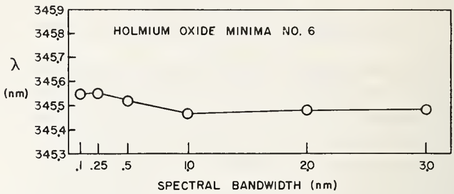


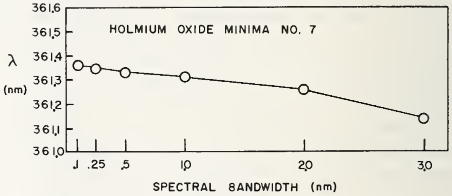


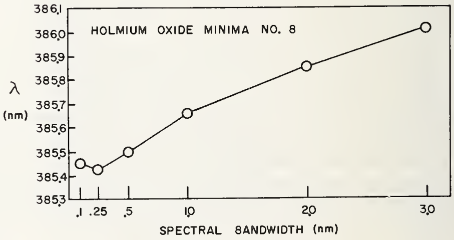


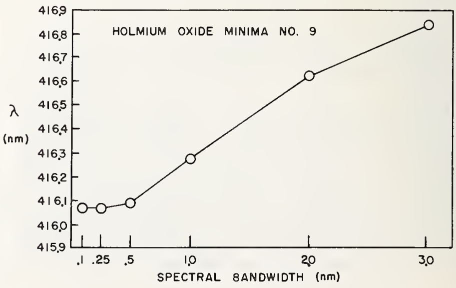


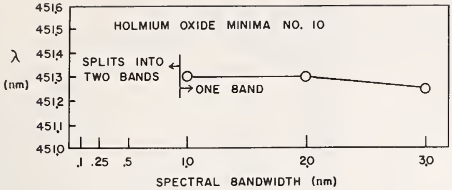


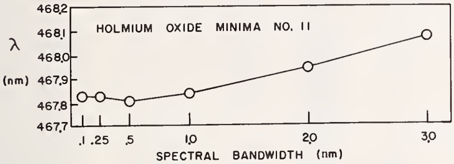


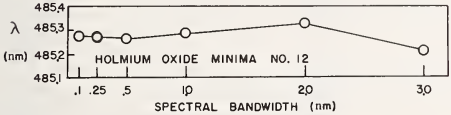


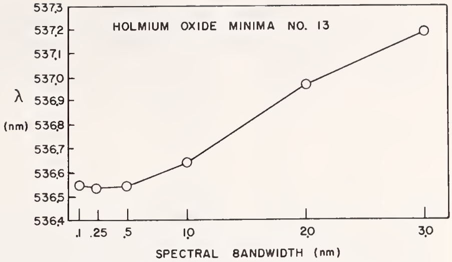












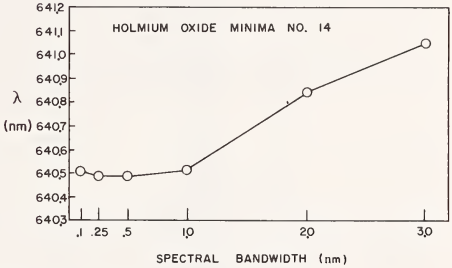


Table 3. Results of holmium oxide solution analysis for transmittance minima 1 through 7 for spectral bandwidths of 0.1, 0.25, 0.5, 1, 2, and 3 nm. 4% holmium oxide in a 10% perchloric acid solution.

Minimum	Temp.	Spectral Bandwidths					
No.	(°C)	0.1 nm	0.25 nm	0.5 nm	1 nm	2 nm	3 nm
1	20	240.99 nm			241.13 nm		
1	25	240.99	240.97 nm	241.01 nm	241.13	241.08 nm	240.90 nm
1	30	240.99			241.15		
2	20	249.79			249.88		
2	25	249.83	249.78	249.79	249.87	249.98	249.92
2	30	249.79			249.86		
3	20	278.14			278.09		
3	25	278.15	278.14	278.13	278.10	278.03	278.03
3	30	278.14			278.09		
4	20	286.99			287.17		
4	25	287.01	287.00	287.01	287.18	287.47	287.47
4	30	287.01			287.18		
5	20	333.48			333.45		
5	25	333.47	333.44	333.43	333.44	333.40	333.32
5	30	333.48			333.45		
6	20	345.57			345.47		
6	25	345.55	345.55	345.52	345.47	345.49	345.49
6	30	345.62			345.49		
7	20	361.38			361.33		
7	25	361.36	361.35	361.33	361.31	361.16	361.04
7	30	361.38			361.31		

Note: The uncertainty in the wavelengths of minimum transmittance is ±0.1 nm. An extra decimal place is given for rounding purposes.

Table 4. Results of holmium oxide solution analysis for transmittance minima 8 through 14 for spectral bandwidths of 0.1, 0.25, 0.5, 1, 2, and 3 nm. 4% holmium oxide in a 10% perchloric acid solution.

Minimum	Temp.	Spectral Bandwidths					
No.	(°C)	0.1 nm	0.25 nm	0.5 nm	1 nm	2 nm	3 nm
8	20	385.45 nm			385.66 nm		
8	25	385.45	385.42 nm	385.50 nm	385.66	385.86 nm	386.01 nm
8	30	385.44			385.68		
9	20	416.10			416.27		
9	25	416.07	416.07	416.09	416.28	416.62	416.84
9	30	416.13			416.30		
10	20	-----*			451.30		
10	25	-----*	-----*	-----*	451.30	451.30	451.24
10	30	-----*			451.34		
11	20	467.83			467.84		
11	25	467.82	467.82	467.80	467.83	467.94	468.07
11	30	467.84			467.84		
12	20	485.30			485.29		
12	25	485.28	485.28	485.27	485.29	485.33	485.21
12	30	485.31			485.30		
13	20	536.53			536.65		
13	25	536.54	536.53	536.54	536.64	536.97	537.19
13	30	536.45			536.63		
14	20	640.48			640.52		
14	25	640.51	640.49	640.49	640.52	640.84	641.05
14	30	640.52			640.49		

* Splits into 2 minima for spectral bandwidths less than 1 nm.

Note: The uncertainty in the wavelengths of minimum transmittance is ±0.1 nm. An extra decimal place is given for rounding purposes.

racy spectrophotometer in some applications. The wavelength scale of the spectrofluorimeter has been carefully calibrated by extensive measurements of emission line sources and is known to have an uncertainty of ±0.1 nm for spectral bandwidth of 0.1 nm.

This reference instrument was used to confirm the results obtained with the calibrated high-precision commercial spectrophotometer used for the holmium oxide measurements. A comparison was made to confirm the results for three of the transmittance minima at one spectral bandwidth setting. The results of this comparison are shown in table 5. The two instruments provided data for these three transmittance minima that agree to within 0.1 nm. The wavelength scale uncertainty for both instruments is ±0.1 nm.

4.6 Other Measurements Outside NBS

A list of the wavelengths of minimum transmittance of the holmium oxide solution reported by other workers is given in table 6. (Ref. 10 offers details of these measurements.)

Table 5. Comparison of values for holmium oxide transmittance minima with values obtained with the NBS Reference Spectrofluorimeter.

	(spectral bandwidth = 0.1 nm)		
Instrument	Minima No. 1	Minima No. 7	Minima No. 14
Varian (Cary) Model 2390	240.995 nm	361.361 nm	640.507 nm
NBS Reference Spectrofluorimeter	240.970 nm	361.313 nm	640.469 nm

The data shown in table 6 indicate that workers in other laboratories are in generally good agreement with one another and that the NBS data also agree well with these workers' previously published data. These workers also found that the wavelengths of minimum transmittance of holmium oxide in similar solution in perchloric acid are not sensitive to variations in temperature and concentration. They also concluded that the wavelengths of minimum transmittance were least affected by changes in spectral bandwidth for band-

Table 6. Comparison of values for holmium oxide transmittance minima with values obtained by other workers (see Reference 10).

Min. No.	NBS Transmittance Minima	(1)	(2)	(3)	(4)
1	240.99 nm*	241.15 nm	241.0 nm	241.1 nm	241.1 nm
2	249.83 *	249.75	250.0	249.7	249.7
3	278.15 *	278.2	277.8	278.7	278.2
4	287.01 *	287.15	287.5	287.1	287.2
5	333.47 *	333.5	333.3	333.4	333.3
6	345.55 *	345.6	345.5	345.5	345.0
7	361.36 *	361.5	361.0	361.5	361.2
8	385.45 *	385.6	385.6	385.5	385.6
9	416.07 *	416.2	416.0	416.3	416.6
10	451.30 #	450.7	450.4	450.8	451.0
11	467.82	467.75			468.0
12	485.28	485.25	485.2	485.8	485.2
13	536.54	536.3			536.8
14	640.51	640.5			

* Spectral bandwidth = 0.1 nm
# Spectral bandwidth = 1 nm

widths less than 1 nm, but that large shifts can be encountered at bandwidths in excess of 1 nm.

5. Conclusions

The reported wavelengths of minimum transmittance of the holmium oxide solution appearing in tables 3 and 4 are estimated to be uncertain by no more than ±0.1 nm at the 95% confidence limit. These wavelengths of minimum transmittance were found to be essentially unaffected by changes in temperature at 25 °C ±5 °C. They were also unaffected by variations in the concentration for solutions containing 2%, 4%, and 6% holmium oxide. The critical parameter affecting the measured values of minimum transmittance was found to be the spectral bandwidth setting of the spectrophotometer. For spectral bandwidths less than 1 nm the wavelength shift is generally less than 0.2 nm. Users can most effectively determine the wavelength error associated with their instrument by using the NBS data listed in tables 3 and 4 that are representive of the spectral bandwidth setting ordinarily used with the instrument. The wavelengths of minimum transmittance of the holmium oxide solution for spectral bandwidths greater than 3 nm have not been evaluated.

The authors gratefully acknowledge the assistance of Chenq-Tsong Chang, guest worker from Taiwan, for his contribution in measuring the transmittance minima of the holmium oxide solution on the NBS Reference Spectrofluorimeter; Jack J. Hsia, for constructive discussions; Robert W. Burke, for assistance in the preparation of various Ho_2O_3 solutions; and the NBS Optical Shop for preparation of the fused silica cuvettes.

6. References

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Appendix A-1. Standard Reference Materials for Spectrophotometry Provided by the National Bureau of Standards.

SRM	Material	Composition
Solid Filters		
930 Glass Filters for Spectrophotometry	Solid filters made of Schott NG-4 and NG-5 optically neutral glass. Obtained from the Schott-Jenaer Glaswerk, Mainz, Germany.	Three filters with nominal transmittances of 10, 20, 30%. Each filter is mounted in a black anodized aluminum holder provided with front and rear shutters. The filters are stored in a cylindrical aluminum container.
2031 Metal-on-Quartz Filters for Spectrophotometry	Solid filters made of semi-transparent evaporated chromium-on-fused silica (non-fluorescent) plates.	Three filters with nominal transmittances of 10, 30, 90%. Each filter is made from two fused silica plates assembled by optical contact. The 90% filter is made by assembling 2 clear plates. The 10% and 30% filters are made by assembling a fused silica plate which carries a film of semitransparent chromium to produce the desired transmittance, and a clear plate. Each filter assembly is placed in a metal holder provided with shutters.
Liquids and Cuvettes		
931b Liquid Absorbance Standards for Ultraviolet and Visible Spectrophotometry	Liquid filters made from a solution of Co and Ni metals dissolved in a mixture of nitric-perchloric acids. The pH of the solution is about 1. Prepared in the NBS Center for Analytical Chemistry.	Three solutions and a blank were delivered in sealed ampoules and have nominal absorbances of 0.1 to 0.9.
935 Crystalline Potassium Dichromate for Use as an Ultraviolet Absorbance Standard	Crystalline potassium dichromate of established purity obtained from the J. T. Baker Chemical Co., Phillipsburg, New Jersey.	The crystalline material of reagent grade purity is offered in glass bottles provided with plastic screw caps.

Certification	Use	Remarks
The transmittance of each filter is measured with the high-accuracy spectrophotometer at λ 440, 465, 546.1, 590, 635 nm, using spectral bandpasses of 2.2, 2.7, 6.5, 5.4, 6.0 nm respectively.	This SRM is intended as a reference source for the verification of the transmittance and absorbance scales of conventional spectrophotometers.	The use of this SRM is limited to the visible spectrum and requires narrow spectral bandpasses. For further details, consult the Certificate included in the Appendix; see also NBS Special Publication 260-51.
The transmittance of each filter is measured with the high-accuracy spectrophotometer at λ 250, 280, 340, 360, 400, 465, 500, 546.1, 590, 635 nm.	This SRM is intended as a reference source for the verification of the transmittance and absorbance scales of conventional spectrophotometers in the ultraviolet and visible spectral region.	SRM 2031 has good optical neutrality and can be used to calibrate spectrophotometers with wide spectral bandpasses from about 200 to 800 nm (with possible extension to λ=3 μm). For further details, consult the Certificate in the Appendix; see, also NBS Special Publication 260-68.
The absorbance of each solution was measured at λ 302, 395, 512, 678 nm using spectral bandpasses of 1.0 1.7, 2.0, 6.5 nm respectively. The certification is made with an uncertainty of about ±1.0%.	This SRM is primarily intended for the verification of the accuracy of transmittance or absorbance scales of spectrophotometers.	Requires the use of SRM 932 and of narrow spectral bandpasses. Provides only one certified value in the ultraviolet. For further details consult the Certificate included in the Appendix.
The apparent specific absorbance is certified for five concentrations at λ 235, 257, 313, 345, 350 nm using spectral bandpasses of 1.2, 0.8, 0.8, 0.8, 0.8 nm respectively, using SRM 932.	This SRM is intended to be used as a reference standard for the verification of the accuracy and linearity of the absorbance scale of spectrophotometers.	Requires accurate preparation of solutions of potassium dichromate in 0.001N perchloric acid. Requires the use of SRM 932 and of narrow spectral bandpasses. Can be used only in the ultraviolet. For further details, consult the Certificate included in the Appendix; see also NBS Special Publication 260-54.

Appendix A-1. Continued.

SRM	Material	Composition
932 Quartz Cuvette for Spectrophotometry	All-quartz cuvette made of non-fluorescent fused silica of optical quality.	Each cuvette is made of fused silica and is provided with a Teflon stopper. The transparent windows are attached to the body of the cuvette by direct fusion. The cuvettes are stress-relieved by proper annealing. The cuvette is stored in a transparent plastic container.
Stray Light		
2032 Potassium Iodide for Use as a Stray Light Standard	Crystalline potassium iodide of established purity, obtained from the J. T. Baker Chemical Co., Phillipsburg, New Jersey.	The reagent-grade, crystalline KI is 99.8% pure as indicated by the manufacturer. Moisture is 0.007%. Homogeneity was determined by absorbance measurements at 265, 267, and 270 nm, and was found adequate.
2033 Potassium Iodide for Use as a Stray Light Standard with Radiation Attenuator	Crystalline potassium iodide of established purity, and two semi-transparent evaporated metal-on-fused silica filters contained in a metal holder provided with shutters. The KI was obtained from the J. T. Baker Chemical Co., Phillipsburg, New Jersey. The filters and the holder were made by the NBS Instrument Shop.	Potassium iodide as for SRM 2032. Two semi-transparent evaporated chromium-on-fused silica (non-fluorescent) filters of optical quality.
Wavelength		
2009 Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers	The didymium glass was prepared by Corning Glass Works, Corning, New York, as Corning 5120 Filter Glass.	Rare earth oxides in a glass matrix. This filter is 1 cm wide, 3 cm high, and 3 mm thick. Each filter is placed in a metal holder which fits into the cuvette holder of the spectrophotometer.
2010 Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers	Same as SRM 2009.	Rare earth oxides in a glass matrix. This filter is placed 5.1 cm square and 3 mm thick.

Certification	Use	Remarks
The nominal inner pathlength is 10 mm and it is certified at 10 points along the height of the transparent windows with an uncertainty of ±0.0005 mm. The cuvettes are 12.5 mm square and 48 mm high.	To be used when accurate transmittance or absorbance measurements are made on liquid samples. Should be used in conjuntion with SRM's 931, 935, 2032, and 2033.	For further details, consult the Certificate included in the Appendix; see also NBS Special Publication 260-32.
The specific absorbances were measured with the high-accuracy spectrophotometer at 240, 245, 250, 255, 265, 270, and 275 nm at 23.5 °C using a spectral bandpass of 0.2 nm. The measurements were performed using SRM 932 quartz cuvettes.	SRM 2032 is to be used to assess heterochromatic stray light in the ultraviolet region below 260 nm, in absorption spectrophotometers. It is recommended that SRM 932 be used in the measurements.	SRM 2032 should be stored in the original, low-actinic glass bottle and cardboard container, protected from exposure to light and humidity. The estimated stability is 3 years.
Same as for SRM 2032. The transmittance of the evaporated metal-on-fused silica filters was measured at 255 nm with the high-accuracy spectrophotometer. The nominal value is 10% for each filter, and a combined value of 1%.	SRM 2033 is to be used to assess heterochromatic and isochromatic stray light in absorption spectrophotometers.	Same as for SRM 2032. The two semi-transparent evaporated metal-on-fused silica filters in the metal holder should be stored in the plastic container provided with SRM 2033.
The wavelengths of maximum absorption were determined with a high-precision spectrophotometer for bandwidths in the range 1.5 to 10.5 nm and for 14 to 24 wavelengths in the range 400 to 760 nm. The instrument has a wavelength accuracy of 0.04 nm.	The filters are to be used in calibrating the wavelength scale in the visible spectral region for spectrophotometers having nominal bandwidths in the range 1.5 to 10.5 nm.	SRM 2009 was not measured individually. It is recommended for most applications. For further details, consult NBS Special Publication 260-66.
Same as SRM 2009.	Same as SRM 2009.	Same as SRM 2009.

Appendix A-1. Continued.

SRM	Material	Composition
2013 Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers	Same as SRM 2009.	Same as SRM 2009.
2014 Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers	Same as SRM 2009.	Same as SRM 2009.
2034 Holmium Oxide for Use as Wavelength Standard for Spectrophotometry	Holmium oxide of established purity dissolved in perchloric acid.	Holmium oxide is offered in solutions in sealed 10 mm quartz cuvettes.
Fluorescence		
936 Quinine Sulfate Dihydrate	The quinine sulfate dihydrate was a special lot of material obtained from J. T. Baker Chemical Co., Phillipsburg, New Jersey.	SRM 936 contains 1.7% impurities determined by liquid chromatography and believed to be dihydroquinine sulfate dihydrate. The water content is 4.74 ± 0.05% determined by the Karl Fisher method and 4.57 ± 0.04% by weight loss. Theoretical value is 4.60%.

NOTE: For further scientific information concerning the following Standard Reference Materials, write or call at the National Bureau of Standards, Gaithersburg, MD 20899: SRM's 930, 931, 932, 935, 936, 2030, 2031, 2032, and 2033: Dr. R. W. Burke, Inorganic Analytical Research Division, Chemistry Building, Room B222. Phone: 301-921-2141. SRM's 2009, 2010, 2013, and 2014: Mr. K. L. Eckerle, SRM 2034: Mr. V. R. Weidner, Radiometric Physics Division, Center for Radiation Research, Metrology Building, Room A321. Phone: 301-921-2791; for scientific information concerning physical parameters involved in spectrophotometry and luminescence measurements: Dr. K. D. Mielenz, Chief, Radiometric Physics Division, Center for Radiation Research, Metrology Building, Room B304. Phone: 301-921-3864.

For information concerning the availability and purchase of the SRM's discussed in this paper contact: Office of Standard Reference Materials, Chemistry Building, Room B311. Phone: 301-921-2045. The NBS Publications discussed in this paper can be purchased from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20034. Please write for information concerning availability, delivery, and price.

Certification	Use	Remarks
Each filter was measured separately; hence the data provide a more accurate representation of the optical properties of the individual filter.	Same as SRM 2009.	SRM 2013 was measured individually and should be used when assurance in the evaluation of an instrument's wavelength scale is highly critical. For further details, consult NBS Special Publication 260-66.
Same as SRM 2013.	Same as SRM 2009.	Same as SRM 2013.
The wavelengths of maximum absorption of holmium oxide solutions in perchloric acid is determined for various spectral bandpasses at the nominal wavelengths 241, 250, 278, 287, 333, 345, 361, 385, 416, 451, 467, 485, 536, 640 nm.	This SRM is to be used as a reference standard for the verification of the accuracy of the wavelength scale of absorption spectrometers, in the ultraviolet and visible spectral regions.	Holmium oxide solutions in perchloric acid were selected as a wavelength standard because the absorption bands are narrower than those of the holmium oxide glass.
The material is certified for the relative molecular emission spectrum E(λ), in radiometric units for a solution of 1.28×10^{-6} mol/L in 0.105 mol/L perchloric acid, using an excitation wavelength of 347.5 nm. The certified values of the molecular emission spectrum at 5 nm intervals from 375 to 675 nm are given. This certification was made with the NBS reference fluorescence spectrometer.	For evaluation of methods and the calibration of fluorescence spectrometers. A solution of 0.1 mg/mL in 1000 mL 0.105 mol/L perchloric acid is recommended. It should be stored in the dark in a well-stoppered glass bottle. This solution is stable for three months. SRM 936 is for "in vitro" diagnostic use only.	The material should be kept in its original bottle and stored in the dark at 30 °C or less. Under these conditions SRM 936 is stable for three years. See NBS Special Publication 260-64.

U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 930D

Glass Filters for Spectrophotometry

This Standard Reference Material (SRM) is intended as a reference source for the verification of the transmittance and absorbance scales of spectrophotometers. SRM 930D consists of three individual glass filters in separate metal holders and one empty filter holder. The filter holders are provided with shutters that protect the glass filters when not in use. These shutters must be removed at the time of measurement and be replaced after the measurements have been completed. Each metal holder bears a filter number (10, 20, or 30) and a set identification number. The upper left corner of each filter has been removed to indicate correct orientation in the metal holder. The certified transmittance values are given below.

Filter & Set Ident. Number	TRANSMITTANCE (T)					TRANSMITTANCE DENSITY ($-\log_{10}T$)				
	Wavelength and (Bandpass) nm					Wavelength and (Bandpass) nm				
	440.0 (2.2)	465.0 (2.7)	546.1 (6.5)	590.0 (5.4)	635.0 (6.0)	440.0 (2.2)	465.0 (2.7)	546.1 (6.5)	590.0 (5.4)	635.0 (6.0)

Date of Certification:

The uncertainty of the certified transmittance value is ±0.5 percent at the time of certification. This uncertainty includes the effects of the random and systematic errors of the calibration procedure, as well as possible transmittance changes of the filters during the period of calibration.

The transmittance values (T) can be converted to percent transmittance (%T) by multiplying by 100. The transmittance densities are calculated from the measured transmittance (T). These values should be indicated by the absorbance scale of the spectrophotometer if the filters are measured against air. The transmittance values given were measured against air at an ambient temperature of 22.5 °C.

Aging of the glass may cause some filters to change transmittance by about ±1 percent over a period of approximately one year from the date of calibration. Improper storage or handling of the filters may also cause changes [5]. It is recommended that the filters in the holders be handled only by the edges with soft plastic (polyethylene) gloves and optical lens tissue. When not in use they should be stored in their holders and in the container provided for this purpose. Extended exposure to laboratory atmosphere and dusty surroundings should be avoided. In cases where verification is desirable, the filters should be returned to the National Bureau of Standards for cleaning and recalibration.

The research, development, and initial production of this SRM were conducted by R. Mavrodineanu and J.R. Baldwin, NBS Inorganic Analytical Research Division.

The transmittance measurements were performed by R.W. Burke, M.V. Smith, and R. Mavrodineanu, NBS Inorganic Analytical Research Division. Technical leadership for the preparation and measurements leading to certification was provided by R.W. Burke.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J.R. DeVoe, NBS Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by L.J. Powell.

Gaithersburg, MD 20899
August 15, 1984
(Revision of Certificate
dated 8-1-77)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

The transmittance measurements were made using the high-accuracy spectrophotometer designed and built at the NBS Center for Analytical Chemistry [1]. This instrument represents the primary transmittance standard; its transmittance accuracy was established using the double-aperture method of linearity testing [1,3,5,6]. The estimated standard deviation obtained from a set of 20 measurements of the high-accuracy spectrophotometer used for transmittance measurements on glass filters of the SRM 930 type is 5×10^{-5} transmittance units for transmittance between 0.1 and 0.3. The bounds of the systematic error of the instrument are estimated to be $\pm 10^{-4}$ transmittance units.

The neutral glasses for the filters were provided by Schott of Mainz, Germany, and are designated as "Jena Color and Filter Glass." The glass material was selected for best homogeneity and minimum of inclusions and striae. The glass filters were aged at NBS for about one year prior to certification. They are of the type NG-4 and NG-5, and their nominal transmittances are 10, 20, and 30 percent [2,5]. The glasses were selected to provide a means to verify the transmittance scale of conventional spectrophotometers at three levels. The exposed surface of the glass is approximately 29 x 8 mm, measuring from a point 1.5 mm above the base of the filter holder (see figure). The empty filter holder is provided to be used in the reference beam of the spectrophotometer so that approximately equivalent conditions of stray radiation are achieved for both beams.

The transmittance of the filters depends upon the intrinsic properties of the material, wavelength, spectral bandpass, geometry of the optical beam, stray light, temperature, and positioning of the filter. A change of ambient temperature of 1 or 2 °C from 22.5 °C will not significantly affect the calibration [5]. Changes in the transmittance may be caused by surface conditions, aging of the glass, exposure to a harmful atmosphere, or careless handling as indicated on the face of this certificate [2,3,4,5]. The effective spectral bandpass used to determine the certified values was 0.8 nm. The spectral bandpass values indicated in parentheses in this certificate are maximum values that should not be exceeded when accurate measurements are contemplated. The transmittance measurements were made by producing the vertical image of the slit (about 8 mm by 0.5 mm), using a convergent beam geometry with an aperture ratio f:10, in the middle of the entrance face of the glass filter. The filters were measured in the spectrophotometer in a position perpendicular to the incident light beam as shown in the attached figure. Measurements performed outside these specified conditions will produce transmittance values that might differ from the certified data.

SRM 930D is stored in a black anodized aluminum container provided with a threaded cap made of the same metal. Each filter is placed in a cylindrical cavity to prevent any contact between the filter face and the walls of the storage container. Contamination of the glass filter surface with particulate matter due to static charge is minimized through the metallic nature of the container. Each filter holder is provided with a flat leaf spring which is inserted into the cylindrical cavity to minimize damage during transportation. These springs can be removed during normal use in the laboratory.

Prior to certification measurements, each filter was examined for surface defects and thoroughly cleaned [5]. Should the surface of the glass filter become contaminated, no attempt should be made to clean it unless the user has the facilities to demonstrate that the cleaning treatment has not altered the glass surface or degraded the accuracy of the certified values. As the Standard Reference Material is a transfer standard, the only means available to verify its integrity is remeasuring its transmittance with a primary standard instrument similar to that used in this certification [1,4,7]. In most cases where verification, or recertification, of the transmittance values is desirable, it will be most expeditious to return the filters to the National Bureau of Standards for measurement. Prior to shipment, the NBS Office of Standard Reference Materials should be contacted regarding the conditions and cost of verification and/or recertification.

We wish to acknowledge the cooperation of George N. Bowers, Jr., M.D., of Hartford Hospital, Hartford, Connecticut; Royden N. Rand, Ph.D., of the Eastman Kodak Co. Research Laboratories, Rochester, New York; and Donald S. Young, M.D., and Ph.D., of the Mayo Clinic, Rochester, Minnesota.

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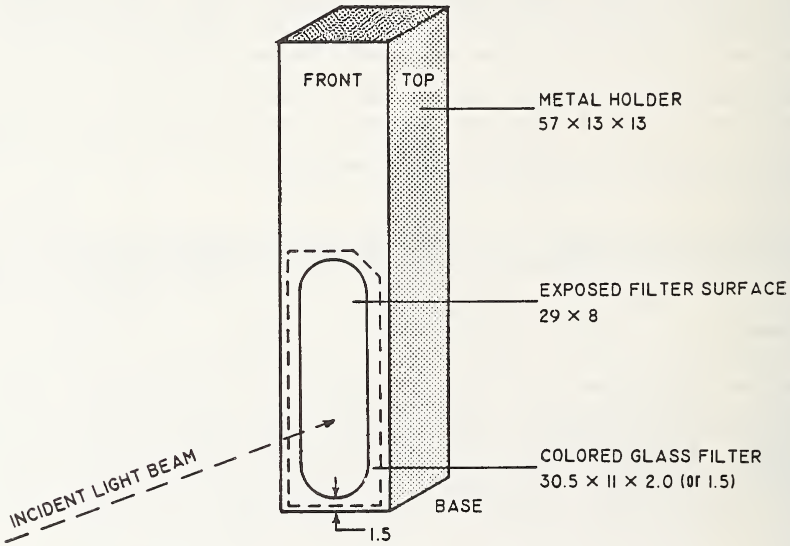
METAL HOLDER FOR THE COLORED GLASS FILTERS*



Nominal Dimensions in mm

*Shutters not illustrated.

U. S. Department of Commerce
Malcolm Baldrige
Secretary
National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 931c

Liquid Absorbance Standards for Ultraviolet and Visible Spectrophotometry

This Standard Reference Material is certified as solutions of known net absorbances at specific spectral wavelengths. It is intended primarily for use in the calibration and checking of accuracy of the photometric scale of narrow bandpass spectrophotometers and for routine critical evaluation of daily working standards used in spectrophotometry. This Standard Reference Material is applicable for calibrating those instruments that provide an effective spectral bandpass of 1.5 nm or less at 320 nm, 2.0 nm or less at 395 nm, 3.3 nm or less at 512 nm, and 8.5 nm or less at 678 nm [1].

Filter	Net Absorbance[a] Wavelength and (Bandpass), nm			
	302(0.8)	395 (0.8)	512(0.8)	678(0.8)
"I" -"Blank"	0.305 ± 0.001	0.309 ± 0.001	0.307 ± 0.001	0.116 ± 0.001
"II" -"Blank"	.608 ± .002	.612 ± .002	.609 ± .002	.232 ± .001
"III"-"Blank"	.938 ± .002	.912 ± .002	.904 ± .002	.346 ± .001

[a] Net absorbances ("I" - "Blank", "II" - "Blank", and "III" - "Blank") were determined using 10.00-mm cuvettes (SRM 932) at 25 °C. See Instructions for Use.

The transmittance measurements leading to the certification of this SRM were performed on the high accuracy spectrophotometer located in the NBS Center for Analytical Chemistry. The design and construction of this instrument have been described previously [2]. This instrument is the primary transmittance standard; its accuracy has been verified using the double aperature radiation-addition principle. The bounds of systematic error of this instrument are estimated to be $\pm 10^{-4}$ transmittance units.

These liquid absorbance filters were calibrated at the wavelengths and conditions indicated by measuring the transmittance, T, of the "Blank" and solutions "I", "II", and "III" against air as a reference. The values of T were used to calculate the corresponding apparent absorbances, A_a, using the relationship $A_a = -\log_{10} T$. The certified net absorbances were obtained by subtracting the apparent absorbances of the "Blank" solution from the apparent absorbances calculated for solutions "I", "II", and "III". The uncertainties of the certified values include all known sources of possible systematic error and the 95 percent confidence level for the mean.

While no long-term stability studies have been made on this lot (931c), studies on previous lots (931, 931a, and 931b) over three-year periods showed no degradation of the material when stored in the original sealed ampoules. Therefore, this material is certified only for use within three years following the date of purchase.

The preparation of the filters was performed by E.R. Deardorff, Inorganic Analytical Research Division, and the transmittance measurements were performed by M.V. Smith, Office of Standard Reference Materials. Technical leadership for the preparation and measurements leading to certification was provided by R.W. Burke, Inorganic Analytical Research Division.

The overall direction and coordination of technical measurements leading to certification were performed under the chairmanship of E.L. Garner.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.W. Seward.

Washington, D.C. 20234
February 12, 1982

George A. Uriano, Chief
Office of Standard Reference Materials

Temperature Dependence

Absorbances at various temperatures (17 to 35 °C) may be calculated using the equation

$$A_t = A_{25}[1+C_A(t-25)]$$

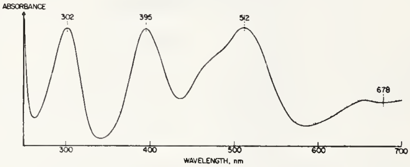
where: A_t = Absorbance at temperature t (°C)
A_{25} = Absorbance certified at 25.0 °C
C_A = Fractional change in absorbance per °C

The values of C_A, at the four wavelengths, are given below. [NOTE: At wavelength 302 nm, absorbance decreases with increasing temperature; at the other wavelengths, absorbance increases with increasing temperature.]

Wavelength, nm	C_A
302	−0.0014
395	+0.0014
512	+0.0018
678	+0.0014

Preparation of Filters

The filters were prepared by dissolving high-purity cobalt and nickel in a mixture of nitric and perchloric acids. The absorbance spectrum of the resulting solution is shown in the following figure. The maxima at 302 and 512 nm are due to absorbance by NO_3^- and $Co(H_2O)_6^{++}$ respectively. The maximum at 395 nm and the plateau at 650-700 nm are due to $Ni(H_2O)_6^{++}$ The pH of these solutions is about 1.



Instructions for Use

This material is for use as a spectrophotometric absorbance standard.

1. Select two clean 10.00 mm cuvettes free of scratches. At least one should be fitted with a ground glass or Teflon stopper to minimize evaporation. Reserve it for all sample measurements.
2. Mark each of the cuvettes to assure the same orientation in the spectrophotometer.
3. Place the cuvettes in their respective holders and fill with distilled water. (Borosilicate Pasteur-type pipettes fitted with rubber bulbs are recommended for transferring all solutions to and from the cuvettes. Soft glass pipettes, which are available commercially, contain residual amounts of ultraviolet absorbing material, but may be used after proper cleaning. Several rinses, first with isopropyl alcohol and then with distilled water, are generally adequate.
4. Obtain the optical mismatch of the cuvettes at 302, 395, 512, and 678 nm, using the spectral bandpass limitations given on the face of the certificate.
5. Empty the cuvettes by suction without removing them from their holders, refill with distilled water and measure the absorbances again at each of the above wavelengths.

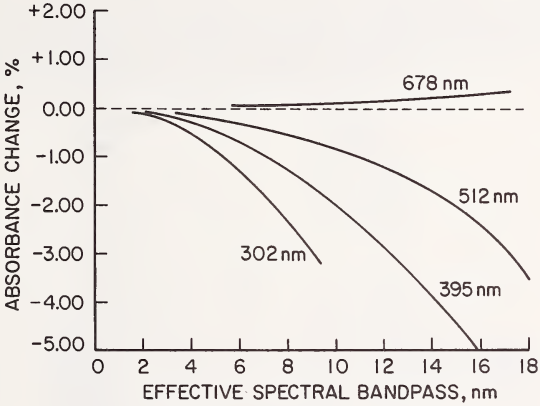
6. Repeat the emptying and refilling operation until constant absorbance readings are obtained.
7. Using the liquid filters provided, measure, in turn, the absorbance of the "Blank", "I", "II", and "III" against distilled water. Shake each ampoule before opening to remix any condensate which may have been collected in the neck. (The ampoules have been prescored directly below the gold band to facilitate opening.)
8. Subtract the appropriate "Blank" reading from the absorbances obtained for "I", "II", and "III". These net absorbances should agree with the certified values within the uncertainties specified. Consult the manufacturer of the instrument if they do not.

The above instructions are for use with the standard 10-mm rectangular cuvette. For calibration of the several spectrophotometric systems used in various automated instruments, the user is referred to the instruction manual for the particular instrument.

The absorbances of these liquid absorbance standards will depend not only on the accuracy of the photometric scale, but also on the wavelength accuracy and the spectral bandpass. A mercury lamp is recommended for checking the wavelength scale. In addition, for those spectrophotometers having a hydrogen (H) or deuterium (D) source, the two emission lines at 486.1 and 656.3 nm (H) or 486.0 and 656.1 nm (D) may provide a convenient check at these wavelengths.

To ensure that the measured absorbances are not significantly different from the certified values, the following restrictions are placed on the size of the spectral bandbass selected: To obtain ±0.1 percent of the true value, the effective spectral bandpass should not exceed 1.5, 2.0, 3.3, and 8.5 nm at 302, 395, 512, and 678 nm, respectively.

For ±0.2 percent, the respective bandpasses should not exceed 2.2, 2.9, 4.8, and 12.3 nm. Additional information on the effect of spectral bandpass on the absorbances of these filters is given in the figure below. These curves are not to be used, however, to correct the measured absorbances.



This Standard Reference Material should be kept in the original sealed ampoules. Once opened, the material should be used immediately. No attempt should be made to reseal the ampoule. In addition, it is recommended that this Standard Reference Material not be used after three years from the purchase date.

References

[1] R. W. Burke, E. R. Deardorff, and O. Menis, J. Research, Nat. Bur. Stand. 76A, 469-482 (1972).

[2] R. Mavrodineanu, J. Research, Nat. Bur. Stand. 76A, 405-425 (1972).

Note: The above papers are also published in NBS Special Publication 378, Accuracy in Spectrophotometry and Luminescence Measurements, R. Mavrodineanu, J. I. Shultz, and O. Menis, Editors, U.S. Government Printing Office, Washington, D.C. 20402, 1973.

U. S. Department of Commerce
Philip M. Klutznick
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 932

Quartz Cuvette for Spectrophotometry

R. Mavrodineanu and J. W. Lazar

This Standard Reference Material consists of a single, accurately calibrated cuvette that is issued for use in the production of accurate spectrophotometric data on liquids. The design and nominal dimensions of the all-quartz cuvette are shown in Figure 1. The pathlength of the cuvette is defined by the distances between the two optically transparent windows measured at several heights within the cuvette. Cuvettes issued as Standard Reference Material 932 have a nominal pathlength of 10 mm. The pathlength and parallelism are certified, at the time of measurements, with an uncertainty of ±0.0005 mm as determined by measurements (at 20 °C) taken at the positions indicated below.

Cuvette number is issued with this certificate. For this cuvette the following measurements were obtained:

Height mm	Pathlength mm

Date of Certification:

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of O. Menis and J. A. Simpson.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D.C. 20234
December 2, 1980
(Revision of certificates
dated 11-5-73, 6-16-77, and 8-2-78)

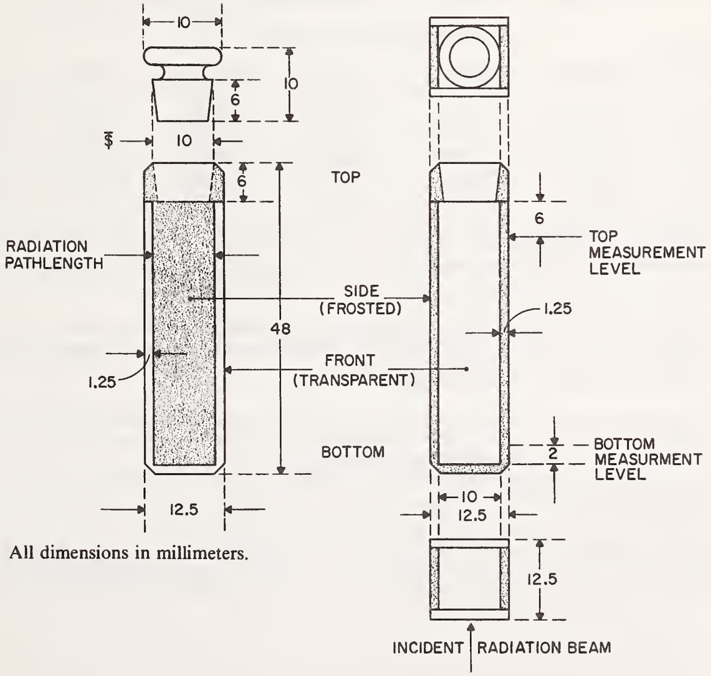
George A. Uriano, Chief
Office of Standard Reference Materials

The original cuvettes were designed and produced at the National Bureau of Standards (NBS) using special techniques and non-fluorescent optical-quality fused quartz as described in NBS SP 260-32[1]. The transparent windows are attached to the body of the cuvette by direct fusion, and the unit was stress-relived by annealing. The radiation pathlength measurements of the cuvette were performed using electronic feeler-gauge type instruments capable of a resolution of 5 parts in 10^6. To preserve the integrity of the certified values, the cuvette must be handled with great care and should be held only by the frosted-quartz side windows. When not in use, it should be stored in the container provided for this purpose. Extended exposure to laboratory atmosphere and dusty surroundings is to be avoided. Improper handling of the cuvette and the use of solutions that can corrode or errode the quartz could degrade the certified pathlength values. In cases where verification of the pathlength is desirable, the cuvette should be returned to NBS for examination and, if needed, recalibration. Prior to shipment the NBS Office of Standard Reference Materials should be contacted regarding the conditions and cost of the verification and/or recertification.

The development and production of SRM 932 is a result of the combined efforts of the Center for Analytical Chemistry. the Center for Mechanical Engineering and Process Technology, and the Instrument Shops Division. The radiation pathlength measurements of all of these cuvettes have been performed, at NBS, by E. G. Erber of the Mechanical Processes Division.

Reference:

[1] Mavrodineanu, R., and Lazar, J. W., Standard Reference Materials; Standard Quartz Cuvettes for High Accuracy Spectrophotometry, Nat. Bur. Stand. (U.S.), Spec. Publ. 260-32 (December 1973). Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402, 55 cents.



All dimensions in millimeters.

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Acting Director

National Bureau of Standards
Certificate

Standard Reference Material 935

Crystalline Potassium Dichromate
for Use as an Ultraviolet Absorbance Standard

R. W. Burke and R. Mavrodineanu

This Standard Reference Material consists of crystalline potassium dichromate of established purity. Solutions of known concentrations of this SRM in 0.001 N perchloric acid are certified for their apparent* specific absorbances**, ϵ_a, at 23.5 °C.

This SRM is intended to be used as a reference standard for the verification of the accuracy and linearity of the absorbance scale of absorption spectrometers that can provide an effective spectral bandpass of 1.2 nm or less at 235 nm, and 0.8 nm or less at 257, 313, 345, and 350 nm. Such verification is accomplished by comparing the measured apparent absorbances, A_a, to the A_a calculated from the certified ϵ_a values as described under "Instructions for Use."

Table 1 gives the certified values of ϵ_a in kg·g^{-1}·cm^{-1} for five concentrations of the SRM 935 potassium dichromate in 0.001 N perchloric acid at 23.5 °C and the indicated wavelengths and spectral bandpasses for a 1-cm internal pathlength.

Table 1. ϵ_a, Apparent Specific Absorbance,[a] kg·g^{-1}·cm^{-1}

Nominal Concentration g·kg^{-1}	Wavelength and (Bandpass) nm					
	235.0(1.2)	257.0(0.8)	313.0(0.8)	345.0(0.8)[b]	350.0(0.8)	Uncertainty[c]
0.020	12.260	14.262	4.805	10.604	10.672	± 0.034
.040	12.304	14.318	4.811	10.603	10.682	± .020[d]
.060	12.347	14.374	4.816	10.602	10.692	± .020[d]
.080	12.390	14.430	4.821	10.601	10.701	± .020[d]
.100	12.434	14.486	4.827	10.600	10.711	± .020[d]

[a] ϵ_a values are given to the third decimal place to preserve the smooth variation of the data with concentration, although the uncertainties are in the second decimal place.

[b] Wavelength 345.0 nm is near one of the two isosbestic points in $HCrO_4^-/Cr_2O_7^=$ spectra. Because it is on the slope of the composite spectrum, reproduction of the ϵ_a values is dependent on wavelength accuracy. Measurements at this wavelength should be made only for verification of the linearity of the absorbance scale.

[c] ϵ_a values are not corrected for the effects of internal multiple reflections within the cuvette, nor have the weights been corrected to vacuum. With these two exceptions, the uncertainties given include all known systematic errors and the 95 percent confidence interval of the mean.

[d] At wavelength 313.0 nm, the uncertainty is reduced to ±0.010.

*The term "apparent" is used because no corrections have been applied to the data for the effects of internal multiple reflections within the cuvette or for buoyancy, i.e., the weights used to express concentrations have not been corrected to vacuum. These combined corrections do not exceed 0.2 percent. The specific absorbances are given in reference 1.

The nomenclature used in this certificate is that recommended by K. D. Mielenz, Anal. Chem. **48, 1093-1094 (1976), which is reproduced in the Appendix of NBS Special Publication 260-54.

Washington, D.C. 20234
June 1, 1977

J. Paul Cali, Chief
Office of Standard Reference Materials

The overall direction and coordination of the technical measurements leading to this certificate were performed under the joint chairmanship of I. L. Barnes and J. R. DeVoe.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. W. Seward.

We wish to acknowledge: The contributions of K. D. Mielenz, NBS Analytical Chemistry Division, for his many valuable discussions and suggestions, and H. H. Ku, NBS Applied Mathematics Division, for his statistical treatment of the data; the cooperation and early support of the National Institute of General Medical Sciences in the research leading to this SRM; and the encouragement of George N. Bowers, Jr., M.D., Hartford Hospital, Hartford, Conn., and Royden N. Rand, Ph.D., Eastman Kodak Co., Research Laboratories, Rochester, N.Y.

PREPARATION AND CERTIFICATION

The details of the preparation and certification of SRM 935 are provided in NBS Special Publication 260-54, Certification and Use of Acidic Potassium Dichromate Solutions as an Ultraviolet Absorbance Standard [2]. This publication should be referred to every time SRM 935 is to be used. Briefly, the transmittances, T, of the solutions prepared from the undried, as received, material were measured with the NBS Institute for Materials Research high-accuracy transmission spectrometer [3].

The ϵ_a values were calculated for each wavelength using the relation:

$$\epsilon_a = \frac{D_s - D_b}{b \times c} = \frac{A_a}{b \times c} \qquad (1)$$

where:

ϵ_a = apparent specific absorbance
A_a = apparent absorbance
D_s = transmittance density of the sample solution, $-\log_{10}T_s$
D_b = transmittance density of the blank solution, $-\log_{10}T_b$
b = internal cuvette pathlength, cm
c = concentration, by weight, of $K_2Cr_2O_7$ solution, $g \cdot kg^{-1}$

The crystalline potassium dichromate used for SRM 935 is a special lot of analytical reagent grade material obtained from the J. T. Baker Chemical Co., Phillipsburg, N.J.

Assay: A coulometric assay of the purity of the undried material was performed by G. Marinenko of the NBS Analytical Chemistry Division. The purity of SRM 935, expressed as an oxidant, was found to be 99.972 ± 0.005 percent where the uncertainty figure represents the 95 percent confidence interval for the mean based on 11 degrees of freedom. In addition, the material was examind by optical emission spectrometry for trace elemental impurities by J. A. Norris of the NBS Analytical Chemistry Division. The only significant impurities detected were sodium and rubidium. Their concentrations were estimated to be in the range of 0.02 and 0.03 percent, respectively. Drying at 105 °C for 12 hours showed that the surface moisture of this material was less than 0.01 percent.

Stability: Solutions prepared from SRM 935 in the concentration range indicated in table 1 and made according to the instructions given in NBS SP 260-54 have been found to be stable within the uncertainties given in table 1 for at least six months when stored at room temperature and protected from evaporation and exposure to light.

INSTRUCTIONS FOR USE

The use of SRM 935 as an absorbance standard requires the careful preparation of a series of solutions of known concentrations, c, of the potassium dichromate in 0.001 N perchloric acid. These solutions are transferred to a quartz cuvette of known pathlength, b, and their apparent absorbances measured at wavelengths 235, 257, 313, and 350 nm, using the spectral bandpass requirements given in table 1. The preparation and measurement of these solutions are described in detail in Section 5 of NBS SP 260-54.

The accuracy of the absorbance scale of the spectrometer being tested is ascertained by comparing the measured apparent absorbances, A_a, of a series of 0.001 N perchloric acid solutions containing 0.020 to 0.100 gram $K_2Cr_2O_7$/kg to the A_a values calculated from the certified ϵ_a values. Although the ϵ_a data in table 1 are given for nominal concentrations of 0.020, 0.040, 0.060, 0.080, and 0.100 g $K_2Cr_2O_7$/kg, the ϵ_a values for concentrations between these nominal concentrations can be determined by linear interpolation. Using the appropriate ϵ_a values, the calculated A_a values at 23.5 °C are obtained from the expression:

$$A_a = \epsilon_a \times b \times c \qquad (2)$$

Calculations:

An example of the calculation of A_a for one concentration of $K_2Cr_2O_7$ under a specified set of conditions is shown below. Calculations of A_a for other concentrations and wavelengths are performed in a similar manner.

Conditions: Wavelength = 350 nm, spectral bandpass 0.8 nm or less
$b = 0.9982$ cm
$c = 0.04375\ \mathrm{g{\cdot}kg^{-1}}$
$t = 23.5$ °C

From column 6, table 1, the ϵ_a for concentrations of 0.040 and 0.060 $\mathrm{g{\cdot}kg^{-1}}$ are 10.682 and 10.692, respectively. The corresponding ϵ_a for $c = 0.04375\ \mathrm{g{\cdot}kg^{-1}}$ is:

$$\epsilon_a = 10.682 + \frac{0.04375 - 0.040}{0.060 - 0.040}(10.692 - 10.682)$$

$$\epsilon_a = 10.682 + 0.0019$$

$$\epsilon_a = 10.684$$

The calculated apparent absorbance, A_a, from equation 2, is:

$$A_a = 10.684 \times 0.9982 \times 0.04375$$

$$A_a = 0.4666$$

The uncertainty, ΔA_a, in the calculated A_a is determined from the combined uncertainties in ϵ_a, b, and c in equation 2, provided no other systematic errors are present. Thus:

$$\Delta A_a = bc\left|\Delta\epsilon_a\right| + \epsilon_a c\left|\Delta b\right| + \epsilon_a b\left|\Delta c\right| \qquad (3)$$

To evaluate ΔA_a, $\Delta\epsilon_a$ is taken from column 7 of table 1 and the Δb and Δc values must be determined experimentally.

In the experiments performed to obtain the ϵ_a values in table 1, the uncertainties for b and c did not exceed 1 part in 10^4 and 2 parts in 10^4, respectively.

The solution of equation 3 gives:

$$\Delta A_a = 1(0.044)\,(0.020) + 10.7(0.044)\,(0.0001) + 10.7(1)\,(0.0000088)$$
$$= 0.0010$$

Thus, the uncertainty of A_a, for the above set of conditions, is ±0.0010.

The correction of the absorbance scale of the absorption spectrometer under test is determined by plotting the differences between A_a measured and A_a calculated as a function of absorbance. A typical plot of such a graph is shown in figure 1. The apparent absorbances measured on this instrument at 350 nm are accurate when the indicated correction is subtracted from the corresponding absorbance scale reading, provided that the conditions of wavelength accuracy, spectral bandpass, and absence of stray light are fulfilled as specified in NBS SP 260-54. Correction curves for wavelengths 235, 257, and 313 nm are obtained in a similar manner.

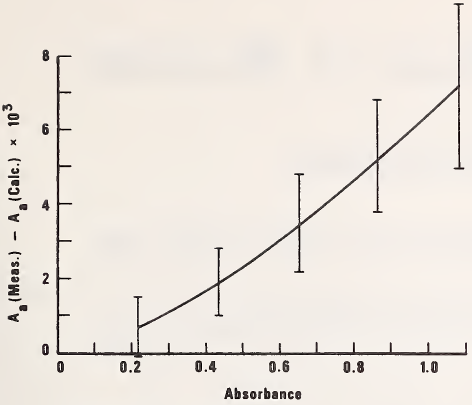


Figure 1. Correction curve for the absorbance scale of a precision spectrometer. The error bars are the sum of the errors arising from the uncertainties in the certified apparent specific absorbances, ϵ_a, cuvette pathlength, b, and concentration, c.

Temperature Correction:

Although ϵ_a values in table 1 are certified at 23.5 °C, SRM 935 can be used as an absorbance standard at other temperatures in the range 20 to 30 °C provided corrections are made to the ϵ_a values. Over this range the apparent specific absorbances decrease linearly with increasing temperature for all the wavelengths given in table 1. The corresponding temperature coefficients, k, for these wavelengths are given in table 2.

Table 2. Variation of ϵ_a with Temperature Over the Range 20 to 30 °C.

λ, nm	Temperature Coefficient, k Percent per degree Celsius
235	−0.05
257	−0.05
313	−0.02
345	−0.08
350	−0.05

The value of ϵ_a at any temperature in the range 20 to 30 °C can be calculated from the certified value and the appropriate temperature coefficient using the relation:

$$\epsilon_a^t = \epsilon_a^{23.5} \left[1 + \frac{k}{100} (t - 23.5) \right]$$

where: ϵ_a^t = apparent specific absorbance at temperature t (°C)

$\epsilon_a^{23.5}$ = apparent specific absorbance certified at 23.5 °C.

k = temperature coefficient, percent per °C.

REFERENCES

1. Burke, R. W., and Mavrodineanu, R., Acidic Potassium Dichromate Solutions as Ultraviolet Absorbance Standards, J. Res. Nat. Bur. Stand. (U.S.), **80A** (Phys. and Chem.), No. 4, 631-636 (July-Aug. 1976).
2. Burke, R. W., and Mavrodineanu, R., Certification and Use of Acidic Potassium Dichromate Solutions as an Ultraviolet Absorbance Standard, NBS Spec. Publ. 260-54 (1977). Copies may be obtained from the Office of Standard Reference Materials, National Bureau of Standards, Washington, D.C. 20234.
3. Mavrodineanu, R., An Accurate Spectrophotometer for Measuring the Transmittance of Solid and Liquid Materials, J. Res. Nat. Bur. Stand. (U.S.), **76A** (Phys. and Chem.), No. 5, 405-425 (1972).

U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 936

Quinine Sulfate Dihydrate

R. A. Velapoldi and K. D. Mielenz

This Standard Reference Material is intended for use in the evaluation of methods and the calibration of fluorescence spectrometers. It is certified for the relative molecular emission spectrum, E(λ), in radiometric units for a solution of 1.28×10^{-6} mol/L quinine sulfate dihydrate in 0.105 mol/L perchloric acid using an excitation wavelength of 347.5 nm. The certified values of the molecular emission spectrum at 5 nm wavelength intervals from 375 to 675 nm are given in table 1. These values have been corrected for instrument and sample parameters, including the spectral responsivity of the detection system, monochromator bandwidth, photomultiplier tube nonlinearity, monochromator wavelength error, solvent refractive index, and cell window transmittance. The relative standard error in E(λ), RSE [E(λ)], is given in table 1. The estimate of the relative systematic error limits in the molecular emission spectrum, RSEL [E(λ)], is also given in table 1 and was determined by the addition of the absolute values of the estimated systematic errors. These relative error limits include uncertainties in the calibration values for the spectral responsivity, the wavelength position of the emission peak maximum, and in the corrections applied for instrument and sample parameters.

From the certified values of E(λ), values may be calculated for the molecular emission spectrum in the various photon, radiometric, wavelength, and wavenumber units using the following equation: [1,2]

$$E(\lambda) = \frac{E_p(\lambda)}{\lambda} = \frac{E(\tilde{\nu})}{\lambda^2} = \frac{E_p(\tilde{\nu})}{\lambda^3}$$

These values have been calculated and are given in NBS Special Publication 260-64.

The technical emission spectrum, $E^T(\lambda)$, i.e., the emission spectrum corrected for instrument parameters only, is also given in SP 260-64. The quinine sulfate dihydrate used for SRM 936 was a special lot of material obtained from the J. T. Baker Chemical Co., Phillipsburg, N.J.

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears and R. W. Seward.

Washington, D.C. 20234
April 1, 1979

George A. Uriano, Chief
Office of Standard Reference Materials

Table 1. The Molecular Emission Spectrum, E(λ), of Quinine Sulfate Dihydrate in 0.105 mol/L $HClO_4$, the Relative Standard Error, RSE, and the Estimated Relative Systematic Error Limits, RSEL, in the E(λ) Values.

λ,nm	E(λ)	RSE [E(λ)]	RSEL [E(λ)]	λ,nm	E(λ)	RSE [E(λ)]	RSEL [E(λ)]
375.0	0.005	0.019	0.087	525.0	0.302	0.001	0.029
380.0	.012	.006	.078	530.0	.264	.003	.029
385.0	.028	.003	.071	535.0	.231	.003	.029
390.0	.057	.003	.064	540.0	.201	.002	.029
395.0	.103	.002	.059	545.0	.175	.002	.029
400.0	.170	.002	.054	550.0	.153	.001	.029
405.0	.257	.003	.049	555.0	.132	.001	.029
410.0	.359	.003	.045	560.0	.116	.001	.029
415.0	.471	.003	.041	565.0	.101	.002	.029
420.0	.586	.003	.037	570.0	.088	.002	.029
425.0	.694	.003	.034	575.0	.076	.003	.029
430.0	.792	.002	.031	580.0	.065	.003	.029
435.0	.874	.002	.028	585.0	.057	.001	.029
440.0	.940	.001	.026	590.0	.050	.003	.030
445.0	.984	.001	.024	595.0	.043	.004	.030
450.0	.999	.001	.023	600.0	.037	.006	.030
455.0	.997	.001	.023	605.0	.032	.002	.030
460.0	.982	.001	.024	610.0	.028	.006	.030
465.0	.947	.001	.024	615.0	.024	.003	.030
470.0	.897	.001	.025	620.0	.021	.011	.030
475.0	.838	.002	.026	625.0	.018	.003	.030
480.0	.782	.002	.027	630.0	.016	.015	.030
485.0	.719	.002	.027	635.0	.014	.014	.030
490.0	.657	.002	.027	640.0	.011	.037	.030
495.0	.595	.003	.027	645.0	.010	.015	.030
500.0	.541	.002	.027	650.0	.009	.027	.030
505.0	.486	.001	.028	655.0	.008	.035	.031
510.0	.434	.003	.028	660.0	.007	.073	.031
515.0	.386	.003	.028	665.0	.006	.046	.032
520.0	.342	.002	.028	670.0	.005	.053	.032
				675.0	.004	.065	.033

SUPPLEMENTARY DATA

The following data for the specific molar absorbances, water content, photon yields, and fluorescence lifetimes are considered to be supplementary and are not to be considered certified values.

The quinine sulfate dihydrate (QSD) used for SRM 936 was found to be homogeneous to better than 0.5% by thin-layer chromatography with development by two solvent systems and the determination of specific molar absorbances, ϵ, at three different wavelengths. The SRM contains approximately 1.7% of an impurity as determined by high performance liquid chromatography using absorbance and fluorescence detection. This impurity is believed to be dihydroquinine sulfate dihydrate, which has optical characteristics that are similar to those of the quinine sulfate dihydrate. The ultraviolet absorption spectrum of SRM 936 in 0.105 mol/L $HClO_4$ exhibits the following absorption maxima:

$$250.0\ \text{nm},\ \epsilon_{max} = 56{,}990 \pm 90\ \text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$$

$$347.5\ \text{nm},\ \epsilon_{max} = 10{,}810 \pm 20\ \text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$$

and, on the side of a peak:

$$365.0\ \text{nm},\ \epsilon_{obs} = 6{,}920 \pm 10\ \text{L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$$

The water content of this material was measured by two methods. The average of six determinations by the Karl-Fischer method gave a value of (4.74 ± 0.05%), while the average of four determinations by a weight loss procedure gave a value of (4.57 ± 0.04%). The theoretical value for water in quinine sulfate dihydrate is 4.60%.

The photon yield, Q, and the fluorescence lifetime, τ, of SRM 936 were compared to values obtained for a sample of purified quinine sulfate dihydrate and are summarized below:

	Q 0.5 mol/L H_2SO_4	τ, ns 0.5 mol/L H_2SO_4
SRM 936, QSD	0.544 ± 0.03	19.1 ± 0.1
Purified QSD	0.546[a]	19.2 ± 0.1

[a] Melhuish, W. H., J. Phys. Chem. 65, 229 (1961); *ibid,* New Zealand J. Sci. Tech. 37, 142 (1955).

PREPARATION AND USE OF SRM 936

This Standard Reference Material is for "in vitro" diagnostic use as a clinical laboratory standard. A "stock" standard solution containing 0.1 mg/mL of quinine sulfate may be prepared as follows: Weigh 0.100 g of SRM 936 to the nearest one-tenth milligram and quantitatively transfer it to a 1000-mL volumetric flask. Dilute to the calibrated volume with 0.105 mol/L $HClO_4$, to give a solution that is 1.28×10^{-4} mol/L (0.1 mg/mL) in quinine sulfate. Store this solution in the dark in a well-stoppered, glass bottle. A "working" standard solution containing 1 μg/mL may be prepared by transferring 10 mL of the above "stock" standard solution to a 1000-mL volumetric flask and diluting to the calibrated volume with 0.105 mol/L $HClO_4$ to give a solution that is 1.28×10^{-6} mol/L (1 μg/mL) in quinine sulfate. Store this solution in the same manner as the above "stock" standard solution.

Several opinions regarding the stability of quinine sulfate solutions have appeared in the literature [3]. NBS considers the 0.1 mg/mL "stock" standard solution prepared from SRM 936 to be stable for 3 months when stored as specified; and the 1 μg/mL "working" standard solution to be stable for 1 month when so stored.

SRM 936 should be kept in its original bottle and stored in the dark at room temperature (30 °C or less). It should not be subjected to heat or direct sunlight during storage. Experience at NBS indicates that under proper storage this material is stable for at least 3 years. If this material degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that the material not be used after 3 years from the date of purchase.

References:

[1] Ejder, E. J., J. Opt. Soc. Amer. **59**, 223 (1969).

[2] Melhuish, W. H., J. Res. Nat. Bur. Stand. (U.S.) **76A**, No. 6, 547 (1972).

[3] Melhuish, W. H., J. Phys. Chem. **65**, 229 (1961); Gill, J. E., Photochem. and Photobiol. **9**, 313 (1969); Birks, J. B., J. Res. Nat. Bur. Stand. (U.S.) **80A**, 389 (1976); Heller, C. A., Henry, R. A., McLaughlin, B. A., and Bless, D. E., J. Chem. Eng. Data **19**, 214 (1974); West, M. A., and Kemp, D. R., Int'l. Lab., p. 27 (May/June 1976); and White, J. U., Pittsburgh Conf. Abstracts, Paper 488 (1977).

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Washington, D.C. 20234

The date of issuance and certification of SRM 936 was April 1, 1979

U. S. Department of Commerce
Malcolm Baldrige
Secretary
National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 2010a

Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers

SERIAL NO. ____________

Kenneth L. Eckerle

This Standard Reference Material (SRM) is a 51 x 51 x 3 mm didymium glass filter. SRM 2010a is intended for use in calibrating the wavelength scale in the visible wavelength region of scanning spectrophotometers having nominal bandwidths in the range 1.5 to 10.5 nm. Depending upon the bandwidth of the spectrophotometer, 12 to 22 wavelength corrections can be determined from 389 to 760 nm. Detailed instructions on the use of this SRM and examples of its use are given in the accompanying NBS Special Publication 260-66. Each didymium glass filter is identified by serial number.

All measurements on these filters were made at 25 °C with a high-accuracy reference spectrophotometer that has a wavelength accuracy of 0.04 nm.

Table 1 gives the wavelengths of the transmittance minima as obtained from measurements on two filters representative of this melt of didymium glass. These values are given for seven equally spaced values of the half-height width of triangular passbands. The minima numbers are shown on Figure 1, which illustrates the spectral transmittance as a function of wavelength.

Table 2 gives the wavelength values of nine points of inflection on the spectral transmittance curve as obtained on two filters. These inflection points are representative of this glass melt and are also shown on Figure 1. These inflection points should only be used with the transmittance minima as described in Sections 2.2 and 2.3 in SP 260-66. Table 2 also indicates the range of the measured wavelengths of the inflection points.

Table 3 gives the estimated random and systematic errors of the transmittance minima given in Table 1 (as obtained from 4 sets of measurements on a single filter).

Table 4 gives the spectral transmittance as a function of wavelength for a filter representative of this melt. These values are not certified but are provided for use as specified in SP 260-66. They should not be used to check the photometric scale of a spectrophotometer.

Trial calibrations made on several instruments, using both minima and inflection points, indicate that wavelength corrections made with these SRM's can be accurate to 0.2 nm. The uncertainty of a calibration, however, will depend upon the stability and other characteristics of a particular instrument.

This filter should be handled only by its edges; when not in use it should be stored in the box provided. If cleaning is necessary, wet the filter with water and rub gently with optical lens tissue soaked with a mild soap solution, rinse with distilled water, rinse with isopropyl alcohol, and rinse again with distilled water. Dry after each rinsing by wiping lightly with optical lens tissue.

July 25, 1984
Gaithersburg, MD 20899

Stanley D. Rasberry, Chief
Office of Standard Reference Material

Figure 1. Spectral transmittance as a function of wavelength. The numbers indentify minima, and the letters identify selected inflection points.

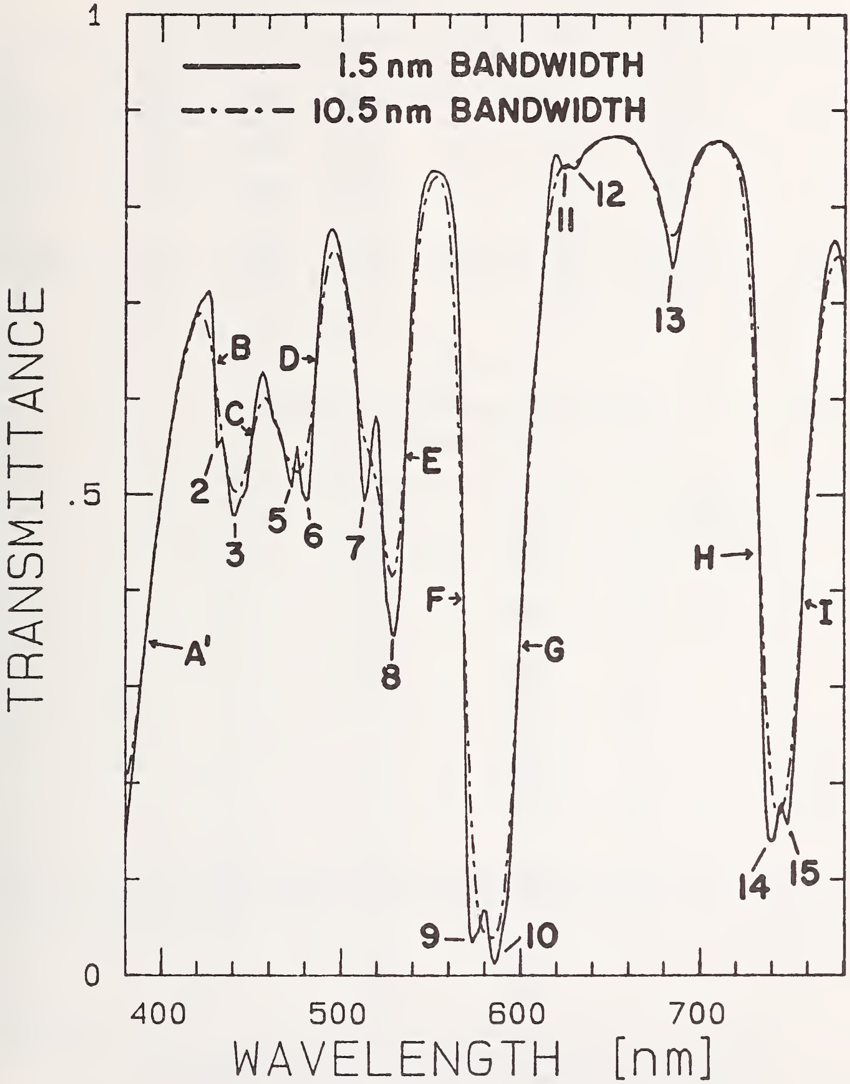


TABLE 1

Certified Wavelengths (nm) of the Transmittance Minima for the Indicated Bandwidths

Bandwidth Minimum No.	1.5 nm	3.0 nm	4.5 nm	6.0 nm	7.5 nm	9.0 nm	10.5 nm
1	- -	- -	- -	- -	- -	- -	- -
2	431.58	- -	- -	- -	- -	- -	- -
3	440.78	440.98	441.41	441.94	442.19	442.17	441.87
4	- -	- -	- -	- -	- -	- -	- -
5	472.67	472.43	472.31	- -	- -	- -	- -
6	481.20	480.68	480.37	479.67	478.36	477.26	476.18
7	513.66	513.76	513.94	514.25	514.87	- -	- -
8	529.37	529.47	529.26	529.11	529.08	528.98	528.74
9	573.12	573.88	574.75	575.72	- -	- -	- -
10	585.50	585.68	585.86	586.03	585.91	585.29	584.39
11	623.85	624.42	- -	- -	- -	- -	- -
12	629.51	629.26	628.17	627.17	627.17	- -	- -
13	684.71	684.74	684.78	684.80	684.77	684.73	684.65
14	739.61	740.20	740.58	741.33	742.55	743.50	744.16
15	748.64	748.48	- -	- -	- -	- -	- -

TABLE 2

Wavelengths and Transmittances at Nine Selected Points of Inflection

Point Identification	Wavelength (nm)	Range* (nm)	Transmittance+
A	388.68	+.02 −.02	0.3027
B	429.67	+.06 −.10	.6268
C	450.21	+.03 −.03	.5569
D	485.75	+.08 −.12	.6210
E	536.39	+.04 −.06	.5597
F	568.40	+.06 −.03	.3969
G	599.57	+.02 −.02	.3375
H	733.55	+.05 −.03	.4493
I	757.22	+.01 −.02	.3959

*The range of wavelengths within which the wavelength for the given transmittance will fall for symmetric triangular passbands with half-height bandwidths from 1.5 to 10.5 nm.

+These values of transmittance are not certified.

TABLE 3

Estimated Random and Systematic Errors of the Transmittance Minima

Band Number	Nominal Wavelength of Minimum Transmittance	Standard Deviation for Indicated Bandwidth						
		1.5 nm	3.0 nm	4.5 nm	6.0 nm	7.5 nm	9.0 nm	10.5 nm
1	402 nm	- -	- -	- -	- -	- -	- -	- -
2	431	0.013 nm (0.06)*	0.010 nm	- -	- -	- -	- -	- -
3	440	0.021 (0.05)	0.013	0.050 nm	0.028 nm	0.009 nm	0.005 nm	0.007 nm (0.25)
4	446	- -	- -	- -	- -	- -	- -	- -
5	473	0.011 (0.08)	0.012	0.032	- -	- -	- -	- -
6	481	0.015 (0.15)	0.013	0.009	0.009	0.009	0.014	0.017 (0.25)
7	513	0.022 (0.08)	0.016	0.014	0.013	0.010	- -	- -
8	530	0.012 (0.21)	0.010	0.010	0.011	0.010	0.011	0.010 (0.25)
9	573	0.004 (0.05)	0.010	0.010	0.012	0.014	- -	- -
10	585	0.007 (0.06)	0.004	0.007	0.008	0.008	0.007	0.007 (0.25)
11	624	0.058 (0.06)	0.061	- -	- -	- -	- -	- -
12	630	0.210 (0.02)	0.120	0.171	0.133	0.091	- -	- -
13	685	0.019 (0.05)	0.029	0.024	0.017	0.014	0.014	0.012 (0.25)
14	740	0.009 (0.25)	0.013	0.011	0.010	0.009	0.009	0.010 (0.25)
15	748	0.020 (0.04)	0.016	- -	- -	- -	- -	- -

* Values in parentheses are estimates of the systematic error. The estimates for the 1.5 nm bandwidth were obtained from the data taken at 1.5 nm intervals as compared to data taken at 0.15 nm intervals. The method of estimating the systematic errors for the 10.5 nm bandwidth is described in Section 3.4 in SP 260-66.

TABLE 4

Transmittance (T) as a function of wavelength from 380 to 780.5 nm for a bandwidth of 1.5 nm. The estimates of random and systematic errors are also provided. These data are not certified.

Wavelength (nm)	T	Δ_T Standard Error	Δ_T Systematic	Wavelength (nm)	T	Δ_T Standard Error	Δ_T Systematic
380.00	.14580	.000027	.00010	381.50	.17003	.000030	.00010
383.00	.19554	.000013	.00010	384.50	.22249	.000015	.00010
386.00	.25140	.000030	.00010	387.50	.28048	.000036	.00010
389.00	.30929	.000054	.00010	390.50	.33718	.000059	.00010
392.00	.36465	.000036	.00010	393.50	.39159	.000027	.00010
395.00	.41903	.000065	.00010	396.50	.44370	.000034	.00010
398.00	.46633	.000062	.00010	399.50	.48705	.000055	.00010
401.00	.50766	.000057	.00010	402.50	.52624	.000068	.00010
404.00	.54799	.000057	.00010	405.50	.56861	.000032	.00010
407.00	.58745	.000094	.00010	408.50	.60604	.000060	.00010
410.00	.62234	.000038	.00010	411.50	.63656	.000068	.00010
413.00	.64951	.000059	.00010	414.50	.66131	.000065	.00010
416.00	.67152	.000046	.00010	417.50	.67987	.000057	.00010
419.00	.68701	.000067	.00010	420.50	.69555	.000103	.00010
422.00	.70151	.000030	.00010	423.50	.70430	.000041	.00010
425.00	.70965	.000033	.00010	426.50	.71287	.000023	.00010
428.00	.70285	.000049	.00010	429.50	.63752	.000022	.00010
431.00	.54947	.000024	.00010	432.50	.55494	.000013	.00010
434.00	.55895	.000046	.00010	435.50	.54368	.000026	.00010
437.00	.52045	.000033	.00010	438.50	.49497	.000054	.00010
440.00	.47961	.000032	.00010	441.50	.47848	.000021	.00010
443.00	.48766	.000036	.00010	444.50	.49576	.000022	.00010
446.00	.49920	.000024	.00010	447.50	.50825	.000046	.00010
449.00	.53230	.000030	.00010	450.50	.56323	.000034	.00010
452.00	.58958	.000041	.00010	453.50	.60902	.000039	.00010
455.00	.62269	.000032	.00010	456.50	.62779	.000015	.00010
458.00	.62218	.000031	.00010	459.50	.60829	.000019	.00010
461.00	.59143	.000041	.00010	462.50	.57854	.000026	.00010
464.00	.57327	.000014	.00010	465.50	.56913	.000036	.00010
467.00	.55998	.000018	.00010	468.50	.54595	.000016	.00010
470.00	.53141	.000028	.00010	471.50	.51638	.000030	.00010
473.00	.50873	.000021	.00010	474.50	.53083	.000021	.00010
476.00	.55022	.000031	.00010	477.50	.52368	.000028	.00010
479.00	.50331	.000028	.00010	480.50	.49395	.000037	.00010
482.00	.49597	.000022	.00010	483.50	.53422	.000031	.00010
485.00	.59664	.000041	.00010	486.50	.65144	.000060	.00010
488.00	.69368	.000055	.00010	489.50	.72897	.000037	.00010
491.00	.75482	.000024	.00010	492.50	.76958	.000047	.00010
494.00	.77582	.000044	.00010	495.50	.77650	.000051	.00010
497.00	.77208	.000054	.00010	498.50	.76286	.000025	.00010

TABLE 4 (Continued)

Transmittance (T) as a function of wavelength from 380 to 780.5 nm for a bandwidth of 1.5 nm. The estimates of random and systematic errors are also provided. These data are not certified.

Wavelength (nm)	T	ΔT Standard Error	ΔT Systematic	Wavelength (nm)	T	ΔT Standard Error	ΔT Systematic
500.00	.75007	.000038	.00010	501.50	.73534	.000045	.00010
503.00	.71949	.000049	.00010	504.50	.70383	.000045	.00010
506.00	.68654	.000029	.00010	507.50	.66262	.000024	.00010
509.00	.62231	.000040	.00010	510.50	.56707	.000031	.00010
512.00	.51720	.000029	.00010	513.50	.49346	.000056	.00010
515.00	.50708	.000031	.00010	516.50	.53946	.000027	.00010
518.00	.56851	.000043	.00010	519.50	.58209	.000030	.00010
521.00	.57281	.000036	.00010	522.50	.51543	.000052	.00010
524.00	.43289	.000017	.00010	525.50	.39072	.000015	.00010
527.00	.37679	.000020	.00010	528.50	.35483	.000018	.00010
530.00	.35279	.000016	.00010	531.50	.37015	.000025	.00010
533.00	.39916	.000017	.00010	534.50	.45853	.000042	.00010
536.00	.54124	.000035	.00010	537.50	.61975	.000031	.00010
539.00	.68105	.000047	.00010	540.50	.72896	.000044	.00010
542.00	.76653	.000033	.00010	543.50	.79325	.000059	.00010
545.00	.81171	.000029	.00010	546.50	.82444	.000040	.00010
548.00	.83233	.000043	.00010	549.50	.83615	.000081	.00010
551.00	.83719	.000045	.00010	552.50	.83660	.000048	.00010
554.00	.83571	.000022	.00010	555.50	.83426	.000029	.00010
557.00	.83133	.000029	.00010	558.50	.82586	.000025	.00010
560.00	.81679	.000044	.00010	561.50	.80277	.000038	.00010
563.00	.78036	.000034	.00010	564.50	.74242	.000038	.00010
566.00	.67208	.000029	.00010	567.50	.53386	.000026	.00010
569.00	.31171	.000019	.00010	570.50	.11909	.000013	.00010
572.00	.04466	.000020	.00010	573.50	.03514	.000016	.00010
575.00	.04138	.000008	.00010	576.50	.04491	.000012	.00010
578.00	.05465	.000013	.00010	579.50	.06821	.000009	.00010
581.00	.06696	.000015	.00010	582.50	.04148	.000013	.00010
584.00	.01915	.000006	.00010	585.50	.01262	.000006	.00010
587.00	.01731	.000007	.00010	588.50	.03031	.000012	.00010
590.00	.04805	.000019	.00010	591.50	.06187	.000007	.00010
593.00	.08229	.000008	.00010	594.50	.12409	.000025	.00010
596.00	.18771	.000025	.00010	597.50	.25829	.000021	.00010
599.00	.31676	.000014	.00010	600.50	.37306	.000031	.00010
602.00	.43501	.000046	.00010	603.50	.49640	.000049	.00010
605.00	.55363	.000037	.00010	606.50	.59925	.000042	.00010
608.00	.63107	.000066	.00010	609.50	.65692	.000042	.00010
611.00	.68896	.000030	.00010	612.50	.73245	.000043	.00010
614.00	.78043	.000024	.00010	615.50	.82013	.000027	.00010
617.00	.84419	.000022	.00010	618.50	.85409	.000056	.00010

TABLE 4 (Continued)

Transmittance (T) as a function of wavelength from 380 to 780.5 nm for a bandwidth of 1.5 nm. The estimates of random and systematic errors are also provided. These data are not certified.

Wavelength (nm)	T	ΔT Standard Error	ΔT Systematic	Wavelength (nm)	T	ΔT Standard Error	ΔT Systematic
620.00	.85400	.000056	.00010	621.50	.84757	.000045	.00010
623.00	.84081	.000045	.00010	624.50	.84027	.000029	.00010
626.00	.84212	.000048	.00010	627.50	.84185	.000048	.00010
629.00	.83965	.000035	.00010	630.50	.84039	.000047	.00010
632.00	.84500	.000046	.00010	633.50	.85109	.000062	.00010
635.00	.85677	.000038	.00010	636.50	.86029	.000032	.00010
638.00	.86270	.000045	.00010	639.50	.86501	.000022	.00010
641.00	.86684	.000035	.00010	642.50	.86847	.000026	.00010
644.00	.86991	.000050	.00010	645.50	.87114	.000069	.00010
647.00	.87189	.000053	.00010	648.50	.87232	.000097	.00010
650.00	.87268	.000030	.00010	651.50	.87294	.000108	.00010
653.00	.87322	.000052	.00010	654.50	.87322	.000064	.00010
656.00	.87320	.000078	.00010	657.50	.87276	.000059	.00010
659.00	.87216	.000017	.00010	660.50	.87106	.000029	.00010
662.00	.86924	.000035	.00010	663.50	.86636	.000018	.00010
665.00	.86192	.000120	.00010	666.50	.85616	.000054	.00010
668.00	.84947	.000030	.00010	669.50	.84273	.000043	.00010
671.00	.83688	.000020	.00010	672.50	.83157	.000038	.00010
674.00	.82668	.000032	.00010	675.50	.82088	.000031	.00010
677.00	.81184	.000039	.00010	678.50	.79967	.000058	.00010
680.00	.78648	.000047	.00010	681.50	.77033	.000071	.00010
683.00	.75090	.000047	.00010	684.50	.73735	.000031	.00010
686.00	.74509	.000039	.00010	687.50	.76335	.000040	.00010
689.00	.77874	.000044	.00010	690.50	.79470	.000040	.00010
692.00	.81070	.000036	.00010	693.50	.82509	.000060	.00010
695.00	.83668	.000063	.00010	696.50	.84584	.000038	.00010
698.00	.85296	.000065	.00010	699.50	.85807	.000074	.00010
701.00	.86156	.000057	.00010	702.50	.86388	.000070	.00010
704.00	.86559	.000080	.00010	705.50	.86695	.000087	.00010
707.00	.86777	.000188	.00010	708.50	.86848	.000069	.00010
710.00	.86848	.000063	.00010	711.50	.86809	.000113	.00010
713.00	.86701	.000069	.00010	714.50	.86522	.000073	.00010
716.00	.86278	.000142	.00010	717.50	.85960	.000047	.00010
719.00	.85552	.000123	.00010	720.50	.85002	.000116	.00010
722.00	.84260	.000084	.00010	723.50	.83304	.000031	.00010
725.00	.81990	.000114	.00010	726.50	.80031	.000070	.00010
728.00	.77176	.000077	.00010	729.50	.72617	.000057	.00010
731.00	.65583	.000101	.00010	732.50	.54906	.000059	.00010
734.00	.41047	.000065	.00010	735.50	.27146	.000070	.00010
737.00	.17783	.000032	.00010	738.50	.14350	.000025	.00010

TABLE 4 (Continued)

Transmittance (T) as a function of wavelength from 380 to 780.5 nm for a bandwidth of 1.5 nm. The estimates of random and systematic errors are also provided. These data are not certified.

Wavelength (nm)	T	Δ_T Standard Error	Δ_T Systematic	Wavelength (nm)	T	Δ_T Standard Error	Δ_T Systematic
740.00	.14029	.000039	.00010	741.50	.14205	.000027	.00010
743.00	.15422	.000013	.00010	744.50	.17506	.000027	.00010
746.00	.17950	.000044	.00010	747.50	.16393	.000038	.00010
749.00	.15817	.000026	.00010	750.50	.17714	.000030	.00010
752.00	.21278	.000036	.00010	753.50	.25589	.000065	.00010
755.00	.30634	.000047	.00010	756.50	.36590	.000050	.00010
758.00	.42896	.000027	.00010	759.50	.48660	.000056	.00010
761.00	.53598	.000089	.00010	762.50	.57747	.000099	.00010
764.00	.61508	.000065	.00010	765.50	.65041	.000071	.00010
767.00	.68350	.000108	.00010	768.50	.71193	.000061	.00010
770.00	.73432	.000079	.00010	771.50	.75005	.000086	.00010
773.00	.76019	.000081	.00010	774.50	.76505	.000141	.00010
776.00	.76438	.000145	.00010	777.50	.75658	.000066	.00010
779.00	.74102	.000081	.00010	780.50	.71767	.000142	.00010

U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Calibration

Standard Reference Material 2031

Metal-on-Quartz Filters for Spectrophotometry

This Standard Reference Material (SRM) is intended for use in the verification of the transmittance and absorbance scales of conventional spectrophotometers in the ultraviolet and visible regions of the electromagnetic spectrum. SRM 2031 consists of three individual filters in their metal holders and one empty filter holder. Two filters, having nominal transmittances of 10 and 30 percent, were produced by evaporating different thicknesses of chromium metal on 1.5-mm thick fused silica plates that had been precision ground and polished. These metal films are protected by 1.5-mm clear fused silica cover plates optically contacted to the base plates. The third filter is a single fused silica plate 3-mm thick, having a nominal transmittance of 90 percent. The metal holders for these filters are provided with shutters to protect the filters when not in use. The shutters must be removed at the time of measurement and be replaced after the measurements have been completed. Each filter holder bears a filter number (10, 30, or 90) and a set identification number.

Set and Filter Ident. Number	TRANSMITTANCE (T)									
	Wavelength, nm									
	250.0	280.0	340.0	360.0	400.0	465.0	500.0	546.1	590.0	635.0

Set and Filter Indent. Number	TRANSMITTANCE DENSITY ($-\log_{10}T$)									
	Wavelength, nm									
	250.0	280.0	340.0	360.0	400.0	465.0	500.0	546.1	590.0	635.0

Date of Certification:

Gaithersburg, MD 20899
October 22, 1984
(Revision of Certificates
dated 6-1-79 and 9-20-82)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

The transmittance values (T) can be converted to percent transmittance (%T) by multiplying by 100. The transmittance densities were calculated from the measured transmittance (T). These transmittance densities should be indicated by the absorbance scale of the spectrophotometer when the filters are measured against air. All of the certified transmittance values were obtained by measuring against air at an ambient temperature of 23.5 °C.

The uncertainty of the certified transmittance values of the two metal film filters is ± 1.0 percent. This uncertainty includes 0.5 percent for random and systematic errors of the calibration procedure, as well as 0.5 percent for possible changes in the transmittance with time. The total uncertainty of the certified transmittance values for the clear quartz plate is 0.5 percent (see note below). The long-term stability of the filters with the chromium film has not been rigorously established. Measurements to date, however, suggest that the transmittance of the chromium-coated filters could change by as much as 0.5 percent in the first year after calibration. Therefore, this SRM should be returned to the National Bureau of Standards for free verification of the transmittance values on the first anniversary of the certification date. Information regarding subsequent verifications or recalibrations, including costs for such service, will be included with this first verification report. Before returning this SRM to NBS, information regarding such shipment should be obtained from the Service Analysis Coordinator, Center for Analytical Chemistry, Room B222, Chemistry Building, National Bureau of Standards, Gaithersburg, MD 20899. Telephone: (301) 921-2141.

When not in use, the filters should be stored in their holders with the shutters in place and in the metal container provided for this purpose. Extended exposure to laboratory atmosphere and dirty surroundings should be avoided.

The transmittance measurements were made using the high-accuracy spectrophotometer designed and built in the NBS Center for Analytical Chemistry [1]. This instrument is a primary transmittance standard; its transmittance accuracy was established using the double-aperture method of linearity testing [1,3,5,6].

Transmittance measurements for SRM 2031 were made by producing the vertical image of the slit (about 8 mm by 1.5 mm), using a convergent beam geometry with an aperture ratio f:10, in the middle of the entrance face of the filter. The filters were measured in the spectrophotometer in a position perpendicular to the incident light beam. A spectral bandpass of 1.6 nm was used for measurements at all wavelengths. Because the transmittances of these filters exhibit an appreciable optical neutrality, the dependence of transmittance on bandpass is not critical and wider bandpasses may be used in routine measurements. For a quantitative discussion of this subject, the user should consult reference 5, pp. 32 and 33.

The transmittance, T, was measured against air in the reference beam; hence it includes the reflection losses that occur at the air-filter interface. Under these circumstances the measured transmittance, T, has a corresponding transmittance density, defined as $-\log_{10} T$. The internal transmittance, T_i, of a material is defined as the transmittance of the material corrected for reflection losses. This is obtained experimentally when the measurements are made against a blank sample in the reference beam. The absorbance, A, of a material is related to this internal transmittance, T_i, by the expression, $A = -\log_{10} T_i$.

The exposed surface of each filter is approximately 29 x 8 mm, measuring from a point 1.5 mm above the base of the filter holder (see figure). The empty filter holder provided is to be used in the reference beam of the spectrophotometer so that approximately equivalent conditions of stray radiation are achieved for both beams. The transmittance of the filters depends upon the intrinsic properties of the material, wavelength, spectral bandpass, geometry of the optical beam, temperature, and positioning of the filter. While changes in ambient temperature of 1 or 2 °C from 23.5 °C have not significantly affected the calibration, the effect of temperature variations exceeding 2 °C have not been investigated. Changes in the transmittance may be caused by surface conditions, aging of the material, exposure to a harmful atmosphere, or careless handling [2,3,4,5,7].

SRM 2031 is stored in a black-anodized aluminum container provided with a threaded cap made of the same metal. Each filter is placed in a cylindrical cavity to prevent any contact between the filter face and the walls of the storage container. Contamination of the filter surface with particulate matter due to static charges is minimized through the metallic nature of the container. A flat leaf spring is inserted into the cylindrical cavity with each filter holder to minimize damage during transportation. These springs can be removed during normal use in the laboratory.

NOTE: In some commercial instruments, the metal-on-quartz filters can generate reflection effects in the sample compartment that can degrade the accuracy of the measured transmittances. During the development of SRM 2031, the presence and magnitude of reflection effects were studied and were found negligible, within the uncertainty specified, in all spectrophotometers tested (see Ref. 5 page 4, pp. 16-30 for additional details of this study). However for certain instruments, these effects could become significant. If such effects are detected or suspected, the user should contact R.W. Burke, NBS Inorganic Analytical Research Division, for assistance and instructions.

The filter is shown in the assembled unit with its front surface facing up. The filter, in its filter holder, should be placed in the cuvette compartment of the spectrophotometer with its front surface facing the incident light beam and the rear surface facing the photodetector.

Top: Cylindrical container with its screw cap, both made of black-anodized aluminum alloy. Four filter holders can be stored in the cylindrical container. Bottom (from left to right): Aluminum alloy filter holder, 12.5 mm square and 58 mm high; Metal-on-quartz filter; Retaining spring of beryllium-copper with nylon screw and washer; Assembled unit; and two Delrin shutters. All metal and plastic parts are flat black.

Prior to certification measurements, each filter was examined for surface defects and the condition of the optical contact [5]. Should the surface of the filter become contaminated, no attempt should be made to clean it unless the user has the facilities to demonstrate that the cleaning treatment will not alter the surface or degrade the accuracy of the certified values. As SRM 2031 is a transfer standard, the only means available to verify its integrity is to remeasure its transmittance with a primary standard instrument similar to that used in this certification [1,5]. In most cases, where verification or recertification of the transmittance values is desirable, it will be most expeditious to return the filters to the National Bureau of Standards for measurement.

Further information concerning the selection, preparation, and properties of SRM 2031 will be found in reference 5.

The research, development, and initial production of this SRM were conducted by R. Mavrodineanu and J.R. Baldwin, NBS Inorganic Analytical Research Division.

The transmittance measurements were performed by R.W. Burke and M.V. Smith, NBS Inorganic Analytical Research Division. Technical leadership for the preparation and measurements leading to certification was provided by R.W. Burke.

The overall direction and coordination of technical measurements leading to certification were performed under the chairmanship of J.R. DeVoe, NBS Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by L.J. Powell.

We wish to acknowledge the cooperation of George N. Bowers, Jr., M.D., of Hartford Hospital, Hartford, Connecticut; Royden N. Rand, Ph.D., of the Eastman Kodak Co. Research Laboratories, Rochester, New York; and Donald S. Young, M.D., Ph.D., of the Mayo Clinic, Rochester, Minnesota.

The contributions of H.E. Bennett and J.M. Bennett of the Michelson Laboratory, Physical Optics Branch, Naval Weapons Center, China Lake, California, who helped in the initial production of SRM 2031 are also gratefully acknowledged.

References

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3. K.S. Gibson, Spectrophotometry, NBS Circ. 484 (Sept. 1949).
4. R. Mavrodineanu, Considerations for the Use of Semi-Transparent Metallic Thin Films as Potential Transmittance Standards in Spectrophotometry. NBS Journal of Research 80A, No. 4, 637-641 (1976).
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U.S. Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 2032

Crystalline Potassium Iodide

Heterochromatic Stray Radiant Energy Standard for Ultraviolet Absorption Spectrophotometry

K. D. Mielenz and R. Mavrodineanu

This Standard Reference Material consists of reagent-grade, crystalline potassium iodide (KI) to be used to assess heterochromatic stray radiant energy (stray light) in ultraviolet absorption spectrophotometers in the spectral region below 260 nm. Stray light is assessed by measuring the spectral absorbance of aqueous solutions of SRM 2032 of known pathlengths and concentrations, and comparing the result with the certified values of the specific absorbance, ϵ.

Specific Absorbance[a]

ϵ (L g^{-1} cm^{-1}), vs Wavelength, λ (nm), at 23.5 °C

λ	240	245	250	255	260	265	270	275
ϵ	25.6	10.67	3.66	1.05	0.260	0.0560	0.0121	0.0031

[a] "Specific absorbance" is defined here as absorbance per unit pathlength and unit concentration. The term "absorptivity" has been avoided since it is ambiguously defined. See K. D. Mielenz, Anal. Chem. 48, 1093-1094 (1976).

The estimated uncertainty of these values is ± 5%, which includes the random and systematic errors of the calibration procedure, as well as variations due to possible instability of the KI. (See, Certification Procedure.)

The material used to produce SRM 2032 was obtained from the J. T. Baker Chemical Co., Phillipsburg, N.J., in two bottles, as reagent-grade (99.8% purity) potassium iodide.

SRM 2032 was issued with the technical assistance of J. R. Baldwin, R. W. Burke, A. L. Cummings, B. I. Diamondstone, and G. A. Sleater, and under the overall direction of I. L. Barnes.

The technical and support aspects concerning preparation, certification, and issuance of this SRM were coordinated through the Office of Standard Reference Materials by R. W. Seward.

Washington, D.C. 20234
October 31, 1979

George A. Uriano, Chief
Office of Standard Reference Materials

Supplementary Information

Material Testing:

Material homogeneity was tested by measuring the absorbance of 1% aqueous solutions of the KI sampled from the top, middle, and bottom of each bottle. These measurements, at 265, 267, and 270 nm, showed no evidence of inhomogeneity. Tests for moisture content (Karl Fischer method) gave an average of 0.007%. This value was sufficiently small that all certified data are based on weighed samples of the undried, as received, material.

Stability of the material to UV and visible light was tested by exposing the KI salt in a low-actinic glass bottle to radiation from a 3 kW xenon-arc lamp at a distance of 25 cm for four days at room temperature. The average absorbances at five wavelengths between 240 and 270 nm of aqueous solutions of the exposed material were 1.1% lower than those of solutions of the unexposed material. In view of the severity of this test, this change was considered minor. Nonetheless, the 1.1% was included in the overall uncertainty of the certified values.

Certification Procedure:

The certified specific absorbances were measured in the NBS Center for Analytical Chemistry high-accuracy spectrophotometer [1, 2], equipped with a deuterium lamp and UV averaging sphere [3, 4]. Thirteen aqueous solutions of KI with concentrations ranging from 0.03 to 30 g L^{-1} were prepared and measured against distilled water, using standard fused-silica cuvettes with pathlengths between 10.001 and 10.009 mm[b]. The absorbance measurements were performed at the temperature, $t = 23.5 \pm 0.5$ °C. A 0.2 nm bandpass was used, and wavelength settings were made with an accuracy of 0.05 nm. The measurements were made on at least three concentrations at each wavelength. These concentrations were chosen so that most absorbance readings fell between 0.1 and 1.0 to minimize stray light and bandwidth errors. The data were found to obey Beer's law.

Thus, the equation,

$$\epsilon = A/c\ell \tag{1}$$

was used to calculate the specific absorbances, ϵ, from the measured absorbances, A, and the known values of concentration, c, and pathlength, ℓ .

Limits to random error (two standard deviations) of these measurements (including the photometric imprecision of the spectrophotometer, errors in the repositioning of cuvettes, concentration errors, pathlength uncertainties, temperature errors, Beer's law uncertainties, and bandwidth errors) were determined to be ± 2.4%. The error due to the 0.05 nm uncertainty of the wavelength calibration of the spectrophotometer is ± 1.1%.

The uncertainty of 5% stated on the face of this certificate represents the sum of these errors and the above mentioned 1.1% uncertainty due to instability of the KI. No corrections were applied for the effects of internal reflections inside the sample and reference cuvettes, nor were the weights corrected to vacuum. These and all other sources of error were considered negligible.

The temperature coefficient at 260 nm was determined to be

$$\frac{1}{\epsilon}\,\frac{d\epsilon}{dt} = 0.031\ ^{\circ}C^{-1}. \tag{2}$$

For precision measurements, it is recommended that SRM 2032 be used in thermostated cuvettes at 23.5 °C, or that a temperature correction according to Eq. (2) be applied.

[b] Issued by NBS as SRM 932, Quartz Cuvettes for Spectrophotometry.

Instructions for Use

Storage and Preparation:

SRM 2032 should be stored in the original, low-actinic glass bottle and the cardboard container in which it was issued to protect it from unnecessary exposure to light and humidity. When so stored, the expected stability of this material is at least three years. Thus, until additional stability data are obtained, this material should be used within three years of the date of purchase.

All solutions prepared from SRM 2032 should be made in borosilicate glass containers using distilled water and transfer pipettes (Pasteur type) of the same glass and fitted with rubber bulbs[c]. Use clean spectrophotometer cuvettes, free of scratches, made of non-fluorescent fused silica, and fitted with ground-glass or Teflon stoppers to minimize evaporation; or preferably use NBS SRM 932. Mark all cuvettes to assure the same orientation in the spectrophotometer, and place them into their respective holders. Using transfer pipettes of the type mentioned, rinse each cuvette several times with distilled water. Prepare a solution of KI in distilled water (e.g., 1%), fill the sample cuvette with the KI solution and the reference cuvette with distilled water. Measure the absorbance, leaving the cuvettes in their holders, empty them (using the pipettes), and repeat the rinsing and filling operations until constant absorbance readings are obtained. Fresh solutions should be made before every test.

Measurements:

A 1% KI solution ($c = 10\ \mathrm{g\ L^{-1}}$) with a 1 cm pathlength exhibits a sharp cutoff in transmittance near 260 nm; i.e., it transmits more than 90% above 273 nm, but less than 0.01% below 258 nm. Therefore, with the monochromator set for a wavelength below 260 nm, any appreciable amount of light detected is heterochromatic stray light, which consists of wavelengths above the cutoff. The amount of stray light in the spectrophotometer at wavelength λ may be determined from the equations:

$$T'(\lambda) = \frac{T(\lambda) + x(\lambda)}{1 + x(\lambda)} \tag{3}$$

$$x(\lambda) = \frac{T'(\lambda) - T(\lambda)}{1 - T'(\lambda)} \tag{4}$$

where:

$x(\lambda)$ is the stray light ratio,

$T'(\lambda)$ is the apparent transmittance,

$T(\lambda)$ is the true transmittance.

The stray light ratio, $x(\lambda)$, is the proportion of heterochromatic stray radiant energy in the spectrophotometer for the wavelength setting λ. Equation (4) forms the theoretical basis for the determination of the stray light ratio by comparing apparent transmittance to true transmittance. For SRM 2032, the true transmittance, $T(\lambda)$, of KI solutions may be calculated as:

$$T(\lambda) = 10^{-\epsilon(\lambda) c \ell}$$

where $\epsilon(\lambda)$ is the certified specific absorbance given on the face of this certificate.

---

[c] Soft glass containers and pipettes contain residual amounts of UV absorbing material, but can be used after proper cleaning. Several rinses, first with isopropyl alcohol and then with distilled water, are generally adequate.

The apparent transmittances of KI solutions depend not only on the stray light ratio of the spectrophotometer, but also on the wavelength accuracy and the spectral bandwidth, which can significantly affect the results obtained because of the steep slope of the absorbance cutoff of KI. A low-pressure mercury discharge lamp is suggested for verifying the wavelength scale, and a bandwidth as narrow as compatible with adequate signal-to-noise ratios should be used.

Many instruments do not permit the direct measurement of transmittances below certain limits, e.g., 1% or 0.1%. For such instruments, the use of SRM 2032 will only measure stray light ratios above these limits. To measure stray light ratios below these limits, the reference beam of the spectrophotometer must be attenuated to extend the transmittance scale into the low-transmittance region.

References

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U. S. Department of Commerce
Philip M. Klutznick
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 2033

Crystalline Potassium Iodide with Attenuator Heterochromatic and Isochromatic Stray Radiant Energy Standard for Ultraviolet Absorption Spectrophotometry

K. D. Mielenz and R. Mavrodineanu

This Standard Reference Material consists of reagent-grade crystalline potassium iodide (KI) and a radiation attenuator to be used to assess the heterochromatic stray radiant energy (stray light) in ultraviolet absorption spectrophotometers in the spectral region below 260 nm.. The radiation attenuator consists of two semitransparent evaporated metal-on-fused silica (non-fluorescent) filters, each having a nominal transmittance of 10%. One filter is mounted in a cuvette-style holder that can be inserted into the sample compartment of the spectrophotometer. The other is mounted in one of the two shutters of the holder. The use of this attenuator permits the expansion of the transmittance scale into the low-transmittance region by providing a two-step attenuation of the reference beam of the spectrophotometer to about 1%. The attenuator can also be used to assess isochromatic stray light by following the instructions given in this certificate.

The heterochromatic stray light is assessed by measuring the spectral absorbance of aqueous solutions of SRM 2033 of known pathlengths and concentrations, and comparing the results with the certified values of the specific absorbance, ϵ.

Specific Absorbance[a]

	ϵ (L g^{-1} cm^{-1}), vs Wavelength, λ (nm), at 23.5 °C							
λ	240	245	250	255	260	265	270	275
ϵ	25.6	10.67	3.66	1.05	0.260	0.0560	0.0121	0.0031

[a] "Specific Absorbance" is defined here as absorbance per unit pathlength and unit concentration. The term "absorptivity" has been avoided since it is ambiguously defined. See K. D. Mielenz, Anal. Chem. 48, 1093-1094 (1976).

The estimated uncertainty of these values is ± 5%, which includes the random and systematic errors of the calibration procedure, as well as variations due to possible instability of the KI. (See Certification Procedure.)

The material used to produce SRM 2033 was obtained from the J.T. Baker Chemical Co., Phillipsburg, N.J., in two bottles, as reagent-grade (99.8% purity) potassium iodide.

The transmittance of the filter mounted in the holder at λ255 nm is __________;
the transmittance of both filters at λ255 is __________.

SRM 2033 was issued with the technical assistance of J. R. Baldwin, R. W. Burke, A. L. Cummings, B. I. Diamondstone, and G. A. Sleater, and under the overall direction of E. L. Garner, NBS Inorganic Analytical Research Division.

The technical and support aspects concerning preparation, certification, and issuance of this SRM were coordinated through the Office of Standard Reference Materials by R. W. Seward.

Washington, D.C. 20234
May 9, 1980

George A. Uriano, Chief
Office of Standard Reference Materials

Potassium Iodide Material Testing:

Material homogeneity was tested by measuring the absorbance of 1% aqueous solutions of the KI sampled from the top, middle, and bottom of each bottle. These measurements, at 265, 267, and 270 nm, showed no evidence of inhomogeneity. Tests for moisture content (Karl Fischer method) gave an average of 0.007%. This value was sufficiently small that all certified data are based on weighed samples of the undried, as received, material.

Stability of the material to UV and visible light was tested by exposing the KI salt in a low-actinic glass bottle to radiation from a 3 kW xenon-arc lamp at a distance of 25 cm for four days at room temperature. The average absorbances at five wavelengths between 240 and 270 nm of aqueous solutions of the exposed material were 1.1% lower than those of solutions of the unexposed material. In view of the severity of this test, this change was considered minor. Nonetheless, the 1.1% was included in the overall uncertainty of the certified values.

Potassium Iodide Certification Procedure:

The certified specific absorbances were measured in the NBS Center for Analytical Chemistry high-accuracy spectrophotometer [1,2], equipped with a deuterium lamp and UV averaging sphere [3,4]. Thirteen aqueous solutions of KI with concentrations ranging from 0.03 to 30 g L^{-1} were prepared and measured against distilled water, using standard fused-silica cuvettes with pathlengths between 10.001 and 10.009 mm[b]. The absorbance measurements were performed at the temperature, $t = 23.5 \pm 0.5\,°C$. A 0.2 nm bandpass was used, and wavelength settings were made with an accuracy of 0.05 nm. The measurements were made on at least three concentrations at each wavelength. These concentrations were chosen so that most absorbance readings fell between 0.1 and 1.0 to minimize stray light and bandwidth errors. The data were found to obey Beer's law.

Thus, the equation,

$$\epsilon = A/c\ell \tag{1}$$

was used to calculate the specific absorbances, ϵ, from the measured absorbances, A, and the known values of concentration, c, and pathlength, ℓ .

Limits to random error (two standard deviations) of these measurements (including the photometric imprecision of the spectrophotometer, errors in the repositioning of cuvettes, concentration errors, pathlength uncertainties, temperature errors, Beer's law uncertainties, and bandwidth errors) were determined to be ± 2.4%. The error due to the 0.05 nm uncertainty of the wavelength calibration of the spectrophotometer is ± 1.1%.

The uncertainty of 5% stated on the face of this certificate represents the sum of these errors and the above mentioned 1.1% uncertainty due to instability of the KI. No corrections were applied for the effects of internal reflections inside the sample and reference cuvettes, nor were the weights corrected to vacuum. These and all other sources of error were considered negligible.

The temperature coefficient at 260 nm was determined to be

$$\frac{1}{\epsilon} \cdot \frac{d\epsilon}{dt} = 0.031\ °C^{-1}. \tag{2}$$

For precision measurements, it is recommended that SRM 2033 be used in thermostated cuvettes at 23.5 °C, or that a temperature correction according to Eq. (2) be applied.

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[b] Issued by NBS as SRM 932, Quartz Cuvette for Spectrophotometry.

Instructions for Use of Potassium Iodide

Storage and Preparation:

SRM 2033 should be stored in the original, low-actinic glass bottle and the cardboard container in which it was issued to protect it from unnecessary exposure to light and humidity. When so stored, the expected stability of this material is at least three years. Thus, until additional stability data are obtained, this material should be used within three years of the date of purchase.

All solutions prepared from SRM 2033 should be made in borosilicate glass containers using distilled water and transfer pipettes (Pasteur type) of the same glass and fitted with rubber bulbs[c]. Use clean spectrophotometer cuvettes, free of scratches, made of non-fluorescent fused silica, and fitted with ground-glass or Teflon stoppers to minimize evaporation; or preferably use NBS SRM 932. Mark all cuvettes to assure the same orientation in the spectrophotometer, and place them into their respective holders. Using transfer pipettes of the type mentiond, rinse each cuvette several times with distilled water. Prepare a solution of KI in distilled water (e.g., 1%), fill the sample cuvette with the KI solution and the reference cuvette with distilled water. Measure the absorbance, leaving the cuvettes in their holders, empty them (using the pipettes), and repeat the rinsing and filling operations until constant absorbance readings are obtained. Fresh solutions should be made before every test.

Measurements:

A 1% KI solution ($c = 10 \text{ g L}^{-1}$) with a 1 cm pathlength exhibits a sharp cutoff in transmittance near 260 nm; i.e., it transmits more than 90% above 273 nm, but less than 0.01% below 258 nm. Therefore, with the monochromator set for a wavelength below 260 nm, any appreciable amount of light detected is heterochromatic stray light, which consists of wavelengths above the cutoff. The amount of stray light in the spectrophotometer at wavelength λ may be determined from the equations:

$$T'(\lambda) = \frac{T(\lambda) + x(\lambda)}{1 + x(\lambda)} \tag{3}$$

$$x(\lambda) = \frac{T'(\lambda) - T(\lambda)}{1 - T'(\lambda)} \tag{4}$$

where:

$x(\lambda)$ is the stray light ratio,

$T'(\lambda)$ is the apparent transmittance,

$T(\lambda)$ is the true transmittance.

The stray light ratio, $x(\lambda)$, is the proportion of heterochromatic stray radiant energy in the spectrophotometer for the wavelength setting λ. Equation (4) forms the theoretical basis for the determination of the stray light ratio by comparing apparent transmittance to true transmittance. For SRM 2033, the true transmittance, $T(\lambda)$, of KI solutions may be calculated as:

$$T(\lambda) = 10^{-\epsilon(\lambda)\, c\, \ell}$$

where $\epsilon(\lambda)$ is the certified specific absorbance given on the face of this certificate.

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[c] Soft glass containers and pipettes contain residual amounts of UV absorbing material, but can be used after proper cleaning. Several rinses, first with isopropyl alcohol and then with distilled water, are generally adequate.

The apparent transmittances of KI solutions depend not only on the stray light ratio of the spectrophotometer, but also on the wavelength accuracy and the spectral bandwidth, which can significantly affect the results obtained because of the steep slope of the absorbance cutoff of KI. A low-pressure mercury discharge lamp is suggested for verifying the wavelength scale, and a bandwidth as narrow as compatible with adequate signal-to-noise ratios should be used.

Optical Attenuator:

Many instruments do not permit the direct measurement of transmittances below certain limits, e.g., 1% or 0.1%. To measure stray light ratios below these limits, the reference beam of the spectrophotometer must be attenuated to extend the transmittance scale into the low-transmittance region.

As mentioned on the face of this Certificate, the optical attenuator consists of two filters, each of which has a nominal transmittance of 10 percent. One filter is mounted in a metal holder that can be inserted in the sample compartment of the spectrophotometer (5). This holder is provided with a front and rear shutter, one of which has a window in which the second filter is mounted. The holder and shutters are flat black. This unit was produced in the NBS Instrument Shops.

Instructions for Use of the Attenuator

This unit can be used to attenuate the incident radiation in the reference beam of the spectrophotometer in two steps by a total factor of about 100, corresponding to about 1 percent transmittance (6). To attenuate by a factor of 10, the attenuator is inserted in the sample compartment of the spectrophotometer facing the incident beam, with both shutters removed. A further attenuation by a factor of about 10 is obtained when the shutter carrying the second filter is inserted in the holder, in front of the first filter. Under these conditions, and with an instrument that cannot measure transmittances lower than 1 percent, the use of the attenuator will permit heterochromatic stray light to be measured down to about 0.01 percent. This attenuation procedure can be used with spectrophotometers capable of scale expansion.

Isochromatic Stray Light:

The radiation attenuator can be used to assess the isochromatic stray light that results from reflection of the incident radiation at the surface of the sample and various optical components, and reaches the photodetector without passing through the sample. It is implicitly assumed that the sample compartment of the spectrophotometer is light-tight.

The measurement is performed by placing the attenuator in the sample beam of the spectrophotometer, with the opaque shutter placed at the rear of the filter holder. Under these conditions, if a signal is detected, it is caused by reflection at the surface of the filter exposed to the incident radiation. This radiation is scattered from the walls and other components of the sample compartment and reaches the photodetector without passing through the sample (5). This signal is the isochromatic stray light.

Isochromatic stray light, which passes through the sample, is generally caused by interreflections between lenses and/or other sample compartment elements. Tests for this stray light component, which is not detected by the methods described above, are discussed in references 7 and 8.

References:

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3. K. D. Mielenz, R. Mavrodineanu, and E. D. Cehelnik, J. Res. NBS 78A, 631-635 (1974).
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6. Estimating Stray Radiant Energy, ASTM Manual on Recommended Practices in Spectrophotometry, pp. 94-105, 3rd. Edition, 1969, 1916 Race St., Philadelphia, Pa. 19103.
7. K. D. Mielenz, J. Res. NBS 76A, 455-467 (1972).
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U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate

Standard Reference Material 2034

Holmium Oxide Solution Wavelength Standard

From 240 to 650 nm

This Standard Reference Material (SRM) is intended as a reference material for establishing the accuracy of the wavelength scale of conventional spectrophotometers in the spectral range 240 to 650 nm. It consists of a solution of 4 percent holmium oxide in 10 percent perchloric acid in water, sealed in a nonfluorescent fused silica cuvette of nominal 10 mm light path. (The cuvette fits most conventional spectrophotometers; see note 1.) The cuvette should be handled only by the tubular end, and, while not in use it should be stored in the container provided. The certified wavelengths of minimum transmittance, expressed in nanometers, are given in Table 1 for six spectral bandwidths from 0.1 to 3.0 nm for 14 bands. They are illustrated in Figure 1. The estimated uncertainty of the measured minimum transmittance wavelengths is believed to be no greater than ± 0.1 nm at the measurement temperature of 25 °C ± 5 °C, and includes the random and systematic errors of the measuring procedure. For details concerning the materials, instrumentation, and method used in the certification of SRM 2034 the user is referred to NBS Special Publication 260-102. This Special Publication also describes the influence of purity and concentration of the holmium oxide solution and temperature, and describes the procedure used for assessment of the wavelengths of minimum transmittance and the establishment of the accuracy of the wavelength scale of the spectrometer used.

The initial research at NBS concerning the potential use of the Ho_2O_3 - $HClO_4$ solution as a wavelength standard was performed by K.D. Mielenz and R.A. Velapoldi.

The transmittance measurements given in this certificate and investigations on the various chemical and physical parameters that might influence the results were performed by V.R. Weidner and R. Mavrodineanu.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of K.D. Mielenz, NBS Radiometric Physics Division, Center for Radiation Research.

The technical and support aspects involved in the issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.L. McKenzie and L.J. Powell.

Gaithersburg, MD 20899
June 20, 1985

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

CAUTION:

If the cuvette of SRM 2034, which contains holmium oxide as well as perchloric acid, is accidentially broken, the following action should be taken:

"Perchloric acid spills should be diluted immediately with water, taken up with swabs (preferably wool) and then washed with generous amounts of water. The swabs should also be washed with water before discarding. Swabs of cotton or other cellulose material contaminated with perchloric acid should be regarded as fire or explosion hazards if not washed thoroughly with large amounts of water" (1,2).

References:

(1) Schilt, Alfred A., Perchloric Acid and Perchlorates, the G. Frederick Smith Chemical Co., Publisher, 867 McKinley Ave., Columbus, Ohio 43223, pg. 157 (1979).

(2) Schumacher, Joseph C., Perchlorates, Their Properties, Manufacture and Use, Reinhold Publishing Corp., NY; Chapman & Hall, LTD, London, pg. 187 (1960).

NOTES:

(1) If the cuvette is too tall for the sample compartment of the spectrophotometer, a piece of black opaque cloth can be used to provide a temporary lightproof enclosure.

(2) The cuvettes have been individually tested for leaks. Should a leak subsequently develop, follow the procedure recommended above (under "CAUTION").

Table 1

Wavelengths of Minimum Transmittance at Six Spectral Bandwidths for a 4 Percent Holmium Oxide Solution in 10 Percent Perchloric Acid in Water.

Minimum Transmittance Band No.	Spectral Bandwidths (nm)					
	0.1	0.25	0.5	1	2	3
1	240.99	240.97	241.01	241.13	241.08	240.90
2	249.83	249.78	249.79	249.87	249.98	249.92
3	278.15	278.14	278.13	278.10	278.03	278.03
4	287.01	287.00	287.01	287.18	287.47	287.47
5	333.47	333.44	333.43	333.44	333.40	333.32
6	345.55	345.55	345.52	345.47	345.49	345.49
7	361.36	361.35	361.33	361.31	361.16	361.04
8	385.45	385.42	385.50	385.66	385.86	386.01
9	416.07	416.07	416.09	416.28	416.62	416.84
10	-----	-----	-----	451.30	451.30	451.24
11	467.82	467.82	467.80	467.83	467.94	468.07
12	485.28	485.28	485.27	485.29	485.33	485.21
13	536.54	536.53	536.54	536.64	536.97	537.19
14	640.51	640.49	640.49	640.52	640.84	641.05

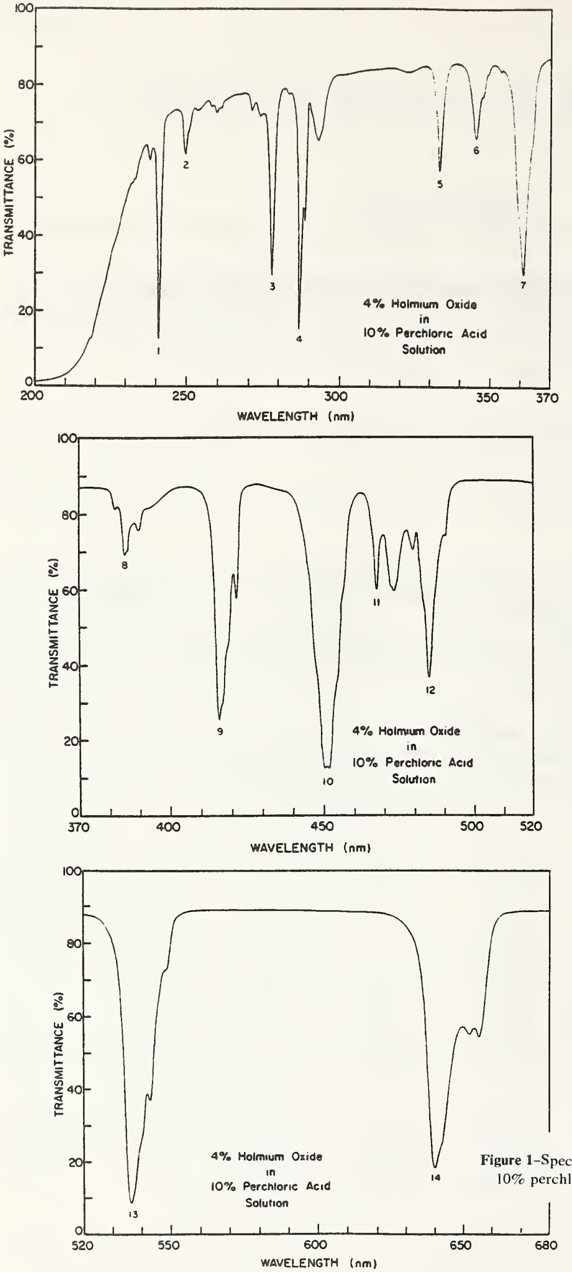

Figure 1–Spectral transmittance of a 4% solution of holmium oxide in 10% perchloric acid in water, beginning at right.

NBS-114A (REV. 2-80)

U.S. DEPT. OF COMM. **BIBLIOGRAPHIC DATA SHEET** *(See instructions)*	1. PUBLICATION OR REPORT NO. NBS/SP-260/102	2. Performing Organ. Report No.	3. Publication Date July 1986

4. TITLE AND SUBTITLE

Holmium Oxide Solution Wavelength Standard From 240 to 640 nm - SRM 2034

Victor R. Weidner, Radu Mavrodineanu, Klaus D. Mielenz,
Rance A. Velapoldi, Kenneth L. Eckerle, Bradley Adams

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

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10. SUPPLEMENTARY NOTES

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☐ 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)*

The work describes the methods and procedures used to determine the wavelengths of minimum transmittance of holmium oxide in perchloric acid solution. Measurements of spectral transmittance of the solutions were made by means of a high precision spectrophotometer over the wavelength range 200 nm to 680 nm. The wavelength scale accuracy of this instrument was verified by extensive measurements of mercury and deuterium emission lines. The measurements of spectral transmittance of the holmium oxide solutions were made as a function of temperature, purity, concentration, and spectral bandwidth. Analysis of the uncertainties associated with these parameters and the uncertainties associated with the calibration of the instrument wavelength scale and the data analysis have resulted in an estimated uncertainty of ±0.1 nm for the determination of the wavelengths of minimum transmittance of the holmium oxide solution.

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

holmium oxide; spectral bandwidth; spectrophotometer calibration; spectral transmittance; wavelength calibration; wavelength standard

13. AVAILABILITY

☒ Unlimited

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