National Institute of Standards & Technology

Certificate

Standard Reference Material 2034

Holmium Oxide Solution Wavelength Standard from 240 to 650 nm

Series No. 94

This Standard Reference Material (SRM) is a certified transfer standard intended for the verification and calibration of the wavelength scale of ultraviolet and visible absorption spectrometers having nominal spectral bandwidths not exceeding 3 nm. SRM 2034 is batch-certified for wavelength location of minimum transmittance of 14 bands in the spectral range from 240 to 650 nm for six spectral bandwidths from 0.1 to 3 nm.

SRM 2034 is an aqueous solution prepared to contain 4 percent by weight of holmium oxide (Ho_2O_3) in 10 percent (V/V) perchloric acid ($HClO_4$). The solution is flame-sealed in a non-fluorescent, fused-silica cuvette of optical quality by fusion of the tubular end of the cuvette. The square cuvette has a nominal 10 mm pathlength so that it fits in the sample compartment of most conventional absorption spectrometers (see Note 1 under "Instructions for Use").

Certification: The certified wavelengths are given in Table 1 of this Certificate and apply to Series No. 94 as well as to all preceding series.

Intrinsic Wavelength Stability and Storage: The certified wavelengths listed in Table 1 in this Certificate are expected to be valid as long as the SRM 2034 solution is chemically stable. Wavelength verification measurements have been performed in the NIST Inorganic Analytical Research Division on original SRM 2034 solutions maintained under typical laboratory conditions. The results of these wavelength verification studies indicate that the SRM 2034 solutions are stable for at least 8 years. When not in use, SRM 2034 should be stored in the container provided, and at a temperature between 20 - 30 °C.

CAUTION: SRM 2034 contains perchloric acid as well as holmium oxide which are considered hazardous. Caution should be exercised when handling this SRM (see section on Safety Precautions).

The research, development, and initial production and certification of SRM 2034, including the investigations on the various chemical and physical parameters that might influence the certified measurements and results, were performed by V.R. Weidner, NIST Radiometric Physics Division, and R. Mavrodineanu, NIST Inorganic Analytical Research Division.

The production and certification renewal of SRM 2034 were performed by J.C. Travis, M.V. Smith, and N.K. Winchester, NIST Inorganic Analytical Research Division. The technical leadership for the production and measurements leading to the certification of this SRM was provided by J.C. Travis.

The overall direction and coordination of technical measurements leading to certification was performed in the NIST Inorganic Analytical Research Division by J.C. Travis and R.L. Watters, Jr.

The support aspects involved in the issuance of this SRM were coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
February 17, 1994
(Revision of certificate dated 12-2-93)

Thomas E. Gills, Acting Chief
Standard Reference Materials Program

(over)

Certification Values

The transmittance spectrum of SRM 2034, referenced to air, for a 0.1 nm spectral bandwidth, is illustrated in Figure 1-3 in this Certificate. The certified wavelengths of minimum transmittance, expressed in nanometers (nm), for 14 bands from 240 to 650 nm and for six spectral bandwidths from 0.1 to 3.0 nm are given below in Table 1. Wavelengths for band No. 10 for the three narrowest spectral bandwidths are not given because this band splits into two transmittance minima for nominal spectral bandwidths less than 1 nm.

Table 1. Certified Wavelengths (nm) of Minimum Transmittance of 14 Bands for SRM 2034 at Six Spectral Bandwidths, Referenced to Air

SRM 2034 Band No.	Spectral Bandwidth (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

Certification Uncertainty

The expanded uncertainty (U) for all of the wavelength values given in Table 1 is U = $\pm$ 0.1 nm, determined from the root-mean-square combination of component standard uncertainties (i.e., estimated standard deviations) and a "coverage factor" (k) of k=2. The coverage factor is based on the student's t-distribution for >30 effective degrees of freedom, to define an interval within which the unknown value of the band-minimum wavelength can be asserted to lie with a level of confidence of approximately 95%. This uncertainty includes "Type A" components of uncertainty, which are evaluated by statistical methods, and "Type B" components of uncertainty, which are evaluated by other means. Component standard uncertainties for SRM 2034 are u_A = $\pm$ 0.025 nm for the calibration of the NIST spectrophotometer wavelength scale against atomic spectral lines, U_{B1} = $\pm$ 0.03 for the estimation of absorption band minima, and u_{B2} = $\pm$ 0.03 nm for possible wavelength shifts with temperature and concentration of the solution. The methods used to combine standard uncertainties are described in NIST Technical Note 1297.[1]

The certified wavelengths listed in Table 1 in this Certificate are not valid if SRM 2034 is used outside the range of experimental conditions investigated. For example, the wavelengths of minimum transmittance of SRM 2034 for spectral bandwidths greater than 3 nm have not been evaluated.

Production and Certification Procedure

Specific details concerning the materials, instrumentation, and method used in the certification of SRM 2034, are given elsewhere.[2,3] NBS Special Publication 260-102 [2] discusses the influence of temperature as well as the purity and concentration of the holmium oxide solution on the certified wavelengths. The procedures used for the assessment of the wavelengths of minimum transmittance and the establishment of the accuracy of the wavelength scale of the reference spectrometer used for the certification are also described in Special Publication 260-102.

Instructions for Use

To maintain the integrity of SRM 2034, the cuvette should be handled only by the tubular end or by its opposing frosted sides. While not in use, SRM 2034 should be stored in the container provided.

SRM 2034 is to be carefully inserted in the cuvette holder of the sample beam within a lightproof sample compartment of the spectrometer being tested (see **Note 1** below). The desired bands are scanned to measure their locations of minimum transmittance for known spectral bandwidth conditions. The transmittance spectrum is to be acquired at a laboratory temperature of 25 $\pm$ 5 °C, and is referred to air, i.e., no cuvette or blank is to be placed in the reference position. The measured wavelength of the minimum transmittance of a specific band is compared to the certified wavelength in Table 1 for that band; the certified wavelength for the spectral bandwidth that is most representative of the spectral bandwidth of the spectrometer being tested is used as the reference value. Taking into account the certification uncertainty of $\pm$ 0.1 nm for SRM 2034, any significant differences between the measured and the certified wavelengths for the peaks measured may then be used to recalibrate the wavelength scale.

NOTE 1. If the cuvette is too tall for the sample compartment of the absorption spectrometer being tested, a piece of black opaque cloth placed over the sample compartment can be used to provide a temporary lightproof enclosure during the wavelength verification procedure.

Wavelength Verification

Good laboratory practice in the handling and storage of SRM 2034 is highly recommended to help maintain the integrity of the certified wavelength values given in Table 1 in this Certificate. If the user determines at any time that this SRM has been exposed to adverse conditions that could affect the chemical stability of the solution, and perhaps invalidate the certified wavelength values, this SRM may be returned to the NIST Inorganic Analytical Research Division for wavelength verification. Prior to return shipment of this SRM, however, the NIST Inorganic Analytical Research Division should be contacted directly by telephone at (301) 975-4117 to obtain the necessary information regarding recommended shipping instructions and verification measurement fees.

Safety Precautions

SRM 2034 contains perchloric acid as well as holmium oxide in cuvettes that have been individually vacuum-tested for leaks. Should a leak in the cuvette subsequently develop, or if the cuvette is accidentally broken, the general recommendation is to carefully treat the perchloric acid spill immediately with copious amounts of water. [4-6] Specifically, the following remedial 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."[5]

Acknowledgments

The initial research at NIST concerning the potential use of the Ho_2O_3-$HClO_4$ solution as a wavelength standard was performed by K.D. Mielenz of the NIST Radiometric Physics Division and R.A. Velapoldi of the NIST Surface and Microanalysis Science Division.

The vacuum-testing and flame-sealing of the fused-silica cuvettes for this SRM were performed by J. Anderson, of the NIST Fabrication Technology Division.

REFERENCES

[1] Barry N. Taylor and Chris E. Kuyatt, "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, (1993).

[2] V.R. Weidner, R. Mavrodineanu, K.D. Mielenz, R.A. Velapoldi, K.L. Eckerle, and B. Adams, Holmium Oxide Solution Wavelength Standard from 240 to 640 nm - SRM 2034, NBS Special Publication 260-102, (1986).

[3] V.R. Weidner, R. Mavrodineanu, K.D. Mielenz, R.A. Velapoldi, K.L. Eckerle, and B. Adams, Spectral Transmittance Characteristics of Holmium Oxide in Perchloric Acid, J. Res. Natl. Bur. Stds., Vol. 90, No. 2, 115-125 (1985).

[4] J.C. Schumacher, Ed., Perchlorates: Their Properties, Manufacture and Uses, Reinhold Publishing Corp., New York, NY, p. 180 (1960).

[5] A.A. Schilt, Perchloric Acid and Perchlorates, The G. Frederick Smith Chemical Co., Publisher, Columbus, OH 43223, p. 157 (1979).

[6] GFS Chemicals 90/91 Catalog, GFS Chemicals, Powell, OH 43605, p. 458.

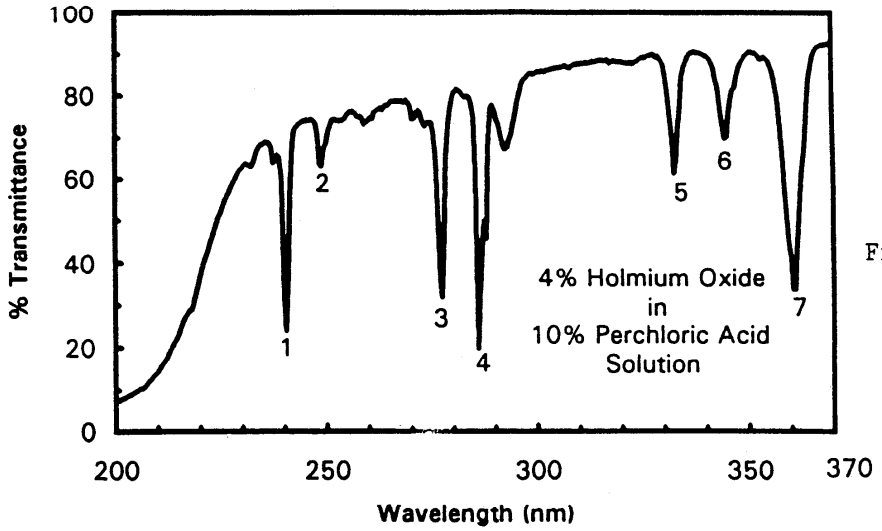

Fig. 1

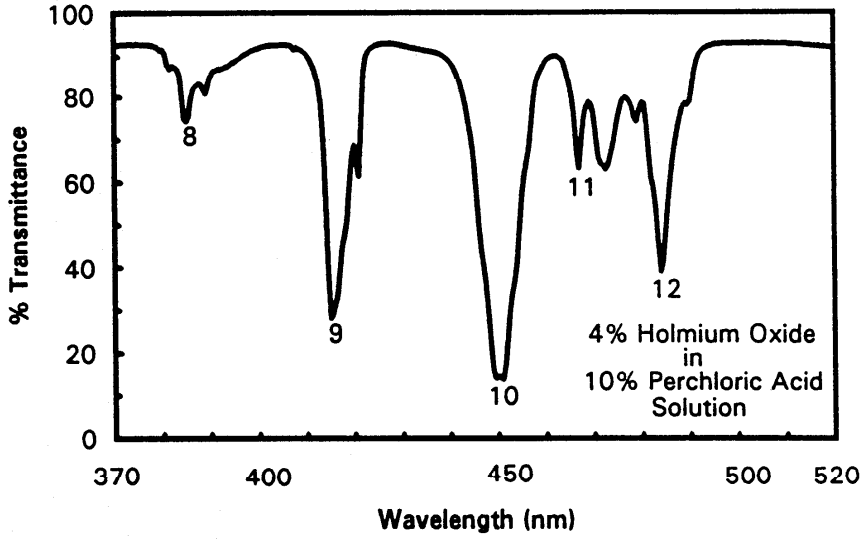

Fig. 2

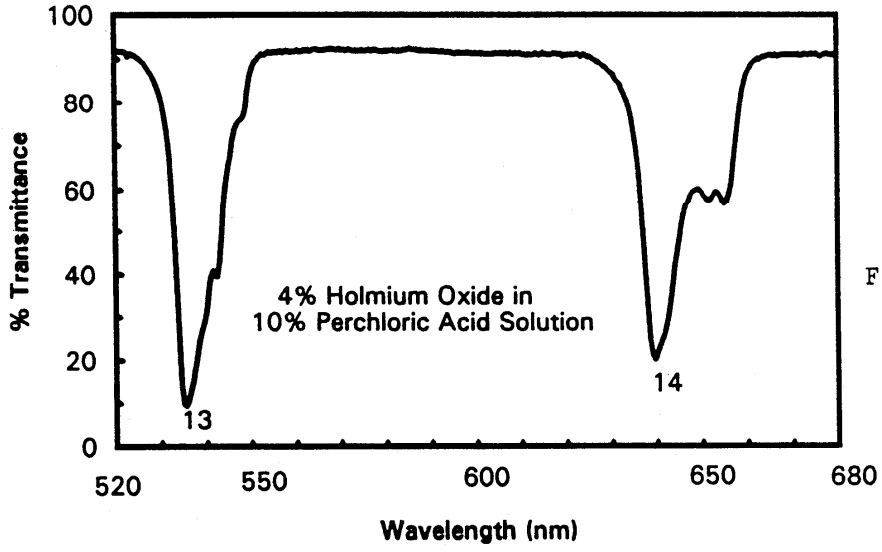

Fig. 3

Figure 1-3. Spectral transmittance of a 4% solution of holmium oxide in 10% perchloric acid solution, 200-370 nm, 370-520 nm, and 520-680 nm.