National Institute of Standards & Technology

Certificate

Standard Reference Material® 2034

Holmium Oxide Solution Wavelength Standard from 240 nm to 650 nm

Series No. 99

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 spectrophotometers having nominal spectral bandwidths not exceeding 3 nm. SRM 2034 is certified for the wavelength location of minimum transmittance for 14 bands in the spectral range from 240 nm to 650 nm and at six spectral bandwidths from 0.1 nm to 3 nm.

SRM 2034 is an aqueous solution containing 4 % (mass fraction) holmium oxide (Ho_2O_3) in 10 % (volume fraction) perchloric acid ($HClO_4$). The solution is contained in a flame-sealed, nonfluorescent, fused-silica cuvette of optical quality (parallel to ~0.9 mrad and flat to ~1 μm). A protective cap is glued over the fused end of the cuvette. The square-bottomed (12.5 mm x 12.5 mm) cuvette has a nominal pathlength of 10 mm and fits in the sample compartment of most conventional absorption spectrophotometers.

CAUTION: The cuvette contains a perchloric acid solution of holmium oxide and has been individually vacuum-tested for leaks. If a leak in the cuvette should subsequently develop, or if the cuvette is accidentally broken, carefully treat the spill immediately with copious amounts of water. The remedial action described in the MSDS accompanying the SRM unit should be taken.

Certification: The certified wavelengths for Series No. 99 are given in Table 1. These certified values apply to all previously issued series of SRM 2034.

Expiration of Certification: The certification of this SRM is valid, within the measurement uncertainties specified, until **31 December 2009**, provided the SRM is used in accordance with the instructions given in this certificate. However, the certification will be nullified if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The overall direction and coordination of technical measurements leading to certification were performed by J.C. Travis of the NIST Analytical Chemistry Division.

The production and certification of SRM 2034 Series No. 99 were performed by J.C.Travis and M.V. Smith of the NIST Analytical Chemistry Division with the assistance of P.C. DeRose, D.F. Mildner, and M.D. Maley of the NIST Analytical Chemistry Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.W.L. Thomas.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 01 March 2000

Thomas E. Gills, Director
Office of Measurement Services

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.

Certified Values: The certified wavelengths of minimum transmittance for 14 bands from 240 nm to 650 nm and for six spectral bandwidths from 0.1 nm to 3.0 nm are given below in Table 1. It should be recognized that the wavelengths for Band No. 10 for the three narrowest spectral bandwidths are not given because this band resolves into two transmittance minima for spectral bandwidths of nominally less than 1 nm. The transmittance spectrum of SRM 2034, referenced to air, for a 0.1 nm spectral bandwidth, is illustrated in Figures 1-3 of this Certificate.

NOTE: The wavelengths of minimum transmittance of SRM 2034 for spectral bandwidths greater than 3 nm have not been evaluated. Therefore, extrapolation of the certified values listed in Table 1 beyond 3 nm is not valid.

Table 1. SRM 2034 Certified Wavelengths (nm) of minimum transmittance for 14 bands 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 $U = \pm 0.10$ nm, determined from the root-mean-square combination of component standard uncertainties (i.e., estimated standard deviations) and a "coverage factor" of $k = 2$ computed according to the ISO guide [1]. The coverage factor is based on the Student's t-distribution for >30 effective degrees of freedom and defines the interval within which the unknown value of the band minimum wavelength can be asserted to lie with a level of confidence of approximately 95 %. Components of the measurement uncertainty include: calibration of the NIST spectrophotometer wavelength scale against atomic spectral lines, estimation of absorption band minima, and possible wavelength shifts due to temperature and concentration of the solution.

Production and Certification Procedure: Specific details concerning the materials, instrumentation, and method used in the certification of SRM 2034 are given in references 2 and 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 spectrophotometer used for the certification are also described in reference [2].

Storage and Handling: Proper handling and storage of SRM 2034 is essential to maintain the integrity of the certified wavelength values given in Table 1. 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, discontinue use of the SRM and dispose of properly.

To maintain the integrity of SRM 2034, the cuvette should be handled only by the capped end or by its opposing frosted sides. Avoid unnecessary stress to the glue seal of the cuvette cap. While not in use, SRM 2034 should be

stored in the container provided at a temperature between 20 °C and 30 °C.

Instructions for Use: Carefully insert SRM 2034 into the sample beam of the spectrophotometer being tested and leave the reference beam empty. Scan the desired bands to measure their locations of minimum transmittance for known spectral bandwidth conditions. Take all measurements at a temperature of 25 °C ± 5 °C.

Compare the measured wavelength of the minimum transmittance of a specific band to its certified wavelength in Table 1 for the spectral bandwidth most representative of the spectrophotometer being tested. Taking into account the certification uncertainty of ± 0.1 nm for SRM 2034, any significant biases indicated by subtracting the certified wavelengths from the corresponding measured wavelengths may be plotted as a function of the wavelengths (either measured or certified, since the difference is small). This plot, with a smooth line drawn through the fourteen measured bias points, represents a bias correction wavelength calibration for the instrument under test. To find the true wavelength corresponding to an indicated wavelength, subtract the bias corresponding to the wavelength from the indicated wavelength. To find the indicated wavelength corresponding to a desired true wavelength, add the bias value corresponding to the wavelength to the true wavelength.

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994); (available at http://physics.nist.gov/Pubs/).

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

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

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.

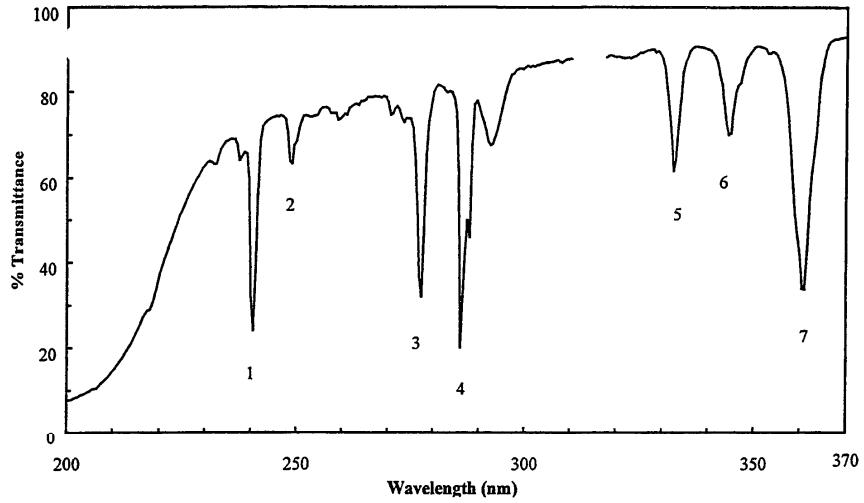

Fig. 1

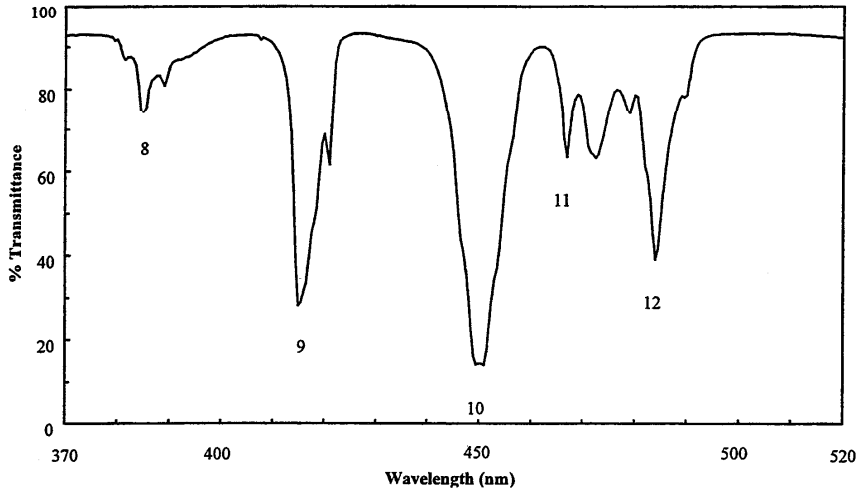

Fig. 2

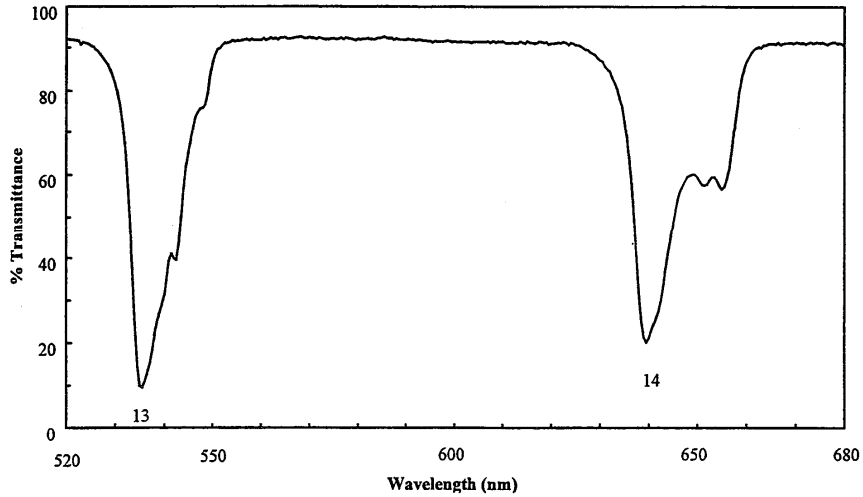

Fig. 3

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