



National Institute of Standards & Technology

Certificate

Standard Reference Material® 2034

Holmium Oxide Solution Wavelength Standard from 240 nm to 650 nm

Series No. 96

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 prepared in 10 % (volume fraction) perchloric acid (HClO_4) to contain 4 % (mass fraction) holmium oxide (Ho_2O_3). The solution is contained in a flame-sealed, nonfluorescent, fused silica cuvette of optical quality. 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 path length of 10 mm and fits in the sample compartment of most conventional absorption spectrophotometers.

CAUTION: SRM 2034 is a perchloric acid solution of holmium oxide in cuvettes that have 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. 96 are given in Table 1 of this Certificate and have been shown to apply to all preceding series as well.

Expiration of Certification: The certified wavelengths are valid for at least 10 years, provided the SRM is handled and stored in accordance with the Instructions for Use. Wavelength verification at 5-year intervals is recommended after the first 10 years of use (see section on Wavelength Verification). Wavelength verification is performed periodically at NIST on control samples from each previous series of SRM 2034 to confirm stability.

The production and certification of SRM 2034 Series No. 96 were performed by J.C. Travis and M.V. Smith of the NIST Analytical Chemistry 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.

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

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

Gaithersburg, MD 20899
Certificate Issue Date: July 30, 1996
(Revision of certificate dated 6-6-95)

Thomas E. Gills, Chief
Standard Reference Materials Program

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. Note 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" (k) 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 change in 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. NIST 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 Special Publication 260-102.

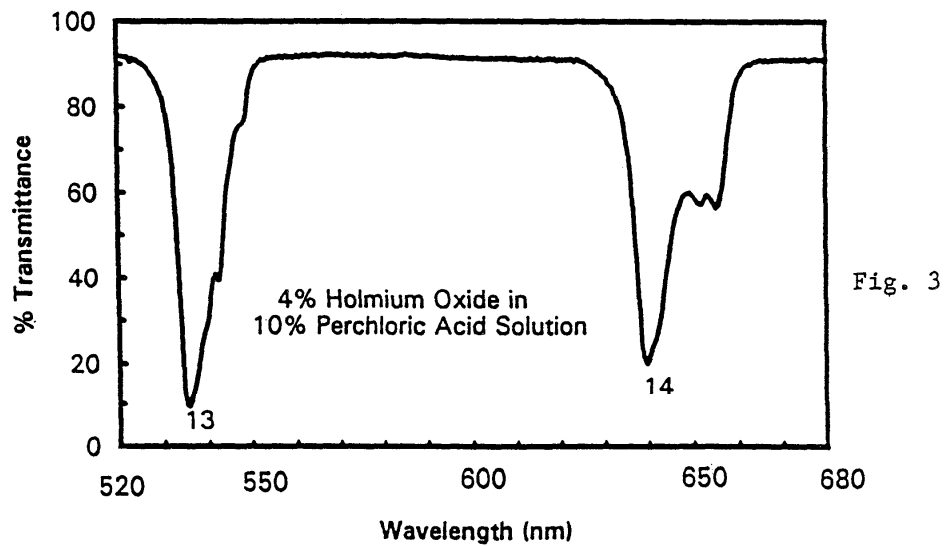
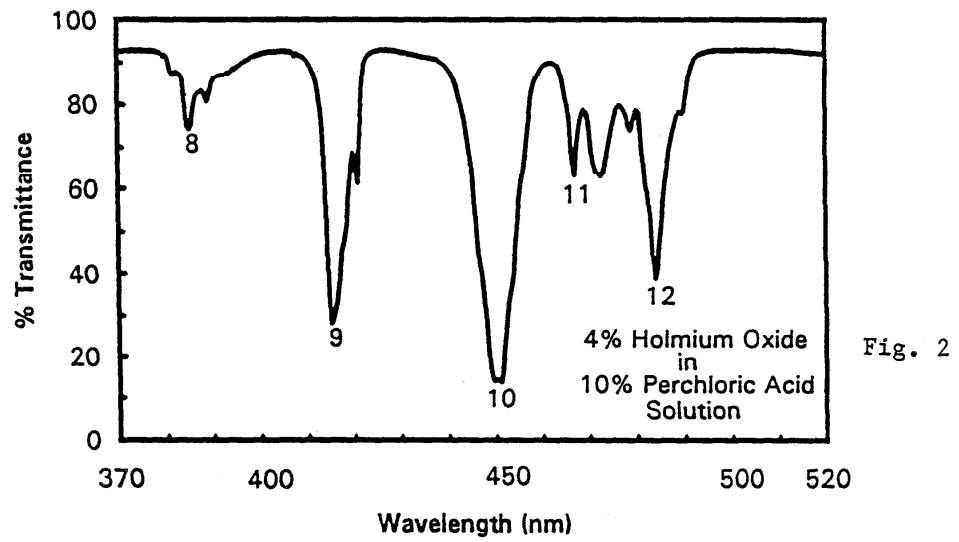
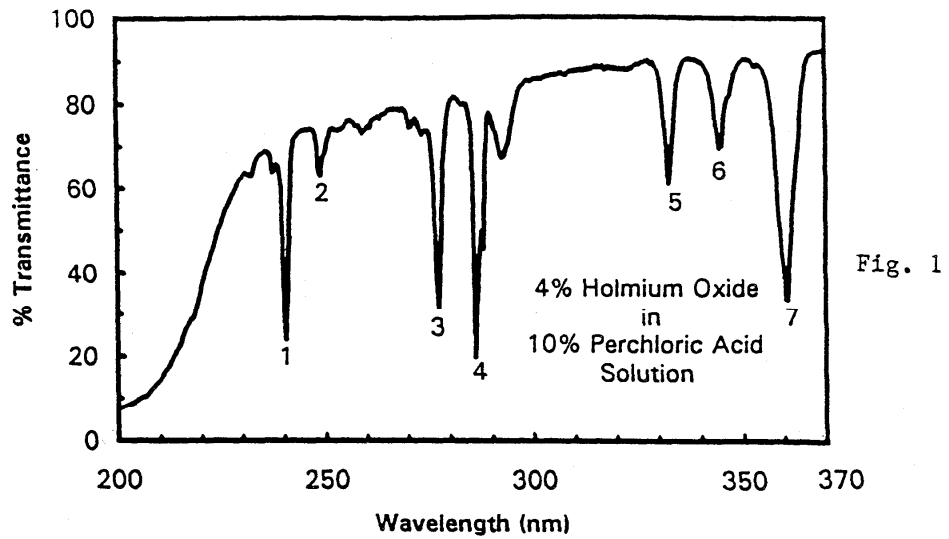


Figure 1-3. Spectral transmittance of a 4% solution of holmium oxide in 10% perchloric acid solution, 200-370 nm, 370-520 nm, and 520-680 nm.

Instructions for Use: 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.

Carefully insert SRM 2034 into the cuvette holder of the sample beam of the spectrometer being tested. Then scan the desired bands to measure their locations of minimum transmittance for known spectral bandwidth conditions. Acquire the transmittance spectrum at a laboratory temperature of $25\text{ °C} \pm 5\text{ °C}$, referenced to air; that is, no cuvette or blank is to be placed in the reference position. 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 $\pm 0.1\text{ nm}$ 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.

Wavelength Verification: Good laboratory practice in the 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, and perhaps invalidate the certified wavelength values, the unit should be returned to NIST for wavelength verification. However, prior to shipment of this SRM, M.V. Smith of the NIST Analytical Chemistry Division should be contacted directly by telephone at (301) 975-4115 or fax at (301) 977-0587 to obtain verification measurement fees and recommended shipping instructions.

REFERENCES

- [1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993).
- [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.*, Vol. 90, No. 2, p. 115-125, (1985).