National Institute of Standards & Technology

Certificate

Standard Reference Material® 2034

Holmium Oxide Solution Wavelength Standard from 240 nm to 650 nm

Series No.: 04-B

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). The SRM is identified on a frosted surface of the cuvette. A protective cap is glued over the fused end of the cuvette. The square-bottomed (12.5 mm × 12.5 mm) cuvette has a nominal pathlength of 10 mm and fits in the sample compartment of most conventional absorption spectrophotometers. *The height of 60 mm precludes the use of SRM 2034 in some instruments.*

CAUTION: SRM 2034 is a perchloric acid solution of holmium oxide. Each SRM 2034 cuvette has been individually vacuum-tested for leaks. If a leak in the cuvette should develop or if the cuvette is accidentally broken, carefully treat the spill immediately with copious amounts of water. The remedial action described in the accompanying MSDS should be undertaken.

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 in Table 1. These certified values apply to previously issued series of SRM 2034 for a period of 10 years from the production year indicated by the series number. The transmittance spectrum of SRM 2034 for a 1 nm spectral bandwidth, referenced to air, is illustrated in Figure 1 of this certificate.

Expiration of Certification: The certification of this SRM is valid, within the measurement uncertainties specified, until **31 December 2014**, provided the SRM is used in accordance with the instructions given in this certificate. However, the certification will be nullified if the cuvette itself is damaged or contaminated.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

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

The production and certification of this SRM were performed by J.C. Travis, D.L. Duewer, 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.R. Anderson of the NIST Fabrication Technology Division.

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Certificate Issue Date: 07 April 2005

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Statistical consultation for this SRM was provided by H-k. Liu of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by C.S. Davis of the NIST Measurement Services Division.

Table 1. SRM 2034 Certified Wavelengths (nm) of Minimum Transmittance and Uncertainties[a] for 14 Bands at 6 Spectral Bandwidths (Referenced to Air)

Band No.	0.1 nm	0.3 nm	0.5 nm	1 nm	2 nm	3 nm
1	240.97 ± 0.05	240.98 ± 0.05	241.02 ± 0.05	241.12 ± 0.05	241.12 ± 0.05	241.04 ± 0.05
2	249.78 ± 0.05	249.79 ± 0.05	249.81 ± 0.05	249.89 ± 0.05	250.03 ± 0.05	250.07 ± 0.05
3	278.15 ± 0.05	278.15 ± 0.05	278.15 ± 0.05	278.13 ± 0.05	278.10 ± 0.05	278.05 ± 0.05
4	287.03 ± 0.05	287.04 ± 0.05	287.08 ± 0.05	287.22 ± 0.05	287.52 ± 0.05	287.57 ± 0.05
5	333.48 ± 0.04	333.47 ± 0.05	333.47 ± 0.05	333.48 ± 0.05	333.47 ± 0.05	333.47 ± 0.05
6	345.46 ± 0.05	345.45 ± 0.05	345.43 ± 0.05	345.38 ± 0.05	345.42 ± 0.05	345.53 ± 0.05
7	361.27 ± 0.05	361.27 ± 0.05	361.27 ± 0.05	361.25 ± 0.05	361.12 ± 0.05	361.11 ± 0.05
8	385.36 ± 0.05	385.39 ± 0.04	385.45 ± 0.04	385.61 ± 0.04	385.80 ± 0.04	386.00 ± 0.04
9	416.02 ± 0.05	416.04 ± 0.05	416.07 ± 0.05	416.25 ± 0.05	416.57 ± 0.05	416.89 ± 0.05
10	-----[b]	-----[b]	-----[b]	451.45 ± 0.05	451.32 ± 0.04	451.36 ± 0.04
11	467.78 ± 0.04	467.79 ± 0.04	467.80 ± 0.04	467.82 ± 0.04	467.90 ± 0.04	468.11 ± 0.04
12	485.20 ± 0.04	485.21 ± 0.04	485.21 ± 0.04	485.23 ± 0.04	485.25 ± 0.04	485.21 ± 0.04
13	536.42 ± 0.04	536.43 ± 0.04	536.45 ± 0.04	536.56 ± 0.04	536.86 ± 0.04	537.21 ± 0.04
14	640.41 ± 0.04	640.41 ± 0.04	640.43 ± 0.04	640.50 ± 0.04	640.79 ± 0.04	641.15 ± 0.04

[a] The uncertainties represent U_{95}, the expanded uncertainty calculated in accordance with reference 1.
[b] The wavelengths for the three narrowest spectral bandwidths for Band No. 10 are not given because this band resolves into two minima for spectral bandwidths of nominally less than 1 nm.

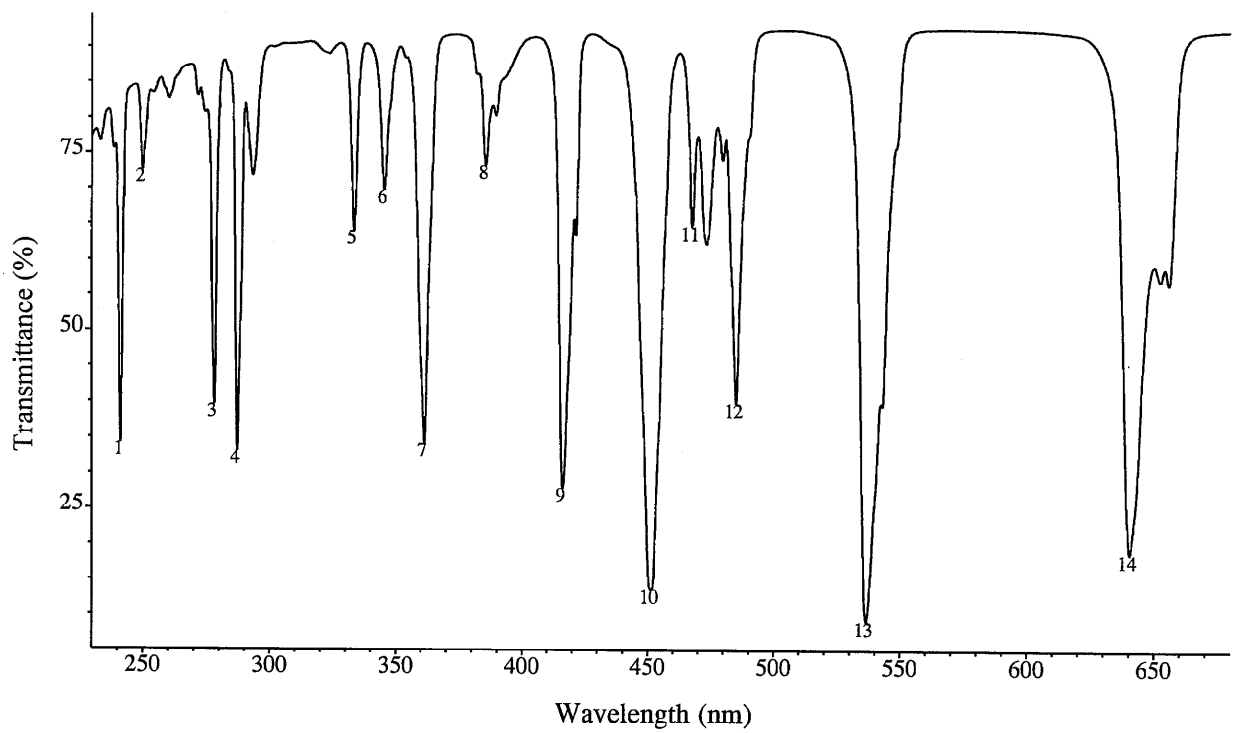

Figure 1. Spectral transmittance at 1 nm spectral bandpass of a 4 % (mass fraction) solution of holmium oxide in 10 % (volume fraction) perchloric acid.

Production and Certification Procedure: Specific details concerning the materials and methods used in the production of SRM 2034 are given in references 2 and 3. The wavelengths of minimum transmittance have been newly assigned based on a multi-center, multinational study involving 22 research-grade spectrophotometers calibrated against independent wavelength standards [4]. 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.

Certification Uncertainty: The uncertainties for SRM 2034 are based upon multiple determinations of the transmittance minima using the independently calibrated instruments of the multi-center study [4]. The expanded uncertainty, U_{95}, for each of the wavelength values given in Table 1 is the estimated standard deviation of the mean of 15 values, with a coverage factor based on Student's *t*-distribution. The expanded uncertainty defines 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 % [1]. Components of the measurement uncertainty including reproducibility, the calibration of the wavelength scale of different spectrophotometers against atomic spectral lines, and the estimation of absorption band minima are confounded into the final result by the experimental design. Wavelength shifts due to temperature and concentration of the solution have been shown to be negligible.

The certified values of Table 1 are repeated in Table 2, but with measurement uncertainty shown as a prediction interval at a level of confidence of 95 % for a single future data acquisition with an instrument from the population that produced the certified values [5]. This table is presented as an aid in the normal application of this material to a single instrument and data set (see "Instructions for Use").

Table 2. SRM 2034 Certified Wavelengths (nm) of Minimum Transmittance and Prediction Intervals[a] for 14 Bands at 6 Spectral Bandwidths-Referenced to Air

Band No.	0.1 nm	0.3 nm	0.5 nm	1 nm	2 nm	3 nm
1	240.97 ± 0.21	240.98 ± 0.21	241.02 ± 0.21	241.12 ± 0.23	241.12 ± 0.29	241.04 ± 0.38
2	249.78 ± 0.21	249.79 ± 0.21	249.81 ± 0.21	249.89 ± 0.22	250.03 ± 0.27	250.07 ± 0.34
3	278.15 ± 0.20	278.15 ± 0.20	278.15 ± 0.20	278.13 ± 0.22	278.10 ± 0.27	278.05 ± 0.35
4	287.03 ± 0.20	287.04 ± 0.20	287.08 ± 0.20	287.22 ± 0.22	287.52 ± 0.27	287.57 ± 0.35
5	333.48 ± 0.18	333.47 ± 0.18	333.47 ± 0.19	333.48 ± 0.20	333.47 ± 0.26	333.47 ± 0.34
6	345.46 ± 0.20	345.45 ± 0.19	345.43 ± 0.19	345.38 ± 0.20	345.42 ± 0.25	345.53 ± 0.33
7	361.27 ± 0.19	361.27 ± 0.19	361.27 ± 0.19	361.25 ± 0.21	361.12 ± 0.26	361.11 ± 0.33
8	385.36 ± 0.19	385.39 ± 0.18	385.45 ± 0.18	385.61 ± 0.19	385.80 ± 0.24	386.00 ± 0.31
9	416.02 ± 0.21	416.04 ± 0.20	416.07 ± 0.21	416.25 ± 0.22	416.57 ± 0.27	416.89 ± 0.35
10	-----[b]	-----[b]	-----[b]	451.45 ± 0.24	451.32 ± 0.23	451.36 ± 0.30
11	467.78 ± 0.15	467.79 ± 0.16	467.80 ± 0.16	467.82 ± 0.17	467.90 ± 0.21	468.11 ± 0.28
12	485.20 ± 0.17	485.21 ± 0.17	485.21 ± 0.17	485.23 ± 0.10	485.25 ± 0.23	485.21 ± 0.30
13	536.42 ± 0.17	536.43 ± 0.17	536.45 ± 0.17	536.56 ± 0.19	536.86 ± 0.23	537.21 ± 0.31
14	640.41 ± 0.15	640.41 ± 0.15	640.43 ± 0.15	640.50 ± 0.16	640.79 ± 0.20	641.15 ± 0.25

[a] The uncertainties represent P_{95}, the approximate 95 % prediction interval for the next single measurement from a participating instrument, calculated in accordance with reference 5.

[b] The wavelengths for the three narrowest spectral bandwidths for Band No. 10 are not given because this band resolves into two transmittance minima for spectral bandwidths of nominally less than 1 nm.

Handling and Storage: 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. If the cuvette is damaged, discontinue use of the SRM and dispose of it properly.

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.

Wavelength Scale Verification: Compare the measured wavelength of a specific band's minimum transmittance to its certified wavelength in Table 1, for the spectral bandwidth most representative of the spectrophotometer being tested. The absolute value of the difference between the measured and certified value for each band should not exceed the certification uncertainty given in Table 1 by more than the wavelength accuracy tolerance specified for the instrument under test or the desired application. The prediction intervals of Table 2 represent realistic lower limits for establishing accuracy tolerances for the various bands. In the event that verification of the native wavelength scale is not obtained over the wavelength range, an external calibration curve may be constructed.

Wavelength Scale Calibration: 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] ISO; *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; 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.; Adams, B.; *Holmium Oxide Solution Wavelength Standard from 240 nm 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.; Adams, B.; *Spectral Transmittance Characteristics of Holmium Oxide in Perchloric Acid*; J. Res. Natl. Bur. Stds., Vol. 90, No. 2, pp. 115–125 (1985).

[4] Travis, J.C.; Acosta, J.C.; Andor, G.; Bastie, J.; Blatner, P.; Chunnilall, C.; Crosson, S.C.; Duewer, D.L.; Early, E.A.; Hengstberger, F.; Kim, C-S; Liedquist, L.; Manoocheri, F.; Mercader, F.; Mito, A.; Monard, L.A.G.; Nevas, S.; Nilsson, M.; Noel, M.; Rodriquez, A.C.; Ruiz, A.; Schirmacher, A.; Smith, M.V.; Valencia, G.; van Tonder, N.; Zwinkels, J.; *Intrinsic Wavelength Standard Absorption Bands in Holmium Oxide Solution for UV/Visible Molecular Absorption Spectrophotometry*; J. Phys. Chem. Ref. Data in (press for 2005).

[5] Walpole, R.E.; Meyers, R.H.; *Probability and Statistics for Engineers and Scientists*, 5th ed.; Prentice Hall, Inc.: Englewood Cliffs, NJ (1993).

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; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.