National Institute of Standards & Technology

Certificate

Standard Reference Material® 2034

Holmium Oxide Solution Wavelength Standard (240 nm to 650 nm)

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. A unit of SRM 2034 consists of a solution 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 × 12.5 mm) cuvette has a nominal pathlength of 10 mm and the height is 60 mm, which fits in the sample compartment of most conventional absorption spectrophotometers. The solution contains 4 % (mass fraction) holmium oxide (Ho_2O_3) in 10 % (volume fraction) perchloric acid ($HClO_4$).

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 all series of SRM 2034 as well as the present one. The transmittance spectrum of SRM 2034 for a 1 nm spectral bandwidth, referenced to air, is illustrated in Figure 1. The measurands are the certified wavelengths in Table 1 and are metrologically traceable to the SI derived units expressed as nanometers.

Expiration of Certification: The certification of **SRM 2034** is valid indefinitely, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). Accordingly, periodic recalibration or recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this material. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of technical measurements leading to certification were performed by S.J. Choquette of the NIST Office of Reference Materials.

Production and certification of this SRM were performed by J.C. Travis, M.V. Smith, and B. Lang of the NIST Biosystems and Biomaterials Division and D.L. Duewer of the NIST Chemical Sciences Division.

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

Support aspects involved with the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Vacuum testing and flame sealing of the fused-silica cuvettes for this SRM were performed by A. Kirchhoff of the NIST Fabrication Technology Division.

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Table 1. SRM 2034 Certified Wavelengths (nm) of Minimum Transmittance and Uncertainties[a] for 14 Bands at Six 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 l.
[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.

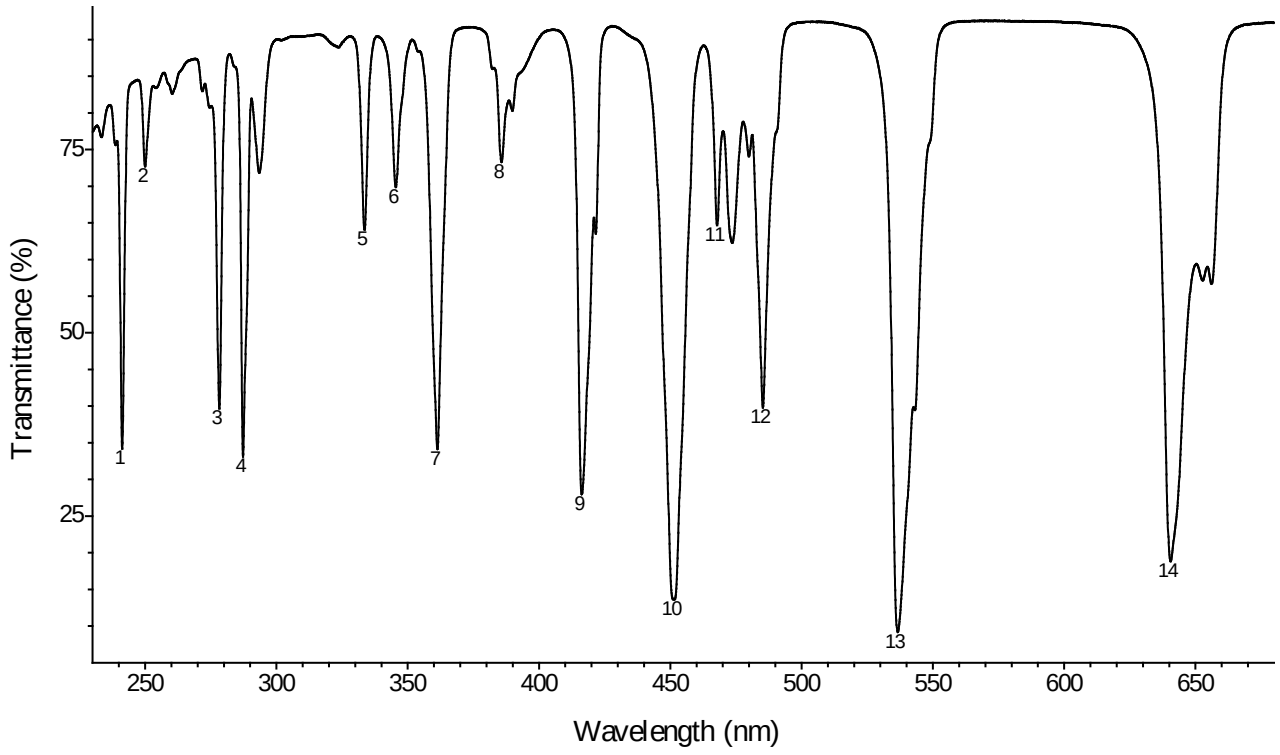

Figure 1. Spectral transmittance at 1 nm spectral bandpass of a 4 % solution of holmium oxide in 10 % perchloric acid solution.

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 the 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. Possible wavelength shifts due to temperature and concentration of the solution have been shown to be negligible.

Information Values: Information values for the prediction intervals at an approximate 95 % confidence level for the certified wavelengths are given in Table 2. These values are intended for a single future data acquisition with an instrument from the population of 22 pen-lamp calibrated research-grade spectrophotometers that produced the certified values [4]. This table is presented as an aid in the normal application of this material to a single instrument and data set (see "Instructions for Handling, Storage, and Use"). Information values cannot be used to establish metrological traceability.

Table 2. Information Values of the Approximate 95 % Prediction Intervals[(a)] for 14 Bands at Six Spectral Bandwidths, Referenced to Air for SRM 2034

Band No.	0.1 nm	0.3 nm	0.5 nm	1 nm	2 nm	3 nm
1	240.76 – 241.18	240.77 – 241.19	240.81 – 241.23	240.89 – 241.35	240.83 – 241.41	240.66 – 241.42
2	249.57 – 249.99	249.58 – 250.00	249.60 – 250.02	249.67 – 250.11	249.76 – 250.30	249.73 – 250.41
3	277.95 – 278.35	277.95 – 278.35	277.95 – 278.35	277.91 – 278.35	277.83 – 278.37	277.70 – 278.40
4	286.83 – 287.23	286.84 – 287.24	286.88 – 287.28	287.00 – 287.44	287.25 – 287.79	287.22 – 287.92
5	333.30 – 333.66	333.29 – 333.65	333.28 – 333.66	333.28 – 333.68	333.21 – 333.73	333.13 – 333.81
6	345.26 – 345.66	345.26 – 345.64	345.24 – 345.62	345.18 – 345.58	345.17 – 345.67	345.20 – 345.86
7	361.08 – 361.46	361.08 – 361.46	361.08 – 361.46	361.04 – 361.46	360.86 – 361.38	360.78 – 361.44
8	385.17 – 385.55	385.21 – 385.57	385.27 – 385.63	385.42 – 385.80	385.56 – 386.04	385.69 – 386.31
9	415.81 – 416.23	415.84 – 416.24	415.86 – 416.28	416.03 – 416.47	416.30 – 416.84	416.54 – 417.24
10	-----[(b)]	-----[(b)]	-----[(b)]	451.21 – 451.69	451.09 – 451.55	451.06 – 451.66
11	467.63 – 467.93	467.63 -467.95	467.64 -467.96	467.65 – 467.99	467.69 – 468.11	467.83 – 468.39
12	485.03 -485.37	485.04 -485.38	485.04 -485.38	485.13 – 485.33	485.03 – 485.48	484.91 – 485.51
13	536.25 – 536.59	536.26 – 536.60	536.28 – 536.62	536.37 – 536.75	536.63 – 537.09	536.90 – 537.52
14	640.26 – 640.56	640.26 – 640.56	640.28 – 640.58	640.34 – 640.66	640.59 – 640.99	640.90 – 641.40

[(a)] The uncertainties represent P_{95}, the approximate 95 % prediction interval for the next single measurement from a participating instrument.

[(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.

CAUTION: SRM 2034 is a perchloric acid solution of holmium oxide. Each SRM 2034 cuvette has been individually vacuum-tested for leaks. If the cuvette should develop a leak or if the cuvette is accidentally broken, refer to the Safety Data Sheet (SDS) for safe handling instructions.

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 HANDLING, STORAGE, AND USE

Handling and Storage: 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 it properly.

Use: Carefully insert SRM 2034 into the sample beam of the spectrophotometer being tested and leave the reference beam empty. *The height of 60 mm precludes the use of SRM 2034 on some instruments.* 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] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Aug 2016); 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://www.nist.gov/pml/pubs/index.cfm (accessed Aug 2016).

[2] Weidner, V.R.; Mavrodineanu, R.; Mielenz, K.D.; Velapoldi, R.A.; Eckerle, K.L.; Adams, B.; *Holmium Oxide Solution Wavelength Standard from 240 to 640 nm - SRM 2034*; NBS Special Publication 260-102 (1986); available at http://www.nist.gov/srm/publications.cfm (accessed Aug 2016).

[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, Vol. 34, pp. 41–56 (2005).

Certificate Revision History: 29 August 2016 (Removed series number; removed expiration date; change certified values in Table 2 to information values; editorial changes); 07 April 2005 (Series 04-B); 21 October 2004 (Series 04-A); 18 December 2002 (Series 02); 04 February 2002 (Update Table l for Series 01); 28 November 2001 (Series 01); 01 March 2000 (Series 99); 15 December 1998 (Series 98); 02 December 1997 (Series 97); 30 July 1996 (Series 96); 06 June 1995 (Series 95); 17 February 1994 (Series 94 revision); 02 December 1993 (Series 94); 03 February 1992 (Series No. 91); 20 June 1985 (Original certificate date.)

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.