National Institute of Standards & Technology

Certificate

Standard Reference Material® 1822a

Refractive Index Standard

This Standard Reference Material (SRM) is a soda-lime glass plate, approximately 25 mm × 25 mm × 3 mm, with ground faces. It is designed to be gradually consumed in use by breaking off pieces that are crushed into small particles for oil immersion refractometry. SRM 1822a is intended for use in checking the accuracy of visible refractometers based on the immersion of small glass particles in oil, which are used extensively in the forensic science community. A unit of SRM 1822a consists of one plate inside a protective box.

Certified Refractive Index and Thermo-optic Coefficient Values: The certified values for the refractive index, *n*, at 22 °C for six visible wavelengths are listed in Table 1.

Table 1. Certified Refractive Index Values for SRM 1822a.

Vacuum Wavelength (nm)	Refractive Index (n) at 22 °C
480.1254	1.526132 ± 0.000016
501.7077	1.524468 ± 0.000016
508.7240	1.523971 ± 0.000016
546.2260	1.521629 ± 0.000016
587.7254	1.519535 ± 0.000016
644.0250	1.517277 ± 0.000016

In order to facilitate the use of the SRM at other wavelengths in-between the measured values, a least-squares fit of the six data points was performed to the following standard functional form:

$$n(\lambda) = a_1 - a_2\lambda^2 + \frac{a_3}{\lambda^2} + \frac{a_4}{\lambda^4}, \qquad (1)$$

with $a_1 = 1.508373$, $a_2 = 2.679 \times 10^{-3}$, $a_3 = 4.051 \times 10^{-3}$, and $a_4 = 4.2703 \times 10^{-5}$. The residuals to the fit are less than 2×10^{-6}, and it is believed that the fit values are at least as accurate as the average measured values over the spectral range from 480.1 nm to 644.0 nm. The thermo-optic coefficient, dn/dT, has been determined at two visible wavelengths. The certified values are dn/dT = 1.2×10^{-6}/°C at 632.8 nm and 2.2×10^{-6}/°C at 543.5 nm, with an expanded uncertainty of 3×10^{-7}/°C.

Expiration of Certification: The certification of SRM 1822a is deemed to be valid, within the measurement uncertainties specified, until **31 December 2011**, provided the SRM is stored and handled in accordance with the instructions given in this certificate (see "Notice and Warning to Users"). Certification will be nullified if the SRM is damaged or contaminated. NIST reserves the right to withdraw, amend, or extend this certification at any time.

The overall direction and coordination of the technical measurements were performed by R.U. Datla of the NIST Optical Technology Division.

The technical measurements leading to certification were performed by S.G. Kaplan of the NIST Optical Technology Division.

Gerald T. Fraser, Chief
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See Certificate Revision History on Page 4

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Statistical consultation was provided by D.D. Leber of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Maintenance of SRM Certification: The refractive index of the glass material has been measured by NIST over a period of several years, and no significant change has been noted. NIST will continue to 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. Registration (see attached sheet) will facilitate notification.

NOTICE AND WARNINGS TO USERS

Storage and Handling: SRM 1822a should be handled with clean plastic or thin rubber gloves. When not in use, the SRM should always be kept in its accompanying protective box. It is recommended that the SRM be stored in a dessicator if available. Do NOT store at or above 100 °C.

INSTRUCTIONS FOR USE

Test Measurements: A small piece will be broken off from the glass plate and crushed into small particles for examination in an oil-immersion microscope. It is recommended that a sufficient number of particles be examined to elucidate any possible effects of residual strain from the crushing process, as well as any effects of temperature inhomogeneity in the immersion medium.

Comparison with Certified Values: The primary purpose of this SRM is to check the accuracy of the user's immersion refractometer. If the user's values differ from the certified values by less than the quadrature sum of the certified expanded uncertainties and the expanded uncertainty of the mean of the user's values, then the user's values are accurate to this level, and no correction should be attempted on the basis of comparison with this SRM. If the user's measured values differ significantly from the certified values, then recalibration of the instrument or reevaluation of the immersion oil may be indicated.

Source of Material: The glass plates for this SRM were obtained from a subset of SRM 1830 Soda-Lime Float Glass (0.1 % Al_2O_3), a glass chemical composition standard. The plates were found to have measurable strain birefringence due to the float process used to manufacture the glass. Also, the surface layer that was in contact with the molten tin is known to change its refractive properties because of the incorporation of tin. These potential refractive index uniformity problems were eliminated by (a) grinding several micrometers of material from the faces of each plate, and (b) annealing the plates to remove the residual strain effects, verified by observing the plates through crossed polarizers.

CERTIFICATION ANALYSIS

Measurement Conditions: The certified refractive index values were measured using a Wild[1] manually operated visual minimum-deviation prism refractometer. The temperature of the instrument and surrounding air was approximately 22 °C, and the atmospheric pressure and humidity were monitored in order to correct for changes in the refractive index of the air [1]. Measurements were performed on six 60° prisms, cut from a randomly selected subset of the SRM plates, at six visible wavelengths emitted by low-pressure Hg, He, and Cd lamps. Repeated measurements were made on each sample of the apex angle, α, and minimum-deviation angle, Δ, for each wavelength to determine the refractive index, n, according to the following formula:

$$n = \frac{\sin\left(\frac{\alpha + \Delta}{2}\right)}{\sin\left(\frac{\alpha}{2}\right)} n_{air}, \qquad (2)$$

where n_{air} is the refractive index of the surrounding air.

[1]Certain commercial equipment, instruments, or materials are identified in this certificate in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for this purpose.

Thermo-optic coefficient measurements at two laser wavelengths (632.8 nm and 543.5 nm) were carried out using another minimum-deviation refractometer in which the sample temperature was varied from 10 °C to 30 °C with 0.01 °C stability and 0.05 °C uncertainty. Repeated measurements were made at both wavelengths on two 30° prisms at a set of temperatures, and dn/dT was determined by a linear least-squares fit to the data.

Determination of Uncertainty in Refractive Index: The combined uncertainty includes "Type A" uncertainties, which are evaluated by statistical methods, and "Type B" uncertainties, which are determined by other means [2]. The apex angle, α, was measured by repeatedly observing the retro-reflected beams from each prism face, rotating the central table to different positions in order to reduce errors due to nonlinearities in the angular scale. The prisms were also flipped over and remeasured to assess the effects of pyramidal error on the measured value of α. Repeated observations of Δ were made on both sides of the prism, and the alignment was checked and corrected between each set of observations. In addition, two of the samples (2 and 4) were removed, replaced, and remeasured on another day to assess the reproducibility of the results. Samples 5 and 6 were also flipped over and remeasured on the Hg line (546.226 nm).

Table 2 lists the components of uncertainty, including repeatability of the deviation angle measurements, σ_{Δ}, reproducibility of the index measurement for each sample, σ_{repro}, uncertainty of the apex-angle measurement, σ_{apex}, temperature drift, σ_{temp}, sample-to-sample material variation, σ_{sample}, and the estimated uncertainty in n of the surrounding air, σ_{air}. The final expanded uncertainty U (coverage factor k = 2) is calculated from the following formula with a resultant value of 1.6×10^{-5}, which can be used over the wavelength range of 480.1 nm to 644.0 nm [2].

$$U = 2\sqrt{\frac{\sigma_{\Delta}^{2}}{6} + \frac{\sigma_{repro}^{2}}{2} + \frac{\sigma_{apex}^{2}}{2} + \sigma_{temp}^{2} + \sigma_{sample}^{2} + \sigma_{air}^{2}} \qquad (3)$$

Table 2. Sources of Uncertainty in the Refractive Index Measurements with Estimated Values for Each Component and Final Expanded Uncertainty in n

Uncertainty Source	Type	Estimated Value	Uncertainty in n
Repeatability of Deviation Measurements	A	0.7 arc sec	3×10^{-6}
Reproducibility of Index Measurements	A	2×10^{-6}	2×10^{-6}
Apex Angle	A	2 arc sec	6.5×10^{-6}
Temperature Drift	B	0.3 °C	6×10^{-7}
Sample-to-Sample Variation	B	3×10^{-6}	3×10^{-6}
Index of Air	B	5×10^{-7}	5×10^{-7}
Expanded Uncertainty (k = 2)			**1.6×10^{-5}**

For the thermo-optic coefficient, the measured angular shifts were graphed and fit to a linear form using least-squares analysis. The value of dn/dT was derived from the slope of the line and averaged for the two samples. The resulting values are dn/dT = 1.2×10^{-6}/°C at 632.8 nm and 2.2×10^{-6}/°C at 543.5 nm. Drift in the temperature of the room air and mechanical drift in the refractometer detector arm are the important contributors to variability in the measured values of dn/dT, yielding an expanded uncertainty of 3×10^{-7}.

REFERENCES

[1] Edlén, B; *The Refractive Index of Air*; Metrologia, Vol. 2, No. 2, pp. 71–80 (1966).

[2] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization of 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).

Certificate Revision History: 14 November 2008 (Editorial changes to constant values in equation 1); 24 January 2007 (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 at telephone (301) 975-2200 fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.