



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 936a

Quinine Sulfate Dihydrate

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and the calibration of fluorescence spectrometers. It is certified for the relative molecular emission spectrum ($E(\lambda)$) in radiometric units for a solution of 1.28×10^{-6} mol/L quinine sulfate dihydrate in 0.105 mol/L perchloric acid using an excitation wavelength of 347.5 nm. The certified values of the molecular emission spectrum at 5 nm wavelength intervals from 375 to 675 nm are given in Table 1. These values have been corrected for the instrument and sample parameters, including the spectral responsivity of the detection system, monochromator bandwidth, photomultiplier tube nonlinearity, monochromator wavelength error, solvent refractive index, and cell window transmittance. The relative standard error (RSE) in $E(\lambda)$, $RSE[E(\lambda)]$, is given in Table 1. The estimate of the relative systematic error limits (RSEL) in the molecular emission spectrum, $RSEL[E(\lambda)]$, is also given in Table 1 and was determined by the addition of the absolute values of the estimated systematic errors. These relative error limits include uncertainties in the calibration values for the spectral responsivity, the wavelength peak maximum, and in the corrections applied for instrument and sample parameters. A unit of SRM 936a consists of 1 g of crystalline material.

From the certified values of $E(\lambda)$, values may be calculated for the molecular emission spectrum in the various photon, radiometric, wavelength, and wavenumber units using the following equation: [1,2]

$$E(\lambda) = \frac{E_p(\lambda)}{\lambda} = \frac{E_\nu}{\lambda^2} = \frac{E_p(\tilde{\nu})}{\lambda^3}$$

These values have been calculated and are given in NBS Special Publication 260-64 [3]. The technical emission spectrum, $E^T(\lambda)$, i.e., the emission spectrum corrected for instrument parameters only, is also given in SP 260-64.

The SRM contains $1.515 \pm 0.047\%$ (95% confidence interval for the mean) of an impurity as determined by high performance liquid chromatography using a UV/VIS detector with a tungsten lamp that was used to monitor the absorbance at 348 nm. The impurity is believed to be dihydroquinine sulfate dihydrate, which has optical characteristics that are similar to those of the quinine sulfate dihydrate.

Source of Material: The quinine sulfate dihydrate used for SRM 936a was a special lot of material obtained from the J.T. Baker Chemical Co., Phillipsburg, N.J.

The original technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.W. Mears and R.W. Seward. The revision and update of this certificate was coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
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Thomas E. Gills, Chief
Standard Reference Materials Program

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The experimental work leading to the certification of this material was performed by R.A. Velapoldi and K.D. Mielenz of the NIST Analytical Chemistry Division. The experimental work on the revision of this SRM was performed by K.S. Sharpless of the NIST Analytical Chemistry Division. The coordination of this effort was under the supervision of S.A. Wise of the NIST Analytical Chemistry Division.

Statistical analysis on the revision of this certificate was provided by S.B. Schiller of the NIST Statistical Engineering Division.

Storage of Crystalline Material: SRM 936a should be kept in its original bottle and stored in the dark at room temperature (30 °C or less). It should not be subjected to heat or direct sunlight during storage.

Expiration of Certification: This certificate is valid within the specified uncertainty limits for five years from the date of shipment from NIST. If this material degrades beyond the limits certified, purchasers will be notified by NIST. Please return the attached registration card to facilitate notification.

Preparation of Stock Solutions and Use of SRM 936a: This SRM is for "in vitro" diagnostic use as a clinical laboratory standard. A "stock" standard solution prepared using a weighed amount of the crystalline SRM and diluted with appropriate solvents, containing 0.1 mg/mL of quinine sulfate, may be prepared as follows: Weigh 0.100 g of SRM 936a to the nearest one-tenth milligram and quantitatively transfer it to a 1000 mL volumetric flask. Dilute to the calibrated volume with 0.105 mol/L HClO_4 to give a solution that is 0.1 mg/mL (1.28×10^{-4} mol/L) in quinine sulfate.

Storage of Stock Solutions: Store this solution in the dark in a well-stoppered, glass bottle.

Preparation of Working Solutions: A "working" standard solution containing 1 $\mu\text{g/mL}$ may be prepared by transferring 10 mL of the above "stock" standard solution to a 1000 mL volumetric flask and diluting to the calibrated volume with 0.105 mol/L HClO_4 to give a solution that is 1 $\mu\text{g/mL}$ (1.28×10^{-6} mol/L) in quinine sulfate. Store this solution in the same manner as the above "stock" standard solution.

Stability of Quinine Sulfate Solutions: Several opinions regarding the stability of quinine sulfate solutions have appeared in the literature [4]. NIST considers the 0.1 mg/L "stock" standard solution prepared from SRM 936a to be stable for 3 months when stored as specified, and the 1 $\mu\text{g/mL}$ "working" standard solution to be stable for 1 month when so stored.

Table 1. The Molecular Emission Spectrum, $E(\lambda)$, of Quinine Sulfate Dihydrate in 0.105 mol/L HClO_4 , the Relative Standard Error, RSE, and the Estimated Relative Systematic Error Limits, RSEL, in the $E(\lambda)$ Values.

λ , nm	$E(\lambda)$	RSE [$E(\lambda)$]	RSEL [$E(\lambda)$]	λ , nm	$E(\lambda)$	RSE [$E(\lambda)$]	RSEL [$E(\lambda)$]
375.0	0.005	0.019	0.087	525.0	0.302	0.001	0.029
380.0	0.012	0.006	0.078	530.0	0.264	0.003	0.029
385.0	0.028	0.003	0.071	535.0	0.231	0.003	0.029
390.0	0.057	0.003	0.064	540.0	0.201	0.002	0.029
395.0	0.103	0.002	0.059	545.0	0.175	0.002	0.029
400.0	0.170	0.002	0.054	550.0	0.153	0.001	0.029
405.0	0.257	0.003	0.049	555.0	0.132	0.001	0.029
410.0	0.359	0.003	0.045	560.0	0.116	0.001	0.029
415.0	0.471	0.003	0.041	565.0	0.101	0.002	0.029
420.0	0.586	0.003	0.037	570.0	0.088	0.002	0.029
425.0	0.694	0.003	0.034	575.0	0.076	0.003	0.029
430.0	0.792	0.002	0.031	580.0	0.065	0.003	0.029
435.0	0.874	0.002	0.028	585.0	0.057	0.001	0.029
440.0	0.940	0.001	0.026	590.0	0.050	0.003	0.030
445.0	0.984	0.001	0.024	595.0	0.043	0.004	0.030
450.0	0.999	0.001	0.023	600.0	0.037	0.006	0.030
455.0	0.997	0.001	0.023	605.0	0.032	0.002	0.030
460.0	0.982	0.001	0.024	610.0	0.028	0.006	0.030
465.0	0.947	0.001	0.024	615.0	0.024	0.003	0.030
470.0	0.897	0.001	0.025	620.0	0.021	0.011	0.030
475.0	0.838	0.002	0.026	625.0	0.018	0.003	0.030
480.0	0.782	0.002	0.027	630.0	0.016	0.015	0.030
485.0	0.719	0.002	0.027	635.0	0.014	0.014	0.030
490.0	0.657	0.002	0.027	640.0	0.011	0.037	0.030
495.0	0.595	0.003	0.027	645.0	0.010	0.015	0.030
500.0	0.541	0.002	0.027	650.0	0.009	0.027	0.030
505.0	0.486	0.001	0.028	655.0	0.008	0.035	0.031
510.0	0.434	0.003	0.028	660.0	0.007	0.073	0.031
515.0	0.386	0.003	0.028	665.0	0.006	0.046	0.032
520.0	0.342	0.002	0.028	670.0	0.005	0.053	0.032
				675.0	0.004	0.065	0.033

Supplementary Data: The following data for the specific molar absorbances, water content, photon yields, and fluorescence lifetimes are considered to be supplementary and are not to be considered certified values, but are provided as information on the characterization of the SRM.

The quinine sulfate dihydrate (QSD) used for SRM 936a was found to be homogeneous to better than 0.5% by thin-layer chromatography with development by two solvent systems and the determination of specific molar absorbances, ϵ , at three different wavelengths. The ultraviolet absorption spectrum of SRM 936a in a 0.105 mol/L HClO₄ exhibits the following absorption maxima:

$$\begin{aligned} 250.0 \text{ nm, } \epsilon_{\text{max}} &= 56,990 \pm 90 \text{ L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1} \\ 347.5 \text{ nm, } \epsilon_{\text{max}} &= 10,810 \pm 20 \text{ L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1} \end{aligned}$$

and, on the side of a peak:

$$365.0 \text{ nm, } \epsilon_{\text{obs}} = 6,920 \pm 10 \text{ L}\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$$

The water content of this material was measured by two methods. The average of six determinations by the Karl-Fischer method gave a value of $4.74 \pm 0.05\%$, while the average of four determinations by a weight loss procedure gave a value of $4.57 \pm 0.04\%$. The theoretical value for water in quinine sulfate dihydrate is 4.60%.

The photon yield, Q , and the fluorescence lifetime, τ , of SRM 936a were compared to values obtained for a sample of purified quinine sulfate dihydrate and are summarized below:

	Q	τ , ns
	0.5 mol/L H ₂ SO ₄	0.5 mol/L H ₂ SO ₄
SRM 936a, QSD	0.544 ± 0.03	19.1 ± 0.1
Purified QSD	0.546 [5]	19.2 ± 0.1

REFERENCES

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