U.S. Department of Commerce
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National Bureau of Standards
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National Bureau of Standards

Certificate

Standard Reference Material 936

Quinine Sulfate Dihydrate

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This Standard Reference Material 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(λ), 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 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 in E(λ), RSE [E(λ)], is given in table 1. The estimate of the relative systematic error limits in the molecular emission spectrum, RSEL [E(λ)], 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 position of the emission peak maximum, and in the corrections applied for instrument and sample parameters.

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

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

These values have been calculated and are given in NBS Special Publication 260-64.

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

The technical and support aspects concerning the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears and R. W. Seward.

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George A. Uriano, Chief
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Table 1. The Molecular Emission Spectrum, E(λ), 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(λ) Values.

λ,nm	E(λ)	RSE [E(λ)]	RSEL [E(λ)]	λ,nm	E(λ)	RSE [E(λ)]	RSEL [E(λ)]
375.0	0.005	0.019	0.087	525.0	0.302	0.001	0.029
380.0	.012	.006	.078	530.0	.264	.003	.029
385.0	.028	.003	.071	535.0	.231	.003	.029
390.0	.057	.003	.064	540.0	.201	.002	.029
395.0	.103	.002	.059	545.0	.175	.002	.029
400.0	.170	.002	.054	550.0	.153	.001	.029
405.0	.257	.003	.049	555.0	.132	.001	.029
410.0	.359	.003	.045	560.0	.116	.001	.029
415.0	.471	.003	.041	565.0	.101	.002	.029
420.0	.586	.003	.037	570.0	.088	.002	.029
425.0	.694	.003	.034	575.0	.076	.003	.029
430.0	.792	.002	.031	580.0	.065	.003	.029
435.0	.874	.002	.028	585.0	.057	.001	.029
440.0	.940	.001	.026	590.0	.050	.003	.030
445.0	.984	.001	.024	595.0	.043	.004	.030
450.0	.999	.001	.023	600.0	.037	.006	.030
455.0	.997	.001	.023	605.0	.032	.002	.030
460.0	.982	.001	.024	610.0	.028	.006	.030
465.0	.947	.001	.024	615.0	.024	.003	.030
470.0	.897	.001	.025	620.0	.021	.011	.030
475.0	.838	.002	.026	625.0	.018	.003	.030
480.0	.782	.002	.027	630.0	.016	.015	.030
485.0	.719	.002	.027	635.0	.014	.014	.030
490.0	.657	.002	.027	640.0	.011	.037	.030
495.0	.595	.003	.027	645.0	.010	.015	.030
500.0	.541	.002	.027	650.0	.009	.027	.030
505.0	.486	.001	.028	655.0	.008	.035	.031
510.0	.434	.003	.028	660.0	.007	.073	.031
515.0	.386	.003	.028	665.0	.006	.046	.032
520.0	.342	.002	.028	670.0	.005	.053	.032
				675.0	.004	.065	.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.

The quinine sulfate dihydrate (QSD) used for SRM 936 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 SRM contains approximately 1.7% of an impurity as determined by high performance liquid chromatography using absorbance and fluorescence detection. This impurity is believed to be dihydroquinine sulfate dihydrate, which has optical characteristics that are similar to those of the quinine sulfate dihydrate. The ultraviolet absorption spectrum of SRM 936 in 0.105 mol/L $HClO_4$ exhibits the following absorption maxima:

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

and, on the side of a peak:

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

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 936 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_2SO_4	0.5 mol/L H_2SO_4
SRM 936, QSD	0.544 ± 0.03	19.1 ± 0.1
Purified QSD	0.546[a]	19.2 ± 0.1

[a]Melhuish, W. H., J. Phys. Chem. **65**, 229 (1961); *ibid,* New Zealand J. Sci. Tech. **37**, 142 (1955).

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PREPARATION AND USE OF SRM 936

This Standard Reference Material is for "in vitro" diagnostic use as a clinical laboratory standard. A "stock" standard solution containing 0.1 mg/mL of quinine sulfate may be prepared as follows: Weigh 0.100 g of SRM 936 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 1.28×10^{-4} mol/L (0.1 mg/mL) in quinine sulfate. Store this solution in the dark in a well-stoppered, glass bottle. A "working" standard solution containing 1 μg/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 1.28×10^{-6} mol/L (1 μg/mL) in quinine sulfate. Store this solution in the same manner as the above "stock" standard solution.

Several opinions regarding the stability of quinine sulfate solutions have appeared in the literature [3]. NBS considers the 0.1 mg/mL "stock" standard solution prepared from SRM 936 to be stable for 3 months when stored as specified; and the 1 μg/mL "working" standard solution to be stable for 1 month when so stored.

SRM 936 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. Experience at NBS indicates that under proper storage this material is stable for at least 3 years. If this material degrades beyond the limits certified, purchasers will be notified by NBS. It is recommended that the material not be used after 3 years from the date of purchase.

References:

[1] Ejder, E. J., J. Opt. Soc. Amer. **59**, 223 (1969).

[2] Melhuish, W. H., J. Res. Nat. Bur. Stand. (U.S.) **76A**, No. 6, 547 (1972).

[3] Melhuish, W. H., J. Phys. Chem. **65**, 229 (1961); Gill, J. E., Photochem. and Photobiol. **9**, 313 (1969); Birks, J. B., J. Res. Nat. Bur. Stand. (U.S.) **80A**, 389 (1976); Heller, C. A., Henry, R. A., McLaughlin, B. A., and Bless, D. E., J. Chem. Eng. Data **19**, 214 (1974); West, M. A., and Kemp, D. R., Int'l. Lab., p. 27 (May/June 1976); and White, J. U., Pittsburgh Conf. Abstracts, Paper 488 (1977).

This Standard Reference Material has been measured and certified at the laboratories of the National Bureau of Standards, Gaithersburg, Maryland. All inquiries should be addressed to:

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