

National Bureau of Standards Certificate

Standard Reference Material 931 Liquid Absorbance Standards for Ultraviolet and Visible Spectrophotometry

R. W. Burke and E. R. Deardorff

These liquid filters are intended as reference solutions for checking the accuracy of the photometric scale of narrow bandpass spectrophotometers. They are applicable for calibrating those instruments that can provide an effective spectral bandpass of 1.5 nm or less at 302 nm, 2.0 nm or less at 395 nm, 3.3 nm or less at 512 nm and 8.5 nm or less at 678 nm.

Net Absorbance^a

Filter	Wavelength and (Bandpass), nm			
	302(1.0)	395(1.7)	512(2.0)	678(6.5)
"A"—"Blank"	0.307±0.003	0.304±0.003	0.303±0.003	0.115±0.002
"B"—"Blank"	0.608±0.005	0.605±0.005	0.606±0.005	0.229±0.003
"C"—"Blank"	0.906±0.007	0.907±0.007	0.911±0.007	0.345±0.003

^aNet absorbances ("A"—"Blank," "B"—"Blank," and "C"—"Blank") were determined using 10.00 mm cuvettes at 25.0 °C, see Instructions for Use.

Absorbance measurements were performed on a high precision double-beam spectrophotometer equipped with a double monochromator. The accuracy of the photometric scale of this instrument was established with the NBS high-accuracy spectrophotometer described by R. Mavrodineanu [1]. The uncertainties of the certified values are twice the standard deviation, commonly referred to as the "95 percent confidence level."

These filters are certified for absorbance at 25.0 °C. Absorbances at other temperatures in the range 17 to 37 °C may be calculated using the formula on page 2.

While no long-term stability studies have been performed on this particular lot of filters, studies of similar preparations have indicated that these filters should be stable for at least one year.

The overall direction and coordination of technical measurements leading to certification were performed under the chairmanship of O. Menis and J. I. Shultz.

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

Washington, D.C. 20234
January 17, 1972

J. Paul Cali, Chief
Office of Standard Reference Materials

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Absorbance at various temperatures (17 to 35 °C)

$$A_t = A_{25} [1 + C_A (t - 25)]$$

where: A_t = Absorbance at temperature t (°C)
 A_{25} = Absorbance certified at 25.0 °C
 C_A = Fractional change in absorbance per °C

The values of C_A , at the four wavelengths, are given below. [NOTE: At wavelength 302 nm, absorbance decreases with increasing temperature; at the other wavelengths, absorbance increases with increasing temperature.]

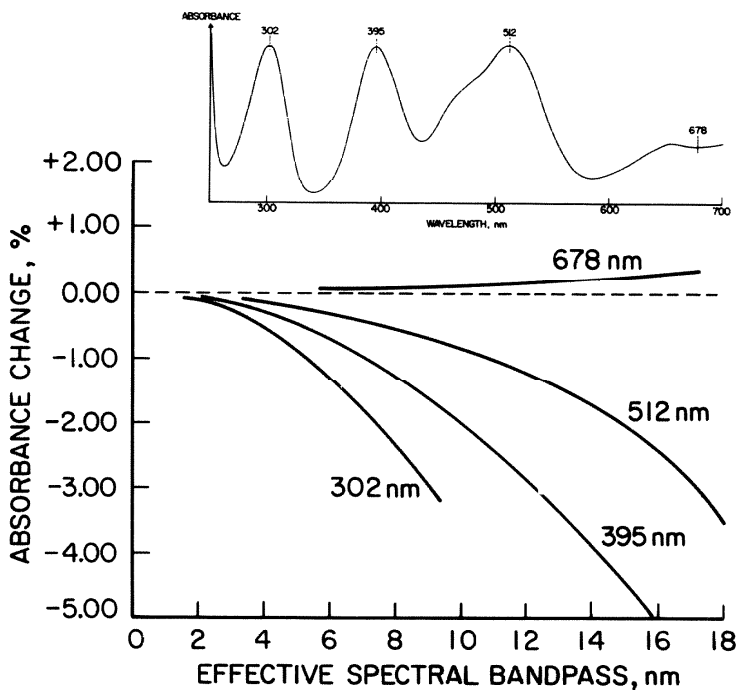
<u>Wavelength, nm</u>	<u>C_A</u>
302	-0.0014
395	+0.0014
512	+0.0018
678	+0.0014

Instructions for Use

1. Select two clean 10.00 mm cuvettes free of scratches. At least one should be fitted with a ground glass or Teflon stopper to minimize evaporation. Reserve it for all sample measurements.
2. Mark each of the cuvettes to assure the same orientation in the spectrophotometer.
3. Place the cuvettes in their respective holders and fill with distilled water. (Borosilicate Pasteur-type pipettes fitted with rubber bulbs are recommended for transferring all solutions to and from the cuvettes. Soft glass pipettes, which are available commercially, contain residual amounts of ultraviolet absorbing material, but may be used after proper cleaning. Several rinses, first with isopropyl alcohol and then with distilled water, are generally adequate.)
4. Obtain the optical mismatch of the cuvettes at 302, 395, 512, and 678 nm, using the spectral bandpass limitations given on the face of the certificate.
5. Empty the cuvettes by suction without removing them from their holders, refill with distilled water and measure the absorbances again at each of the above wavelengths.
6. Repeat the emptying and refilling operation until constant absorbance readings are obtained.
7. Using the liquid filters provided, measure, in turn, the absorbances of the "Blank," "A," "B," and "C" against distilled water. Shake each ampoule before opening to remix any condensate which may have collected in the neck (the ampoules have been prescored directly below the gold band to facilitate opening).
8. Subtract the appropriate "Blank" reading from the absorbances obtained for "A," "B," and "C." These net absorbances should agree with the certified values within the uncertainties specified. Consult the manufacturer of the instrument if they do not.

The absorbances of these filters will depend not only on the accuracy of the photometric scale but also on the wavelength accuracy and the spectral bandpass. A mercury lamp is recommended for checking the wavelength scale. In addition, for those spectrophotometers having a hydrogen (H) or deuterium (D) source, the two emission lines at 486.1 and 656.3 nm (H) or 486.0 and 656.1 nm (D) may provide a convenient check at these wavelengths.

To insure that the measured absorbances are not significantly different from the certified values, the following restrictions are placed on the size of the spectral bandpass selected: To obtain ± 0.1 percent of the true value, the effective spectral bandpass should not exceed 1.5, 2.0, 3.3, and 8.5 nm at 302, 395, 512, and 678 nm, respectively. For ± 0.2 percent, the respective bandpasses are 2.2, 2.9, 4.8, and 12.3 nm. Additional information on the effect of spectral bandpass on the absorbances of these filters is given in the figure below. These curves are not to be used, however, to correct the measured absorbances.



Preparation of filters

The filters were prepared by dissolving high purity cobalt and nickel in a mixture of nitric and perchloric acids. The absorption spectrum of the resulting solution is shown in figure above. The maxima at 302 and 512 nm are due to absorption by NO_3 and $\text{Co}(\text{H}_2\text{O})_6^{2+}$, respectively. The maximum at 395 nm and the plateau at 650-700 nm is due to $\text{Ni}(\text{H}_2\text{O})_6^{2+}$. The pH of these filters is about 1.

Reference

- [1] R. Mavrodineanu, NBS Technical Note 584, O. Menis and J. I. Shultz, ed., pp. 2-21, U.S. Government Printing Office, Washington, D.C. 20402 (Dec. 1971).