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Designation: E 925 – 02

Standard Practice for the Periodic Monitoring the Calibration of Narrow Band-Pass Ultraviolet-Visible Spectrophotometers whose Spectral Slit Width does not Exceed 2 nm¹

This standard is issued under the fixed designation E 925; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

^{ε1} ~~Note—Section 11 was added editorially in June 1994.~~

INTRODUCTION

In the application of spectrophotometric methods of analysis it is the responsibility of the analyst to verify and validate that the instrument is functioning properly and is capable of providing acceptable analytical results. It is preferable that the verification of instrument performance be accomplished through the use of ~~readily available, stable~~ reference materials whose properties have been accurately ~~measured by a standardizing laboratory. Several such certified standards determined.~~ Such materials are currently available from the National Bureau of Standards ~~readily available,~~ and their use in the tests and measurements described in this practice is satisfactory for evaluating the performance of spectrophotometers whose spectral slit width does not exceed the value for which the ~~intrinsic or certified properties are valid. A compromise maximum permissible spectral slit width of 2 nm is recommended for the reference materials and error tolerances recommended here.~~

This practice covers some of the essential instrumental parameters that should be evaluated to ensure the acceptability of the analytical data routinely obtained on the instrument. These parameters include the accuracy of the wavelength and absorbance scales and stray radiant power levels.

The accuracy of the wavelength scale in both the UV and ~~VIS~~ visible regions is determined using the sharp absorption bands of a holmium oxide glass or solution filter. ~~The wavelength of maximum absorbance of these bands has been determined.² The accuracy of the absorbance scale is determined using certified standards available through the National Bureau of Standards. The absorbance scale accuracy in the UV region (235–350 nm) is determined using acidic solutions of potassium dichromate (solid potassium dichromate is available from dichromate. In the National Bureau of Standards as SRM 935) while in the visible region (440–635 nm) the absorbance accuracy is determined using three individually certified neutral density glass filters (National Bureau of Standards SRM 930 series).³ filters.~~ The use of these reference materials provides a valid and relatively simple means to test the ~~accuracy of errors in~~ the wavelength and absorbance scales of ~~narrow band-pass small spectral slit width~~ spectrophotometers in the spectral ranges indicated. ~~A simplified version of the opaque filter method is provided as a test for excessive stray radiant energy.~~

¹ This practice is under the jurisdiction of ASTM Committee E13 on Molecular Spectroscopy and is the direct responsibility of Subcommittee E13.01 on Ultraviolet and Visible Spectroscopy.

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² McNeirney, J., and Slavin, W., *Applied Optics*

² *Annual Book of ASTM Standards*, Vol 1, 1962, p. 365. 03.06.

³ SRM 935 and 930 may be purchased

³ Available from the Office of Standard Reference Materials, National Bureau of Standards, Washington, D. C. 20234; Technical Information Service, Port Royal Road, Springfield, VA 22161.

1. Scope

1.1 This practice covers the parameters of spectrophotometric performance that are critical for testing the adequacy of

instrumentation for most routine tests and methods⁴ within the wavelength range of 200 to 700 nm and the absorbance range of 0 to 2. The recommended tests provide a measurement of the important parameters controlling results in spectrophotometric methods, but it is specifically not to be inferred that all factors in instrument performance are measured.

1.2 This practice may be used as a significant test of the performance of instrumentation. ~~While this procedure is instruments~~ for which the spectral slit width does not ~~designed to extensively evaluate~~ exceed 2 nm and for which the manufacturer's specifications for wavelength and absorbance accuracy do not exceed the performance tolerances employed here. This practice employs an illustrative tolerance of $\pm 1\%$ relative for the error of the absorbance scale over the range of 0.2 to 2.0, and of ± 1.0 nm for the error of the wavelength scale. A suggested maximum stray radiant power ratio of 4×10^{-4} yields $<1\%$ absorbance bias at an instrument, ~~it may~~ absorbance of 2. These tolerances are chosen to be compatible with many chemical applications while comfortably exceeding the uncertainty of the certified values for the reference materials and typical manufacturer's specifications for error in the wavelength and absorbance scales of the instrument under test. The user is encouraged to develop and use tolerance values more appropriate to the requirements of the end use application. This procedure is designed to verify quantitative performance on an ongoing basis and to compare one instrument's performance with that of other similar units. Refer to Practice E 275 to extensively evaluate the performance of an instrument.

1.3 This practice should be performed on a periodic basis, the frequency of which depends on the physical environment within which the instrumentation is used. Thus, units handled roughly or used under adverse conditions (exposed to dust, chemical vapors, vibrations, or combinations thereof) should be ~~calibrated~~ tested more frequently than those not exposed to such conditions. This practice should also be performed after any significant repairs are made on a unit, such as those involving the optics, detector, or radiant energy source.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

E 131 Terminology Relating to Molecular Spectroscopy²

⁴ Available from Beckman Instruments (Part No. 96157) and from distributors from Corning Glass Works (Catalogue No. 3131).

⁴ Routine tests are defined as those in which absorbance data obtained on a sample are compared to those of a standard sample preparation.

E 169 Practices for General Techniques of Ultraviolet/Visible Quantitative Analysis³

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers³

E 387 Test Method for Estimating Stray Radiant Power Ratio of Spectrophotometers by the Opaque Filter Method³

E 1866 Guide for Establishing Spectrophotometer Performance Tests³

2.2 *Other Publications:*

NBS Special Publication 260-54: Certification and Use of Acidic Potassium Dichromate Solutions As An Ultraviolet Absorbance Standard⁵

NBS Special Publication 260-102: Holmium Oxide Solution Wavelength Standard from 240 to 640 nm—SRM 2034⁴

NIST Special Publication 260-116: Glass Filters as a Standard Reference Material for Spectrophotometry—Selection, Preparation, Certification, and Use of SRM 930 and SRM 1930⁴

NIST Special Publication 260-140: Technical Specifications for Certification of Spectrophotometric NTRMs⁴

3. Terminology

3.1 *Definitions:*

3.1.1 For the definitions of terms used in this practice, refer to Terminology E 131.

3.1.2 For a description of the instrumental parameters evaluated in this practice, refer to Practice E 275.

3.1.3 For a description of quantitative ultraviolet spectrophotometric techniques, refer to Practice E 169.

4. Significance and Use

4.1 This practice permits an analyst to compare the performance of an instrument to the manufacturer's supplied performance specifications and to verify its suitability for continued routine use. It also provides generation of calibration monitoring data on a periodic basis, forming a base from which any changes in the performance of the instrument will be evident.

5. Reference to this Calibration-Monitoring Procedure

5.1 Reference to this practice in any spectrophotometric calibration-monitoring scheme shall constitute due notification that the adequacy of the spectrophotometer performance has been evaluated by means of this practice. Performance is considered to be adequate when the ~~calibration~~ data obtained are ~~in agreement with~~ within the stated tolerances from the true values.

6. Instrument Operation

6.1 In obtaining spectrophotometric calibration data the analyst must select the proper instrumental operating conditions to realize satisfactory instrument performance. Operating conditions for individual instruments are best obtained from the manufacturer's literature because of variations in instrument design.

6.2 When using ~~calibration standards, reference materials,~~ all the components of the spectrophotometer must be functioning properly. In addition, the temperature of the specimen compartment should be between ~~23° 20~~ and ~~30°C, 25°C.~~ Matched solution cells should be used for calibration purposes.

6.3 Each of the above factors in instrument operation is important in the determination of wavelength and absorbance accuracy.

7. Determination of Wavelength Accuracy in the Ultraviolet and Visible Spectral Regions

7.1 *Discussion*—The holmium oxide glass filter **(1, 2)**⁶ or solution standard (NBS Special Publication 260-102) may be used for evaluating wavelength accuracy. ~~The wavelengths (nm) monitored are:~~

279.4

287.5

360.9

418.7

453.2

536.2

637.5

~~NOTE 1—Depending on the base glass of and solution standards are both widely available commercially in the filter, a peak at 241.5 nm may also be cuvette format (a cuvette-shaped metal holder is used for the glass), as well as from NIST for high-accuracy needs. Holmium glass filters for wavelength calibration purposes.~~

~~7.1.1 If checks are available from several distributors of glass color filters and possibly from different glass-manufacturers. A purchaser should require certification by the supplier that the wavelengths of the absorption bands are within 0.2-nm of the values given in Reference (2) , and reported below. The NIST Calibration Services program provides individually verified glass wavelength standards through Service ID No. 38050C. The appropriate solution standard is found to be less accurate than 4 %~~

⁶Routine tests are defined as those

⁶The boldface numbers in which absorbance data obtained on a sample are compared parentheses refer to those the list of a standard sample preparation. references at the end of this standard.

(mass fraction) holmium oxide in 10 % (volume fraction) perchloric acid, contained in a 10-mm pathlength cuvette. Band positions for the † solution reference material were determined as locations of the transmittance minima of specified bands in the instrument manufacturer's specifications; transmittance spectrum of the material in the original certification of NIST SRM 2034. Absorbance maxima or transmittance minima must be located within ± 1 nm of the wavelengths given below:

<u>Glass Filter^A</u>	<u>Dilute Acidic Solution^B</u>
<u>241.5 nm^C</u>	<u>241.1 nm</u>
<u>...</u>	<u>249.9 nm</u>
<u>279.3 nm</u>	<u>278.1 nm</u>
<u>287.6 nm</u>	<u>287.2 nm</u>
<u>333.8 nm</u>	<u>333.4 nm</u>
<u>...</u>	<u>345.5 nm</u>
<u>360.8 nm</u>	<u>361.3 nm</u>
<u>385.8 nm</u>	<u>385.7 nm</u>
<u>418.5 nm</u>	<u>416.3 nm</u>
<u>453.4 nm</u>	<u>...</u> ^D
<u>459.9 nm</u>	<u>467.8 nm</u>
<u>...</u>	<u>485.3 nm</u>
<u>536.4 nm</u>	<u>536.6 nm</u>
<u>637.5 nm</u>	<u>640.5 nm</u>

^A Wavelengths taken from Reference (2) for Corning Glass Works Code 3130 glass, superceded by Corning Glass Works Code 3131 glass and Kopp Glass Code 3131 glass, for which the wavelengths are also valid.

^B Wavelengths rounded to 0.1 nm from the results given in NBS SP 260-102 for a 1-nm spectral slit width.

^C May not be usable, depending on the base glass of the filter.

^D Peak omitted because it resolves into a doublet at spectral slit width values less than 1 nm.

7.1.1 If the observed absorption bands of the holmium oxide glass or solution deviate by more than ±1 nm from the values stated above (see Note 2) stated, then corrective service must be performed on the instrument by a qualified service person. If the user performs this service himself, he should carefully follow service, the manufacturer's recommended procedure.

~~Note 2—The larger of these tolerance values procedure should be used as the criterion of instrumental wavelength accuracy at each wavelength checked, followed carefully.~~

7.1.2 The wavelength accuracy is dependent on the spectral bandwidth slit width and thus on the physical slit width. Spectral bandwidths slit widths may be determined from the manufacturer's specifications.

7.1.3 Computer based peak location algorithms that may be used to assign absorbance maxima or transmittance minima are discussed in section 7.6 of Guide E 1866. It should be noted that peak asymmetries in the holmium oxide reference materials are such that digital filter widths should be smaller than the full-width-half-maximum recommendation of that Guide.

7.1.4 In the absence of drift or slippage in the wavelength drive train, repeatability of the band positions should be on the order of ±0.1 nm for a given instrument, especially with the use of a computer based peak location algorithm.

7.2 Procedure:

7.2.1 Examine the holmium oxide filter reference material and remove any surface contamination using a soft brush or lens paper. Measure the temperature of the sample compartment by placing a thermometer an appropriate sensor into the cell compartment of a stabilized instrument and replacing the compartment cover securely. Place the thermometer bulb sensor as close as possible to the actual position that will be occupied by the standard. After a suitable period of time record the temperature reading, remove the thermometer, sensor, and resume normal operations.

7.2.2 Record the blank absorbance or transmittance (air versus air) readings spectrum at the desired resolution and at the appropriate wavelength intervals and scan speeds, in order to perform any necessary baseline adjustments. The wavelength intervals should be no greater than the spectral slit width used. Acquire the appropriate spectrum of the holmium oxide reference material with respect to air and baseline correct if necessary using the blank spectrum. Record the wavelengths of maximum absorbance as a function the positions of wavelength at each appropriate wavelength interval the relevant bands, and compare these values to the expected values. If large discrepancies (>1 nm) exist between the true and measured wavelengths, repeat the procedure at a slower scan speed and narrower smaller spectral slit width, if possible, to verify the nonconformity.

7.2.3 Report the wavelength calibration data in the manner of Table 1, given as an example for the holmium oxide glass reference material.

8. Evaluation of Stray Radiant Power Ratio (SRPR)

8.1 Discussion—A portion of the unwanted stray radiant power detected by the photodetector can be measured using the following sharp cut-off solution filters in 1-cm cells:

<u>Solution</u>	<u>Wavelength</u>
<u>KI, 1.0 g/L in H₂O</u>	<u>220 nm</u>
<u>K₂Cr₂O₇, 0.25 g/L in aqueous</u>	<u>370 nm</u>
<u>—0.05 N KOH</u>	
<u>0.05 M KOH</u>	

8.1.1 Reagent grade materials should be used for these solutions. They are essentially opaque at the indicated wavelengths; any observed transmittance is equivalent to the SRPR. However, it should be recognized that these solutions absorb most of the stray

TABLE 1 UV-VIS Spectrophotometers Wavelength and Stray Radiant Power Ratio Calibration

Instrument _____
 Instrument _____
 Date _____
 Date _____
 Temperature _____
 Temperature _____
 Analyst _____
 Analyst _____

Wavelength Calibration: Holmium Oxide Filter

True Wavelength (nm)-Range	Observed Wavelength (nm)	Difference (nm)	Conforms	Does Not Conform
278.4-280.4	241.5 ± 1			
286.5-288.5	279.3 ± 1			
359.9-361.9	287.6 ± 1			
417.7-419.7	333.8 ± 1			
452.2-454.2	360.8 ± 1			
535.2-537.2	385.8 ± 1			
636.5-638.5	418.5 ± 1			
	453.4 ± 1			
	459.9 ± 1			
	536.4 ± 1			
	637.5 ± 1			

Stray Radiant Power Ratio

Wavelength (nm)	Transmittance or Absorbance	Conforms	Does Not Conform
220	Transmittance or Absorbance	Conforms	Does Not Conform
370			

radiation within 40 nm of the indicated wavelengths and a significant fraction of still more distant stray radiation. Consequently the tests underestimate the true SRPR. The underestimation may be severe in instruments using relatively narrow-band blocking filters. effective SRPR.

8.1.2 An acceptable level of SRPR depends on the spectral character and absorbance level of the sample under investigation. Whenever However, an upper limit of 4×10^{-4} is consistent with a worst-case absorbance bias of ~1 % at the SRPR exceeds upper limit of the manufacturer's specification absorbance range ($0 < A \leq 2$) covered by this practice, and is suggested in the absence of other criteria.

8.1.3 While the stray radiant power ratio is equivalent to the transmittance described previously, it is often more convenient to make the measurement in the absorbance mode and mathematically convert absorbance to transmittance.

8.1.4 An excessive SRPR usually arises from dust, scratches, or corrosion on the collimator or disperser, or both. Correction of this problem Qualified personnel should be performed by qualified personnel: correct this problem. Care should be taken to discriminate between SRPR and light leaks. The latter most often originate in the sample compartment and can be detected by blocking the sample beam alternately at the ports on the source and detector sides of the sample compartment. Any difference in the detected signals indicates a light leak.

8.1.3 While the stray radiant power ratio is equivalent to the transmittance described previously, it is often preferable to make the measurement in the absorbance mode and mathematically convert absorbance to transmittance.

8.2 Procedure:

8.2.1 Use the visible light source lamp in the 370 nm region and the ultraviolet light source lamp in the 220 nm region.

8.2.2 Determine the transmittance or absorbance of each solution at the appropriate wavelength using the indicated solvents for reference.

8.2.3 Refer to Standard Test Method E 387 if the dynamic range of the readout electronics of the instrument is not adequate for the direct measurement of SRPR as described here.

8.2.4 In the manner of Table 1, report the transmittance or absorbance of these solutions. Note whether the measured effective stray radiant power ratio exceeds the manufacturer's specification, suggested tolerance of 4×10^{-4} or the user-defined tolerance.

9. Determination of the Absorbance Scale Accuracy in the Ultraviolet and Visible Spectral Regions

9.1 *Discussion*—The accuracy of the absorbance scale is determined using certified standards available through the National Bureau of Standards, reference materials with known absorbances. The absorbance scale accuracy in the ultraviolet region (235 to 350 nm) is determined using acidic solutions of potassium dichromate (as described in National Bureau of Standards SRM 935) while in (NBS) Special Publication 260-54. In the visible region (440 to 635 nm) the absorbance accuracy is determined using three essentially certified neutral density glass filters (National Bureau of Standards SRM 930 series), as described in NIST Special Publication 260-116 and NIST Special Publication 260-140. The certified absorbances should be traceable to the regular transmittance scale maintained by a national metrology institute (NMI).

9.1.1 If the measured blank-corrected absorbances ($A_{measured,corr}$) of the standards are outside the acceptable range, then corrective service must be performed on the instrument by a qualified service person. If the user performs this service himself, then he should carefully follow service, the manufacturer's recommended procedure should be followed carefully.

9.1.2 An acceptable absorbance range for each standard for any instrument must be determined based on the instrument manufacturer's specifications and on the analytical demands of the end-use application of the instrument. As a guide to the acceptability of photometric accuracy data a tolerance of $\pm 1.50\%$ relative ($0.2 \leq A \leq 2.0$) is recommended and employed in this practice. The user is based on encouraged to establish tolerance limits more appropriate to the random errors inherent application in the preparation question, and use of these standards.

9.1.3 The data obtained during the calibration tables of this practice as templates for custom tables that reflect the absorbance accuracy in both the visible and ultraviolet regions can be used to construct absorbance scale correction curves. The absorbance scale correction curve is useful in high accuracy work when an analyst needs to convert a measured absorbance reading into a corrected absorbance reading. Rigorous appropriate tolerances.

9.1.3 Rigorous treatment of the construction and use of this an absorbance correction curve for high accuracy work is beyond the scope of this practice but the generation and use of such a calibration curve is described in the NBS special publication 260-54-practice.

9.1.4 Studies by the NBS NIST have indicated that properly prepared and stored solutions of SRM 935 acidic potassium dichromate are stable for at least six months. Series of SRM 930 months when prepared in the manner described in 9.3.2.1 and stored in the dark in well-stoppered 1-L volumetric flasks. Neutral density glass filters as well as SRM 935 (stored in a desiccator over magnesium perchlorate) are stable certified by different sources for one periods of from two to se five years, with appropriately adjusted uncertainties.

9.2 *Visible Region*—The absorbance scale in the visible region is tested using glass filters, filters of a proprietary neutral glass. The wavelengths (nm) construction and certification of such filters is described in some detail in NIST Special Publications SP 260-116 and SP 260-140. Traceability of the certified absorbance values to the transmittance scale maintained by the Analytical Chemistry Division (ACD) of NIST is supported by NIST for commercial participants in the NIST Traceable Reference Materials (NTRMSM) program, or by self assertion for other commercial sources. Traceability of these filters is normally maintained through NIST SRM 930 filters with nominal absorbances of 0.5, 0.7 and 1.0 and SRM 1930 filters with absorbances of 0.3, 1.5, and 2.0. (A letter series designation for SRM 930 is periodically adjusted without significant effect for this practice.) The wavelengths for which certified absorbance values are reported for individual filters are close to local extrema in the nearly-neutral glass to minimize the effect of wavelength error on the measured transmittance:

440(2.2)440.0 nm

465(2.7)465.0 nm

590(5.4)546.1 nm

635(6.0)590.0 nm

9.2.1 Newer635.0 nm

Neutral density glass filters are also have certified absorbance values at 546.1 nm available from the National Physical Laboratory (NPL) of the UK and data at this wavelength should also be recorded. These from commercial sources asserting traceability to the regular transmittance scale maintained by NPL.

9.2.1 These filters have individually certified absorbance values and the precautionary notes stated in the certificate which that accompanies this practice the filters should be followed. In cases where recertification of the absorbance values of these filters is desired (for example, due required (due to rough handling, expiration or improper storage; or hand-aligne) they should be returned to the NBS certifying laboratory for cleaning and recalibration. recertification.

9.2.2 *Procedure:*

9.2.2.1 Examine the glass filters for surface contamination and clean with a soft brush or lens paper bulb-type of air puffer if necessary. Any other attempt to clean the filters invalidates the certification. Measure the temperature of the sample compartment as described in Section 7.

9.2.2.2 Determine the absorbance blank (air versus air) using an appropriate scan speed air absorbance value) at the indicated wavelengths. Record these measurements. If large (>0.005_{1A}) blank values are observed for recording instruments, adjust the baseline. observed, use these to blank-correct measured apparent absorbances by subtraction. Measure the apparent absorbance of each filter at each wavelength and record, versus air. Each filter should be oriented in the same manner in the sample holder. If a corrected absorbance reading is outside the acceptable absorbance range, repeat the procedure with a slower scan speed longer

integration time and narrower smaller spectral slit width, if possible, to verify the nonconformity.

9.2.3 Report the visible region calibration validation data in the manner of Table 2, constructed for a set of three filters of the nominal absorbances of NIST SRM 930.

9.3 *Ultraviolet Region*—The absorbance scale in the ultraviolet region is tested using acidic solutions of potassium dichromate (available from NIST as SRM 935a). The wavelengths (nm) of interest and maximum spectral bandwidths (nm) are:

- 235(1.2)235 nm
- 257(0.8)257 nm
- 313(0.8)313 nm
- 345(0.8)350 nm
- 350(0.8)

NOTE 1—Acidic potassium dichromate solutions specifically prepared for spectrophotometric validation are also available commercially in sealed ampoules and cuvettes. Portions of the procedure below, for the powder form, will not be required for these forms. Certified values and expiration dates that accompany such preparations should be observed.

9.3.1 The precautionary notes stated in the certificate that accompanies and the standard material safety data sheet (MSDS) for SRM 935a should be observed. These documents are available from the NIST internet site at www.nist.gov under the Standard Reference Materials Program online catalog.

9.3.2 *Procedure:*

TABLE 2 UV-Vis Spectrophotometers Absorbance Calibration—Visible Region (NBS SRM)

Instrument _____
 Instrument _____
 Date _____
 Date _____
 Temperature _____
 Temperature _____
 Analyst _____
 Analyst _____

Wavelength (nm) and Special Bandwidth (nm)	Filter No.	Calculated Certified Absorbance Range	Absorbance of Filter	Absorbance Blank	A_{nom} Measured ^A	Scan Speed	$\frac{\Delta A_{cert}}{\Delta A_{meas}}$	A_{corr} ^{A true}	Bias ^{B)}	Tolerance
EDon-formes	Does Not									
440.0		—0.5 —Not Con-1.0				—0.005 form				
440.0		0.5 0.7 1.0				0.005 form				
0.007 0.010	440.0 (2.2)									
465.0 (2.7)										
590.0 (5.4)					6.1					
54)					6.1					
635.0 (6.0)										
590.0)										
635.0		0.5 0.7 1.0				0.005 0.007 0.010				

^A A_{nom} = nominal absorbance; A_{cert} Measured = (Absorbance of Filter—Absorbance Blank). A_{corr} = measured absorbance, blank corrected as necessary.

^B $A_{corr} - A_{cert}$.

^C Tolerance taken for example as 1 % of the nominal. User to assign as appropriate for each application.

^D True = mid-value of calculated certified absorbance range.

9.3.2.1 Prepare the absorbance standard ~~s~~ solutions of potassium dichromate (~~SRM 935~~) by transferring 200.0 ± 0.3 , ~~250.0~~ 300.0 ± 0.3 , 400.0 ± 0.3 , and 500.0 ± 0.3 mg of ~~SRM 935~~ the powder to four separate 100 mL volumetric flasks and dilute to volume with distilled water (Absorbance Standard Stock Solutions). Stopper the solutions and mix well. Dilute these solutions by pipetting 20.0 mL of each solution separately to four 1-L volumetric flasks, adding 1 mL of 1M HClO₄ (8.6 mL of 70 % HClO₄/100 mL H₂O) and diluteing to volume with distilled water (Absorbance Standard Sample Calibration Solutions). These final calibration solutions contain 40, ~~50~~, ~~60~~ 60, 80, and 100 mg of ~~SRM 935~~ potassium dichromate per litre of solution, respectively. Prepare a blank solution by diluting 1 mL of 1 M HClO₄ to one L with the same distilled water. Stopper the solutions and mix well.

9.3.2.2 Clean and match the 1-cm solution cells (cuvettes). Measure the temperature of the sample compartment as described in Section 7.

9.3.2.3 Determine the apparent absorbance blank at the indicated wavelengths using solvent in each cuvette. Record these measurements. If large ($>0.01A$) blank values are ~~observed for recording instruments, adjust~~ observed, re-cleaning the baseline or re-clean the cuvettes, or both. cuvettes may be necessary. Measure the apparent absorbance of each Absorbance Standard Sample Calibration Solution of potassium dichromate in the sample cuvette at each wavelength and record. Rinse the cuvettes several times with the solutions to be measured before they are placed in the sample compartment and maintain the same orientation of ~~the a~~ a cuvette throughout the procedure. If a corrected apparent absorbance value (~~$A_{measured}$~~) A_{corr}) of an Absorbance Standard Sample Calibration is outside the acceptable range, repeat the reading ~~at with a slower scan speed longer integration time and narrower smaller spectral slit width, if possible.~~ If the absorbance readings at all wavelengths for a solution are unacceptable, prepare a fresh solution at the concentration of interest and repeat the absorbance measurements. If non-conformities are verified, corrective service must be performed by a qualified service person. If the user performs this ~~service himself, he should carefully follow service,~~ the manufacturer's recommended procedure should be followed carefully.

9.3.3 Report the ultraviolet region calibration data in the manner of Table 3

TABLE 3 UV-Vis Spectrophotometer Absorbance Calibration—Ultraviolet Region (NBS SRM 935 Potassium Dichromate)

Nominal Concentration of Calibration Solution (mg/L)										Absorbance Sample Calibration (mg/L)
Instrument _____ Instrument _____ Data _____ Date _____ Temperature _____ Temperature _____ Analyst _____ Analyst _____										
Wavelength (nm) and Spectral Bandwidth (nm)	Nominal Solution Composition (mg/L)	Calculated Certified Apparent Absorbance Range	Apparent Absorbance of Solution	Absorbance Blank	$A_{cert}^{measured A}$	Scan $SpA_{measured}^B$	ΔA ($A_{blank}^{measured} - A_{cor}^{true B}$)	Bias ^C	Tolerance ^D	
Does Not Conform										
235 (1.2)	400.485-0.499	0.607-0.625								
235.0	400.485-0.499									
	50									
	60									
	0.73									
	0-0.752									
	60									
	80									
	100-752									
	700.853-0.879									
	0.492									
	0.79									
257 (0.8)	40	0.564-0.582								
257 (41)	40	0.564-0.582								
0.991										
1.243										
	50	0.706-0.728								
	60	0.840-0.875								
	70	0.993-1.023								
		0.005								
		0.007								
		0.010								
		0.012								
313 (0.8)	40	0.189-0.195								
	50				0.237-0.245					
257.0	50				0.237-0.245					
	600.2									
	85-0.293									
	0									
	60									
	80-293									
	70	0.332-0.342								
	0.573				0.006					
100	0.862				0.009					
	1.154				0.012					
	1.449				0.014					
345 (0.8)	20	0.207-0.216								
	400.419-0.431									
313.0	400.419-0.431									
	50	0.523-0.539								
	60									
	0.627-									
	0.647									
	60									
	80									
	10-647									

10. Documentation of Data

10.1 Spectral charts and ~~Tables 1–3~~ tables should be retained for reference.

11. Keywords

11.1 absorbance; molecular spectroscopy; reference materials; spectrophotometers; UV/visible; wavelength

REFERENCES

- (1) McNeirney, J., and Slavin, W., *Applied Optics*, Vol 1, 1962, p. 365.
- (2) Keegan, H. J., Schleiter, J. C., and Weidner, V. R., *J. Opt. Soc. Am.*, Vol 51, 1961, p. 1470.

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