

UNITED STATES
DEPARTMENT OF
COMMERCE
PUBLICATION



NBS SPECIAL PUBLICATION **260-32**

Standard Reference Materials:

**STANDARD QUARTZ CUVETTES
FOR HIGH ACCURACY SPECTROPHOTOMETRY**

QC
100
157
260-32
973
e. 2

U.S.
DEPARTMENT
OF
COMMERCE
National
Bureau
of
Standards

NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards¹ was established by an act of Congress March 3, 1901. The Bureau's overall goal is to strengthen and advance the Nation's science and technology and facilitate their effective application for public benefit. To this end, the Bureau conducts research and provides: (1) a basis for the Nation's physical measurement system, (2) scientific and technological services for industry and government, (3) a technical basis for equity in trade, and (4) technical services to promote public safety. The Bureau consists of the Institute for Basic Standards, the Institute for Materials Research, the Institute for Applied Technology, the Institute for Computer Sciences and Technology, and the Office for Information Programs.

THE INSTITUTE FOR BASIC STANDARDS provides the central basis within the United States of a complete and consistent system of physical measurement; coordinates that system with measurement systems of other nations; and furnishes essential services leading to accurate and uniform physical measurements throughout the Nation's scientific community, industry, and commerce. The Institute consists of a Center for Radiation Research, an Office of Measurement Services and the following divisions:

Applied Mathematics — Electricity — Mechanics — Heat — Optical Physics — Nuclear Sciences² — Applied Radiation² — Quantum Electronics³ — Electromagnetics³ — Time and Frequency³ — Laboratory Astrophysics³ — Cryogenics³.

THE INSTITUTE FOR MATERIALS RESEARCH conducts materials research leading to improved methods of measurement, standards, and data on the properties of well-characterized materials needed by industry, commerce, educational institutions, and Government; provides advisory and research services to other Government agencies; and develops, produces, and distributes standard reference materials. The Institute consists of the Office of Standard Reference Materials and the following divisions:

Analytical Chemistry — Polymers — Metallurgy — Inorganic Materials — Reactor Radiation — Physical Chemistry.

THE INSTITUTE FOR APPLIED TECHNOLOGY provides technical services to promote the use of available technology and to facilitate technological innovation in industry and Government; cooperates with public and private organizations leading to the development of technological standards (including mandatory safety standards), codes and methods of test; and provides technical advice and services to Government agencies upon request. The Institute consists of a Center for Building Technology and the following divisions and offices:

Engineering and Product Standards — Weights and Measures — Invention and Innovation — Product Evaluation Technology — Electronic Technology — Technical Analysis — Measurement Engineering — Structures, Materials, and Life Safety⁴ — Building Environment⁴ — Technical Evaluation and Application⁴ — Fire Technology.

THE INSTITUTE FOR COMPUTER SCIENCES AND TECHNOLOGY conducts research and provides technical services designed to aid Government agencies in improving cost effectiveness in the conduct of their programs through the selection, acquisition, and effective utilization of automatic data processing equipment; and serves as the principal focus within the executive branch for the development of Federal standards for automatic data processing equipment, techniques, and computer languages. The Center consists of the following offices and divisions:

Information Processing Standards — Computer Information — Computer Services — Systems Development — Information Processing Technology.

THE OFFICE FOR INFORMATION PROGRAMS promotes optimum dissemination and accessibility of scientific information generated within NBS and other agencies of the Federal Government; promotes the development of the National Standard Reference Data System and a system of information analysis centers dealing with the broader aspects of the National Measurement System; provides appropriate services to ensure that the NBS staff has optimum accessibility to the scientific information of the world. The Office consists of the following organizational units:

Office of Standard Reference Data — Office of Technical Information and Publications — Library — Office of International Relations.

¹ Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D.C. 20234.

² Part of the Center for Radiation Research.

³ Located at Boulder, Colorado 80302.

⁴ Part of the Center for Building Technology.

Standard Reference Materials:

Standard Quartz Cuvettes For High Accuracy Spectrophotometry

R. Mavrodineanu

Analytical Chemistry Division
Institute for Materials Research

and

J. W. Lazar

Optical Physics Division
Institute for Basic Standards

National Bureau of Standards
Washington, D.C. 20234



U.S. DEPARTMENT OF COMMERCE, Frederick B. Dent, *Secretary*
NATIONAL BUREAU OF STANDARDS, Richard W. Roberts, *Director*

Issued December 1973

Library of Congress Catalog Number: 73-600200

National Bureau of Standards Special Publication 260-32

Nat. Bur. Stand. (U.S.), Spec. Publ. 260-32, 26 pages (Dec. 1973)

CODEN: XNBSAV

U.S. GOVERNMENT PRINTING OFFICE
WASHINGTON: 1973

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402
(Order by SD Catalog No. C13.10:260-32). Price 55 cents.

PREFACE

Standard Reference Materials (SRM's) as defined by the National Bureau of Standards are "well-characterized materials, produced in quantity, that calibrate a measurement system to assure compatibility of measurement in the nation." SRM's are widely used as primary standards in many diverse fields in science, industry, and technology, both within the United States and throughout the world. In many industries traceability of their quality control process to the national measurement system is carried out through the mechanism and use of SRM's. For many of the nation's scientists and technologists it is therefore of more than passing interest to know the details of the measurements made at NBS in arriving at the certified values of the SRM's produced. An NBS series of papers, of which this publication is a member, called the NBS Special Publication - 260 Series is reserved for this purpose.

This 260 Series is dedicated to the dissemination of information on all phases of the preparation, measurement, and certification of NBS-SRM's. In general, much more detail will be found in these papers than is generally allowed, or desirable, in scientific journal articles. This enables the user to assess the validity and accuracy of the measurement processes employed, to judge the statistical analysis, and to learn details of techniques and methods utilized for work entailing the greatest care and accuracy. It is also hoped that these papers will provide sufficient additional information not found on the certificate so that new applications in diverse fields not foreseen at the time the SRM was originally issued will be sought and found.

Inquiries concerning the technical content of this paper should be directed to the author(s). Other questions concerned with the availability, delivery, price, and so forth will receive prompt attention from:

Office of Standard Reference Materials
National Bureau of Standards
Washington, D.C. 20234

J. Paul Cali, Chief
Office of Standard Reference Materials

OTHER NBS PUBLICATIONS IN THIS SERIES

- NBS Spec. Publ. 260, Catalog of Standard Reference Materials, July 1970. 75 cents.* (Supersedes NBS Misc. Publ. 260, January 1968 and NBS Misc. Publ. 241, March 1962.)
- NBS Misc. Publ. 260-1, Standard Reference Materials: Preparation of NBS White Cast Iron Spectrochemical Standards, June 1964. 30 cents.*
- NBS Misc. Publ. 260-2, Standard Reference Materials: Preparation of NBS Copper-Base Spectrochemical Standards, October 1964. 35 cents.*
- NBS Misc. Publ. 260-3, Standard Reference Materials: Metallographic Characterization of an NBS Spectrometric Low-Alloy Steel Standard, October 1964. 20 cents.* (Out of print).
- NBS Misc. Publ. 260-4, Standard Reference Materials: Sources of Information on Standard Reference Materials, February 1965. 20 cents.* (Out of print).
- NBS Misc. Publ. 260-5, Standard Reference Materials: Accuracy of Solution X-Ray Spectrometric Analysis of Copper-Base Alloys, March 1965. 25 cents.* (Out of print).
- NBS Misc. Publ. 260-6, Standard Reference Materials: Methods for the Chemical Analysis of White Cast Iron Standards, July 1965. 45 cents.*
- NBS Misc. Publ. 260-7, Standard Reference Materials: Methods for the Chemical Analysis of NBS Copper-Base Spectrochemical Standards, October 1965. 60 cents.*
- NBS Misc. Publ. 260-8, Standard Reference Materials: Analysis of Uranium Concentrates at the National Bureau of Standards, December 1965. 60 cents.* (Out of print).
- NBS Misc. Publ. 260-9, Standard Reference Materials: Half Lives of Materials Used in the Preparation of Standard Reference Materials of Nineteen Radioactive Nuclides Issued by the National Bureau of Standards, November 1965. 15 cents.*
- NBS Misc. Publ. 260-10, Standard Reference Materials: Homogeneity Characterization on NBS Spectrometric Standards II: Cartridge Brass and Low-Alloy Steel, December 1965. 30 cents.*
- NBS Misc. Publ. 260-11, Standard Reference Materials: Viscosity of a Standard Lead-Silica Glass, November 1966. 25 cents.*
- NBS Misc. Publ. 260-12, Standard Reference Materials: Homogeneity Characterization of NBS Spectrometric Standards III: White Cast Iron and Stainless Steel Powder Compact, September 1966. 20 cents.*
- NBS Misc. Publ. 260-13, Standard Reference Materials: Mossbauer Spectroscopy Standard for the Chemical Shift of Iron Compounds, July 1967. 40 cents.*
- NBS Misc. Publ. 260-14, Standard Reference Materials: Determination of Oxygen in Ferrous Materials - SRM 1090, 1091, and 1092, September 1966. 30 cents.*
- NBS Misc. Publ. 260-15, Standard Reference Materials: Recommended Method of Use of Standard Light-Sensitive Paper for Calibrating Carbon Arcs Used in Testing Textiles for Colorfastness to Light, June 1967. 20 cents.*
- NBS Spec. Publ. 260-16, Standard Reference Materials: Homogeneity Characterization of NBS Spectrometric Standards IV: Preparation and Microprobe Characterization of W-20% Mo Alloy Fabricated by Powder Metallurgical Methods, January 1969. 35 cents.*
- NBS Spec. Publ. 260-17, Standard Reference Materials: Boric Acid; Isotopic and Assay Standard Reference Materials, February 1970. 65 cents.*
- NBS Spec. Publ. 260-18, Standard Reference Materials: Calibration of NBS Secondary Standard Magnetic Tape (Computer Amplitude Reference) Using the Reference Tape Amplitude Measurement "Process A", November 1969. 50 cents.*
- NBS Spec. Publ. 260-19, Standard Reference Materials: Analysis of Interlaboratory Measurements on the Vapor Pressure of Gold (Certification of Standard Reference Material 745), January 1970. 30 cents.*
- NBS Spec. Publ. 260-20, Standard Reference Materials: Preparation and Analysis of Trace Element Glass Standards. (In preparation)
- NBS Spec. Publ. 260-21, Standard Reference Materials: Analysis of Interlaboratory Measurements on the Vapor Pressures of Cadmium and Silver, January 1971. 35 cents.*
- NBS Spec. Publ. 260-22, Standard Reference Materials: Homogeneity Characterization of Fe-3Si Alloy, February 1971. 35 cents.*

- NBS Spec. Publ. 260-23, Standard Reference Materials: Viscosity of a Standard Borosilicate Glass, December 1970. 25 cents.*
- NBS Spec. Publ. 260-24, Standard Reference Materials: Comparison of Redox Standards, January 1972. \$1.*
- NBS Spec. Publ. 260-25, Standard Reference Materials: A Standard Reference Material Containing Nominally Four Percent Austenite, February 1971. 30 cents.*
- NBS Spec. Publ. 260-26, Standard Reference Materials: National Bureau of Standards—U.S. Steel Corporation Joint Program for Determining Oxygen and Nitrogen in Steel, February 1971. 50 cents.*
- NBS Spec. Publ. 260-27, Standard Reference Materials: Uranium Isotopic Standard Reference Materials, April 1971. \$1.25.*
- NBS Spec. Publ. 260-28, Standard Reference Materials: Preparation and Evaluation of SRM's 481 and 482 Gold-Silver and Gold-Copper Alloys for Microanalysis, August 1971. \$1.*
- NBS Spec. Publ. 260-29, Standard Reference Materials: Calibration of NBS Secondary Standard Magnetic Tape (Computer Amplitude Reference) Using the Reference Tape Amplitude Measurement "Process A-Model 2", June 1971. 60 cents.*
- NBS Spec. Publ. 260-30, Standard Reference Materials: Standard Samples Issued in the USSR (A Translation from the Russian), June 1971. \$1.*
- NBS Spec. Publ. 260-31, Standard Reference Materials: Thermal Conductivity of Electrolytic Iron SRM 734 from 4 to 300 K, November 1971. 35 cents.*
- NBS Spec. Publ. 260-32, Standard Reference Materials: The Cooperative Study of Temperature Scale Standards for DTA by ICTA and NBS. (In preparation)
- NBS Spec. Publ. 260-33, Standard Reference Materials: Comparison of Original and Supplemental SRM 705, Narrow Molecular Weight Distribution Polystyrene, H. L. Wagner, May 1972. 35 cents.*
- NBS Spec. Publ. 260-34, Standard Reference Materials: Thermoelectric Voltage, April 1972. 40 cents.*
- NBS Spec. Publ. 260-35, Standard Reference Materials: Thermal Conductivity of Austenitic Stainless Steel, SRM 735 from 5 to 280 K, April 1972. 35 cents.*
- NBS Spec. Publ. 260-36, Standard Reference Materials: A Reference Method for the Determination of Calcium in Serum. SRM 915, May 1972. \$1.25.*
- NBS Spec. Publ. 260-37, Standard Reference Materials: Methods of Analysis of NBS Clay Standards, June 1972. 75 cents.*
- NBS Spec. Publ. 260-38, Standard Reference Materials: Preparation and Calibration of Standards of Spectral Specular Reflectance, May 1972. 60 cents.*
- NBS Spec. Publ. 260-39, Standard Reference Materials: The Eddy Current Decay Method for Resistivity Characterization of High-Purity Metals, May 1972. 55 cents.*
- NBS Spec. Publ. 260-40, Standard Reference Materials: Selection of Thermal Analysis Temperature Standards Through a Cooperative Study (SRM 758, 759, 760), August 1972. 65 cents.*
- NBS Spec. Publ. 260-41, Standard Reference Materials: Use of Standard Light-Sensitive Paper for Calibrating Carbon Arcs used in Testing Textiles for Colorfastness to Light, August 1972. 30 cents.*
- NBS Spec. Publ. 260-42, Standard Reference Materials: The Characterization of Linear Polyethylene, SRM 1475, September 1972. 45 cents.*
- NBS Spec. Publ. 260-43, Standard Reference Materials: Preparation and Homogeneity Characterization of an Austenitic Iron-Chromium-Nickel Alloy, November 1972. 45 cents.*
- NBS Spec. Publ. 260-44, Standard Reference Materials: Preparation and Use of Superconductive Fixed Point Devices, SRM 767, December 1972. 75 cents.*
- NBS Spec. Publ. 260-45, Standard Reference Materials: Powdered Lead-Based Paint, SRM 1579. (In preparation)

*Send order with remittance to: Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402. Remittance from foreign countries should include an additional one-fourth of the purchase price for postage.

CONTENTS

		PAGE
I.	INTRODUCTION	1
II.	EXPERIMENTAL	2
III.	APPLICATIONS	6
IV.	REFERENCES	17

LIST OF TABLES

<u>Table No.</u>		PAGE
1.	Radiation pathlength expressed in mm and determined on 17 quartz cuvettes made at NBS.	5
2.	Radiation pathlength expressed in mm and determined on 10 quartz cuvettes made by a commercial manufacturer	7

LIST OF FIGURES

<u>Figure No.</u>		PAGE
1.	Fused silica material and parts used for constructing the cuvettes for high-accuracy spectrophotometry	11
2a.	Standard instrument for measuring the thickness of plates and length of quartz segments	12
2b.	Close-up of figure 2a	13
3.	General shape and nominal dimensions (mm) of NBS quartz standard cuvette for spectrophotometry.	14
4a.	General view of the standard instrument used to measure the radiation pathlength of cuvettes.	15
4b.	Close-up of figure 4a	16

STANDARD REFERENCE MATERIALS:

STANDARD QUARTZ CUVETTES FOR HIGH ACCURACY SPECTROPHOTOMETRY

R. Mavrodineanu

Analytical Chemistry Division
Institute for Materials Research
National Bureau of Standards
Washington, D. C. 20234

and

J. W. Lazar

Optical Physics Division
Institute for Basic Standards
National Bureau of Standards
Washington, D. C. 20234

Accurate knowledge of lightpath and parallelism of cuvettes used in spectrophotometry is one of the indispensable parameters which must be determined when accurate transmittance measurements of liquid materials is considered. A description is given of the design and techniques developed at NBS for the production of quartz cuvettes having a nominal radiation pathlength of $10 \text{ mm} \pm 0.03 \text{ mm}$. For each cuvette the pathlength and parallelism are certified with an uncertainty of $\pm 0.0005 \text{ mm}$. The method and instrumentation used to determine these parameters is also described in the paper.

Key words: Cuvette, spectrophotometry; lightpath; pathlength; quartz, cuvette; radiation pathlength.

I. INTRODUCTION

True transmittance values can be obtained only when using accurate measuring techniques and by taking into consideration all factors which can affect and distort the data [1]. Since transmittance is the ratio of two radiation flux intensities, it is necessary that the photometric scale of the spectrophotometer used be accurate. Other important conditions which must be satisfied are: wavelength accuracy, use of adequate spectral bandpass, use of collimated radiation, freedom from reflections (interreflection), fluorescence, polarization, light scatter, optical interferences, particulate matter. Surface conditions and the temperature at which the measurements are performed must be defined, and the material subjected to measurements must be homogeneous (freedom of strain) and stable.

When liquid samples are measured the pathlength of the radiation passing through the solution must be accurately known. This last condition is particularly important, not only when accurate transmittance measurements are contemplated, but also when the molar absorptivity of a chemical species is sought. In these cases, an uncertainty of 0.1 mm in a nominal radiation pathlength of 10 mm for instance, will result in an error of 1 percent in the absorbance measurements, and, similarly, an uncertainty of 0.01 mm will result in an error of 0.1 percent absorbance. The object of the work discussed in this paper is to describe the procedures developed and used in the construction of quartz cuvettes having a nominal radiation pathlength of $10 \text{ mm} \pm 0.03 \text{ mm}$, parallelism between the two transparent plates within 0.002 mm, plate flatness of 2 to 3 fringes (mercury green line) and parallelism of these plates within 0.002 mm over the whole length of the plates.

Additional conditions to be met were free from fluorescence and strain, and the use of fused silica of optical grade.

The fulfillment of these specifications would practically eliminate the radiation pathlength error when absorbance measurements are performed at an uncertainty level of 0.1 percent. Such an accuracy seems to be at the limit obtainable by the present state of the art when measurements of absorbance are performed on liquid samples, and include the errors of sample preparation and handling, as well as the instrument uncertainty.

II. EXPERIMENTAL

Several designs were contemplated for constructing quartz cuvettes which would satisfy the rather tight specifications mentioned above. The technique illustrated in figure 1 was finally selected, and was developed in association with E. P. Muth of NBS Optical Shop, who also made all the parts required for the cuvettes discussed in this work. The final assembly was performed by E. I. Klein from NBS Glassblowing Shop.

A rectangular-shaped fused-silica structure 48.0 mm high, 13.5 mm wide and 300 mm long was used to make the body of the cuvettes. Segments approximately 13 mm long were cut from this material and ground and polished, using conventional optical shop techniques, to produce a rectangular segment $10 \text{ mm} \pm 0.03 \text{ mm}$ long. The rectangular edges of each segment were finished to a flatness of 1 to 2 fringes (mercury green line), and at one end a small orifice was provided to permit air expansion during the assembly operation.

The length of every segment was checked for accuracy with a standard measuring instrument, illustrated in

figure 2a and b, capable of determining this length with an accuracy of $\pm 0.5 \mu\text{m}$.

Two plates made of non-fluorescent fused silica 51 mm long, 16 mm wide and 2 mm thick were ground and polished in a similar manner to a width of 12.5 mm, a thickness of 1.1 mm with a parallelism of 0.002 mm and a flatness of about 2 fringes (mercury green line) as shown in figure 1. These plates were checked for thickness with the instrument illustrated in figure 2a, b and were attached to the rectangular body by careful welding with city gas-oxygen torch using a needle shape flame.

The top of the assembled unit was then cut to produce a cuvette 43 mm high, and a rectangular fused silica block of 6 mm was attached to the open end of the cuvette by fusion. This block was provided with a standard tapered opening with a diameter of 10 mm at the top. A standard-taper Teflon stopper was used to provide tight closure of the cuvette (fig. 1). The cuvette was finished by grinding the two side walls and bevelling the edges. Its nominal dimensions are given in figure 3. The unit was stress-released by proper annealing.

The cuvette was then measured to determine the radiation pathlength and the parallelism of the unit. For this purpose the instrument illustrated in figure 4a and b was used, all measurements being performed at a temperature of $20^\circ \pm 0.05^\circ \text{C}$. The measurements were made as described below.

The cuvettes were lightly clamped in a fixture which was bolted to the table of a Moore No. 3 Measuring Machine*

*The identification of commercial instruments and products is given only to permit reproduction of the work described in this paper. In no instances does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the particular equipment or product is necessarily the best available for the purpose.

and were adjusted until the two plates which define the radiation path length were vertical within the limits of their parallelism. An electronic lever type gage, with a resolution of $0.125 \mu\text{m}$ was mounted on the spindle. To reach the bottom of the cuvette, a longer lever was fabricated and mounted on the gage. Use of the longer lever required that a correction factor be applied to all measurements which decreased the resolution of the gaging system to $0.312 \mu\text{m}$.

As shown in Figure 4b, a combination of gage blocks and end plates were wrung together so that the distance between the inside faces of the end plates was $10.0000 \pm 0.0001 \text{ mm}$. The gage blocks were then positioned beneath the center of the spindle and the gage lever was offset from the center of rotation of the spindle so that when the spindle was rotated 180° and the lever contacted each end plate the meter readout of the gaging system was at its null (zero) position.

The machine table was then moved to position the cuvette beneath the spindle. The spindle was lowered until the gage lever was at its first measuring position inside the cuvette. The table was moved until the lever contacted the face of the cuvette and the meter was at its zero position. The spindle was rotated 180° until the gage lever contacted the opposite side of the cuvette. The meter reading multiplied by the correction factor plus the nominal 10 mm path length gave the true pathlength at that point. Measurements were made approximately every 4 mm by lowering the spindle.

Seventeen quartz cuvettes were made and measured at NBS according to the procedures described in this paper, and the values found are presented in table 1.

Table 1. Radiation pathlength expressed in mm and determined on 17 quartz cuvettes made at NBS. The measurements were performed between the internal faces of the transparent plates, at 20°C.

Cuvette	1 Top	2	3	4	5	6	7	8	9	10	11	12	13 Bottom
11	9.9978	9.9981	9.9981	9.9988	9.9991	9.9997	9.9991	10.0003	10.0003	10.0003	10.0003	10.0003	10.0003
12	9.9978	9.9972	9.9975	9.9975	9.9975	9.9981	9.9984	9.9981	9.9978	9.9975	9.9978	9.9978	9.9984
13	10.0087	10.0006	9.9997	9.9994	10.0003	10.0003	10.0012	10.0018	10.0022	10.0022	10.0015	10.0018	10.0025
14	9.9988	9.9966	9.9960	9.9957	9.9954	9.9950	9.9947	9.9947	9.9947	9.9944	9.9944	9.9947	9.9954
15	9.9978	9.9978	9.9978	9.9972	9.9972	9.9966	9.9969	9.9972	9.9972	9.9969	9.9969	9.9972	9.9978
16	9.9991	9.9981	9.9974	9.9974	9.9981	9.9984	9.9991	9.9991	9.9994	9.9994	9.9997	9.9994	9.9991
17	9.9957	9.9947	9.9957	9.9966	9.9975	9.9981	9.9988	9.9991	9.9991	9.9991	9.9994	9.9994	9.9988
18	9.9966	9.9960	9.9956	9.9950	9.9944	9.9944	9.9947	9.9947	9.9947	9.9944	9.9947	9.9950	-
19	9.9956	9.9956	9.9963	9.9966	9.9972	9.9972	9.9975	9.9975	9.9972	9.9978	9.9978	9.9975	9.9972
20*	9.9857	9.9838	9.9820	9.9813	9.9810	9.9817	9.9813	9.9817	9.9817	9.9820	9.9829	9.9851	9.9891
21	9.9984	9.9984	9.9988	9.9988	9.9989	9.9991	9.9989	9.9984	9.9984	9.9969	9.9978	9.9981	9.9975
22	9.9994	9.9984	9.9978	9.9981	9.9981	9.9981	9.9978	9.9975	9.9972	9.9972	9.9975	9.9975	9.9981
23*	9.9916	9.9900	9.9894	9.9891	9.9891	9.9891	9.9882	9.9863	9.9857	9.9863	9.9854	9.9879	9.9891
24	9.9978	9.9975	9.9978	9.9978	9.9978	9.9972	9.9988	9.9991	9.9994	9.9991	9.9981	9.9991	9.9997
25	9.9988	9.9984	9.9981	9.9988	9.9991	9.9991	9.9994	10.0003	10.0003	10.0006	10.0006	10.0006	10.0003
26	9.9984	9.9988	9.9988	9.9994	9.9997	10.0003	9.9997	10.0003	10.0003	10.0006	10.0009	10.0009	10.0015
27*	9.9966	9.9960	9.9963	9.9963	9.9963	9.9966	9.9966	9.9969	9.9969	9.9975	9.9981	9.9984	9.9991

*The values obtained for these cuvettes exceed the limits specified for acceptance.

From these data it can be concluded that only three of the 17 cuvettes, or less than 18 percent of the NBS cuvettes, were below the specifications established for acceptance.

The same measurements were performed on 10 quartz cuvettes of commercial origin. The results assembled in table 2 show that only 4 of the 10 cuvettes were acceptable by the standards established in this paper, which indicate a rejection of 60 percent. It must be noted, however, that 9 of 10 cuvettes were within the specifications of the manufacturer which indicate a tolerance of nominal light-path of 10 mm \pm 0.01 mm.

III. APPLICATIONS

As a result of these measurements, it was decided to produce at NBS a number of quartz cuvettes, identical to those described in this work, and to certify them for radiation pathlength and parallelism. These cuvettes would then constitute a Standard Reference Material (SRM) in spectrophotometry, and be made available to the public as SRM 932. Each cuvette will have a certificate of calibration of which a reproduction is given here.

Table 2. Radiation pathlength expressed in mm and determined on 10 quartz cuvettes made by a commercial manufacturer. The measurements are performed between the internal faces of the transparent plates, at 20 °C.

Cuvette	1	2	3	4	5	6	7	8	9	10	Bottom
1	10.020 ₈	10.020 ₂	10.021 ₂	10.021 ₂	10.020 ₈	10.021 ₈	10.021 ₅	10.021 ₂	10.021 ₈	10.022 ₁	10.022 ₁
2	10.016 ₂	10.018 ₇	10.016 ₅	10.015 ₂	10.015 ₉	10.016 ₅	10.016 ₈	10.016 ₈	10.017 ₁	10.017 ₁	10.017 ₁
3*	10.012 ₁	10.015 ₆	10.016 ₉	10.015 ₆	10.014 ₈	10.015 ₉	10.016 ₉	10.015 ₁	10.013 ₃	10.011 ₀	10.011 ₀
4	10.002 ₄	10.003 ₄	10.004 ₂	10.003 ₇	10.003 ₂	10.003 ₉	10.004 ₂	10.004 ₂	10.003 ₉	10.002 ₉	10.002 ₉
5*	10.014 ₆	10.016 ₉	10.017 ₉	10.015 ₄	10.013 ₆	10.015 ₄	10.012 ₈	10.015 ₈	10.012 ₃	10.012 ₈	10.012 ₈
6	10.007 ₂	10.009 ₅	10.009 ₃	10.009 ₃	10.010 ₀	10.009 ₃	10.008 ₈	10.010 ₃	10.009 ₃	10.010 ₃	10.010 ₃
7*	10.015 ₁	10.015 ₉	10.013 ₁	10.013 ₆	10.016 ₁	10.016 ₄	10.017 ₇	10.017 ₄	10.021 ₂	10.016 ₄	10.016 ₄
8*	10.006 ₂	10.006 ₀	10.007 ₀	10.008 ₈	10.006 ₇	10.006 ₂	10.006 ₇	10.008 ₈	10.008 ₂	10.000 ₆	10.000 ₆
9*	10.001 ₆	10.002 ₂	10.006 ₇	10.006 ₅	10.007 ₂	10.006 ₅	10.010 ₈	10.012 ₃	10.014 ₄	10.013 ₃	10.013 ₃
10*	10.009 ₈	10.010 ₀	10.008 ₈	10.008 ₂	10.009 ₃	10.011 ₃	10.010 ₈	10.008 ₂	10.008 ₅	10.006 ₂	10.006 ₂

* The values obtained for these cuvettes exceed the limits specified for acceptance.

National Bureau of Standards

CERTIFICATE

STANDARD REFERENCE MATERIAL 932
QUARTZ CUVETTE FOR SPECTROPHOTOMETRY

R. Mavrodineanu and J. W. Lazar

This Standard Reference Material is intended as a reference source when production of accurate spectrophotometric data on liquid samples is considered. It consists of an all-quartz rectangular cuvette designed to fit the holder of conventional spectrophotometers. It is provided with two optically transparent and parallel windows defining a nominal radiation pathlength of 10 mm + .03 mm. The inner surfaces of the windows are parallel within .002 mm. The pathlength and parallelism are certified with an uncertainty of ± 0.0005 mm as determined by measurements at positions equally spaced 2 mm from the bottom to within 6 mm of the stopper top. These measurements are made at 20 °C, and the distance between each position is about 4 mm.

Position of Measurement	Radiation Pathlength, mm
Top	9.999
9	9.999
8	10.000
7	9.999
6	10.000
5	9.999
4	9.998
3	9.998
2	9.998
1	9.997
Bottom	9.997

The general shape and nominal dimensions of the cuvette are illustrated in the figure.*

The cuvette must be handled with great care and should be held only by the frosted quartz side windows. When not in use, it should be stored in the container provided for this purpose. Extended exposure to laboratory atmosphere and dusty surroundings is to be avoided.

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

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.

J. Paul Cali, Chief
Office of Standard Reference Materials

Washington, D. C. 20234

March 1, 1973

*Note: This figure is identical with figure 3 of this paper.

The cuvette was designed and produced at the National Bureau of Standards by special techniques and from non-fluorescent optical-quality fused silica. The transparent windows are attached to the body of the cuvette by direct fusion, and the unit was strain-released by proper annealing. The overall flatness of the transparent windows is within two fringes (546-nm Hg line), and their parallelism is within two micrometers. The radiation pathlength measurements were performed before and after the assembly of the cuvette using electronic feeler-gage type instruments capable of a resolution of 5 parts in 10^6 . SRM 932 is a result of the combined efforts of the Analytical Chemistry Division, Institute for Materials Research, the Optical Physics Division of the Institute for Basic Standards, and the Instrument Shops Division.

E. P. Muth and E. I. Klein designed and assembled the cuvette respectively. The radiation pathlength measurements were performed by E. G. Erber.

SRM 932 was developed to meet the needs expressed by the Standards Committee of the American Association of Clinical Chemists under the chairmanship of George N. Bowers, Jr., M. D., Hartford Hospital, Hartford, Connecticut.

This work was initiated and performed with the approval and encouragement of O. Menis, Section Chief, Analytical Chemistry Division.

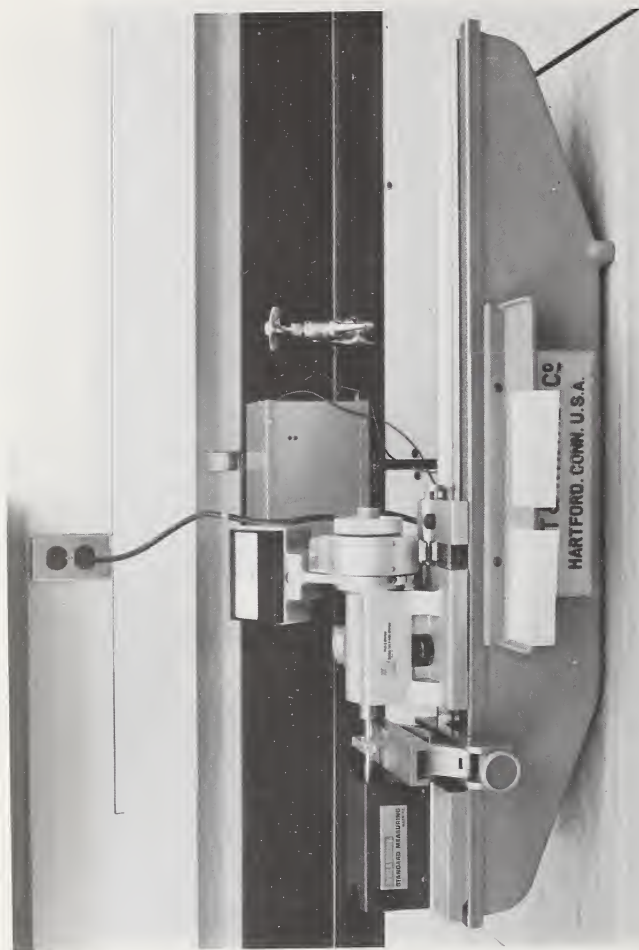


Figure 2a. Standard instrument for measuring the thickness of plates and length of quartz segments.

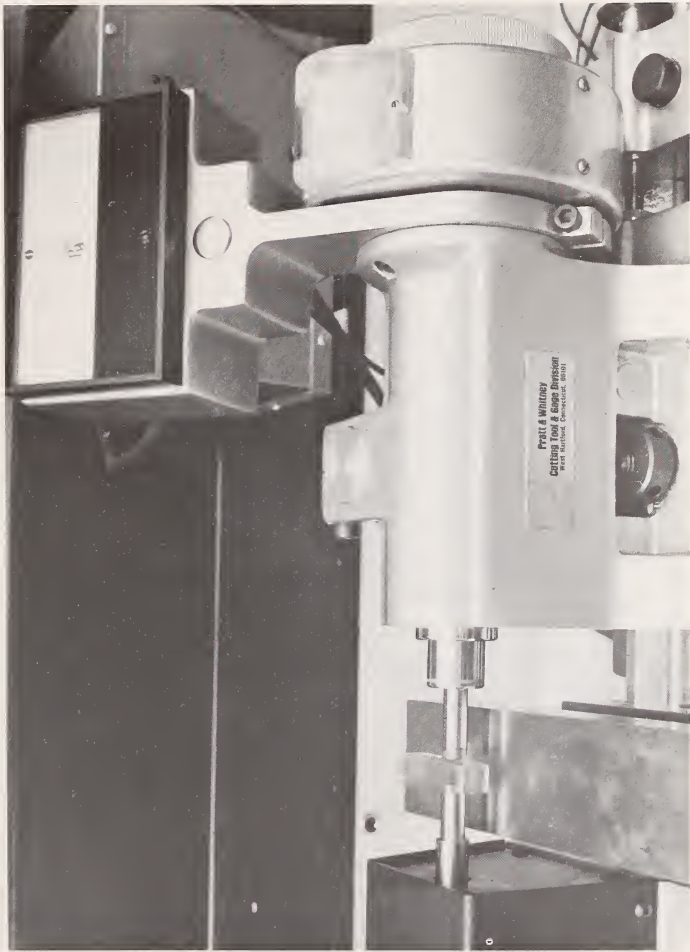


Figure 2b. Close-up of figure 2a.

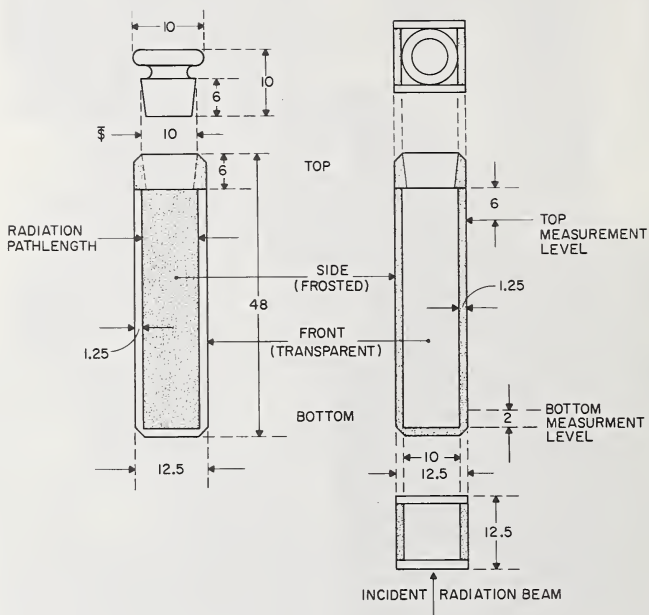


Figure 3. General shape and nominal dimensions (mm) of NBS quartz standard cuvette for spectrophotometry.

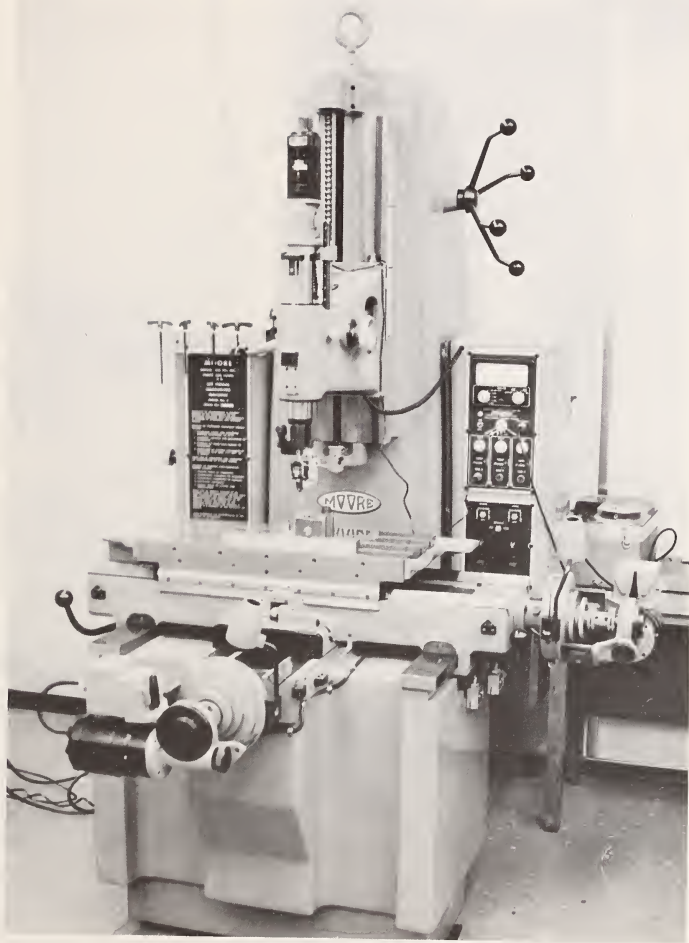


Figure 4a. General view of the standard instrument used to measure the radiation pathlength of cuvettes.

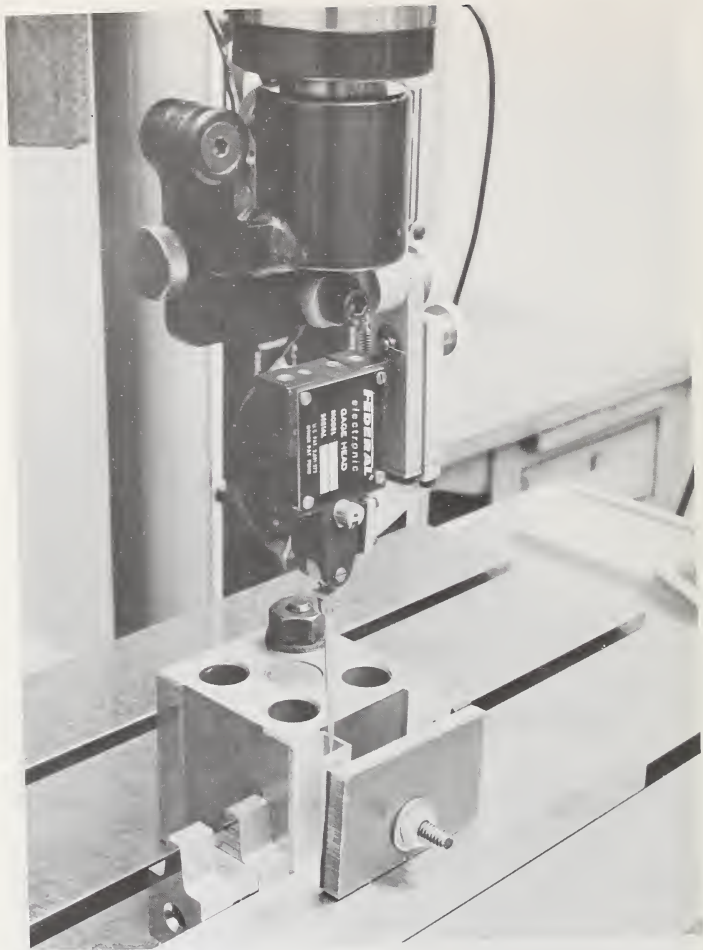


Figure 4b. Close-up of figure 4a.

IV. REFERENCES

- [1] Mavrodineanu, R., An Accurate Spectrophotometer for Measuring the Transmittance of Solid and Liquid Materials, J. Res. Nat. Bur. Stand. (U.S.), 76A, 405-425 (1972).
- Clarke, F. J. J., High-Accuracy Spectrophotometry, J. Res. Nat. Bur. Stand. (U.S.), 76A, 375-403 (1972).
- Mielenz, K. D., Physical Parameters in High-Accuracy Spectrophotometry, J. Res. Nat. Bur. Stand. (U.S.), 76A, 455-467 (1972).
- Burke, R. W., Deardorff, E. R. and Menis, O., Liquid Absorbance Standards, J. Res. Nat. Bur. Stand. (U.S.), 76A, 469-482 (1972).
- Burnett, R. W., Accurate Measurement of Molar Absorptivity, J. Res. Nat. Bur. Stand. (U.S.), 76A, 483-489 (1972).

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET		1. PUBLICATION OR REPORT NO. SP 260-32	2. Gov't Accession No.	3. Recipient's Accession No.
4. TITLE AND SUBTITLE Standard Quartz Cuvettes for High Accuracy Spectrophotometry			5. Publication Date December 1973	6. Performing Organization Code
7. AUTHOR(S) R. Mavrodineanu and J. W. Lazar			8. Performing Organization	
9. PERFORMING ORGANIZATION NAME AND ADDRESS NATIONAL BUREAU OF STANDARDS DEPARTMENT OF COMMERCE WASHINGTON, D.C. 20234			10. Project/Task/Work Unit No.	
			11. Contract/Grant No.	
12. Sponsoring Organization Name and Address Same as Block 9.			13. Type of Report & Period Covered Final	
			14. Sponsoring Agency Code	
15. SUPPLEMENTARY NOTES				
16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.) Accurate knowledge of lightpath and parallelism of cuvettes used in spectrophotometry is one of the indispensable parameters which must be determined when accurate transmittance measurements of liquid materials is considered. A description is given of the design and techniques developed at NBS for the production of quartz cuvettes having a nominal radiation pathlength of 10 mm + 0.03 mm. For each cuvette the pathlength and parallelism are certified with an uncertainty of + 0.0005 mm. The method and instrumentation used to determine these parameters is also described in the paper.				
17. KEY WORDS (Alphabetical order, separated by semicolons) Cuvette, spectrophotometry; lightpath; pathlength; quartz, cuvette; radiation pathlength.				
18. AVAILABILITY STATEMENT <input checked="" type="checkbox"/> UNLIMITED. <input type="checkbox"/> FOR OFFICIAL DISTRIBUTION. DO NOT RELEASE TO NTIS.			19. SECURITY CLASS (THIS REPORT) UNCL ASSIFIED	21. NO. OF PAGES 27
			20. SECURITY CLASS (THIS PAGE) UNCL ASSIFIED	22. Price \$.55

NBS TECHNICAL PUBLICATIONS

PERIODICALS

JOURNAL OF RESEARCH reports National Bureau of Standards research and development in physics, mathematics, and chemistry. Comprehensive scientific papers give complete details of the work, including laboratory data, experimental procedures, and theoretical and mathematical analyses. Illustrated with photographs, drawings, and charts. Includes listings of other NBS papers as issued.

Published in two sections, available separately:

• Physics and Chemistry (Section A)

Papers of interest primarily to scientists working in these fields. This section covers a broad range of physical and chemical research, with major emphasis on standards of physical measurement, fundamental constants, and properties of matter. Issued six times a year. Annual subscription: Domestic, \$17.00; Foreign, \$21.25.

• Mathematical Sciences (Section B)

Studies and compilations designed mainly for the mathematician and theoretical physicist. Topics in mathematical statistics, theory of experiment design, numerical analysis, theoretical physics and chemistry, logical design and programming of computers and computer systems. Short numerical tables. Issued quarterly. Annual subscription: Domestic, \$9.00; Foreign, \$11.25.

DIMENSIONS, NBS

The best single source of information concerning the Bureau's measurement, research, developmental, cooperative, and publication activities, this monthly publication is designed for the layman and also for the industry-oriented individual whose daily work involves intimate contact with science and technology—for engineers, chemists, physicists, research managers, product-development managers, and company executives. Annual subscription: Domestic, \$6.50; Foreign, \$8.25.

NONPERIODICALS

Applied Mathematics Series. Mathematical tables, manuals, and studies.

Building Science Series. Research results, test methods, and performance criteria of building materials, components, systems, and structures.

Handbooks. Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

Special Publications. Proceedings of NBS conferences, bibliographies, annual reports, wall charts, pamphlets, etc.

Monographs. Major contributions to the technical literature on various subjects related to the Bureau's scientific and technical activities.

National Standard Reference Data Series. NSRDS provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated.

Product Standards. Provide requirements for sizes, types, quality, and methods for testing various industrial products. These standards are developed cooperatively with interested Government and industry groups and provide the basis for common understanding of product characteristics for both buyers and sellers. Their use is voluntary.

Technical Notes. This series consists of communications and reports (covering both other-agency and NBS-sponsored work) of limited or transitory interest.

Federal Information Processing Standards Publications. This series is the official publication within the Federal Government for information on standards adopted and promulgated under the Public Law 89-306, and Bureau of the Budget Circular A-86 entitled, Standardization of Data Elements and Codes in Data Systems.

Consumer Information Series. Practical information, based on NBS research and experience, covering areas of interest to the consumer. Easily understandable language and illustrations provide useful background knowledge for shopping in today's technological marketplace.

BIBLIOGRAPHIC SUBSCRIPTION SERVICES

The following current-awareness and literature-survey bibliographies are issued periodically by the Bureau:

Cryogenic Data Center Current Awareness Service (Publications and Reports of Interest in Cryogenics).

A literature survey issued weekly. Annual subscription: Domestic, \$20.00; foreign, \$25.00.

Liquefied Natural Gas. A literature survey issued quarterly. Annual subscription: \$20.00.

Superconducting Devices and Materials. A literature survey issued quarterly. Annual subscription: \$20.00.

Send subscription orders and remittances for the preceding bibliographic services to the U.S. Department of Commerce, National Technical Information Service, Springfield, Va. 22151.

Electromagnetic Metrology Current Awareness Service (Abstracts of Selected Articles on Measurement Techniques and Standards of Electromagnetic Quantities from D-C to Millimeter-Wave Frequencies). Issued monthly. Annual subscription: \$100.00 (Special rates for multi-subscriptions). Send subscription order and remittance to the Electromagnetic Metrology Information Center, Electromagnetics Division, National Bureau of Standards, Boulder, Colo. 80302.

Order NBS publications (except Bibliographic Subscription Services) from: Superintendent of Documents, Government Printing Office, Washington, D.C. 20402.

U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
Washington, D.C. 20234

OFFICIAL BUSINESS

Penalty for Private Use, \$300

POSTAGE AND FEES PAID
U.S. DEPARTMENT OF COMMERCE
COM-215

