

UNITED STATES
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COMMERCE
PUBLICATION



NBS SPECIAL PUBLICATION **260-32**

Standard Reference Materials:

**STANDARD QUARTZ CUVETTES
FOR HIGH ACCURACY SPECTROPHOTOMETRY**

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Standard Reference Materials:

Standard Quartz Cuvettes For High Accuracy Spectrophotometry

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STANDARD REFERENCE MATERIALS:

STANDARD QUARTZ CUVETTES FOR HIGH ACCURACY SPECTROPHOTOMETRY

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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.

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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

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*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.

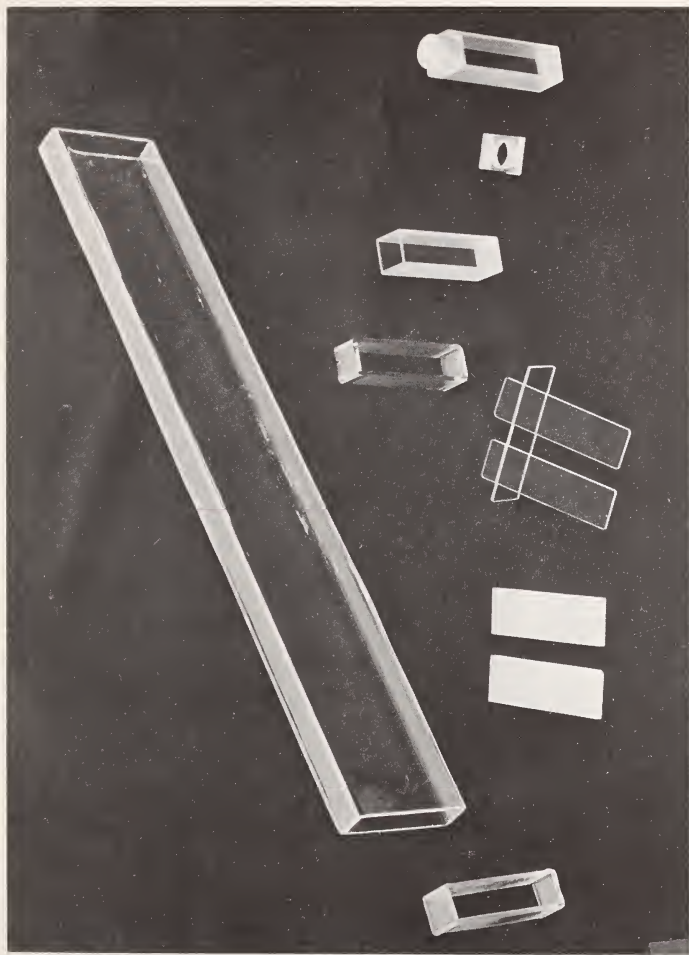


Figure 1. Fused silica material and parts used for constructing the cuvettes for high-accuracy spectrophotometry

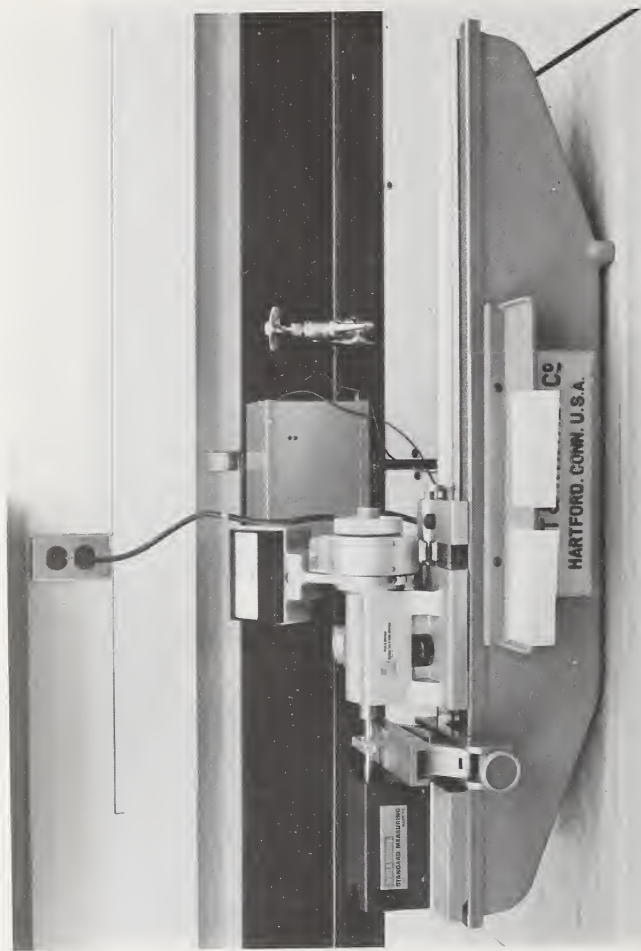


Figure 2a. Standard instrument for measuring the thickness of plates and length of quartz segments.

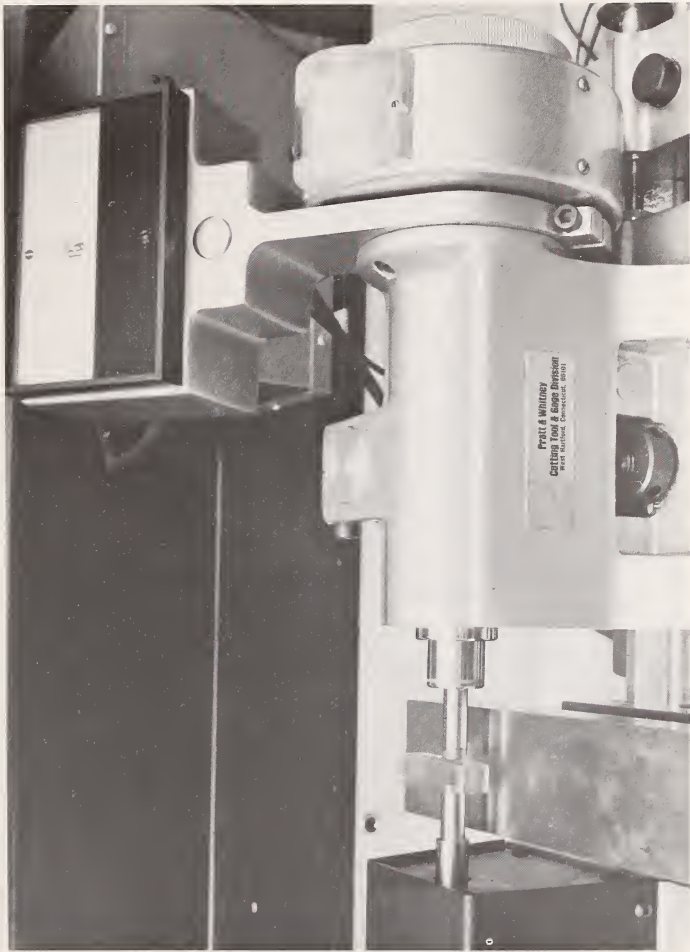


Figure 2b. Close-up of figure 2a.

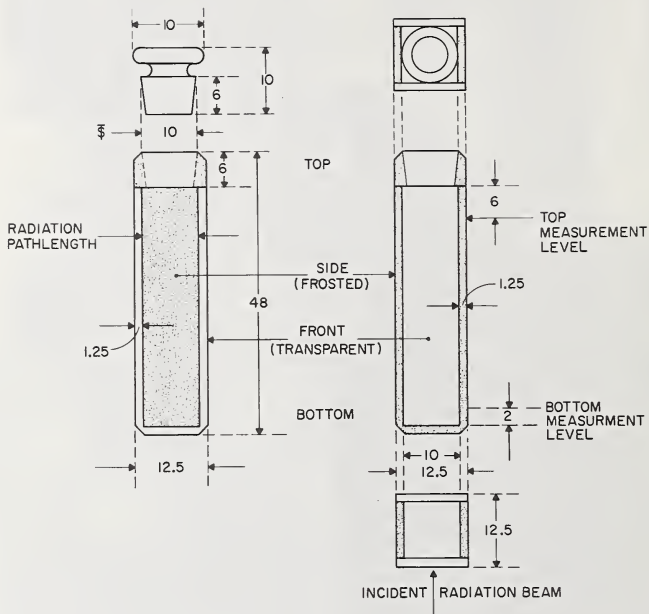


Figure 3. General shape and nominal dimensions (mm) of NBS quartz standard cuvette for spectrophotometry.

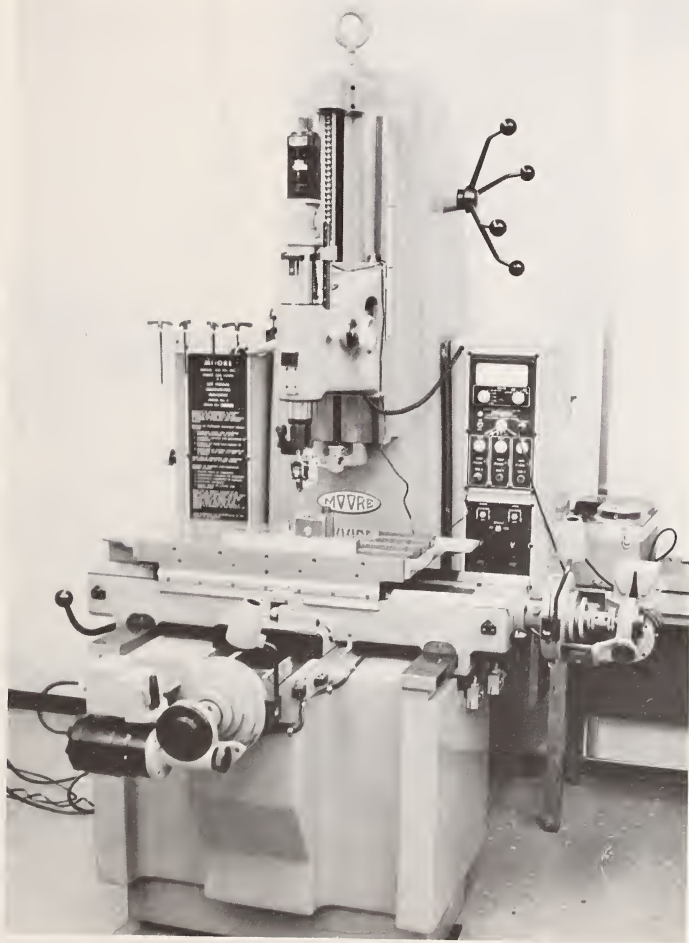


Figure 4a. General view of the standard instrument used to measure the radiation pathlength of cuvettes.

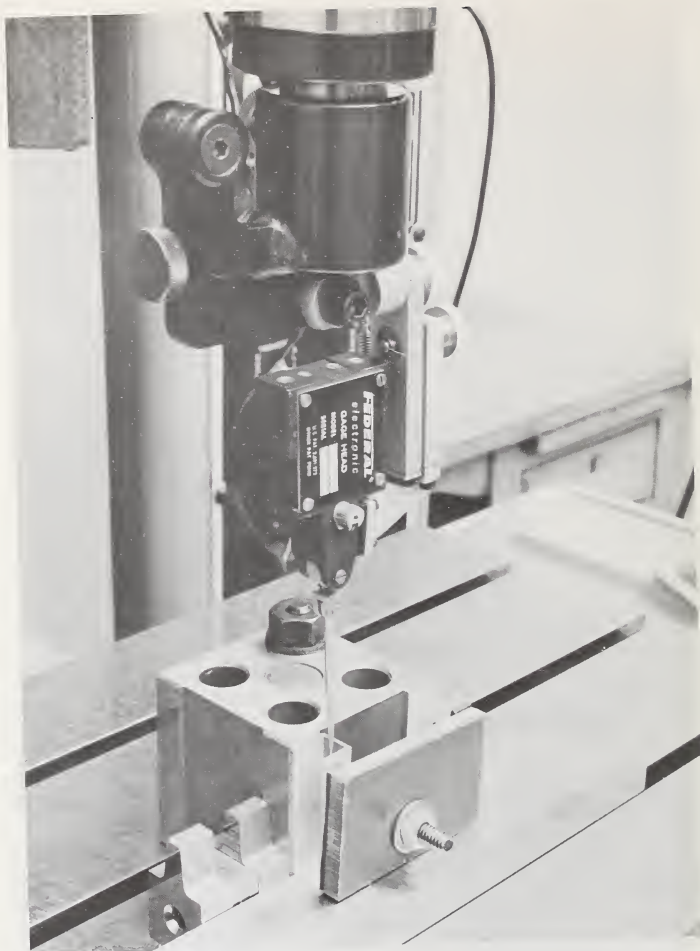


Figure 4b. Close-up of figure 4a.

IV. REFERENCES

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