

General Disclaimer

One or more of the Following Statements may affect this Document

- This document has been reproduced from the best copy furnished by the organizational source. It is being released in the interest of making available as much information as possible.
- This document may contain data, which exceeds the sheet parameters. It was furnished in this condition by the organizational source and is the best copy available.
- This document may contain tone-on-tone or color graphs, charts and/or pictures, which have been reproduced in black and white.
- This document is paginated as submitted by the original source.
- Portions of this document are not fully legible due to the historical nature of some of the material. However, it is the best reproduction available from the original submission.

Spectrophotometric Attachment for the Vacuum Ultraviolet

NORMAN N. AXELROD*
Goddard Space Flight Center, Washington, D. C.

(Received January 9, 1961)

An absorption spectrophotometric attachment to a vacuum ultraviolet monochromator has been built and tested. With an empty sample chamber, the ratio of the radiant flux through the sample chamber to the radiant flux through the reference chamber was measured. By optimizing conditions at the entrance slit, the ratio was constant within experimental error over the region 1000-1600 Å. The transmittance of thin celluloid films was measured with the attachment.

IN order to facilitate and speed the taking of absorption measurements in the vacuum ultraviolet, an attachment to a monochromator has been built and tested. The attachment makes use of the top and bottom halves of the exit beam of the monochromator. The phosphor-covered ends of two light pipes are placed in front of the top and bottom halves of the vertical exit slit, and the phosphorescence is piped to two separate photomultiplier tubes. Small samples are introduced in front of the phosphor-covered ends of the light pipes, and the signals can then be read simultaneously on two pen recorders, or the outputs of the amplifiers can be fed into a ratio recorder. Although sodium salicylate^{1,2} was used in the measurements described below, other phosphors³ can be employed and the ratio of the outputs of the two photomultipliers, with no sample in either half of the beam, would remain constant as a function of wavelength.

It can be seen from the diagram in Fig. 1 that the possibility of cross talk beyond the sample holder is eliminated by use of mechanical obstructions. The light pipes were made of Pyrex, coated with evaporated

aluminum, and sprayed with paint for durability. The photomultiplier tubes were E.M.I. 9256B, operated at 940 v. The only vacuum seals necessary were the O-ring seals on the faceplate and on the plunger, and the glyptal seals around the light pipes. The vertical position of the samples was adjusted by means of a hole-and-pin arrangement in the plunger, and the azimuth was fixed by milling flat the sample holder and having a clearance of 0.005 in. between that flat and another flat located in front of the light pipes.

The monochromator used was a McPherson 220 1-m-radius-mount normal-incidence vacuum monochromator with an aluminum-coated replica grating, 600 lines/mm. With 100- μ slits, the measured full width of a line at half maximum was 2.6 Å. The operating pressure in the main chamber was less than 10^{-4} mm. The outputs of the phototubes were recorded simultaneously on two Brown recorders.

RESULTS AND DISCUSSION

The first tests were made using a continuously operated 0.25-amp dc discharge through a water-cooled

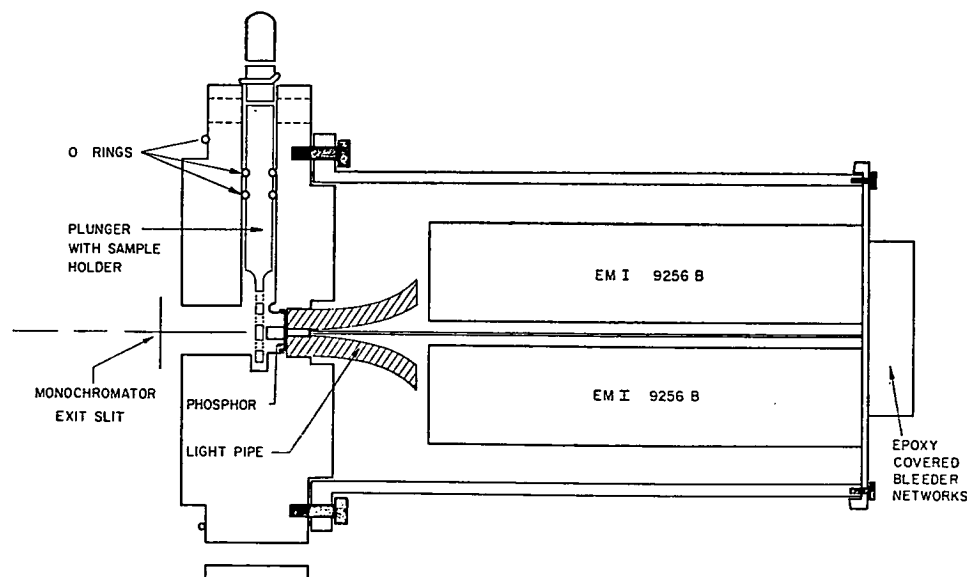


FIG. 1. Schematic illustration of the spectrophotometric attachment.

* Present address: Royal Holloway College, University of London, Englefield Green, Surrey, England.

¹ K. Watanabe and E. C. Y. Inn, *J. Opt. Soc. Am.* 43, 32 (1953).

² E. Krokowski, *Naturwissenschaften* 45, 509 (1958).

³ R. Conklin, *J. Opt. Soc. Am.* 49, 669 (1959), and references contained therein.

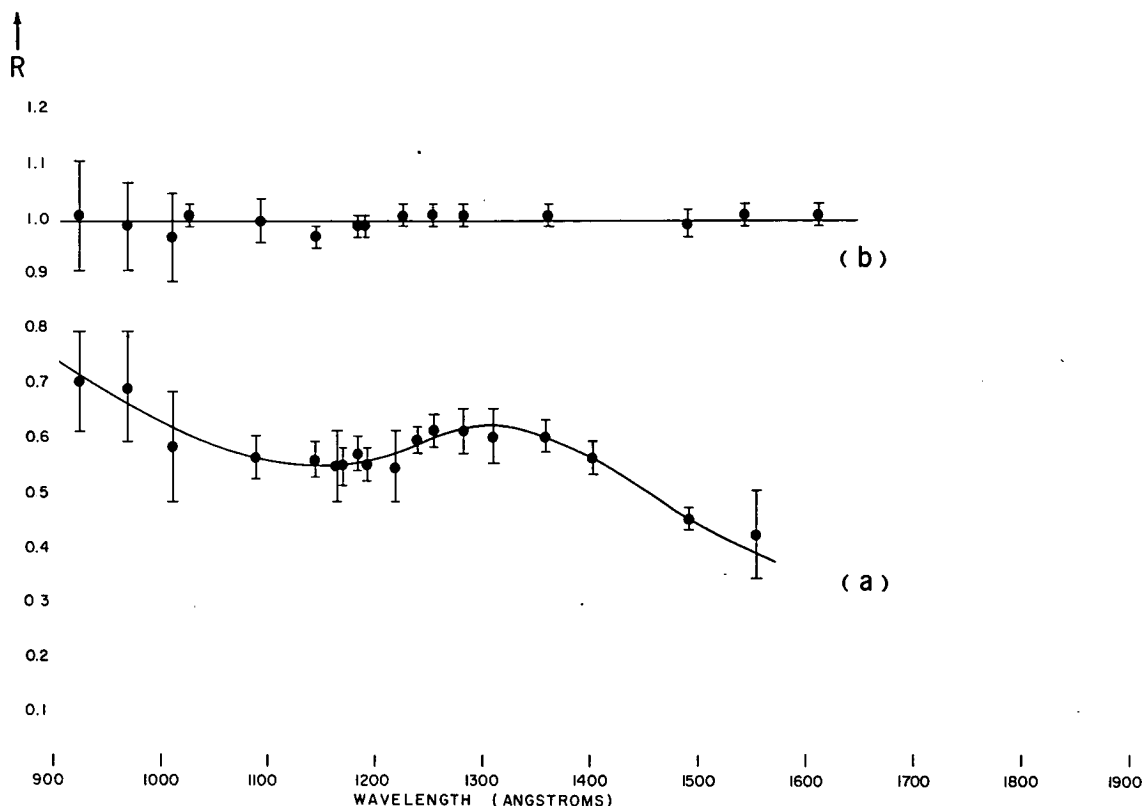


FIG. 2. The ratio R of the radiant flux through the sample chamber to the radiant flux through the reference chamber versus wavelength; (a) for the dc discharge through the capillary, (b) for the 2450 Mc discharge in 22-mm tubing.

capillary⁴ as a source of radiation. The fluxes were measured by scanning a line and using the peak value minus the minimum value. The ratio R of the signal from the bottom photomultiplier to the signal from the top photomultiplier was measured and was found to vary as a function of wavelength. In fact, R varied between 0.4 and 0.7 in a reproducible fashion, as shown in Fig. 2(a). When the attachment was rotated 180° about the center of the exit slit of the monochromator, the ratio R still varied in the same manner, and had approximately the same values. This indicated that the variation was not inherent in the attachment, but was due to different spectral distributions at the top and bottom of the slit.

In order to reduce the variation in R , a new source with a different geometry was constructed. A 125-w, 2450-Mc Raytheon diathermy unit was used to power a low-pressure discharge in a cylindrical Pyrex tube of 2.2 cm inner diameter. An aluminum cylinder, which was suspended from the Pyrex tube by means of a tungsten wire, was 1 cm in diameter, 2 cm in length, and 2½ cm from the aperture, and had the same axis as the Pyrex tube. The Pyrex tube was mounted with an O ring on a grooved aluminum plate which had a circular aperture of 9 mm diameter. The aperture was 1¼ cm from the entrance slit. The discharge appeared

to be uniform between the aperture and the aluminum cylinder. By contrast, the capillary tube was 4 mm in diameter, 9½ cm long, and its mouth was approximately 1½ cm from the slit. The exit and entrance slits were 12 mm by 100 μ .

With the new source, R was a constant, within experimental uncertainty. Measurements taken using the microwave powered source with flowing hydrogen at 400 μ are shown in Fig. 2(b). For the measurements taken using a dc discharge, as shown in Fig. 2(a), there was 350 μ of hydrogen in the discharge tube. The dependence of R on wavelength for the dc and microwave discharges did not change when the carrier gas used was nitrogen at similar pressures. With the microwave tube, the best results were obtained when aluminum foil was wrapped around the outside of the Pyrex tube, leaving an uncovered region between the aluminum plate and the mouth of the aluminum tube where the microwave power from the antenna came through the Pyrex. The foil served to confine the discharge to a region nearer the slit, and also to make the photomultiplier signal quieter.

In Figs. 3(a) and (b), the product of the linear absorption coefficient with the sample thickness of thin films of collodion as a function of wavelength is shown. The collodion films were prepared by dissolving aged collodion Merck in isoamyl acetate. The films formed by placing a drop of the solution on the surface of

⁴ F. S. Johnson, K. Watanabe, and R. Tousey, *J. Opt. Soc. Am.* 41, 702 (1951).

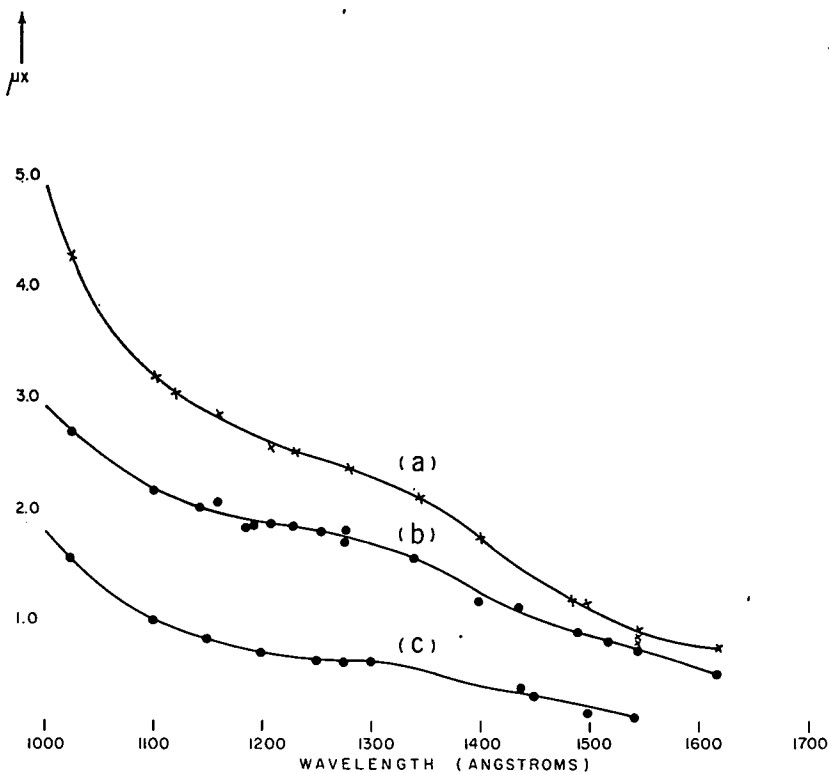


FIG. 5. The linear absorption coefficient times thickness as a function of wavelength; (a) for a collodion film 480-Å thick, (b) for a collodion film 290-Å thick, (c) for the difference between (b) and (a).

distilled water were lifted by a 300-mesh screen which had 39% transmittance between 1000 and 1600 Å and in the visible region. The transmittance of the films was measured with no correction being made for the reflectance. The films prepared here appear to be the same as those prepared by H. M. O'Bryan.⁵ Using his constants and Fresnel's equations, the reflectance at 1000 Å is found to be approximately 10%. Since the absorption coefficient is smoothly varying, the reflectance can also be expected to change in a regular manner. In Fig. 3(c) is shown the product of the linear absorption coefficient with the film thickness versus wavelength obtained by subtracting curve (b) from curve (a). It is seen to have the same shape as (a) and (b). If there were a systematic error of the same magnitude in (a) and (b), it could be shown by a simple calculation that the subtraction process would have removed the systematic error in much the same fashion as it would have removed the effect of the reflectance. Since these curves are identical, within experimental error, the systematic error caused by the spectrophotometer is seen to be negligible.

The film thicknesses were determined by extrapolating the curves in Figs. 3(a) and (b) to 1000 Å, and using the absorption coefficient determined by O'Bryan.⁵

⁵ H. M. O'Bryan, J. Opt. Soc. Am. 22, 739 (1932).

The thickness of sample (a) was found to be 480 Å, and that of sample (b) 290 Å.

EXTENSIONS AND SUGGESTIONS

For use with a ratio recorder, it is important that the image of the entrance slit be made parallel with the exit slit. This is considerably more important in the case of a source with a rapidly varying spectral distribution than in the case of a slowly varying continuum.

If it is felt necessary to reduce the dark current and noise of the photomultipliers, light pipes using fiber bundles could be used to transmit the luminescence over longer distances, and the tubes could conveniently be mounted in suitable cryostats.

A possible error that could occur with use of this attachment would be that caused by the reflection of luminescence from the sample back into the light pipe. This could easily be resolved by tilting the sample such that the reflected light went onto a black surface or into a light trap.

ACKNOWLEDGMENTS

The author is pleased to acknowledge stimulating and informative conversations with Dr. Bertram Donn and Dr. William Behring. The assistance of Alfred K. Stober with the mechanical design is also gratefully acknowledged.