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Fifth

Semi-Annual Report

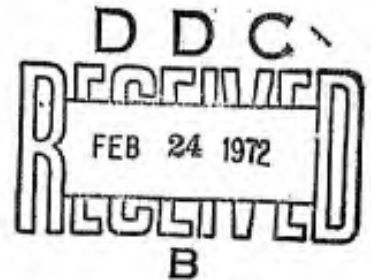
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Lawrence N. Hadley  
Professor, Department of Physics  
Principal Investigator  
Colorado State University  
Fort Collins, Colorado



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## INTRODUCTION

A problem experienced for some years in this work has been the precision measurement of the reflectance of a thin film sample. In the following discussion this quantity will be denoted by the symbol "R" in accordance with common usage. Some years ago, a Beckman DK-2 A Spectroreflectometer was purchased (with funds external to this contract) for the measurement of the reflectance R. This instrument utilizes an integrating sphere and measures either the total reflectance  $R_t$  or the diffuse reflectance  $R_d$  by reorienting the sample. The difference,  $R_t - R_d$ , should then be the desired specular reflectance  $R_s$ . The accuracy in measurement of  $R_s$  was not high, particularly for values of  $R_s$  which were in the range of a few percent. Uncertainties might run as high as 25% of the measured values.

Because of this, an absolute reflectometer has been constructed which measures the square of  $R_s$ . It is a slight modification of the type described in ref.(1), and is shown schematically in fig. (1). It is believed

*... a part because of design and alignment  
... the reflectometer is used in the research.*

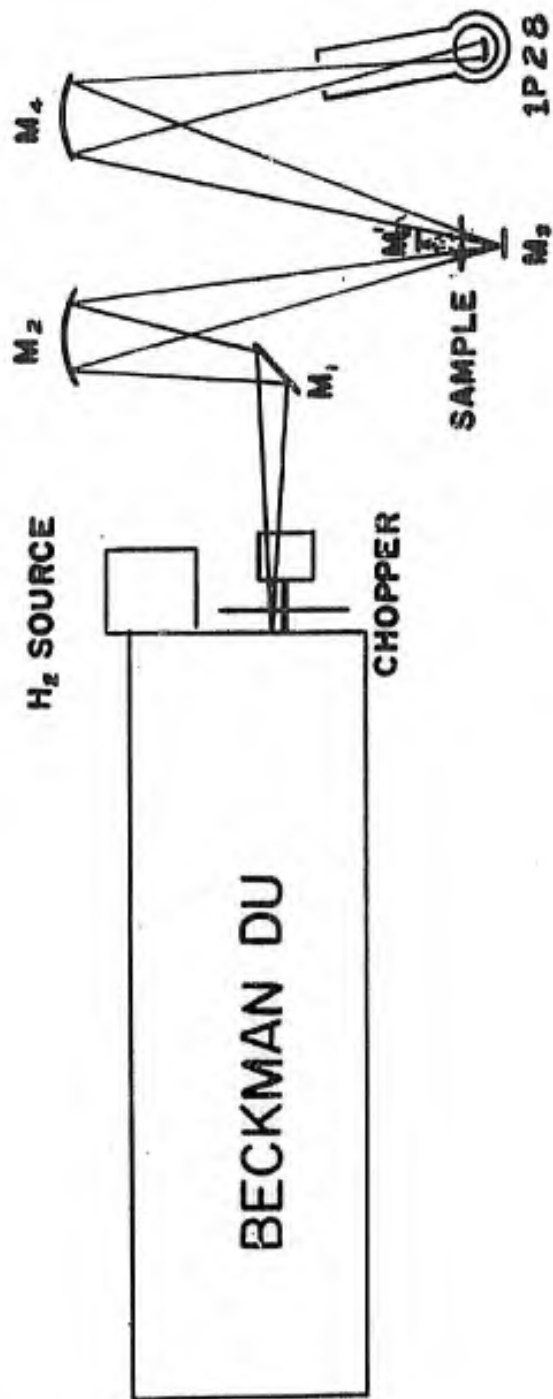


Figure 1. Optical Arrangement of Spectrometer and Reflectometer.

that the accuracy of this instrument is better than one percent. The repeatability is good to approximately 0.1%.

- (1) John Strong, *Procedures in Experimental Physics*, p. 376, Prentice-Hall, 1939.

### THE REFLECTOMETER

The monochromator is the optics of an ancient Beckman DU Spectrophotometer. The detector utilizes the photomultiplier from the previously mentioned spectroreflectometer and the associated preamplifier. The required 480 Hz signal is obtained from a chopper driven by a synchronous motor. The chopper is placed directly beyond the exit slit of the monochromator. The output of the preamplifier is read directly with a Hewlett-Packard Vacuum-Tube Voltmeter Model 400H.

It will be noted from Fig. (1) that the divergent beam from the monochromator is bent by mirror  $M_1$  (as a matter of convenience), and strikes the concave mirror  $M_2$ . The mirror  $M_2$  then focuses an image of the exit slit of the monochromator onto the mirror  $M_3$ , which then sends a divergent beam to the concave mirror  $M_4$ , which, in turn, forms a second image of the exit slit a few centimeters in front of the detector. The detector can then be either the 1-P-28 or the Pb S cell of the above mentioned system, or without using the chopper, the photocell of a Welch Densichron.

When using the 1-P-28 photocell or the Pb S cell, the HP 400 H Vacuum Tube Voltmeter is used only as an

indicating instrument. The procedure for measurement consists of adjusting the slit for full scale reading on a particular scale of the vacuum-tube voltmeter. Normally the 10-volt scale was chosen if sufficient energy was available from the monochromator. This scale reading represented a value considerably below saturation of the preamplifier, but well above the noise level. This slit adjustment was made with mirror  $M_3$  in its reference position. A General Radio Kelvin-Varley Bridge (Type 1454-A), driven by a Hewlett-Packard oscillator (Type 200-AB) was set to full output, and the gain of the oscillator (set at a frequency of 480 Hz) adjusted so that the full scale reading of the VTVM could be duplicated. The input to the Kelvin-Varley bridge was monitored with a Simpson Model 260 V-O-M, to ensure its constancy. The sample was then swung into place, and mirror  $M_3$  was moved to the position  $M'_3$ , and the new reading of the VTVM was observed, on an appropriate scale. The Kelvin-Varley bridge was then adjusted to duplicate this reading on the VTVM, and the ratio noted. This ratio is then recorded directly as the value of  $R^2$ .

As a check, the sample was then swung out of place, and  $M_3$  was returned to its original position to verify that the initial intensity remained unchanged. In each observation, when the sample was in position, the light exiting the monochromator was blocked so that a zero reading (including both stray light and noise) could be obtained. In only a few cases at the extreme short wavelength range was it necessary to apply any zero correction.

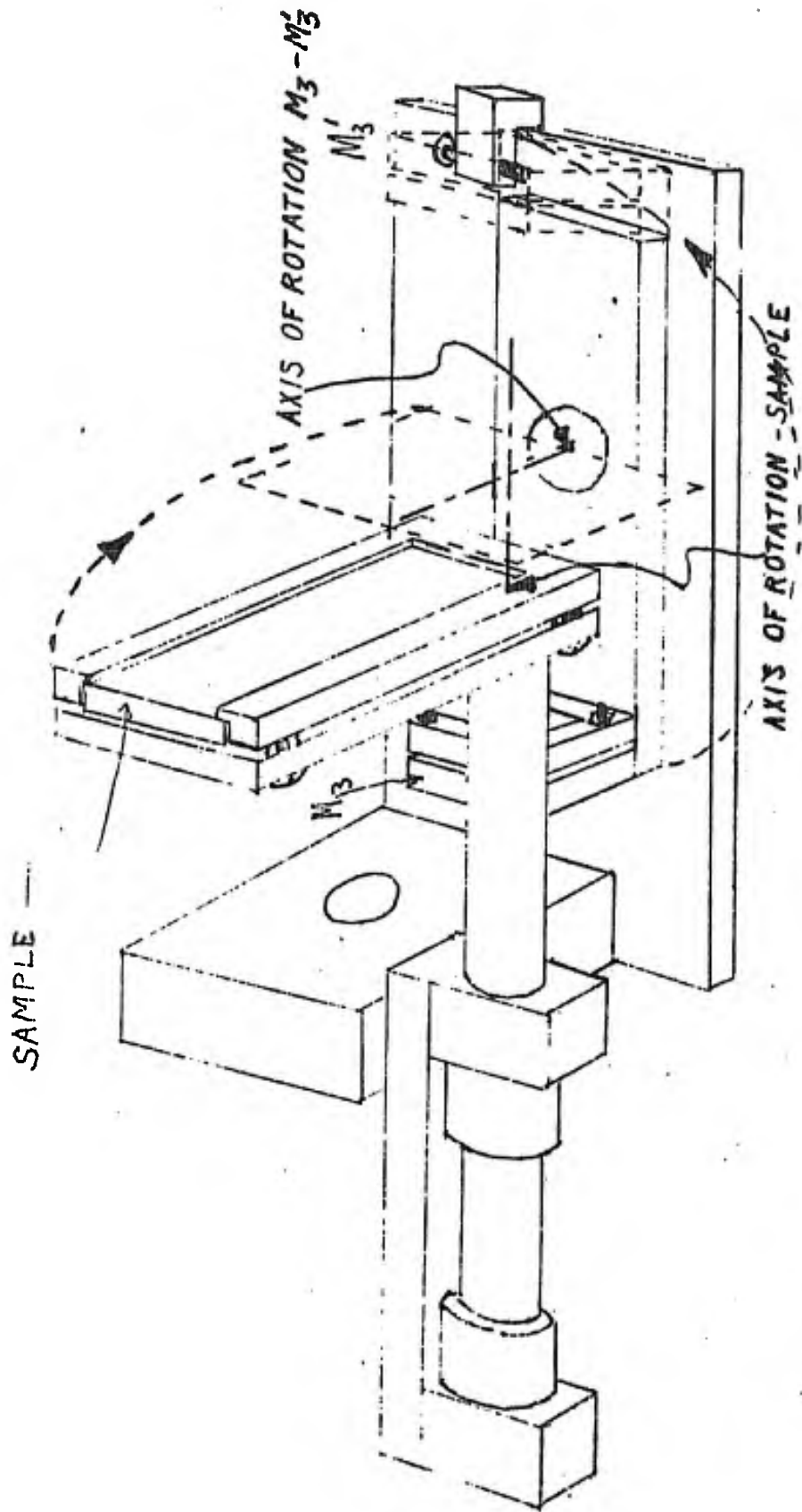


FIG. 2. DETAIL OF REFLECTOMETER HEAD

### ALIGNMENT

An alidade was converted into an autocollimator by refashioning the original eyepiece into a Gaussian eyepiece with an added light source. The alidade telescope had a magnification of approximately 15, and the sensitivity of the instrument as an autocollimator was slightly better than one minute of arc. The alignment procedure consisted of using the autocollimator to adjust mirror  $M_3$  (see figures (1) and (2) so that it was parallel to its axis of rotation, by successively placing it in the  $M_3$  and  $M_3'$  positions. Stop-screws limiting its rotation were also adjusted at the same time to ensure that the two positions  $M_3$  and  $M_3'$  were separated by  $180^\circ$  to an accuracy of less than one minute of arc. The platform and bearing which supported and controlled this rotation were found to be quite reliable in alternating between the two positions of  $M_3$ .

With the mirror  $M_3$  in the initial position, the remainder of the system was then aligned to maximize the detector reading, at the same time making sure that the beam was appropriately centered on each of the mirrors. The angle of incidence of the central

ray of the beam onto mirror  $M_3$  was adjusted so that the beam just barely, but certainly, cleared the supports for  $M_3$  when in the  $M'_3$  position with the sample in place. The angle of incidence under these circumstances was about  $13^\circ$  and the total spread of the beam was about  $3.5^\circ$ .

The sample was then also aligned by means of the autocollimator to be parallel to the positions of  $M_3$  and  $M'_3$ . This alignment was also made to a precision of better than one minute of arc. Subsequent tests showed that having made all alignment to this accuracy, a variation of the angular position of the sample of 2-3 minutes about either a horizontal or vertical axis through the sample produced a variation of less than 0.4% in the measured value of the reflectance. In actual practice during measurements, the alignment was maintained to an accuracy of approximately 0.5 minute of arc.

The mechanism for placing the sample in and out of the beam in such a manner that its introduction into the beam was repeatable in position to high accuracy is shown schematically in figure (2). The shaft supporting the sample holder was approximately

parallel to a line joining the positions  $M_3$  and  $M'_3$ , and placed above and to one side of this line. Thus a rotation of this shaft by  $90^\circ$  was sufficient to remove or insert the sample. The two bearings supporting the shaft, and the shaft itself were machined to a very slight taper in order to minimize any error in repeating sample placement.

In a similar manner, the platform and bearing supporting the rotation of mirror  $M_3$  was machined to an accuracy to support repeated positioning to an accuracy of less than one minute of arc. All of the above positioning precautions were found to be necessary as a result of experiments with previous versions of this monochromator.

It is also necessary that the sample surface be located such that the axis of rotation of  $M_3 - M'_3$  be parallel to, and pass through this surface. This is a circular nut atop the bearing for this axis of rotation. (See fig. (2)) A measuring microscope, inclined at a slight angle from the vertical, was focussed on the left edge of this nut, with the sample out of position. When the sample is then placed in the measuring position, the measuring microscope then sees a reflected image of the right side of this nut.

The sample is then translated horizontally until  
the direct and reflected images coincide.

## RESULTS

To test the reflectometer, a standard rhodium mirror supplied to us some years ago by the Night Vision Laboratory was used as a test sample. The standard mirror was cleaned by scrubbing in a detergent solution, followed by a thorough rinse in hot water, followed by an ultrasonic bath using isopropyl alcohol and finally by rinsing in a hot vapor bath of isopropyl alcohol.

The solid curve in figure (3) is plotted from the original calibration data supplied with the standard mirror. The circled points were measured using the procedures described previously, and using a quartz mercury arc as a source. The greatest deviation (.6%) occurred at  $0.365\mu$ . Data was taken at eight selected wavelengths in the range  $0.2536\mu$  to  $0.5461\mu$ .

These values and figures are given in order that the data in the ensuing report concerning the measurements of the optical constants of some Titanium Dioxide films may be better evaluated and understood.

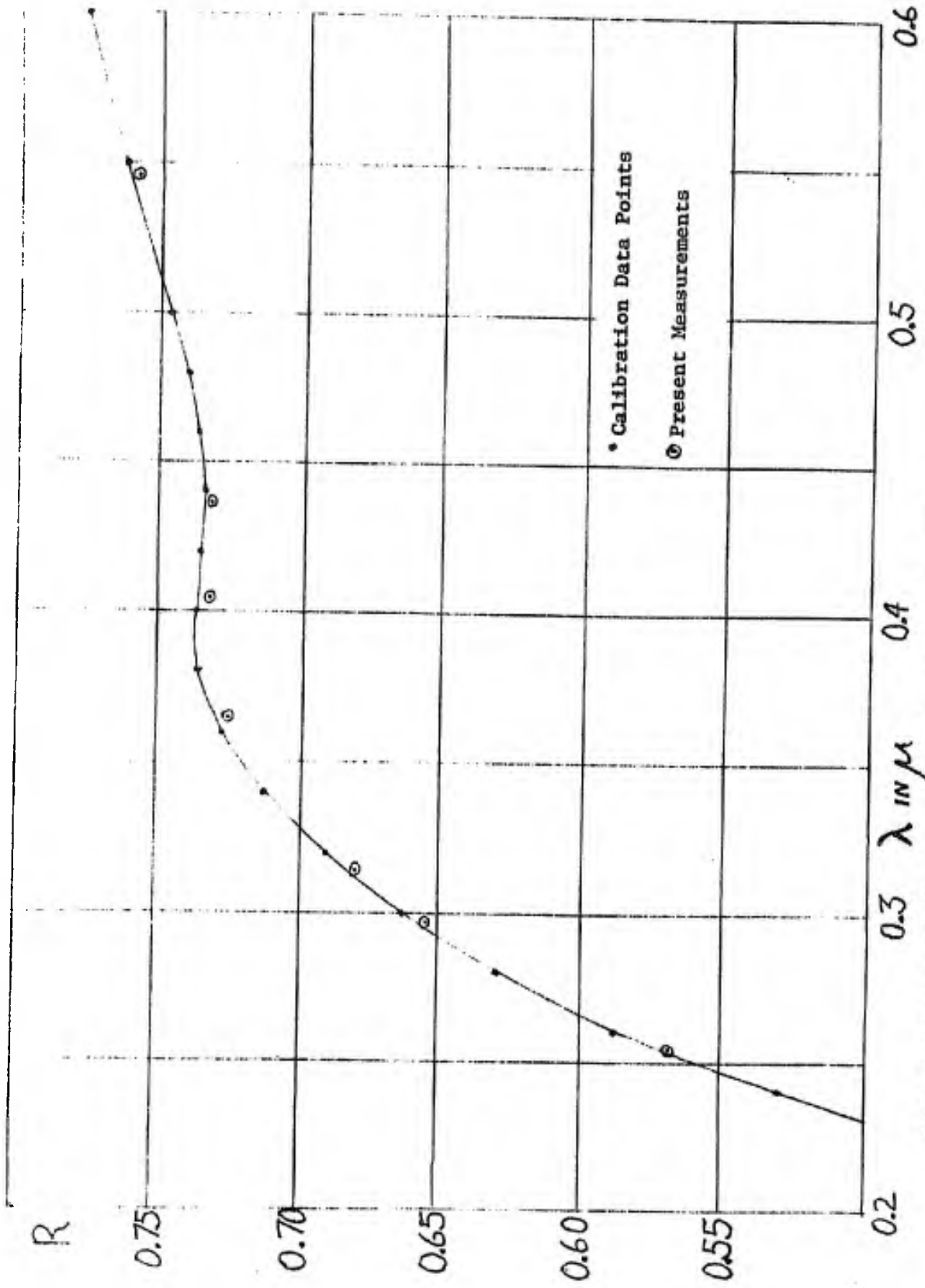


Fig. 3 Measured Reflectance of Standard Rhodium Mirror