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TRANSMISSION MEASUREMENTS THROUGH LIQUIDS IN A WHITE CELL

BY

DAVID EARL HURLEY, 1949

A THESIS

Presented to the Faculty of the Graduate School of the

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ABSTRACT

The suitability of using the White cell for determining the extinction coefficient of liquids was examined by measuring the attenuation of electromagnetic radiation, in the visible part of the spectrum, through distilled water. The analysis of the transmittance measurements through liquids in a White cell does not provide a sufficient number of independent equations to solve directly for the extinction coefficient. Reasonable estimates however can be made by employing a correction which accounts for changes in the mirror reflectance due to a liquid-mirror interface.

The reduction in the transmittance of distilled water due to an oil film was studied for fuel oils number 2 and 3 and midwestern crude oil. Reduction depends on the thickness and the extinction coefficient of the oil. It was found that a small film of crude oil, approximately six thousandths of an inch thick, can reduce the transmittance to almost zero in the range where the water's transmittance is a maximum (0.45μ - 0.50μ).

Errors resulting from using transmittance measurements to determine the extinction coefficient of liquids were examined. The analysis reveals that transmittance measurements through two cells which differ in their optical path by 1.3 will yield the highest level of accuracy.

ACKNOWLEDGEMENT

The author would like to thank everyone who helped and encouraged him through this endeavor, especially Dr. Bassem Farid Armaly for his patience, guidance, and hard work. He would like to express his gratitude to Dr. Dwight Look, Jr. for his suggestions and technical advisement and Dr. Alfred Crosbie for his professional counseling. The author would like to thank the Cloud Physics Department for the distilled water and the National Science Foundation for providing the grant that made this work possible (GK32679). He would also like to express his appreciation to Mrs. Jo Crumly for typing the manuscript.

The author dedicates this work to his mother, father, wife, and daughter.

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NOMENCLATURE

Symbol		Units
$\bar{n}_\lambda$	Spectral complex index of refraction	
n_λ	Spectral real part of the complex index of refraction	
k_λ	Spectral imaginary part of the complex index of refraction	
β_λ	Spectral extinction coefficient	1/cm
λ	Wavelength	cm
$I_{o\lambda}$	Spectral incident intensity	Watt/cm^2
$I_{1\lambda}$	Spectral transmitted intensity at position 1	Watt/cm^2
$I_{2\lambda}$	Spectral transmitted intensity at position 2	Watt/cm^2
T_λ	Spectral transmittance	
x_1	Distance from datum to position 1	cm
x_2	Distance from datum to position 2	cm
ΔI_λ	Spectral uncertainty in intensity	Watt/cm^2
Δx	Uncertainty in distance	cm
$\beta_{h\lambda}$	Spectral upper bound of extinction coefficient	1/cm
$\beta_{l\lambda}$	Spectral lower bound of extinction coefficient	1/cm
$\Delta\beta_\lambda$	Spectral uncertainty in extinction coefficient	1/cm
M	Accuracy of intensity measurement	
L	Accuracy of length measurement	
τ_λ	Spectral change in optical depth	
$I_{4a\lambda}$	Spectral transmitted intensity through air with four passes in a White cell	Watt/cm^2
$\rho_{a\lambda}$	Spectral reflectance of mirror-air interface	
$I_{8a\lambda}$	Spectral transmitted intensity through air with eight passes in a White cell	Watt/cm^2

Nomenclature (continued)

Symbol		Units
$I_{4w\lambda}$	Spectral transmitted intensity through water with four passes in a White cell	Watt/cm^2
$\rho_{w\lambda}$	Spectral reflectance of mirror-water interface	
$I_{8w\lambda}$	Spectral transmitted intensity through water with eight passes in a White cell	Watt/cm^2
$I_{12a\lambda}$	Spectral transmitted intensity through air with twelve passes in a White cell	Watt/cm^2
$I_{12w\lambda}$	Spectral transmitted intensity through water with twelve passes in a White cell	Watt/cm^2
$I_{w\lambda}$	Spectral transmitted intensity through water	Watt/cm^2
$I_{1\lambda}$	Spectral transmitted intensity through an oil film and water	Watt/cm^2
$T_{1\lambda}$	Spectral transmittance of an oil film	
ρ_{λ}	Spectral reflectance	
$n_{1\lambda}$	Spectral real part of the complex index of refraction for medium 1	
$k_{1\lambda}$	Spectral imaginary part of the complex index of refraction for medium 1	
$n_{2\lambda}$	Spectral real part of the complex index of refraction for medium 2	
$k_{2\lambda}$	Spectral imaginary part of the complex index of refraction for medium 2	
$\beta_{A\lambda}$	Spectral approximate extinction coefficient	
F.O.	Fuel oil	
C.O.	Crude oil	
t	Thickness of the oil film	

I. INTRODUCTION

Noticing man's increasing strain on the environment, people are beginning to question the effects of this contamination and are asking what damage this contamination will do and what can be done about correcting it. Air and water pollution affect every form of life on earth and a great deal of research is in process to determine the effects various pollutants have on the environment. From a solar radiation point of view, air pollution influences the rate and spectral distribution of solar energy that penetrates the atmosphere and reaches the earth's surface thus affecting the rate and type of physiological developments at the earth's surface. Similarly, water pollution can influence the surface reflection of solar energy and its attenuation to various depths below the surface. The first factor plays an important role in studies dealing with remote sensing and global energy balance. The second factor influences the type and the degree of life development that can naturally exist at various water depths, because even the smallest microorganisms at the bottom of the food chain are dependent upon sunlight as a source of energy.

Seventy-five percent (75%) of the earth's surface is covered by water in forms of oceans, lakes, rivers and reservoirs. Their use as a source of energy and food is increasing rapidly with the increase in population. The solar energy transmitted to different depths below the water surface is responsible for all plant and animal life within the water. Thus changes in the radiative properties of water due to foreign additives or pollution might influence its role in the earth's global energy balance and life requirement. For example, life could not exist below the water surface if its transmissivity to solar energy was reduced to zero.

Photosynthesis is a chemical reaction which uses solar energy to transform water and carbon dioxide to glucose (plant food) and oxygen. It is an essential step in the chain of food supply and represents a measure of physiological productivity. Energy in the visible region of the spectrum (0.4μ - 0.7μ) is the only portion that is suitable for this conversion. The rate of reaction at any water depth depends on the magnitude of energy available. The major factor affecting the transmittance of this energy to various water depths is a property known as the extinction coefficient. This property increases rapidly with an increase of foreign substance, thus reducing the depth to which energy can penetrate and reducing the productive volume of the water. The extinction coefficient of naturally existing bodies of water, which are a conglomerate of dissolved substances, plant and animal life, varies with location and depends on a multitude of other factors. Accurate values for their extinction coefficient can be obtained only from measurements taken on site so as not to disturb the suspended particles and the aquatic life. Their biological productivity can be estimated if both the incident solar energy and their extinction coefficient are known. Distilled water, on the other hand, is the purest form of that substance and its extinction coefficient can act as a lower limit for all naturally existing water. It has been used extensively in the laboratory and its optical constants have been reported over a wide range of wavelengths. It has been used as a solute for determining the influence of various pollutants on these properties, and in some cases this information was used to determine the type of pollutants existing in natural waters.

The accidental spills and the intentional dumping of oil in the ocean leaves a thin film on the water surface. It is obvious that this oil cover affects the transmission of solar energy to the ocean's depths and thus disturbs in some degree the ongoing life processes. Unfortunately, these changes in transmittance characteristics have not been investigated either experimentally or analytically. The objective of the study is to examine the suitability of the "White cell" for determining the extinction coefficient of distilled water in the visible part of the spectrum and to measure the decrease in the transmitted energy when an oil film is placed on this water surface.

II. REVIEW OF LITERATURE

The use of reflectance and transmittance measurements for determining the optical constants of liquids has been successfully demonstrated by several investigators on ocean and distilled water.[1-8] Detailed review of the literature and a summary of the results for distilled water have been made by Irvine and Pollack[9] and more recently by Hale and Querry[10]. Reflection methods have been the most popular because they allow the determination of both the real and imaginary part of the complex index of refraction. It has been established[11] however that the use of the reflection methods for determining the optical constants for water is suitable only when the imaginary part of the complex index of refraction is larger than 0.02. When this criteria is violated, transmission methods should be used. Transmission methods on the other hand are not suitable when the absorption is strong due to the difficulties of maintaining a very thin cell with parallel windows. In addition when the absorption is very weak, a long absorption path is required to accurately determine this property from the transmittance measurements. A summary of the various techniques that can be used to deduce this property is presented by Avery[12] and Ochoa[13].

One major weakness of the existing laboratory transmission measurements through water, in the visible region of the spectrum[1-4], is the relatively short length of the cell used and the calibration procedure. Sullivan[4] and Hulburt[2] are the only ones who have used more than one cell length; an essential step for accurately predicting the extinction coefficient of liquids from transmission measurements.

Also, they accounted for the differences between air-window (or mirror) interface and water-window (or mirror) interface. Unfortunately, the cells of Sullivan were relatively short and he could not determine accurately the extinction coefficient in the region where the absorption is weak, 0.40 to 0.58 microns.

Figure 1 shows the results of Sawyer[3], Clark and James[1], James and Birge[2], Hulburt[2], and Tyler, Smith and Wilson[14]. The results of Tyler, Smith and Wilson[14] are for clear natural water from Crater Lake in Oregon. Since this water is not distilled, it is understandable that their results are higher than those of the other investigators. As can be seen in Figure 1 there are considerable discrepancies in the results, especially in the region where the transmissivity is a maximum (0.45 - 0.5μ). Tyler, Smith and Wilson[14] point out that part of these discrepancies might be due to differences in scattering centers since in this region scattering can play as important a role as absorption. The other part is the fact that insufficient cell length was used to obtain these transmittance measurements. It is clear from Figure 1 and the fact that the last laboratory measurements of distilled water throughout the visible spectrum were taken over 25 years ago that a new and more rigourous experiment is needed to bring into proper prospective the extinction coefficient of distilled water.

Review of the literature indicates that the transmission characteristics of an oil layer on a water substrate have not been examined experimentally. The lack of accurate and reliable data on the optical constants of various types of oils makes it impossible even to estimate this behavior. Preliminary results on this problem will be reported in this study.

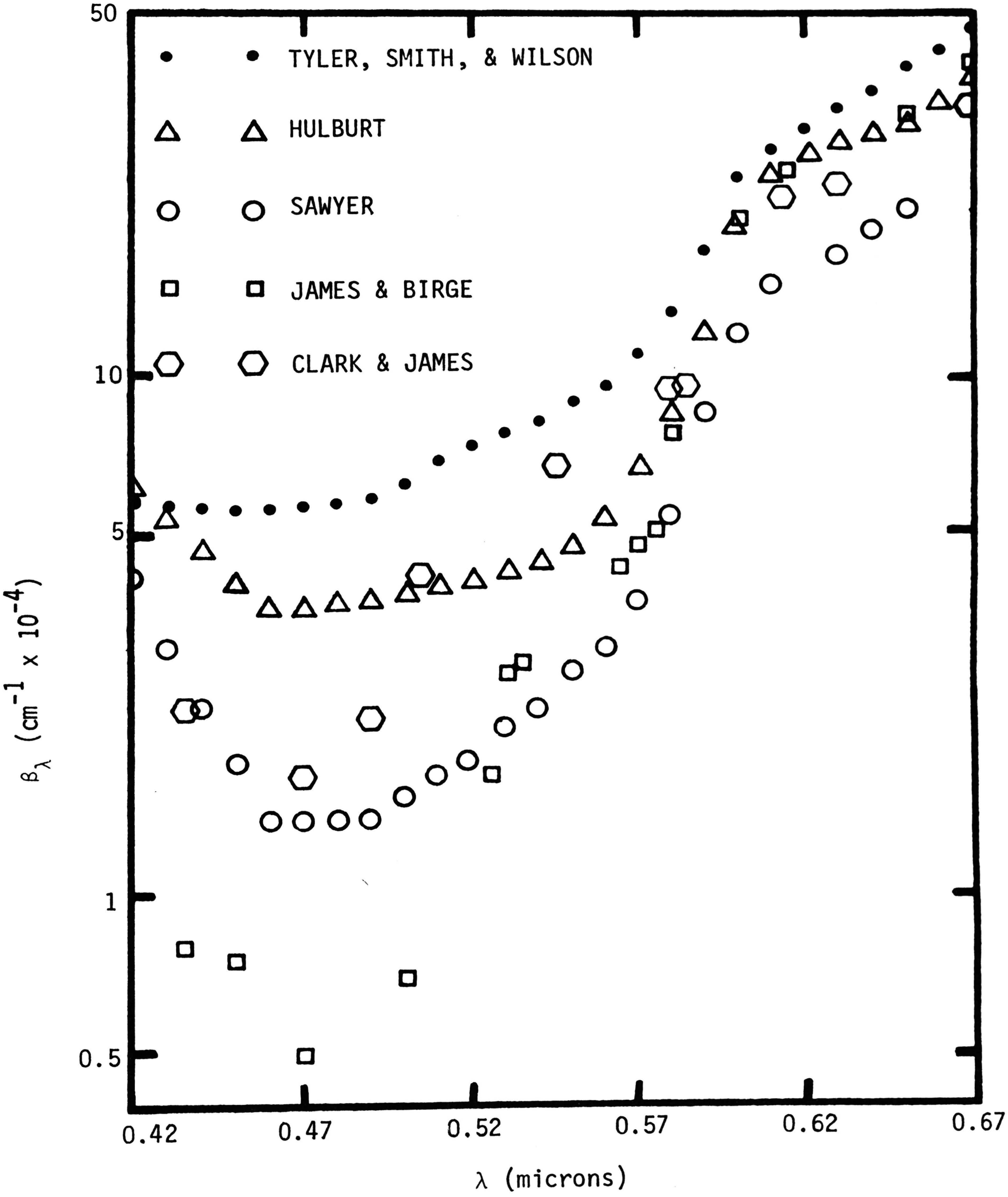


Figure 1. Extinction Coefficient of Distilled Water

III. ERRORS IN EXTINCTION COEFFICIENT WHEN DETERMINED FROM TRANSMISSION MEASUREMENTS

The complex index of refraction, $\overline{n}_\lambda$, consisting of a real part, n_λ, and an imaginary part, k_λ, is a physical property of a substance which governs the reflections of electromagnetic radiation at the interface between two substances, and the attenuation of radiation through these substances. These optical constants (n_λ, k_λ) cannot be measured directly, but rather must be deduced from other measured quantities. Small experimental errors in these measurements can sometimes be amplified through the mathematical manipulations and reductions, resulting in larger errors in the determined optical constants. The objective of this section is to examine the errors that could result in the extinction coefficient, β_λ, (related to the imaginary part of the complex index of refraction by $\beta_\lambda = 4\pi k_\lambda/\lambda$) when it is determined from the measurement of transmittance.

There are two basic methods for determining the imaginary part of the complex index of refraction; one is to use reflection measurements from the interface while the other uses the transmittance measurements through the medium. The reflection measurements method has the advantage of allowing the simultaneous deduction of both real and imaginary parts of the complex index of refraction as described in the summary of Avery[12] and Ochoa[13]. It has been pointed out by Look[11] et al. that for water, the magnitude of interface reflectances are not sensitive to changes in k_λ when $k_\lambda \leq 0.02$. This fact will prevent the accurate deduction of that property from these measurements in that range. For this reason, reflection methods are not suitable for deducing the imaginary part of the refractive index of a weakly absorbing medium and should be limited to strongly absorbing mediums such as metals, heavy oils, and water in the infrared. The transmission measurements method cannot be used to deduce the real part of the refractive index; it is limited to the deductions of the imaginary part only. It should be used with weakly absorbing medium such as gases and light colored liquids in the visible where reflection methods are not suitable. When used with a strongly absorbing medium, very small optical paths are required and errors in this length and non-parallelism in the windows will introduce large errors in the deduced value of k.

When the extinction coefficient of a liquid is to be determined by transmission methods, two sample cells of different length are normally needed. Measurements of the transmitted intensity through these cells can be expressed by Beer's Law as:

$$I_{1\lambda} = I_{o\lambda}\ T_{\lambda} e^{-\beta_{\lambda} x_1} , \tag{1}$$

and

$$I_{2\lambda} = I_{o\lambda}\ T_{\lambda} e^{-\beta_{\lambda} x_2} . \tag{2}$$

$I_{o\lambda}$ is the incident intensity, T_{λ} is the transmittance of the two windows, $I_{1\lambda}$ and $I_{2\lambda}$ are the measured transmitted intensities through sample cell of length x_1 and sample cell of length x_2, respectively, and β_{λ} is the extinction coefficient. The extinction coefficient is then deduced from these measurements by taking their ratio resulting in the following relation:

$$\beta_{\lambda} = \ln\ (I_{1\lambda}/I_{2\lambda})/(x_2 - x_1) . \tag{3}$$

In reality one can only measure the intensity and cell lengths to some degree of accuracy which is imposed by the experimental apparatus and the limitations of the instruments. In addition, small fluctuations in the light source will introduce uncertainties in the measured intensities. These errors will produce uncertainties in the determined extinction coefficient. The success of the experiment will be in knowing the magnitude of these errors and their effect on the accuracy of the determined extinction coefficient.

To determine how these uncertainties affect the extinction coefficient, perturbations in the measured intensities and lengths are introduced as follows:

$$I_{1\lambda}' = I_{1\lambda} \pm \Delta I_{\lambda} = I_{o\lambda}\, T_{\lambda}\, e^{-\beta_{\lambda} x_1'} , \tag{4}$$

$$I_{2\lambda}' = I_{2\lambda} \pm \Delta I_{\lambda} = I_{o\lambda}\, T_{\lambda}\, e^{-\beta_{\lambda} x_2'} \tag{5}$$

where

$$x_1' = x_1 \pm \Delta x \tag{6}$$

$$x_2' = x_2 \pm \Delta x \tag{7}$$

The ΔI_{λ} and Δx are the uncertainties in the intensity and length respectively. An upper bound, $\beta_{h\lambda}$, and a lower bound, $\beta_{l\lambda}$, can be determined for the extinction coefficient due to these uncertainties resulting in:

$$\beta_{h\lambda} = \frac{\ln\left(\dfrac{I_{1\lambda} + \Delta I_{\lambda}}{I_{2\lambda} - \Delta I_{\lambda}}\right)}{(x_2 - x_1) - 2\Delta x} \tag{8}$$

$$\beta_{1\lambda} = \frac{\ln\left(\dfrac{I_{1\lambda} - \Delta I_\lambda}{I_{2\lambda} + \Delta I_\lambda}\right)}{(x_2 - x_1) + 2\Delta x} \tag{9}$$

The uncertainty in the extinction coefficient can then be deduced by combining equations (8) and (9) through the following definition:

$$\frac{\Delta\beta_\lambda}{\beta_\lambda} = \frac{\beta_{h\lambda} - \beta_{1\lambda}}{\beta_\lambda} = \frac{\ln\left(\dfrac{I_{1\lambda} + \Delta I_\lambda}{I_{2\lambda} - \Delta I_\lambda}\right)}{\beta_\lambda\left\{(x_2 - x_1) - 2\Delta x\right\}} - \frac{\ln\left(\dfrac{I_{1\lambda} - \Delta I_\lambda}{I_{2\lambda} + \Delta I_\lambda}\right)}{\beta_\lambda\left\{(x_2 - x_1) + 2\Delta x\right\}} \tag{10}$$

For convenience define

$$M = \frac{\Delta I_\lambda}{I_{1\lambda}} \tag{11}$$

$$L = \frac{2\Delta x}{(x_2 - x_1)} \tag{12}$$

$$\tau_\lambda = \beta_\lambda\ (x_2 - x_1) \tag{13}$$

where M and L are the relative errors in intensity and length, respectively and τ_λ is the change in optical depth. Also using equations (3) and (10), the error in the extinction coefficient can be expressed by

$$\frac{\Delta\beta_\lambda}{\beta_\lambda} = \frac{\ln\left(\dfrac{1 + M}{e^{-\tau_\lambda} - M}\right)}{\tau_\lambda\ (1 - L)} - \frac{\ln\left(\dfrac{1 - M}{e^{-\tau_\lambda} + M}\right)}{\tau_\lambda\ (1 + L)} \tag{14}$$

The above error in the extinction coefficient becomes a function of three variables: M, L, and τ_λ. The fixing of any two will permit their evaluation as a function of the third.

Several values for the relative error in intensity and length measurements were selected and substituted into equation (14) to evaluate the relative error introduced into the extinction coefficient as a function of optical depth, τ_λ. The results are shown in Figure 2 for a $\pm$ 0.1% error in length measurement, L, and $\pm$ 0.1, $\pm$ 0.5, $\pm$ 1.0, $\pm$ 5.0, and $\pm$ 10% error in the intensity measurement, M. Three important conclusions can be drawn from Figure 2. Number one, the relative error in the extinction coefficient increases with increasing relative error in the intensity measurements. This is also true for an increase in error in the length measurement although the results shown are only for one value of L. Number two, the minimum error in the extinction coefficient occurs at essentially the same value of τ, approximately 1.3 for the given range of errors in intensity or length measurements. Number three, in the neighborhood of $\tau = 1.3$ (the optimum change in optical depth) the slope of the curves is small and so deviations from that optimum change create only a small increase in the error of the determined extinction coefficient.

The above results are also good when one uses a single cell with length equivalent to $(x_2 - x_1)$. One cell is all that is required when the medium to be tested has an index of refraction similar to air, such as gases. For liquids one must use two cells to correct for the window-medium interface. Based on this analysis the first cell should be as small as possible, thus absorbing only a little of the incident intensity. The second cell should be made long enough so that the difference in length places the change in optical depth close to the optimum value, $\tau = 1.3$. Since one is usually interested in a wavelength range and not one particular wavelength, one should design the cells in such a manner that in the wavelength range under

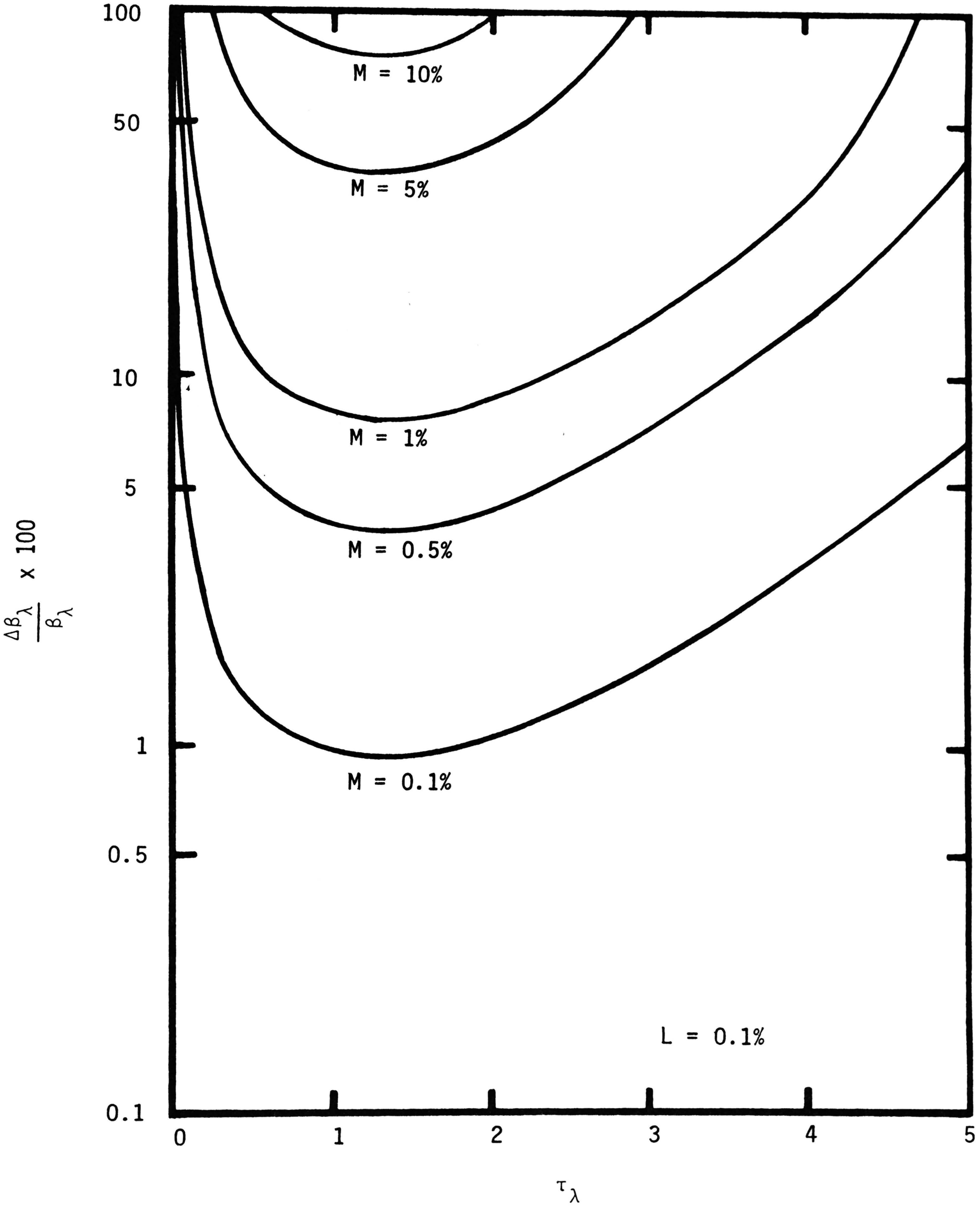


Figure 2. Percentage Error in Extinction Coefficient Versus Change in Optical Depth (L = 0.1%)

consideration the changes in optical depth should lie between 0.5 and 2.5. This will permit a change in extinction coefficient equivalent to five times the magnitude of the smallest value. This, of course, assumes that one knows the approximate values of the extinction coefficient before starting the measurements, which is a rarity. Under these conditions it would be wise to estimate these values by using any available cell and taking crude measurements to determine the range of magnitudes for the extinction coefficient over the wavelength interval of interest before the final cell design is made.

Figure 3 represents the simple plot of the extinction coefficient, β_λ, versus the changes in length, $(x_2 - x_1)$, for three fixed values of τ_λ as governed by $\tau_\lambda = \beta_\lambda(x_2 - x_1)$. For any given value of extinction coefficient, one can determine directly the changes in length, $(x_2 - x_1)$, that will produce the least error in the extinction coefficient by using the line for $\tau_\lambda = 1.3$. Assume $\beta_\lambda = 10$ cm^{-1}, then going to Figure 3 one finds $(x_2 - x_1)$ should be 0.13 cm. In addition, for a fixed error level of 0.1% and 1% in length and intensity measurements, respectively, the region of 10% error in extinction coefficient is presented by the bounding curves, $\tau_\lambda = 0.56$ and $\tau_\lambda = 2.35$. Therefore, if one has chosen $(x_2 - x_1)$ to be 0.13 cm, then β_λ can range from 4.3 cm^{-1} to 18.1 cm^{-1} and still be accurate to within 10% error. Also, the minimum error at $\tau_\lambda = 1.3$ for this particular case is 7.4% (Fig. 2 M = 1%). It is clear from Figure 2 that large changes in optical depth around the optimum value is possible without increasing

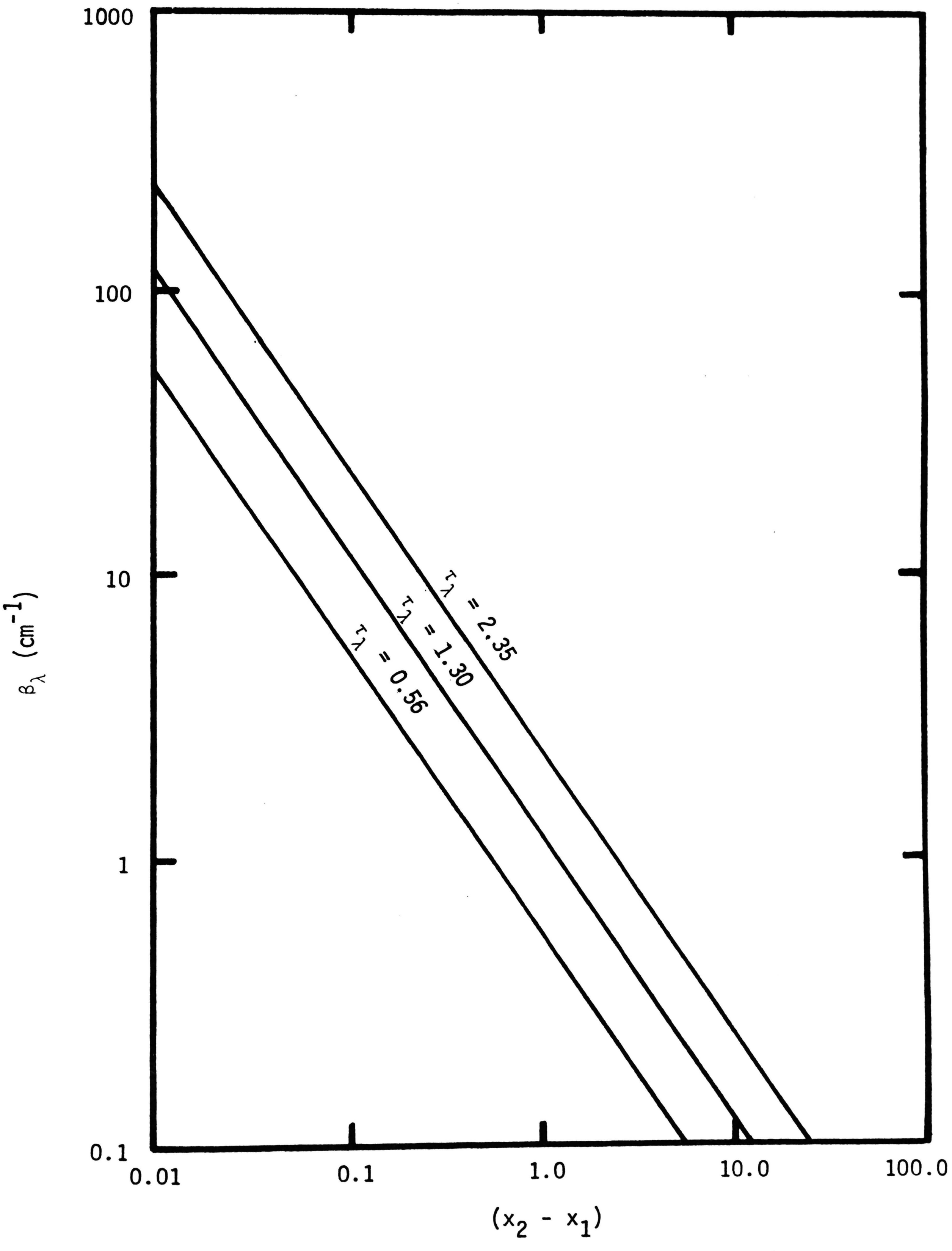


Figure 3. Extinction Coefficient Versus $(x_2 - x_1)$, Region of Ten Percent Error for M = 1% and L = 0.1%

drastically the error level. Thus for this case, a range of extinction coefficients that differ by a factor of 4.2 (2.35/0.56) can be determined to an accuracy level of less than 10% by using only two cells. If, in the wavelength range under consideration, the magnitudes of the extinction coefficients vary by a factor larger than 4.2 and error levels less than 10% are desired, then more than two cells will be required.

Using the above criterion, results reported by previous investigators were analyzed to determine the magnitude of errors that could exist in their final results. It is assumed for this evaluation that uncertainties in length and intensity measurements are 0.1% which is extremely good. Sullivan[4] reported experimental measurements of extinction coefficient for water in the wavelength range between $\lambda = 0.40\mu$ and $\lambda = 0.79\mu$. The differences in length between the two cells used was 30 cm resulting in a change in optical depth of 0.099 to 0.616. Based on our analysis, his results at large τ should be accurate to 1.1% and at small τ should be accurate to 4.4%. Hulburt[2] reported measurements of extinction coefficients for water in the wavelength range between 0.4μ and 0.7μ. The difference in his cell lengths was 340 cm making the range for τ to be between 0.122 to 1.96. The error level that could be predicted for his results ranges from 0.92% to 3.7% at the lowest value of extinction coefficient. If, however, their intensity measurements were accurate to within 1%, then Sullivan's[4] errors would range from 9.4% to 42.3% and Hulburt's[2] would range from 7.4% to 35.6%. These large errors for the lowest value of the extinction coefficient can explain why there is a lack of agreement at these low values. No one has used a sufficiently long path to reduce the large uncertainties that exist in the extinction coefficient for water in the range between $\lambda = 0.4$ to $\lambda = 0.5\mu$.

IV. TRANSMITTANCE MEASUREMENTS

Transmittance measurements through liquids have been used successfully by several investigators[1-5] to determine the extinction coefficient. This method offers an advantage of higher accuracy over the reflection methods when the liquid is weakly absorbing as discussed earlier in section II. Under these conditions a very long optical path is required to determine accurately this property. All the reported transmittance measurements through liquids have been made using a one pass cell which makes the apparatus bulky and the measurements difficult. An attempt is made here to use a multiple pass cell, which has been used frequently with gases[15], in an effort to make the apparatus compact and the measurements feasible.

Early attempts to obtain long optical path cell in small volume were made by both Smith and Marshall[16] and Katz and Mack.[17] Smith and Marshall used a system consisting of two plane mirrors at one end and one spherical mirror at the other end. Light entering the system is reflected first by the spherical mirror toward one of the plane mirrors which redirects it back to the spherical mirror via the adjacent plane mirror. This path can be repeated several times to achieve the desired length. Katz and Mack achieved the same effect by replacing the two plane mirrors by a totally reflecting prism. White[18] proposed a system which can perform the previous task by using three spherical mirrors having the same radius of curvature. This system was named after the inventor and is known as the "White cell." Two of the spherical mirrors are located on one end of the cell and are placed equidistant from the optical axis of the cell as defined by the center point of the third mirror which is located one radius of curvature away from the other two mirrors. This system is capable of producing a multiple of four paths by adjusting the inclination angle of the two adjacent mirrors. The length of each path is equal to the mirror's radius of curvature. The suitability of the White cell for determining the extinction coefficient of liquids will be examined by performing measurements on distilled water. Details of the apparatus and the results are reported below.

A. APPARATUS

The apparatus consisted of a low resolution spectrometer, detector, source, White cell and appropriate mirrors to complete the optical path. It is shown schematically in Figure 4 and pictorially in Figure 5. The spectrometer was a Perkin Elmer model 112 and was mounted on an adjustable base which allows smooth and accurate positioning of the entrance slit on the focused image of the source. A 1P21 photomultiplier was used as a detector with Perkin Elmer amplifier model 107. The output was read on a Hickok model 3301 digital multimeter. The source was a 54 Watt tungsten ribbon filament instrumentation lamp mounted in a light baffled enclosure. A series of mirrors were used to collect radiant energy from the source, pass it through the White cell and then refocus the transmitted beam on the slit of the spectrometer as seen in Figure 4. The slit width was maintained at 0.03 mm. Corresponding to a wavelength resolution smaller than 0.01 microns in the experimental wavelength range.

The White cell which was used in this experiment was designed and partially constructed by Ochoa[13] and is shown schematically in Figure 4. The cell forms a rectangular box 10 x 20 x 45 inches high.

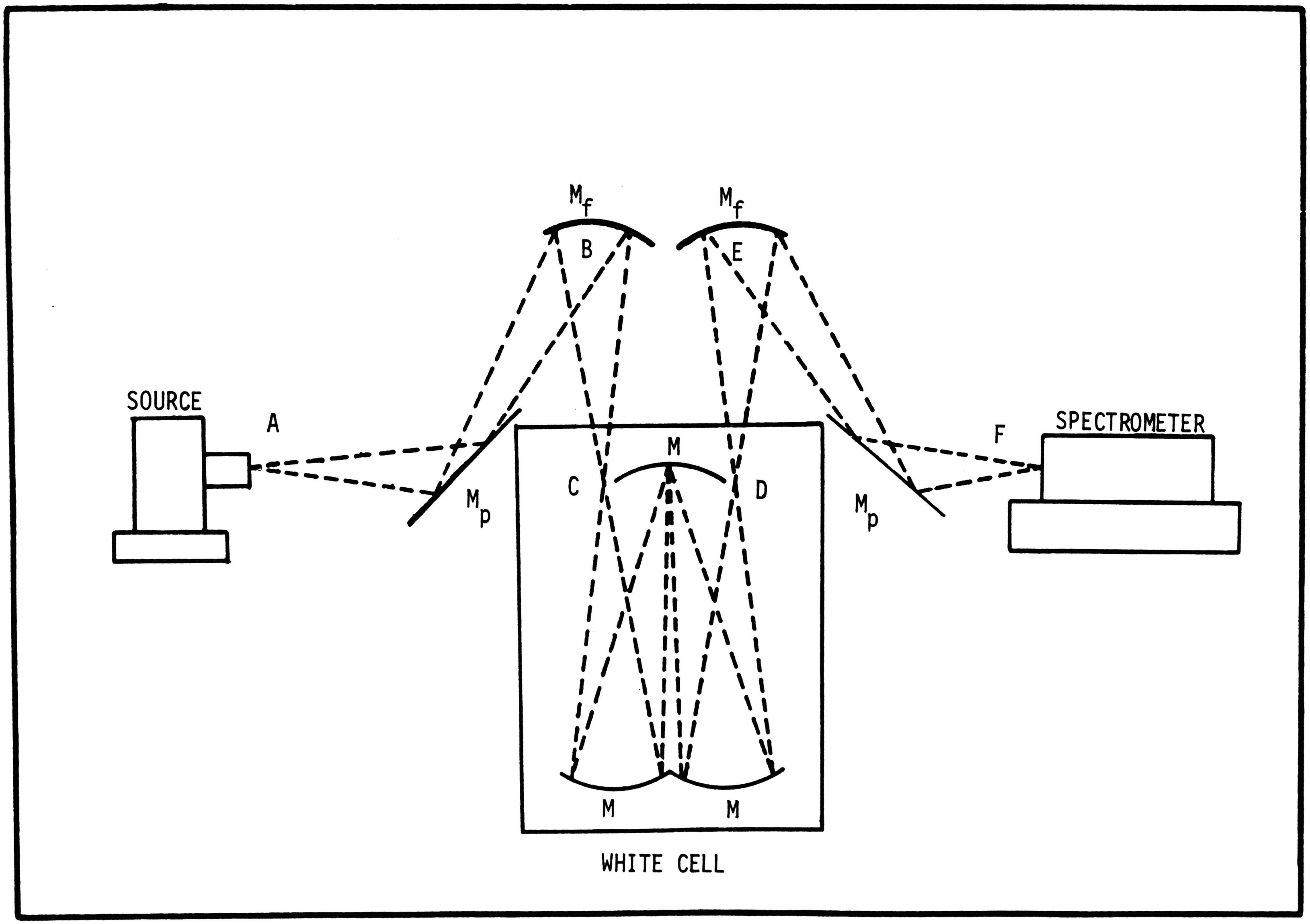


Figure 4. Schematic of Apparatus

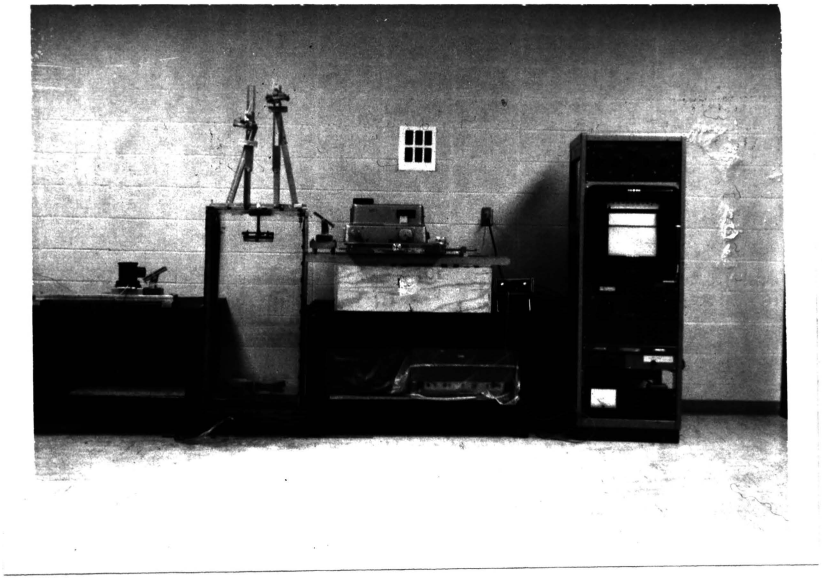
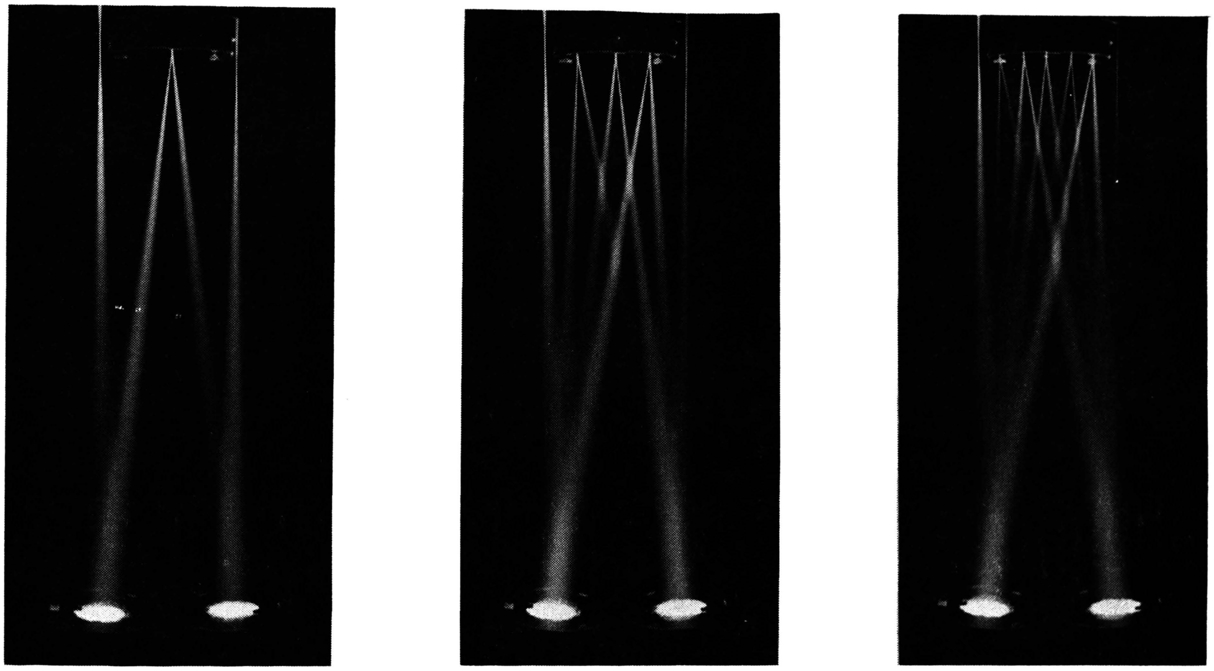


Figure 5. A Front View of Apparatus

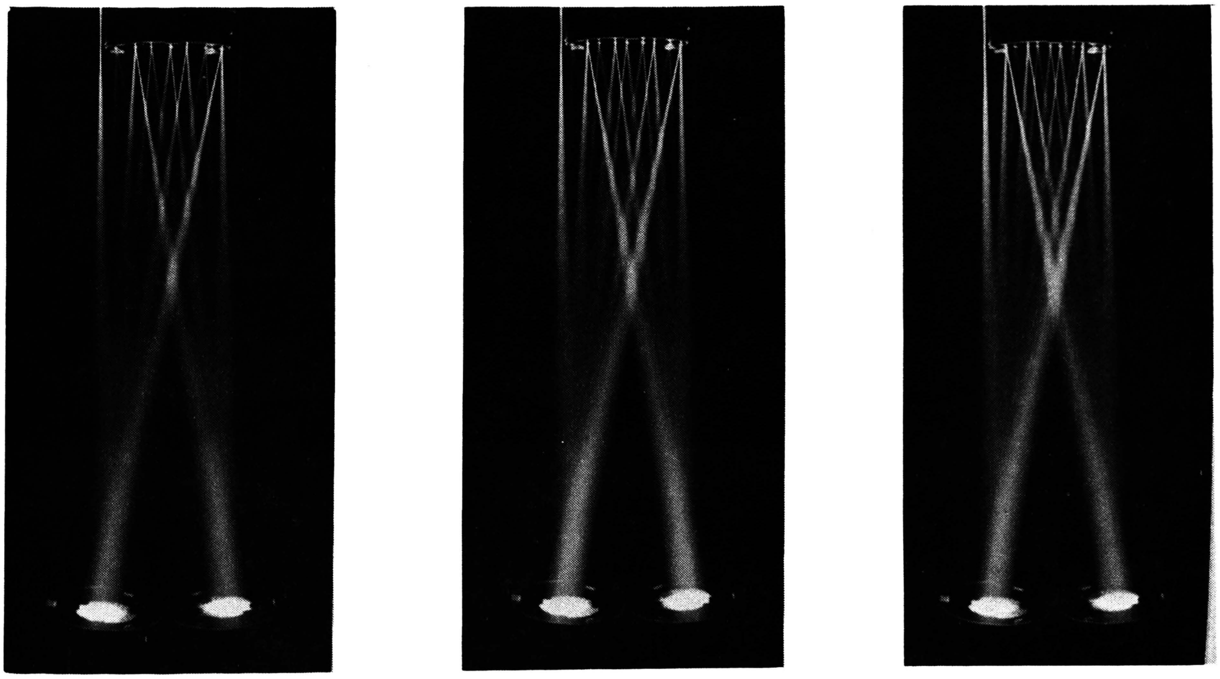
Two of its sides were made of 1/4 inch plexiglass plate for visual observation. All metallic parts within the cell were made of aluminum and they were coated with epoxy paint to prevent water contamination. The seams of the cell were sealed internally and externally by silicon sealant to make it water tight. The three spherical mirrors, M, are 6 inches in diameter and have a 32 inch radius of curvature. The two remaining spherical mirrors, M_f, also have a 32 inch radius of curvature but their diameter is only 4.25 inches. These two mirrors and the plane mirrors, M_p, formed the external optics which were used to direct the beam into the cell and then focus the transmitted beam onto the slit of the spectrometer. The path length can be changed by changing the inclination angle of the two adjacent spherical mirrors. A micrometer screw at the top of the cell and a connecting wire may be used to lift one side of each mirror and rotate it about the hinge to adjust their inclination angle. The optical distance from the source, point A, to the first focusing mirror, point B, is 39 inches; the distance from the focusing mirror to the focus point in the White cell, point C, is 26 inches; the distance from the other focus point of the White cell, point D, to the second focusing mirror, point E, is 27 inches; and the distance from this focusing mirror to the entrance slit of the spectrometer, point F, is 45 inches. Figure 6 shows pictorially the path followed by the light beam when the number of passes is changed from 4 to 28 corresponding to a change in length from 128 to 768 inches.

B. ALIGNMENTS AND CALIBRATION

The optical alignment of the apparatus and the spectrometer calibration are needed prior to any data acquisition. The first step in



4 Paths (128")　8 Paths (256")　12 Paths (384")



16 Paths (512")　20 Paths (640")　24 Paths (768")

Figure 6. Optical Paths in a White Cell

the alignment of the apparatus was to place all mirrors in the same optical plane and separate the spherical mirrors, which are inside the White cell, by a distance equivalent to their radius of curvature, 32 inches. Mirrors inclination angles and distances were selected and fixed, to focus the light beam from the source on the entrance point, C, of the White cell. This point must be in the same plane as the centers of the spherical mirrors, should coincide with the surface of the upper spherical mirror and should be placed as close as possible to the edge of that mirror. The bottom left spherical mirror is then adjusted such that the diverging beam from point C is centered on it. The inclination angle of this mirror, controlled by one of the micrometer screws, was then adjusted to reflect the incident beam and focus it at the center of the upper spherical mirror. Adjustments on the upper mirror were then made to center the reflected and diverging beam on the bottom right spherical mirror. The inclination angle of that mirror, controlled by a second micrometer screw, and its orientation were adjusted to reflect and focus the incident beam at point D which is symmetric, relative to the optical axis, to point C. To insure that the bottom mirrors rotate in the same plane, the inclination angle of each mirror was changed by using the micrometer screw, and the focused image was brought to coincide with point C. If these points coincide the White cell is properly aligned.

When the White cell is properly aligned one can change equally the inclination angles of the two bottom mirrors, by using the micrometer screws, and a series of images will appear on the top mirror all in line and evenly spaced. Figure 7 shows the case where the two bottom mirrors do not rotate in the same plane resulting in staggered set of

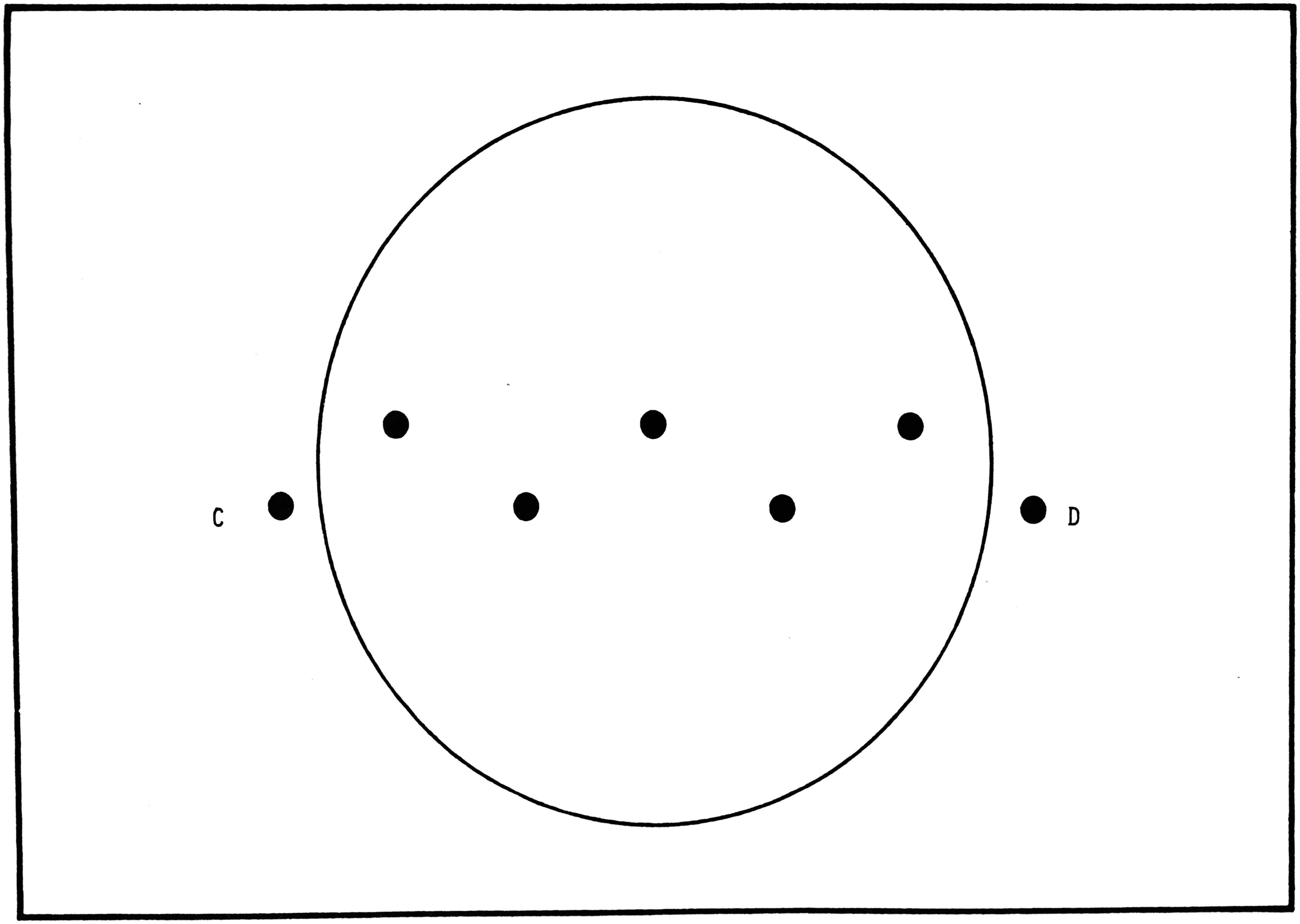


Figure 7. Staggered Images in the White Cell

images. Figure 8 shows the case where the two bottom mirrors are not at the same angle of inclination resulting in unevenly spaced images. Figure 9 shows the correct positioning of these images which would result from a properly aligned system.

Drum number calibration versus wavelength was performed by using calibrated transmittance Kodak Wratten gelatin filters number 58, 49, 47, 30, 25, 2A, and a sample of holium oxide. The results of such a calibration are shown in Figure 10. The repositioning of the spectrometer, which was required every time the number of passes through the cell were changed did not alter this calibration. The measured photomultiplier output when the cell was empty with four passes is shown in Figure 11. This represents the maximum output and the wavelength range that could be attained with the present apparatus. The low output at the extremities of this wavelength range makes the measurements there questionable and narrows the usable range of this spectrometer.

C. PROCEDURE AND ANALYSES

The White cell was aligned to provide four and eight passes and the transmitted signals through the empty cell were recorded over the wavelength range. At the beginning of each run, a source monitoring photodiode was used and adjustments were made to insure that the source strength was maintained at the same value throughout the experiment. Each time the number of passes through the cell was changed from four to eight and vice versa, the spectrometer was repositioned to obtain the maximum output at a selected drum number. The measurements obtained with the empty cell could be used to evaluate the average

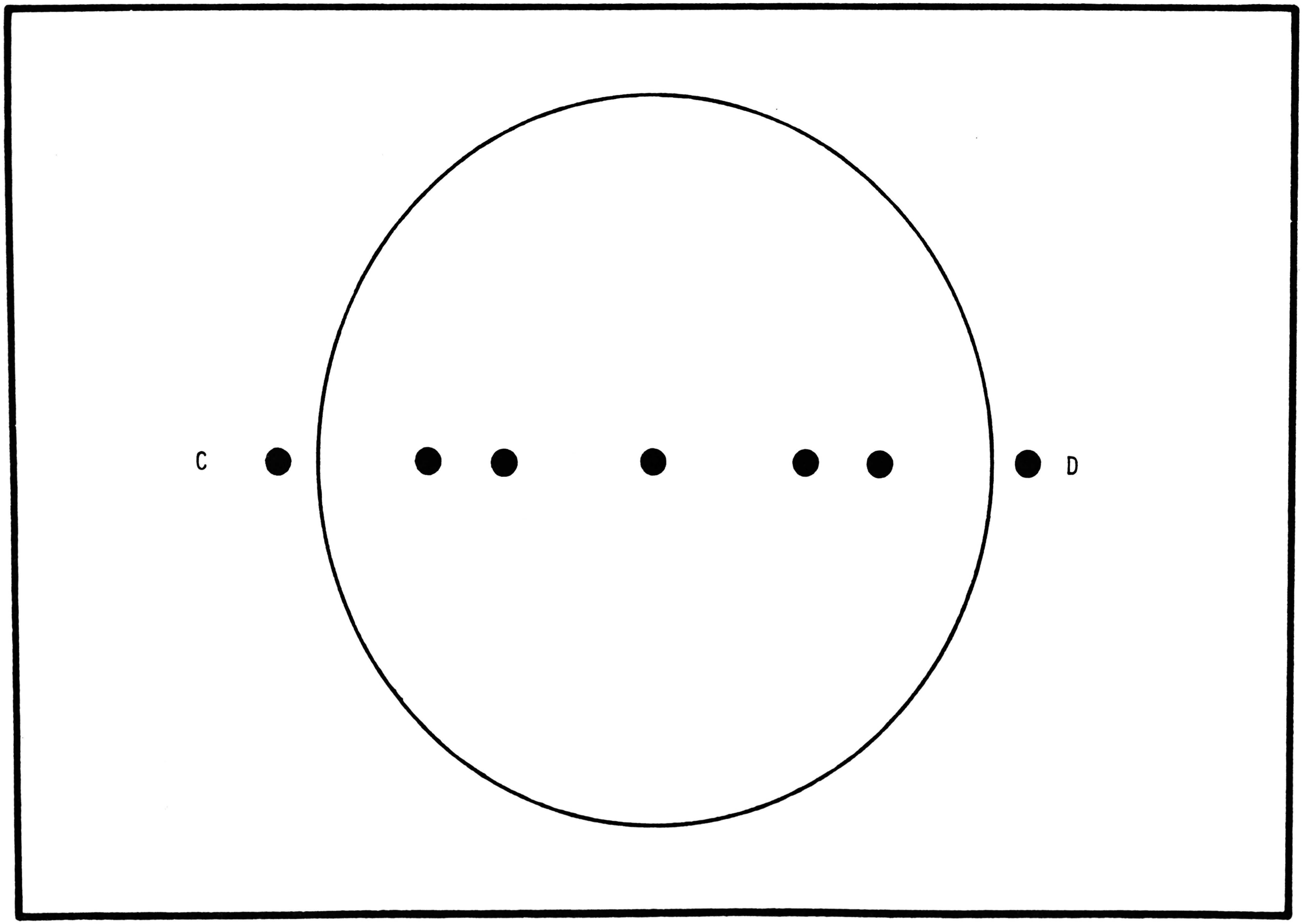


Figure 8. Unevenly Spaced Images in the White Cell

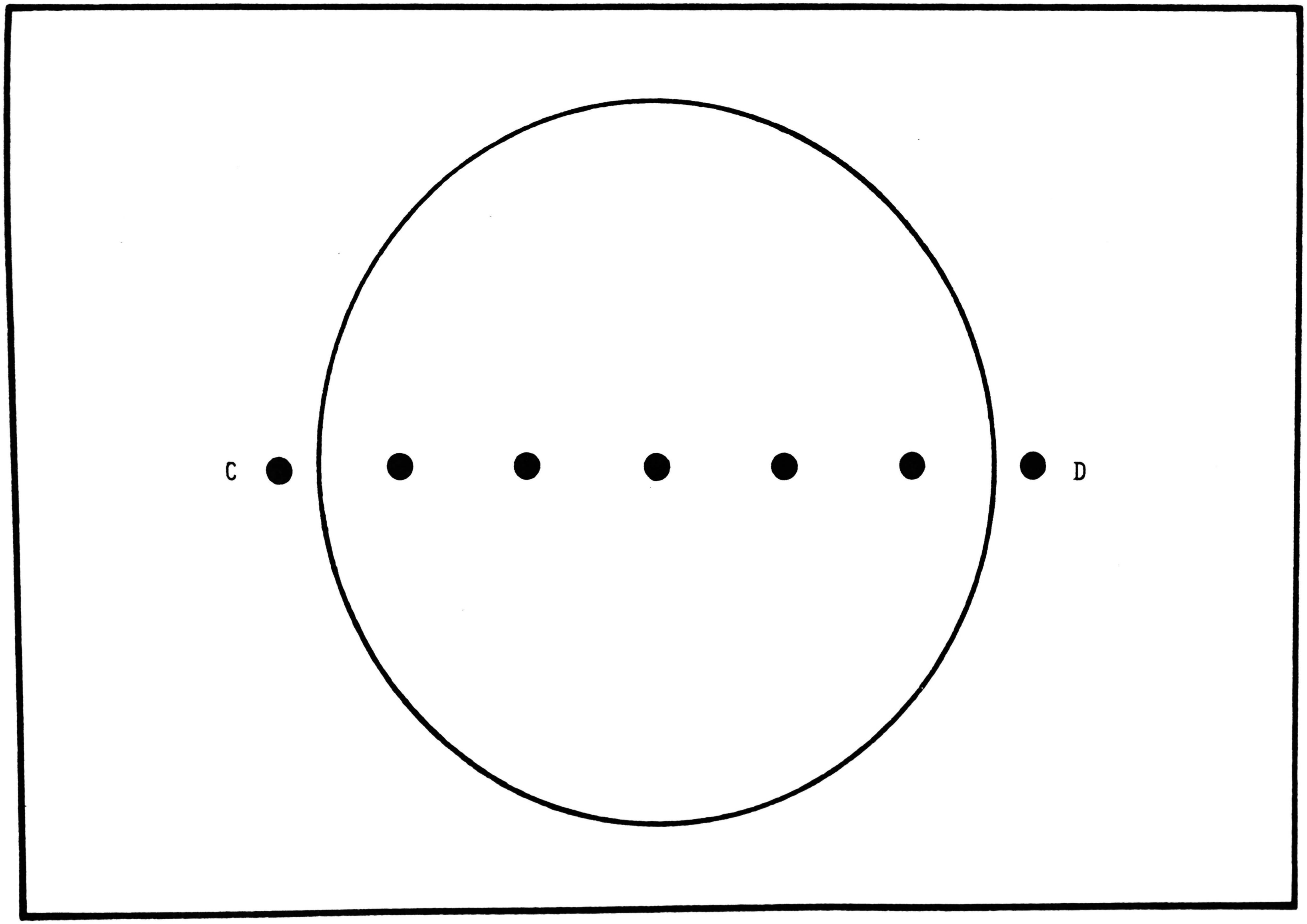


Figure 9. Correct Positions of Images in the White Cell

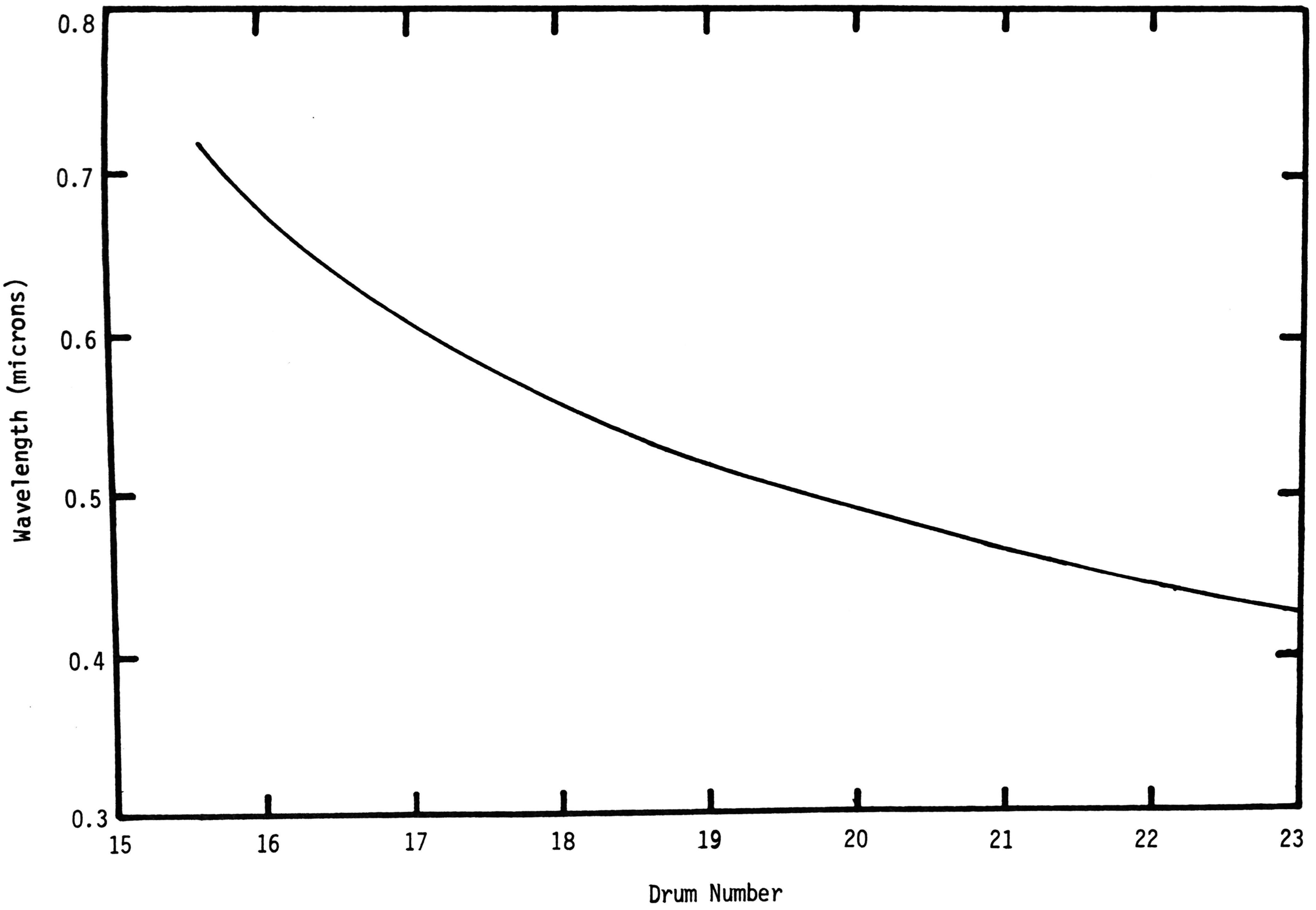


Figure 10. Drum Number Calibration Versus Wavelength

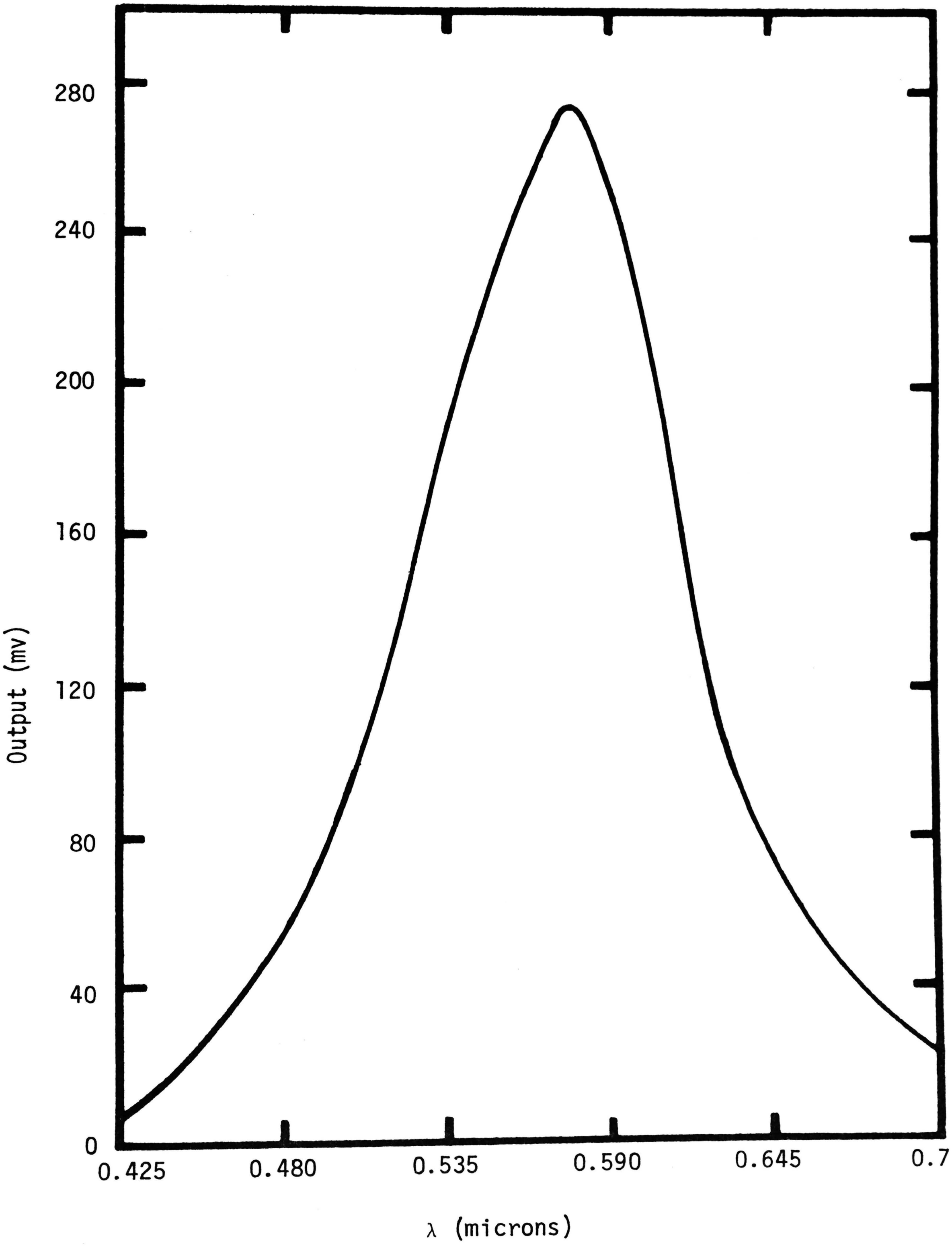


Figure 11. Measured Output for an Empty White Cell with Four Passes

mirror reflectance versus wavelength. By assuming that the three mirrors have identical properties and the reflectance from each one is the same, the intensity leaving the empty cell after four passes, $I_{4a\lambda}$, can be expressed in terms of the inlet intensity, $I_{o\lambda}$, which was maintained constant throughout the experiment, by

$$I_{4a\lambda} = I_{o\lambda}\,\rho_{a\lambda}^{3} \tag{15}$$

where $\rho_{a\lambda}$ is the average mirror-air interface reflectance. The intensity leaving the same cell after eight passes, $I_{8a\lambda}$, is given by

$$I_{8a\lambda} = I_{o\lambda}\,\rho_{a\lambda}^{7} \; . \tag{16}$$

The average mirror-air reflectance can be deduced by taking the ratio of the above equations:

$$\rho_{a\lambda} = \left(\frac{I_{8a\lambda}}{I_{4a\lambda}}\right)^{1/4} \; . \tag{17}$$

The White cell was then filled with distilled water up to the surface of the upper spherical mirror where the incident and the departing light beam focuses, i.e., points C and D. It was found that when the cell is only partially filled with water, the water refracts the light in such a way that proper alignment could not be achieved. The filled cell was aligned to provide four and eight passes and the transmitted signals were recorded versus wavelength. This arrangement provided a path length of x_1 = 325 cm. and the other, x_2 = 650 cm. By assuming that attenuation through the water follows Beer's law, the intensity leaving the cell after four passes, $I_{4w\lambda}$, can be expressed in terms of the inlet intensity by

$$I_{4w\lambda} = I_{o\lambda}\, T_{\lambda}^{2}\, \rho_{w\lambda}^{3}\, e^{-\beta_{\lambda} x_{1}} \tag{18}$$

where T_{λ} is water-air interface transmittance, β_{λ} is the extinction coefficient, x_1 is the path length with four passes and is equivalent to 325 cm. and $\rho_{w\lambda}$ is the mirror-water interface reflectance. For eight passes the intensity leaving the cell, $I_{8w\lambda}$, is given by

$$I_{8w} = I_{o\lambda}\, T_{\lambda}^{2}\, \rho_{w\lambda}^{7}\, e^{-\beta_{\lambda} x_{2}} \tag{19}$$

where x_2 is the path length with eight passes and is equivalent to 650 cm.

Equations (15), (16), (18), and (19) are four independent equations with five unknowns, $I_{o\lambda}$, T_{λ}^{2}, $\rho_{w\lambda}$, $\rho_{a\lambda}$, and β_{λ}, for which solutions could not be obtained. The acquisition of additional data by making measurements with twelve passes through the White cell provides two additional equations; however, these equations are not independent from the previously available four. For example, measurements with twelve passes while the cell is empty would provide the following equation:

$$I_{12a\lambda} = I_{o\lambda}\, \rho_{a\lambda}^{11} \tag{20}$$

where $I_{12a\lambda}$ is the intensity leaving the empty cell with twelve passes. The variables appearing in this equation are the same as the ones appearing in equations (15) and (16) which are sufficient for providing a solution for each $I_{o\lambda}$ and $\rho_{a\lambda}$. Measurements with twelve passes when the cell is filled with water would provide the following relation:

$$I_{12w\lambda} = I_{o\lambda}\, T_\lambda^{\,2}\, \rho_{w\lambda}^{\,11}\, e^{-\beta_\lambda x_3} \qquad (21)$$

where x_3 is the path length with 12 passes and is equivalent to 975 cm. and $I_{12w\lambda}$ is the intensity leaving the cell at that setting. Equations (18), (19), and (21) might lead one to believe that they are independent and as such should provide for the solution of the three unknowns, $\rho_{w\lambda}$, β_λ, and the combination of $I_{o\lambda}\, T_\lambda^{\,2}$. However, upon further investigation and the fact that $x_3 = 3x_1$ and $x_2 = 2x_1$, one can show that equation (21) is a linear combination of the other two equations (18) and (19); for example:

$$\frac{I_{12w\lambda}}{I_{8w\lambda}} = \left(\frac{I_{12w\lambda}}{I_{4w\lambda}}\right)^{1/2} = \frac{I_{8w\lambda}}{I_{4w\lambda}} = \rho_{w\lambda}^{\,4}\, e^{-\beta x_1} \qquad (22)$$

The above fact makes the White cell unsuitable for determining the extinction coefficient for liquids unless additional information is available from other sources to define and evaluate one of the unknown parameters. Faced with this dilema the measured data was used and implemented with other available data to estimate the extinction coefficient for distilled water. Equations (15), (16), (18), and (19) were combined to yield the following relation for the extinction coefficient:

$$-\beta_\lambda = \frac{1}{x_1} \ln\left[\frac{(I_{8w\lambda}/I_{8a\lambda})}{(\rho_{w\lambda}/\rho_{a\lambda})^4\,(I_{4w\lambda}/I_{4a\lambda})}\right] \qquad (23)$$

where the ratios of the reflectances $(\rho_{w\lambda}/\rho_{a\lambda})$ will be estimated from a source other than this experimental data. Note that if gas filled the White cell in place of the liquid, then there will be essentially no change in the mirror reflectances and the reflectances' ratio appearing in equation (23) becomes equal to unity. This is what makes such a cell suitable for determining the extinction coefficient for gases. The important factors in equation (23) are ratios of measured intensities at the same path length so minor errors in cell alignment will cancel and will not drastically affect the magnitude of these ratios. Amplification of errors is minimized since only one term in the equation is raised to a power higher than unity.

The measured data can be reduced from the same four equations by eliminating the reflectances' ratio and expressing the final results in terms of the air-water interface transmittance which is also unknown. Such a reduction will yield the following relations:

$$\beta_\lambda = \frac{1}{x_1} \ln \left[\frac{(I_{8w\lambda}/I_{8a\lambda})^3 \, T_\lambda{}^8}{(I_{4w\lambda}/I_{4a\lambda})^7} \right] . \qquad (24)$$

Note that in this relation each term is raised to a different power and the unknown parameter, T_λ, is raised to the eighth power. This fact will lead one to believe that equation (24) will amplify strongly any errors in intensity measurements and interface transmittance calculations. Indeed, when one compares the results of equation (24) with those deduced from equation (23), large differences exist. Based on the above fact and the desire to dampen rather than amplify experimental and calculated errors, equation (23) was used to evaluate the extinction coefficient from the experimental results.

To examine experimentally the influence of an oil film on the energy transmitted through the water interface, the White cell was filled with distilled water to a level of 9 cm. above the surface of the top spherical mirror and was aligned to provide four passes. A glass ring 3 inches in diameter with a float was located at the surface of the water and in the path of the light beam. This ring was used to confine the oil film and permitted the light beam to pass once through the oil layer. Measurements through such a system were taken first when the ring did not contain an oil film which accounted for any surface tension effects inside the ring. This measurement provided data for the water transmittance capability. A specified volume of oil was then added inside the ring to form a thin and uniform layer in the center of the ring. Transmittance measurements through this system were taken over the wavelength range in a procedure similar to the one used when there was no oil film. The thickness of the oil film was increased and the influence of that increase on the transmitted energy was measured. The above procedure was repeated for crude oil and fuel oils number 2 and 3.

The thickness of the oil film versus volume added was calibrated for the three types of oil used by using a micrometer. It was found that in order to produce a uniform film cover inside the ring and over the water surface, a minimum of two cubic centimeters needed to be added. This represented a thickness of approximately 0.01 inches.

The calibration curve for these oils is presented in Figure 12 and the accuracy of their measurements should be within 0.001 inches. The curve for crude oil appears somewhat non-linear. It is believed that this is due to the higher viscosity of the oil.

The intensity leaving the cell when the glass ring has no oil cover, $I_{w\lambda}$, can be expressed using equation (18) by

$$I_{w\lambda} = I_{o\lambda} T_{\lambda}^{2} e^{-\beta x} \rho_{w\lambda}^{3} \qquad (25)$$

where x is the path length and is equivalent to 343 cm. The intensity leaving the cell when the glass ring has an oil cover, $I_{1\lambda}$, can be expressed by

$$I_{1\lambda} = I_{o\lambda} T_{1\lambda} T_{\lambda} e^{-\beta x} \rho_{w\lambda}^{3} \qquad (26)$$

where $T_{1\lambda}$ is the oil film transmittance. The ratio of equations (25) and (26) provides a measure of the oil film transmittance in comparison with air-water interface transmittance

$$\frac{I_{1\lambda}}{I_{w\lambda}} = \frac{T_{1\lambda}}{T_{\lambda}} \quad . \qquad (27)$$

D. RESULTS AND DISCUSSION

As discussed in the previous section, transmission measurements through the White cell do not provide sufficient information to evaluate the extinction coefficient of liquids. Additional information, that could be provided by performing an additional experiment which will determine the ratio of the mirror-air to mirror-water interface reflectance, is needed in order to make the White cell suitable for

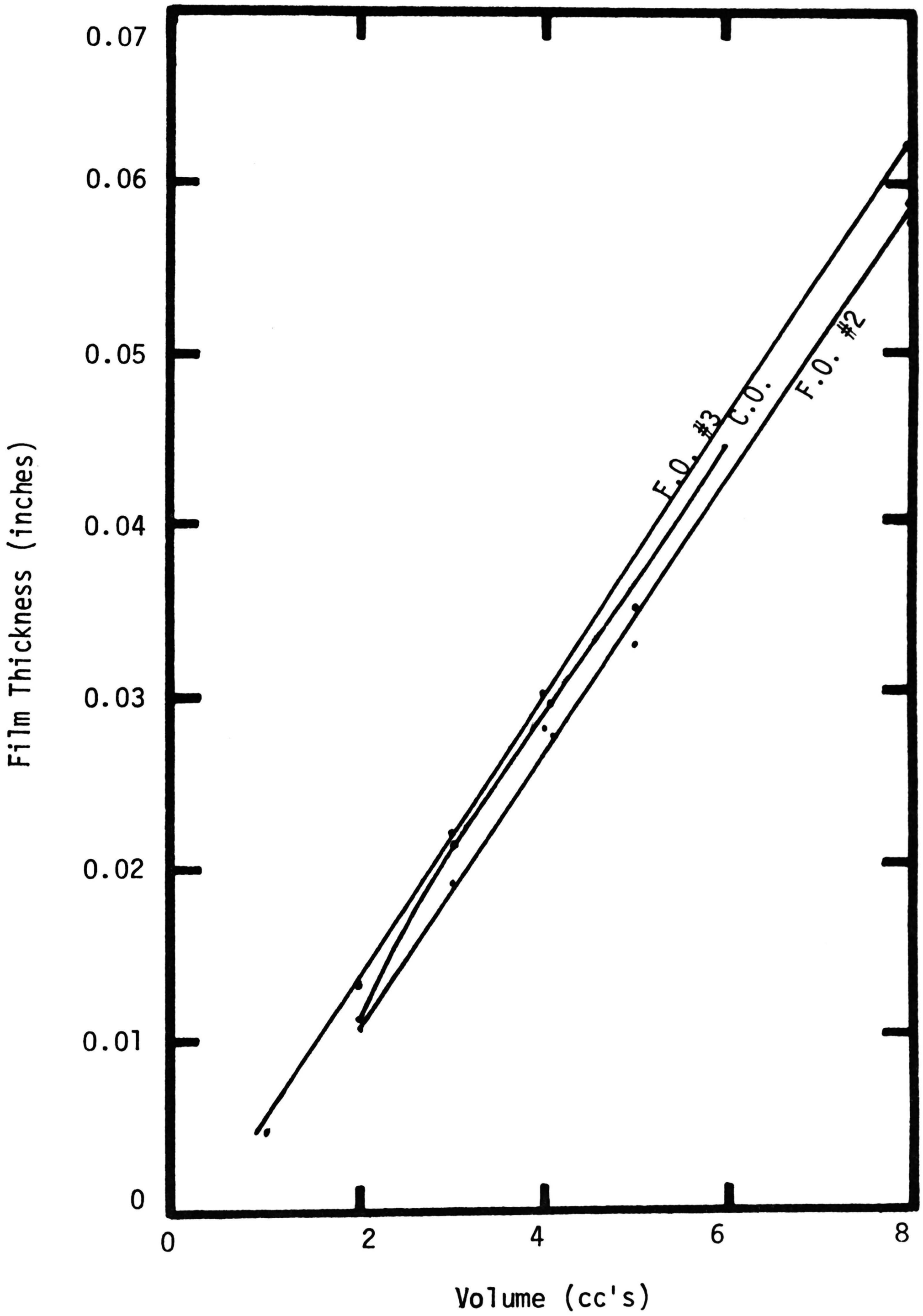


Figure 12. Oil Film Calibration Curves (Thickness Versus Volume)

liquids. Such an experiment was not performed in this study; however, the correction was theoretically predicted from data appearing in the literature. This fact makes the reported results for the extinction coefficient of distilled water as only approximate ones. The results dealing with the oil film, however, are not affected by the above difficulties and they should reflect accurate behavior.

Equation (23) was used to evaluate the extinction coefficients of distilled water from the measured transmission data. Due to the fact that the index of refraction of water is always higher than that of air, the mirror-air interface reflectance will always be larger than the mirror-water interface reflectance. This fact can be used to generate an upper bound for the extinction coefficient by forcing the reflectances to be equal to each other. Under these conditions equation (23) reduces to the following form:

$$-\beta_{u\lambda} = \frac{1}{x_1} \ln \left[\frac{I_{8w\lambda} \quad I_{4a\lambda}}{I_{8a\lambda} \quad I_{4w\lambda}} \right] \tag{28}$$

where $\beta_{u\lambda}$ stands for the upper bound value of the extinction coefficient. The results of these calculations are presented in Figure 13 and in Table 1 and are compared with those of Hulburt[2]. As one should expect they follow the same trend but have a higher magnitude.

In an effort to account in some way for the changes in the mirror reflectances $(\rho_{w\lambda}/\rho_{a\lambda})$, an expression was derived from the Fresnel relations. For normally incident radiation, which is a realistic approximation of the apparatus, surface reflectance, ρ_λ, can be generally evaluated by using the following relation:

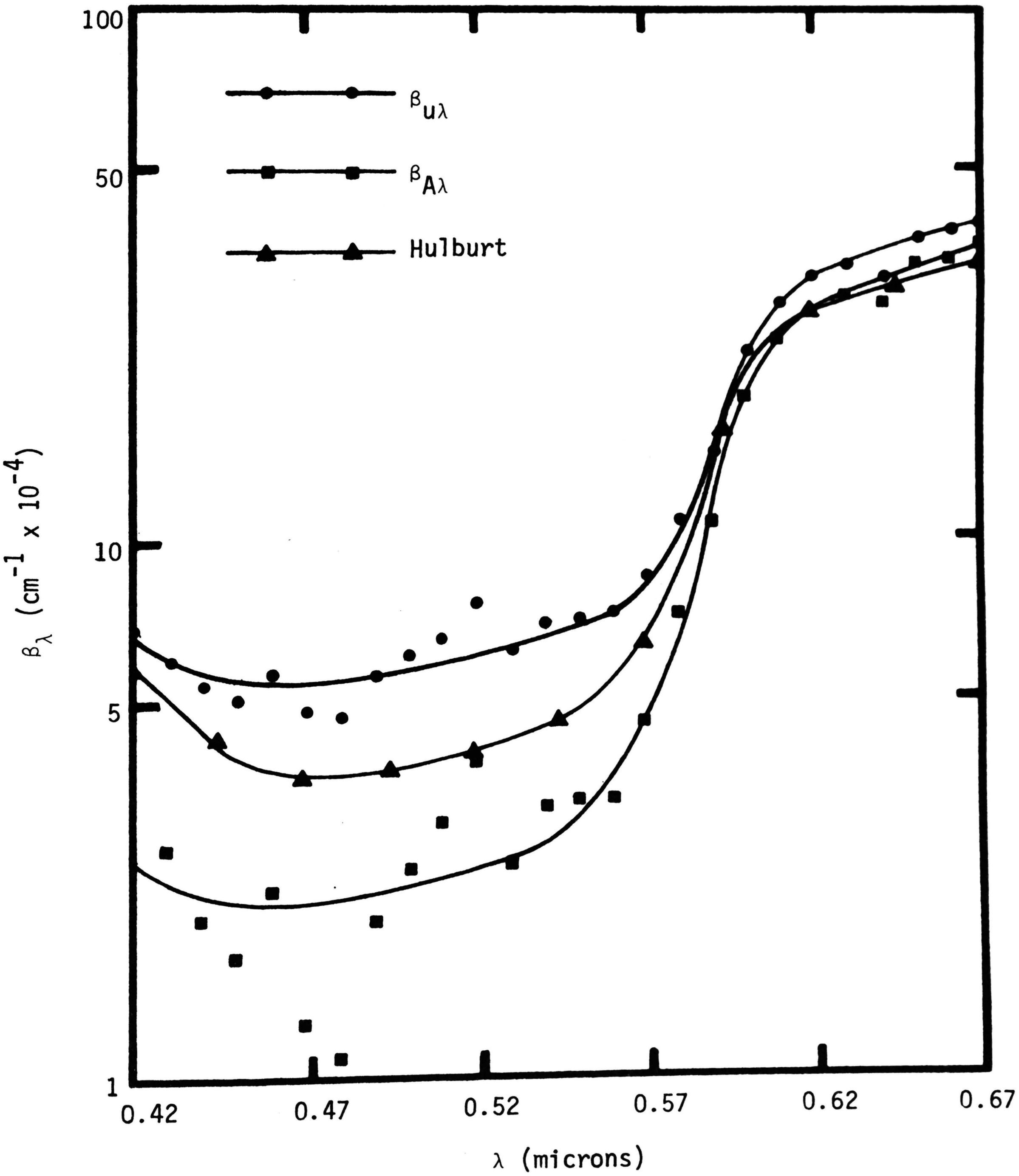


Figure 13. Experimental Extinction Coefficient of Distilled Water

TABLE 1

Experimental Extinction Coefficient of Distilled Water ($cm^{-1} \times 10^{-4}$)

λ	$\beta_{u\lambda}$	$\beta_{A\lambda}$
0.43	6.0	2.7
0.44	5.4	2.0
0.45	5.1	1.7
0.46	5.7	2.2
0.47	4.8	1.3
0.48	4.7	1.1
0.49	5.6	2.0
0.50	6.2	2.5
0.51	6.7	3.0
0.52	7.7	3.8
0.53	6.3	2.5
0.54	7.1	3.2
0.55	7.2	3.2
0.56	7.3	3.3
0.57	8.7	4.7
0.58	11.4	7.3
0.59	14.8	10.7
0.60	23.0	18.9
0.61	28.2	24.1
0.62	31.7	27.5
0.63	32.9	28.7
0.64	31.5	27.3
0.65	37.5	33.3
0.66	38.8	34.7
0.67	40.9	36.7
0.68	51.6	47.5

$$\rho_\lambda = \frac{(n_{2\lambda} - n_{1\lambda})^2 + (k_{2\lambda} - k_{1\lambda})^2}{(n_{2\lambda} + n_{1\lambda})^2 + (k_{2\lambda} + k_{1\lambda})^2} \tag{29}$$

where n_λ and k_λ are the real and imaginary part of the complex index of refraction. Subscript 2 refers to the mirror surface and 1 refers to the bounding medium. When the bounding medium is air, $n_{1\lambda} = 1$ and $k_{1\lambda} = 0$ resulting in the following expression for the reflectance:

$$\rho_{a\lambda} = \frac{(n_{2\lambda} - 1)^2 + k_{2\lambda}{}^2}{(n_{2\lambda} + 1)^2 + k_{2\lambda}{}^2} \tag{30}$$

When the bounding medium is water, $n_{1\lambda} = 1.33$ and $k_{1\lambda} = 0$ is a good approximation in the experimental wavelength range. The mirror reflection becomes

$$\rho_{w\lambda} = \frac{(n_{2\lambda} - 1.33)^2 + k_{2\lambda}{}^2}{(n_{2\lambda} + 1.33)^2 + k_{2\lambda}{}^2} \ . \tag{31}$$

By replacing the constant 1.33 appearing in the above equation by (1 + 0.33), the reflectance can be expressed as follows:

$$\rho_{w\lambda} = \frac{(n_{2\lambda} - 1)^2 + k_{2\lambda}{}^2}{(n_{2\lambda} + 1)^2 + k_{2\lambda}{}^2} \left\{ \frac{1 + \left[0.0989 - 0.66\ (n_{2\lambda} - 1)\right] / \left[(n_{2\lambda} - 1)^2 + k_{2\lambda}{}^2\right]}{1 + \left[0.0989 + 0.66\ (n_{2\lambda} + 1)\right] / \left[(n_{2\lambda} + 1)^2 + k_{2\lambda}{}^2\right]} \right\} \tag{32}$$

The first term in the above equation is in fact the mirror-air interface reflectance. The reflectance ratio is then given by

$$\frac{\rho_{w\lambda}}{\rho_{a\lambda}} = \frac{1 + \left[0.0989 - 0.66\,(n_{2\lambda} - 1)\right] / \left[(n_{2\lambda} - 1)^2 + k_{2\lambda}{}^2\right]}{1 + \left[0.0989 + 0.66\,(n_{2\lambda} + 1)\right] / \left[(n_{2\lambda} + 1)^2 + k_{2\lambda}{}^2\right]} \qquad (33)$$

This ratio was calculated using the optical constants for aluminum film, which is a common mirror surface, reported by Weistein[19] and presented below.

λ	$n_{2\lambda}$	$k_{2\lambda}$
0.4	0.38	3.90
0.5	0.62	4.85
0.6	0.97	5.85
0.7	1.35	7.00

Values at an intermediate point were interpolated from the above results. The ratio calculated from equation (33) varied monotomically between 0.876 to 0.898 over the experimental wavelength range. These values were used with equation (23) to evaluate a corrected but approximate value for the extinction coefficient of distilled water, $\beta_{A\lambda}$, from the measured transmittance data. These results are also presented in Figure 13 and in Table 1. They are within 100% of Hulburt's data and exhibit a maximum deviation at λ = 0.42 microns.

Equation (27) was used to deduce the ratio of the oil film transmittance to air-water interface transmittance. These calculations are presented in Figures 14 through 17 for fuel oil 2 and 3 and mid-western crude oil. In all the results this ratio decreases as the wavelength decreases due to the increase in the extinction coefficient of the oil. The reductions increased with an increase in

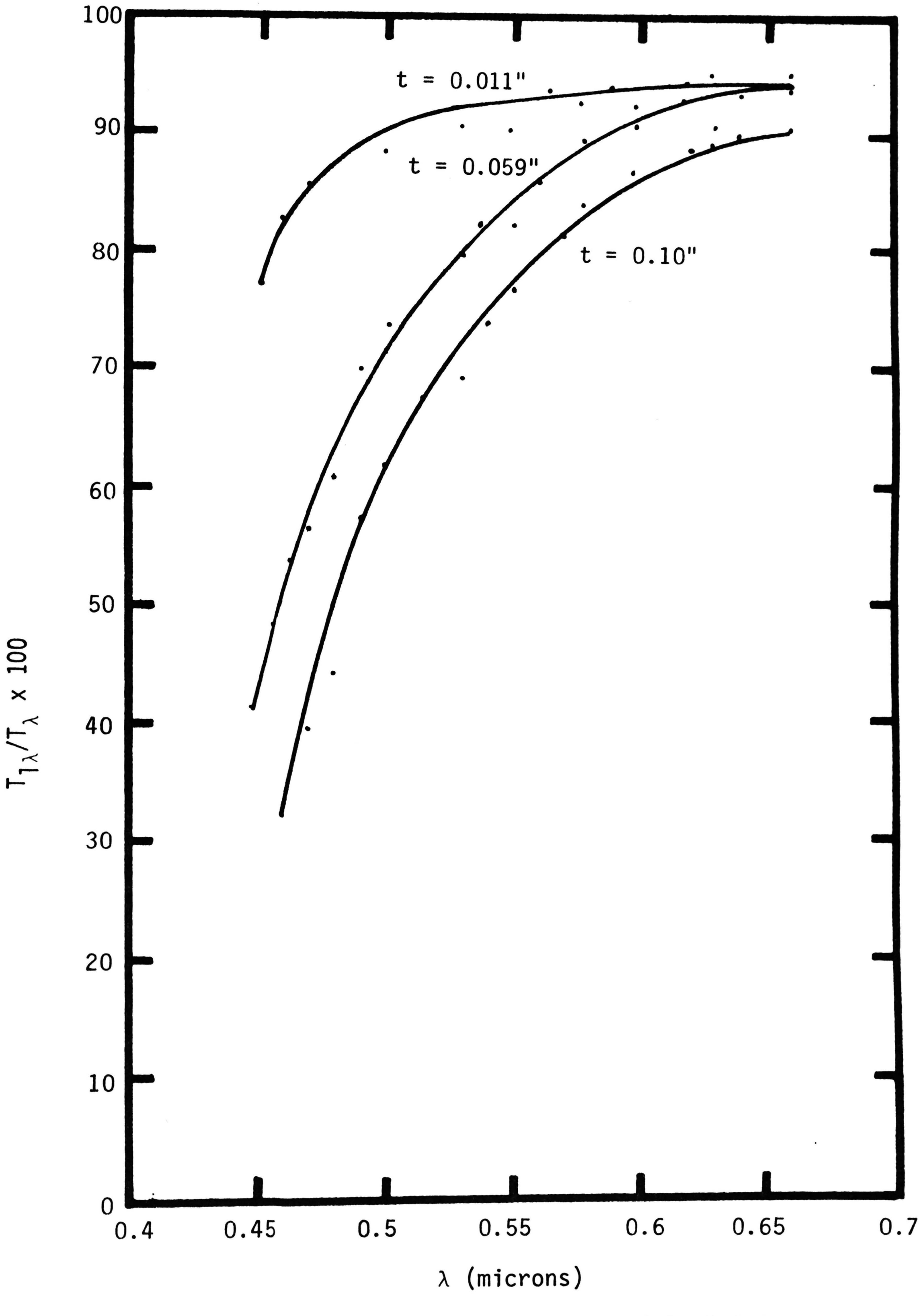


Figure 14. Film Transmittance of Fuel Oil #2

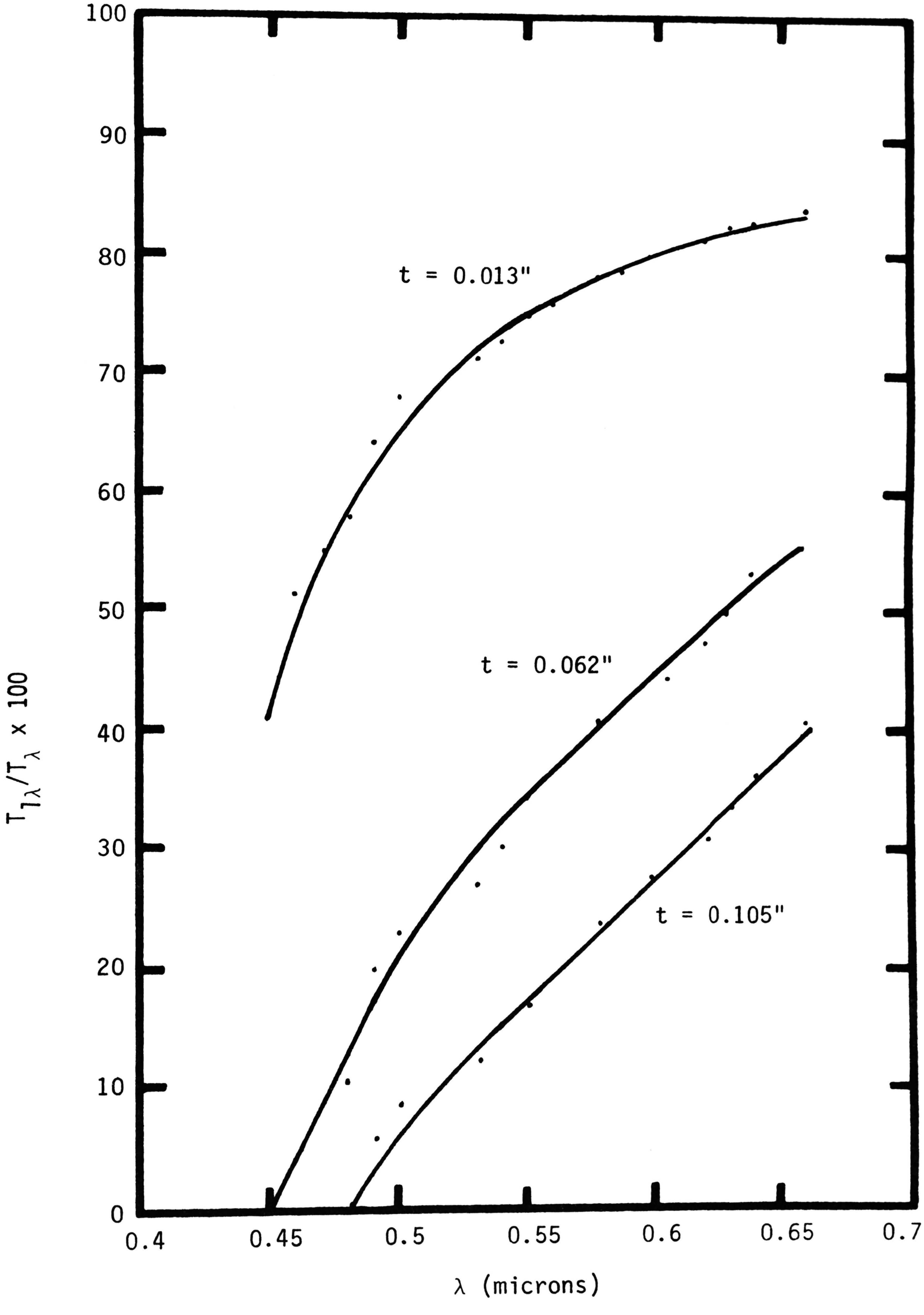


Figure 15. Film Transmittance of Fuel Oil #3

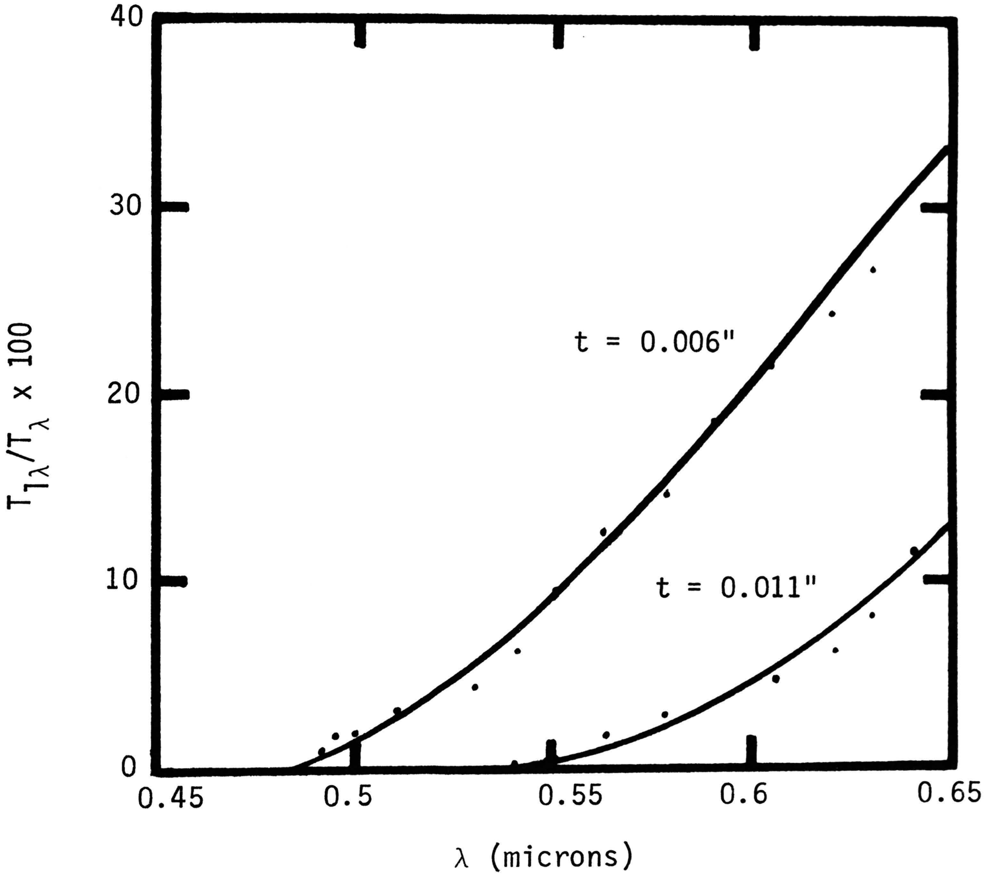


Figure 16. Film Transmittance of Midwestern Crude Oil

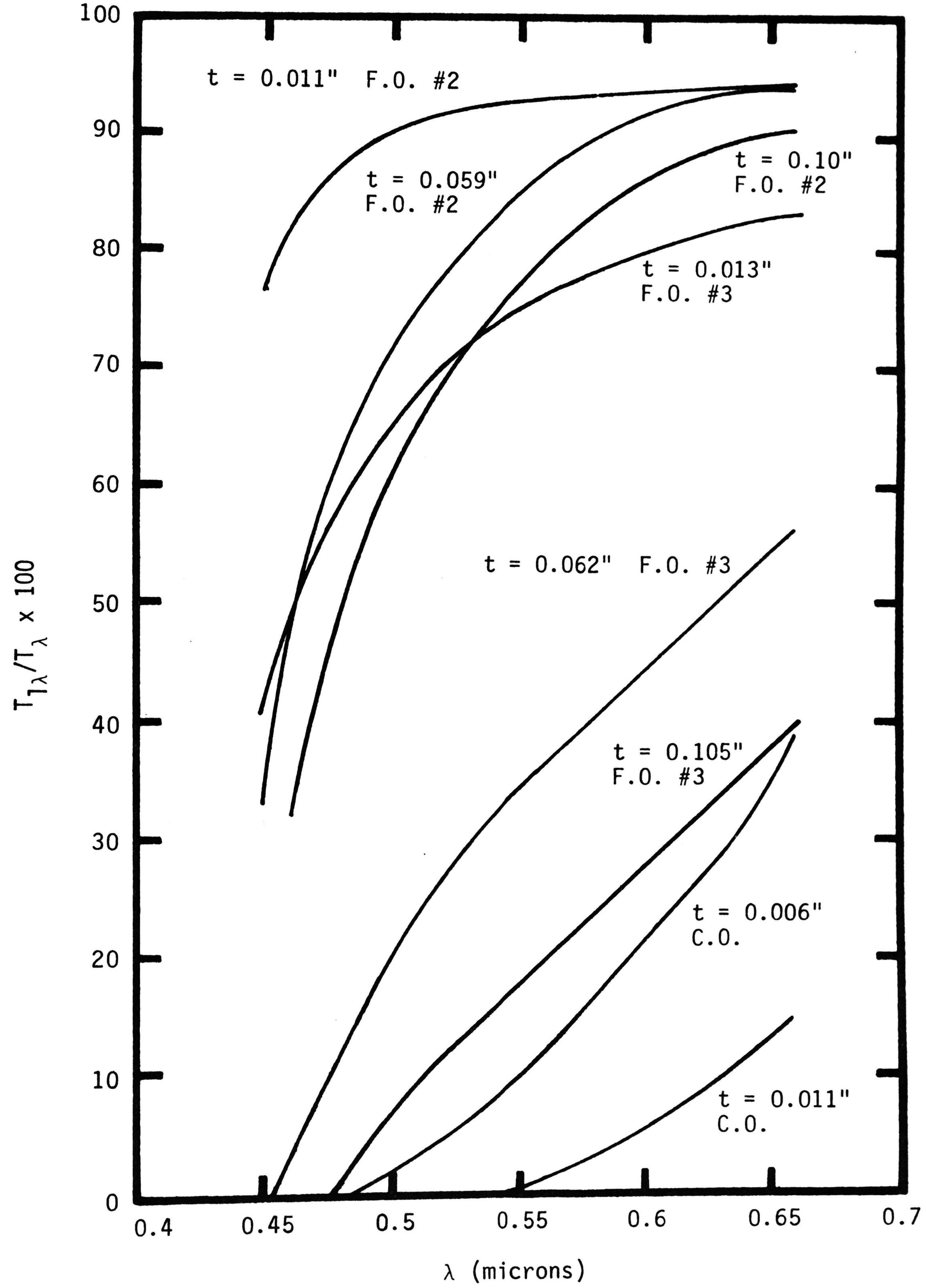


Figure 17. Film Transmittance of Fuel Oil #2, #3, and Midwestern Crude Oil

the fuel oil number and with the film thickness. The thicknesses of the oils used were fuel oil number 2 -- 0.011, 0.059, and 0.1 inches; fuel oil number 3 -- 0.013, 0.062, and 0.105 inches; and midwestern crude oil -- 0.006 and 0.011 inches. All the thicknesses are accurate to 0.001 inches except for 0.006 inches of crude oil. It is accurate to 0.002 inches.

V. CONCLUSIONS AND RECOMMENDATIONS

Transmission measurements through distilled water in a White cell were performed in the visible region of the spectrum with four and eight cell passes. These measurements did not provide sufficient information to evaluate the extinction coefficient of distilled water. The ratio of mirror-water to mirror-air interface reflectances which were needed to uniquely evaluate the above property from the transmission data was considered unity to provide an upper limit. The reported extinction coefficients are within the scattered range of literature values.

The influence of oil film on interface transmittance was experimentally measured for fuel oils 2 and 3 and midwestern crude oil. The influence increases with an increase in fuel oil number. Crude oil affected the interface transmittance more than the other two fuel oils. Generally, the effect increases as the wavelength decreases due to a sharp increase in the extinction coefficient of oil.

The experiment reveals that transmission measurements through liquids in a White cell is not sufficient for determining its extinction coefficient. Results from an additional experiment, which will determine the ratio of mirror-water to mirror-air interface reflectances will be needed to make these measurements usable. A one pass cell appears to be more suitable for that purpose and will provide more accurate values due to the fewer number of required experimental measurements. Analyses reveal that for best results, the second cell should provide an optical path which is 1.3 longer than the small cell. The small cell can be as small as practically possible and will provide for elimination of the window-liquid transmittance.

To improve the quality of future experiments, the following recommendations are made:

1. An attempt should be made to stabilize the light source. This would reduce the fluctuations in the intensity measurements and yield more accuracy in these measurements.
2. A series of different sources and detectors should be used which would allow an extension of the wavelength range.
3. The spectrometer slit width should be reduced so one can obtain a higher resolution.
4. A more accurate means of measuring film thickness and a more critical determination of the oil's extinction coefficient are needed before meaningful results can be interpreted from the study of oil films on water.

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