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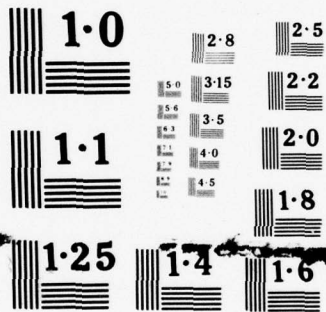
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SALISBURY, SOUTH AUSTRALIA

TECHNICAL REPORT 1711 (A)

A REFLECTOMETER FACILITY FOR THE MEASUREMENT OF TRANSMISSION  
AND REFLECTION OF LIGHT AT NORMAL INCIDENCE

J.R. VENNING

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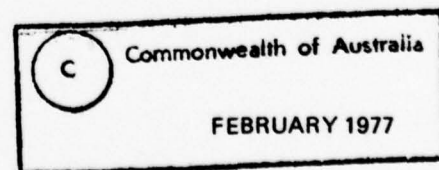
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#### S U M M A R Y

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The reflectometer facility and the procedure for alignment of the reflectometer is described.

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The reflectometer facility and the procedure for alignment of the reflectometer is described.

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## 1. INTRODUCTION

A reflectometer facility has been designed and built to measure the reflection and transmission of light from flat samples. These measurements are a necessary part of the study of the optical and physical properties of thin film interference filters. Data from this facility is used to assess the performance of thin film filters and to calculate the optical constants ( $n$  and  $k$ ) of the materials used to make them.

The physical and optical properties of some of the materials used in the manufacture of thin films have been widely reported in the literature, however, there are large variations in the published data and very little information concerning some commonly used materials. Zirconium dioxide, a useful material, is not very well reported in the literature and the optical constants of this material have been measured and are included in this report. It is generally accepted that the properties of thin film materials are affected by the pressure within the vacuum chamber during deposition, the rate of deposition, the temperature of the substrate, the purity of the source material and the thickness of the deposited layer, and it is possibly for these reasons that the published data is so variable.

The theory of thin film filter systems is well developed and the calculation of the theoretical performance of any given system is readily accomplished using a computer(ref.1,2,3,4,5). The reflection and transmission characteristics of a thin film filter can be calculated using the optical constants of the materials used to construct the filter. These calculated values can be compared with transmission and reflection measurements made of the actual filter and the degree of agreement achieved in this comparison depends on the accuracy of the values of the optical constants used in the calculation. Transmission curves calculated from published values of  $n$  and  $k$  do not always match the curves derived from actual films. This can be due to the way in which the films are made or inaccuracies in the published data. A knowledge of the optical constants of materials in the form in which they are actually used is therefore essential for the successful design of thin film systems.

There are many methods of measuring and deducing the optical constants of materials in thin film form. These can be roughly divided into two main categories: reflection/transmission methods and polarisation methods. Each method has its own distinctive limitations either in difficulty of measurement or in subsequent data reduction and interpretation.

An instrument which measures transmission and reflection at normal incidence has a distinct advantage because the measurements may be used directly to define the performance of a thin film system as well as providing data which can be used to calculate the optical constants of the materials used. The reduction of reflection and transmission data to determine the optical constants of thin films has been simplified by re-arranging the Fresnel equations as suggested by Denton, Campbell, Tomlin(ref.6,7,8).

This technical report describes the normal incidence reflectometer facility developed at W.R.E. and discusses the alignment procedure employed. Results are given for the optical constants of zirconium dioxide and the reflectance of aluminium and aluminium over-coated with glass.

## 2. SINGLE AND DOUBLE BEAM MEASUREMENT SYSTEMS

The simplest method of measuring the reflection or transmission of a sample is to measure the flux in an incident beam of radiation and to compare this measurement with a measurement of the flux reflected or transmitted by the sample when it is placed in the beam. The accuracy with which these ratios may be obtained using a single beam arrangement is limited by variations in the detector, the source, and the electronics. The dependence of the output of incandescent

lamps and the photomultipliers on their supply voltages is discussed briefly in Appendix II.

A simple two beam system uses a beam splitter to separate the incoming beam into two paths; a reference path and a sample path, each path ending on a separate detector. Fluctuations in the intensity of the radiation from the source can be allowed for, or the intensity of the source may be adjusted by a feedback loop from the reference beam. The accuracy of this type of system depends on the selection of two detectors with matching spectral sensitivities, the characteristics of which drift equally with time, temperature and voltage. The accuracy is further affected because separate amplifiers and a ratiometer are required. During the short time taken to measure reflection and transmission at each wavelength, large changes in the lamp and detector characteristics would not be expected. Therefore, the two detector arrangement could be used with equipment like the reflectometer.

In a null balance system, the input radiation is shared between the reference and sample paths and then re-combined onto a single detector. The input beam can be time-shared using a mirror chopper and the two signals compared using phase sensitive detection. Alternatively, a beam splitter may be used to separate the beams and then one beam can be modulated to identify it for signal detection. In both arrangements the difference of the signals from the two beams is used to drive an optical attenuator until a null condition is reached. These systems avoid the inaccuracies due to the use of two detectors, separate amplifiers and the ratiometer, but they are limited in accuracy by the electro-mechanical linkage and the resolution and accuracy of the optical attenuator.

The simplest of the multiplexed systems is similar to the null balance system except the demultiplexed reference and sample signals are not used to drive a servo mechanism. The signals are compared in a ratiometer to obtain the ratio of the sample to reference signals. The accuracy with which ratios of electrical signals can be measured with a ratiometer is better than the accuracy of readout of most electro-mechanical linkage systems.

In all of these systems the input radiation can be modulated by an opaque chopper and tuned amplifiers or phase sensitive detection used to improve the signal to noise ratio of the output signal from the photomultiplier.

### 3. REFLECTOMETER FACILITY

The reflectometer facility which has been developed is used to measure the spectral reflection and transmission of flat samples. It consists of three main units: the reflectometer, the electronic detection system of the single detector multiplexed signal type and the monochromator - see figures 1 and 2. The monochromator in use is a Sirospec grating monochromator.

#### 3.1 The reflectometer

The reflectometer is a true normal incidence reflectometer based on the design by Shaw and Blevin(ref.9). The main features of the reflectometer are that it is an absolute instrument working at normal incidence, it is easy to use and reasonably quick when used to measure reflection, transmission, and reflection plus transmission at a single wavelength setting.

##### 3.1.1 Description of the reflectometer

Radiant flux from a Quartz Iodine lamp is focused onto the entrance slit of the monochromator as an  $f/10$  cone by lens  $L_1$  (see figure 2). The light from the exit slit of the monochromator is directed via lens  $L_2$ , spherical mirrors  $M_1$ ,  $M_3$ , and plane mirrors  $M_2$  and  $M_4$  to irradiate the sample,  $S$ , at normal incidence. A clear silica plate  $P_1$  with plane parallel faces is mounted in the beam at an angle  $40^\circ$  to the beam in order to reflect a fraction of the flux reflected from the sample to the photocell,  $C$ .



Lens  $L_2$  and mirrors  $M_1$  and  $M_3$  image the grating at the sample within aperture  $A_1$  and image the exit slit of the monochromator near aperture  $A_2$ . This slit image is about 12 mm high and the grating image is approximately 10 mm square. Laterally separated images of the slit are formed near  $A_2$  due to reflection at both surfaces of the plate  $P_1$ . Only the flux reflected by the inner surface of  $P_1$  is passed by aperture  $A_2$ , the remaining images being vignetted. The sample is mounted on a motor-driven sliding carriage and can be moved in and out of the beam. A second silica plate  $P_2$ , nominally identical with  $P_1$ , is mounted symmetrically behind aperture  $A_1$  so that, for a transparent sample or with the sample removed, it reflects a fraction of the incident beam towards photocell C. Again laterally separated images of the slit are formed near  $A_2$  due to reflection from both surfaces of the plate  $P_2$  and only the flux from the inner surface is passed by  $A_2$ . With proper alignment of the instrument both the transmitted and reflected beams from the sample illuminate the same area of the photocell. An alignment method to achieve this is described in Appendix I. It is possible to measure the flux reflected from the inner surfaces of plates  $P_1$  and  $P_2$  either separately or together by positioning shutters  $SH_1$  and  $SH_2$  appropriately. The shutters are fitted with solenoids and can be electrically or manually operated.

The reference beam is reflected by the three bladed chopper  $C_2$  to spherical mirror  $M_5$  and then to the photocell C.

In addition to the images of the grating and slit in apertures  $A_1$  and  $A_2$  are images of the grating near  $M_1$  and the slit near  $M_2$ . These extra images are useful for controlling the grating image size at the sample and for chopping the beam at 3 Hz. With the sample located in the beam some of the reflected component is transmitted by silica plate  $P_1$ . This beam forms an image of the slit between mirrors  $M_4$  and  $M_3$  and a small plane mirror  $M_6$  is located at this image to remove the unwanted return beam from the main optical path.

### 3.1.2 Measurement of reflection and transmission of a sample

A series of measurements of the flux incident on the photocell for a sequence of positions for the shutters  $SH_1$  and  $SH_2$  and the sample S allows the calculation of normal incidence reflection and transmission to be made. Scattered flux from the sample is lost from the main beams and the measurements cannot be used directly to distinguish between scattering and absorption.

Let  $r_1$  and  $r_2$  be the reflectances of the inner surfaces of silica plates  $P_1$  and  $P_2$  respectively for  $40^\circ$  incidence and the state of polarisation of the incident beam. Let beam 1 and beam 2 be the beams reflected from the inner surfaces of  $P_1$  and  $P_2$  respectively. With unit flux incident on the sample, the flux  $F_t$  in beam 2 is given by:

$$F_t = Tr_2 \quad (1)$$

and the flux  $F_r$  in beam 1 is given by:

$$F_r = Rr_1 \quad (2)$$

where T and R are the fractional transmission and reflection of the sample. With the sample removed from the beam, the flux  $F_i$  in beam 2 is given by:

$$F_i = r_2 \quad (3)$$

The transmission of the sample is determined directly by substituting for  $r_2$  in equation (1)

$$T = \frac{F_t}{F_i}$$

The determination of the reflection from the sample is affected by the relative values of  $r_1$  and  $r_2$  as follows:

$$\frac{F_r}{F_i} = \frac{Rr_1}{r_2}$$

i.e.

$$R = \frac{F_r}{F_i} \cdot \frac{r_2}{r_1} \quad (4)$$

If  $r_2 = r_1$  then  $R = \frac{F_r}{F_i}$  directly. If  $r_2 \neq r_1$  consider the silica plates to be interchanged. Then:

$$F'_r = Rr_2$$

and

$$F'_i = r_1$$

giving

$$\frac{F'_r}{F'_i} = \frac{Rr_2}{r_1}$$

i.e.

$$R = \frac{F'_r}{F'_i} \cdot \frac{r_1}{r_2} \quad (5)$$

From equations (4) and (5):

$$R^2 = \frac{F'_r}{F'_i} \cdot \frac{F_r}{F_i}$$

and

$$\left(\frac{r_2}{r_1}\right)^2 = \frac{F_i}{F_r} \cdot \frac{F'_r}{F'_i}$$

R can then be calculated from equation (4) above:

$$R = \frac{F_r}{F_i} \cdot \frac{r_2}{r_1}$$

when  $\frac{r_2}{r_1}$  is a recorded measured function of wavelength.

The losses in the sample can be calculated from:

$$1 = R + T + L$$

where L is the loss of flux due to scattering and absorption.

If a dielectric substrate is coated on one side then from measurements of reflection and transmission the reflectance and transmittance of the coated surface may be calculated (see Section 4.4.2).

### 3.2 Electronic instrumentation

The electronic system now in use with the reflectometer has been built because of difficulties experienced earlier with simpler systems. Some minor constraints were imposed on the selection of a detection system by the physical construction of the reflectometer unit.

#### 3.2.1 Detection system for the reflectometer

The physical arrangement of the reflectometer is suitable for a single beam system, a two detector system or a single detector multiplexed system. Single beam a.c. and d.c. systems have been used with the reflectometer but variations in signal, not traceable to the power supplies, limited the speed and accuracy of measurement. The choice of a single detector multiplexed arrangement was considered preferable to the use of two detectors.

The signal detection system used for the reflectometer is a multiplexed system with feedback from the reference beam to control the gain of the signal amplifier. The input beam is chopped at 1000 Hz and it is time-shared between the sample and reference beams at a frequency of 3 Hz. The beams are incident on a single photomultiplier.

Ratio comparisons of signals are made using analogue technology. The output is available in both analogue and digital form. The output from the ratiometer is a measurement of the ratio between the sample beam and the reference beam.

The overall system can be used in three modes:

- (1) signal detection using a 1000 Hz tuned amplifier;
- (2) phase sensitive detection at 1000 Hz;
- (3) the full ratio mode with the reference beam and dual frequency modulation.

#### 3.2.2 Ratiometer

The ratiometer has been designed to accept signals over a wide range of levels and to cope with widely varying ratios of reference to sample signals. A simplified block diagram is given in figure 3.

##### (a) Input stage

The signal from the photomultiplier goes through a voltage follower, to a variable gain pre-amplifier and a fixed gain 1000 Hz tuned amplifier and detector. The output of the 1000 Hz detector is displayed on a meter. In operation, the gain of the pre-amplifier is adjusted to give nearly full scale deflection of the meter when only the reference beam modulated at 3 Hz is incident



on the photomultiplier. This ensures that the succeeding stages of the instrument will not be overloaded. This stage can be used as an independent measuring system if the 3 Hz chopper is not operating. The output of the 1000 Hz detector can be switched to give analogue and digital data.

(b) Automatic gain control

The a.g.c. amplifier is controlled by the 3 Hz demultiplexed signal from the reference beam. The 1000 Hz reference beam signal is converted to a d.c. signal through a phase sensitive detector (PSD). The output of the PSD goes to the summing integrator for comparison with a stable -1 V reference. The gain of the a.g.c. amplifier is adjusted through the feedback loop to maintain the output of the reference channel at +1 V. Variations in the reference beam signal from the photomultiplier due to changes in photomultiplier gain or lamp intensity are compensated automatically. The response time of the a.g.c. system is determined by the time constants associated with the PSD and the summing integrator.

The a.g.c. system fails to function when the input signal from the reference channel is too low. The control voltage is displayed on a front panel meter indicating if the a.g.c. feedback loop is operating satisfactorily or not.

(c) Ratio output stage

The 1000 Hz signal from the sample beam, modulated at 3 Hz by the low frequency chopper, shares the same amplifiers as the reference beam until it is demultiplexed. It is then converted to a d.c. signal in a separate phase sensitive detector before being amplified through a variable gain d.c. amplifier. The signal from this amplifier can be switched to give both analogue and digital output data.

The variable gain d.c. amplifier is used to adjust the signal level of the output to a suitable level for input to a digital or analogue panel meter. It is convenient to adjust the gain of the amplifier to make the digital panel meter (DPM) read 1.000 when the sample is out of the sampling beam. The external clock controlling the reading cycle of the digital panel meter is derived from the 3 Hz chopper so that the DPM is synchronised with the sampling cycle. This reduces the effect of any 3 Hz ripple on the input to the DPM.

Fluctuations in the reference beam signal due to variations in the photomultiplier or lamp circuits cause changes in the gain of the a.g.c. amplifier. For simplicity in the following analysis only changes in the intensity of the lamp are considered as a single detector system cannot distinguish between changes in the lamp or detector.

The reference beam and the sample beam signals at the input to the appropriate phase sensitive detectors are  $S_B = g_a \beta I$  and

$S_A = g_a a I$  respectively where

$I$  = intensity of incident radiation,

$a$  = transmission factor of the sample beam,

$\beta$  = transmission factor of the reference beam,

$g_a$  = gain of a.g.c. amplifier

The feedback loop keeps  $S_B$  constant when  $I$  varies by adjusting  $g_a$ . Over the period of averaging or smoothing, the product  $g_a I$  for the reference and sample beams remains constant giving:

$$S_A = S_B \frac{a}{\beta}$$

It then follows that the d.c. output signal of the ratiometer

$$S'_A = k_1 \frac{a}{\beta}$$

where  $k_1$  includes  $S_B$  and the effects of conversion to d.c. through the PSD and subsequent amplification. For measurements of transmission and reflection of a sample at one wavelength, the ratios of  $a/\beta$  for the 'sample out' and 'sample in' conditions must be made without changing the gain of the output stage between readings. Then the transmission or reflection of the sample is obtained from the ratio of the relevant readings.

(d) Low frequency detection stage

The signal from the a.g.c. amplifier contains both 1000 Hz and 3 Hz modulation. The averaged value of the high frequency component of the signal is proportional to  $g_a I(\beta + a)$  or  $g_a I\beta (\frac{a}{\beta} + 1)$ . The averaged value of the low frequency component is  $g_a I(\beta - a)$  or  $g_a I\beta (1 - \frac{a}{\beta})$ . The feedback loop holds  $g_a I\beta$  constant and therefore, after synchronous detection, the signals available are proportional to  $(1 + \frac{a}{\beta})$  and  $(1 - \frac{a}{\beta})$ .

The low frequency detection stage, consisting of a 3 Hz detector amplifier, PSD and d.c. amplifier, provides an output of the  $(1 - \frac{a}{\beta})$  function.

(e) Choice of modulation frequencies

The high and low chopping frequencies of 1000 Hz and 3 Hz were chosen for the following reasons:

- (1) The large difference between the two frequencies avoids having to synchronise high and low frequency choppers to minimise low frequency beat problems.
- (2) A reasonably long sampling time is provided but the sampling rate is still kept high enough to permit the system to respond to low frequency variations.

#### 4. MEASUREMENTS

The reflectometer facility has been used to measure several different types of samples. Measurements that have been made are:

- (a) reflectance of laser mirrors, protected and unprotected mirror surfaces and metal mirrors prepared in different ways;

- (b) reflection and transmission of bloomed surfaces, Fabry-Pérot filters and thin films of single materials;
- (c) reflectance of samples of gold, germanium, molybdenum and nichrome; the optical constants of which had been determined using ellipsometry(ref.10) and the reflectances calculated from them for comparison with actual measurement.

At the present stage of development of the measurement facility the losses due to scattering and absorption are indistinguishable from one another. The effect of surface and bulk scattering of light from thin films deposited on well polished substrates can be assumed to be less than half a percent of the incident radiation for most materials used in thin film filters. With proper care this assumption does not lead to the introduction of significant errors in the analysis of the data taken from the reflectometer.

#### 4.1 Optical constants of zirconium dioxide

Transmission and reflection measurements of films of single materials can be used directly to assess the optical quality of the films. Processing of these measurements is needed to determine the optical constants of the film material and the thickness of the film. The Fresnel equations, rearranged as suggested by Tomlin(ref.6,7), have been used to calculate the optical constants and thickness of films of zirconium dioxide. The optical constants of zirconium dioxide are listed in Table 1. It is difficult to determine absorption coefficients less than 0.001 with this method as the effect of very small absorption coefficients is quite small in thin films of single materials. The optical constants calculated for the zirconium dioxide films have been used to recalculate the reflection and transmission of films of the same optical thickness as the sample films and also for films of different thickness for which transmission measurements were available. The comparisons between calculated and measured spectral transmission curves were consistently good: much better than that achieved with published data and previous data available at W.R.E.

TABLE 1. OPTICAL CONSTANTS OF ZIRCONIUM DIOXIDE

Wavelength (nm)	(a)		(b)		n	k(g)
	n	k	n	k		
350	2.000	0	2.052	0		
400	1.962	0	2.009	0		
500	1.935	0	1.968	0	2.05(d)	-
500	-	-	-	-	2.23(e)	-
546	1.926	0	1.961	0	1.97(f)	-
550	1.925	0	1.960	0	2.10(c)	-
600	1.916	0	1.957	0		
630	1.911	0	1.955	0	1.95(f)	-
800	1.899	0	1.950	0		
1000	1.893	0	1.946	0		

where

- (a) Optical constants calculated from reflectometer data
- (b) Khawaja(ref.14)
- (c) Cox, Hass, Ramsey(ref.16)
- (d) Stetter et.al.(ref.17)
- (e) Hiraga et.al.(ref.18)
- (f) Clapham(ref.19)
- (g) k values not given



TABLE 2. REFLECTANCE OF ALUMINIUM MIRRORS

Wavelength (nm)	Aluminium age (days)						Aluminium + schott 8329 glass ( $3/8 \lambda @ 500 \text{ nm}$ )			
	1	2	6	21	80	>3 years*	2	6	21	80
350		0.969	0.928	0.925	0.916	0.786	0.916	0.917	0.917	0.916
400		0.925	0.924	0.924	0.919	0.824	0.929	0.928	0.928	0.926
450		0.922	0.920	0.920	0.918	0.842	0.918	-	0.920	0.918
500	0.919	0.917	0.918	0.917	0.914	0.846	0.906	0.907	0.908	0.908
550	0.914	0.914	0.915	0.915	0.913	0.848	0.883	-	0.885	0.882
600	0.901	0.905	0.909	0.908	0.904	0.848	0.849	0.850	0.850	0.850
650		0.906	0.907	0.906	0.896	0.847	0.821	-	0.823	0.822
700		0.883	0.888	0.889	0.844	0.841	0.789	0.790	0.790	0.789

\* The mirror measured was known to be at least 3 years old



## 4.2 Spectral reflectance of aluminium

Thin opaque films of aluminium coated on optical components are used as mirrors. The reflectivity of an aluminium film decreases as the film ages and it is desirable in some applications to overcoat the metal surface to inhibit the ageing process. Typically, a protective coating of silicon dioxide or glass is evaporated onto the freshly deposited aluminium. This protective coating can lower the spectral reflectivity of the mirror. Reflectance measurements are being made of protected and unprotected films as they age so that the amount of change to be expected with time is known and any long term advantages to be gained from protective coatings can be assessed. Preliminary measurements are reported in Table 2.

## 4.3 Laser mirror materials

Laser mirrors are made of dielectric materials in order to be highly reflecting and non-absorbing. With the high radiation power in many laser beams, slight absorption in the mirror causes heating in the surface layers and this can destroy the mirror. The presence of absorption in thin films depends on several factors. Assuming that the bulk materials are non-absorbing and of high quality, absorption can still be introduced by the deposition process. The rate of deposition, the pressure in the vacuum chamber, the temperature of the substrate and the introduction of impurities or defects during deposition all influence the amount of absorption in the final product. The direct measurement of very low absorption is a difficult task but it is an important task when evaluating methods of manufacture and materials used in the making of laser mirrors.

Some thin film systems are very sensitive to small values of absorption coefficient  $k$ . One of these is the Fabry-Pérot filter which exhibits a relatively large absorption peak for quite small values of  $k$ . For example zinc sulphide and cryolite are two materials considered to be non-absorbing at 766 nm. A simple all-dielectric Fabry-Pérot filter made from these materials to have a peak transmission at 766 nm also had a peak absorption value of 20% at this wavelength. Calculations show that an absorption coefficient  $k$  of less than 0.001 in the spacer layer can account for the presence of this absorption peak. It is shown that absorption coefficients of less than 0.001 can be determined from reflection and transmission measurements made of Fabry-Pérot filters(ref.11). These results can then be used in an objective evaluation of materials and methods used in the manufacture of laser mirrors.

## 4.4 Calculation of parameters of substrates and thin film systems from simple measurements of transmission and reflection

For many applications of thin film measurements the reflectance and transmittance of the coated surface is required. These parameters can be found from simple inter-reflection theory.

### 4.4.1 Dielectric substrates - refractive index

Consider a flat dielectric substrate of sufficient thickness to disregard interference effects - see figure 4. The intensity reflection and transmission coefficients  $R_1$  and  $T_1$  for a single surface of an uncoated substrate are:

$$R_1 = \left( \frac{n_0 - n_1}{n_0 + n_1} \right)^2 \quad (6)$$

$$T_1 = 1 - R_1 = \frac{4 n_0 n_1}{(n_0 + n_1)^2} \quad (7)$$

where  $n_0$  is the refractive index of the substrate and  $n_1$  is the refractive index of air. Taking into account multiple reflections within the substrate the total reflection is given by the expression

$$\begin{aligned} R_T &= R_1 + T_1^2 R_1 + T_1^2 R_1 (R_1^2) + T_1^2 R_1 (R_1^4) + \dots \\ &= R_1 + \frac{T_1^2 R_1}{1 - R_1^2} \\ &= R_1 + \frac{(1 - R_1)^2}{1 - R_1^2} R_1 \\ &= \frac{2R_1}{1 + R_1} \end{aligned}$$

i.e.

$$R_1 = \frac{R_T}{2 - R_T} \quad (8)$$

and the total transmission by

$$\begin{aligned} T_T &= T_1^2 + T_1^2 R_1^2 + T_1^2 R_1^4 + \dots \\ &= \frac{T_1^2}{1 - R_1^2} \\ &= \frac{(1 - R_1)^2}{1 - R_1^2} \\ &= \frac{1 - R_1}{1 + R_1} \end{aligned}$$

i.e.

$$R_1 = \frac{1 - T_T}{1 + T_T} \quad (9)$$

Therefore, provided there is negligible loss, the refractive index of the substrate can be determined from measurements of total reflection or transmission using equations (8) or (9) with equation (6) rearranged as

$$n_0 = n_1 \frac{1 + \sqrt{R_1}}{1 - \sqrt{R_1}}$$

Using equations (6) and (9) it can also be shown that

$$T_T = \frac{2n_0 n_1}{n_0^2 + n_1^2} = \frac{2n_0}{n_0^2 + 1} \quad \text{when } n_1 = 1 \text{ for air.}$$

A table of  $n_0$  and  $T_T$  for this function provides a convenient method for determining the refractive index of the substrate from a measurement of transmission.

#### 4.4.2 Dielectric substrate coated with dielectric thin film

Consider a plane dielectric substrate coated with a dielectric thin film system. The intensity reflection coefficient of the coated surface is to be determined from transmission or reflection measurements. Referring to figure 5 the total reflection,  $R_T$ , is given by the expression:

$$\begin{aligned} R_T &= R_2 + T_2^2 R_1 + T_2^2 R_1 (R_1 R_2) + T_2^2 R_1 (R_1 R_2)^2 + \dots \\ &= R_2 + \frac{T_2^2 R_1}{1 - R_1 R_2} \end{aligned}$$

where  $R_2$  and  $T_2$  are the intensity reflection and transmission coefficients of the coated surface.

Now

$$T_2 = 1 - R_2$$

therefore

$$R_T = R_2 + \frac{R_1 - 2R_1 R_2 + R_1 R_2^2}{1 - R_1 R_2}$$

and

$$R_2 = \frac{R_T - R_1}{1 - R_1 (2 - R_T)} \quad (10)$$

It can be seen that if  $R_1$  and  $R_T$  are small then the approximation

$$R_2 = \frac{R_T - R_1}{1 - 2R_1} \text{ could be used.}$$

The total transmission,  $T_T$ , is given by

$$T_T = 1 - R_T$$

substitution for  $R_T$  in equation (10) gives

$$R_2 = \frac{1 - T_T - R_1}{1 - R_1 (1 + T_T)} \quad (11)$$

Therefore the reflectance of the surface coated with a dielectric film can be calculated from measurements of total reflection or transmission using equations (10) or (11).

## 5. DATA ACQUISITION

The arrangement of this facility gives relatively rapid results at a single wavelength. However, the operation becomes very tedious when a spectral scan is required because the tasks involved are repetitious. The recording and initial processing of data to determine the reflection and transmission of the sample are time consuming and boring, brought about by the very basic method of measurement used. A data link has recently been established between the reflectometer facility and an IBM370/168 computer(ref.12). This data link operates as a time-sharing facility capable of transmitting 25 16-bit words/second. The data rate from the reflectometer is well below this at 4 16-bit words/second. The use of the data link is an optional facility and has several advantages. The data can be processed immediately for values of reflection and transmission and the answers displayed at the reflectometer as well as being stored in the computer for further processing. This further processing is directed from a time-sharing computer terminal and can include the presentation of the results in graphical or tabulated forms or both. In general, it is not necessary to use the computer facility when only a small number of measurements are required.

## 6. CONCLUSION

The reflectometer facility has been set up to measure the spectral transmission and reflection of flat substrates in the spectral wavelength range of 0.3 to 1  $\mu\text{m}$ . Reflection and transmission measurements can be made with an accuracy of 0.2%. Data from the reflectometer has been used in the assessment and investigation of thin film filters and materials. The optical constants of dielectric materials in thin film form have been calculated and the results for zirconium dioxide are tabulated in Table 1. Measurements of the reflection and transmission of Fabry-Pérot filters enables the small absorption coefficients in low loss materials to be determined.

The reflectometer facility is a valuable tool in the assessment of thin films. Established methods of data processing are currently being used and the development of improved data processing methods is proceeding.



## NOTATION

A	tabulated constants
B, B <sub>2</sub> B <sub>3</sub> , B <sub>4</sub> B <sub>5</sub> , B <sub>6</sub>	tabulated constants
F, F <sub>0</sub>	luminous output of incandescent lamp at voltage V or V <sub>0</sub>
F <sub>i</sub>	flux reflected from silica plate P <sub>2</sub>
F <sub>r</sub>	flux reflected from sample and silica plate P <sub>1</sub>
F <sub>t</sub>	flux transmitted through sample and reflected from silica plate P <sub>2</sub>
F' <sub>i</sub>	flux reflected from silica plate P <sub>1</sub> in changed position
F' <sub>r</sub>	flux reflected from sample and silica plate P <sub>2</sub> in changed position
G	gain of photomultiplier
I	intensity of incident radiation
L	fraction of flux lost from beams 1 and 2 due to scattering and absorption
L, L <sub>0</sub>	life of incandescent lamp at voltage V or V <sub>0</sub>
N <sub>λ</sub>	spectral radiance of black body in terms of wavelength
P, P <sub>0</sub>	power output (watts) of incandescent lamp at voltage V or V <sub>0</sub>
R	fraction of flux reflected from sample
R <sub>1</sub> , R <sub>2</sub>	intensity reflection coefficients
R <sub>T</sub>	fraction of flux reflected from sample
S <sub>A</sub>	reference beam signal
S <sub>B</sub>	sample beam signal
S' <sub>A</sub>	d.c. output signal of ratiometer
T	fraction of flux transmitted through sample
T	absolute temperature of black body
T, T <sub>0</sub>	absolute temperature of filament in incandescent lamp at voltage V or V <sub>0</sub>
T <sub>1</sub> , T <sub>2</sub>	intensity transmission coefficients

$T_T$	fraction of flux transmitted through sample
$V, V_0$	voltage of incandescent lamp suppl.
$V_s$	interdynode voltage of photomultiplier
$a$	empirical 'constant' for dynode of photomultiplier
$c$	velocity of light
$c_1$	first radiation constant
$c_2$	second radiation constant
$e$	base of naperian logarithms = 2.718 ...
$f$	electron collection efficiency between the photocathode and first dynode of photomultiplier
$g$	electron collection efficiency between dynodes of photomultiplier
$g_a$	gain of a.g.c. amplifier
$h$	Planck's constant
$i, i_0$	current of incandescent lamp at voltage $V$ or $V_0$
$k$	Boltzmann's constant
$k$	absorption coefficient $\tilde{n} = n - ik$
$k_1$	a.c. to d.c. gain of PSD and variable gain amplifier
$m$	number of dynodes in photomultiplier
$n$	real part of complex refractive index
$n_0$	refractive index of substrate
$n_1$	refractive index of medium
$\tilde{n}$	complex refractive index $\tilde{n} = n - ik$
$r_1, r_2$	fraction of flux reflected from inner surfaces of silica plates $P_1, P_2$
$x$	empirical 'constant' for dynode of photomultiplier
$\alpha$	transmission factor of the sample beam
$\beta$	transmission factor of the reference beam
$\lambda$	wavelength

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## APPENDIX I

## ALIGNMENT OF THE REFLECTOMETER

The successful operation of the reflectometer requires that the two silica plates ( $P_1$  and  $P_2$  in figure 2) be used at the same angle of incidence and that the diffuser in front of the detector be illuminated at equal angles by the light reflected from each. The main constructional features of the reflectometer are:

- (1) The silica plates have a three point spring-loaded mounting in the vertical plane.
- (2) The silica plate mounts are kinematically mounted on turntables.
- (3) The guide rail for the sample mount has its pivot point on the line joining the centres of the turntables.
- (4) The sample carrier is adjustable in the vertical plane.
- (5) The reflectometer base is kinematically mounted.

The alignment of the reflectometer was carried out in the following manner: (refer to figure 6).

- (1) The turntables were used to define the three reference planes of the reflectometer. For convenience the alignment was carried out on a surface table with the turntable surfaces parallel to the surface table.
- (2) The silica plates were adjusted in their mounts so that the inside reflecting faces were located over the axes of the turntables when mounted. An auto-collimator was set in position 1 (figure 6), its axis made normal to the axis of the 'R' turntable with the aid of a reference mirror mounted perpendicular to its own base. One silica plate in its mount was placed on the turntable and the foot pad of the turntable kinematic mount adjusted to make the silica plate vertical. The turntable position was adjusted to make the silica plate normal to the axis of the auto-collimator. The second plate, in its mount, was then placed on the same turntable and the adjusting screws of the mount adjusted to make this plate normal to the auto-collimator. The plates, in their mounts, were then interchangeable.
- (3) An alignment telescope was set in position 3 (figure 6) with its axis normal to the axes of the turntables and intersecting them. A mirror with ruled cross lines was mounted centrally on the sample holder and the limit screw of the sample holder adjusted until the centre of the sample holder was in line with the axes of the turntables. Using the alignment telescope as an auto-collimator, the adjusting screws on the sample carrier and the sample carrier guide rail assembly were adjusted to make the sample mirror normal to the optical axis of the telescope.
- (4) The silica plates in their mounts were mounted on the turntables and the alignment telescope shifted to accommodate the shift of the image of the mirror cross lines caused by refraction through the quartz. A cross line graticule was mounted centrally in the aperture  $A_2$ . The 'R' turntable was rotated until the image of the graticule was centred in the telescope. The graticule was checked and adjusted for height before sliding the sample carrier out of the sampling beam. With the sample mirror out of position, the 'T' turntable was rotated and the foot pad of the kinematic mount adjusted until the graticule image was centred in the telescope.
- (5) The alignment procedure to this point ensured that:

- (a) the basic reference planes had been established;
  - (b) the sample could be positioned centrally between the silica plates and normal to the line between the turntables;
  - (c) the silica plates in their mounts were interchangeable between the 'R' and 'T' positions without any detectable difference in alignment;
- and (d) the reflected and transmitted beams from the sample were incident on the same area of the diffuser.

At this stage the silica plates were not necessarily set at equal angles of incidence. The auto-collimator was set in position 2 with the optical axis of the collimator parallel to the axis of the telescope. Using a large flat test mirror as a transfer reference, the telescope was moved step by step to position 4. An aluminised right angle prism was then located in the plane of the diffuser with one face normal and the other face parallel to the optic axis of the telescope. The alignment telescope was transferred to position 3 again using the mirror as the transfer reference. The telescope alignment was checked against the auto-collimator and the sample mirror to confirm that the transfer had been successful. It can be seen from figure 7 that unless the silica plates are inclined equally to the input beam the auto-collimator graticule image as seen through the telescope will appear to be shifted laterally when the sample mirror is moved in and out of the beam. In figure 7 the front face of each silica plate is drawn in its correct position and the ideal ray path represented by continuous lines. The dashed lines represent the ray path when the silica plates are rotated clockwise through a small angle and show the resultant angular shift of the image resulting from incorrect alignment. The rotational positions of the turntables were adjusted, sharing the adjustment equally between the turntables, to eliminate the shift of the image observed when the sample mirror was moved in and out of the beam. The graticule was then placed in the plane of the diffuser and rechecked with the telescope. No further adjustment was required.



## APPENDIX II

## STABILITY OF LAMP AND PHOTOMULTIPLIER IN TERMS OF VOLTAGE

For the visible and near infra-red region of the spectrum, photomultipliers are a common detector. Several parameters influence the operating conditions of a photomultiplier. Johnson noise, shot noise, and statistical variations in the intensity of the incident radiation are unavoidable problems, the effect of which can be minimised by good circuit design and suitably high flux levels. Dark current limits the lowest intensity of radiation that can be detected and its major source is thermionic emission from the photocathode.

## II.1 Effect of EHT variations on photomultipliers

The selection of the EHT supply and the determination of the stability actually required in the supply is dependent on the dynode characteristics of the photomultiplier to be used.

Empirical equations to relate the interdynode voltage and the secondary emission coefficient of the dynodes as well as typical values are given by E.M.I. Electronics Ltd(ref.13). The gain,  $G$ , of the photomultiplier is related to the interdynode voltage through an empirical relationship of the form:

$$G = f g^m a^m (V_s)^{xm}$$

where:  $f$  is the electron collection efficiency between the photocathode and the first dynode;

$g$  is the electron collection efficiency between dynodes;

$m$  is the number of dynodes;

$a$  and  $x$  are constants dependent on the dynode material;

and  $V_s$  is the interdynode voltage.

Differentiation gives:

$$\frac{dG}{G} = x m \frac{dV_s}{V_s}$$

For the photomultipliers used with the reflectometer the product  $xm$  is about 10.

## II.2 Effect of lamp supply on spectral radiance

The National Bureau of Standards relate the various parameters of incandescent tungsten lamps to the supply voltage of the lamp using the following empirical formulae(ref.14).

$$\log(F/F_0) = A_2 [\log(V/V_0)]^2 + B_2 \log(V/V_0)$$

$$\log(P/P_0) = A_3 [\log(V/V_0)]^2 + B_3 \log(V/V_0)$$

$$\log(i/i_0) = A_4 [\log(V/V_0)]^2 + B_4 \log(V/V_0)$$

$$\log(T/T_0) = A_6 [\log(V/V_0)]^2 + B_6 \log(V/V_0)$$

where  $F, F_0$  = luminous output at voltage  $V$  or  $V_0$

$P, P_0$  = power (watts) at voltage  $V$  or  $V_0$

$i, i_0$  = lamp current (amps) at voltage  $V$  or  $V_0$

$T, T_0$  = absolute temperature of filament at voltage  $V$  or  $V_0$

$A, B$  = tabulated constants

Where less accuracy can be tolerated, simpler empirical relationships can be written:

$$F/F_0 = (V/V_0)^{B_2} \quad B_2 = 3.513$$

$$P/P_0 = (V/V_0)^{B_3} \quad B_3 = 1.5805$$

$$i/i_0 = (V/V_0)^{B_4} \quad B_4 = 0.5805$$

$$T/T_0 = (V/V_0)^{B_6} \quad B_6 = 0.350$$

$$L/L_0 = (V/V_0)^{-B_5} \quad B_5 = 13.5$$

where  $L$  and  $L_0$  = life of lamp at voltage  $V$  or  $V_0$ .

The values of  $B$  are for gas filled lamps of 40 to 50 W.

The parameter most relevant for spectral measurements is the spectral radiance of the filament. The spectral radiance is related to the actual temperature of the radiating filament. While the emissivity of tungsten is a function of wavelength and must be taken into account for radiometric measurements, it will be omitted from the calculations to establish the approximate dependence of the spectral radiance of a tungsten lamp on the supply voltage.

The spectral radiance of a black body is given by:

$$N_\lambda = \frac{2\pi hc^2}{\lambda^5 [\exp(hc/k\lambda T) - 1]} \quad (\text{planck radiation law})$$

$$= \frac{c_1}{\lambda^5 [\exp(c_2/\lambda T) - 1]}$$

where  $N_\lambda$  = spectral radiance in terms of wavelength

$h$  = Planck's constant

$c$  = velocity of light

$k$  = Boltzmann's constant

$T$  = absolute temperature of black body

$c_1$  = first radiation constant

$$= 2\pi hc^2 = 3.741 \times 10^{-16} \text{ mK}$$

$c_2$  = second radiation constant

$$= hc/k = 1.439 \times 10^{-2} \text{ mK}$$

When  $T$  is small in relation to  $c_2$  then  $\exp c_2/\lambda T$  is large compared to 1 and the  $(-1)$  term in the expression for  $N_\lambda$  can be neglected.  
i.e.

$$N_\lambda \approx \frac{c_1}{\lambda^5} \cdot e^{-c_2/\lambda T} \quad (\text{Wien radiation law})$$

differentiation gives:

$$\frac{dN_\lambda}{N_\lambda} = \frac{c_2}{\lambda T} \cdot \frac{dT}{T}$$

Differentiating

$$\frac{T}{T_0} = \left(\frac{V}{V_0}\right)^{B_6}$$

gives:

$$\frac{dT}{T} = B_6 \cdot \frac{dV}{V}$$

i.e.

$$\frac{dN_\lambda}{N_\lambda} = \frac{c_2}{\lambda T} \cdot B_6 \cdot \frac{dV}{V}$$

i.e. the variation of spectral radiance with lamp voltage is inversely proportional to wavelength. For a filament temperature of 2854K and an emissivity of 1 then:

$$\begin{aligned} \frac{dN_\lambda}{N_\lambda} &= \frac{1.439 \times 10^{-2} \times 0.35}{2.854 \times 10^3} \cdot \frac{dV}{V} \cdot \frac{1}{\lambda} \\ &= \frac{1.76 \times 10^{-6}}{\lambda} \cdot \frac{dV}{V} \end{aligned}$$

Thus for  $\lambda = 300 \text{ nm}$ ;  $\frac{dN_\lambda}{N_\lambda} = 5.8 \frac{dV}{V}$

$\lambda = 600 \text{ nm}$ ;  $\frac{dN_\lambda}{N_\lambda} = 2.9 \frac{dV}{V}$

$\lambda = 900 \text{ nm}$ ;  $\frac{dN_\lambda}{N_\lambda} = 2.0 \frac{dV}{V}$

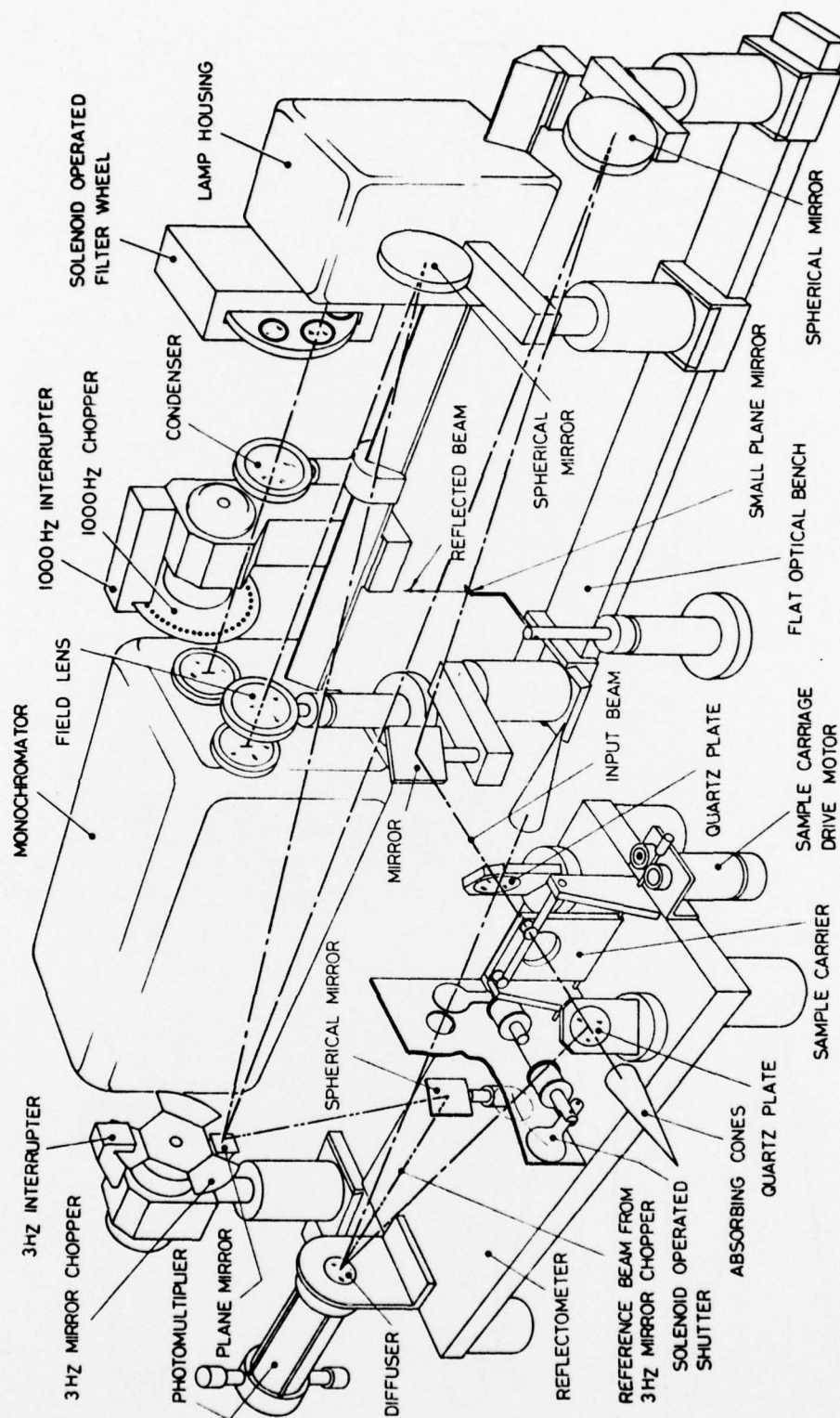


Figure 1. Normal incidence reflectometer facility



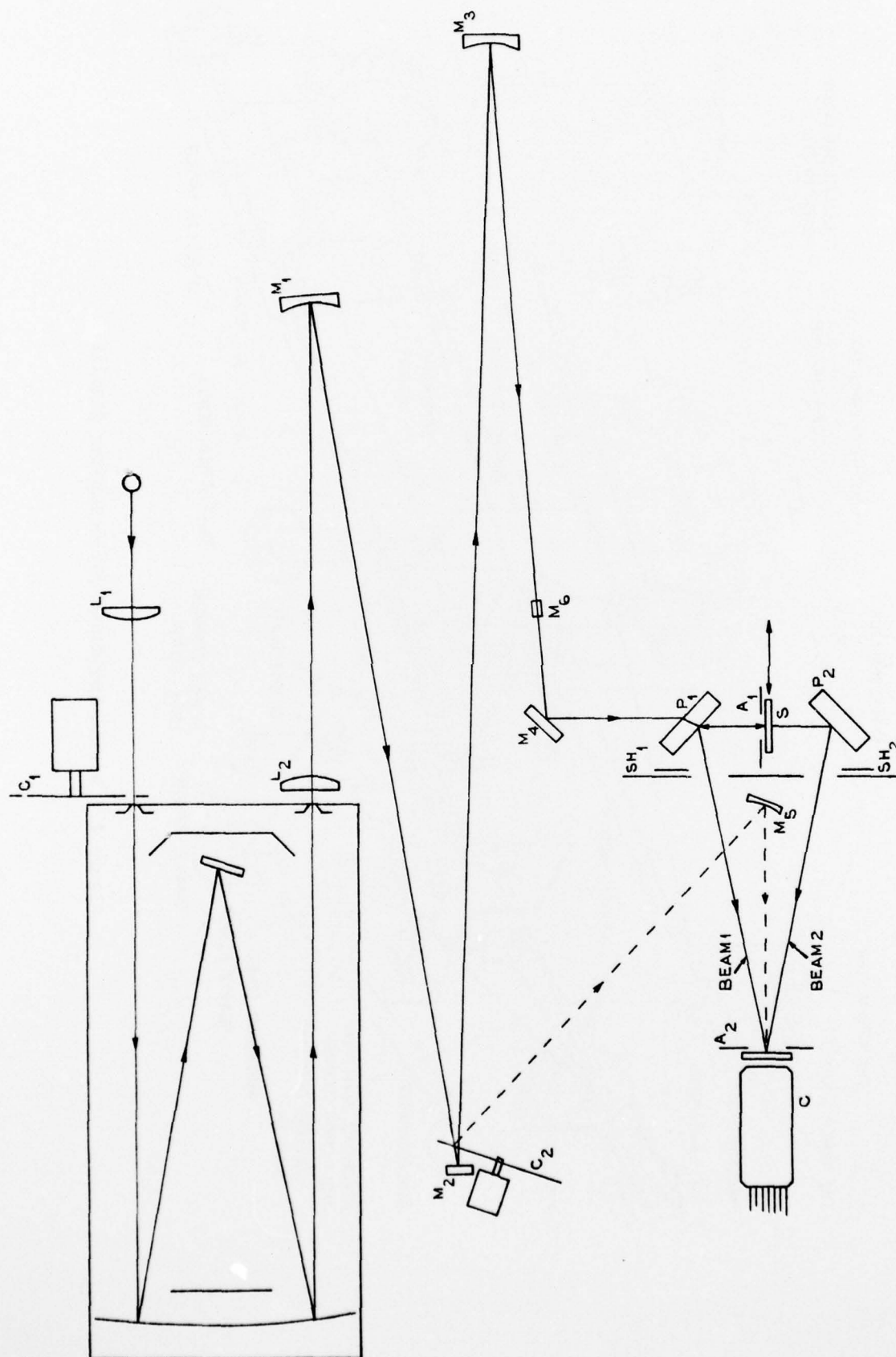


Figure 2. Schematic layout of the reflectometer

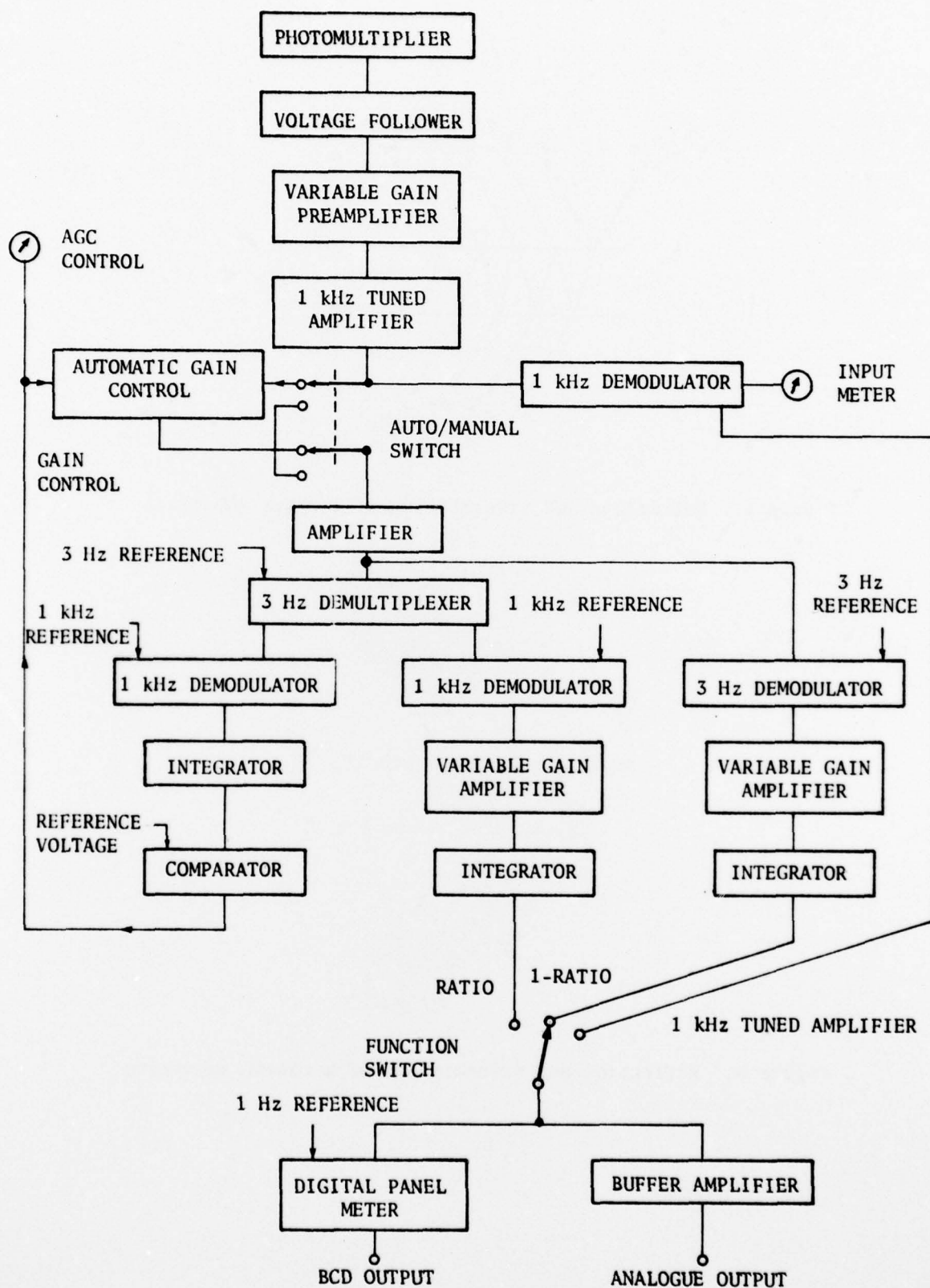


Figure 3. Simplified block diagram of the ratiometer

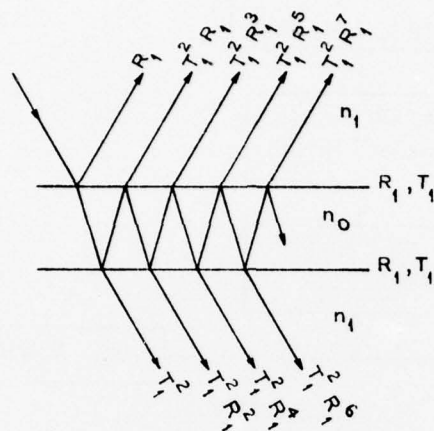


Figure 4. Reflection and transmission of a thick substrate

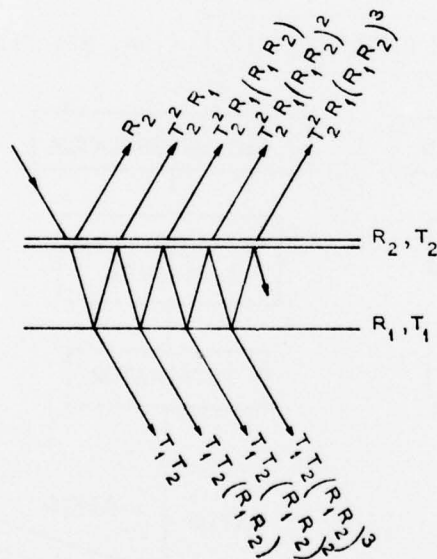


Figure 5. Reflection and transmission of a coated substrate

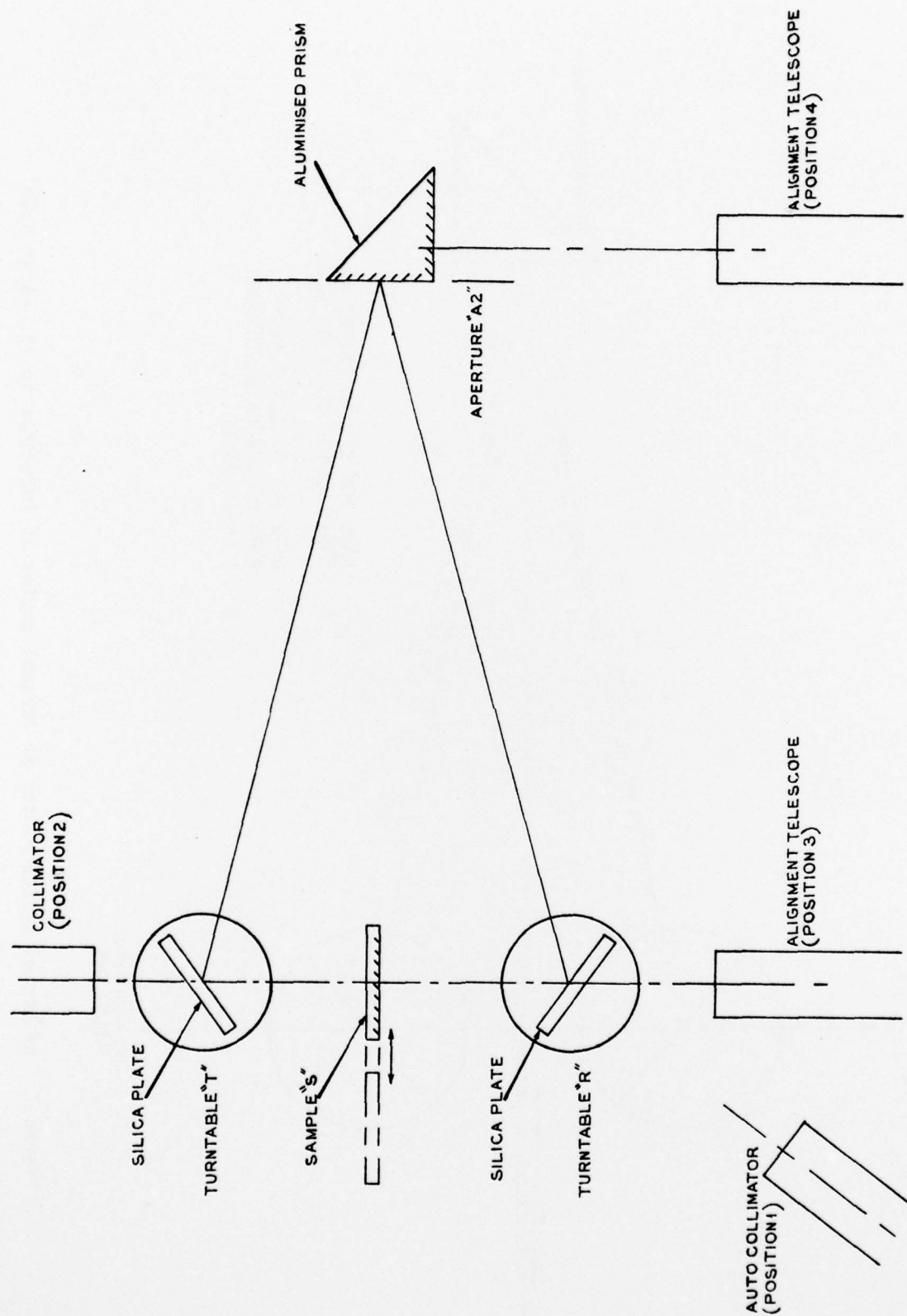


Figure 6. Alignment of absolute reflectometer



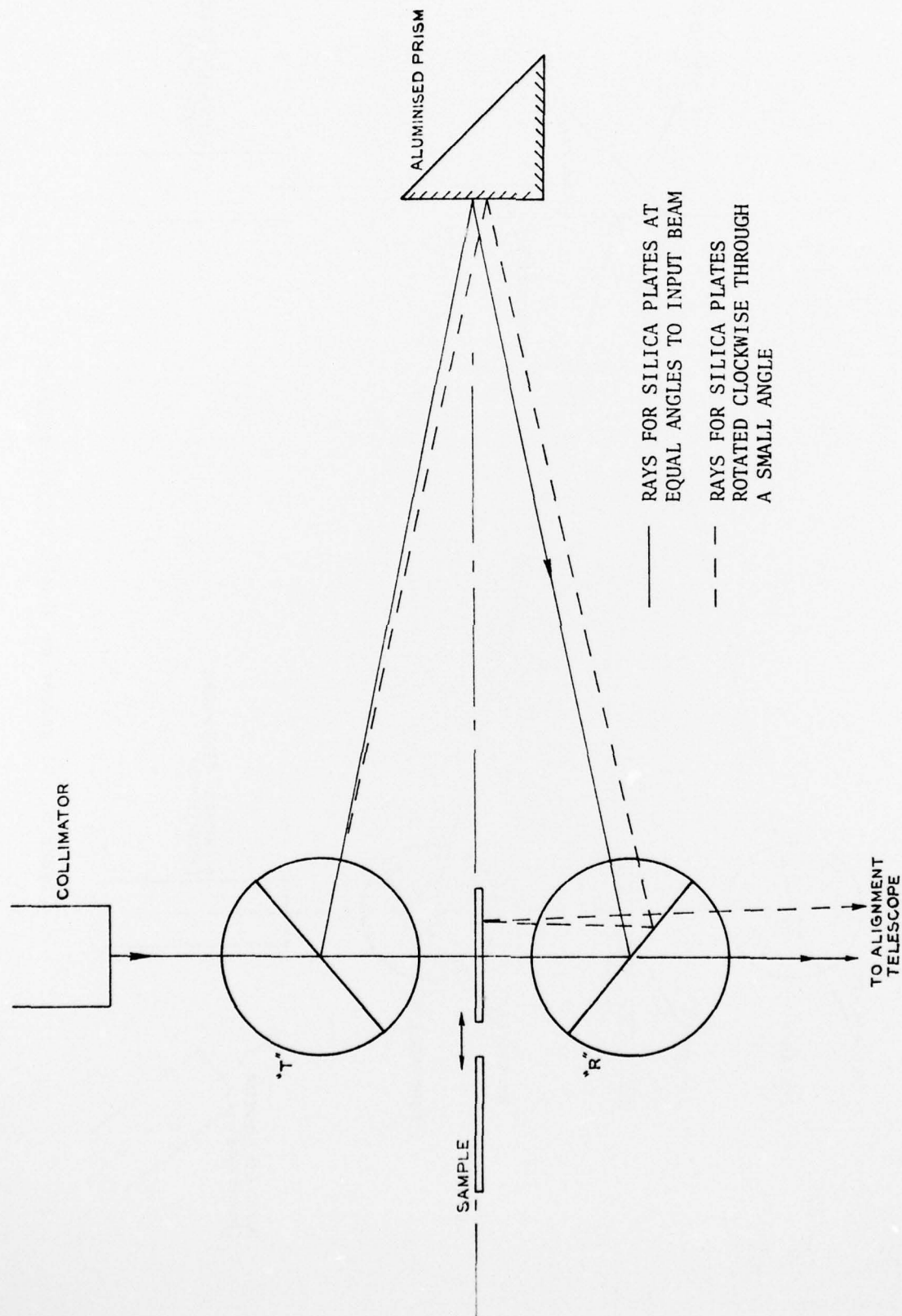


Figure 7. Effect of silica plates at unequal angles of incidence to the input beam

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