SOME OPTICAL PROPERTIES
OF THIN FILMS OF SELENIUM

by

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ABSTRACT

A method for the preparation of thin films of selenium by means of vacuum evaporation is described. Films deposited on thin glass and quartz plates were used in the spectrographic observation of absorption in the visible and ultraviolet regions of the spectrum. Films deposited on the compensating plates of the Michelson interferometer were used in measuring the refractive index of selenium at four wave-lengths. Measurements of reflection and transmission were made at various wavelengths with a photomultiplier tube.

I

INTRODUCTION

This study of some optical properties of selenium was undertaken, following a gift of some purified selenium, because of the unusual effects of light upon the behaviour of the element. The material was used in the form of thin films deposited by evaporation in a vacuum; this technique has so far been little used in the investigation of selenium.

Selenium (1) was discovered in 1817 by Berzelius (2), who found it in the sediment at the bottom of the sulphuric acid lead chambers at Gripsholm. Since the properties of the element closely resembled those of tellurium, Berzelius called it "Selenium", from the Greek word Σελήνη, the moon (while "Tellus" is the name of the earth). Selenium occurs in the sixth group of the periodic table, along with oxygen, sulphur, tellurium and polonium, and resembles sulphur both in the various forms of the element and in its compounds. Its atomic weight is 78.96, its atomic number 34. The boiling point is 688 degrees C., the melting point of the gray metallic form 220 degrees C. (3). Vitreous selenium consists of chain molecules, and is described as a super-cooled liquid of great viscosity. The red, crystalline monoclinic variety is made up of 8 - membered ring molecules. Metallic selenium is gray and crystalline; it is the stable form of the element at all temperatures up to 220 degrees C.

In 1873 Willoughby Smith (4) announced the discovery that the resistance of selenium altered materially on exposure to light. This aroused widespread interest in the following years, and many conflicting results were found.

Selenium has a number of chemical uses. It gives coloured glass a pale violet colour, or red (used for signal lights on railroads), and as a decolourizer overcomes the natural green colour of ordinary glass. It is effective in preventing combustion and has been used to flame-proof insulated wires. It will flame-proof wall-papers, etc. but the large amounts required make the expense too great. It has been used in the vulcanization of rubber, and the manufacture of pigments.

Selenium compounds have a physiological effect similar to that of arsenic, but their toxicity is much less. Selenium is used as an insecticide. Selenates, unlike sulphates, are highly poisonous.

Selenium is used in some kinds of barrier layer photocells, for instance the Weston "Photronic" cell, in exposure meters and light meters, and in selenium rectifiers, e.g. for battery chargers. A type of photographic reproduction has been developed which depends on its photoconductivity(5).

Most of the work on the optical properties of selenium has been done with the metallic variety or the red crystalline one, and little with the vitreous form used in this study. Wood (6) used thin prisms of vitreous selenium to measure the dispersion of the red end of the spectrum. Using an interferometer method with thin films, Wood found a dispersion curve for the range 7500A to 4000A which rises as the wavelength is decreased and has a turning point in the green. He found the absorption rising gradually as the wavelength is decreased, from 7100A to 3400A. Edmunds,(7) observing the elliptical polarization of light reflected from vitreous selenium, found ageneral transition from vitreous to metallic reflection with decreasing wavelength. Analytical interpretation gave absorption and dispersion curves of the same form as Wood's. The experiments as well as the application of the dispersion formulae seemedto indicate a number of overlapping absorption bands in the ultra-violet. Edmunds found that there is apparently no selective reflection in the ultra-violet although this is suggested by the high absorption.

Weiner (8) points out that little attention has been given to the possibility of photo-conduction in the red amorphous form of selenium, the usual statement being that it is an insulator showing no photo-conductivity and he reports that it is a photo-conductor with a spectral response peaked in the blue-green portion of the spectrum.

II

PREPARATION OF SELENIUM FILMS

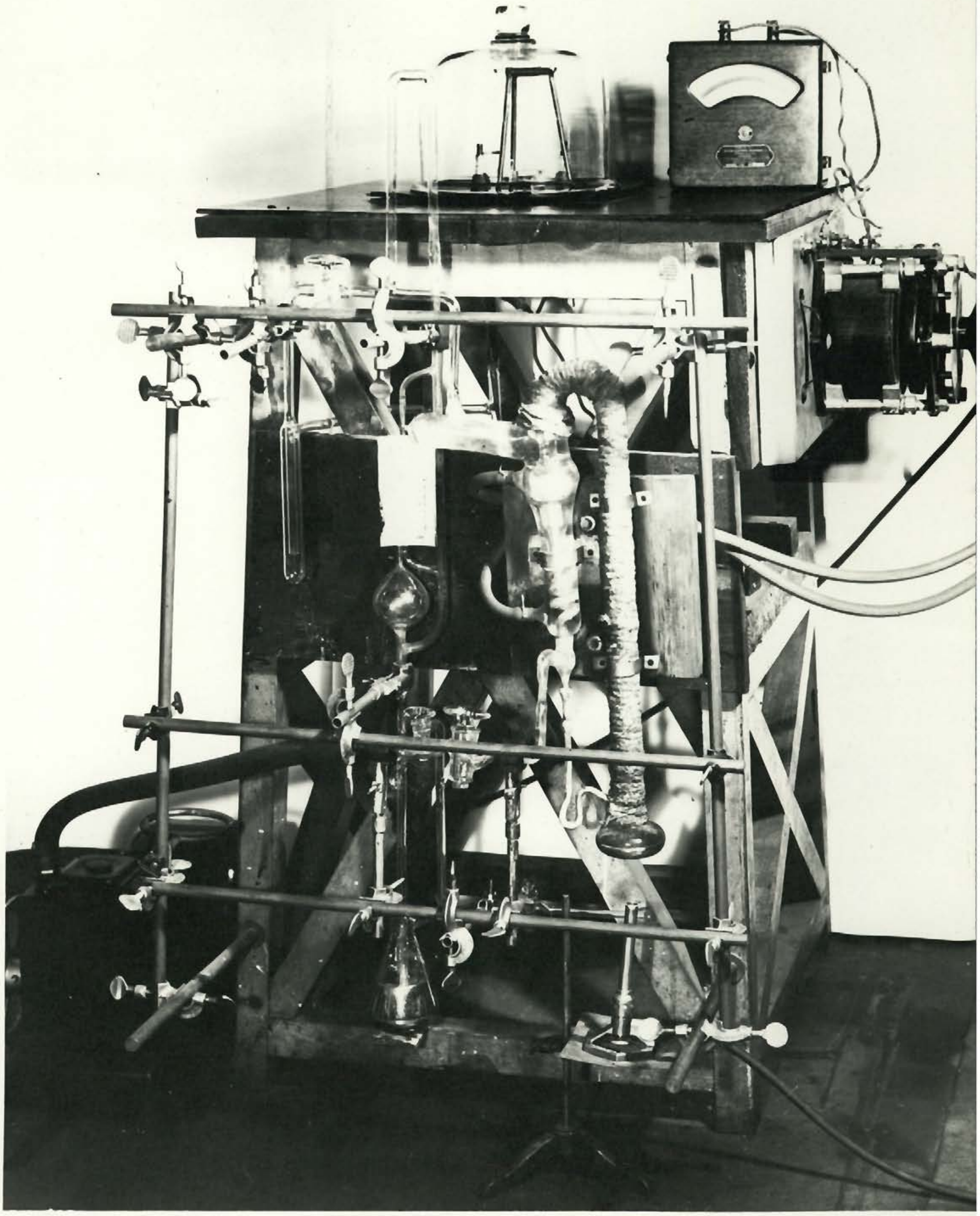
The thin films of selenium used in the following work were prepared by vacuum evaporation (9,10) of selenium onto quartz plates, three-inch glass microscope slides, or the compensating plates of the Michelson interferometer. Purified selenium manufactured by the Canadian Copper Refiners and given to the laboratory by Professor J.U. MacEwan was used for the earlier films, and double-distilled in quartz (DDQ Brand) selenium presented by Canadian Copper Refiners for the later ones. The films used for measurements were made from this second sample.

The Evaporation Apparatus

The vacuum apparatus used for making the selenium films (Figure 1) is described by D. S. Smith (11), and was previously used for evaporating aluminum. The vacuum line was rebuilt on the same design by Mr. R. Lorimer. The system is evacuated by a glass three-stage mercury diffusion pump backed by a Cenco Hyvac pump; a pressure of 10^{-5} mm. of mercury or less can be produced. A mercury trap which is in the line was not used in this experiment.

The evaporating chamber (Figures 2 and 3) is formed by a glass bell-jar resting on a brass plate, through which a tube leads to the vacuum line. Two spark plugs, threaded into the base-plate, provide insulated electrical lead-ins.

Figure I - THE VACUUM EVAPORATION APPARATUS



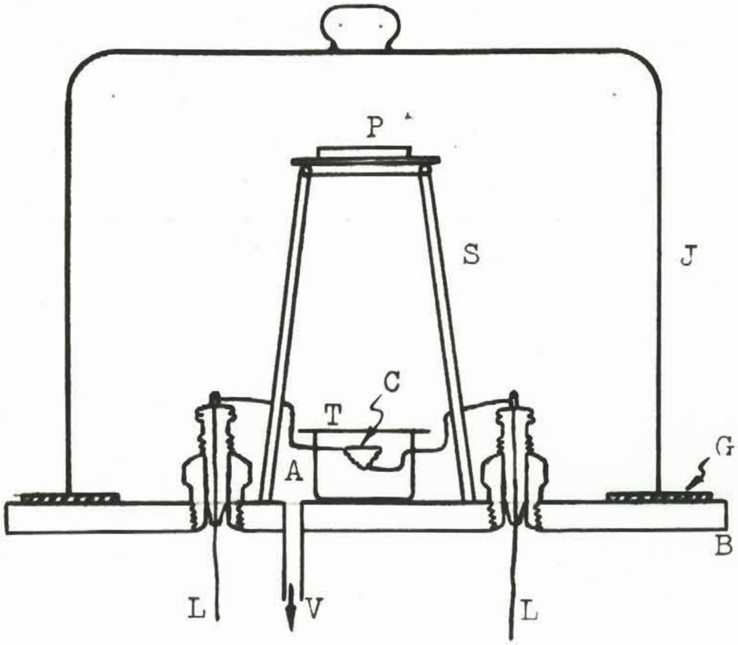


FIG. II THE EVAPORATING CHAMBER

P - Plate to be coated

S - Stand

C - Crucible

A - Pyrex Cup

T - Cover

B - Base Plate

G - Gasket

J - Bell-Jar

V - to Vacuum Line

L - Leads

Figure III THE EVAPORATING CHAMBER

The joint between bell-jar and plate is sealed with a neoprene gasket, coated with Apiezon M stopcock grease. This joint is very satisfactory, easily opened and re-sealed, and seldom needs re-greasing.

The selenium to be evaporated is placed in a small crucible, one cm. top diameter, made by applying Alundum cement to a conical basket formed of 25-mil tungsten wire, and sintering for a few minutes at 1200-1500 degrees C., as measured by an optical pyrometer, after drying(12). Chromel wire was found unsatisfactory for making these crucibles, since it usually melted or broke during the sintering. A conical wire basket of bare tungsten did not hold the selenium, which ran through when melted. The crucible is connected by heavy copper wires to the terminals of the spark-plugs, and the heating current, measured by a 0-15 amp. ammeter, is supplied at low voltage by an eleven hundred watt Variac connected to the 110-volt line. The crucible is supported, and enclosed at the bottom and sides, by a glass cup. This is made from a Pyrex beaker of one inch diameter, cut off one inch above the base, with two slots cut down three-eighths inch from the top to permit passage of the wires. This cup is covered with a piece of tin-plate. With this arrangement, the selenium can be melted and given a preliminary de-gassing while covered, without danger of depositing any impurities on the plate, and the cover removed by means of a magnet outside the bell-jar when the evaporation of the film is to commence.

The plate, which is to receive the film, is supported about 10 cm. above the crucible containing selenium upon a stand which is made of glass tubing. If a thin strip only is to be coated, the stand holds two microscope slides held apart by spacers, and the plate rests on these.

Cleaning of Plates

All plates were thoroughly cleaned with hot $KMnO_4$ solution (4%), followed by dilute HCl, and rinsed in tap water and distilled water. They were then rinsed in alcohol, and dried with cotton wool and alcohol (13). A glow discharge, for cleaning by ionic bombardment, was produced in the chamber at a pressure of about 2×10^{-3} mm. (the lower limit of the fore-pump) by applying the terminal of a Tesla coil "leak-hunter" to one of the leads of the spark plugs for about fifteen minutes. It is difficult to assess the value of the discharge in this experiment. No significant difference was noted between films on plates which had been exposed to the discharge and those not so treated. The usual result of poor cleaning is films which peel off or show pinholes, and neither of these difficulties was encountered with the selenium films.

Evaporation Procedure

After cleaning the plate and arranging the apparatus as described above, the vacuum chamber was sealed and the fore-pump started. When the system was pumped down, the discharge was started and run for fifteen minutes. When this was completed, the mercury diffusion pump was started and in one-half to one hour the pressure was reduced to less than 5 x 10^{-5} mm. The heating current was then turned on: only very low voltages were necessary with the particular crucibles and leads used. The current was immediately raised to 8 amps, the Variac reading less than 2 volts, to melt the selenium. (The DDQ selenium was supplied in the form of small pellets, called "shotted" selenium). This takes only a few minutes and although accompanied by heavy deposition of selenium on the walls of the glass shielding cup, may be observed by eye. The current was then lowered to about 4 amps for fifteen minutes to out-gas the selenium and remove any volatile impurities. The cover was removed by means of a permanent magnet outside the bell-jar, and the current set at about 5 amps for the evaporation. This deposited a thin layer in five to ten minutes. The thickness of the film was estimated during deposition by observing the succession of the interference colours, preferably by reflected sky light, as the thickness increased. The pressure immediately after the evaporation was usually slightly higher than before, but still less than 5 x 10^{-5} mm. The plate was left in the chamber, with the pumps going, for at least twenty minutes, to avoid possible absorption of gas on the heated surfaces. The surface of the selenium in the crucible was not uniformly shiny after heating, suggesting slight contamination, and although no effect of this on the films was observed, the surface layer was removed before subsequent evaporation of selenium.

General Observations on Selenium Films

The films formed by the above procedure show red colourings by transmitted light. The thinner ones are a light red-brown, medium ones a brownish red, and the thicker ones a deep ruby-red. The selenium surfaces have a metallic lustre. The selenium in these films is evidently of the amorphous allotropic modification (vitreous form), and Keck (14) comments that this is the form of selenium deposited by vacuum evaporation when the plates are at temperatures lower than 50 degrees C. during coating.

The films are easily scratched and may be scraped off the plate, but adhere firmly under ordinary handling. Indeed, the films that formed on the bell jar and other objects in the evaporating chamber were often extremely difficult to remove: thin layers were fairly soluble in xylol and could be rubbed off, while thicker ones were slowly dissolved by aqua regia. The thin deposit which always appeared on the upper side of a plate was removed by wiping with xylol.

Observation with a microscope at a magnification of 1000 X showed no structure in the films.

Approximate observations on film thickness were made by several methods. The interference fringes could be counted easily for films which thinned gradually to nothing, although in the case of strips with sharp edges they could be counted only during formation. Comparisons between films can be made in this way, although the determination of the geometrical thickness would require knowledge of the refractive index and the phase change on reflection. The order of magnitude of the thickness of one film was found by the scale on the fine adjustment of the microscope as one micron, of another, by the spherometer, as three microns. However, thinner films were used for the optical measurements, and these methods of direct measurement could not be applied to them.

III

SPECTROGRAPHIC MEASUREMENT OF ABSORPTION

A semi-quantitative method(15) was used to measure the absorption of light by selenium films. Series of exposures on the spectrograph were made which differed in length by powers of two. A short series (say three) was first made with a clear portion of a plate (glass or quartz) in front of the slit. Then on the same photographic plate a longer series, of longer exposures, was made with a coated glass or quartz plate before the slit. For various spectral lines, the reference spectrum was matched with the exposure through the film which gave the same density. The comparison of exposure times gives a measure of the absorption, since the intensity of the source was relatively constant throughout.

The transmission, T, is defined(16) as the ratio of transmitted intensity to incident intensity.

$$T = I/I_o$$

Neglecting reflection, the absorption, A, is 1 - T. A quantity often used is the extinction, E, defined by

$$E = \log_{10}\frac{I_o}{I} = \log_{10}\frac{1}{T}$$

The reciprocity law(17) states that the amount of a photochemical reaction depends on the light energy absorbed - i.e. the product of the light intensity, I, and the time of irradiation, t. This law applies to photographic plates to a good approximation.

Therefore, when densities are the same, the intensity of light falling on the slit is inversely proportional to the exposure time. Thus, $T = \frac{I}{I_o} = \frac{t_o}{t}$, where t_o is the exposure time of the reference spectrum and t is the exposure time of the matching density in a spectrum taken through the absorbing medium.

The instruments used were a Hilger medium Quartz spectrograph with a selenium film on a quartz plate, and a Hilger Glass Constant Deviation spectrograph with a selenium film, or a glass plate. The films were of approximately the same thickness, as measured by the interference colours.

A print of a plate taken on the Hilger medium quartz spectrograph is shown in Figure 4, with matching densities marked. The results are shown graphically in Figure 5. It may be seen that the selenium film on glass, for which the measurements at longer wavelengths were taken, is somewhat thicker than the other.

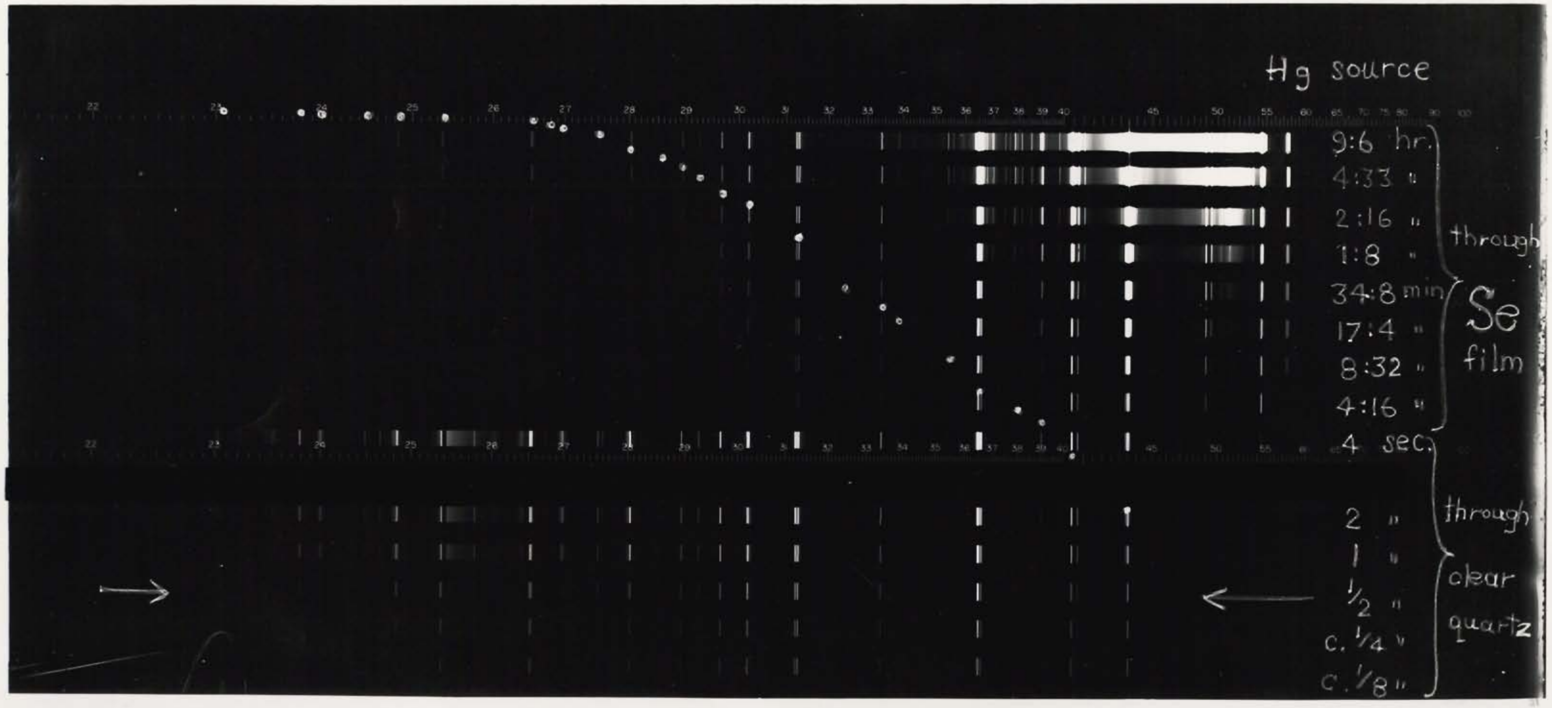
These results are in agreement with Wood's observations(6) of the absorption in vitreous selenium. He found that in the range from 7100A to 3400A the absorption rises slowly from very low values at the long wavelengths.

The curve found here rises steadily from very low values in the red, and appears to level off at about 2600A. However, the observations at wavelengths shorter than 2600A are estimated from meagre data, and are accordingly less reliable.

Although reflection has been neglected, it will be seen in Chapter V that it is not inconsiderable.

FIGURE IV SPECTROGRAM SHOWING ABSORPTION

Taken on the Hilger medium quartz spectrograph. Six short exposures through clear quartz; eight long ones through selenium film. The scale reads from 2200A. to 10,000A. The dots mark densities matching the reference spectrum, which is marked by an arrow.


Hg source
9:6 hr.
4:33 "
2:16 "
1:8 "
34:8 min
17:4 "
8:32 "
4:16 "
4 sec.
through Se film
2 "
1 "
1/2 "
c. 1/4 "
c. 1/8 "
through clear quartz
22
23
24
25
26
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28
29
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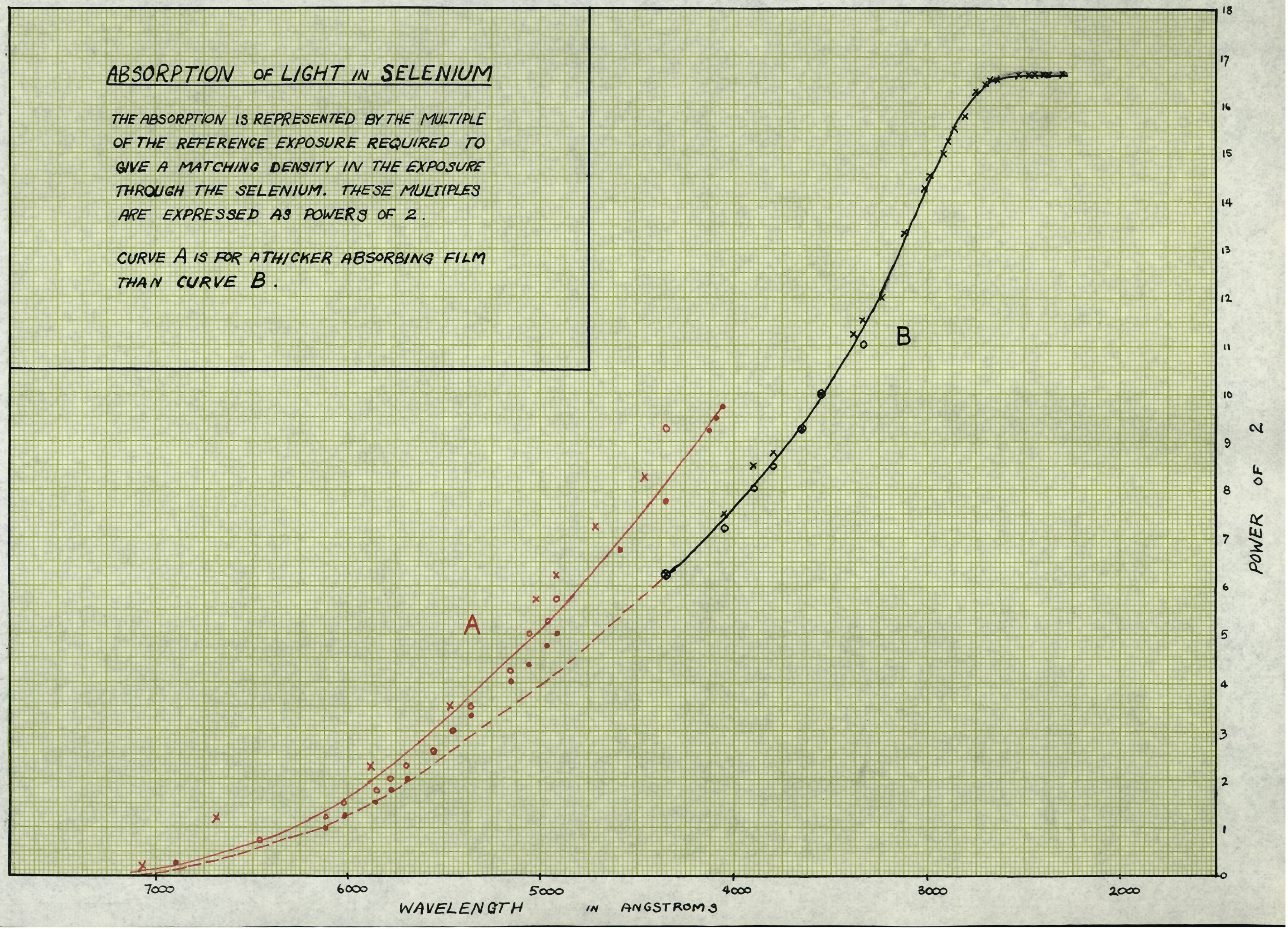
90
100



ABSORPTION OF LIGHT IN SELENIUM

THE ABSORPTION IS REPRESENTED BY THE MULTIPLE OF THE REFERENCE EXPOSURE REQUIRED TO GIVE A MATCHING DENSITY IN THE EXPOSURE THROUGH THE SELENIUM. THESE MULTIPLES ARE EXPRESSED AS POWERS OF 2.

CURVE A IS FOR A THICKER ABSORBING FILM THAN CURVE B.

IV

REFRACTIVE INDEX MEASUREMENTS

The Michelson interferometer (18) was used in an attempt to measure the refractive index of the selenium films. A horizontal strip of selenium was deposited directly on the compensating plate of the interferometer, and using vertical straight-line fringes, the fringe shifts were measured by means of a micrometer eyepiece. The apparatus is shown in Figure 6. A lens is used to focus the fringes in the plane of the cross-hairs. A ground glass screen, and a filter where necessary, are placed between the light source and the interferometer. The selenium film may be seen in Figure 6 as a dark band on the compensating plate. Pictures of typical fringe patterns are shown in Figure 7.

The effect of placing a thin film of refractive index μ and thickness t in the path of one of the interfering beams (19) is to displace the fringes laterally by an amount proportional to the change in optical length of the path. For plate at an angle of 45 degrees the path change is $1.41\left[2(\mu-1)t\right]$ and this divided by λ, the wavelength in air, is the displacement x, in terms of fringe widths. Thus

$$x\lambda = \left[2(\mu-1)t\right] 1.41$$

and if one of μ or t is known, the other can be found from measurements of x. The ratio of two film thicknesses can be found from measurements at the same wavelength.

FIGURE VI THE MICHELSON INTERFEROMETER AS USED FOR REFRACTIVE INDEX MEASUREMENTS

The selenium film may be seen as a dark band on the right-hand rectangular plate (the compensating plate.)

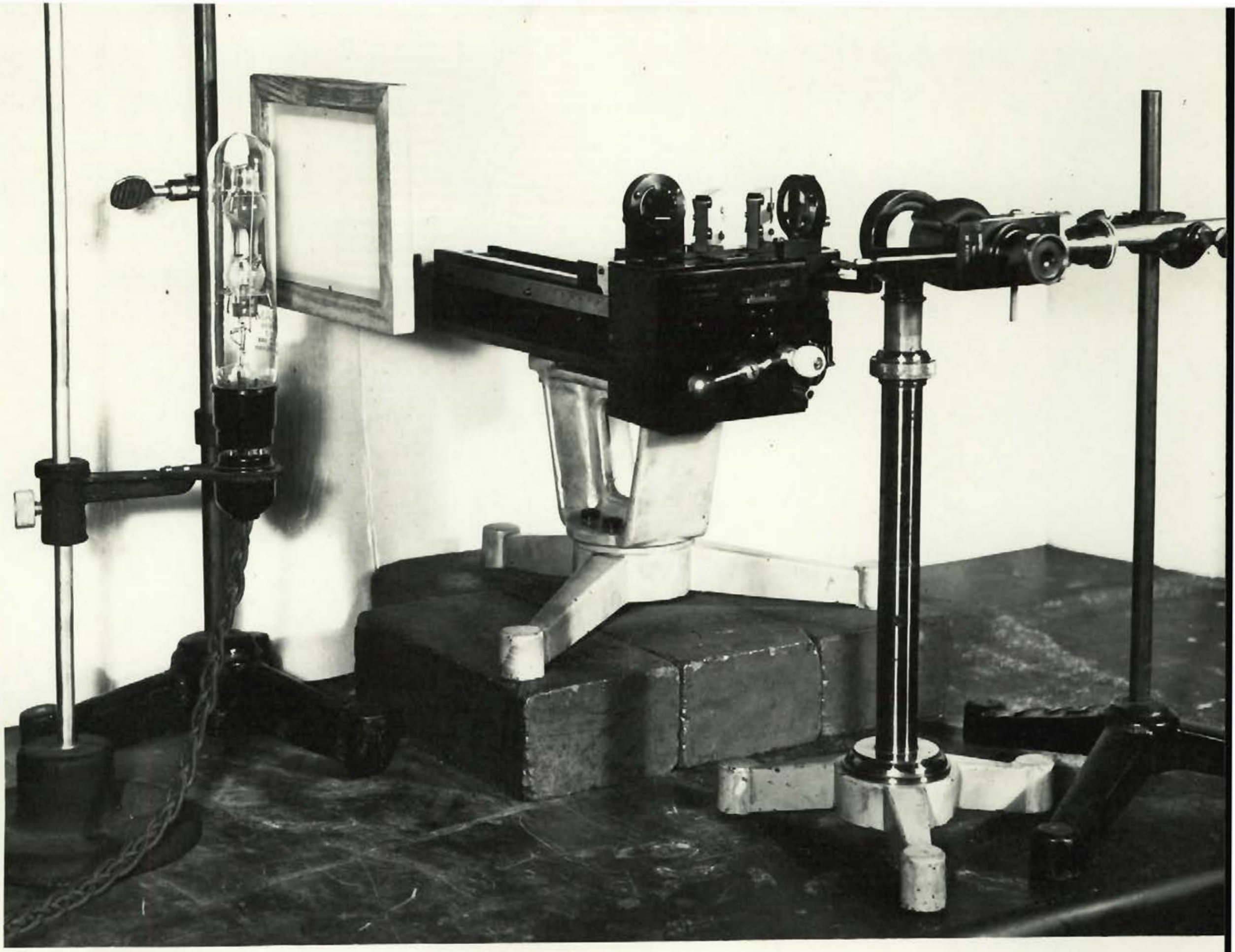


FIGURE VII SOME INTERFEROMETER FRINGE PATTERNS

1. Fringe pattern made by sodium light. At top and bottom are fringes produced by light passing through the clear portions of the compensating plate and in the middle by light passing through the selenium film. The latter fringes are displaced to the right because of the film and are curved at the ends because the displacement produced by the thinning edge of the film is less.

2. Fringe pattern made by sodium light. The pattern is viewed from above the horizontal axis and therefore some fringes are produced by light which has passed only once through the selenium film and once through the clear portion of the plate, and others by light which has passed through the film twice. In the former case the displacement is half.

3. White light fringes. The central dark fringe may be followed through the pattern to determine the number of fringe-widths displacement. This fringe is clearest in the upper portion of the picture.

Observations were made on three films of different thicknesses. The interferometer was set for white-light fringes, and the central dark fringe could be observed in its normal position and as displaced by the strip of selenium. This gives the approximate fringe shift, as, for example, three fringe widths. Measurements were made with monochromatic light, setting the micrometer cross-hair on the dark fringes of either set in order across the field. Four wavelengths were used: sodium yellow, and mercury red, green and blue. Visibility, and therefore accuracy, were best for the yellow and green.

The thickness of one film was measured by Mr. D.S. Smith, of the National Research Council, Ottawa, using a Pulfrich interferometer. This instrument measures the geometrical thickness plus the phase shift on reflection at the air-selenium surface(11). Measurements were made with six wavelengths and the mean used for the calculation of refractive indices. The thicknesses of the other films were calculated in terms of this one from the thickness ratios.

A Hilger interferometer was used and monochromatic light was produced as follows:

Red - 6230 - Mercury lamp with filter No.2, Cambosco Spectro-Radioscopic filters.

Yellow - 5890 Osram sodium lamp, no filter

Green - 5460 Mercury lamp with No.7 Cambosco and Wratten No.62.

Blue - 4360 Mercury lamp with No. 10 Cambosco and Zeiss No. BG 12.

The values found for **u** and t are given in the following table.

TABLE OF VALUES OF REFRACTIVE INDEX FOUND BY THE MICHELSON INTERFEROMETER

Film Number:	I	II	III	Mean
λ = 6230 A.	μ = 2.36	2.53	2.27	2.39
5890	2.68	2.74	2.60	2.67
5460	3.14	3.09	3.37	3.20
4360	--	3.44	3.40	3.42
Thickness:	3070*	924	2260 A.	

* measured by Pulfrich interferometer

The third figure is not accurate. Measurements were made on the accuracy of the setting of the cross-hairs on the fringes, and the error was found to be less than the scatter of the results. The accuracy of the values should be greatest for film No. II, the thinnest, since it gave the clearest fringes and the least absorption.

V

PHOTOMULTIPLIER MEASUREMENTS OF TRANSMISSION AND REFLECTION

Measurements of optical transmission and reflection were made using a photomultiplier tube as described by Kuhn(20) (see Figures 8 and 9).

The photomultiplier tube is mounted on the moveable arm of a spectrometer, and the quartz plate with the selenium film on it is fixed vertically on the spectrometer table so that the axis of rotation passes through the surface of the film. A diaphragm with an aperture about 1 cm. by 4 cm. is placed in front of the film. The photomultiplier is shielded from stray light by a round can with a slit in one side. Light from a Hilger quartz monochromator with a mercury lamp for source is focussed on the plate by a quartz lens. A source with filters may be used directly in place of the monochromator.

The photomultiplier tube, an R.C.A. 931-A(21), was run from a 1000 volt power supply with 100 volts per stage. A Sola voltage regulator stabilized the voltage to the power supply and the mercury lamp and a 500-watt Variac controlled the voltage for the power supply. The photomultiplier current was read on a microammeter.

Three readings were taken, one with the source, stop and multiplier in line (I in Figure 8) without the selenium film, the second in the same way with the film in place, and the third after the photomultiplier had been moved into position II in which the light reflected at a small angle reaches the tube.

Transmissions, reflections, and absorptions are plotted on page 26. The transmission coefficient T is the per cent of the incident light transmitted, R that reflected and A that absorbed, where

$$T + R + A = 100 \text{ per cent}.$$

The transmission coefficient decreases to very low values in the ultra-violet, while the reflection coefficient rises. The transmission curve is thus in agreement with that found spectrographically. The absorption, which is large in the green and blue, apparently decreases in the ultra-violet. Weiner(8) reports that the photoconductive response is greatest in the blue-green region of the spectrum.

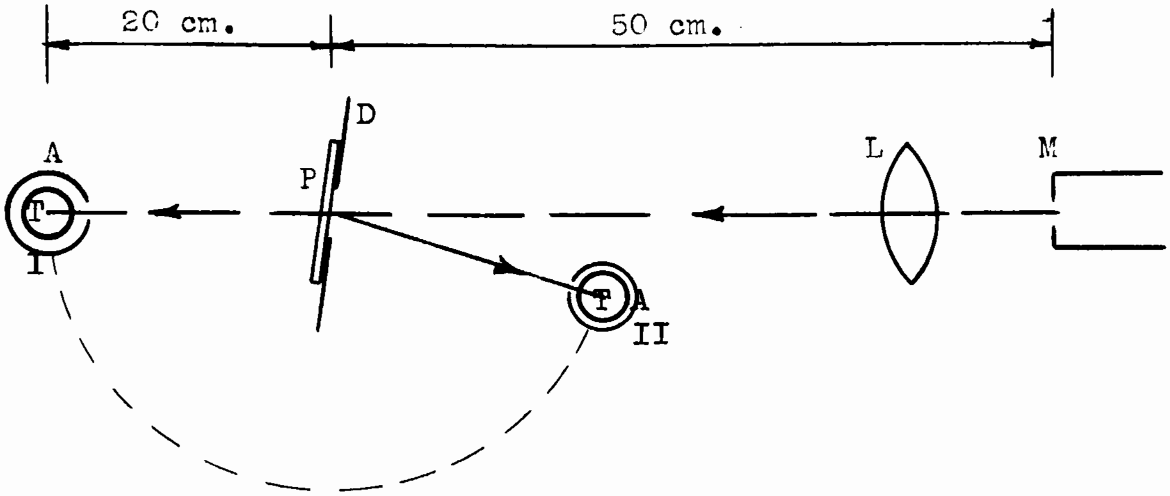


FIG. VIII APPARATUS FOR MEASUREMENT OF ABSORPTION AND REFLECTION

M - Slit of Monochromator

L - Quartz Lens

D - Diaphragm

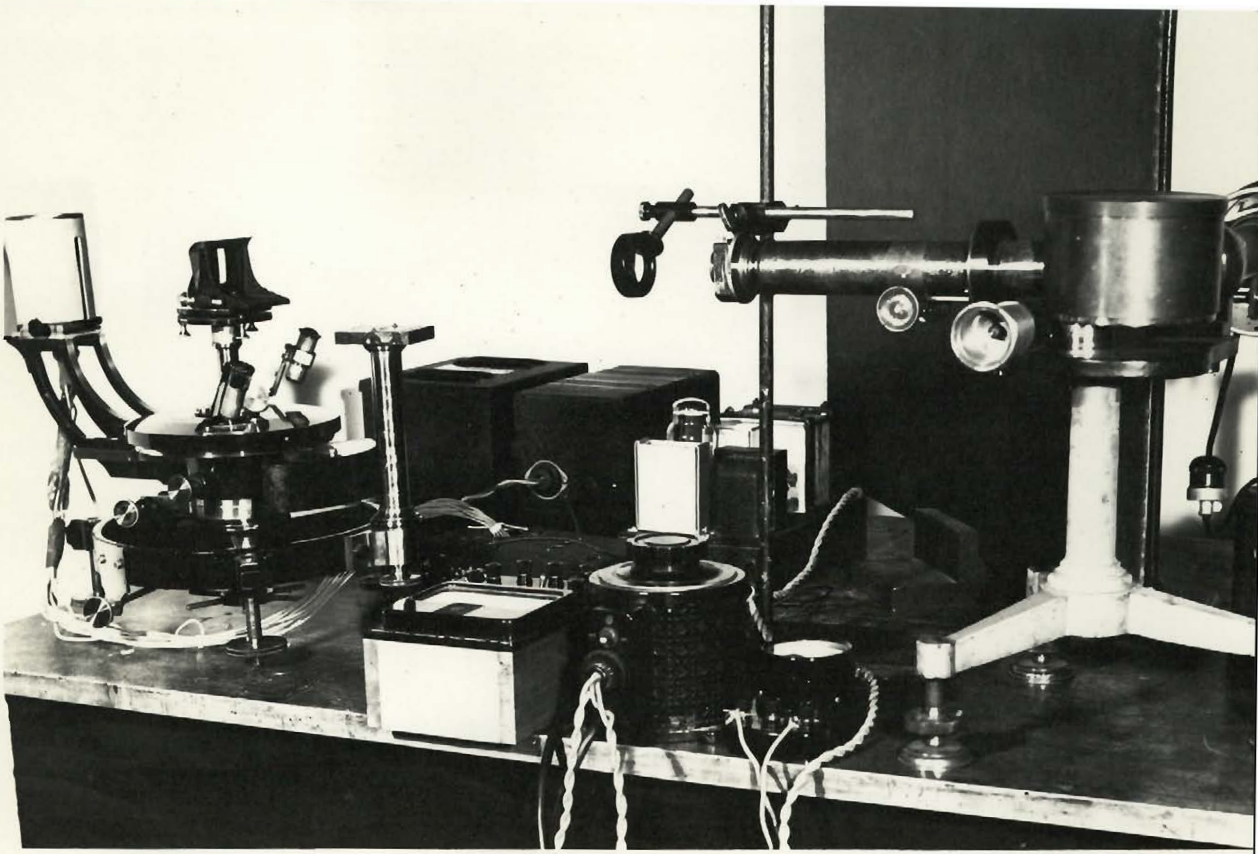
P - Plate

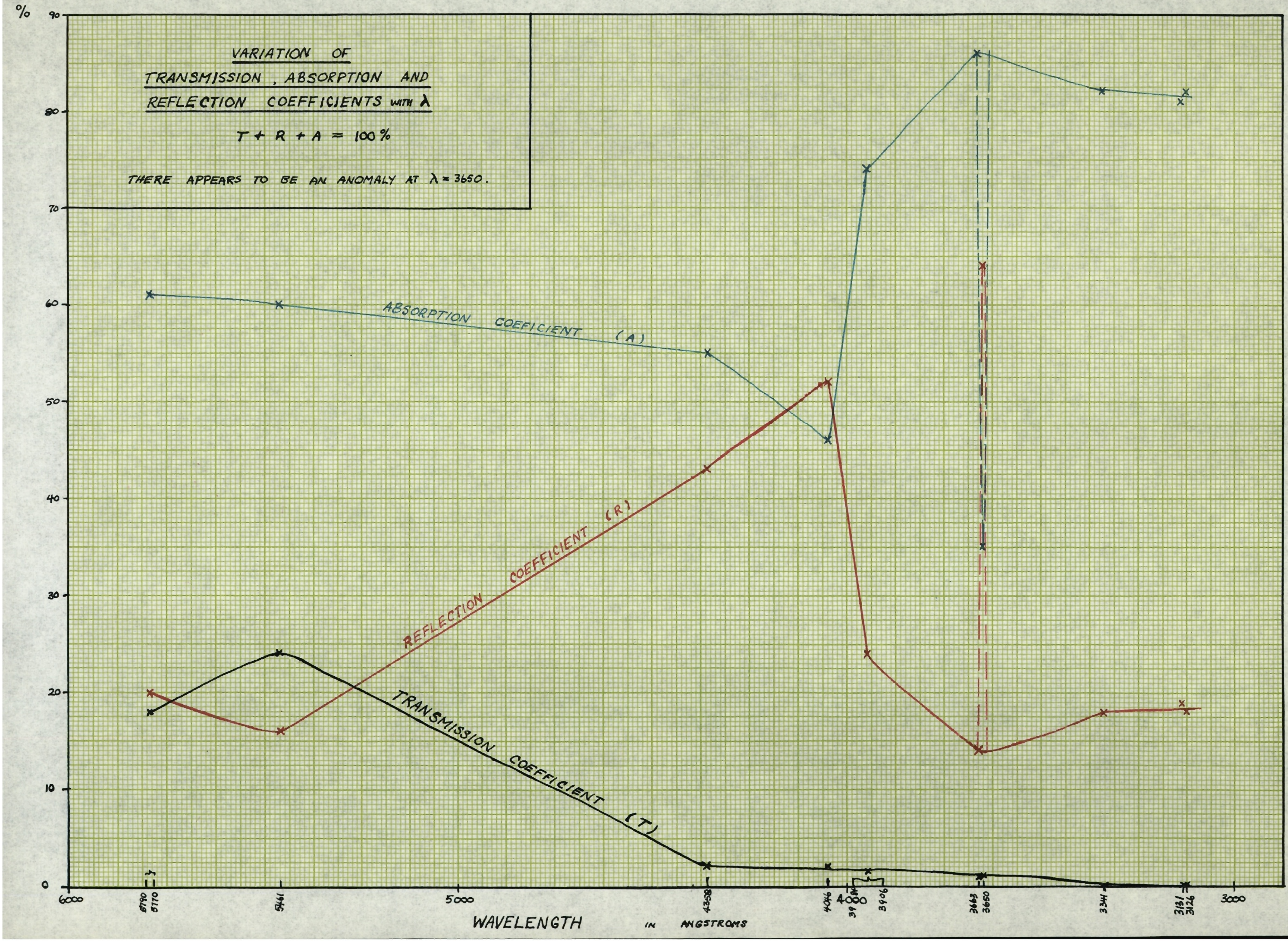
A - Shield for Photomultiplier

T - Photomultiplier Tube

FIGURE IX APPARATUS FOR MEASUREMENT OF TRANSMISSION AND REFLECTION

The photomultiplier tube mounted on the spectrometer arm at left is hidden by the cylindrical shield. The monochromator and mercury lamp are at the right.





VARIATION OF
TRANSMISSION , ABSORPTION AND
REFLECTION COEFFICIENTS with λ
T + R + A = 100%
THERE APPEARS TO BE AN ANOMALY AT λ = 3650.
%
90
80
70
60
50
40
30
20
10
0
ABSORPTION COEFFICIENT (A)
REFLECTION COEFFICIENT (R)
TRANSMISSION COEFFICIENT (T)
6000
5790
5770
5461
5000
4358
4046
4000
3984
3906
3663
3650
3341
3131
3126
3000
WAVELENGTH IN ANGSTROMS

FIGURE X

-VI-

CONCLUSIONS

The evaporation technique has proved useful for the production of thin films for measurements of various kinds. The method of determining refractive indices with the Michelson interferometer is promising, although the results presented are not very accurate. It would be interesting to examine the effects of ageing on the properties of the films, especially in view of the hygroscopic nature of selenium and the possibility of change of allotropic form. Such changes might account for the scatter in the measured refractive indices

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