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Title

AN APPARATUS FOR MEASURING THE REFLECTION, TRANSMISSION, AND PHOTOELECTRON YIELDS OF THIN METALLIC FILMS IN THE EXTREME VACUUM ULTRAVIOLET

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CHAPTER I

INTRODUCTION

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During the past several years, people in this laboratory have been studying the reflection, transmission, and photoelectron yields of thin metallic films in the far vacuum ultraviolet region of the electromagnetic spectrum.[1] In addition to their direct application to vacuum ultraviolet spectroscopy and space physics, the results obtained may be used as evidence for recent advances in electron and solid state physics. Unfortunately, however, lack of sufficient incident light intensity below 450Å has limited our investigations to the portion of the spectrum above this wavelength. This report describes the equipment which is presently being readied to extend these measurements to fifty Angstroms.

The specific experiments for which the equipment has been designed are: reflection, transmission, and photoelectron yield measurements of thin films in the wavelength range between 600 and 50 Angstroms, and measurement of the efficiencies of various gratings over the same spectral region. Since light dispersed by the grazing incidence

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[1] O. P. Rustgi. Ph.D. Dissertation, University of Southern California, Los Angeles, 1960.

monochromator should be highly plane polarized, provision has been made to allow rotating the target in two mutually perpendicular planes so that effects of polarization can also be studied. Author

CHAPTER II

APPARATUS

A simplified diagram of the apparatus is shown in Fig. 1. The major components are: a light source; a grazing incidence monochromator of the Vodar-Romand type; a reflectometer designed for reflection, transmission, and possibly photoelectron yield measurements; and an auxiliary experimental chamber which has been equipped to make grating efficiency measurements, but which can be modified easily for other experiments.

Monochromator

The monochromator is a Vodar-Romand type grazing incidence instrument designed and built at the Laboratoires de Bellevue du Centre National de la Recherche Scientifique in France. Its dispersing element is a three-meter concave grating ruled with 600 lines/mm. With this grating, the instrument will cover the spectral interval from 970Å to 72Å. A grating with 1200 lines/mm will theoretically extend this to 36Å but there is some question as to whether this is possible with an 82° angle of incidence.

A schematic diagram of the Vodar mounting is shown in Fig. 2. The entrance slit, grating, and exit slit are denoted E, R, and S, respectively. To avoid the inconvenience of a moving experimental chamber, the exit slit is

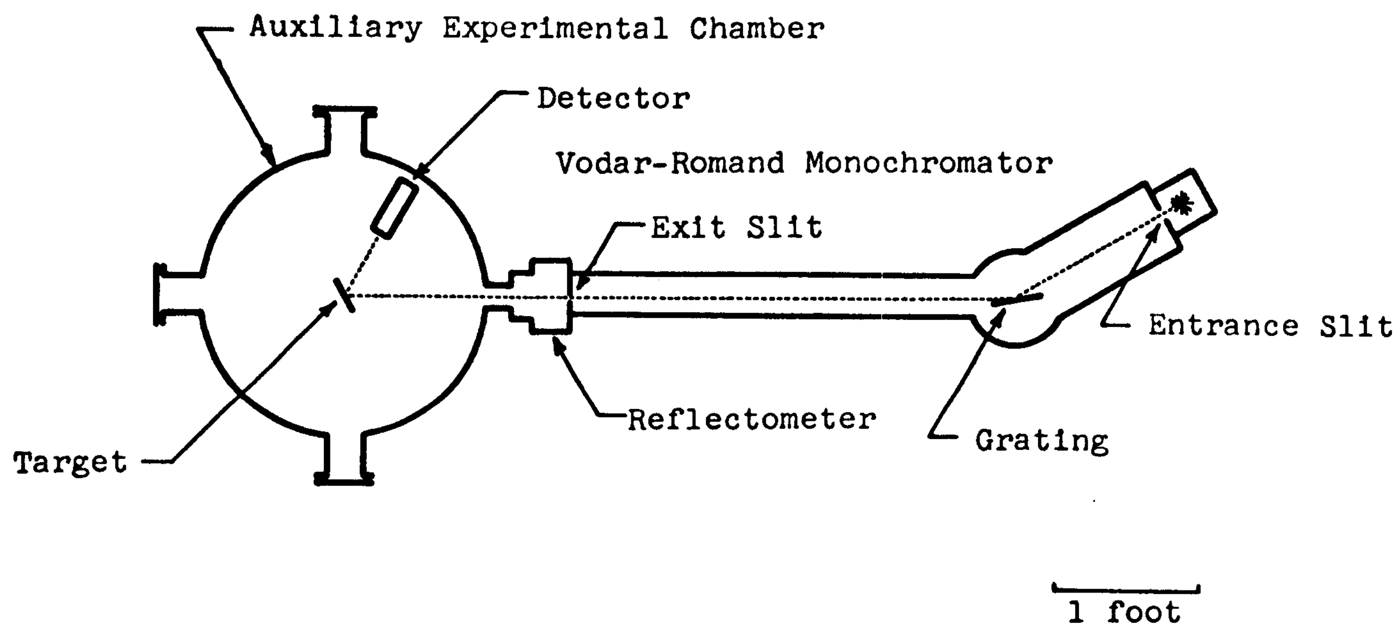

FIG. 1. THE EXPERIMENTAL ARRANGEMENT

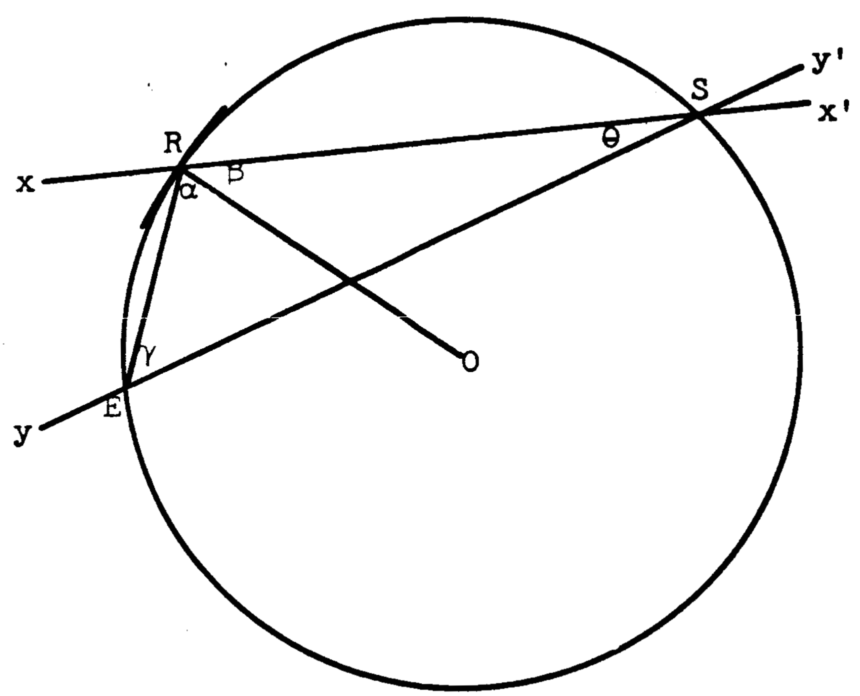

FIG. 2. A SCHEMATIC DIAGRAM OF THE VODAR MOUNTING

spacially fixed. According to simple grating theory, a focused image of the entrance slit will appear at the exit slit if, and only if, both slits lie on a circle, the Rowland circle, which is tangent to the grating and whose diameter is equal to the radius of curvature of the grating. The plane of the Rowland circle is perpendicular to the slits and the planes of the grating's rulings. Since, by the law of sines,

$$\frac{\overline{ER}}{\sin\theta} = \frac{\overline{RS}}{\sin\gamma} = \left\{\begin{array}{l}\text{the diameter of the circle passing}\\ \text{through points E, R, and S,}\end{array}\right. \qquad (1)$$

the Vodar mounting will be focused if

$$\frac{\overline{ER}}{\sin\theta} = 2\overline{OR} = 3 \text{ meters.} \qquad (2)$$

This condition is met by fixing the distance $\overline{ER}$ and constraining the entrance slit to move along the fixed track y,y', and the grating housing to move along the other fixed track x,x'.

The particular wavelength, λ, appearing at the exit slit is determined by the equation

$$\lambda = b\ (\sin\alpha \pm \sin\beta), \qquad (3)$$

where b is the grating constant. From equations (1) and (2), and since $\alpha = 82^\circ$, while $\theta = 8^\circ$,

$$\lambda = \left[b \sin 82^\circ \pm \sin\left(\cos^{-1}\frac{\overline{RS}}{3\text{ meters}}\right)\right]. \qquad (4)$$

On the actual instrument, there is a scale running parallel to x,x' and the monochromator is limited to inside orders. Therefore, the working equation becomes

$$\lambda = \left[b \sin 82^{\circ} - \sin \left(\cos^{-1} \frac{x + 513}{3000}\right) \right], \qquad (5)$$

where x is the scale reading in millimeters, and the constant 513 mm is a scale zero correction.

Fig. 3 is a simplified diagram of the instrument, supported by a cast aluminum bench. The grating housing moves along the two rails x,x' while the entrance slit is guided by a third rail y,y'. Telescoping tubes connect the exit slit with the grating chamber. These tubes are sealed with double O-ring seals equipped with inbetween pump-outs.

Both the slits have symmetric openings and are adjusted by means of external micrometer screws which are calibrated in units of microns. Between each slit and its sputtering shield there is a sliding gate valve. The valve assembly can be removed, and the samll area between the shield and the slit can be used as a differential pumping chamber.

The grating chamber is driven by a screw which is in turn driven by a variable speed motor, and the scanning speed can be varied between approximately 0.04 and 2Å/sec. The grating carriage can be rapidly traversed by releasing it from the screw. An oil-damped counterweight prevents atmospheric pressure from moving the carriage when it is released.

Reflectometer

The reflectometer, whose design is based upon

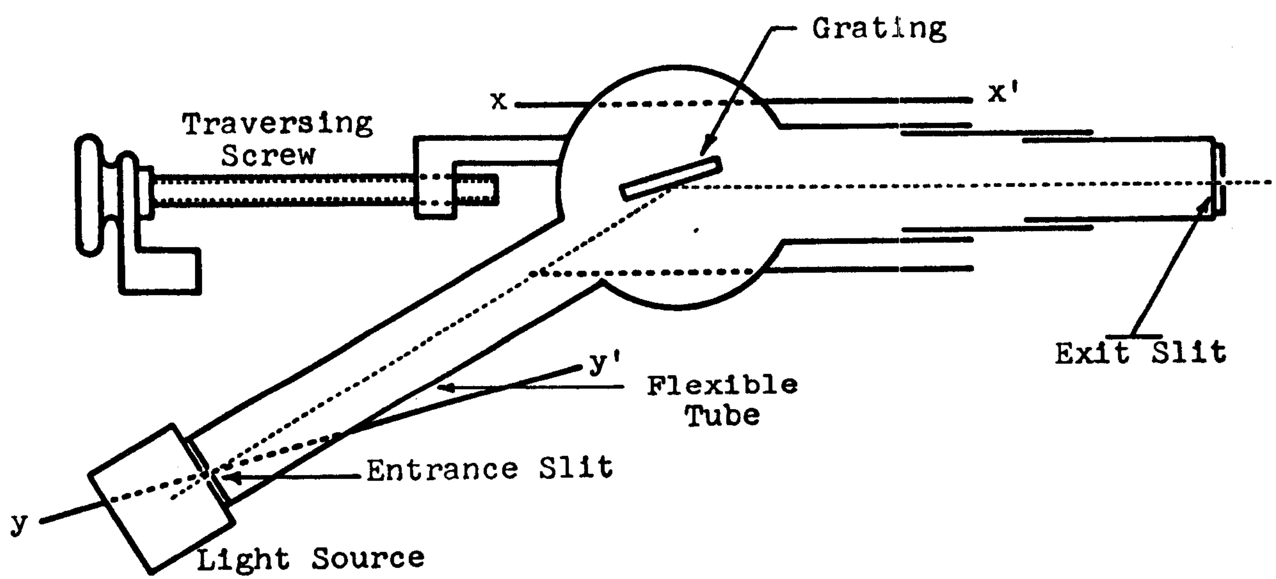

FIG. 3. THE VODAR-ROMAND MONOCHROMATOR

similar units built by Smith[2] and Rustgi,[3] is shown in Fig. 4. Its primary features are that the angle of incidence of the light on the thin film can be varied externally between eight and eighty-two degrees while the entire unit can rotate about the optic axis, thereby effectively changing the angle of polarization of the incident light. The unit is compact and the thin film is close to the exit slit of the monochromator so that the illuminated area is small.

The sample film under study is supported on a shaft passing through the wall of the chamber and sealed with an O-ring. The film can be removed from the light path by partially retracting the shaft from the chamber.

A light pipe bent in the shape of a question mark is mounted through a wall of the chamber. By means of an external knob, it can be rotated through 360° about an axis coincident with the axis of rotation of the sample. The upper end of the light pipe looks at the thin film and is coated with sodium salicylate. In the presence of vacuum ultraviolet radiation, this phosphor fluoresces at about 4000Å. This secondary radiation is transmitted through the light pipe to a photomultiplier. Since it is a true image conduit containing 73,000 filaments, the light pipe does not require an external coating of aluminum.

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[2]Abbott Smith, University of Rochester, AF 49(638)-433, TN b 4 (1960).

[3]O. P. Rustgi (private communication).

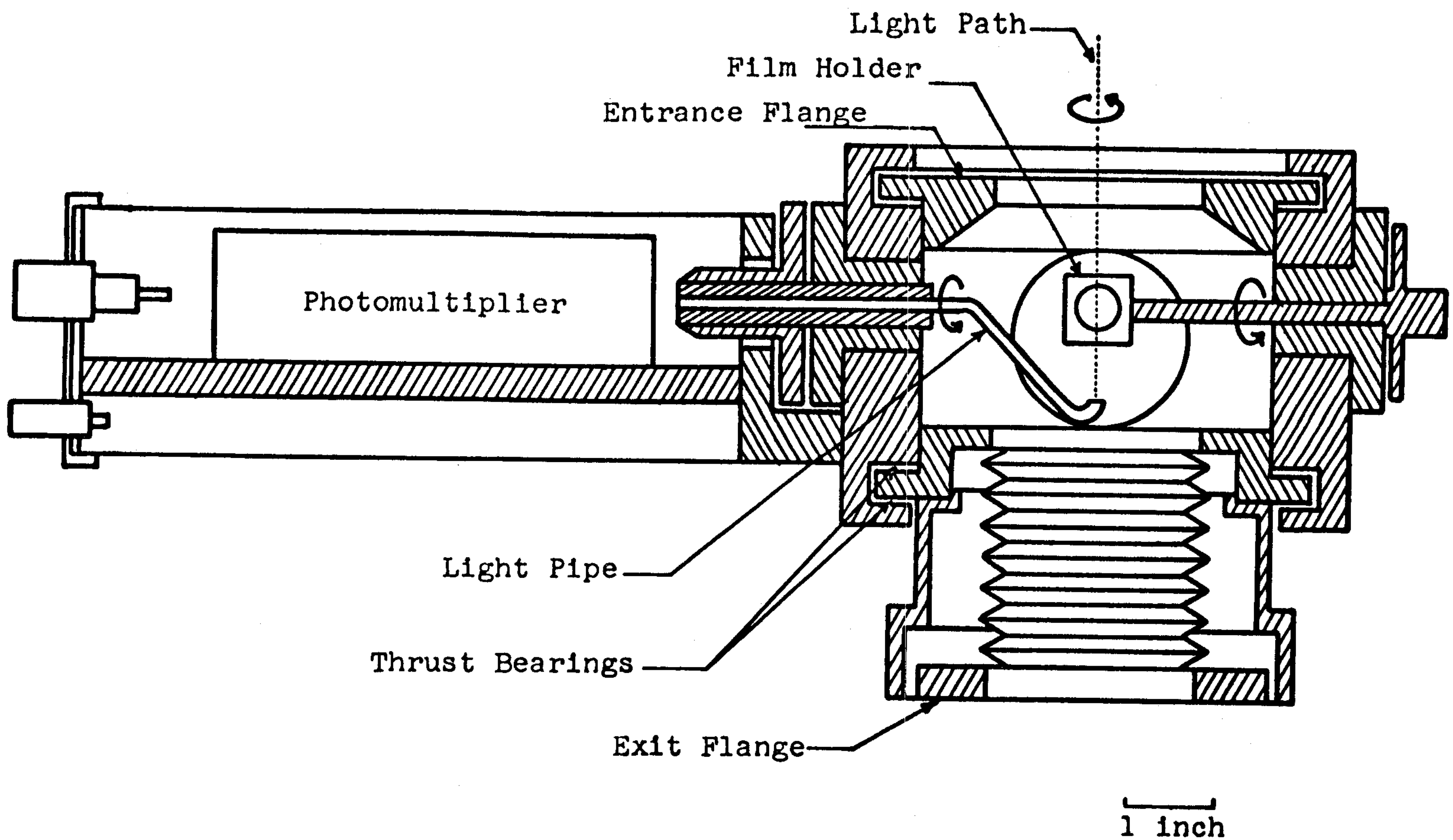

FIG. 4. THE REFLECTOMETER

A DuMont No. 6291 end window type photomultiplier looks at the lower end of the image conduit. The photomultiplier is enclosed in a light-tight electrostatically shielded housing which also contains a cathode follower amplifier.

As shown in the figure, behind the thin film there is a port to which an evaporator can be attached for forming films in situ. Opposite this port there is another one which presently serves as a window. It can, however, be used as a mounting for a thermocouple for absolute intensity measurements;[4] or an electron gun for heating the thin film substrate during evaporation to control crystal formation.[5]

By replacing the light pipe with an electron collector, the reflectometer could be used for photoelectron yield measurements. No provisions have been made to cool the sample holder, however, so that thermoelectric emission could be a serious problem.

Auxiliary Experimental Chamber

The auxiliary experimental chamber shown in Fig. 5 is essentially a closed cylinder two feet in diameter and one foot high. A photomultiplier facing toward the center of the chamber is mounted near the edge of a circular table

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[4] H. Prugger, "Photon Flux Measurements," Technical Report, University of Southern California, 1963

[5] O. A. Weinreich and G. Dermit, J. Appl. Phys. 34, 225 (1963).

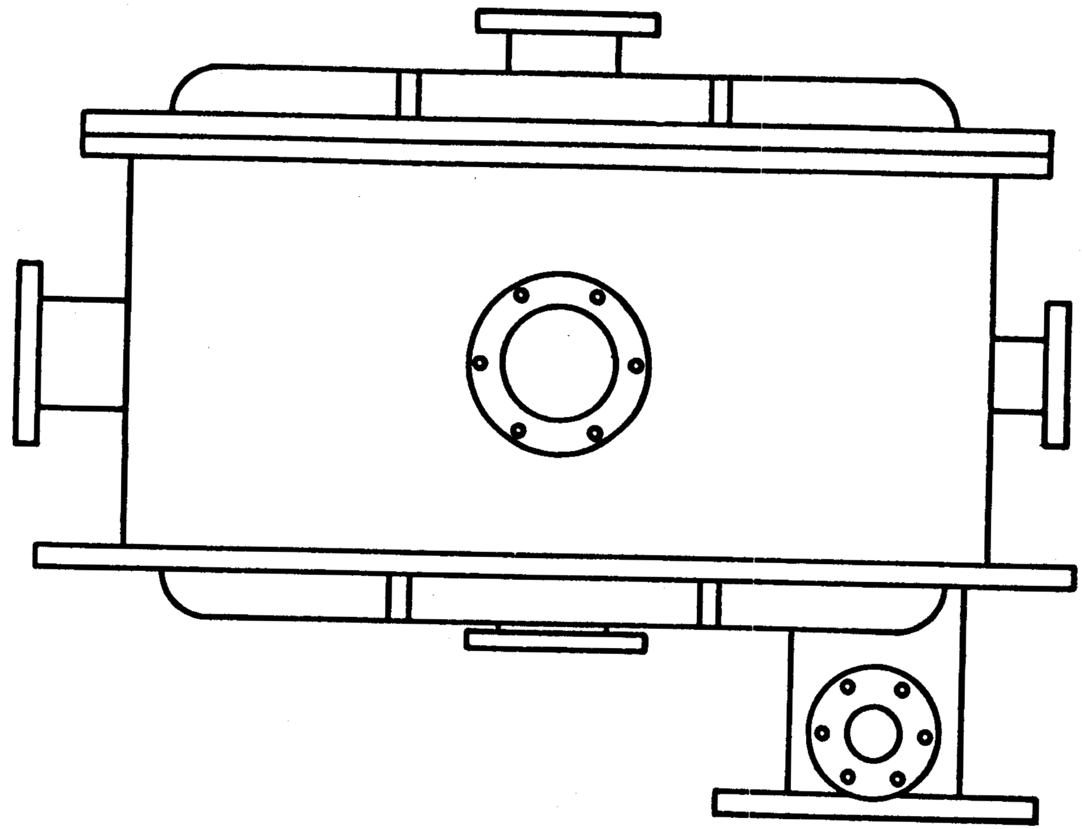

FIG. 5. THE AUXILIARY EXPERIMENTAL CHAMBER

which is supported by roller bearings and is coupled to a shaft passing through the lower central port. The object under study is supported from a port in the removable top plate and can be rotated about an axis coincident with the axis of rotation of the photomultiplier table. One of four side ports is used for the admission of the light beam while the remaining three can be used as windows or connecting auxiliary apparatus such as an evaporator.

Light Source

At the present time, a capillary discharge source is attached to the monochromator. This source was chosen because it gives an intense line spectrum of the gas being used. Such a spectrum is a definite aid during focusing and calibration of the monochromator. However, it has the disadvantages of not producing much light below 200Å and of operating at a fairly high pressure.

The light source housing supplied with the monochromator was designed to accommodate a vacuum discharge source of the Vodar type. While this type of source does produce sufficient illumination and operates at 10^{-4} to 10^{-5} mm Hg, several promising sources have been developed recently, and these will be tried before one is finally chosen for the experiments.

Detection Equipment

As has been stated previously, the reflected or transmitted light will be detected by a photomultiplier sensitized with sodium salicylate. The maximum photon flux which can be expected to strike the photocathode of the photomultiplier is of the order of 10^9 to 10^{10} photons per second and typical photon fluxes will probably be only 10^8 to 10^9 photons per second. Since the resolving time of a typical photomultiplier is 10^{-8} seconds, the output of the tube at light levels below 10^8 photons per second will be a series of pulses of constant amplitude, each pulse corresponding to an electron leaving the photocathode. The amplitude of these pulses is equal to the amplitude of noise pulses originating at the photocathode.[6] The light pulses last 10 microseconds and have a maximum repetition frequency of 100 pulses per second so that the average current from the photmultiplier will be low. For these reasons, the pulses out of the photomultiplier are integrated with a resistance capacitance network so that total charge rather than average current is measured by the readout equipment.

There are two major types of noise in a photomultiplier: stray electron emission from the photocathode and the first several dynodes, and electron emission from the glass enevlope.[7] The latter can be eliminated by keeping all metal

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[6] Fitz-Hugh Marshall, J. W. Coltman, and A. I. Bennett, Rev. Sci. Instru. 19, 744 (1948).

[7] DuMont Multiplier Photo Tubes (Allen B. DuMont Laboratories Inc., Clifton, New Jersey, 1960), p.8.

structures that are in contact with the outside of the envelope at the same potential as the photocathode. Stray electron emission is, for the most part, thermoelectric in origin and can be reduced by cooling the photocathode. It has been reported,[8] however, that cooling below $-30^{o}C$ does little good. At room temperature, there are about 10^5 noise pulses per second in a photomultiplier such as the 931A. These pulses would completely obscure the signal if the output of the photomultiplier were integrated over an appreciable time. However, since the signal lasts only 10 microseconds, if the photomultiplier output is integrated over the same period only a few noise pulses will contribute. This can be done by either gating the integrator or by adjusting its discharge RC time constant to 10 microseconds. Since the latter can be easily done, it has been chosen.

The output of the integrator will consist of a series of pulses. The pulses corresponding to incident light pulses will have a rise time equal to the length of a light pulse, a 10 microsecond decay time, and a peak amplitude proportional to the total number of photons striking the photocathode during one light pulse. The constant of proportionality depends primarily upon the effeciency of the

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[8] James P. Rodman and Harlan J. Smith, Applied Optics 2, 181 (1963).

photocathode, the photomultiplier gain, the shape of the light pulse, and the RC time constant of the integrator. The noise pulses will have rise times of 10^{-8} seconds and decay times of 10 microseconds. Their amplitudes will generally be lower than those of the signal pulses but a few, arising from several electrons leaving the photocathode simultaneously,[9] will have amplitudes which are quite large. These can be discriminated against by limiting the rise time of the peak reading voltmeter.

The peak reading voltmeter (diagrammed in Fig. 6) is essentially the circuit used by Williams *et al.*[10] except that a transistor emitter follower and a high-speed high-conductance silicon diode are used to decrease the charging time constant to 10 microseconds. The discharge time constant has been set at one second to accommodate the recorder which has a full scale deflection time of one second. The unit operates linearily only between 0.6 and 8 volts so that a pulse amplifier is used between it and the photomultiplier. The output of the unit is connected to an RCA ultrasensitive microammeter and a chart recorder.

Vacuum System

A side view of the equipment is shown in Fig. 7.

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[9] *Ibid.*

[10] S. E. Williams, M. R. Meharvy, V. W. Maslen, and R. L. Fakener, J. Opt. Soc. Am. **44**, 654 (1954).

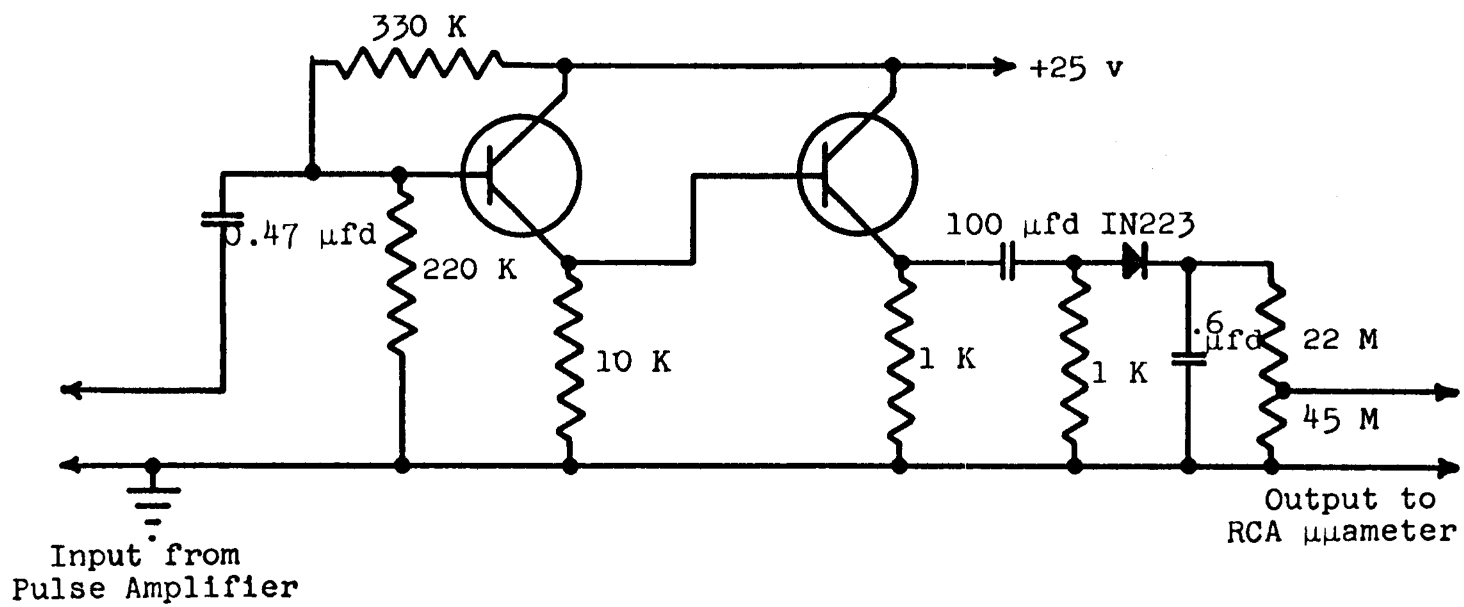

FIG. 6. PEAK READING VOLTMETER

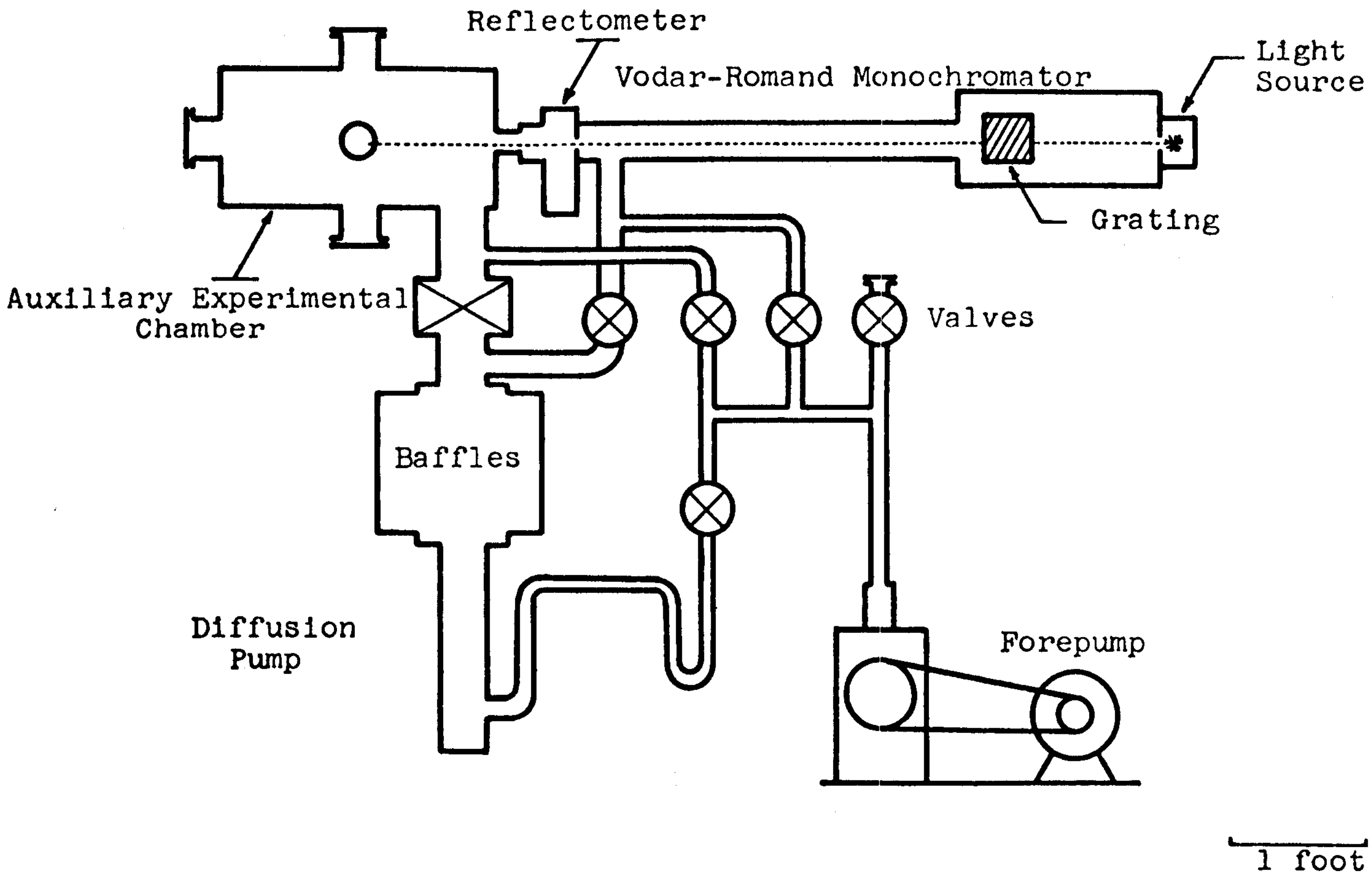

FIG. 7. DIAGRAM OF THE VACUUM SYSTEM

One oil diffusion pump evacuates both experimental chambers and the monochromator. Both air and liquid nitrogen cooled baffles are used to restrict oil backstreaming and to aid pumping. Valves have been included so that either the experimental chambers or the monochromator can be isolated from the diffusion pump. This system should produce a vacuum of 10^{-6} mm Hg provided the light source pressure is reduced to 10^{-5} mm Hg.

The light source is evacuated by a small oil diffusion pump which moves with the source. It is not baffled but will produce a vacuum of 10^{-5} mm Hg in the source chamber.

CHAPTER III

MONOCHROMATOR FOCUSING PROCEDURE

Since the exit slit and the tracks, along which the entrance slit and grating housing move, are fixed, focusing the monochromator consists essentially of positioning the grating so that its Rowland circle is coincident with the one defined by the geometry instrument. The directions supplied with the monochromator were inadequate, so the following procedure was developed.

The grating was centered in its holder and the holder was positioned so that the center of the grating lay on the axis of rotation of the grating support table. This was done to within 0.0005 inch with the aid of a micrometer gauge.

A floodlight was used as a light source, and the grating table was rotated until the central image appeared on a translucent screen in the exit tube of the monochromator. Because the central image does not fall within the range of movement of the grating housing, the screen was placed seven centimeters inside the tube. The image thus observed had well defined sides but was limited in height only by the diameter of the exit tube. Nevertheless, its intensity diminished at the top and bottom; and the grating was tilted until the brightest portion was centered vertically on the screen.

The grating was now rotated in its holder until the planes of the rulings were parallel to the edges of the central image and therefore the entrance slit. The rulings, which cannot be seen with the naked eye, were defined by the visible spectrum produced with a flashlight bulb suspended in the grating housing. A cathetometer was used to compare the planes of the rulings and the edges of the central image.

Finally, the exit slit was placed on the exit tube, and the grating rotated until the 584.3Å line of helium appeared at the exit slit when the grating housing scale read 379 mm.

CHAPTER IV

OPTICAL TESTS

After the preliminary focusing had been completed, a capillary discharge light source was used to check the operation of the monochromator and its associated equipment. For this purpose, the oil diffusion pump without a liquid nitrogen cooled baffle was connected directly to the monochromator. Neither the reflectometer nor the auxiliary experiment chamber was used.

A 931A photomultiplier was placed at the exit slit in a small chamber which was evacuated by the diffusion pump through the monochromator's roughing line. The output of the 931A was connected to the input of a Tektronix 545 oscilloscope through a plug-in preamplifier which had been modified to change the polarity of the pulses. The integration time provided by this arrangement was 150 microseconds. This allowed the rise time of the peak reading voltmeter, which was connected to the oscilloscope's output, to be set at 100 microseconds to discriminate against the stray pulses picked up by the oscilloscope from the light source firing circuit.

Runs were made using air, helium, and nitrogen at various pressures in the light source. A typical run using nitrogen is shown in Fig. 8. The increase in the background below 200Å is due to stray light from the central image which begins to strike the wall of the exit tube at this point. This probably could be eliminated by a diaphragm in the grating housing. The absence of lines above 600Å is somewhat surprising. The most likely cause is that the slits and the planes of the grating's rulings are not perpendicular to the Rowland circle defined by the geometry of monochromator. Since the slits cannot be rotated and measurements are desired only between 50Å and 600Å, it will probably not be worthwhile to rotate the grating to its proper position should this prove to be the problem.

When helium was used at 3×10^{-1} mm Hg in the light source with a forty micron entrance slit and a sixty micron exit slit, the instrumental half widths of the 584.3Å, 303.8Å, 256Å lines were 0.67Å, 0.60Å, and 0.89Å, respectively. These compare with 0.45Å, 0.26Å, and 0.30Å obtained by Romand during the tests he performed on the instrument in France.

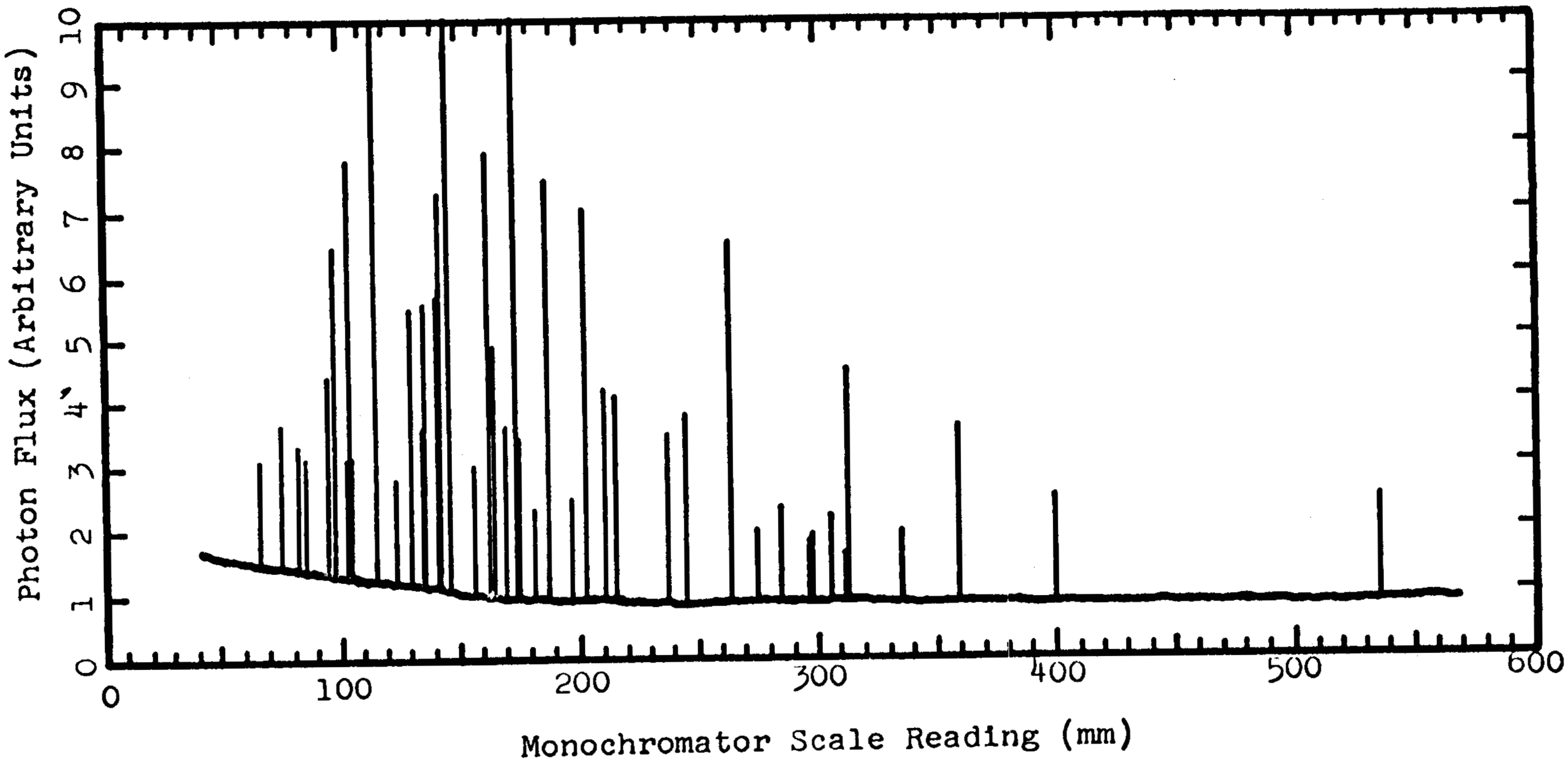

FIG. 8. SPECTRAL OUTPUT OF THE MONOCHROMATOR WITH A CAPILLARY DISCHARGE LIGHT SOURCE

CHAPTER V

CONCLUSION

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Although neither the reflectometer nor the auxiliary experimental chamber has been tested, very little remains to be done before the equipment can be put into full operation. The results of the tests on the monochromator leave little doubt that sufficient illumination can be obtained below 100Å to perform all the experiments for which the equipment has been designed. ~~Author~~