THE TRANSMISSION CHARACTERISTICS OF SOME OPTICAL CRYSTALS IN THE EXTREME ULTRAVIOLET

by

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V. INTRODUCTION

In an effort to measure radiation levels existing in space, photon counters have been developed to measure selective regions of the X-ray (1), ultraviolet (2,3), and infrared (4) spectrums. In all of these regions, the transmittance of the counter window has an important role in determining the short wavelength cutoff of the detector. In the extreme ultraviolet (1050-2000 Å) some of the more common materials used for windows are LiF, CaF_2, BaF_2, sapphire and quartz (5). Outside of this small group of crystals, very few crystals are known to transmit in this spectral region.

When LiF was found to be transparent in the ultraviolet (6), it became feasible to operate a monochromator with parts of the system evacuated with LiF windows separating various parts of the system. In addition the short wavelength region of gaseous discharge tube sources such as argon, xenon and others could be extended by the use of LiF windows (7). However, it would be useful to determine if other materials would permit a further penetration of the ultraviolet region or would have other properties that would make them superior to LiF as a window material.

A number of crystals have been obtained including several fluorides and transmission measurements were

closely analyzed for those optical crystals transmitting in the extreme ultraviolet. The main point of interest in this study was the determination of the short-wave length cutoff of the transmission curve and the characteristics of the curve near cutoff. The results obtained in this work provide a significant amount of new data on optical materials which transmit in this region.

VI. THEORY

When light is normally incident on a crystal surface it is reflected, absorbed and/or transmitted. In order for a photon to be absorbed, its energy must exceed that of the band gap between the valance band and the lowest conduction band. Thus one might expect that a photon with an energy of $E + \delta E$ could be absorbed while one of $E - \delta E$ might be transmitted (where E equals the band gap energy). This sharp discontinuity in the slope in the transmission curve is not found as it takes a finite range of wavelengths (50 - 100 A) to complete the switch from transmitting to absorbing.

There are however, two factors which have not been mentioned that play an important role in broadening the transmission cutoff region. The first is impurities in the crystal and the second is the creation of exitons.

Impurities can add levels slightly below the conduction band or above the valance band. This had the result of lowering the band gap energy in these local regions. But with impurities in the order of ten parts per million, there is a limit as to how many photons can be absorbed at the lower energy compared to the number absorbed at higher energies.

Patterson (13) experimentally found a linear relationship between the log of the absorption coefficient (α) and the wave length for pure crystals. Impurities cause this relationship to vary from a linear one, as shown by the dashed line in Fig. 1. Similar behavior was found to hold for all thin films of ionic crystals tried in the extreme ultraviolet.

The other factor which had been ignored was that of exiton formation. An electron-hole pair is produced for each photon absorbed in the crystal. If the photon has an energy greater than the gap energy, the electron and "hole" might be considered to be independent of one another. However, the equivalent coulomb attraction which exists between an electron and a "hole" leads to a theoretical situation in which the electron and the "hole" rotate about the common center of mass. In this case they are not free to move independently. As in the case of atoms, the energy of the exiton is quantized. This results in a series of specific levels directly below the conduction band continuum.

The preceeding discussion should lead us to expect that not all crystals should lead to the same results near cutoff and even with the same type crystals, different behavior might be found.

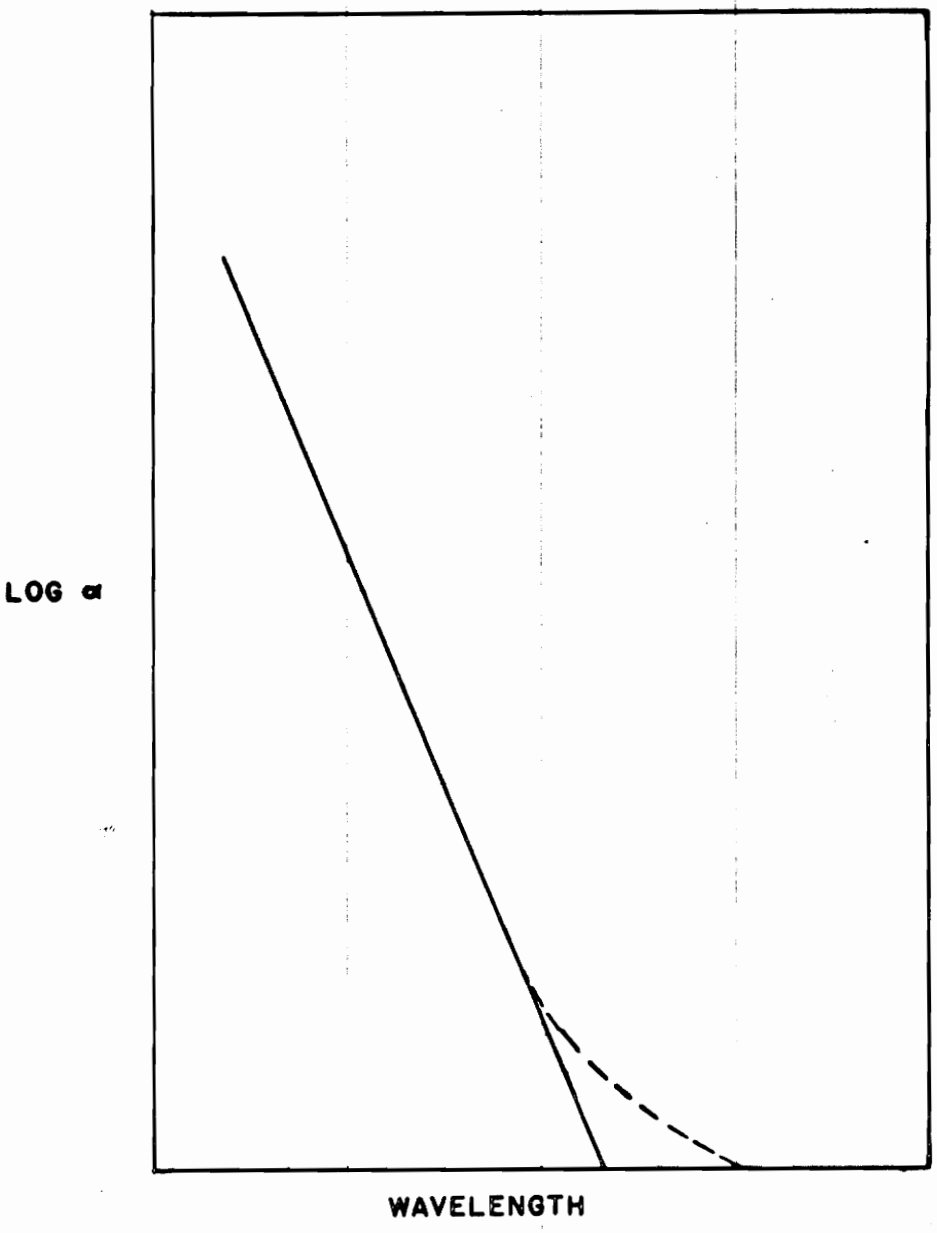

Figure 1 - Experimental curve of the absorption of extreme ultraviolet in thin films.

VII. INSTRUMENTATION AND EXPERIMENTAL PROCEDURE

Various techniques for obtaining monochromatic light have been known for some time. In 1819, Fraunhofer studied the diffraction of parallel light by gratings which provided a means of obtaining a spectral distribution of the incident radiation other than by prisms. Later Rowland (8) developed a technique for ruling a grating directly on a metallic spherical mirror. This eliminated the need for light to pass through a transparent medium and allowed regions of the ultraviolet to be studied for which glass lenses were opaque.

The theory of concave gratings has been treated extensively and will not be discussed further here. For example, Namioka (9) has recently published a comprehensive treatment. Based on the theories covered in this reference as well as in others several different grating mountings have been developed for particular applications.

Of these arrangements a type known as the Eagle (12) mounting has become dominant in vacuum monochromators as it requires fewer moving parts and much less space than other types. The Eagle mounting places all the moving parts on one axis by introducing the idea of rotating only the grating. This results in a number of structural

and optical advantages, such as ease in building a vacuum system, but there are a few disadvantages: 1) Light is not incident normally on the photographic plate, and 2) The spectrum is not linear. Between the calibration points of 1216 $\mathring{A}$ and 1608 $\mathring{A}$ the maximum error in a measured wavelength was 0.5 angstroms due to this non-linearity. The data may be adjusted for the non-linearity by employing the formula

$$\lambda = a + bs + cs^2.$$

If λ_1, λ_2, and λ_3 are three wave lengths corresponding with scale readings of s_1, s_2, and s_3 where

$$S_2 \sim \frac{s_1 + s_3}{2}$$

then $$\lambda = [a - \frac{e}{4}(s_1 - s_3)^2] + bs + e(s - s_m)^2$$

where $$b = \frac{\lambda_1 - \lambda_2}{s_1 - s_3}$$

$$a = \lambda_1 - bs_1$$

$$e = \frac{\lambda_2 - a - bs_2}{(s_2 - s_1)(s_2 - s_3)}$$

and

$$s_m = \frac{s_1 + s_2}{2}$$

It is found that this configuration can be improved by mounting the grating in such a way that the principle

ray does not coincide with the principle axis of the grating. This type of mounting is referred to as an off-plane Eagle mounting (12, 18). The Seya-Namioka vacuum scanning monochromator used in this work is an improved mounting of this last type. It has a 30,000 line per inch 3 1/4 inch wide grating with a radius of curvature of 0.50 meters. It is blazed for maximum reflection at 1100 A. The monochromator, with this grating, has a range of 500 A-3000 A. A diagram of the monochromator is shown in Fig. 2.

A crystal holder was constructed so that a large variety of shapes and sizes could be easily accomodated. This holder was turned out of lucite and then painted black. The black paint was a precaution needed only for runs made above 2000 Å. Below this wavelength lucite is totally opaque, and it is not luminescent. It was also constructed so that either a photomultiplier or an ion chamber could be used in recording the photons which pass through the crystals.

The crystal holder was constructed to fit the upper aperture of the exit port which was modified by the addition of a beam splitter directly following the exit slit. This beam splitter is a 42° prism which is coated with aluminum and a thin coat of MgF_2 to protect the aluminum from corrosion. This coating reflects between 50% and 85% of the incident radiation in the region of interest. The prism is actually used as

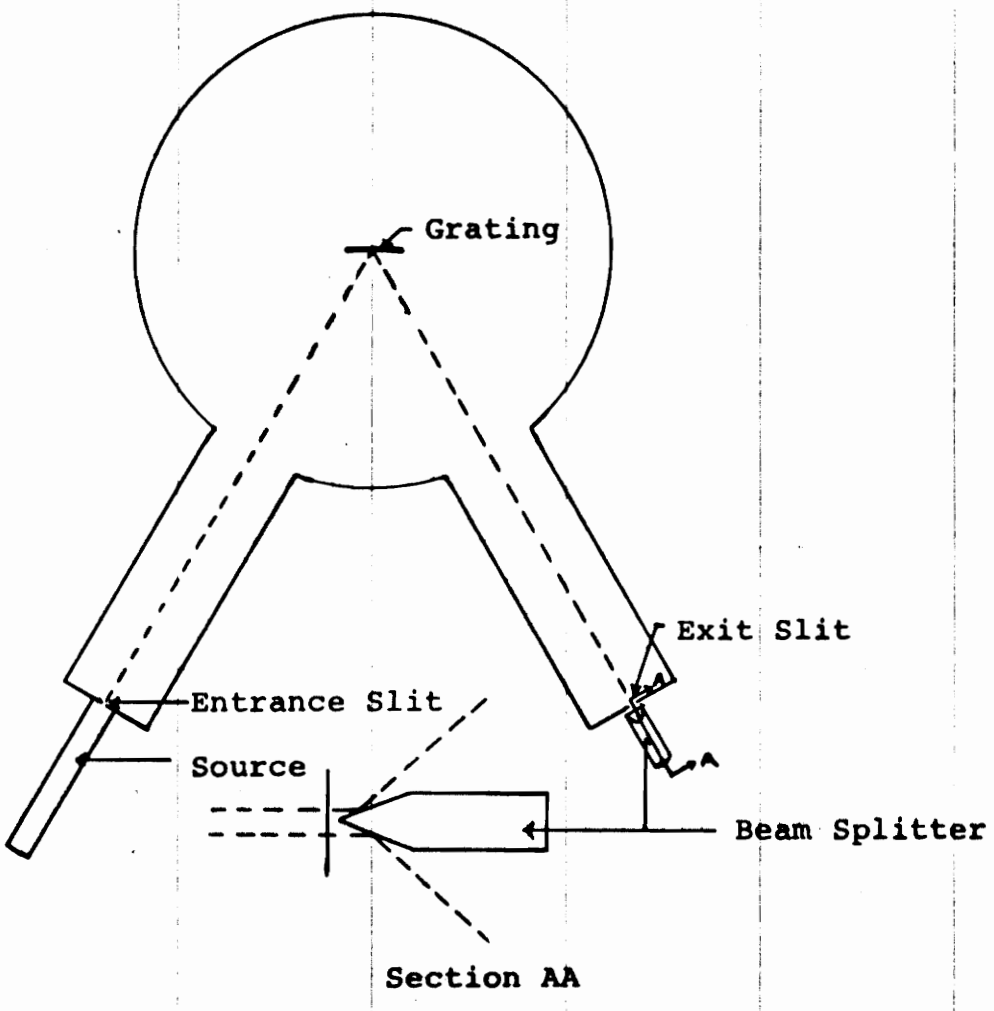

Figure 2 - Sketch of Seya-Namioka Monochromator

two mirrors with their reflecting planes separated by 42°. By changing the adjustment of the position of the prism the ratio of the intensity of incident light on the two exit ports may be set at any desired level. This ratio was set at 1.7 - 1.0 as the upper aperture was to contain the crystal holder and the energy passing through it would be attenuated by a crystal.

The hydrogen source used produced a line spectrum similar to that described by Schubert and Hudson (14). The source was a free flow D.C. arc discharge tube, where the flow rate was monitored on the input gas line. The hydrogen was allowed to flow on into the vacuum pumps. The first pumped directly on the entrance slit and removed the majority of the hydrogen with the second pump removing anything that got past the first. A third pump was used to pump on the exit apertures. These exit apertures were each independently separated from the main system by LiF windows.

The outputs of the detectors were read on Keithly micro-micro ammeters and recorded on chart recorders.

Three types of photon detectors were used. Each had different characteristics that served to make each type of detector suited to a particular task. One was a photomultiplier tube coated with a scintillation material of sodium salicylate. A second type of

detector which proved itself useful was a CsI photocell. Finally ion chambers proved effective for several applications. Each of these will be treated briefly below showing its advantages and disadvantages.

The 14 stage EMI photomultiplier tube coated with sodium salicylate yields an almost flat response of quantum efficiency with respect to wavelength. The dark current of the photomultiplier was kept well below the signal level (a factor of twenty below the manufacturers stated level) by keeping the photomultiplier in a desiccator. Occasionally it was placed in an oven overnight at 150° C.

In order to have a method of determining quickly whether a crystal transmitts any radiation between 1050 A and 1800 Å one needs a detector which will not be affected by visible or near ultraviolet radiation. A CsI photocell has the desired characteristics as the quantum efficiency drops off above 1800 Å. Thus it could be used to measure broad band transmission by turning the grating so that the central image was focused on the exit slit. The transmission for the range was now determined by measuring the total response in the region of interest with and without the crystal in place. The results obtained are listed in Table 1

column 5. The quantum efficiency of the CsI photocell used is shown in Fig. 3.

Further study was done by making scans with the photomultiplier. In many cases, however, these transmission studies were hampered by the fact that the crystal was fluorescent. This fluorescence was in the near ultraviolet (2000-4000 A). The quantum efficiency of the photomultiplier was such that it could easily pick up this radiation. However, a photocell similar to that shown in Fig. 3 had a quantum efficiency of approximately zero in this region and could not pick up this secondary radiation. This, however, did not provide a sufficiently large signal with which to work, so ion chambers were used. These consisted of ion chambers with LiF windows and a NO gas filling. This ion chamber when operated at 45 volts had a gain of unity and at 1000 volts had a gain of about a thousand. However, the chamber was sensitive only over the limited spectral region of 1050 - 1350 A.

Stability

A stability test was made of the total system including both the light source and the recording system. The set up included placing the monochromator scan on the Lyman Alpha (1216 A) peak, and recording the outputs of a standard ion chamber and a photomultiplier.

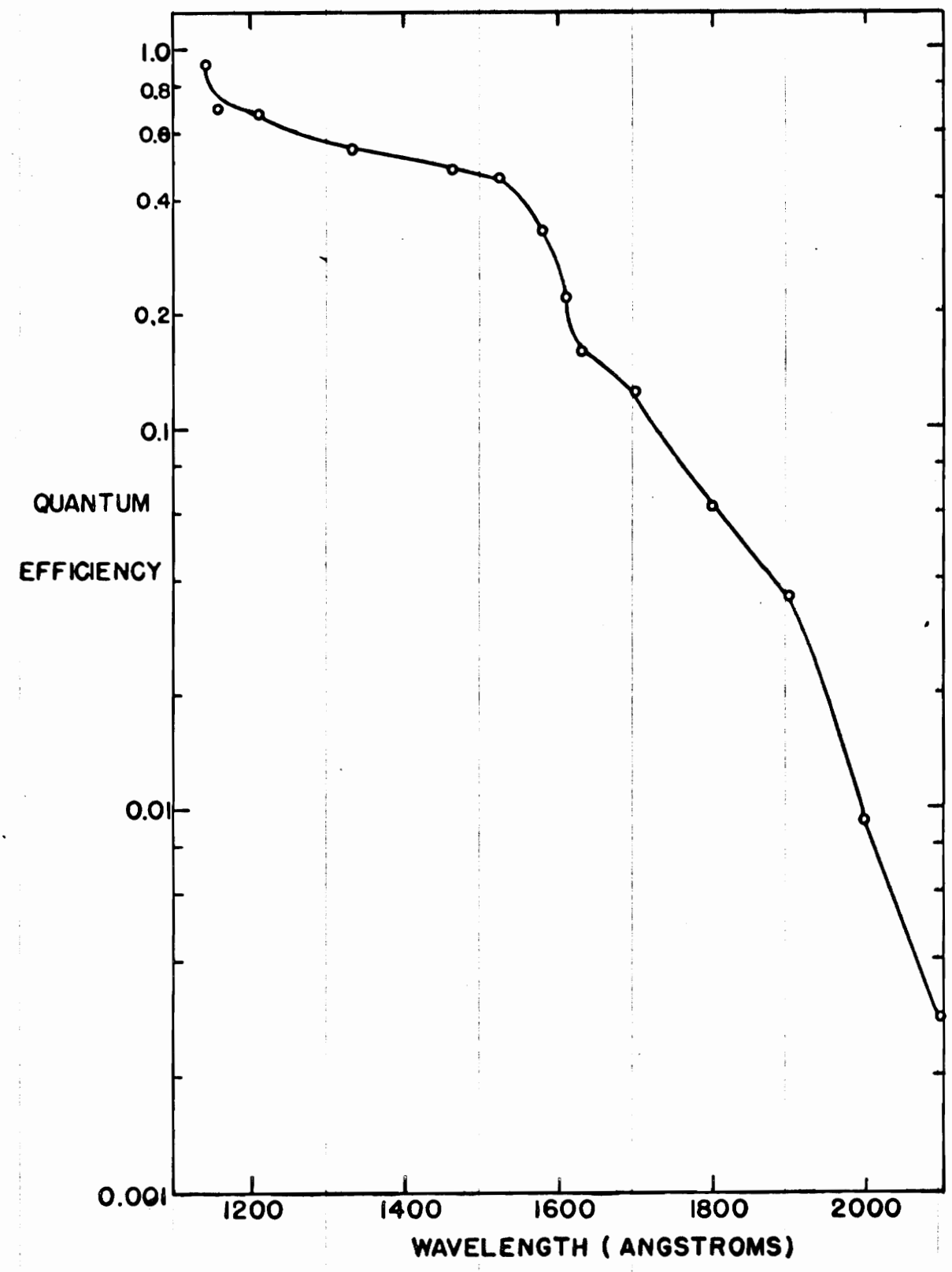

Figure 3 - Quantum efficiency of an experimental CsI photocell.

The run showed a source drift of less than 3% for an 18 hour run. Also, occasionally one of the recordings would shift slightly without the other doing so. These latter changes were attributed to either the Keithley or the Brown recorder but as they were only about 1% in value the errors introduced were considered negligible.

The output of the source varies as a function of several different factors. The most difficult parameter to control is the temperature of the arc. Compressed air circulating in a jacket around the capillary tube was employed in an effort to keep the arc temperature constant.

Crystal Preparation

Some of the crystals were received in the form of polished crystal chips and others were cut and polished from cylindrical boules. The samples which were tested were approximately 1 mm thick. The boules varied from 1/4 inch to 1/2 inch diameter. The chips varied greatly in size. The largest was a square of 1/2 inch on a side (∿1/2 mm thick). The smallest had to be carefully mounted over a 1/8 inch diameter hole in a piece of lucite.

Transmission Runs

The first transmission runs consisted of three parts. First, a calibration scan would be made using the empty

crystal holder. This would be followed by individual scans of those crystals which were of the proper size to fit in this particular holder. Finally, the record would be concluded with a second calibration scan. The lower aperture contained a monitor to check the stability of the system. The fractional transmission was determined by dividing the area under the peak by the area under the calibration peak.

It was experimentally determined that the intensity of the scattered light in the system was independent of wavelength. Thus if the scattered light was subtracted from both the calibration runs and the data runs, one could make a point by point division to obtain the transmission. This can be further justified since Schubert (14) showed the hydrogen spectra is a line spectra on a continuum of about 2% of the peak heights of the major peaks. A constant level was subtracted out by adjusting the zero level on the micro-micro ammeters. Then the outputs of the monitor and recording photomultipliers were arranged in such a way as to give the fractional transmission directly.

Figure 4 shows a comparison of the results of the two techniques. It must be remembered that the manual method is limited in the number of points that can be used for determining the fractional transmission. The

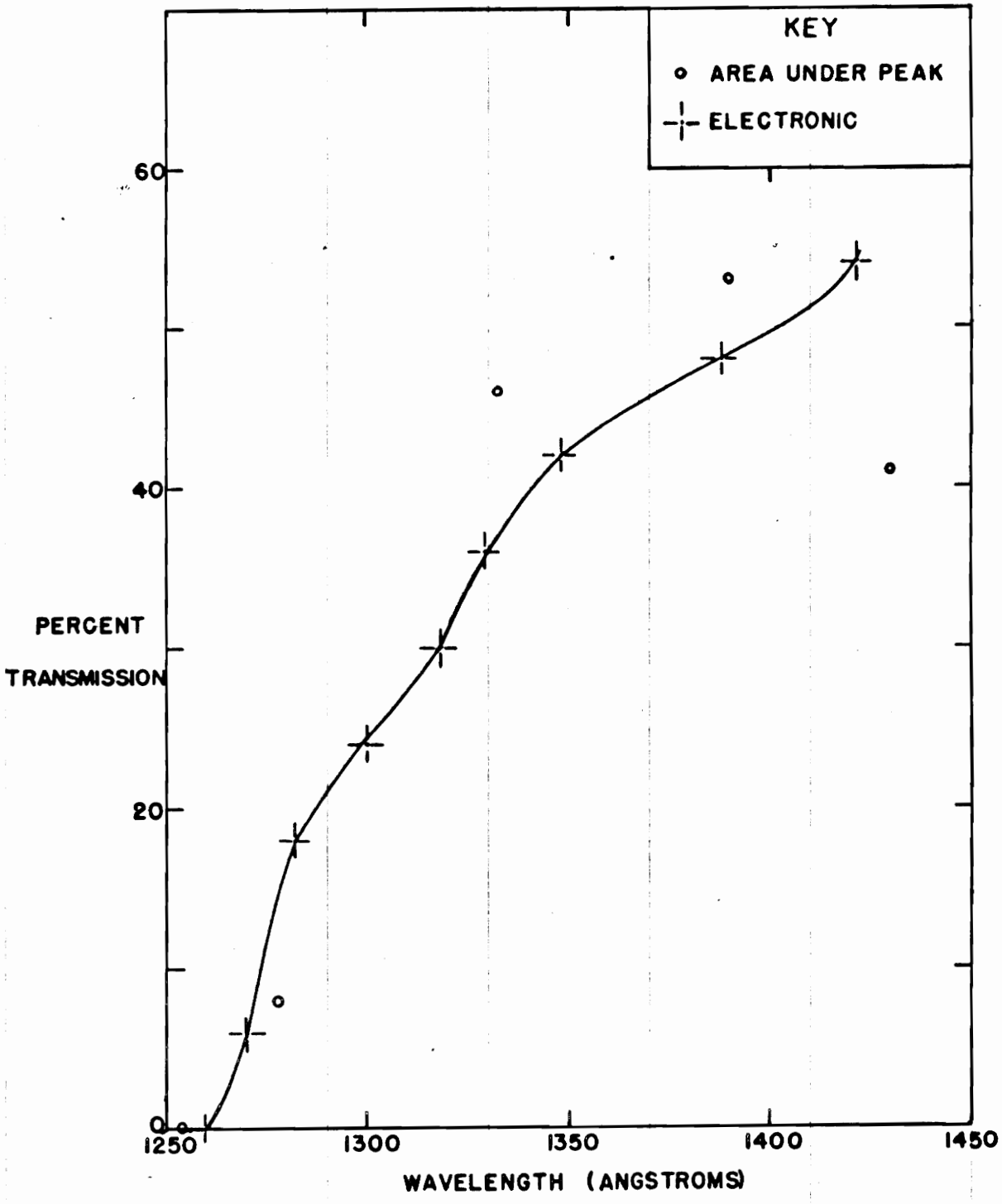

Figure 4.- Comparison of fractional transmission by peak area method to electronic method for sodium fluoride.

latter method, although having some instantaneous deviations from the actual value, can easily be averaged to show the true values.

In order to obtain an idea of the accuracy of the electronic method, a transmission run was made without any crystal in the upper aperture. The resulting curve showed how the recording and electronic system varied from the expected ratio of 1.00. Figure 5 shows the results of this calibration run. Each point represents the average value of the electronic ratio for a wavelength interval of 1 Å. 98% of the points are within 10%, and 79% are within 5%. The larger variations from the actual value of 1.0 are partially caused by the entrance and exit slits not being exactly parallel to one another. This results in an overshoot on the leading edge of a peak and an undershoot on the trailing edge.

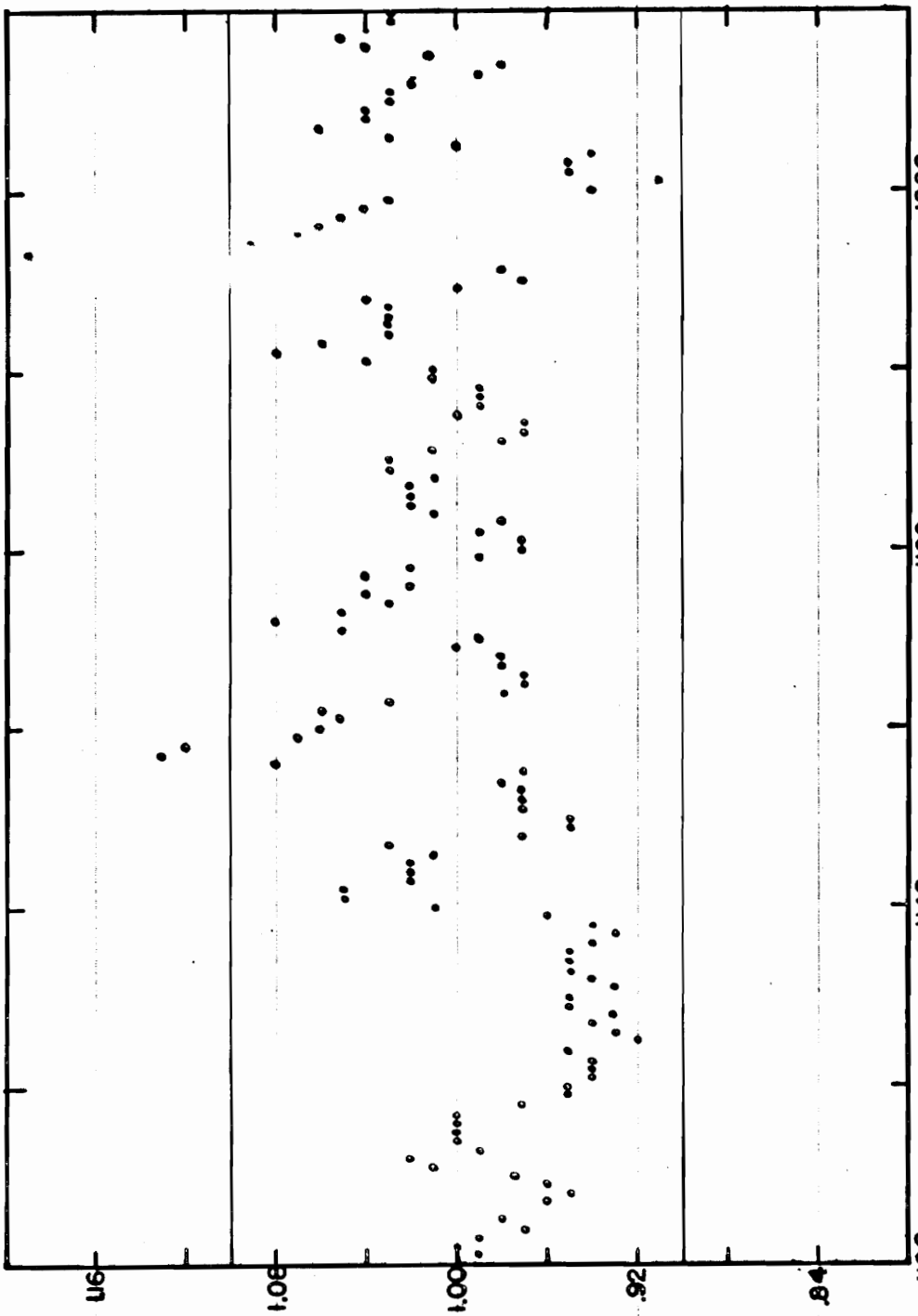

Figure 5 - Calibration check of electronic method.

IX. RESULTS

Thirty five crystals were run and their transmission cutoffs determined accurately for those transmitting in the extreme ultraviolet. The region of the transmission cutoff was noted for many of the other crystals but actual determination of their cutoff points was left to open air instruments. These data are shown in Table 1.

In addition, column 5 of Table 1 shows the results of the central image transmission determination using a photocell. This method of obtaining a fractional transmission for a broad region of the spectra was explained on page 15 and it provides a quick and efficient manner of determining whether a crystal transmits in the region of interest.

Comments on some of the individual crystals follow.

Magnesium Fluoride

Three samples of MgF_2 were tested. In cutting the boules it was found that the sample from Isomet was much more brittle than the other samples. Figure 6 shows a graph comparing the transmissions of the three. The cutoff is sharp and occurs between 1120 A and 1125 A for the three crystals. In an attempt to determine the cause of the different curve shapes shown in Fig. 6, a spectrochemical analysis was run on the three samples. The

Table 1 - Transmission Cut offs of Crystal Run

Crystal No.	Compound	Supplier (5)	Transmission Cutoff (1) (Angstroms)	Percent Transmission (1050-1800A)
1	CuI (2)	A	>2000	0
2	LaF_3 (3)	B	1500	3
3	BeO	C	1250	11
4	NaCl	A	1730	1
5	NaF	A	1265	54
6	NaCl	D	1705	4
7	KBr	A	2000	0
8	KBr	E	1995	0
9	KI	A	>2250	0
10	ADP	E	1770	2
11	SrF_2	E	1312	47
12	$NaNO_3$	E	~2500	0
13	KDP	E	1705	4
14	NaF	E	1272	32
15	CdS	E	>4000	0
16	CdSe	E	>4000	0
17	ZnS	E	>2000	0
18	ZnSe	E	>2000	0
19	IR-1 (2)	F	>2000	0
20	IR-2 (2)	F	>2000	0

Table 1 - Continued

Crystal No.	Compound	Supplier (5)	Transmission Cutoff (1) (Angstroms)	Percent Transmission (1050-1800A)
21	$CaWO_4$	G	(4)	0
22	CdF_2	G	1990	0
23	$CaMoO_4$	G	>2000	0
24	$SrMoO_4$	G	>3000	0
25	LaF_3	D	1300	42
26	MgF_2	D	1120	64
27	Al_2O_3		1430	17
28	MgF_2	A	1121	55
29	PrF_3	D	2105	0
30	Lucite		2000	0
31	CeF_3	D	2850	0
32	NdF_3	D	1727	1
33	MgF_2	G	1124	58
34	KD*P	G	1640	--
35	BaF_3	E	1339	--

1. A transmission of 3% of the maximum transmitted beam was used as the cut off point.
2. This was not a single crystal, but was a pressed conglomeration of many small crystals.

Table 1 - Continued

3. This crystal is very old, and its purity is doubted.
4. This crystal fluoresces for incident radiation greater than 1475 Å.
5. The suppliers are : A - Semi-Elements, B - Varian, C - North American Aviation, D - Optovac, E - Harshaw, F - Kodak, G - Isomet.

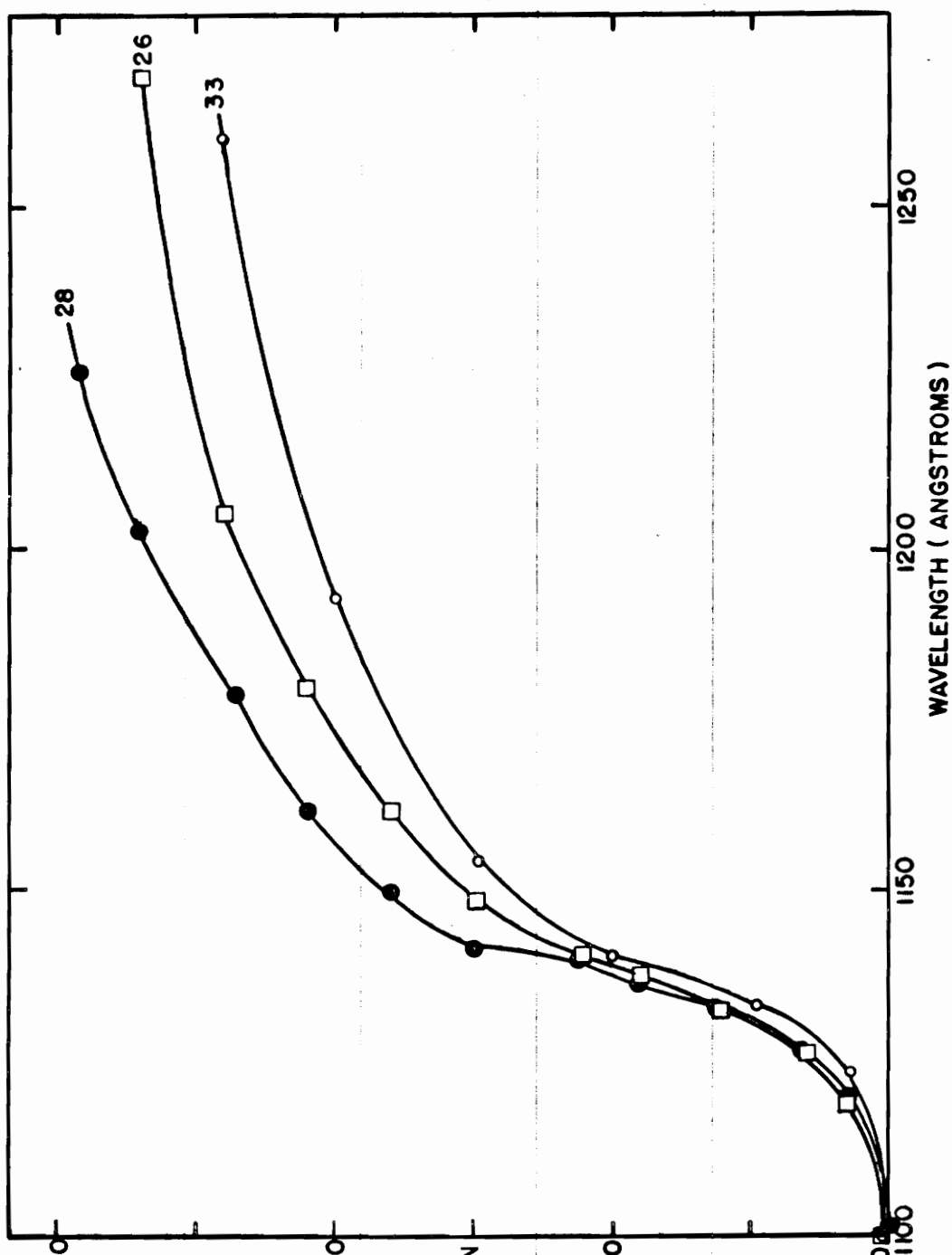

Figure 6 - Transmission of magnesium fluoride.

results of this analysis are shown in Table 2. As can be seen there are some slight differences in the abundances of some of the trace impurity elements present.

These transmission results vary considerable from those obtained by Duncanson (15). He obtained a transmission of 7% at 1100 A using a crystal twice as thick. The absorption band he found indications of at 1320 Å was not found in this work.

Sodium Fluoride

The two samples were cut and polished by the respective companies. Both samples were approximately the same thickness but the transmission of one sample was approximately one third of the other. However, the cutoffs were very similar. Only the data from the higher transmitting crystal are plotted (Fig. 4) and show a sharp cut off with a 3% transmission at 1265 A.

Rare Earth Fluorides

The four rare earth fluoride crystals which were examined were: lathanum fluoride, LaF_3; neodymium fluoride, NdF_3; praeseodymium fluoride, PrF_3; and cerium trifluoride, CeF_3. These four crystals all have hexagonal structure. Also, the lattice spacing constants (a_o and c_o) are approximately the same for the four. This is shown in the data listed on page 30.

Table 2 - Spectrochemical Analysis of MgF_2 Crystals Studied.

Element	Crystal No.		
	26	28	33
Mg	VS	VS	VS
F	VS	VS	VS
Mn	VW	VW	VW
Si	VW	Tr	VW
Fe	VW	Tr	VW
Ni	VW	Tr	VW
Cu	VW	Tr	Tr
V	Tr	Tr	VW
Ti	VW	--	--
Ca	Tr	Tr	Tr
Al	Tr	Tr	Tr
Cr	Tr	FTr	Tr
Ag	Tr	FTr	FTr
Na	FTr	FTr	FTr

KEY

VS - 10. - 100%
VW - .001 - .01%
Tr - .0001 - .001%
FTr - less than 0.0001
-- Not detected

	a_o	c_o	Transmission cutoff
CeF_3	7.11	7.27	2850
LaF_3	7.16	7.32	1300
NdF_3	7.02	7.19	1727
PrF_3	7.06	7.21	2105

It is apparent from the above data that the molecular weight of the specimen plays a major role in determining the transmission cutoff since the lattice spacings of the crystals listed are nearly the same.

All of these crystals were supplied in the form of 3/8 inch diameter boules. NdF_3 and PrF_3 appear to have absorption bands in their fractional transmission curves.

CeF_3 does not transmit in the desired region. However, since it was very similar in structure to NdF_3, LaF_3, and PrF_3, the approximate cutoff was located and the general characteristics noted.

Barium Fluoride

Barium fluoride has been investigated by several investigators (16,17). Their results are in excellent agreement with those obtained here. The BaF_2 used was grown, cut and polished by the manufacturer. It has a transparency of 3% at 1339 A. This compares with the cutoffs of 1335 (16) and approximately 1345 (17) found by other investigators. The difference between Heath's

value of 1335 and the one obtained here can partially be attributed to the definition of the cutoff transmission. Heath does not define what he means by "transmission limit." Chubb's value of 1345 was obtained 10 years ago and the purity of BaF_2 crystals has been improved in the interim. The purity of the crystal not only affects the cutoff point but also the slope.

Oxides

Only two oxides were previously known to transmit in the extreme ultraviolet, Al_2O_3 and SiO_2. One of these two, Al_2O_3, was checked and it showed a gradual cutoff to 1430 A. This is in good agreement with the value of 1425 quoted by Chubb (17).

Beryllium oxide was found to transmit down to 1250 A. It has a broad absorption band centered about 1315 A. The slope of the curve (see Fig. 8) is even more gradual than Al_2O_3. BeO is a very fragile crystal, making it useless as a transmission window.

Phosphates

Potassium dihydrogen phosphate (KDP), potassium dideutereum phosphate (KD^*P), and ammonium dihydrogen phosphate (ADP) provide an opportunity to observe the effects of small changes in the constituents on crystal structure and the cutoff.

The transmission cutoff value obtained for ADP agrees very well with that obtained by Heath (16). Both KDP and ADP exhibit very sharp cutoffs (Fig. 7). The KD*P crystal did not exhibit such a sharp cutoff. However, this lack of sharpness in the cutoff is probably due at least partially to the shape of the crystal and to the fact that the orientation of the optical axis was unknown.

Figure 8 illustrates all of the transmission data obtained for these crystals in the extreme ultraviolet.

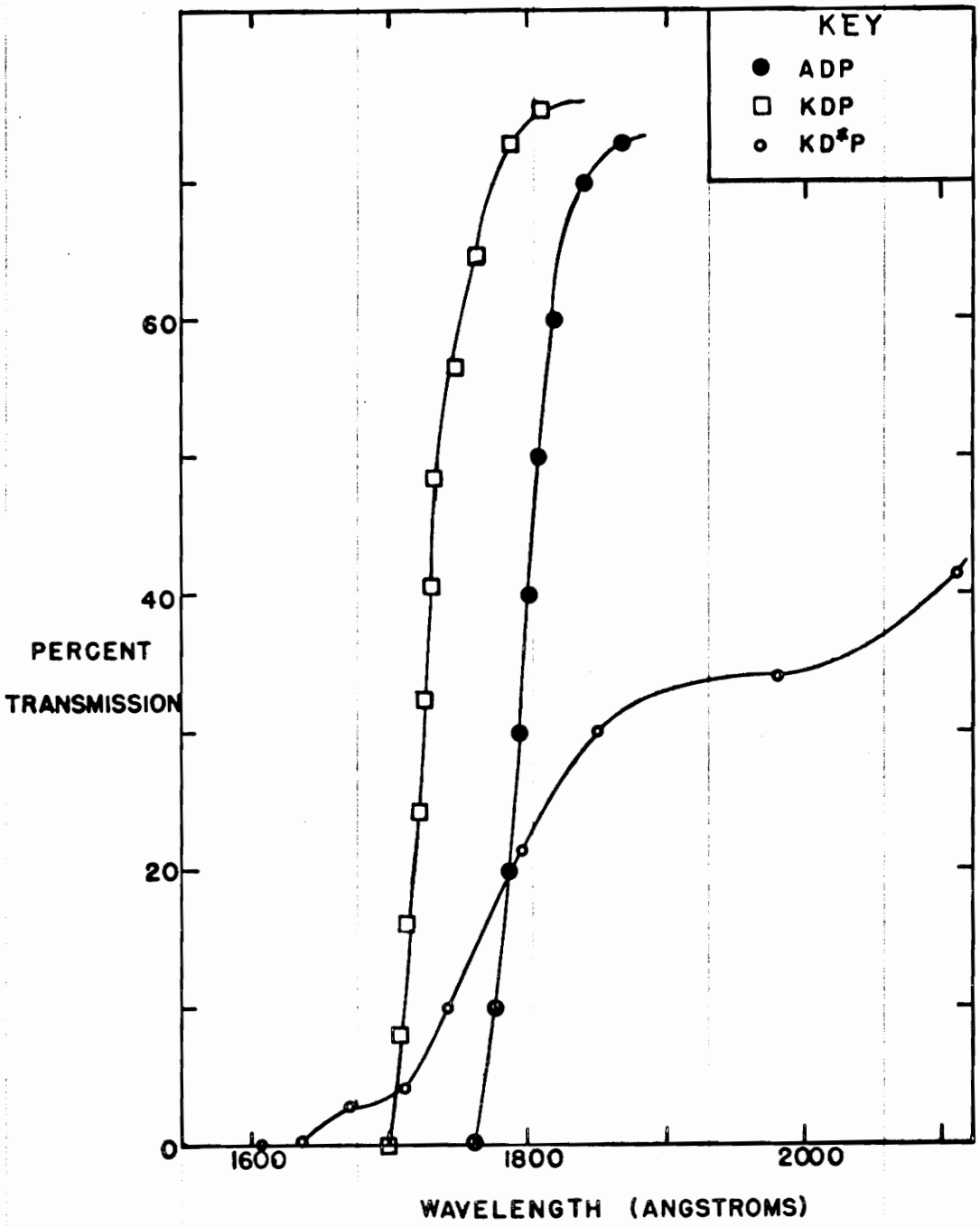

Figure 7 - Transmission of ammonium dihydrogen phosphate, potassium dihydrogen phosphate and potassium dideuterium phosphate.

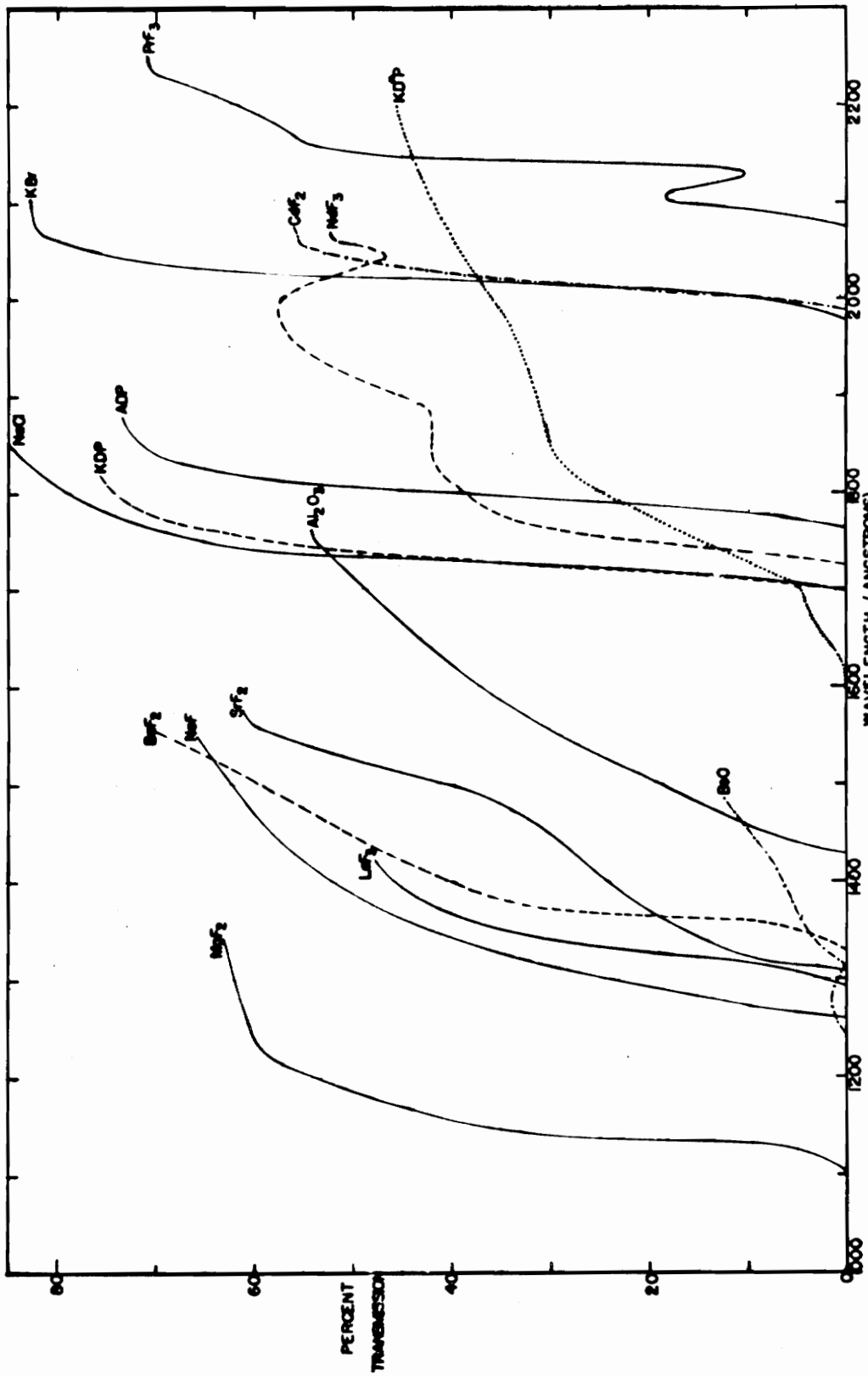

Figure 8 - Transmission of all crystals in region 1100 - 2100 A.

X. CONCLUSIONS

The number of crystals known to transmit in the extreme ultraviolet was increased to over twenty by the results obtained in this experiment. Table 3 is a listing of all crystals known to transmit in the extreme ultraviolet. The crystals are placed in order by transmission cutoff and include not only those from Table 1 which lie in this region but also those previously listed by Chubb (2) and Heath (16).

Crystals which could be used for a comparative study are MgF_2, CaF_2, SrF_2, BaF_2 and CdF_2. These crystals are all cubic and Wyckoff (19) classifies them as being of the CaF_2 type. For the most part all of the cutoff curves for these crystals are sharp, except possibly SrF_2, with the cutoff points increasing in wavelength with increasing mass of the molecule. Similar results may be noted for NdF_3, CeF_3, PrF_3, and LaF_3. It would be interesting to ascertain if other crystals of similar chemical compounds would demonstrate similar behavior.

These data also show a higher degree of reproducability than had been seen in the past (6). This shows up particularly well in the MgF_2 crystals and is a result of better growth and preparation of the samples.

Table 3 - Transmission Cut offs of Crystals Known to Transmit in the Extreme Ultraviolet.

Crystal	Cutoff Point[1]	Remarks
LiF	1050[2]	Sharp
MgF_2	1120	Sharp
CaF_2	1220[2]	Sharp
BeO	1250	Very Gradual
NaF	1265	Sharp
LaF_3	1300	Sharp
SrF_2	1312	Moderately Sharp
BaF_2	1339	Sharp
AL_2O_3	1430	Moderate
Topaz	1550[2]	Moderate
SiO_2	1560[2], 1580[3]	Gradual
Gypsum	1620	Moderate
KD*P	1640	Gradual
NaCl	1705	Very Sharp
KDP	1705	Sharp
NdF_3	1727	Sharp
KCl	1750	Sharp
ADP	1770	Sharp
CdF_2	1990	Sharp
KBr	1995	Very Sharp
Calcite	2030[3]	Sharp
PrF_3	2105	Sharp

(1) See footnote 1 of Table 1 on page 24.

(2) This value is from Table 3 of reference 2.

(3) This value is from Table 2 of reference 16.

XI. ACKNOWLEDGEMENTS

The author wishes to express his sincere gratitude and thanks to his advisors Dr. A. Keith Furr of Virginia Polytechnic Institute and Dr. Talbot A. Chubb of the U.S. Naval Research Laboratory for their many hours of assistance in the completion of this thesis. The financial support and use of the equipment belonging to the U.S. Naval Research Laboratory is deeply appreciated. Thanks are also due Mr. Julian Siomkajlo and Mr. Gilbert Fritz of NRL for their stimulating discussions, and to Mr. Charles Herbert for his quick service in cutting and polishing the crystals.

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XIII. VITA

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ABSTRACT

The Transmission Characteristics of
Some Optical Crystals in
The Extreme Ultraviolet

by

Arthur T. McClinton, Jr.

The transmissions of some optical crystals were determined for the vacuum ultraviolet using a Seya-Namioka monochromator. The characteristic low wavelength cutoffs were determined for crystals transmitting in the region 1050-2000 A. The crystals so studied are: MgF_2, NaF, LaF_3, SrF_2, BaF_2, BeO, Al_2O_3, KD^*P, KDP, ADP, NaCl, NdF_3, KBr, CdF_3 and PrF_3. The techniques employed in the experiment and the data obtained will be presented.