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DESIGN, FABRICATION & PERFORMANCE OF AN ELLIPSOIDAL SPECTROREFLECTOMETER

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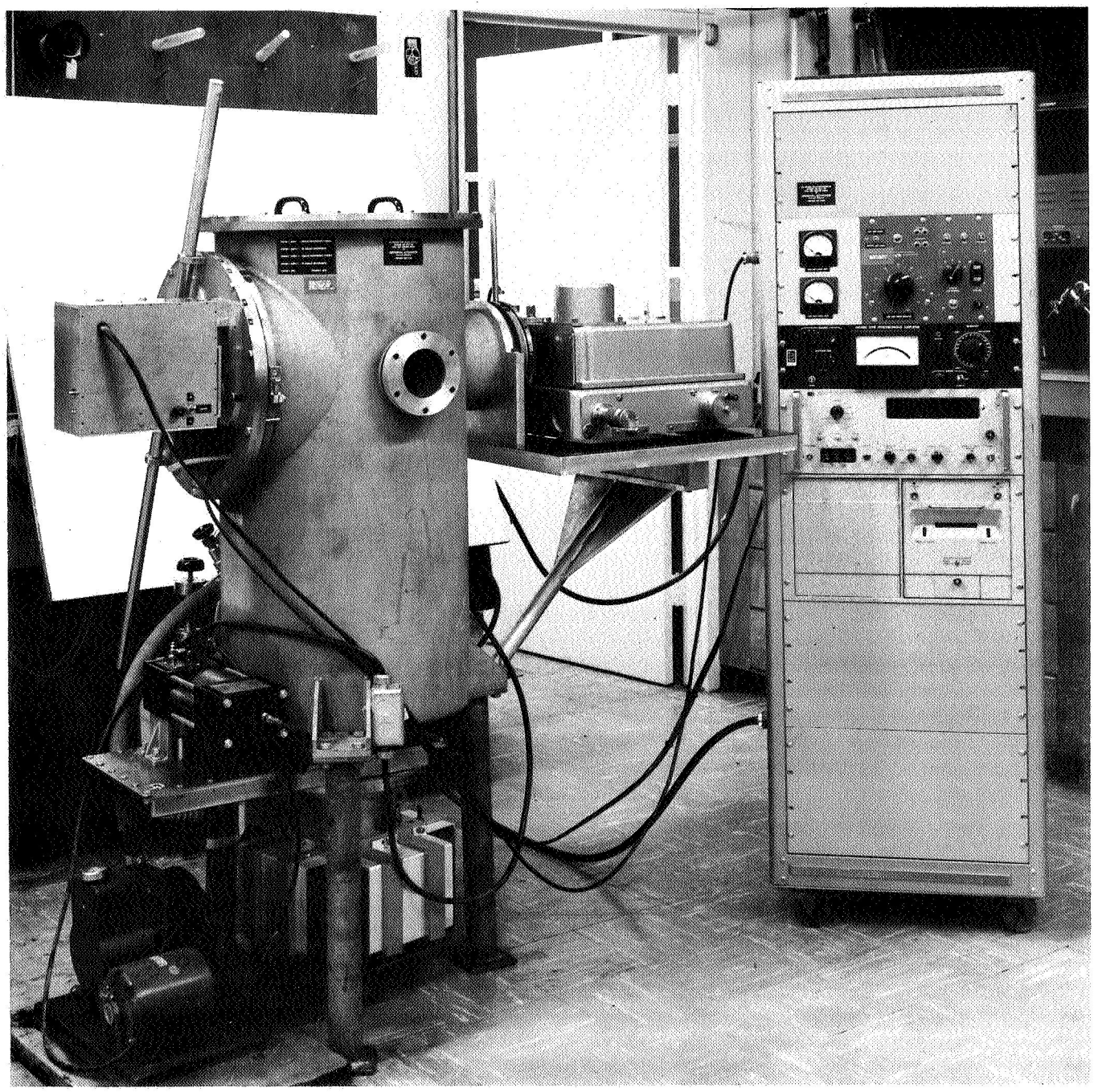

FOREWORD

The writer wishes to express thanks to the large number of people who contributed to the development and construction of this far infrared spectroreflectometer. First, the initial survey analysis that resulted in the selection of the ellipsoidal device was done by Mr. Werner Brandenberg. Mr. Sam Pountney made the general layout and tank design. Mr. Torgeson provided the detail design of the chopper, sample system, mirror mounts, etc., and performed most of the machine work. Mr. Bill Corbin provided large assistance in checkout and correction of difficulties. Mr. R. S. Gilson assisted in all phases of the operation and performed the experimental measurements on the samples. The author further wishes to thank the very large number of people who made suggestions, some usable some not, but all made in good faith and with kindly intentions, on how to improve the instrument.

The construction of the instrument has been a difficult and taxing undertaking. It seems now to the writer that it is perhaps too much to expect for a superior instrument to result directly from a design, worked out on paper, and translated directly into a manufactured instrument. The design is all important if the instrument is ever to be a good one but a new instrument, will evolve step by step as problems arise and are solved. Problems arose and were solved on the reflectometer. The data obtained indicates that good performance can be obtained from the instrument. As experience is gained, the instrument can be further improved.

For some years, the personnel of the Radiative Properties Laboratory, General Dynamics Convair, Space Science Section have been considering the trade-offs and optimizations for this type of an instrument. A theoretical analysis was made of the focusing properties of an ellipsoid for such an application. These background studies culminated in 1966 in the design of a far infrared spectroreflectometer which provides much of the coverage required and which has "growth potential" for considerably greater coverage.

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1.0 INTRODUCTION

As space vehicles become more sophisticated and complex, the requirements increase for refined and extended radiative property measurements. Instrumentation to perform these measurements is not available commercially and to provide the design engineer with the required data, it is necessary to design, and construct instrumentation. A requirement exists for a general purpose device for measuring in a vacuum the absolute total hemispherical reflectance as a function of wavelength, specimen temperature (especially low temperature) and angle. Wavelengths of interest extend from the ultraviolet (UV) beginning at about 0.2 μ and extend through the visible into the infrared (IR). The temperature at which a surface is to be used determines the extent of the coverage required in the IR. For room temperature work, data out to 30 μ is usually satisfactory. For work at low temperatures, greater IR wavelength coverage is required. Angular coverage from close to normal (0°) to near grazing (90°) incidence is required. This is particularly important in that it allows the determination of directional emittance which, on integration over 2π steradian provides total hemispherical emittance as a function of wavelength. Most total hemispherical data presently available was obtained calorimetrically and there is a real need for the data as a function of wavelength. An instrument which will function to obtain data over the complete useful ranges of wavelength, angle, atmosphere (vacuum etc.) and temperature to meet all the requirements of space exploration is, of course, desirable but represents a "big order".

The far infrared spectroreflectometer delivered to NASA provides wavelength coverage from 2.5 to 90 μ; the potential is from 0.2 μ to 360 μ.

Angular coverage is now 6° to 76°; potential 6° to 80°. The sample is now maintained at tap water temperature; elevated temperature measurements seem perfectly feasible; these measurements will probably be limited by the error resulting from heating of the source by radiation from the sample. Reduced temperature measurements are also possible. The problem is to maintain a sample at a known reduced temperature when a radiant flux of some 50 watts is incident on a 1" sample disc. [The direct (as distinct from the reciprocal) operating mode would be highly advantageous in this case.]

2.0 COMPONENT DESCRIPTION

2.1 ELLIPSOID

In the comparative analysis of the focusing properties of the hemisphere versus those of a hemi-ellipsoid, it was determined that a hemi-ellipsoid generates a much better (smaller) image than does a hemisphere and accordingly the use of an ellipsoid was planned for the spectroreflectometer. It was determined that grinding of a hemi-ellipsoid of the type required to be extremely difficult. After some negotiations and discussions, a hemi-ellipsoid of the dimension shown in Figure 2.1-1 was procured.* The hemi-ellipsoid was ground initially in such a way that an ellipsoid of revolution which was symmetrical with respect to the semi-major axis was formed. This shape was then cut in half along the plane passing through the semi-major axis. The two quarters of the ellipsoid were then repositioned to form the desired hemi-ellipsoid as shown in Figure 2.1-1. There is a very thin crack or line created by joining the two halves of the hemi-ellipsoid, but this represents such a small percent of the area of the ellipsoid surface that it does not cause serious error. Aluminum was evaporated on the intersurface of the ellipsoid since it provides high reflectance in all regions of interest and is reasonably resistant to corrosion.

The source is placed at one of the foci of the hemi-ellipsoid, the sample at the other. The source radiance thus illuminates the sample. The isotropy of the illumination is discussed in sections below under 2.3 Source.

*Perkin-Elmer Corporation, Costa Mesa, California

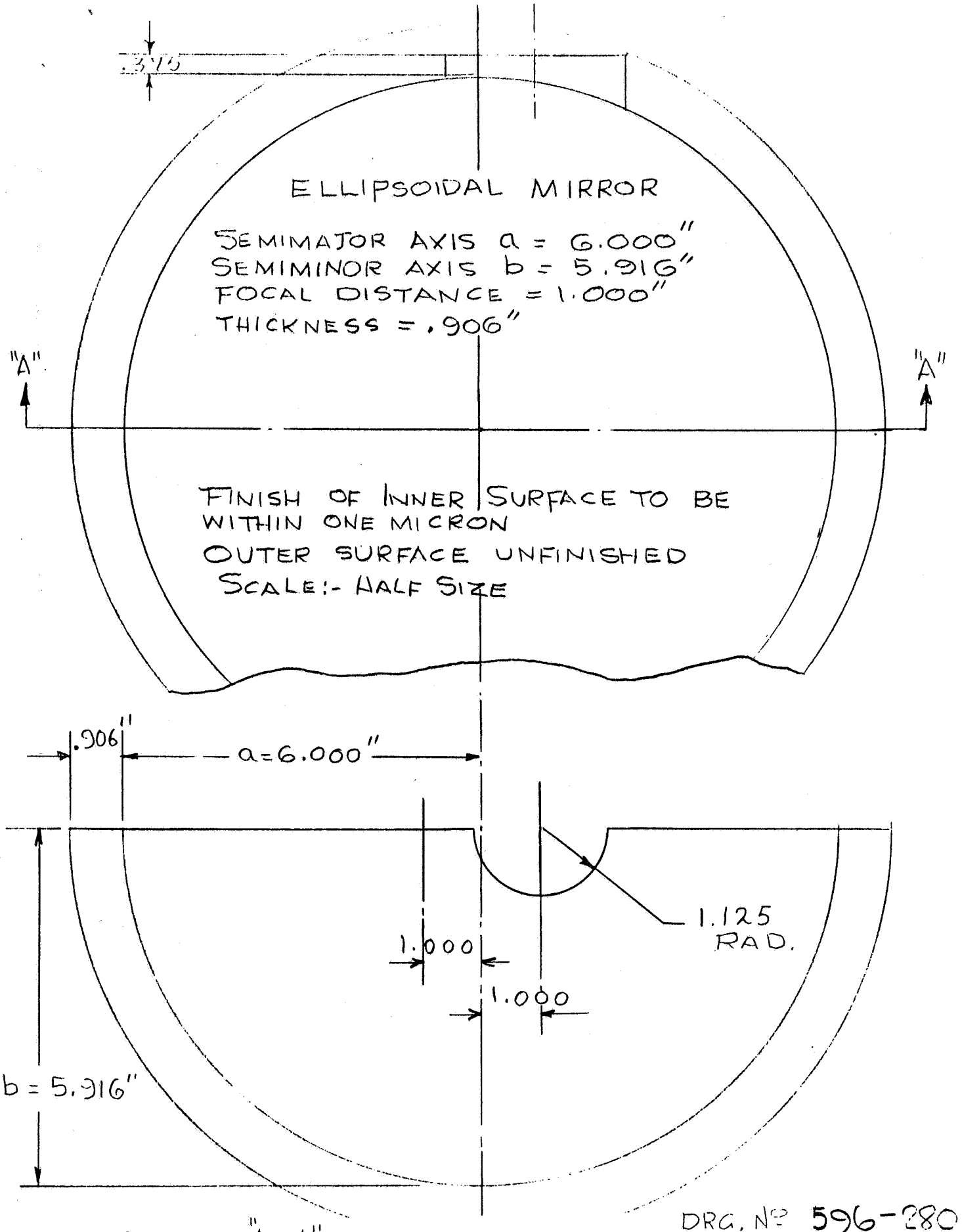


FIGURE 2.1-1 HEMI-ELLIPSOID

The first ellipsoid showed excellent optical properties. Focusing of a point at one of the foci to the other focus was found to be excellent. This is the ellipse which was delivered to NASA/Ames.

2.2 MECHANICAL

Reference is made to Figures 2.2-1 and 2.2-2. The system is enclosed in a large vertical cylindrical tank which mounts on three legs. The source rotation mechanism consists of a large "rotation cylinder" which " necks down" into a conical section to a bearing ring. The cylinder with its axis horizontal fits into the vertical main tank as shown. It is suspended on both ends; on the small end by a roller bearing and on the large end by machined bearing surfaces. The thrust which will be present when the system is under vacuum is borne by a large ball bearing ring. Vacuum seals consisting of double "O" rings with provision for pumping between the seals are provided.

The plate which closes the open end of the cylinder holds the ellipsoid, source, sample, and chopper system. The system is kinematically mounted to the cylinder and vacuum sealed with "O" rings. The plate has a cavity with a semi-circular cross section which projects into the cylinder. The flat part of this cavity is directly under the ellipsoid and serves to support it. The source and sample systems are also supported on the flat top of the cavity. The cavity serves to allow access to the sample system and to supply cooling water and source power directly under the ellipsoid. The rotation required for obtaining angular data and the 100 percent datum is accomplished by turning the rotation cylinder to which the plate cavity, ellipsoid, chopper, etc., are attached.

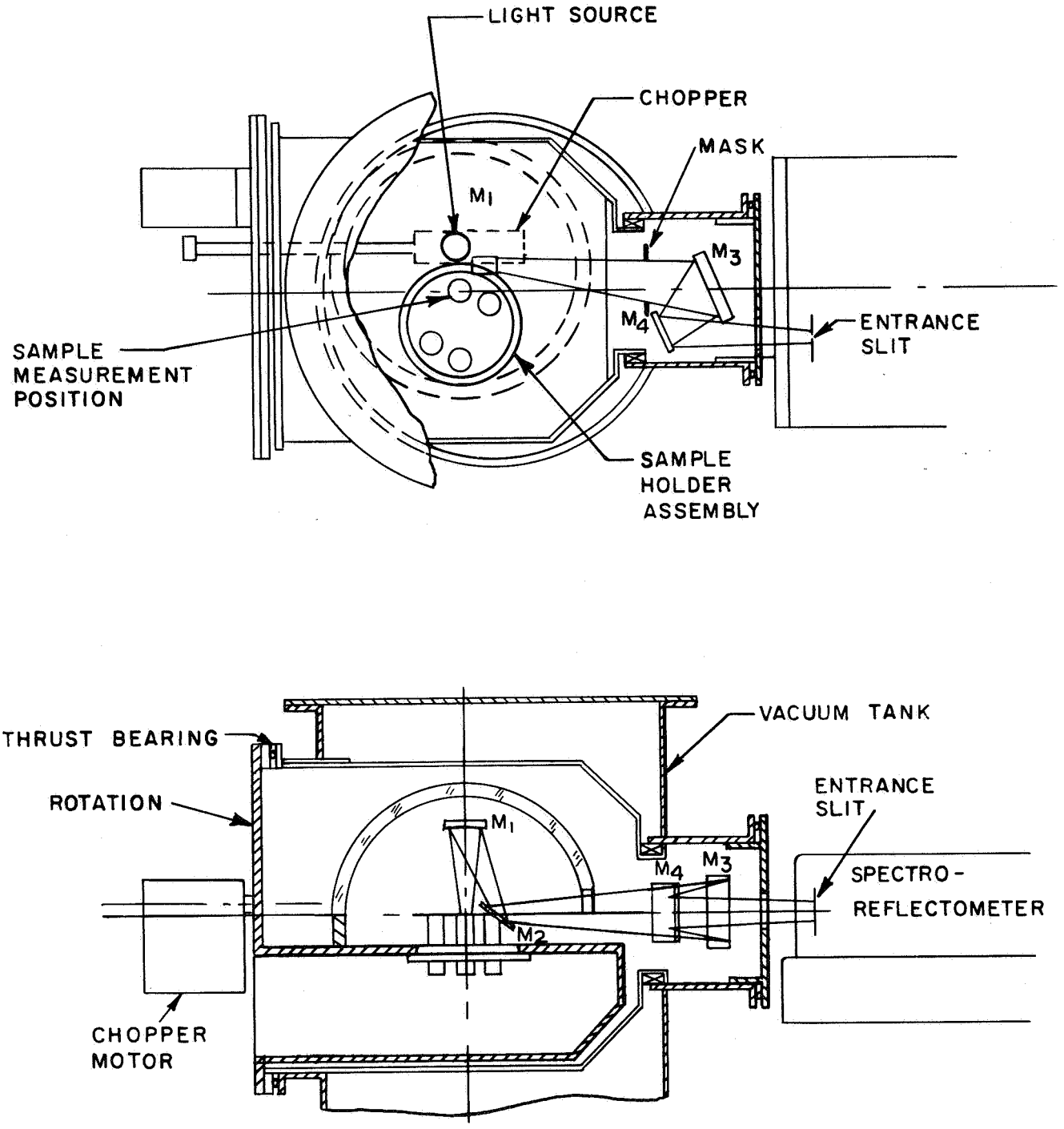


FIG. 2.2-1 : SPECTROREFLECTOMETER - SAMPLE IN MEASUREMENT (I) POSITION

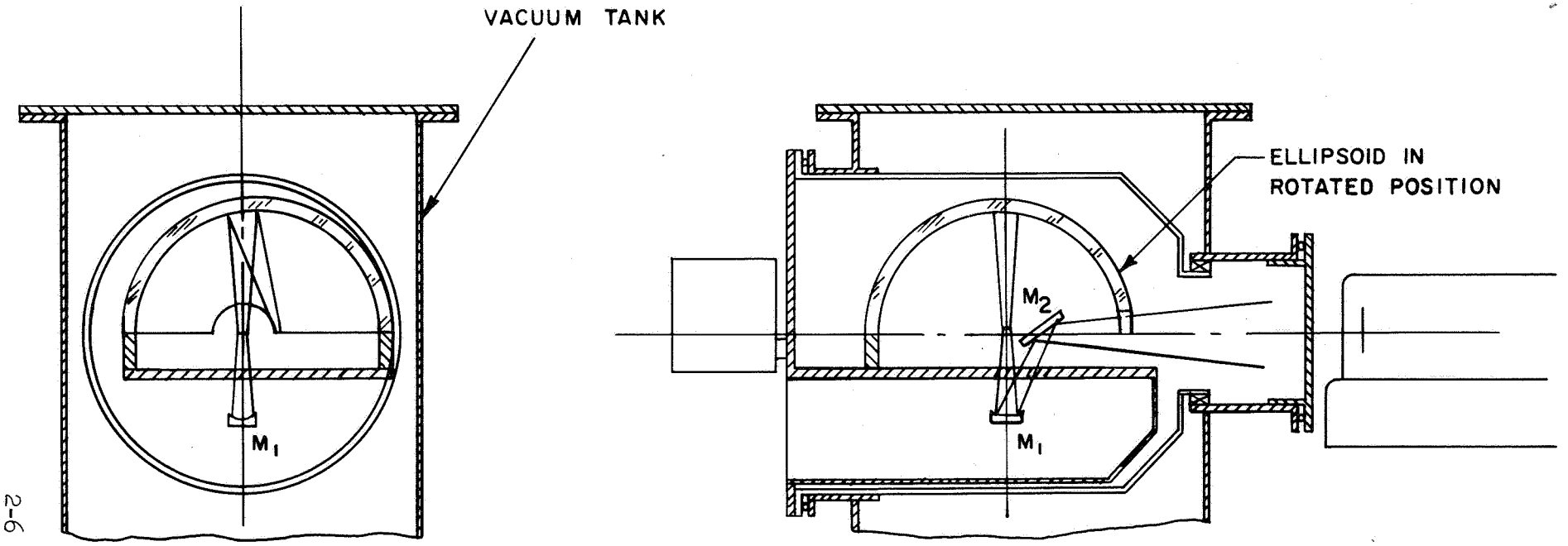


FIG. 2.2-2 : 100 % (I_0) MEASUREMENT POSITION

The top of the main vacuum tank is sealed off with a large flange and made vacuum tight with "O" ring seals. Below the rotating cylinder is a space for a boost-vac vacuum system of the type generally used in conjunction with vac ion systems. The port for the attachment of the vacuum system is at the base of the main vacuum chamber.

The monochromator is supported outside the main vacuum chamber on a plate which fastens to the main tank.

2.3 SOURCE

In the field of infrared spectroscopy, a glow bar or Nernst glower is ordinarily used in the infrared region from 2.5 to 50 μ. For operation past 50 μ a mercury arc such as the type H4* is sometimes employed. Both the heated quartz envelope and the emitting mercury contribute to the radiation. It was initially planned to use a glow bar source for the instrument but on investigation it was determined that the tungsten carbide comprising the glow bar element is subject to a certain amount of evaporation when operated in a vacuum. The evaporated material would, of course, coat the ellipsoid surface and degrade its performance and thus the use of a glow bar was not desirable. After some experimental study and experimentation, it was decided to use as a source a graphite thimble heated on the inside by a tungsten filament. The graphite thimble or dome is placed over a tungsten filament as shown in Figure 2.3-1. This type of source has been demonstrated to work very well for extended periods of time in a vacuum with no graphite evaporation difficulty. The unit also works satisfactorily in a controlled atmosphere for a more limited period of time. The atmosphere must be free of water and oxygen in order for the unit to function satisfactorily.

The use of a tungsten filament heated graphite hemisphere was preferred over the use of the carbon rod source since a carbon rod would require large leads to carry high current at low voltage which could cause noise problems in the amplification and detection systems. Further, the tungsten lends itself to a dome shape which is ideal for this application.

*General Electric Company

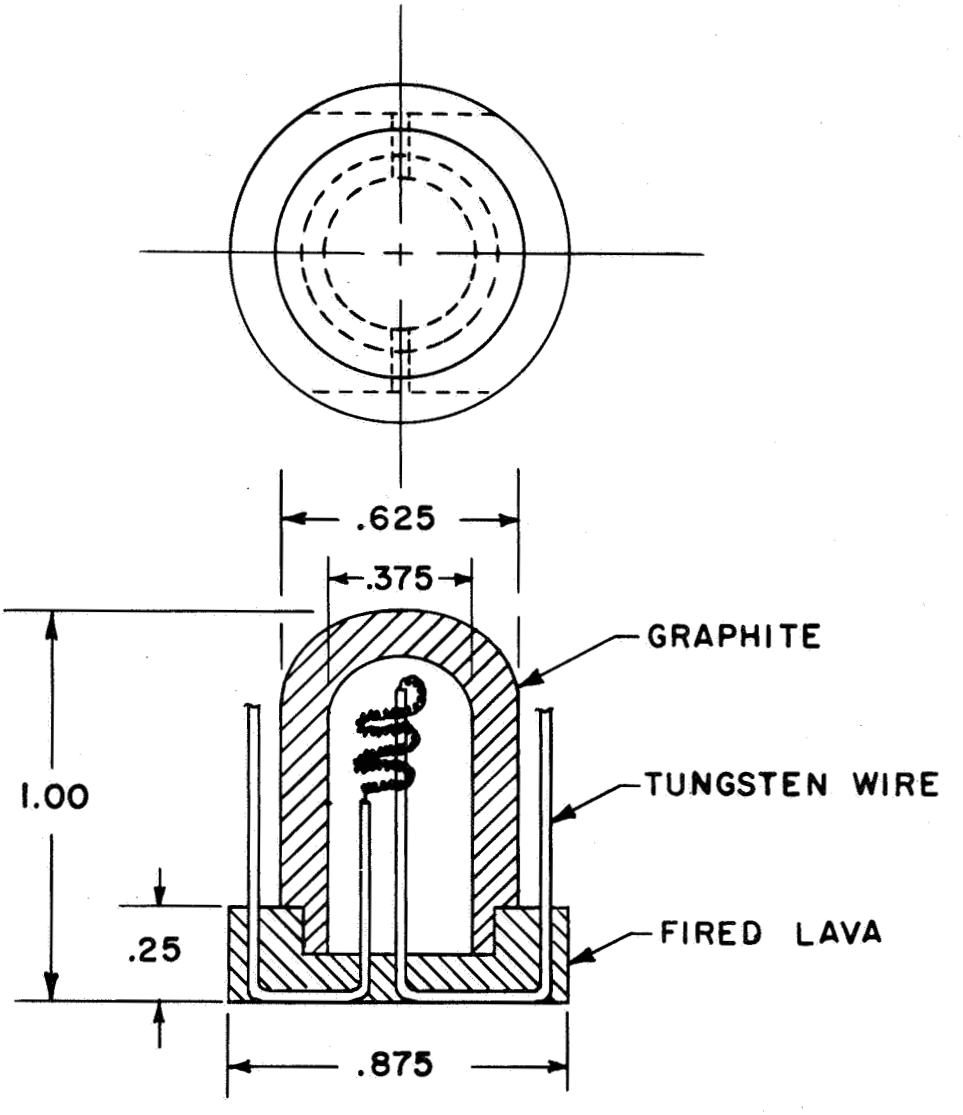


FIGURE 2.3-1 TUNGSTEN HEATED GRAPHITE SOURCE

The instrument is also provided with a H4 mercury arc for the operation in the region from .2 to 2 μ and from 50 to 100 μ. The two sources may be interchanged without breaking vacuum by rotating the source holder. The source holder is rotated by reaching into the sample space under the ellipse and rotating a device in the source cavity with a small rod. There are some reservations regarding the use of the mercury arc and these will be explained in the section on Operation, 4.0.

The area of the source is of importance since it bears on the isotropy of the radiation striking the sample. Radiance for the 100 percent datum is taken from directly below the sample position viewing the same part of the ellipse viewed by a specular sample. This radiance value (watts/cm^2 steradian) should be equal to the radiance striking the sample area from any direction in the 2π steradian angle over the sample.

For many situations, calculation of the isotropy of the radiation striking the sample is very complex. In one case however, it is straightforward; namely the case in which all rays from the part of the sample viewed by the spectrometer are reflected to and are completely encompassed by the source. Consider that a mercury source is placed at the detector position in the monochromator. The green line is run "backward" through the monochromator, comes out the entrance slit, goes through the optical system and is imaged on the sample. Assume that the sample is a perfectly diffuse reflector. The light striking the sample is diffusely reflected from the sample and specularly reflected by the ellipsoid onto the source. If all of the image at the source position is within the boundary of the source, i.e., falls on the source, any and all solid angles over the image

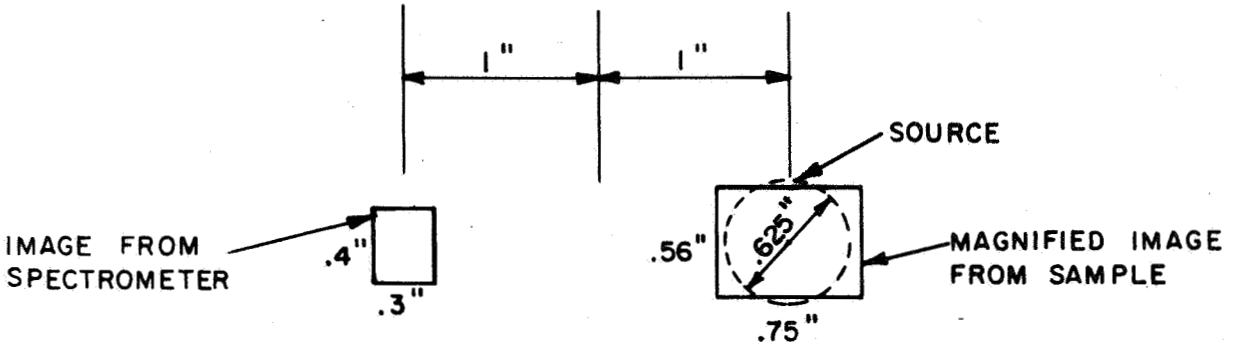


FIG. 2.3-2 : IMAGE MAGNIFICATION ON THE SOURCE

on the sample will be filled with radiation from the source and thus the radiation striking the sample from the source will be the source radiance as attenuated by the reflectance of the ellipsoidal reflector. The illumination of the sample area used will then be isotropic if the source is isotropic and the reflectance of the ellipsoid surface is uniform.

The size of the source and the size of the image of the spectrometer on the sample then becomes important. The image is 0.3" by 0.4". The magnification with the ellipsoid is 1.4 in the "length" direction and 2.5 in the "width" direction. This magnification shown is based on calculations made by a member of the laboratory during the early consideration of the ellipsoidal reflective device (Reference 1).

The magnification depends on which area of the ellipsoid is being used to reflect the light; the least magnification or demagnification occurring when the central part of the ellipsoid above the sample is used. Applying the magnification criteria, a source size as shown in the drawing below, Figure 2.3-2, is required if the source is planar and lies on the equator. The center of the source will be used most of the time since samples, especially in the IR, tend to "bunch" the reflected energy in and around the specular angle which will give essentially a 1 to 1 source sample image relationship. However, if the sample is a diffuse reflector, or if angular measurements are made, then areas other than the "overhead" areas of the ellipse are used and a larger source is required. Larger, rather than greater area is used advisedly here. The rays of light which require a larger source as shown in Figure 2.3-2, are reflected from the lower parts of the ellipse, i.e., nearer the equator. These rays tend to travel across the center point of the source position, however, are "too high or too low" to strike the

point that would provide a one to one image relationship. Accordingly, rather than making the detector area large, it is possible to keep the detector small by allowing it to project a short distance above and a short distance below the equator. To put it another way, it is the requirement to have the source intercept all of the "backward" green line rays reflected from the sample. This is most easily done by making a three-dimensional source rather than making a large area flat source. This device has the further advantage that the critical placement of the source and the sample at the equator of the ellipse is no longer required, since there is a range of positions of the source and sample near the equator where full isotropic illumination is obtained. The use of a three dimensional source does require that a low shield be provided to eliminate direct radiation from the source to the sample; such shields have been provided. The gray body graphite source provides a three-dimensional or volume source. The diameter of this source is indicated in Figure 2.3-2 in dotted form.

It was also noted that the reflectance of the ellipsoid surfaces must be uniform. The ellipsoid is uniformly polished and illuminated and the reflectance should be essentially uniform over its entire surface. Incidentally, this is a distinct advantage of this system as compared to the two parabola systems. The rays from the source to the ellipse to the sample all are reflected at near normal incidence irrespective of whether the sample is diffuse or specular or whether angular measurements are being made. With the double parabola system, the rays are reflected at a variety of angles and reflectance on the aluminized parabola surfaces is a function of angle. The uniformity of irradiation would be degraded in proportion to deviations in reflectance and polarization effects introduced.

2.4 TRANSFER OPTICS

The radiation from the source is reflected by the ellipsoid and illuminates the sample. Radiation reflected from the sample is received by the small spherical mirror M1, (see Figures 2.2-1 and 2.2-2) over the sample, reflected to the M2 diagonal, thence to the toroid mirror M3, and finally, to the monochromator slit by plain mirror M4. In leaving the tank the beam passes through a transparent window. Different windows are required for transmission in different wavelength regions and are shifted into place by a rotating plate. The aperture of the monochromator is thus filled with the radiance reflected from the sample.

The slit of the spectrometer is imaged on the spherical mirror over the sample; the grating of the instrument is imaged on the sample. When narrow slits are used, the image on the small spherical mirror is narrow and if it were desired to limit the "shadowing" error, i.e., the error due to the mirror blocking part of the radiation from the ellipse to the sample, a narrower spherical mirror could be used. For wavelengths out to 40 μ this is entirely possible.

To make angular measurements it is necessary to make the image narrow on the sample. This may be done with a mask in front of the toroid. A mask is provided. The masking could also be done in front of the off axis parabola in the spectrometer or in front of the grating.

The transfer optics are designed to accommodate the full aperture of the monochromator as determined by filling the exit slit aperture with light from a mercury arc. In developing the instrument, it was found that the diagonal mirror past the exit slit was not of sufficient size to allow use of slits wider than about 1.6 dial (dial x 5 mm = slit opening) without the beam falling off the diagonal mirror or Restrahlen plate. The result of

this is a loss of some energy at slits above 1.6 dial (1.6 dial = 8.0 mm). The mirrors in the optical transfer system are fixed and in ordinary operation do not rotate. There is a mechanism for rotating M1 and M2 but this is used only for alignment and checking as discussed under 3.0, Alignment.

Measurements at various angles may be made by rotating the ellipsoid, sample, and chopper system about an axis lying in the sample plane and passing through the center of the sample and perpendicular to the major axis of the ellipsoid.

The 100 percent datum is obtained by rotating the ellipsoid 180° from its sample measurement position after shifting the sample out of position as shown in Figure 2.2-2. Thus the radiance value of the radiation incident on the sample is directly recorded with exactly the same optical system which was used to measure the reflected radiance. The mechanical arrangement for accomplishing this is discussed in detail under Mechanical Design, Section 2.2.

2.5 CHOPPER

The sample receives light and absorbs part of it and reflects the remainder. The reflectance is obtained by determining the ratio of the reflected light to the incident light.

$$\rho = \frac{I}{I_o}$$

I_o = incident beam intensity

I = reflected beam intensity (1)

The sample will also emit energy since it will be at some temperature above absolute zero. Unless an instrument for the measurement of directional reflectance is properly designed, this sample self-emission degrades the accuracy of the reflectance data and the seriousness of this degradation increases as the wavelength and sample temperature increase. Since long wavelength coverage was a design criterion for this instrument, special attention to this problem was required. The following table indicates the ratio of the emitted flux from a 1400°K blackbody (the approximate temperature of a globar source) to the emission from a black sample at T_s (T_s = 300°K, 350°K, 400°K, 450°K).

TABLE 2.5-1

Ratio of Blackbody Flux at 1400°K to Blackbody Flux at Sample Temperature T_s

T_s / λ Microns	300°K	350°K	400°K	450°K
2	1.5×10^8	5.1×10^6	3.8×10^6	5.3×10^5
5	2000	520	190	85
10	68	34	19	13
20	15	10	7.5	5.8
50	6.8	5.7	4.7	4
100	5.8	4.7	4	3.5

It is immediately evident that between 5 and 10 μ the self-emission error will become significant. It is so serious at longer wavelengths that a basic design which eliminates the effect is required.

A well-established method to eliminate sample emission effects in IR absorption spectroscopy is to chop the source radiation between the source and the sample. This method has been adapted to the directional reflectance instrument and is an integral feature of the design.

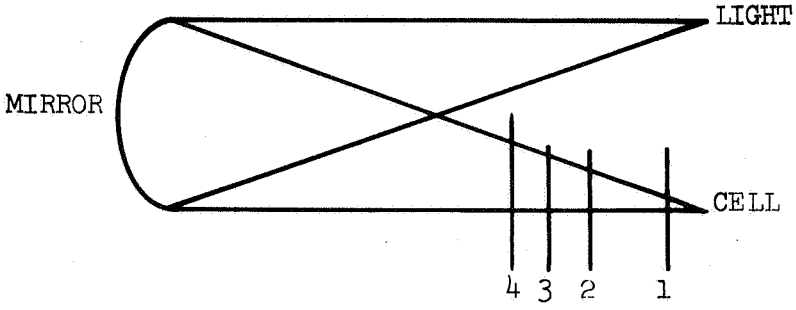
The designing of a chopper to operate between the source and the sample in an ellipsoidal reflectometer presents some mechanical design difficulty. Ordinarily, the chopper for an infrared spectroscopic system is placed close to the source or at or close to a point of focus, so that the cross section of the beam which is chopped is of relatively small size. Using the ellipsoid design with the source at one of the foci and sample at the other focus chopping between the source and samples makes necessary a chopper which is capable of intermittently passing and blocking light radiating from the source into a hemisphere. A system was designed wherein the chopper blade is in the form of a hemicylinder. The source lies within the cylinder and the chopper hemicylinder blade revolves around the source. Thus, when the blade is out of position, the source is for practical purposes completely exposed to the hemisphere. And when the chopper is in the closed position, the beam is completely blocked from the ellipsoid.

The fact that hemispherical radiation rather than just a small beam has to be chopped causes an amplification problem. Consider the optical path between the sample position and the source if the sample is a specular reflector (or if the instrument is in the position to

measure the 100% datum). The beam "seen" by the spectrometer is relatively narrow above the source. If the sample is a diffuse reflector, the light from the source will radiate in all directions and the sample will receive light from all directions over a hemisphere. The beam which is detected in the spectrometer is very wide. It is evident that chopping the beam in the specular or 100% mode will be rapid, i.e., the beam will go from completely blocked to completely open and vice versa in a relatively small fraction of the rotation cycle of the chopper. But for the diffuse sample measurement, the chopper will require more time to completely block or to completely clear the angles into which the detected radiation is emitted. With a chopper which is rotating at constant velocity, the signal from a specular sample will be of considerably different shape from the signal received if a diffuse sample was being measured. While it is true that the "area under the curves" is the same, it has been learned* that the response of these two types of signals would be different. A test was performed by Brower Laboratories. The test sheet furnished by Brower Laboratories is reproduced in Figure 2.5-1. It was found that errors as high as a few percent occur. This is, of course, an unacceptable error, even though it is true that in most cases the error would be no where near this large since most samples are approximately specular or if not specular at least have most of their energy "bunched" in a cone around the specular direction and thus the angular discrepancy between the 100% and the sample would not be so great. But it will be periodically necessary to study samples which

*Mr. Brower, Brower Laboratories, Inc., Westboro, Massachusetts.

A TEST WAS PERFORMED TO DETERMINE THE EFFECT OF CHOPPING ANGLE ON OUTPUT ENERGY AS DETECTED BY A THERMOCOUPLE AND AMPLIFIED BY A BROWER LABS. MODEL #129 SYNCHRONOUS AMPLIFIER.



CHOPPER POSITION	CHOPPING ANGLE	LOSS OF ENERGY %
1	20°	-
2	45°	2.0
3	60°	3.0
4	90°	8.0

FROM POSITION #1 TO POSITION #4 THE OUTPUT ENERGY FROM THE CELL DECREASED BY A FACTOR OF 8%.

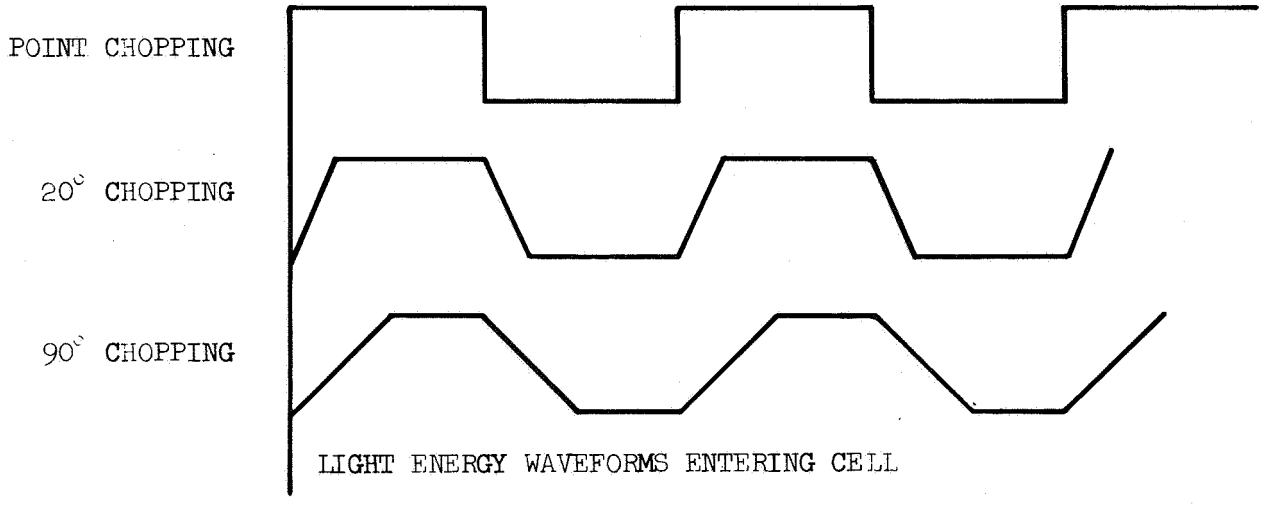


FIGURE 2.5-1

EVALUATION OF ENERGY OUTPUT VS CHOPPED BEAM AREA
(DATA PROVIDED BY BROWER LABORATORIES)

are diffuse in nature and, therefore, a "fix" was required.

This was accomplished through the use of a reciprocating sine wave drive system in the chopper gear train wherein there is a relatively long dwell time of the chopper blade in the closed position and in the open position and rapid transition between the open and the closed positions. Based on Mr. Brower's data, the use of the sine wave motion will reduce the error to under 1% for the most extreme case (for a perfectly diffuse sample). Thus, for ordinary samples, it is anticipated that the error will be considerably smaller than this.

The reciprocating nature of the chopper makes possible another type of error for angular measurements which was not realized when the reciprocating motion was designed. For an "ordinary" sample, not perfectly diffuse but which bunches reflected energy about the specular direction, the performance of angular measurements uses light from different parts (angles) of the source, depending on the angular setting. Depending on whether the chopper approaches from the right-hand side or the left-hand side, the chopper will tend to close off the beam early and open it late or do the opposite and close late and open early. Either way there could be an error since the square wave would no longer by symmetrical. The quick open - quick close characteristics of the chopper tend to minimize this error and no indication of error was found experimentally due to difficulty from reciprocating chopping when making angular measurements.

A parenthetical note might be added at this point. The instrument was designed to operate in the reciprocal mode because no isotropic IR detector was available. If at a later date, such a detector is available,

the modifications to install it in the instrument should be minimal and many problems would be eliminated. The chopper could be installed outside the vacuum tank. The chopper has been the most difficult item of the Far Infrared Spectroreflectometer to develop.

2.6 SAMPLE

It was desired to make the sample as large as feasible in order to allow the performance of angular measurements over a wide angular increment. It was also required that water cooling of the sample be provided in order to maintain the samples at room temperature during measurement. There will be approximately 50 watts radiant energy focused on the center portion of the sample and this heat must be transferred to a coolant liquid in order to prevent the sample from being heated.

A design was evolved in which the sample is 1" in diameter and in which tap water thermostating at the sample back surface is provided. A drawing of the sample holder and cross section is shown in Figure 2.6-1. Four sample holders are provided, each may be positioned in the measurement position without breaking vacuum by the sample rotation mechanism.

The large sample is useful and necessary for angular measurements to near grazing angles. However, a troublesome error was found which is indirectly related to sample size. Radiation from the graphite thimble source is imaged onto the sample area. Usually the source was operated at an output of about 60 watts. Probably at least 80% of this power is radiated and lands on the sample. If the sample is a good reflector the light will reflect back from the sample to the source area. Some of the returning radiation will strike and be absorbed by the source. (The focusing properties of the ellipse are such that a large portion of the returned radiation does not strike the source, however, a significant amount of it does.) The returned radiation which strikes the source and which is not absorbed is reflected from the source and can make another

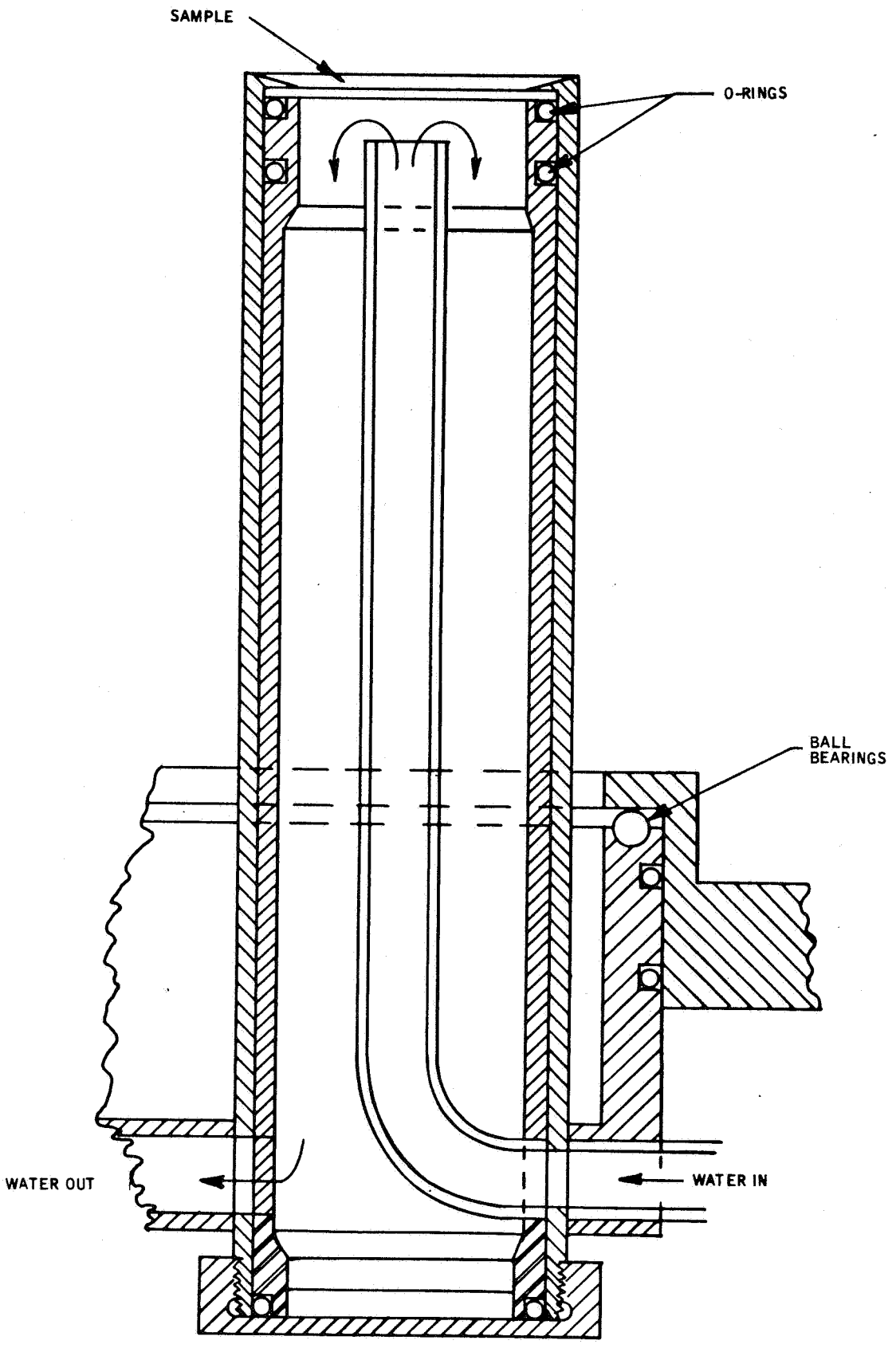


FIGURE 2.6-1 SAMPLE HOLDER DETAIL

pass at the sample thus giving effectively a higher radiant flux from the source. In initial tests it was found that errors of a few percent were occurring due to these phenomena and that reflectances for vacuum deposited aluminum were running the order of 102%, in some wavelength regions. The error due to the reflected light from the source would be eliminated if the source were a blackbody (all the reflected light would be absorbed).

The radiation return to the source also causes another type of error. The source is heated slightly by the sample reflected energy. This heating was demonstrated to occur by viewing the sample with a pyrometer first when it was in the measurement position and then when the sample was removed and the instrument was put in the 100% position. A difference of about 8°C temperature could be detected when the test was made without the sample caps on the samples.

To control the radiation return to the source, special sample caps were fastened to the sample holder cylinders to limit the exposed area of the sample to just slightly larger than the area of the spectrometer image on the sample. It was found that the placement of caps over the sample largely eliminated the source reflectance part of the error. Caps are supplied with the instrument and may be fastened to the sample cylinders and used for all near normal incidence measurements. For angular measurements special caps with a longer slot are provided to accommodate the longer image pattern on the sample.

The heating of the source due to reflected energy probably causes a small error in the reflectance measured in the region from 2.5 to 4 μ.

But past about 4μ the error is negligible due to the characteristics of the radiating bodies in these wavelength regions, i.e., on the long wavelength side of the blackbody peak, changes in temperature cause only very small changes in radiance. It is easy to detect and determine the amount of the source heating error. When the large rotation cylinder is rotated from the 100% position to the sample measurement position and the sample is rotated into position, a value will be obtained for the reflected light which will increase over a period of 15 or 20 seconds. The initial value determined is undoubtedly the essentially correct reflectance value and the increase is due to the heating of the source. It will be found that this occurs only in the initial parts of the spectrum (first couple of microns) as noted above, and the correct value of the reflectance can be obtained by taking the initial reading printed after allowing for the usual detection system lag. (Start the digital recorder printing before the sample is rotated into position and leave it on as the sample is positioned.)

As noted under source, a three-dimensional or volume source rather than a planar source is provided and has many advantages. It does have the disadvantage that radiation could (if not blocked) directly illuminate the sample from a grazing angle. This is clearly undesirable and especially so for diffusely reflecting samples. The small sample caps mentioned above effectively block direct radiation to the sample. The angular sample caps are provided with small lips at the end to accomplish the same purpose.

2.7 SPECTROMETER

In the past, Model 98 or 99* monochromators have been generally employed with reflectance measurements of the type described here. These instruments use salt prisms to disperse light and are flexible and have been generally satisfactory, however, they are subject to certain limitations. The most serious of which is the wavelength coverage in the far IR spectral region.

A thoroughly developed grating-type monochromator, Model 210,* which provides extended wavelength coverage in the far IR and has many other advantages in comparison to the prism instruments is available. This monochromator has been selected for use in this reflectometer. Using interchangeable gratings it will operate in the wavelength region 0.25 μ to 400 μ with the gratings supplied light dispersion from 2.5 to 125 μ is provided. The instrument was successfully operated from 2.5 to 90 μ, and using other Restrahlen plates, filters, and thermocouples the range can probably be extended to past 110 μ.

As delivered, the instrument employs a diamond window thermocouple detector** for the infrared region. Separation of orders of the gratings and stray light are controlled through the use of filters as described under Section 2.11. The monochromator is equipped with a gas inlet plug as to allow purging with an inert atmosphere. By purging the monochromator and using a vacuum or purged atmosphere in the ellipsoid chamber, it is possible to obtain spectral data essentially free from interference of atmospheric absorption bands. If atmospheric absorption

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*Perkin-Elmer Corp., Norwalk, Connecticut.

**Reeder Co., Detroit, Michigan.

is a particularly critical problem for some special work, use of a collar in the area between the window and the monochromator could be easily fashioned.

The effective aperture of the 210 is f/3.8 versus f/4.5 for the prism instrument. The resolution obtainable with the Model 210 is considerably better than with the Model 99.

2.8 DETECTORS

The detector supplied with the instrument is a Model RP-5W thermocouple with a diamond window.

It is not possible to select a thermocouple which is optimum for all wavelengths and tradeoffs are required and performance considerably reduced from optimum is obtained if a thermocouple is procured to cover the full wavelength region from 2.5 to 100 μ. It is encouraging that good performance was obtained with the supplied couple and considerably increased performance potential exists by using optimized couples in given wavelength increments. Considerations involved in optimizing the couple involve target size and window material.

2.8.1 WINDOW MATERIAL

The only material which appears suitable for thermocouple window that transmits from 2.5 to 100 μ is diamond. This material is supplied with the instrument. It has serious and restrictive absorptions as shown in Figure 2.8-1. Past 50 μ, its transmission is reported to be good. Cesium iodide has fairly good transmission from 2.5 to 50 μ, but has rather high reflectance losses. Cesium bromide has lower reflectance losses, but begins to cut off at about 40 μ and probably is not useable at 50 μ. From 2.5 to 50 μ there is certainly considerable performance to be gained by the use of a better window on the thermocouple as shown by Figure 2.8-1.

2.8.2 THERMOCOUPLE TARGET SIZE

The ellipsoid in front of the thermocouple detector reduces the image about 6 to 1. The slit is 10 mm high. The height of the target is reduced to correspond to this (plus a little) and is 2.0 mm wide. On the conventional spectrometers which have slit openings up to 2.0 mm,

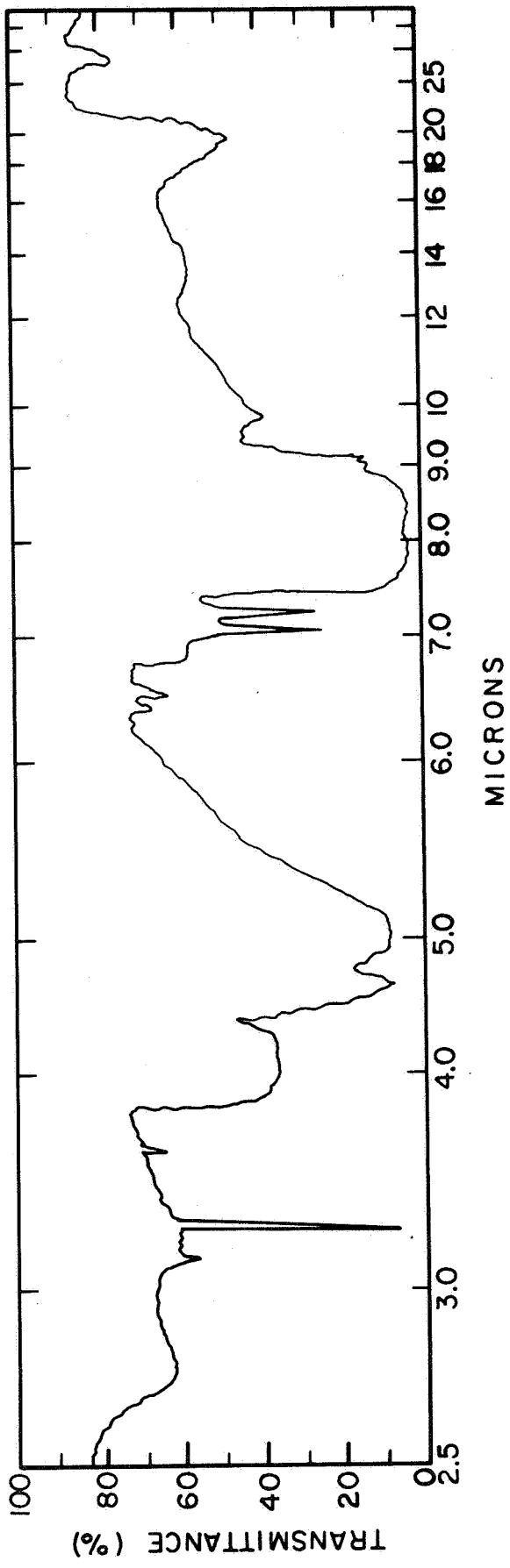

FIG. 2.8-1 : TRANSMISSION OF DIAMOND USED FOR THERMOCOUPLE WINDOW

the target is usually made .2 millimeter wide. The Model 210, of course, has a possibility of providing slit openings up to 10 mm. To allow the full image at the detector position to fall on the thermocouple would require a couple which is about 2 mm × 2 mm. But in many regions of the spectrum a slit nowhere nearly 10 mm wide is required and the use of a couple which is optimized for the full slit width would be relatively insensitive due to the large mass of the detector material. Similarly, a couple which is too narrow would not receive all of the light available at the detector position. The compromise made in the case of the instrument delivered to NASA was to make the diamond window thermocouple target 2.0 mm × 0.8 mm. It is felt very likely and will be tested in our laboratory that the use of a quartz couple and a wider target will allow our operation of the instrument out past 100 μ. A cesium bromide couple with a target size of 2.0 mm × 0.5 mm would probably be optimum for the wavelength of 2.5 to 40 μ.

It is necessary from time to time, making adjustments and so forth, to remove the monochromator from its position and then to replace it. Great care should be exercised in setting the monochromator down in its position on the spectrometer. The thermocouple target can be broken by a small drop ($\frac{1}{4}$") of the monochromator into position. The monochromator is a bit difficult to hold, however, with care it can be inserted into position and removed from position with no mechanical shock.

2.9 AMPLIFIER

Reference is made to the instrument manual regarding the Brower amplifier. It was found during development and checkout that the preamplifier environment was very critical and it was necessary to remove the preamplifier from the relay rack and place it on a small table. A table was not provided for this preamplifier for NASA inasmuch as the exact spatial arrangement of components which would be used was not known. It is recommended that the preamplifier be set on a stable platform which is not subject to any vibrations from pumps, choppers and so forth and is away from any stray electric field.

An open thermocouple manifests itself among other ways by the inability to obtain test calibrating signals on the amplifier. The chopper operates at 5.5 cps and if it is desired to use the amplifier with other equipment and a standard 11 cps Brower chopper, an alternate frequency card must be substituted for the card in the instrument.

2.10 RECORDING

Recording is accomplished by means of the Model 2401C* integrating digital millivolt meter and the Model 66-562AR* digital recorder. These are both very nice instruments and they function well. For further information regarding maintenance and so forth, reference is made to the rather complete manuals supplied with these instruments by the manufacturers.

*Hewlett Packard, Palo Alto, California.

2.11 ELECTRICAL

A simple diagram of the electrical wiring system is shown in Figure 2.11-1. A constant voltage supply transformer is used to supply the sources. The switching is such that either the mercury lamp or the gray body source may be turned on, but not both sources at one time. Care should be exercised to be sure that the water is always turned on before the sources are activated.

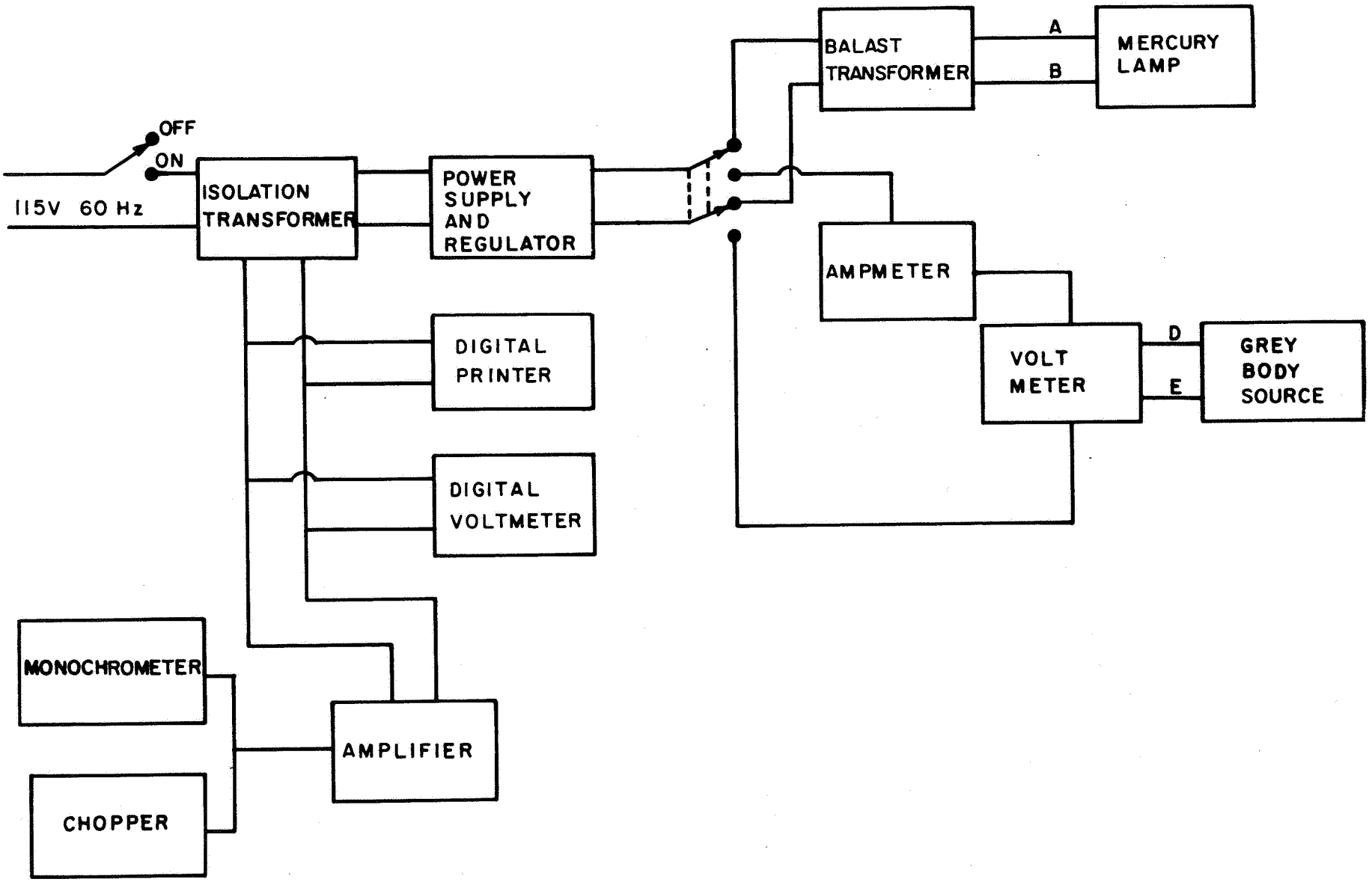

FIG. 2.11-1: WIRING DIAGRAM

2.12 FILTERS AND WINDOWS

Filters 1 through 7 supplied with the instrument cover the range from 2.5 to about 40 μ as shown in the Instrument Settings Table. Transmission curves were not supplied with these filters. They seem to be satisfactory in the wavelength region specified, however, it is suspected that transmission of these filters is not outstanding in certain regions where their use is required. However, overall satisfactory operation is obtained using them.

Past 40 μ there were problems. The manufacturer did not supply filters for the Model 210 in this range, but recommended the use of Restrahlen plates, scatter plates, etc. The use of graphite loaded high density polyethylene was also suggested. This material has properties of sooted mirrors or windows and is much more convenient to use (and better). A literature search was made for suitable materials and several were tested. The generally satisfactory scheme outlined in the Instrument Settings Table was finally evolved.

The filter number 8 is made up of two layers of high density polyethylene. This, used in conjunction with the quartz window, provides satisfactory operation out to 90 μ and perhaps further, depending on the use of optimum Restrahlen plates and detectors. The high density polyethylene cuts off the visible and near IR portion of the spectrum, and the quartz cuts off the radiation between about 7 μ and 40 μ. In checking one layer of the high density polyethylene and quartz filters, it was determined that the place where probably a small amount of energy "gets through" is in the region where the transmission of the quartz is just ending and the transmission of the high density polyethylene is just beginning. As

best as could be determined using the scale expansion on an IR 4,* the two filters leave a small "roof-shaped" area where a small amount of false energy probably leaks through. Doubling the high density polyethylene filters, as was done on the NASA instrument, largely eliminated the difficulty and allowed operation to past 50 μ. A filter with better transmission past 50 μ would be highly desirable. Reportedly, very high performance filters are available,* and a set of these is on order, but has not yet been received. These will be checked and the information regarding them made available to NASA.

The crystal quartz window has its optic axis lying on the face. This type crystal has better far IR transmission characteristics than other cuts.

The KBr and cesium iodide windows provided are of a standard size and the usual precautions regarding moisture apply (see Perkin-Elmer manuals). The fused silica window is supplied for use in inspection and alignment when a clear window is useful. This window could also be useful for operation in the visible and UV portion of the spectrum if instrument capability were extended to those wavelength regions.

*Beckman Instruments, Inc., Fullerton, California.

3.0 ALIGNMENT

Initial alignment of the instrument at the NASA site will be performed by a Convair representative. The procedures involved are as follows: The toroid (see Fig. 2.2-1), M3, and the large diagonal M4 mirror assembly should be placed on the slide mount and located as close to the rotating window plate as possible and fastened in place with the two screws provided. The small rotating window plate is not installed at this time.

The Model 210 grating monochromator is set into position in the three holes provided in the mounting platform (set it down lightly so as not to damage the thermocouple if it is installed in the monochromator). The diagonal mirror past the exit slit is removed from the light path so that a mercury arc lamp source may be focused on the exit slit. Convair uses a special arc housing which bolts directly to the Model 210 monochromator for the H4 lamp and uses a lens in a holder that sits on the monochromator base to focus the light from the H4 onto the exit slit. It is required that the aperture of the exit slit be filled by this light. Failure to fill the aperture can lead to difficulties in the alignment. It is suggested that after the mercury arc is set into position and it appears that the aperture is filled, a white card be placed in front of the entrance slit of the spectrometer and that the position of the mercury-arc image on the exit slit be moved slightly back and forth across the slit. The green line image on the card in front of the spectrometer will move and clearly define the aperture extremities.

The light emerging from the front entrance slit passes into the instrument and is intercepted by the first diagonal, M4. It should pass

as close as possible to the edges of the parabola, M3, without striking it. Because of the fixed position of the monochromator, striking the toroid will probably not be a problem. Check the area illuminated on M4 mirror with a white card. The light should not fall off of the edges of this mirror. If necessary, the first diagonal may be pushed laterally in its holder to cause it to intercept the entire beam. With the first diagonal filled, the light will fall on the toroid, M3. Check with a white tab to be sure the light does not fall off the edges of M3. The light then should fall on the second or small diagonal, M2. Check to make sure that the beam from M3 to M2 does not strike M4; if it does, a lateral adjustment of M4 is required. There is a mask in front of M2 and the beam should approximately center on this mask. (The mask in front of M3 is not installed at this point.) If a small adjustment is required to move the beam onto M3, it may be accomplished by a small adjustment of the toroid, using the Allen screws on the back of the mirror. If a large adjustment is required, probably a rotation of M4 has occurred and should be corrected. This may be accomplished by removing the M4-M3 mirror assembly, loosening the Allen screws on the M4 rotation adjustment, replacing the toroid-diagonal assembly with the Allen wrench in place, adjusting the M4 to center the beam on the mask of M2 and tightening the Allen screws. After alignment at NASA Ames, it is not anticipated that the above adjustments will be required unless the mirrors are removed for aluminizing or changes.

The light from the first diagonal, M2, is reflected up to the overhead mirror, M1 and then down to the sample. The alignment of M1 and M2 is critical and adjustment will be required from time to time; the alignment

should be checked fairly often. Adjustment is as follows: Make sure that the large rotation cylinder with the assembly which holds the ellipse, etc., is "pushed all the way in." (The ellipse is not installed at this time.) If this is neglected when adjusting M1, then, when a vacuum is created in the tank, the cylinder will "pull in" and the image will not fall properly on the sample. Place a small level on the base plate which holds the samples, ellipse, etc. Rotate the cylinder to level the plate. Set the angle pointer to zero. The small spherical mirror M1 should be positioned directly over the sample and the beam from small diagonal M2 should direct the light to the overhead spherical mirror so that it completely fills it and so that none of the beam passes around the edges. This can be checked by holding a white card over the mirror. Also, it should be ascertained that the light does not overfill the first diagonal, M2, and this also can be checked by the use of a white card held in the direction of the beam past the diagonal. With the light properly adjusted on M1 and M2, adjust M1 so that the image falls on the sample and within the limits of the opening of the sample cap.

It is necessary that mirrors M1 and M2 do not touch the sample cylinders or other components on the base plate. To accomplish this by inspection when the large rotation cylinder is turned would be practically impossible due to the inability to obtain a satisfactory view of the components involved. As a result, mirrors M1 and M2 were mounted on a ball bearing arrangement such that for adjustment of these mirrors the M1-M2 mirror assembly could be rotated in a way which is equivalent to the rotation of the large rotation cylinder. After adjustment these two mirrors are held in a fixed position by a screw at the base of the plate which fits into the housing.

To check the mirrors to assure that there is no contact, rotate the sample assembly to a 100% measurement position, remove the screw at the base of the plate, rotate the M1 and M2 mirror assembly and check to make certain that neither mirror strikes the cylinders holding the sample nor the base of the sample holder assembly. M1 should come close to this base but should not touch it. After checking and assuring that fouling does not occur, return the mirrors to their original position and install the screw that blocks the rotation of the mirror adjustment assembly.

The large hemi-ellipsoid kinematically mounts on the base plate by a ball-V groove arrangement. It is fastened to the base by three screws. It was focused before delivery and should require no refocusing or alignment. Focusing the ellipsoid is an involved tedious and time consuming operation. The method used to focus it will be described here, in case focusing adjustment or checking is required at some future date. Due to the three dimensional character of the source, ellipsoid focusing is not highly critical, however, for best operation, the accurate focus of the hemi-ellipsoid is desirable.

The sample, when in the measurement position, must center at one focus of the hemi-ellipsoid. It is necessary that the center of the face of the sample lie on the rotation axis of the large rotation cylinder. To establish this, rotate one of the samples into the exact sample measurement position. This may be checked with a fine scribe line on the ellipse base. This line is perpendicular to the axis of rotation and should project through the center of the sample holder cylinder when the sample is in the measurement position. The sample height may be checked by use of a jig which was shipped with the instrument. The jig consists of a half circular aluminum plate,

and an aluminum bar with a small axle projecting from one end and a cutaway in the center. The aluminum plate fits into the "necked down" part of the large rotational cylinder, and the bar cradles in the position provided for it in the plate and the other end of the bar fastens into the front plate in a hole provided. A screw with a pointed tip locates the center of rotation of the cylinder at the sample position. This should just touch the sample. This was set up properly during manufacture and it is not likely that it will change unless the sample holder or structural parts of the instrument are changed.

After the sample height and position are checked, the sample center is designated as one focus of the hemi-ellipsoid and the mirror is then adjusted to place the other focus at the source position. The ellipse base assembly including source, sample unit, etc. are removed from the instrument and made secure on a table. A sample disc is made with a pin-hole in the center. A lathe is used to make the pin-hole in order to properly locate the hole in the center. The back underpart of the sample is beveled to allow light from as wide an angle as possible to get through the pin-hole. A small light bulb, battery-operated or otherwise, is placed in the sample cylinder against the disc so that the light passes through the hole. This provides essentially a point source at one of the foci of the ellipse. The graphite source is removed from its holder and a white card with crossed lines is placed in the same plane as the top of the sample and the intersection of the crossed lines is located at the other focus, 2" from the sample. A mirror is placed on a holder so that the card face and crossed lines may be viewed from under the edge of the ellipse. The light is turned

on and the position and quality of the image on the card is viewed. The image should fall precisely on the intersection of the cross lines and the focus should be good. The quality of focus obtainable can be determined by moving the card up and down slightly at the focus position. If the focus is not optimum at the source plane, then an adjustment of the ellipse is required.

Focusing of the ellipsoid is accomplished by adjustments on the two semi-circular bracket arrangement which are cemented to the ellipsoid. It was originally intended to focus each quarter of the ellipsoid separately and three adjustments were provided on each semi-circle. With the two quarters of the ellipsoid joined together and cemented only three of the adjustments out of the total six are required. In the procedure used, the setting screws of three of the adjustments were set "loose" so as not to obstruct the movement of the ellipsoid and adjustment was accomplished with the other three setting screws. Reference is made to Figure 3.0-1 showing the adjusting arrangement. The ellipse is bonded to the upper semi-circle with silicone rubber. This semi-circle has a hole arrangement to accommodate the adjusting screw with its large flat cap. This screw fits into the base semi-circle. Up and down adjustments are made by locking the lower clamp ring on the adjusting screw and turning by means of a small axle or rod, the upper clamp ring being tight to the screw but sufficiently below the upper semi-circle to allow turning. Lateral adjustments are made by sliding the upper semi-circle on the head of the adjusting screw. These adjustments are made with some difficulty but can be accomplished. After the

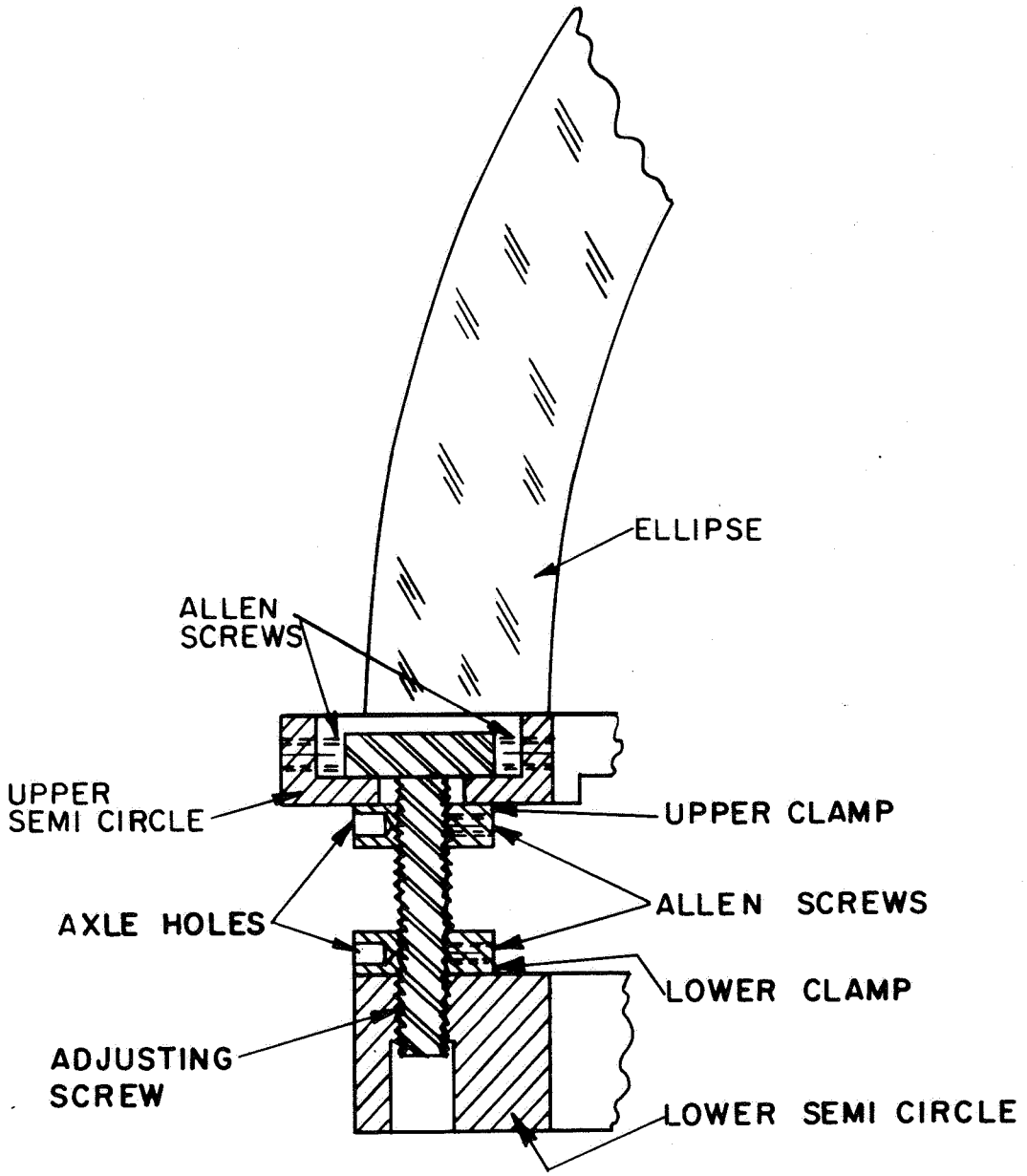

FIG. 3.0-1 : ELLIPSE ADJUSTING ARRANGMENT

ellipse is properly focused, as evidenced by the optimum figure on the card cross lines, the adjustments are tightened in place and the focus again checked before removing the focusing gear.

It is not recommended that focusing of the ellipsoid be undertaken lightly. Clear and definite information should be available that focusing is required. It is not anticipated that focusing will be required throughout the life of the instrument, unless some alterations are made or unless a new ellipse is installed.

4.0 OPERATION

4.1 SAMPLE LOADING

To load samples, first, remove the multiple sample assembly from the opening in the base plate. Blow out as much of the water in the system as possible, using an air line. Holding the sample assembly in a vertical position, unscrew the caps at the base of the sample system and remove the water cooling cylinders. Keeping the cylinders up right limits the wetting of the samples in the holder. There is a threaded hole in the base of these cylinders into which a screw may be placed to facilitate removal of the cylinders. Lubricate the "O" rings and place the sample on top of the cylinders and place the cylinders on a table. It is necessary to turn the inlet and outlet on the water cylinders in the proper direction to allow a passage of water through the system. The proper arrangement is evident upon inspection. There are two types of cylinders; two in which the water comes out at a right angle to the incident direction and two which are straight through. Lower the sample holder unit over the sample and cylinder and then press the cylinder in until the sample seats in its proper position. Then turn the sample holder upside down and screw the cap in place. It is necessary that the screw cap contact the cylinder in order to firmly press the "O" rings against the sample surface. If there are variations in sample thickness, it may be necessary to use washers between the cap and the cylinder. With a little experience, a "feel" can be developed for the cap tightening operation to know when the "O" rings are being sufficiently squeezed to make a water-tight joint. With a little practice, the sample loading can be accomplished so that

water leaks do not occur. After all four samples are loaded, the assembled sample unit should be tested for leaks (run water through system and pinch the hose to give back pressure) before it is put into position in the base plate. The sample holder system is held in place by four Allen screws and made vacuum tight by an "O" ring seal. The addition of the black sample caps, necessary for reduction of reflected light back to the source, elevates slightly the sample holder walls and care is necessary when sliding the sample unit back into position. With the angular sample caps, it may be necessary to remove the caps and replace them from above with the ellipsoid removed from the system.

4.2 SOURCE OPERATION

The mercury arc source is fixed in place on the source holder and will require little maintenance and attention. The gray body or graphite source seats in the water-cooled sample holder opposite the mercury arc and will require replacement of filament from time to time. It has been found in laboratory tests at Convair that this source lasts for days under constant operation in a vacuum. In the purge atmosphere (argon) in which the tests for the NASA samples were made, it was found that the lifetime of a filament was about 8 hours. The difficulty apparently was that the system was not sufficiently pumped out before the inert gas was introduced. Initially, in using inert gas it was found that the lifetime of the filament was only about 2 to 3 hours or less. This was found to be due to impurities in the commercial argon used and was remedied by flowing the argon through a stainless steel tube containing titanium chips heated in a chemical furnace to red heat. Evacuating the tank to 10^{-3} mm of mercury and intro-

ducing argon through the heated titanium, the 8 hours or more of operation of the tungsten filament was obtained. The unit fails through the formation of a moss-like filmy mass inside the graphite thimble. The system would operate apparently satisfactorily in either the 100% or in the sample position, but then when it was inverted, the moss would fall down through the filament and cause difficulties which manifest themselves in fluctuations of the gages showing filament voltage and current, resulting shortly thereafter in the failure of the filament.

The filament used is of a type used in the 1000 watts quartz iodine lamps* and a supply of extra filaments was first furnished with the instrument. Extra filament material has been obtained from the long thin quartz line lamps. It is necessary to develop a technique for putting a new filament into the sample holder, but with some practice this can be accomplished satisfactorily. The gray body source unit may be removed from the instrument by loosening the Phillips head screw and slipping the leads from the source out from the source holder. The graphite cap is removed from the lava rock base by removing two pins, one on either side of the source. The graphite cap is cleaned out and the replacement tungsten filament is put on the two lead-in wires. A small current can be applied across the tungsten filament to heat it just barely to a visible red in order to allow fashioning it into a suitable shape. It is felt very likely that with the use of better vacuum equipment that the inert gas atmosphere operation can be carried on for considerably longer than 8 hours before failure of the filament.

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*General Electric Co., Cleveland, Ohio.

The source operates at about 50 to 60 watts power. Aside from the difficulties of moss in the cap which causes trouble on inversion of the system, the source was found to be stable and functioned well.

4.3 VACUUM AND PURGE ATMOSPHERE

The system was designed for vacuum operation. However, upon testing and checkout, considerable difficulty was found in operating under vacuum conditions and as an expediency, testing was accomplished using an inert argon atmosphere. In reviewing the pros and cons of operating in a vacuum versus an inert atmosphere, there do not seem to be any compelling reasons to use a vacuum and there are some real disadvantages. It was found that the material in the sample covers and the material of the sample itself tended to evaporate in a vacuum and to coat the optics, especially the large ellipsoidal mirror. The rotations of components that are required for the operation of the instrument were found to be more difficult to make under vacuum conditions than with an inert gas. When inert gas was used, a negative pressure in the tank of 1 or 2 psi was maintained to keep the rotation cylinder "pushed in."

4.4 DETERMINATION OF I_o (100%) AND I (REFLECTANCE MEASUREMENT)

With the sample, source, and ellipse installed, and a vacuum or purge atmosphere established, the measurements may be begun. The gray body graphite source is turned into the operation position and turned on and regulated at between 50 to 60 watts. The amplifier and recorder are turned on (see the respective manuals for details). The sample assembly is rotated to a 100% measurement position. The large rotation cylinder is rotated to the 100 percent position. The position occurs at an angle of 173°. In

this position the small spherical mirror is focused by the ellipsoid onto the sample position from which the sample is removed and transfers directly the radiation which would have illuminated the sample into the spectrometer. The chopper is turned on and the signal is read on the recording system. Slit and gain settings are made to provide a full scale reading on the front dial of the Brower Amplifier. After establishing this I_o (100 percent) datum, the large rotation cylinder is rotated to the near normal reflectance angle at 6° and one of the samples is rotated into position. The I (reflectance datum) is then recorded and the sample assembly is rotated to the next sample position and so on in sequence until all four samples are recorded. The sample holder is then rotated into the 100 percent position and the large rotation cylinder is rotated into the 100 percent position and I_o is rechecked. It is possible for the sample cylinders of the sample assembly to foul the mirror, M1, and the mirror bracket if the proper rotation sequencing is not adhered to. In checkout here at Convair, this problem was well known but still in the initial testing fouling occurred several times and the tank had to be opened and M1 adjusted. Later, after some use of the instrument, personnel involved had "learned" and the entire set of data for NASA was obtained without a fouling mishap.

4.5 INSTRUMENT SETTINGS

Table 4.5-1 gives the combinations of gratings, mirror or Restrahlen plates, filters, and windows for determination of reflectance spectra. Table 4.5-2 gives the wavelength vs drum settings, and Table 4.5-3 gives the settings used in the determination of the test reflectance spectra.

TABLE 4.5-1

INSTRUMENT SETTINGS FOR REFLECTANCE MEASUREMENTS

WAVELENGTH IN MICRONS	GRATING NO.	RESTRAHLEN	FILTER	WINDOW
2.5	240 L/mm	Mirror	3	KBr
2.6 - 4.0	240 "	"	3	"
4.1 - 6.4	240 "	"	4	"
6.5 - 10.2	101 "	"	5	"
10.3 - 14.0	101 "	"	6	"
14.1 - 15.0	101 "	"	7	"
15.1 - 20.0	40 "	"	7	"
20.1 - 24.3	40 "	"	7	CsI
24.3 - 30.0	40 "	"	8	"
30.1 - 39.9	20 "	BaF_2	8	"
40.0 - 50.0	20 "	"	2	Crystal Quartz
52.5 - 75.0	10 "	KCl	2	"
77.0 - 90.0	10 "	KBr	2	"

TABLE 4.5-2

WAVELENGTH VS DRUM SETTING FOR OPERATION FROM 2.5 TO 100. μ

$\lambda(\mu)$	Cm^{-1}	Drum Reading	$\lambda(\mu)$	Cm^{-1}	Drum Reading
2.5	4000.	20.360	19.	526.3	13.48
3.0	3333.	15.000	20.	500.0	12.25
3.5	2857.	11.145	21.	476.2	11.12
4.0	2500.	8.360	22.	454.5	10.08
4.5	2222.	6.140	23.	434.8	9.12
5.0	2000.	4.345	24.	416.7	8.31
5.5	1818.2	2.910	25.	400.	7.50
6.0	1666.7	1.710	26.	384.6	6.78
6.5	1538.	17.545	27.	370.4	6.06
7.0	1429.	15.463	28.	357.1	5.44
7.5	1333.	13.640	29.	344.8	4.84
8.0	1250.	12.065	30.	333.3	4.30
8.5	1176.	10.660	35.	285.7	15.78
9.0	1111.	9.41	40.	250.0	12.32
9.5	1053.	8.32	45.	222.2	9.61
10.0	1000.	7.32	50.	200.0	7.52
10.5	952.4	6.40	55.	181.8	5.74
11.0	909.1	5.682	60.	166.7	20.82

TABLE 4.5-2

WAVELENGTH VS DRUM SETTING FOR OPERATION FROM 2.5 TO 100. μ

$\lambda_{(\mu)}$	Cm^{-1}	Drum Reading	$\lambda_{(\mu)}$	Cm^{-1}	Drum Reading
11.5	869.6	4.84	65.	153.8	18.24
12.0	833.3	4.14	70.	142.9	16.18
12.5	800.0	3.52	75.	133.3	14.20
13.	769.2	2.92	80.	125.0	12.66
14.	714.3	1.88	85.	117.6	11.20
15.	666.7	0.096	90.	111.1	9.94
16.	625.0	18.60	95.	105.3	8.76
17.	588.2	16.42	100.	100.0	7.80
18.	555.6	14.94			

TABLE 4.5-3

INSTRUMENT SETTINGS USED FOR DETERMINATION OF
SAMPLE REFLECTANCE AND FALSE ENERGY DETERMINATION

WAVELENGTH IN MICRONS	GAIN	SLIT	RESTRAHLEN	FILTER NO.	WINDOW	FALSE ENERGY	
						FILTER	PERCENT
2.5	25.μv	0.490	Mirror	3	KBr		
3.	"	0.520	"	3	KBr		
3.5	"	0.515	"	3	KBr		
4.	"	0.770	"	3	KBr		
4.5	"	0.945	"	4	KBr		
5.	10.μv	0.970	"	4	KBr		
5.5	"	0.710	"	4	KBr		
6.	"	0.785	"	4	KBr		
6.5	25.μv	0.510	"	5	KBr		
7.	"	0.420	"	5	KBr		
7.5	"	0.675	"	5	KBr		
8.	2.5μv	1.175	"	5	KBr		
8.5	5.μv	1.355	"	5	KBr		
9.	10.μv	1.160	"	5	KBr		
9.5	25.μv	1.43	"	5	KBr		
10.	10.μv	0.755	"	5	KBr	Quartz	0.
10.5	"	0.740	"	6	KBr		
11.	"	0.780	"	6	KBr		
11.5	"	0.890	"	6	KBr		
12.	"	1.070	"	6	KBr		

TABLE 4.5-3

INSTRUMENT SETTINGS USED FOR DETERMINATION OF
SAMPLE REFLECTANCE AND FALSE ENERGY DETERMINATION

WAVELENGTH IN MICRONS	GAIN	SLIT	RESTRAHLEN	FILTER NO.	WINDOW	FALSE ENERGY	
						FILTER	PERCENT
13.			Mirror	6	KBr		
14.			"	6	KBr		
15.	2.5μv	1.00	"	7	KBr	Quartz	0.
16.			"	7	KBr		
17.			"	7	KBr		
18.			"	7	KBr		
19.			"	7	KBr		
20.	5.μv	0.660	"	7	KBr	Quartz	0.
21.			"	7	CsI		
22.			"	7	CsI		
23.			"	7	CsI		
24.			"	7	CsI		
25.	1.μv	0.534	"	8	CsI	NaCl	0.
26.			"	8	CsI		
27.	500.nv	0.515	"	8	CsI		
28.				8	CsI		
29.				8	CsI		
30.	500.nv	0.724		8	CsI	NaCl	0.
35.	500.nv	0.60	BaF_2	8	CsI	NaCl	0.
40.	25.nv	2.00	BaF_2	2	C Quartz	NaCl	0.

TABLE 4.5-3

INSTRUMENT SETTINGS USED FOR DETERMINATION OF
SAMPLE REFLECTANCE AND FALSE ENERGY DETERMINATION

WAVELENGTH IN MICRONS	GAIN	SLIT	RESTRAHLEN	FILTER NO.	WINDOW	FALSE ENERGY	
						FILTER	PERCENT
45.	25.nv	0.800	BaF_2	2	C Quartz		
50.	25.nv	2.00	BaF_2	2	"	KBr	1.
55.	25.nv	1.55	KCl	2	"	KBr	0.5
60.	25.nv	0.810	KCl	2	"	CsBr	0.
65.	25.nv	0.730	KCl	2	"	CsBr	0.
70.	25.nv	1.055	KCl	2	"	CsBr	0.
75.	25.nv	1.28	KCl	2	"	CsBr	1.
75.	25.nv	0.980	KBr	2	"	KBr	0.
80.	25.nv	1.50	KBr	2	"	CsBr	0.
85.	25.nv	1.53	KBr	2	"	CsBr	0.
90.	25.nv	2.00	KBr	2	"	CsBr	8.

4.6 ANGULAR MEASUREMENTS

To make angular measurements, the main tank lid and the ellipse are removed and the near normal incidence sample caps are removed from the two samples for which angular measurements are possible (as listed on the Table on the main tank). The angular sample caps are placed in position and oriented properly so that the sample is shielded from the direct radiation from the source. A restrictive baffle is placed in front of the large toroid, M3. This limits the width of the beam and allows angular measurements to a much greater angle than would otherwise be possible. At this point, the mercury green line should be run backwards through the instrument and the pattern on the sample checked. An adjustment of M1 may be necessary. The angular limit may be determined by rotating the base plate, etc. (keep it "pushed in"). The angle at which the green line strikes the rim of the sample holder cylinder is the maximum permissible angle using a flat sample. If the sample disc were cut specially on a lathe so that it would fit into the sample holder but project slightly above its present level so that the edges of the sample holder would not restrict the angular view, measurements out to greater angles could be made. Then, the maximum angle which could be measured would be an inverse function of the width of the baffle opening. Thus, large angle performance is obtained at the expense of energy to the detector which amounts to a tradeoff between extended wavelength coverage and the size of the angle to be measured.

Using flat (planer) samples, angular measurements out to 77° were studied to wavelengths of 22 μ. If required, the angle can be extended as noted above and the wavelength region can be extended through the use of a different thermocouple.

5.0 DETERMINATION OF SAMPLE REFLECTANCE

5.1 NEAR NORMAL DATA

The results obtained on the NASA-supplied samples are given in Table 5.1-1 and in graphical form in Figures 5.1-1 through 5.1-6. The data is reasonable and as far as can be found consistent with literature reports. The vacuum deposited silver gave very surprising results. In fact, the writer was so surprised during data taking that the instrument tank was opened and the alignment of the instrument was checked. There was, of course, nothing wrong with the alignment. The thought then occurred that the sample was of such a nature that there were interference phenomena present, which should have been obvious in the first place. As an afterthought, on reviewing the data for this report, it seems it would have been useful to have taken the data at much more closely spaced wavelength increments to better resolve the reflection spikes.

During the handling of the vacuum deposited aluminum sample, water was inadvertently allowed to wet the surface of the aluminum. The sample was cleaned in an alcohol vapor bath but did not seem as bright and shiny to the eye as it had beforehand and possibly the sample was degraded by this accident.

In Table 5.1-2 are given the reflectance values obtained on some samples, as determined by the ellipsoidal device and in Table 5.1-3 as run in the hohlraum. These data are presented graphically in Figures 5.1-7 through 5.1-10. On the figures the ellipsoidal data is shown as a series of triangles. There is general agreement, however, there are some discrepancies evident. The hohlraum data tends to be slightly higher

than the ellipsoidal data, especially in the long wavelength regions. A sample self-emission error, which is present in the hohlraum and absent in the ellipsoidal device, would tend to cause this type of behavior. The only thing that is puzzling about it is that it does not occur in all cases. The 3M velvet black paint shows the effect in some measure. The zinc oxide potassium silicate sample shows it very markedly; whereas, the Sherman Williams flat acrylic white paint does not show the effect. There is the further interesting phenomenon that on the reflectance spikes, i.e., the points of relatively high reflectance, in a generally low reflectance sample, the hohlraum data lies higher. This is especially noted in the Sherman Williams flat acrylic white paint but is also noted on the 3M black velvet. The source of this discrepancy will be studied using Convair's instrument. The reasons are not clear. The resolution performance of the Model 210 grating is superior to that of the prism instrument which works with the hohlraum and it does not appear that the error is likely to be due to resolution problems.

The hohlraum data on the gold is not shown but was essentially coincident with the ellipsoidal data.

5.2 ANGULAR DATA

The angular data taken on two NASA-supplied samples, namely, cobalt oxide and 3M black velvet paint are shown in tabular form in Table 5.2-1 and in graphical form in Figures 5.2-1 through 5.2-3. The behavior of the 3M black velvet paint is reasonable and not particularly surprising. The behavior of the angular reflectance of the Tabor solar absorber amazed the operators. Here again, as with the vacuum deposited silver sample,

the instrument was opened up and the alignment checked to be sure that something was not amiss inside. The total hemispherical emittance of this type of sample as calculated from the near normal reflectance data without regard to the angular effects could be in error by a serious amount.

TABLE 5.1-1

PERCENT REFLECTANCE OF NASA SUPPLIED SAMPLES

WAVELENGTH IN MICRONS	V.D. Al ON 5 MIL. S.S. #AQD-17	ZINC OXIDE POTASSIUM SILICATE Z-93 #AHA-9	PARSONS OPTICAL BLACK LACQUER ANA-13	TABOR SOLAR ABSORBER COBALT OXIDE #AUH-12	LANTHANUM OXIDE IN POTASSIUM SILICATE F-1-38	V.D. Ag ON 6 MIL. THICK FUSED SILICA ON Al SUBSTRATE
2.5	0.963	0.563	0.064	0.545	0.360	0.971
3.	0.957	0.168	0.015	0.522	0.063	0.937
3.5	0.962	0.098	0.018	0.590	0.055	0.951
4.	0.960	0.126	0.049	0.784	0.079	0.887
4.5	0.965	0.112	0.063	0.849	0.084	0.638
5.	0.962	0.107	0.060	0.886	0.086	0.040
5.5	0.953	0.080	0.041	0.902	0.064	0.020
6.	0.950	0.049	0.020	0.908	0.038	0.018
6.5	0.952	0.050	0.015	0.916	0.034	0.012
7.	0.952	0.052	0.016	0.934	0.024	0.001
7.5	0.960	0.057	0.016	0.931	0.023	0.001
8.	0.960	0.025	0.015	0.945	0.028	0.061
8.5	0.969	0.012	0.013	0.948	0.021	0.331

TABIE 5.1-1

PERCENT REFIECTANCE OF NASA SUPPLIED SAMPIES

WAVEIENGTH IN MICRONS	V.D. Al ON 5 MIL. S.S. #AQD-17	ZINC OXIDE POTASSIUM SILICATE Z-93 #AHA-9	PARSONS OPTICAL BLACK LACQUER ANA-13	TABOR SOIAR ABSORBER COBAIT OXIDE #AUH-12	IANTHANUM OXIDE IN POTASSIUM SILICATE F-1-38	V.D. Ag ON 6 MIL. THICK FUSED SILICA ON Al SUBSTRATE
9.	0.960	0.013	0.013	0.946	0.017	0.655
9.5	0.962	0.015	0.015	0.949	0.015	0.418
10.	0.961	0.020	0.017	0.949	0.018	0.238
10.5	0.959	0.021	0.019	0.954	0.023	0.137
11.	0.957	0.021	0.021	0.950	0.022	0.104
11.5	0.960	0.023	0.020	0.952	0.022	0.076
12.	0.964	0.025	0.018	0.953	0.022	0.054
13.	0.966	0.018	0.020	0.956	0.016	0.088
14.	0.962	0.030	0.018	0.952	0.012	0.060
15.	0.957	0.029	0.016	0.942	0.018	0.047
16.	0.968	0.023	0.016	0.944	0.029	0.044
17.	0.970	0.005	0.017	0.959	0.029	0.030
18.	0.961	0.026	0.016	0.938	0.021	0.025

TABLE 5.1-1

PERCENT REFLECTANCE OF NASA SUPPLIED SAMPLES

WAVELENGTH IN MICRONS	V.D. Al ON 5 MIL. S.S. #AQD-17	ZINC OXIDE POTASSIUM SILICATE Z-93 #AHA-9	PARSONS OPTICAL BLACK LACQUER ANA-13	TABOR SOLAR ABSORBER COBALT OXIDE #AUH-12	LANTHANUM OXIDE IN POTASSIUM SILICATE F-1-38	V.D. Ag ON 6 MIL. THICK FUSED SILICA ON Al SUBSTRATE
19.	0.969	0.046	0.017	0.953	0.019	0.021
20.	0.960	0.054	0.018	0.965	0.022	0.100
21.	0.962	0.064	0.020	0.962	0.031	0.516
22.	0.964	0.077	0.021	0.967	0.037	0.451
23.	0.963	0.094	0.021	0.971	0.039	0.312
24.	0.969	0.108	0.021	0.966	0.039	0.244
25.	0.965	0.117	0.022	0.972	0.039	0.206
26.	0.968	0.114	0.021	0.965	0.044	0.183
27.	0.963	0.100	0.020	0.971	0.062	0.168
28.	0.967	0.083	0.022	0.965	0.081	0.156
29.	0.964	0.074	0.020	0.965	0.096	0.147
30.	0.969	0.066	0.020	0.970	0.105	0.142
35.	0.985	0.065	0.019	0.970	0.150	0.127

TABLE 5.1-1

PERCENT REFLECTANCE OF NASA SUPPLIED SAMPLES

WAVELENGTH IN MICRONS	V.D. Al ON 5 MIL. S.S. #AQD-17	ZINC OXIDE POTASSIUM SILICATE Z-93 #AHA-9	PARSONS OPTICAL BLACK LACQUER ANA-13	TABOR SOLAR ABSORBER COBALT OXIDE #AUH-12	LANTHANUM OXIDE IN POTASSIUM SILICATE F-1-38	V.D. Ag ON 6 MIL. THICK FUSED SILICA ON Al SUBSTRATE
40.	0.984	0.090	0.017	0.955	0.163	0.267
45.	0.980	0.108	0.018	0.960	0.190	0.113
50.	0.965	0.100	0.037	0.970	0.185	0.184
55.	0.982	0.107	0.033	0.967	0.178	0.248
60.	1.00	0.114	0.034	0.973	0.158	0.260
65.	0.976	0.075	0.026	0.990	0.158	0.068
70.	0.988	0.113	0.020	0.966	0.155	0.103
75.	0.998	0.212	0.030	0.984	0.154	0.133
80.	1.01	0.187	0.033	0.994	0.140	0.280
85.	0.983	0.166	0.050	0.984	0.135	0.330
90.	1.03	0.169	0.046			

TABLE 5.1-2

PERCENT REFLECTANCE OF CONVAIR SUPPLIED SAMPLES

WAVELENGTH IN MICRONS	V. D. GOLD ON QUARTZ CRYSTAL	ZINC OXIDE IN POTASSIUM SILICATE	3M VELVET BLACK PAINT	SHERWIN WILLIAMS FLAT ACRYLIC WHITE PAINT
2.5	0.963	0.503	0.017	0.252
3.	0.955	0.212	0.016	0.074
3.5	0.953	0.122	0.018	0.044
4.	0.954	0.120	0.018	0.110
4.5	0.956	0.100	0.018	0.139
5.	0.957	0.086	0.018	0.112
5.5	0.952	0.058	0.018	0.074
6.	0.947	0.043	0.017	0.043
6.5	0.951	0.036	0.016	0.055
7.	0.959	0.034	0.016	0.037
7.5	0.955	0.027	0.014	0.039
8.	0.962	0.025	0.013	0.038
8.5	0.965	0.012	0.045	0.033
9.	0.946	0.008	0.096	0.038
9.5	0.948	0.007	0.113	0.053
10.	0.960	0.007	0.063	0.159
10.5	0.961	0.009	0.042	0.072
11.	0.956	0.010	0.040	0.054
11.5	0.956	0.010	0.038	0.041
12.	0.957	0.012	0.034	0.032

TABLE 5.1-2

PERCENT REFLECTANCE OF CONVAIR SUPPLIED SAMPLES

WAVELENGTH IN MICRONS	V.D. GOID ON QUARTZ CRYSTAL	ZINC OXIDE IN POTASSIUM SILICATE	3M VELVET BLACK PAINT	SHERWIN WILLIAMS FIAT ACRYLIC WHITE PAINT
12.5	0.957	0.008	0.033	0.023
13.	0.960	0.007	0.036	0.019
14.	0.955	0.006	0.034	0.026
15.	0.954	0.005	0.032	0.085
16.	0.966	0.004	0.033	0.062
17.	0.966	0.003	0.030	0.051
18.	0.965	0.010	0.030	0.049
19.	0.963	0.012	0.030	0.049
20.	0.968	0.016	0.026	0.091
21.	0.966	0.016	0.061	0.151
22.	0.966	0.018	0.085	0.250
23.	0.971	0.020	0.074	0.205
24.	0.966	0.025	0.065	0.163
25.	0.969	0.026	0.065	0.162
26.	0.967	0.030	0.060	0.195
27.	0.973	0.027	0.058	0.157
28.	0.966	0.023	0.056	0.136
29.	0.964	0.023	0.055	0.146
30.	0.972	0.020	0.054	0.151
35.	0.970	0.027	0.055	0.160

TABLE 5.1-2

PERCENT REFLECTANCE OF CONVAIR SUPPLIED SAMPLES

WAVELENGTH IN MICRONS	V.D. GOLD ON QUARTZ CRYSTAL	ZINC OXIDE IN POTASSIUM SILICATE	3M VELVET BLACK PAINT	SHERWIN WILLIAMS FLAT ACRYLIC WHITE PAINT
40.	0.963	0.019	0.065	0.160
45.	0.970	0.040	0.031	0.160
50.	0.968	0.022	0.047	0.161
55.	0.980	0.041	0.063	0.171
60.	0.974	0.027	0.067	0.149
65.	0.976	0.047	0.065	0.157
70.	0.972	0.056	0.058	0.190
75.	0.974	0.043	0.074	0.161
80.	0.991	0.054	0.093	0.241
85.	0.997	0.033	0.075	0.144
90.		0.052	0.061	0.254

TABLE 5.1-3

PERCENT REFIECTANCE OF CONVAIR SUPPLIED SAMPLES HOHLRAUM DATA

WAVELENGTH IN MICRONS	ZINC OXIDE IN POTASSIUM SILLICATE	3M BLACK VELVET	SHERWIN WILLIAMS ACRYLIC FLAT WHITE
2.5	0.520	0.030	0.320
3.	0.130	0.030	0.100
3.5	0.080	0.030	0.075
4.	0.080	0.030	0.170
4.5	0.085	0.045	0.225
5.	0.065	0.035	0.190
5.5	0.050	0.040	0.100
6.	0.045	0.035	0.075
6.5	0.045	0.035	0.090
7.	0.040	0.035	0.065
7.5	0.040	0.035	0.070
8.	0.030	0.040	0.055
8.5	0.020	0.075	0.050
9.	0.020	0.170	0.070
9.5	0.025	0.125	0.080
10.	0.030	0.075	0.130
10.5	0.030	0.060	0.105
11.	0.030	0.065	0.075
11.5	0.040	0.060	0.065
12.	0.020	0.055	0.045

TABLE 5.1-3

PERCENT REFLECTANCE OF CONVAIR SUPPLIED SAMPLES HOHLRAUM DATA

WAVELENGTH IN MICRONS	ZINC OXIDE IN POTASSIUM SILLICATE	3M BLACK VELVET	SHERWIN WILLIAMS ACRYLIC FLAT WHITE
12.5	0.020	0.060	0.040
13.	0.020	0.060	0.035
14.	0.020	0.055	0.045
15.	0.030	0.065	0.115
16.	0.020	0.060	0.075
17.	0.020	0.060	0.075
18.	0.075	0.070	0.090
19.	0.090	0.055	0.105
20.	0.110	0.075	0.140
21.	0.110	0.130	0.250
22.	0.125	0.130	0.260
23.	0.135	0.120	0.230
24.	0.150	0.105	0.200
25.	0.165	0.120	0.175
26.	0.155	0.100	0.205
27.	0.170	0.105	0.200
28.	0.170	0.125	0.200
29.	0.130	0.090	0.185
30.	0.185		

TABLE 5.2-1

ANGULAR MEASUREMENTS ON COBALT OXIDE AND 3M VELVET BLACK PAINT BAFFLE INSTALLED IN FRONT OF TOROID MIRROR

MEASUREMENT ANGLE IN DEGREES	COBALT OXIDE					3M VELVET BLACK PAINT				
	WAVELENGTH IN MICRONS									
	2.5	3.0	5.0	10.	20.	2.5	3.0	5.0	10.	20.
	REFLECTANCE					REFLECTANCE				
6	0.590	0.534	0.936	1.01	1.00	0.017	0.017	0.019	0.072	0.035
10	0.617	0.572	0.980	1.06	1.05	0.017	0.017	0.019	0.073	0.035
20	0.602	0.571	0.974	1.04	1.05	0.018	0.018	0.020	0.075	0.036
30	0.594	0.577	0.984	1.05	1.05	0.020	0.020	0.022	0.078	0.038
40	0.589	0.584	0.992	1.05	1.05	0.023	0.023	0.024	0.084	0.046
50	0.578	0.583	0.978	1.03	1.04	0.027	0.027	0.029	0.095	0.059
60	0.489	0.478	0.736	0.778	0.770	0.037	0.037	0.039	0.116	0.098
70	0.043	0.057	0.026	0.021	0.023	0.057	0.057	0.061	0.136	0.109
73	0.040	0.053	0.027	0.025	0.027	0.065	0.064	0.064	0.121	0.073
75	0.038	0.050	0.036	0.043	0.049	0.072	0.074	0.068	0.106	0.057
77	0.041	0.052	0.049	0.062	0.070	0.080	0.077	0.071	0.100	0.060

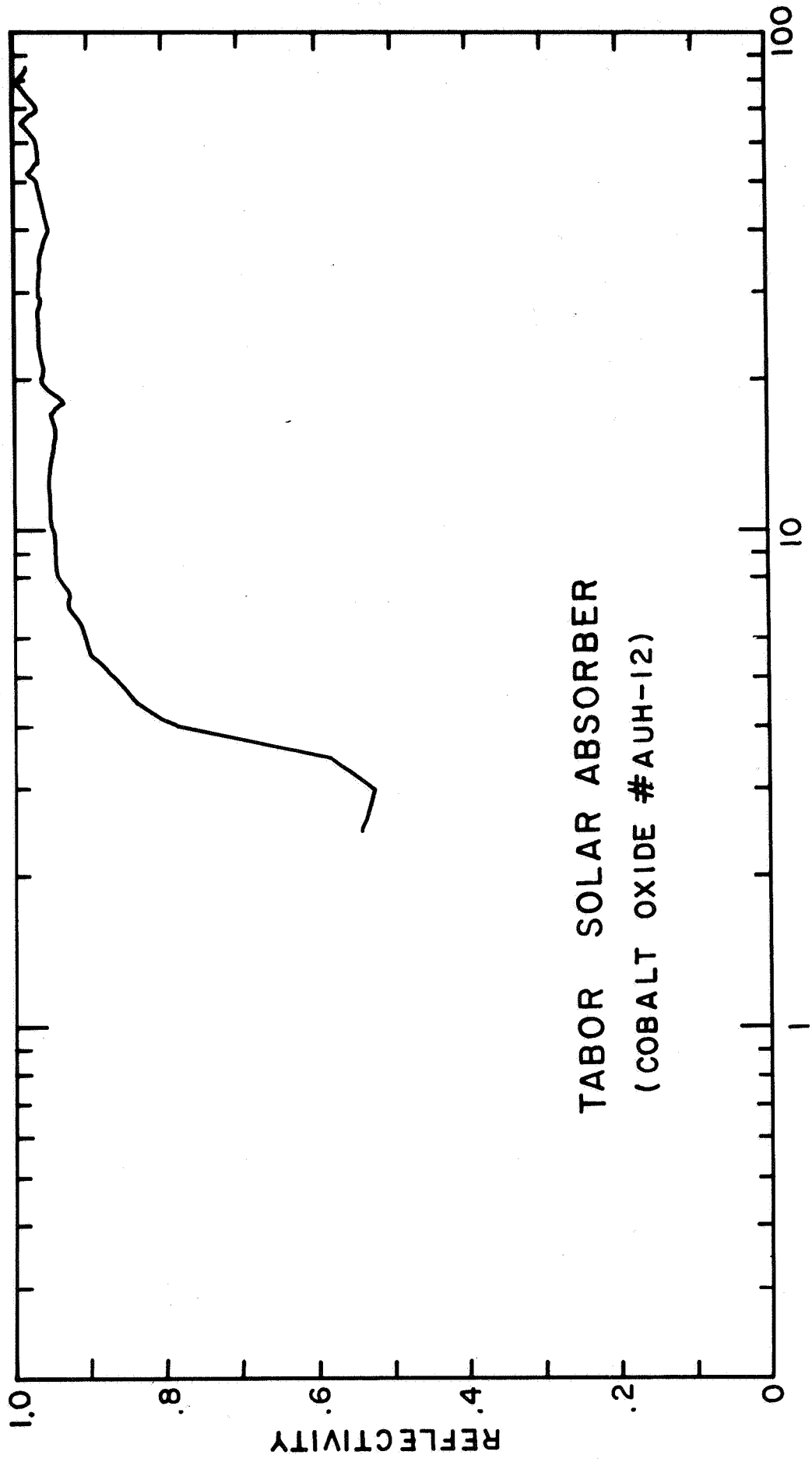
TABOR SOLAR ABSORBER
(COBALT OXIDE #AUH-12)
REFLECTIVITY
1.0
.8
.6
.4
.2
0
1
10
100
WAVELENGTH IN MICRONS

FIGURE 5.1-1

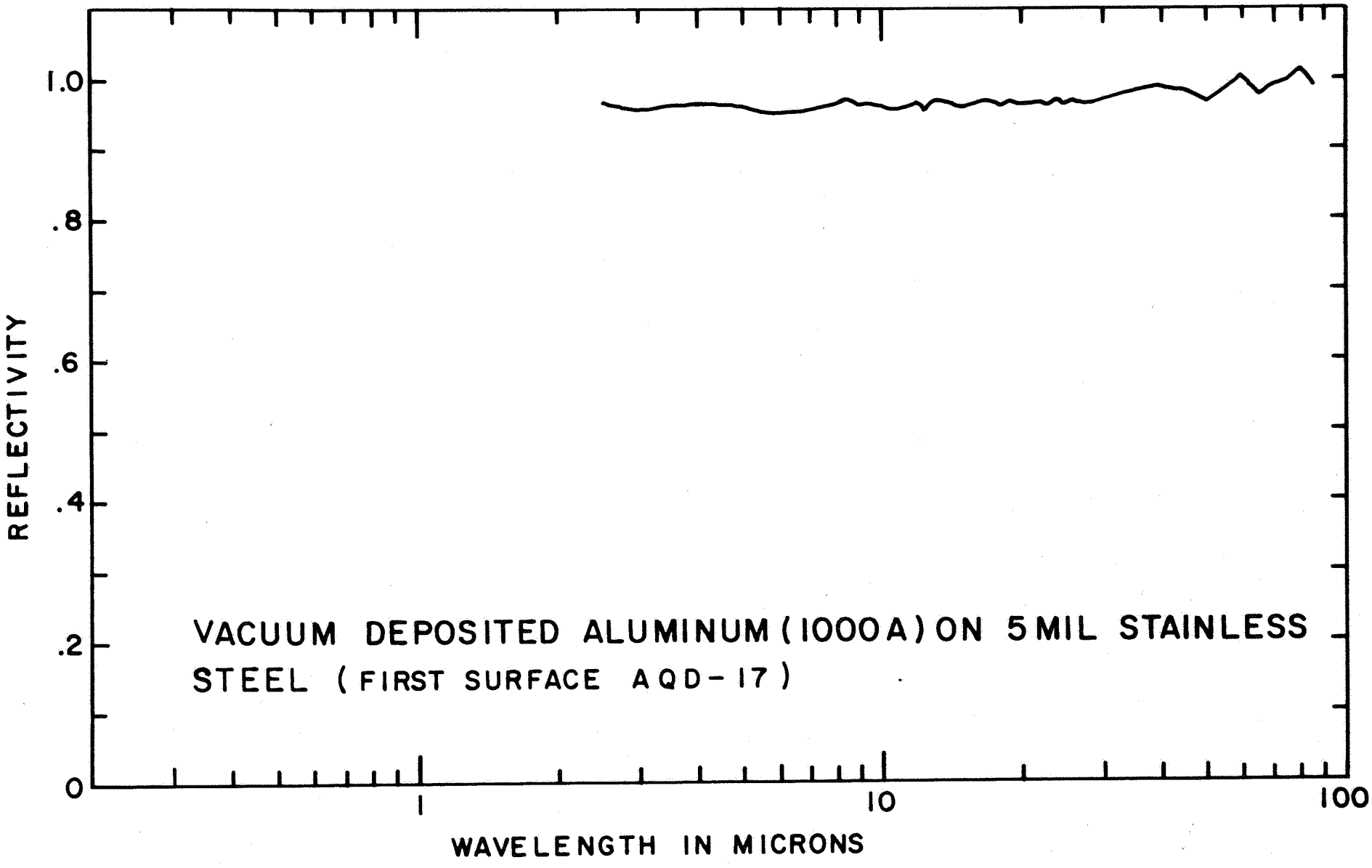
1.0
.8
.6
.4
.2
0
REFLECTIVITY
VACUUM DEPOSITED ALUMINUM (1000A) ON 5 MIL STAINLESS STEEL (FIRST SURFACE AQD-17)
1
10
100
WAVELENGTH IN MICRONS

FIGURE 5.1-2

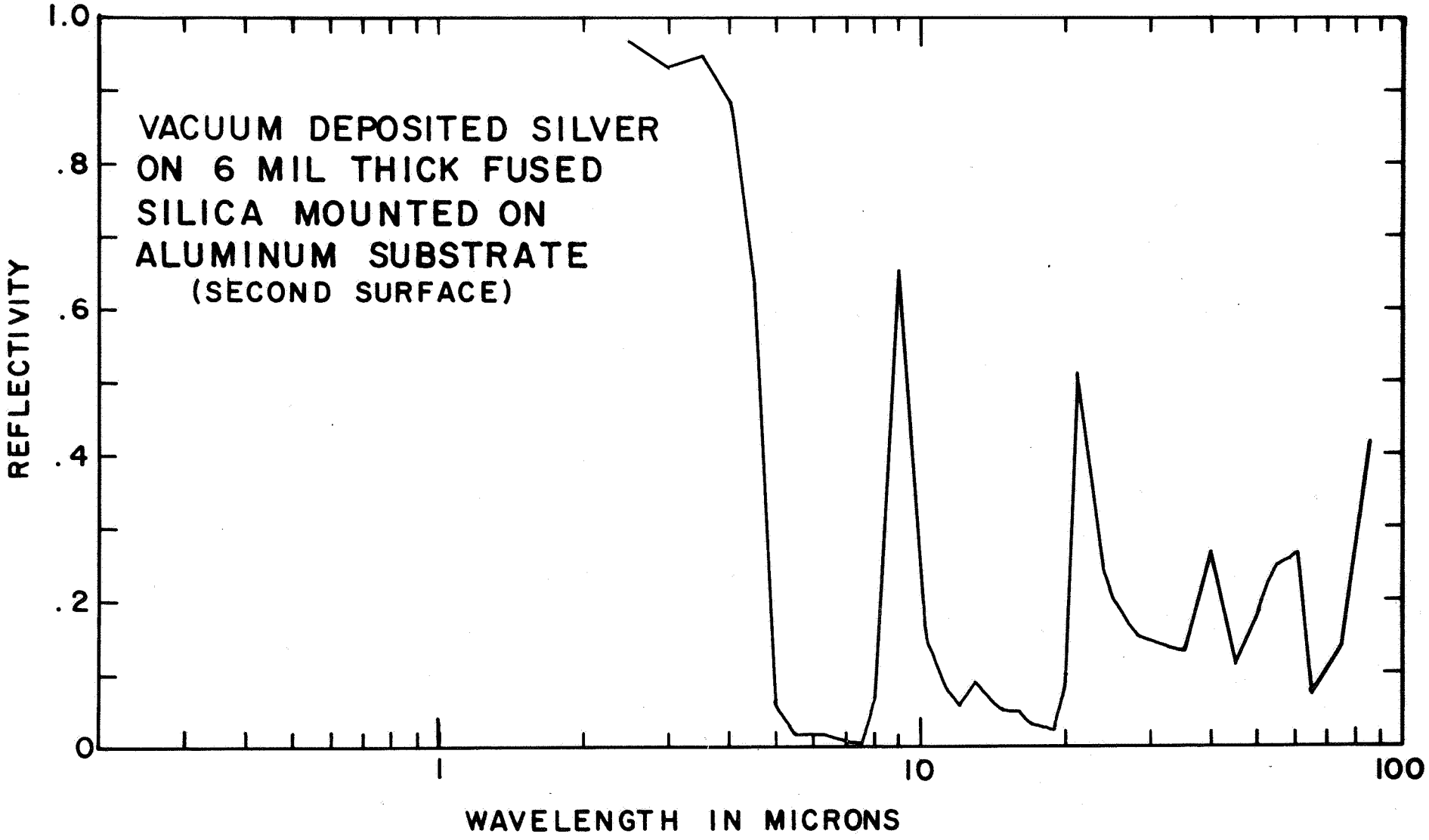
VACUUM DEPOSITED SILVER
ON 6 MIL THICK FUSED
SILICA MOUNTED ON
ALUMINUM SUBSTRATE
(SECOND SURFACE)
REFLECTIVITY
1.0
.8
.6
.4
.2
0
1
10
100
WAVELENGTH IN MICRONS

FIGURE 5.1-3

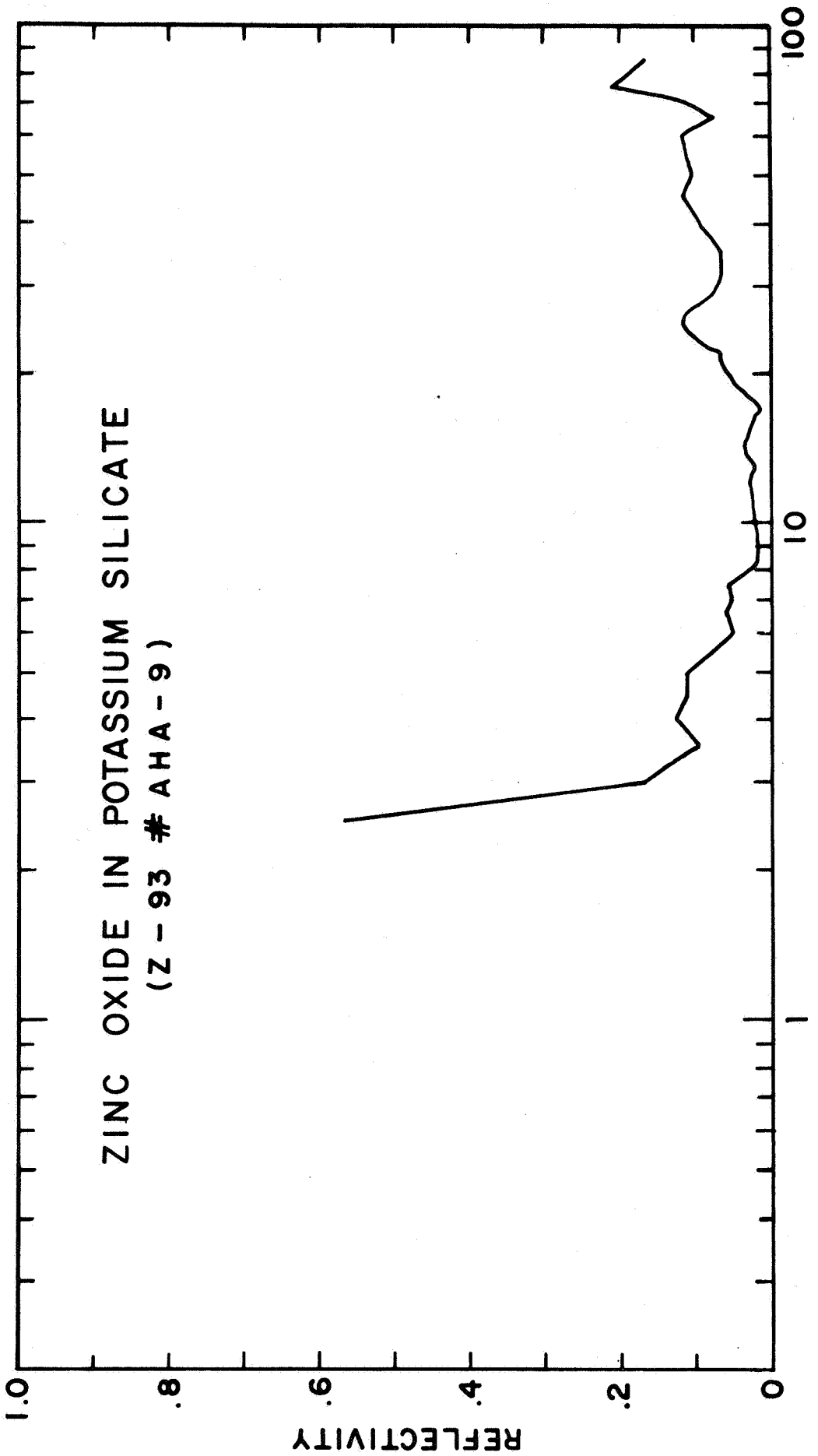
ZINC OXIDE IN POTASSIUM SILICATE
(Z-93 # AHA-9)
REFLECTIVITY
1.0
.8
.6
.4
.2
0
1
10
100
WAVELENGTH IN MICRONS

FIGURE 5.1-4

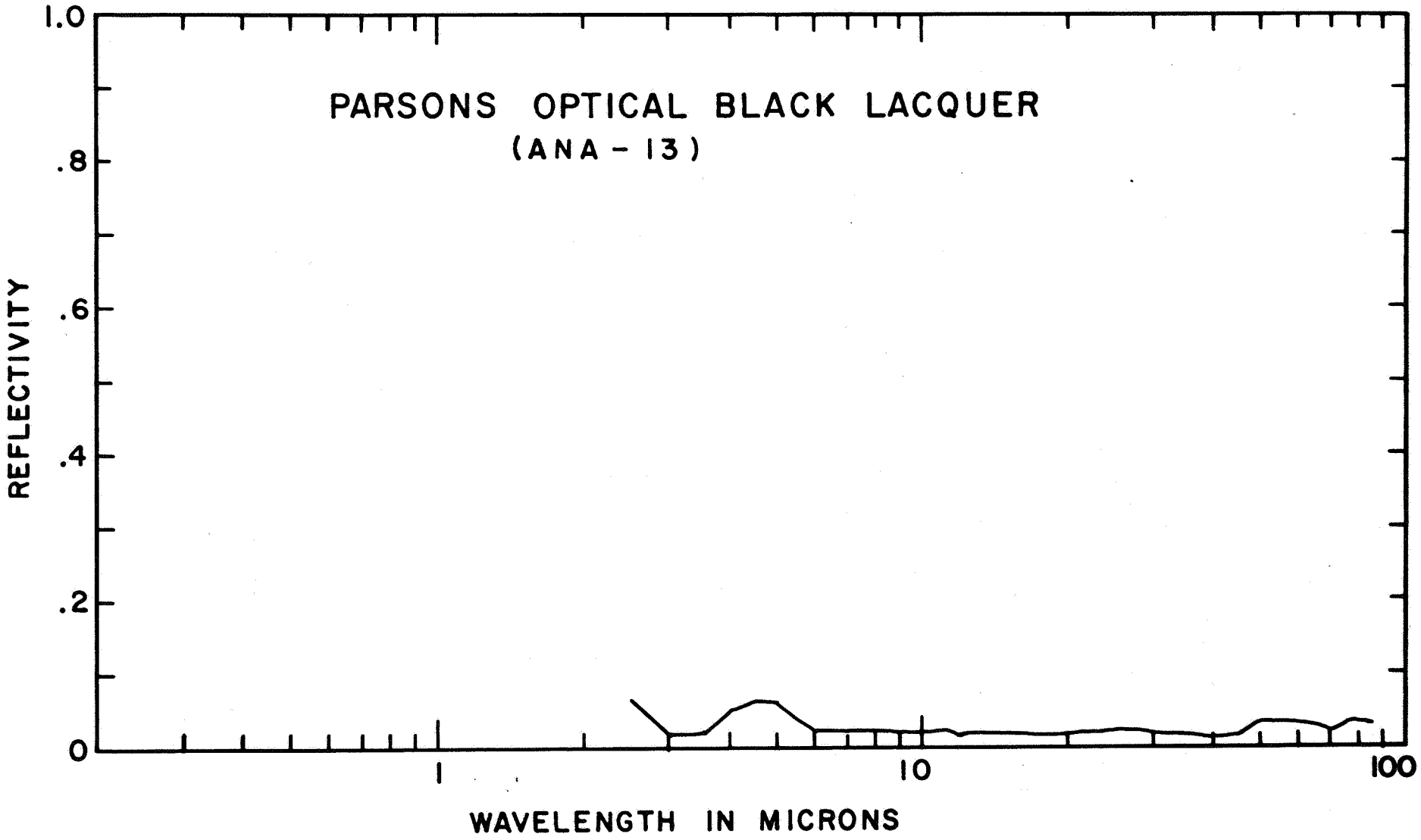
PARSONS OPTICAL BLACK LACQUER
(ANA - 13)
REFLECTIVITY
1.0
.8
.6
.4
.2
0
1
10
100
WAVELENGTH IN MICRONS

FIGURE 5.1-5

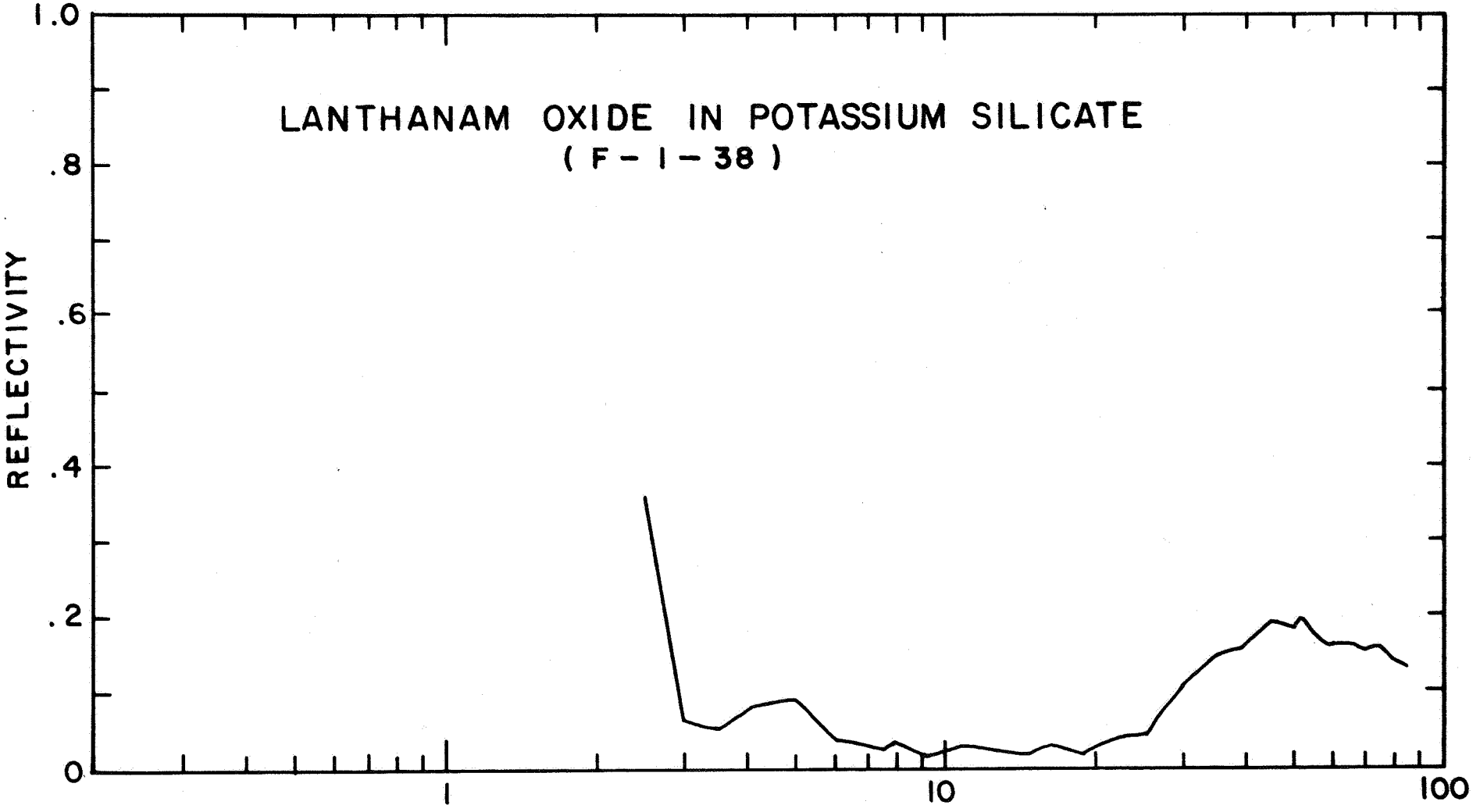
LANTHANAM OXIDE IN POTASSIUM SILICATE
(F - 1 - 38)
REFLECTIVITY
1.0
.8
.6
.4
.2
0
1
10
100
WAVELENGTH IN MICRONS

FIGURE 5.1-6

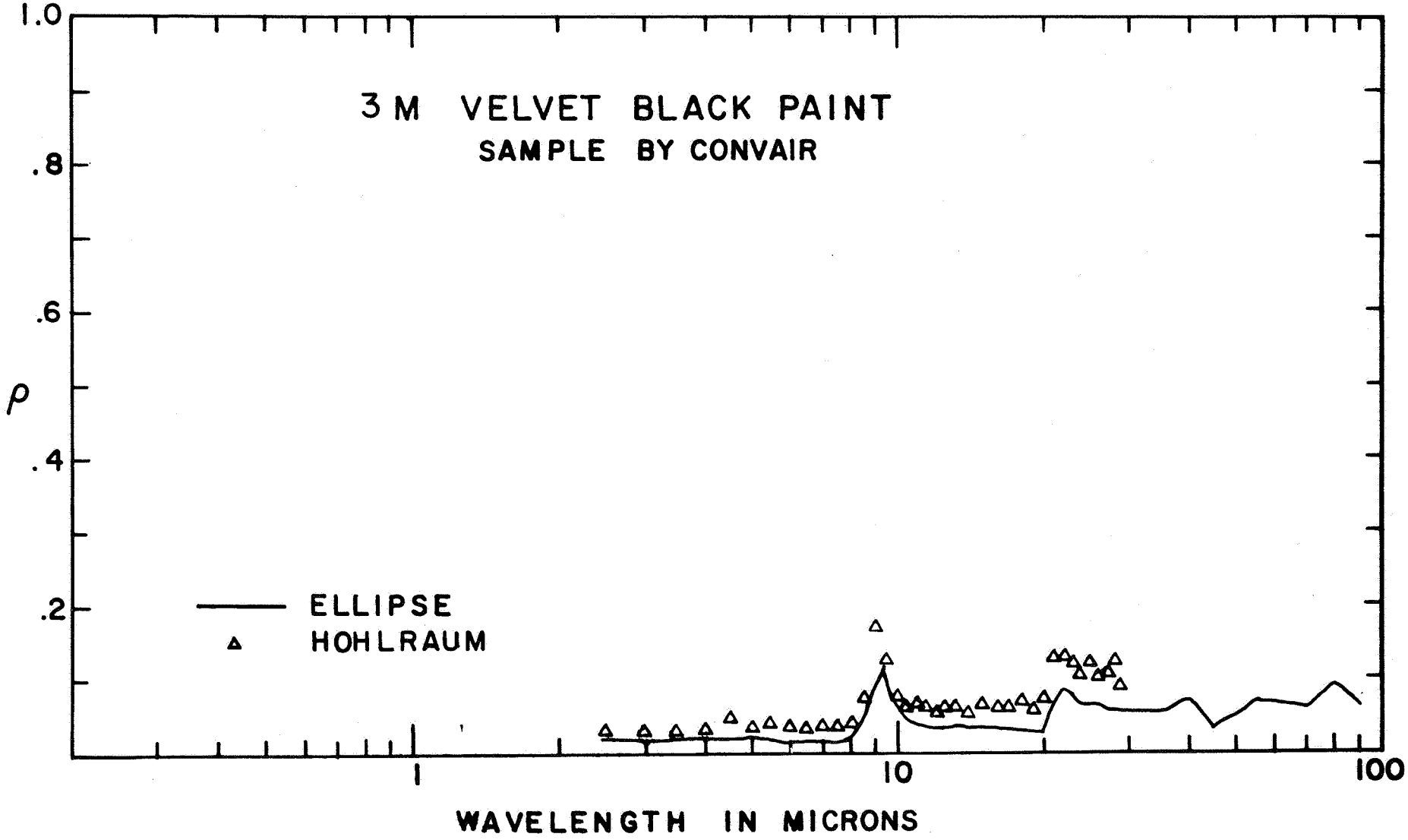
3 M VELVET BLACK PAINT
SAMPLE BY CONVAIR
1.0
.8
.6
.4
.2
ρ
ELLIPSE
HOHLRAUM
1
10
100
WAVELENGTH IN MICRONS

FIGURE 5.1-7

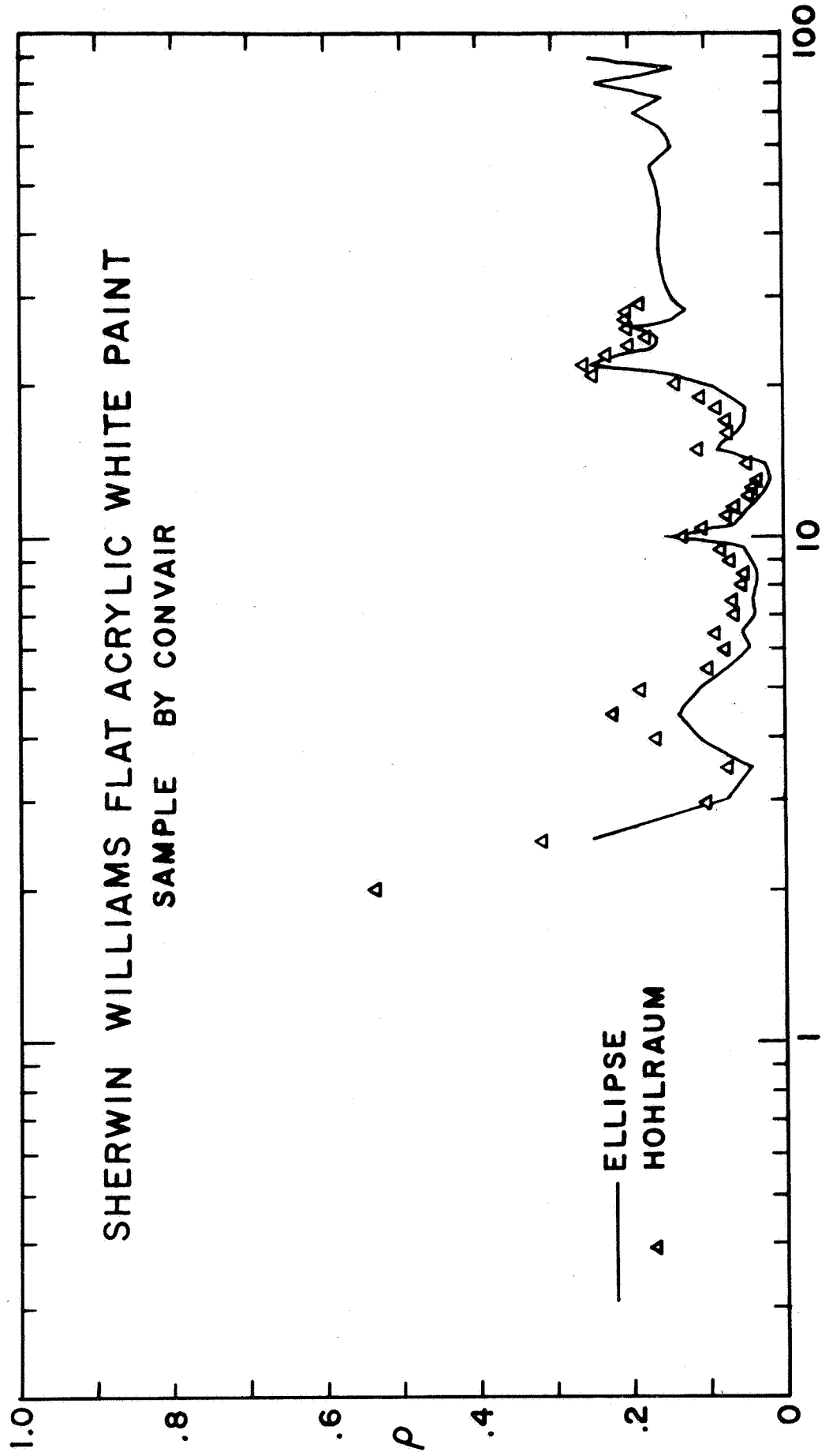
SHERWIN WILLIAMS FLAT ACRYLIC WHITE PAINT
SAMPLE BY CONVAIR
ELLIPSE
HOHLRAUM
ρ
1.0
.8
.6
.4
.2
0
1
10
100
WAVELENGTH IN MICRONS

FIGURE 5.1-8

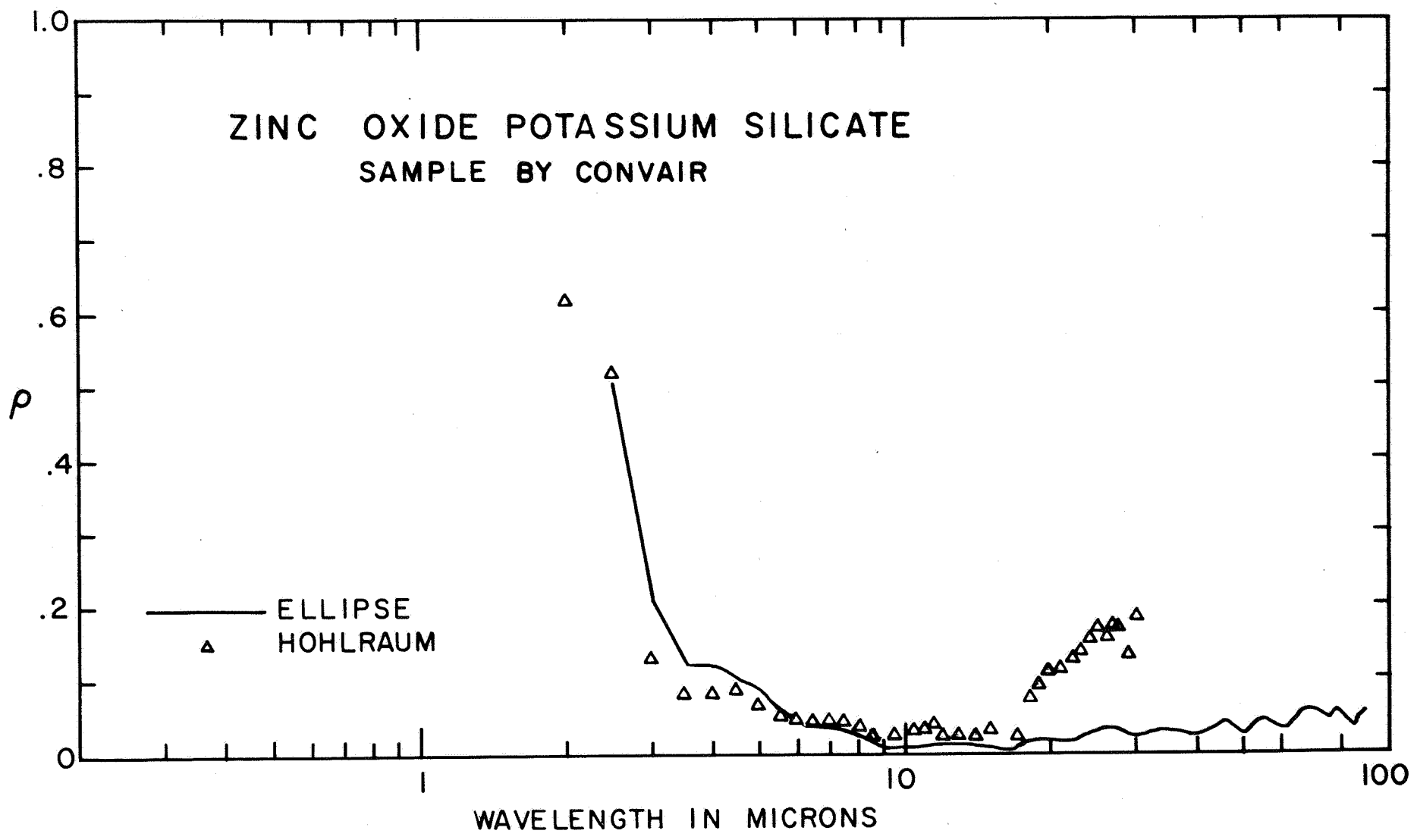
ZINC OXIDE POTASSIUM SILICATE
SAMPLE BY CONVAIR
1.0
.8
.6
.4
.2
0
ρ
ELLIPSE
HOHLRAUM
1
10
100
WAVELENGTH IN MICRONS

FIGURE 5.1-9

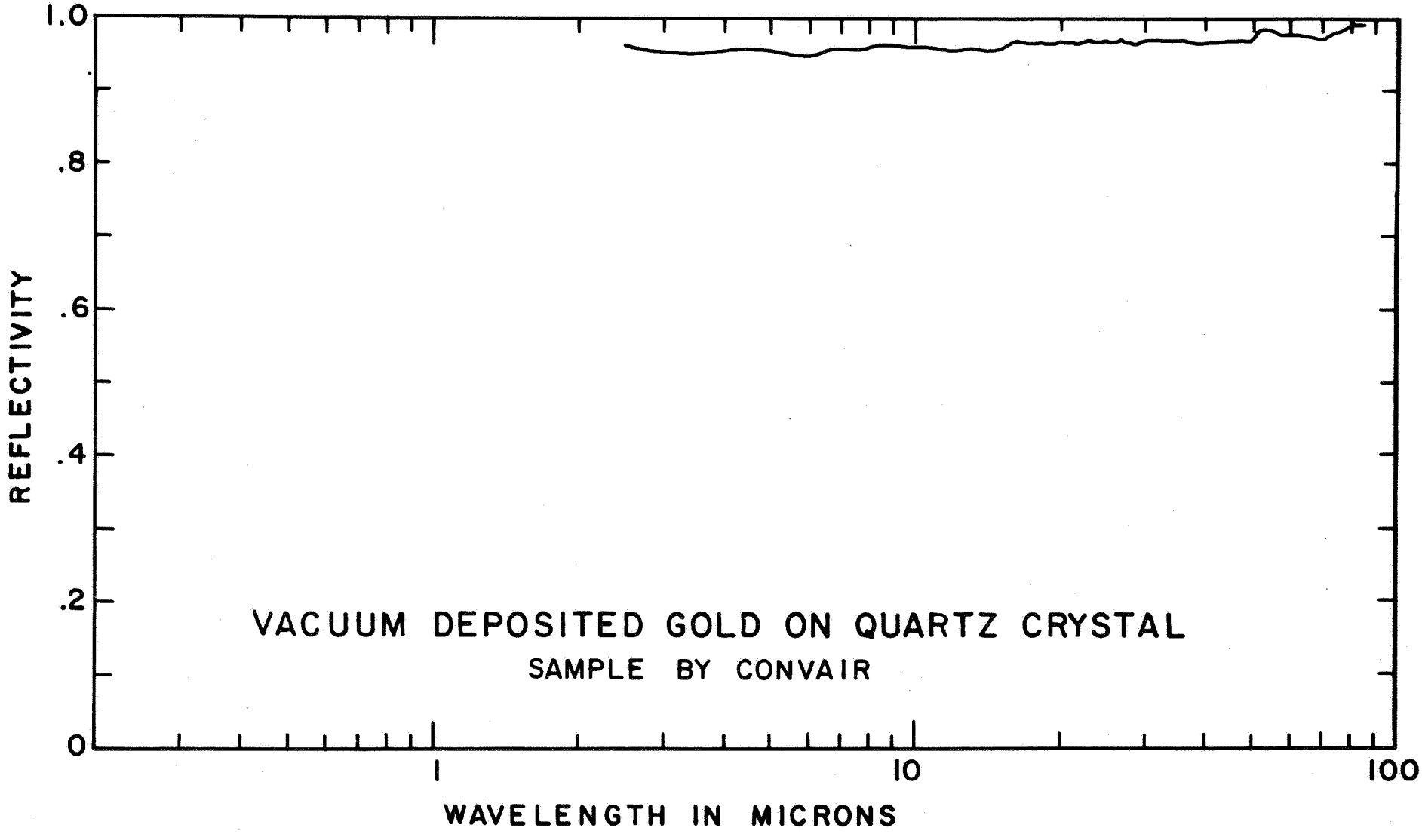
1.0
.8
.6
.4
.2
0
REFLECTIVITY
VACUUM DEPOSITED GOLD ON QUARTZ CRYSTAL
SAMPLE BY CONVAIR
1
10
100
WAVELENGTH IN MICRONS

FIGURE 5.1-10

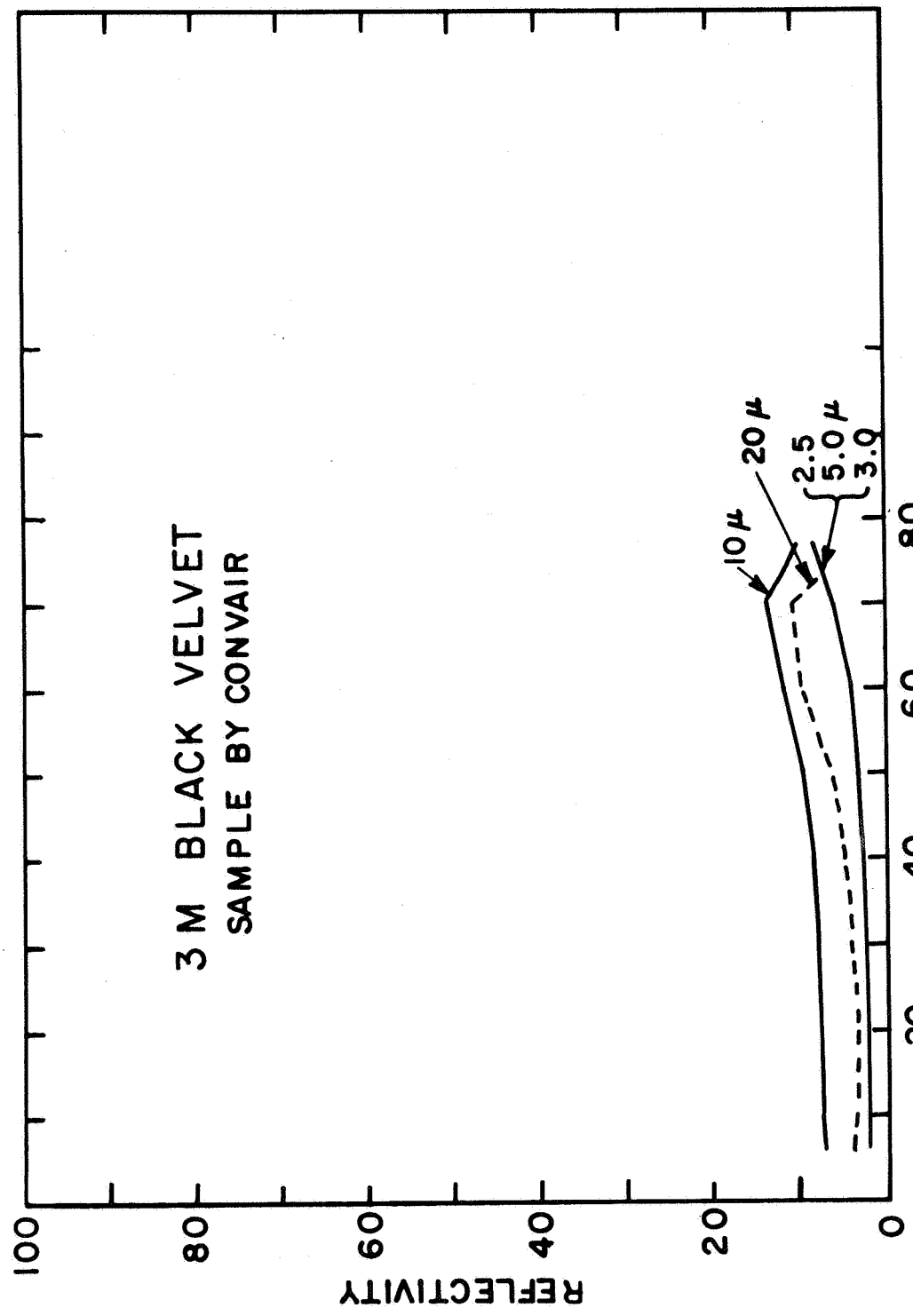
3 M BLACK VELVET
SAMPLE BY CONVAIR
10 μ
20 μ
2.5
5.0 μ
3.0
REFLECTIVITY
0
20
40
60
80
100
ANGLE, DEGREES
20
40
60
80

FIGURE 5.2 – 1

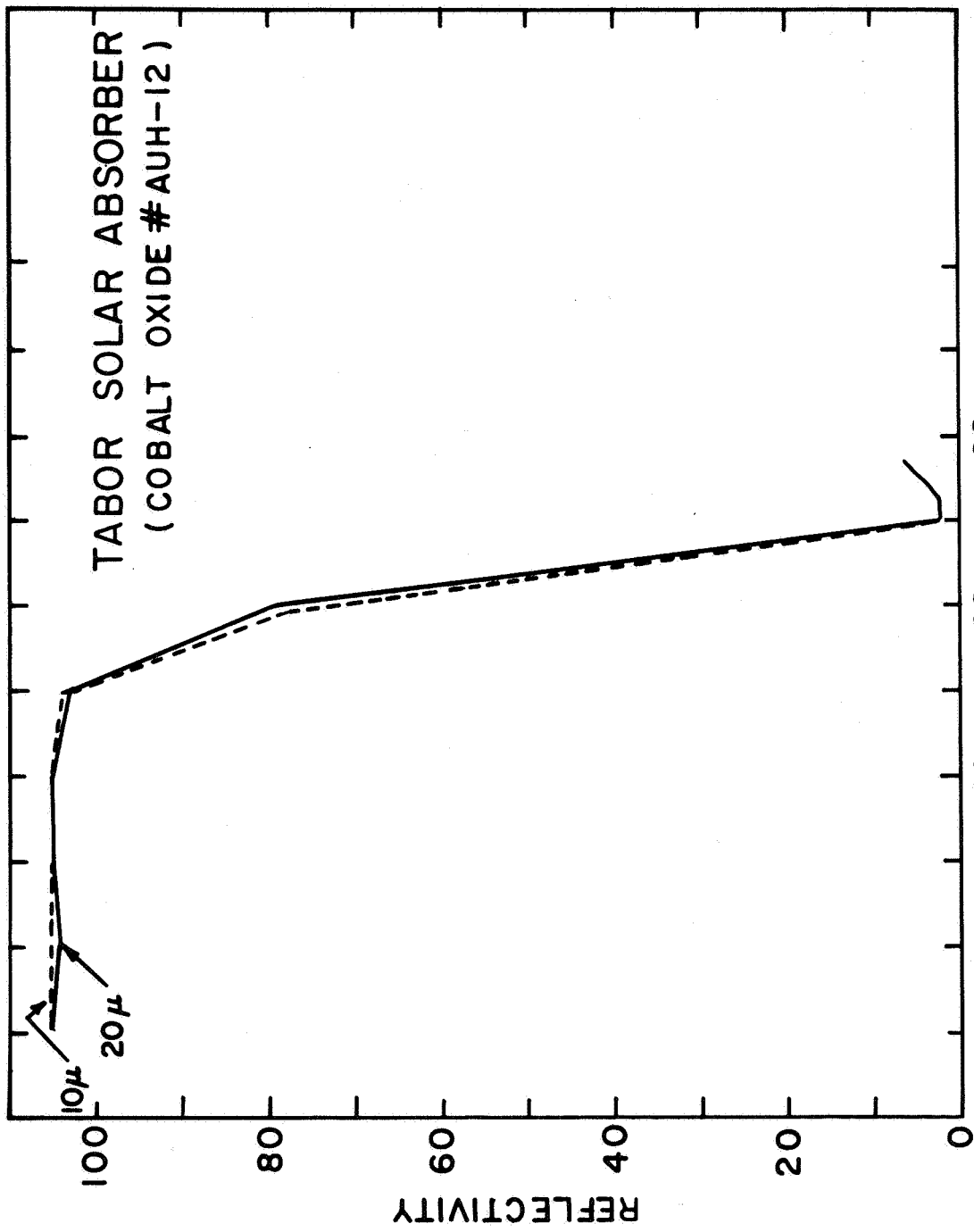
TABOR SOLAR ABSORBER
(COBALT OXIDE #AUH-12)
10μ
20μ
REFLECTIVITY
0
20
40
60
80
100
20
40
60
80
ANGLE, DEGREES

FIGURE 5.2-2

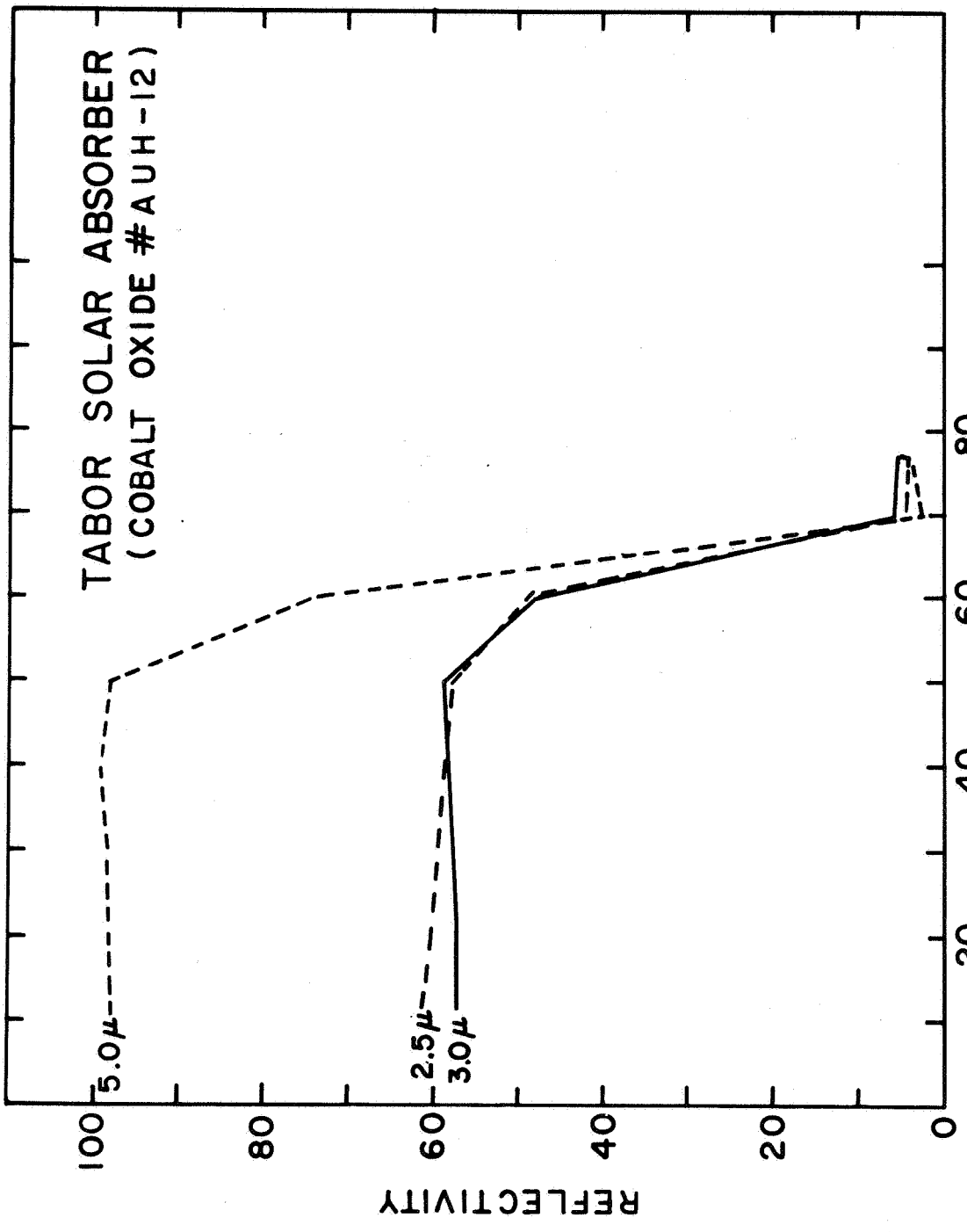
TABOR SOLAR ABSORBER
(COBALT OXIDE #AUH-12)
5.0μ
2.5μ
3.0μ
REFLECTIVITY
100
80
60
40
20
0
20
40
60
80
ANGLE, DEGREES

FIGURE 5.2-3

6.0 MAINTENANCE

For maintenance of the amplifier, digital volt meter and digital recorder, reference is made to the respective manuals.

Perhaps some note regarding the cleaning of the optical surfaces in the tank is required. The mirrors (M1, M2, M3, and M4) if contaminated can be cleaned and reused or the surfaces can be stripped and new aluminum placed on the mirrors. Cleaning of uncoated front surfaced aluminum mirrors is always ticklish and the surface, when cleaned, is never as good as the original surface. It has been found in our laboratories that cleaning can be accomplished satisfactorily by using isopropyl alcohol and a high grade of cotton. The alcohol is poured on the mirror and very lightly rubbed with the cotton. The rubbing is continued until the mirror is dry. This works fairly well, the mirror will end up with very fine scratches which are visible to the eye. This makes no difference for the infrared region of the spectrum. If it were desired to use the instrument in the UV portion of the spectrum they would probably be unacceptable and re-aluminizing of the mirrors would be required. The large hemi-ellipsoid may require cleaning. Actually, it is probably more likely that it will require cleaning than the other mirrors will require cleaning. This can be accomplished in the same fashion as was described for the mirrors - use isopropyl alcohol very carefully, wipe the surfaces with cotton, pour out the alcohol, and continue to wipe the surfaces until they are dry, with soft cotton. If it becomes necessary to re-aluminize the ellipse, Convair's experience in this regard may be of value. The hemi-ellipsoid

was received with a good coating of aluminum but during tests, various accidents and difficulty it has degraded to the point that it was necessary to re-aluminize. Our optical shops stripped the aluminum in the conventional fashion using a sulphuric acid copper sulfate mixture. The surface was then cleaned in the accepted fashion and re-aluminized. The coating looked good on removal from the chamber but the next day aluminum along the crack joining the two aluminum quarter ellipsoids flaked and peeled. This was attributed to improper cleaning and the entire operation was repeated again. The same difficulty was encountered. It was then determined that some of the cleaning solution was trapped in the crack space and that the fumes were causing the aluminum to corrode and peel. The ellipse was soaked with a solution of sodium carbonate to neutralize any acid that remained in the cracks. It was placed in a vacuum chamber and pumped down and left overnight to attempt to dry out any moisture in the cracks, and it was then aluminized again and in this case the aluminization was successful. If re-aluminization is necessary, care should be taken not to introduce corrosive chemicals in the crack between the two quarters.

REFERENCE

1. W. M. Brandenberg, "Focusing Properties of Hemispherical and Ellipsoidal Mirror Reflectometers," J. Opt. Soc. Am. 54, 1235 (1964).

APPENDIX

RESEARCH REPORT

THE SIGNIFICANCE OF INFRARED REFLECTANCE DATA BEYOND 22 MICRONS FOR SPACECRAFT THERMAL ANALYSIS

Ordinarily, the thermal design engineer is concerned with the solar reflectance of the spacecraft surface material and sometimes with the reflectance of earth emitted energy and sunlight reflected from the earth. The solar reflectivity is adequately covered by integrating sphere devices operating from about .3 μ to about 2 μ and data past 22 μ is, of course, not required. Earth emission at wavelengths past 22 μ can be significant, accounting for as much as 20% of the infrared flux.

The engineer is also concerned with the emission from the vehicle. Direct emission measurements from surfaces in the vicinity of 300°K are difficult to make and are not generally practical. As a consequence, reflectance measurements are made and the emittance is calculated from the equation

$$\epsilon_\lambda + \rho_\lambda = 1 \quad .$$

At 300°K a negligible amount of the blackbody energy (.001%) falls at wavelengths shorter than 2.5 μ. However, 22% of the blackbody energy lies at wavelengths past 22 μ. As the temperature of the emitting surface drops, the percentage of the blackbody energy covered out to 22 μ also drops. For example, at 200°K only 55% of the energy of a 200°K blackbody would be obtained out to 22 μ and at 100°K 90% of the energy of a black-

body would lie at wavelengths longer than 22 μ. Thus, it is seen that for 300°K surface temperatures in space the use of wavelengths past 22 μ would be highly desirable and as the temperature of the emitting surface becomes lower, the requirement for greater wavelength coverage increases. The occurrence of temperatures below 300°K is not uncommon in spacecraft practice and for some problems wavelength coverage past 22 μ becomes practically a necessity.

Actually for a rigorous emittance determination, the temperature of the sample should be at the temperature that the emittance value is desired. Ordinarily, however, this is not done. The sample is maintained at the ambient (tap water) temperature and the reflectance is determined and the emittance at various temperatures is calculated from the reflectance vs wavelength curve. This procedure has inherent in it the assumption that the emittance as a function of wavelength does not change rapidly with temperature. This is probably a good assumption for most materials. However, it has not been checked in any great detail and work in this area is probably required.

Some consideration has been given to the storage of liquid hydrogen for deep space missions. Multiple radiation shields facing the sun can reduce the flux from the sun to a very small value and if the radiation from the hydrogen tank equals or exceeds the flux through the multiple shielding, then storage of the hydrogen without loss would be possible. The radiation from the surface at liquid hydrogen temperature is of course, almost infinitesimal. However, the heat penetrating the multiple shields can similarly be reduced so that the actual emittance of

the hydrogen tank surface becomes important. There has been no experimental way to determine reflectances and thus emittances at the wavelengths required, in the vicinity of 300 μ. (Calorimetric methods are also difficult at liquid hydrogen temperature.) This wavelength coverage is, of course, beyond the capabilities of this instrument but it is reasonable that sooner or later requirements will exist for the determination of reflectances and emittances in the 200 to 300 μ wavelength region.

THE IMPORTANCE OF SPECTRAL REFLECTANCE DATA FOR CHARACTERIZING COATING CONSTITUENTS

Paint coatings are made up of pigments and binders. In the research approach to evolving better coatings, the separate study of the binder and the pigment is desirable as well as the determination of the characteristics of the combination of the two. Synergistic effects are possible. The total hemispherical emittance often used for engineering answers to thermal control problems are of only very limited value in this area. The determination of the spectral reflectance provides a means of "finding out what's going on." The wavelength regions of high and low emittance are determined, the separate effects of the binder, and the pigment are determined and so forth. The ellipsoidal far infrared spectroreflectometer is so constructed that it can be used with unconsolidated pigment materials. Relative measurements are made against an aluminized surface and if care is exercised in turning the sample assembly so as not to disturb the powders. Thus, an unconsolidated pigment power could be

studied without the necessity of compacting it, wetting it, and spraying it.

A COMPARISON OF SPECTRAL REFLECTANCE DATA VS CALORIMETRIC TOTAL HEMISPHERICAL DATA FROM MEASUREMENT CONSIDERATIONS

It seems to be one of the maxims of science that no one measurement determines everything desired. This is certainly the case with regards to the comparison between calorimetric measurements and spectral reflectance measurements. The problem is perhaps best discussed from the point of view of the advantages and the disadvantages of each method.

The advantages of the calorimetric method are: 1) In one measurement the total hemispherical emittance is obtained without the requirement for integration procedures. 2) The measurements have no "wavelength coverage" limitations such that part of the data is not obtained due to lack of wavelength coverage. 3) The data are obtained at the temperature desired and the uncertainty of the calculation of emittance at one temperature from an emittance as a function of wavelength curve taken at another temperature does not exist.

The disadvantages of the calorimetric method are: 1) Fairly complex apparatus is involved so the rapid processing of samples is not possible. The data reduction techniques are often rather involved. 2) Further calorimetric measurements do not furnish data which are as useful as spectral reflectance data are for research purposes. One "total answer" is obtained. Little information is obtained as to how to improve, modify, and otherwise change the coating to improve its performance.

The advantages of the spectral reflectance method are: 1) Samples can be expeditiously processed. 2) Information is obtained on which to base changes or modifications to obtain an improved type of coating. Knowledge can be obtained as to where the reflectance is high or low from which conclusions can be drawn regarding as to which component should be altered to attain better performance.

The disadvantages of the spectral reflectance method are: 1) Often the emittance is required at a temperature other than the temperature at which the sample was maintained during measurement and the thermostating of the sample is often difficult or impossible. 2) It is necessary to perform an integration over wavelength to obtain total normal emittance. This is not a serious drawback inasmuch as the high speed computer techniques are available to perform this operation. If total hemispherical emittance is required, then the angular dependence of the reflectance must be determined and integration over angle as well as wavelength is required. This considerably increases the amount of time required to obtain results.

RECOMMENDATIONS OF FURTHER STUDY AREAS FOR LONG WAVELENGTH INFRARED TECHNIQUES

The possible areas of research that come to mind have been touched on in the above paragraphs. One area that needs checking, as noted above, is the dependence of the reflectance vs wavelength curve on the temperature of the sample. The question needs to be answered as to how far from the temperature of the sample is the data useful for calculating emittances at other temperatures, what are the effects for different types of surfaces, metals, and non-metals, etc.

Another area which should be investigated is the experimental checking of spectral reflectance data with calorimetric data. With the possibility of running reflectance at closely spaced angles, it should be possible to very closely match the calorimetrically determined emittance.

A general survey of the most commonly used materials in the wavelength region past 22 μ would be useful inasmuch as the largest part of the data obtained to date has a cutoff point somewhere between 15 and 30 μ. It would be useful to the aerospace thermal control community to have spectral data on the thermal control coatings out to 100 μ.