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BIDIRECTIONAL SPECTROREFLECTOMETER MANUAL

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GENERAL DYNAMICS/CONVAIR

FOREWORD

The Bidirectional Spectroreflectometer was designed and built for NASA, Manned Space Flight Center, Huntsville, Alabama under Contract NAS8-20512.

Bidirectional Spectroreflectometer

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CREDITS

In chronological order, the credit for the bidirectional spectroreflectance hardware and the techniques described in this manual are as follows: (a) The initial concept and optical design of an instrument which uses parallel light to "over-illuminate" and "over-detect" a sample: W. M. Brandenberg; (b) The arithmetic for data reduction using the "over-illumination" system (after a few missteps): W. M. Brandenberg and J. T. Neu with helpful discussions with O. W. Clausen; (c) The design of the general features of the irradiation and detection system: W. M. Brandenberg; (d) Design of the optical spectrometer-to-sample system: J. T. Neu; (e) Mechanical Design, Design Drawings (all), Machining and Assembly: W. Torgeson (80% + of it); (f) Optical Assembly Checkout and Calibration: J. T. Neu and R. E. Gillson; (g) The use of a half silvered mirror to allow "zero" angle measurements was suggested by W. Snoddy of NASA MSFC; (h) Data Computer Reduction Program, William Breckenridge, E. J. Philbin, J. T. Neu, W. M. Brandenberg and W. H. Gallaher; (i) Measurements on the Aluminum Samples: W. H. Gallaher and J. T. Neu.

1.0 DESCRIPTION

The bidirectional spectroreflectometer provides a means of irradiating an opaque sample with monochromatic light, incident at angles of 0 to 90° and permits measurement and recordation of collimated light, reflected at various angles through a hemispherical volume above the sample. In the case of translucent samples, this device permits measurement and recordation of collimated light reflected or transmitted through a spherical volume surrounding the sample. The wave length of the incident flux may be varied from 0.2 to 25 microns.

Three irradiance sources are utilized: a Sylvania 1000 watt, 3400° sun-gun lamp, a General Electric A-H4 mercury arc and a Perkin-Elmer glow bar. An optical schematic of the source system is presented in Fig. 1. A source selection mirror permits the selection of a particular source to the exclusion of the other two sources. The monochromator is the standard Perkin-Elmer Model 98 monochromator provided with two prisms, one a potassium bromide prism and the other a calcium fluoride prism. Reference should be had to the Perkin-Elmer Manual for a detailed description of the monochromator. The monochromator is used in this application in its standard configuration except that a slit height limiter, which may be removed, is placed in the baffle before the entrance slit and a light tube is provided from the exit slit to the housing of the instrument.

By means of the monochromator-to-sample optical system, (Fig. 2) the divergent beam from the spectrometer is reflected by a diagonal plain mirror to an 11° off-axis-parabola which causes the beam to converge and (after being reflected from a plain diagonal mirror) to focus at the aperture disc position, AP-1. Beyond this focal point the beam again diverges and after being reflected from two diagonal mirrors, impinges an off-axis-parabola where it is rendered parallel and directed toward the sample.

The sample-to-detector optical system is similar to the source spatial system. The sample is viewed by an off-axis-parabola which receives parallel light and reflects it to a plain mirror where it is focused on an aperture disc in front of the detector assembly compartment.

Three detectors in three interchangeable detector-assemblies are provided to permit simple and rapid changes of detectors. The detectors are 1) a 1-P28 RCA photomultiplier tube, 2) a lead sulfide detector, and 3) a thermocouple detector. These detector assemblies are mounted at the plane of the detector aperture, AP-2. In the case of the thermocouple and the lead sulfide detectors, an ellipsoidal mirror reflects the light onto the detector while in the case of the phototube, the light strikes a quartz lens which focuses the beam on the face of the phototube. Support equipments such as the amplifier, power supplies, etc., are situated in a relay rack. Reference should be made to the manufacturer's manuals for detailed descriptions of these equipments.

The mechanical design is shown in detail in the instrument blueprints and will not be discussed in this manual.

2.0 UNPACKING

The spectroreflectometer was carefully packed and crated in San Diego to ensure that it arrives in Huntsville undamaged. Care should be exercised in the unpacking and assembly operation and an inspection for obvious damage, broken parts, etc., should be made. With regard to adjustments, it is suggested that the sequential procedure described in Section 4.0, Alignment Procedure, be followed so that unnecessary adjustments are obviated. Certain modifications and adjustments if made, could lead to considerable extra difficulty in the calibration of the instrument e.g., the potassium bromide and the calcium fluoride prisms have been calibrated for this particular instrument as presently aligned and any readjustment of the monochromator alignment which effects its focus, etc., could change this calibration and require a complete recalibration. If any uncertainty arises as to the necessity for making adjustments, it would be advisable to phone Dr. J. T. Neu of the Convair Space Science Laboratory.

2.1 Basic Instrument. This item is in the largest box bolted to the base. Remove the sides of the box, and then remove packing box from under table, wooden props, etc. Remove the basic instrument from base. The bolts holding the instrument to the base are the instrument's leveling bolts and should be replaced in the four table legs after the base is removed.

2.2 The major portion of the spectrometer was shipped in an assembled condition. However, certain items were separately packaged to prevent damage in transit. These separately packaged items are outlined below:

2.2.1 Prisms. These items were shipped in the manufacturers' shipping containers. They should be inspected through the glass cover for any obvious damage and set aside. It is unnecessary to remove the glass cover at this stage.

2.2.2 Detector Assemblies. There is a separate assembly for each detector (thermocouple, lead sulfide and phototube). Each of the assemblies were packed separately. In addition, the lead sulfide and thermocouple detectors were removed from their respective assemblies and packed separately. Check the detector assemblies for any obvious damage and set them aside.

2.2.3 Detectors. The instrument is provided with two thermocouple detectors; one having a target diameter of .5 mm and the other having a .75 diameter target. These are packed separately in the manufacturer's container. They should be inspected to assure that both units are present but they should not be opened at this stage. The thermocouple detector and the lead sulfide detector (to some degree) are both susceptible to mechanical shock. Exercise extreme care to avoid "bumping" the thermocouple detector unit against any object.

2.2.4 Shutter and Stray Light Filter. A shutter and a stray light filter complete with a holder, which clips in front of the monochromator housing, were packed separately. The stray light filter is a silver cloride plate with a smoke film on the front face. This smoke film is susceptible to the accumulation of finger prints and should be handled with extreme care.

2.2.5 Apertures. A set of interchangeable apertures has been provided and will be found in a small container which may be inspected and set aside at this point.

2.2.6 Sample Holders. Two sample holders, one for opaque samples and one for translucent samples have been included. Inspect and set aside.

2.2.7 Slit Height Limitation Clips. Two are included. Inspect and set aside.

2.2.8 Glan-Thompson Prisms. Two prisms 22 x 22 x 64 mm are included. Inspect and set aside.

2.2.9 Rotron Fan. Inspect and set aside. Be certain that the motor radiation shield is in place.

2.2.10 Thermocouple Premp. Inspect and set aside.

2.2.11 Off-Axis-Parabolic Mirrors. Two 23° off-axis-parabola mirrors (round, about 1 3/4"D) and one 11° off-axis-parabola mirror (square) have been removed from the system. They are wrapped in paper with the mounting stem protruding.

2.3 Checkout Gear. The following items comprising alignment and checkout gear have been included:

2.3.1 Pointed rod (3/4" diameter)

2.3.2 3/16" drill rod

2.3.3 Alignment cards

2.3.4 Alignment handle (attaches to parabola mounting stems)

2.4 Relay Rack. The relay rack containing the electronics equipment, etc., has been shipped as a unit in the smaller of the boxes. Cables were left connected at the relay rack terminus. Unpack and inspect for damage.

3.0 ASSEMBLY

Begin assembly by installing the detector transfer-optical system and attaching the photo tube detector unit to it. Install the off-axis-parabola mirrors by inserting their stems into the ball joint holders. The stems are threaded to attach to an allen tool with a handle which has been provided to facilitate installation (and the alignment to be accomplished at a later stage).

Next, unwind the cords attached to the relay rack. Connection of these lines to the instrument may be accomplished merely by finding plugs which fit, since the instrument was designed so that a misapplication of a plug to an unproper socket is impossible with one exception; i.e., it is possible to connect the photo tube and the thermocouple directly to the amplifier rather than to the detector switch (1120028) where they belong. If any difficulty is experienced, refer to the schematic of Figure 3. The relay rack and the instrument should be situated in such a position that the source unit is capable of performing its required rotation without causing the cables to bind. Attach water lines to the glow bar source. It is necessary to supply water to the glow bar only when it is in operation. However, it has been noted that the source end of the instrument becomes rather warm when the 1000 watt sun gun lamp is in use and running water through the glow bar housing might also be advisable when this source is in operation. The sun gun lamp assembly will require insertion of the bulb and positioning of the rotron fan. The sun gun lamp is held by two ceramic holders in the light housing. The bulb can be positioned by the exercise of a little care without removing the housing, etc. Care should be taken not to break the two ceramic holders, since they are rather fragile. After the bulb is set into position, place the sleeve on the housing and finally, position the rotron fan on the housing so as to blow a current of air through the housing when the bulb is in operation. The instrument switches are wired so that the light cannot be turned on unless the fan is also on. Experience has indicated that the sun gun source housing has a half life of from 3 to 5 minutes if there is no air stream.

In a similar manner, assemble the A-H4 mercury arc source. The mercury arc glass envelope should be removed by covering the lamp with a rag and tapping the glass lightly with a hammer. Thereafter, carefully screw the mercury arc into its socket and slip the socket into position in the housing. The glow bar has been assembled in the glow bar housing prior to shipment and no adjustment is required. Install the thermocouple preamp in the clips provided on the upper right hand side of the relay rack. The housing cover of the Model 98 monochromator should be removed and the calcium fluoride prisms installed (refer to the Perkin Elmer manual regarding this operation). The opaque sample holder should now be placed in position in the center of the detection unit. The instrument is now assembled and ready for alignment and checkout. Make certain that all of the switches in front of the instrument are in the off position and plug the instrument into a 120 volt 20 amp circuit. A distinct hum will be noted. This is from the constant voltage transformer and need to cause no concern. The instrument should be left connected to an electrical outlet at all times since there are heaters on the monochromator base which require current. These heaters are activated when the instrument is plugged-in regardless of the position of the main power switch.

4.0 ALIGNMENT PROCEDURE

The alignment of the bidirectional spectroreflectometer requires some degree of skill and dexterity, but it is not beyond the capability of laboratory personnel. It might be compared in difficulty to the aligning and focusing of the Perkin-Elmer Monochromator. For a given project, if the incident and reflected angular accuracies are critical then extreme care must be exercised in properly setting the zero angles. If a high degree of beam collimation is required, then small apertures and careful focusing are required to fully utilize the built-in quality of the highly parallel beam and high angular accuracy of the instrument. However, for most samples of interest for bidirectional study, the ability of the instrument to provide angular accuracy and collimation will far exceed requirements. It is suggested that attention be paid to the angular and the collimation accuracies required so that an undue amount of effort and time is not spent in obtaining more accuracy than is useful or meaningful.

The instrument has been carefully aligned at GD/Convair in San Diego prior to shipment and a representative will check out the alignment after the instrument is received in Huntsville.

To align the instrument, first remove the detector polar angle (θ') lock mechanism by loosening the Allen set screw and turning the hub counter clock-wise until it comes off. (The Allen set screw provides friction to keep the θ' adjustment from "self-locking.") Turn the detector unit in the azimuthal direction until the holes in the detector system lineup with the center of rotation of the axis of the source system. Pass the length of 3/16" drill rod provided through all three holes. Rotate the source system to 70 or 80 degrees and place a level on the short section of rod between the detector system and the source system and adjust the leveling screws on the base of the instrument until the short section of rod is level.

Now, check the section of rod between the pivot points on the detection system. If it is not level, it may be made level by properly adjusting the nuts which fasten the detector system to the base. This adjustment should not be required unless unexpected damage has occurred during shipment. Remove the rod and replace the clamping mechanism. Rotate the source system to $\theta = 0^{\circ}$ and turn on the mercury arc source. Occasionally, the arc does not light the first time the switch is turned on and it becomes necessary to flip the switch several times until the arc fires. When the arc is hot (e.g., immediately after it is turned off) it will not fire until it cools but a spark coil held near the arc will cause it to fire. Do not place the spark coil near the thermocouple or PbS detectors.

Next, adjust the source selection mirror until the image of the mercury arc is centered on the entrance slit. With the monochromator cover removed and with the calcium fluoride prism placed in the instrument, and a 0.5 mm slit, rotate the wave length drum until the green line is seen to pass out of the exit slit. This should correspond to a reading of 1100 on the drum but if it does not, use whatever drum setting is necessary but do not reset the Littrow mirror at this time.

At this point, it would be advisable to quickly check the alignment of the spectrometer. Since the alignment of the spectrometer was not affected by its shipment from Newark, Conn. to Convair in San Diego, it is not anticipated that any misalignment will occur between San Diego and Huntsville.

Before any focus adjustments of the monochromator are made, it should be determined that such adjustments are absolutely necessary, since the wavelength calibration curves provided will have to be redone if focusing adjustments are made on the monochromator. Procedures for checking the alignment are given in the P&E Manual. The pattern of the green line on a white card placed just beyond the exit slit is observed when narrow slits are used. It will be necessary to temporarily remove the "light tube" between the exit slit and the housing when this check is made.

After the focus is checked, replace the light tube and open the slits to about 1.5 microns. Install the slit height limitation clip with the large hole in the opening in the slit baffle in front of the monochromator entrance slit. Replace the monochromator cover and <u>replace screws</u> (always replace screws!) Then remove the cover from the spectrometer-to-sample optical system. A series of white cards, marked A, B, C, etc., is supplied with the instrument. Place Card A in the circular opening just to the right of mirror 1 as indicated in Figure 2. Card A has a small hole in it and is transparent enough to allow the rectangular pattern of the green line to be viewed. This rectangular green line pattern should be centered on the circular hole in Card A. If it is not, this indicates that the spectrometer is shifted on the rotating carriage. It is highly unlikely that this will occur and before an adjustment is made, it should be checked to be sure that there actually has been such a shift. If there has been a shift, the spectrometer can be replaced in the proper position by loosening the three Allen head screws under the spectrometer and shifting it until the rectangular pattern is centered in the circular hole. Now, place Card B lightly over mirror 1 and observe the dot of light that strikes the card. The dot should be located at the point where the two pencil lines on the card intersect.

Remove Card B from mirror 1 and place Card C in front of mirror 2 with Card A still in place. Card C may be inserted just in front of the mirror and the friction of the housing will hold it in place. The dot or the pencil of light which now strikes m_1 and is reflected to m_2 should again be centered at the intersection of the pencil marks. If it is not centered, loosen the Allen head screw that clamps the m_1 ball joint (not the shaft) and adjust it until the dot is centered. It should not be necessary to loosen the Allen head screw in the ball joint. Remove Card A from the circular aperture.

Place Card E (a round card about the size of a dime) in the aperture position and by manipulation of m_2 (using the tool that screws into the stem, and making certain that the handle is firmly attached to the tool so that m_2 cannot "fall out" of the ball joint), approximately focus and approximately center the "green dot" on Card E after removing Card C. The three screws that clamp the ball joint should be just loose enough to allow movement. Focusing is accomplished by translation through the ball while positioning is accomplished by "rotation of the ball." Insert Card C′, place Card D over mirror 3 and observe the point at which the light strikes the card. The light should be incident upon the pencil mark intersection. If required, adjustment of mirror 2 is made by adjusting the three screws that bear against the stem. After the dot is centered on the pencil mark intersection, Card D is removed. The pencil of light reflected from mirror 3 should now be centered on Card E by loosening the Allen head screw clamping the ball joint of mirror 3 and moving the mirror on its ball joint until it is centered on the perforation on Card E. It will be found that this is a rather difficult adjustment to make which requires considerable care and patience.

The image of the slit (reduced by the slit height limiter) will now appear on Card E. Card E′ (similar to E but without a hole in it) is placed in the aperture disc holder. Use a narrow slit and remove Card′ to observe the image. The slit should come to focus on this card and the quality of the focus can be judged from the sharpness of the line at this point. If focusing is required, it may be accomplished by loosening the single allen head screw <u>in</u> the ball joint which holds the shaft of mirror 2 and sliding the mirror backward and forward as required. (Use screw in tool, etc.) If this adjustment is made, it may cause the image to shift slightly off of the center hole and an iterative process involving mirror 2 and 3 may be required. Obtaining a sharp focus and precise centering at this stage is not critical unless it is desired to use one of the small apertures to obtain a very high degree of light collimation. It is anticipated that for most work, energy will be a much greater problem than collimation and that no aperture will be used at this stage.

When the pencil of light is centered and focused on Card E′, replace Card E′ with one of the small aperture discs in the aperture disc holder. Place Card C′ back in position. Now, open the slits to 1.5 mm and place Card F in front of m_4 and center the dot of light on the pencil mark intersection. Then remove Card F and insert Card G in front of mirror 5 and center the dot on the pencil marks again. At this stage leave Card G in place, rotate the spectrometer a few degrees off zero and insert the large pointed alignment rod in the sample holder hole. A card is provided in the alignment rod which makes an angle of 23° with the center line of the rod. Attach the adjustment tool to m_6 and translate it so that it is "as high up" as possible and carefully rotate the angle θ until the spectrometer is again horizontal ($\theta = 0^o$), being certain that the point of the alignment rod is below the surface of m_6. If it appears that the rod point is going to hit m_6 lower the rod in the sample holder. Adjust the rod so that the card's lower right hand extremity is coincident with the dot on m_5. Place Card H over m_6 and hold it in place with small strips of scotch tape or masking tape. Lower m_6 until it is just above the pointer. The projection of the line of the card from the dot of light on Card G to mirror 6 should strike the center of the crossed pencil marks. If it does not, this will indicate that it is necessary to adjust the spectrometer source unit in relationship to the sample holder-detector unit. This latter adjustment will be properly made at Convair before shipping and it is not expected that any displacement will occur. If this should occur, a translation of the detector and sample holder system, toward or away from the spectrometer source unit may be made by means of the nuts which hold the detector unit to the spectrometer table. Care must be exercised in making this adjustment if it is required. The level and alignment of the rod which fits through the axis around which the detector unit rotates and anchors in the source axis must be maintained. After the adjustment is made, remove the large alignment rod and Card G from mirror 5 and observe the position of the dot image on Card H. It should be centered. If it is not, adjustments of mirror 5 should be made to center it. Now replace the large pointed alignment rod in the same holder and center the point of the rod on the dot on Card H by rotating the source system. The zero degree position setting (on the angular dial in front) of the source system should be checked and, if necessary, set so that θ is zero. This adjustment is made by loosening the screws holding the zero line pointer and translating the pointer.

Remove the pointed rod. Remove Card H from mirror 6 and allow the pencil of light now centered on mirror 6 to be deflected toward the sample holder. Place the sample holder in position with a small round card with a center dot at the center of the holder. By loosening the three screws clamping the ball on m_6, adjust the dot until it is centered on the card. Clamp the screws holding the ball in position. Remove Card C′ and the aperture disc. Check the collimation of the beam striking the sample by using a very narrow slit and checking the width of the image at various distances from the mirror. Mirror 6 is a 23° off-axis-parabola mirror and will serve to render the beam parallel providing that the source is a point. The correct focus of the beam can be judged, however, by using very narrow slits and ascertaining the divergence or convergence of the beam as a card is moved away from m_6. Inasmuch as finite source sizes are used, the beam will not be exactly parallel. Actually, the beam should diverge one slit width for each focal length of the off-axis-parabola. (The focal length of the off-axis-parabola is 203 millimeters.) So, for a very narrow slit almost negligible divergence of the beam should occur in the width dimension of the slit. With respect to the length dimension of the slit, considerable convergence and divergence will occur unless a very narrow preslit mask is in place. The preslit masks provided are rather wide and some divergence will still occur in the slit length direction. If desired, aperture AP-1 may be fully illuminated (wide slit and no preslit mask) and a small size aperture disc used to provide a beam of low and uniform divergence. Adjustment may be made by translating m_6 (i.e., by loosening the Allen head screw in the ball and adjusting the shaft up or down).

The source system is now aligned. Rotate the angle θ (source system) to exactly 90° and swing the detector unit into position so that the full beam from the source is incident on the detection system. Remove the flat cover plate from the detector optical system and place Card I in the round entrance aperture of the detector unit and place Card C′ in the source system but with no aperture at AP-1. Adjust the θ' and φ' "zero angle" settings so that they are 0° when the dot is incident upon the crossed pencil marks of Card I. These adjustments are made by loosening the screws holding the zero line pointer and shifting to the "zero" on the angular dial. With the dot centered on the crossed pencil marks, remove Card I and the beam will fall on m_7 and be reflected from m_7 (mirror 7 is an off-axis-parabola, of the same specifications as m_6) onto m_8. Place Card J on m_3 and check the position of the pencil of light at m_8. It should fall on the intersection of the pencil marks. If it does not, loosen the ball of m_7 and adjust it to fall on the cross. Remove Card J from m_8 and allow the pencil of light to strike Card E located in the aperture disc position AP-2. The detector assemblies should not be installed at this point. Mirror 8 is adjusted so as to cause the pencil of light to be incident upon the center of Card E. If the focus of this pencil of light on Card E is not sharp, then mirror 7 may be translated backward or forward in order to make the focus sharp and clear. The source aperture and Card C′ should be removed for this focusing adjustment. If refocusing is necessary, reimaging of the light on the center of Card E will probably be necessary. In the detector optical adjustment, the θ', ϕ' zero settings must be maintained.

Card E is now removed and an aperture disc is inserted in the aperture disc hole on the detector unit. If the phototube detector assembly is used further alignment is not required. The position of the quartz lens has been properly set at Convair and no further adjustment of the optics of the phototube should be required. The procedure for the alignment of the lead sulfide detector and the thermocouple detector is as follows: With no aperture in the source optical system and a fairly wide slit, cover a small aperture disc placed in the detector unit with a bright beam of light. Light passing through the aperture will fall on the ellipsoidal mirror. The operator should examine the thermocouple or the lead sulfide detector assembly and become familiar with the appearance of the target of the detector. In both cases, the target is a small surface area inside the unit. After the operator is familiar with the appearance of the target area, place the detector in position, fasten the three screws in the base and remove the inspection port on the detector barrel. By moving the ellipsoidal mirror back and forth in its mount and laterally in the ball joint it is possible to focus the light onto the detector area. This is a rather difficult task to perform but with a little practice it can be accomplished. It is probably best accomplished by first centering the beam on the detector face without regard to focus and clamping the three screws which lock the ball joint. Then, by slowly moving the ellipsoidal mirror back and forth in the translation mount, bring the unit to focus on the face of the detector unit. It is advisable during this translational adjustment to activate the detector unit, (i.e., turn on the recorder, etc) and using a suitable wave length of light, to maximize the amount of energy, striking the detector. Refer to the thermocouple alignment section in the P&E Manual. Small lateral adjustment on the target face may be made by changing the tension on the three screws which lock the ball joint. If the lateral adjustment is slightly in error, the angular alignment of the detector unit (θ', ϕ') will be similarly in error. When it becomes essential in using the lead sulfide or the thermocouple detectors to have the highest angular accuracy, it might be advisable to first make the translations so that the light is focused in the plane of the detector face and then to set the angular positions of the detector unit by viewing a specular sample. For example, setting θ at 45° and adjusting the detector unit until the light sensed is maximized in setting θ' at exactly 45° and in a similar fashion maximizing the light and setting the proper φ'. As mentioned earlier, adjustments of this kind will not be required unless extreme accuracy is required in the angular positioning of the device.

5.0 OPERATION

5.1 Monochromator. Persons desiring to use the instrument should first completely familiarize themselves with the operation of the Perkin-Elmer spectrophotometer. Knowledge of the operating methods of the spectrophotometer will alleviate difficulties in the operation of the unidirectional spectroreflectometer. The Perkin-Elmer Manual provided with the instrument describes the method of operation of the monochromator.

5.2 Selection of Components. In adjusting the instrument for the determination of the unidirectional reflectance of a sample it will first be necessary to consider the wave length coverage desired and to select suitable instrument components to cover this wave length region. A variety of considerations will go into the selection of these components inasmuch as there are overlaps in certain spectral regions. Selections must be made of source, filter, prism, polarization of the source beam, polarization of the detector beam, and detector.

5.2.1 Sources

5.2.1.1 Photo tube. The RCA 1P28 photo tube provided is capable of recording flux signals from 0.2 microns to between 0.7 and 0.75 microns. The photo tube provides high sensitivity and (compared to the other detectors) low noise. Since the design stage of this project, the author has had occasion to become familiar with photo tube technology in connection with another project. It has been learned that there are photo tubes which may be used much farther into the near infrared and which have very high sensitivity in this region. Had the writer been aware of the existence of these photo tubes at the time the proposal was written an additional interchangeable photo tube detector assembly would have been suggested to permit use of the photo tube beyond 1 micron. It is, of course, entirely feasible for the customer to make a photo tube holder similar to the current assemblies and to obtain one of these high sensitivity photo tubes. The tubes which have useful operating characteristics in the near infrared regions are usually of the "end on" type. The ones having very high sensitivity often require a considerably higher plate voltage than can be supplied by the Perkin-Elmer power supply and an alternate power supply would have to be obtained for them. The RCA 7102 tube is compatible with the current Perkin-Elmer power supply and its range of sensitivity extends into the near infrared. However, it does not have the high sensitivity which is currently available in some of the other tubes. Some experimentation in our own laboratories with higher sensitivity tubes is planned and we will forward any information we obtain on this subject in the future.

5.2.1.2 Lead Sulfide Detector. The lead sulfide detector is used from the cutoff point of the photo tube out to between 2 and 3 microns. This detector provides good sensitivity but is usually noisier than the photo tube.

5.2.1.3 The Thermocouple Detector. The region for the use of the thermocouple detector is from the cutoff point of the lead sulfide detector out to 25 microns.

5.2.2 Filters. A single monochromator (as distinct from a double monochromator) is almost always plagued with the problem of "false energy" or as it is sometimes called "stray light." Excellent articles presenting analyses of this problem have been published and it will not be dealt with in any detail here. The wave length regions in which it can occur depend upon (among other things) the nature of the source and the nature of the detector. To use the infrared region as an example, the glow bar source used in this region has its peak energy in the neighborhood of 2 microns. At some longer wave length, say 20 microns, the source has a much smaller amount of energy available than it had at 2 microns. With the spectrometer set at 20 microns a small fraction of the peak energy which is chopped at 13 CPS gets through the monochromator and strikes the detector. The instrument records the sum of the true and the false signal. In the infrared this problem is handled by a lead sulfide silver chloride filter with a smoked face. This filter may be inserted in front of the monochromator slit to eliminate stray light and should be used from about 15 microns out to 25 microns. Stray light problems may occur in certain other regions of the spectrum and the operator should be constantly aware of this possibility and should check for their existence. It may be desirable to procure blocking filters for certain other regions of the spectrum depending on the interest in spectral purity, etc.

5.2.3 Prisms. Two prisms, a calcium fluoride and a potassium bromide are provided. The calcium fluoride prisms have a useful range of from 0.2 microns out to about 6 microns. In order to obtain this full coverage one must reset the Littrow mirror depending upon which end of the spectrum one desires to cover. Two calibrations are provided, one with the mercury green line set at 1100 on the drum (to cover the UV-visible portion of the spectrum) and another calibration with the mercury green line set at 2300 (to cover the full infrared potential of the prism). The drum setting of the green line is made by using the mercury arc source, removing the cover of the monochromator and observing the light passing through the exit slit. The drum is set at the desired number for the green line and the Allen head screw which adjusts the Littrow mirror is turned until the green line passes the exit slit. At this point the cover is replaced and screwed into position and the small tap provided on the front of the housing is removed and the special Allen screw inserted through this hole and micro adjustments are made on the green line setting. Final setting should be made with very narrow slits and the green line should reach a peak right at the given drum setting. Always make the drum settings by rotating from larger to smaller drum numbers. The potassium bromide prism may be used from the cutoff point of the calcium fluoride prism out to 25 microns. A calibration chart with a green line setting for this prism is provided. It may be desirable to use the KBr prism from about 2 to 25 microns to obtain the greater band pass (more energy) resulting from the lower dispersion.

5.2.4 Polarization. By means of the Glan-Thomson prisms provided it is possible to polarize either or both the source and the detection light beams. The Glan-Thomson prism is potted into a brass sleeve and this sleeve is held in an additional sleeve in the instrument. It is possible to rotate the brass sleeve in which the prism is potted in the sleeve provided with the instrument. In our laboratory no attempt was made to position the polarization prism at any particular angle and the angle desired may be set by the customer. An angular scale is provided for this purpose on both the source and the detection optical system.

5.3 Instrument's Settings. The following list of settings must be made prior to making a measurement.

5.3.1 Select the source and turn the source selection mirror to illuminate the entrance slit with the light from this source. Set the spectrometer slit at the desired width. Set the spectrometer drum to select the proper wave length. Set the angle of the incident polarization if it is desired to illuminate the sample with polarized light. Set the angle of incidence of the light (i.e., set θ). Set the sample azimuthal angle (this setting will have meaning only if the sample has a regular pattern, grain, etc). Set the angle of the detection polarization system if it is desired to polarize the reflected light before it hits the detector. Set the view angle of the sample (i.e., set θ' and ϕ'). Set the zero for the instrument. The latter setting requires adjustment of the Perkin-Elmer amplifier and the digital recorder. For methods of setting the Perkin-Elmer amplifier reference should be made to the Perkin-Elmer Manual. The digital recorder requires no particular settings except selection of the range desired (ordinarily the range from 0 to 10 mV and 0 to 100 mV would be used).

APPENDIX 1

Parts List

Perkin Elmer Corp., 433 West Foothill Blvd., Monrovia, Calif.

1.	Model 98 monochromator	098-0011
2.	Model 107 amplifier	170-0001
3.	Front end less 012-0032	112-0004
4.	Photomultiplier power supply	112-0038
5.	Cables	013-0141
		081-0028
		081-0029
		012-0163
6.	Silver chloride-silver sulfide filter	021-0029
11.	Ellipsoid mirror (2)	021-0126
12.	Thermocouple preamp	088-0054
14.	Internal photomultiplier tube mount	012-0149
15.	PbS preamp and switch	112-0028
16.	Glow bar power supply	112-0033
17.	Mounted calcium fluoride prism	127-1657
18.	Mounted potassium bromide prism	127-1257
19.	Lead sulfide detector with .75 mm sq. target	special
20.	Off-axis parabola mirror 11 deg off-axis	012-0486
21.	Off-axis parabola mirror 23 deg off-axis	137-0169

General Electric

1)	Balast transformer	9T64Y4017G-2
2)	Mercury arc	H100A47

Karl Lamprecht, 3959 N. Lincoln Ave., Chicago 13, Ill.

1. Glan-Thompson Prisms (2) mounted in standard brass tubes.

Charles M. Reeder & Co., 173 Victor Ave., Detroit 3, Michigan

1. Thermocouples to mount in Perkin Elmer Model 112, Round targets, high sensitivity, cesium bromide windows #RDR-F

    Target 0.5 mm dia

    Target 0.75 mm dia

Raytheon, Richards Avenue, So. Norwalk, Connecticut

1. Constant voltage regulator

    Type RVA 10005

Hewlett Packard Co., Neely Sales Corp., San Diego, Calif.

1. Hewlett Packard digital voltmeter #3440a
2. Hewlett Packard high gain/auto range #3443a
3. Hewlett Packard digital recorder #562A
4. Hewlett Packard digital recorder Cable #562A-16c

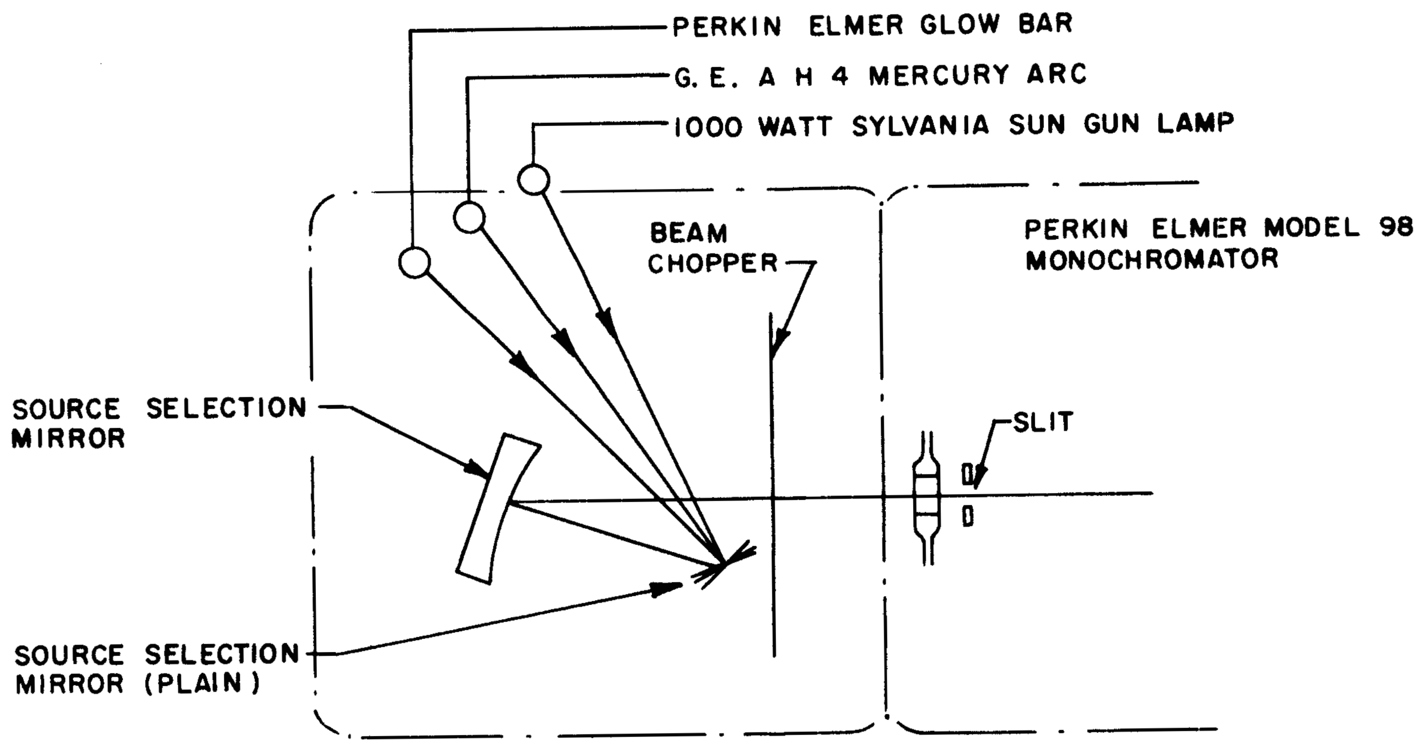
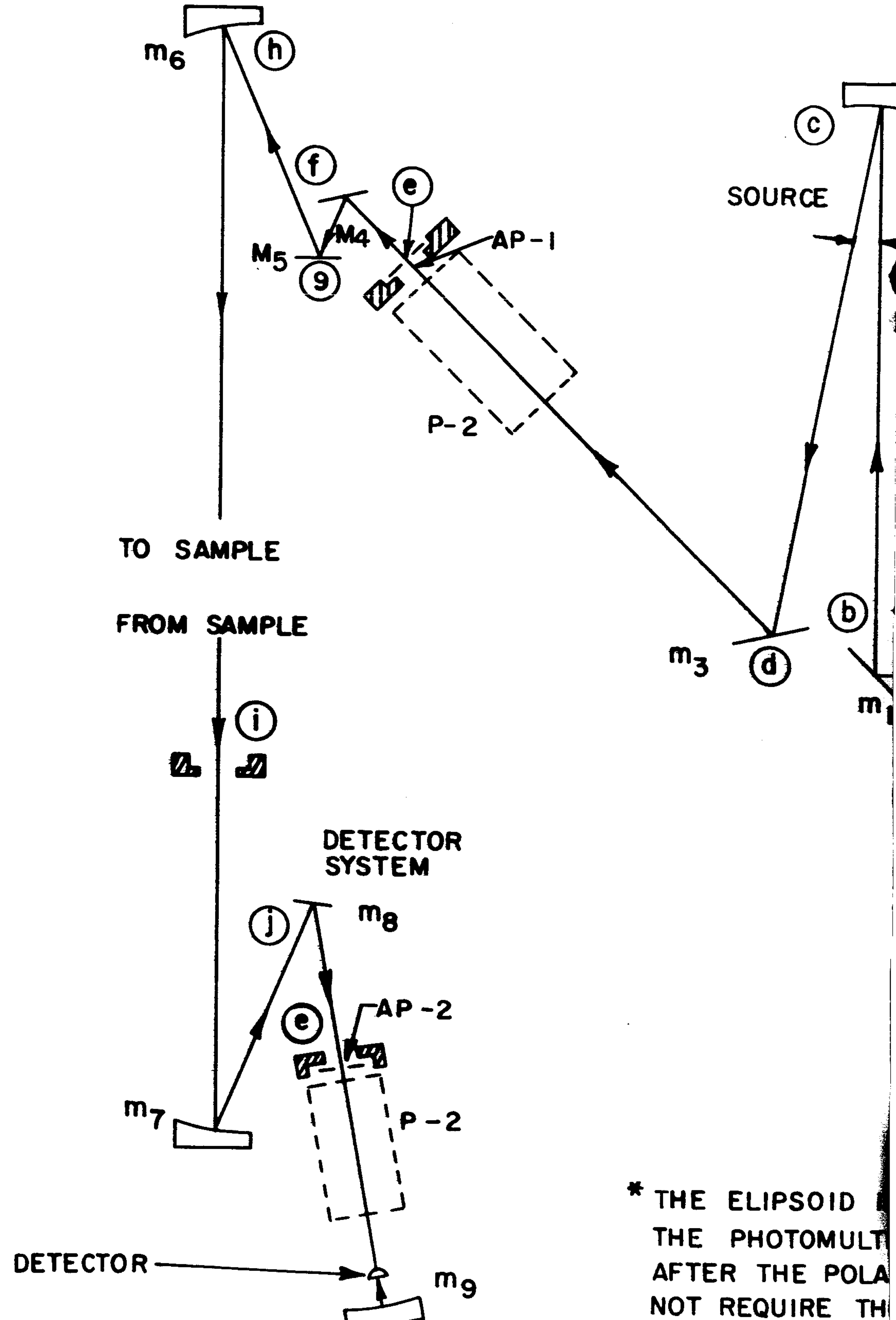
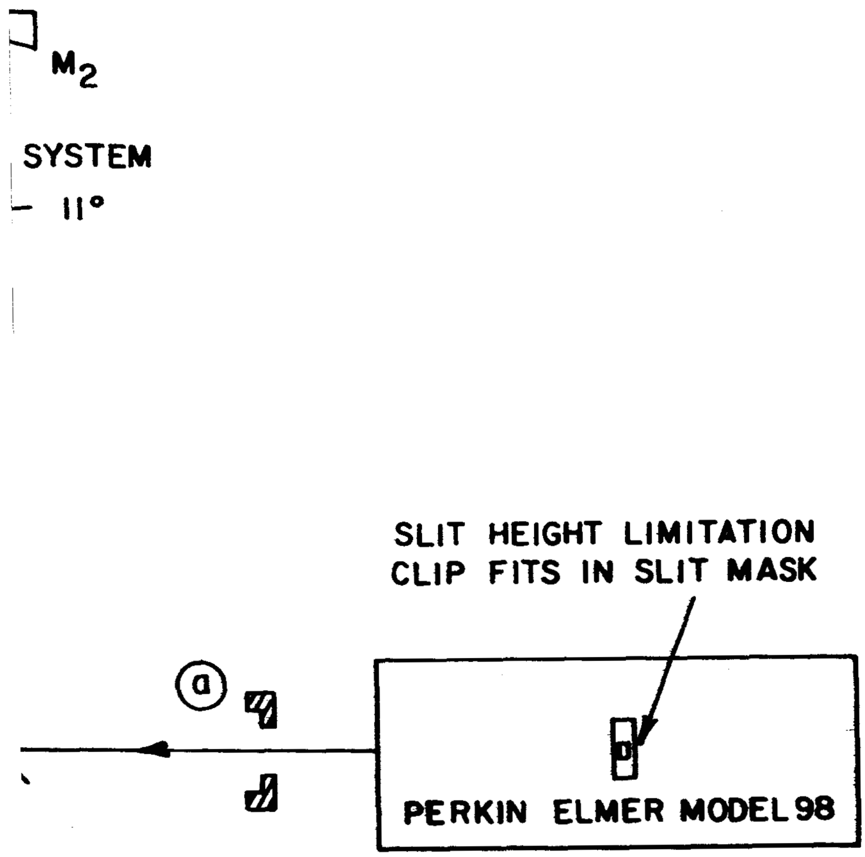


FIGURE 1 : OPTICAL SCHEMATIC - LIGHT SOURCES
CONVAIR MODEL NB-2 BI-DIRECTIONAL SPECTROREFLECTOMETER



* THE ELIPSOID
THE PHOTOMULT
AFTER THE POLA
NOT REQUIRE TH

FIG. 2 : OPTICAL SCHEMATIC –CONVAIR MODEL NB-2



(a)(b)(c) etc –ALIGNMENT CARD POSITIONS

m_2 –11° OFF AXIS PARABOLA f = 267 mm

m_6 , m_7 – 23° OFF AXIS PARABOLA f = 206.2 mm

*m_9 – ELIPSOID f_1 = 1.35" f_2 = 7.19"

m_1 , m_3 , m_4 , m_5 , m_8 –PLAIN MIRRORS

AP–1 –SOURCE APERTURE

AP–2 –DETECTOR APERTURE

P–1 –SOURCE POLARIZING PRISM

P–2 –DETECTOR POLARIZING PRISM

USED FOR THE THERMOCOUPLE AND LEAD SULFIDE DETECTORS
PLIER DETECTOR HAS A LENS (QUARTZ PERKIN –ELMER #027-1718)
RIZATION PRISM TO FOCUS THE BEAM ON THE PHOTOTUBE AND DOES
E ELIPSOID.

BI–DIRECTIONAL SPECTROREFLECTOMETER

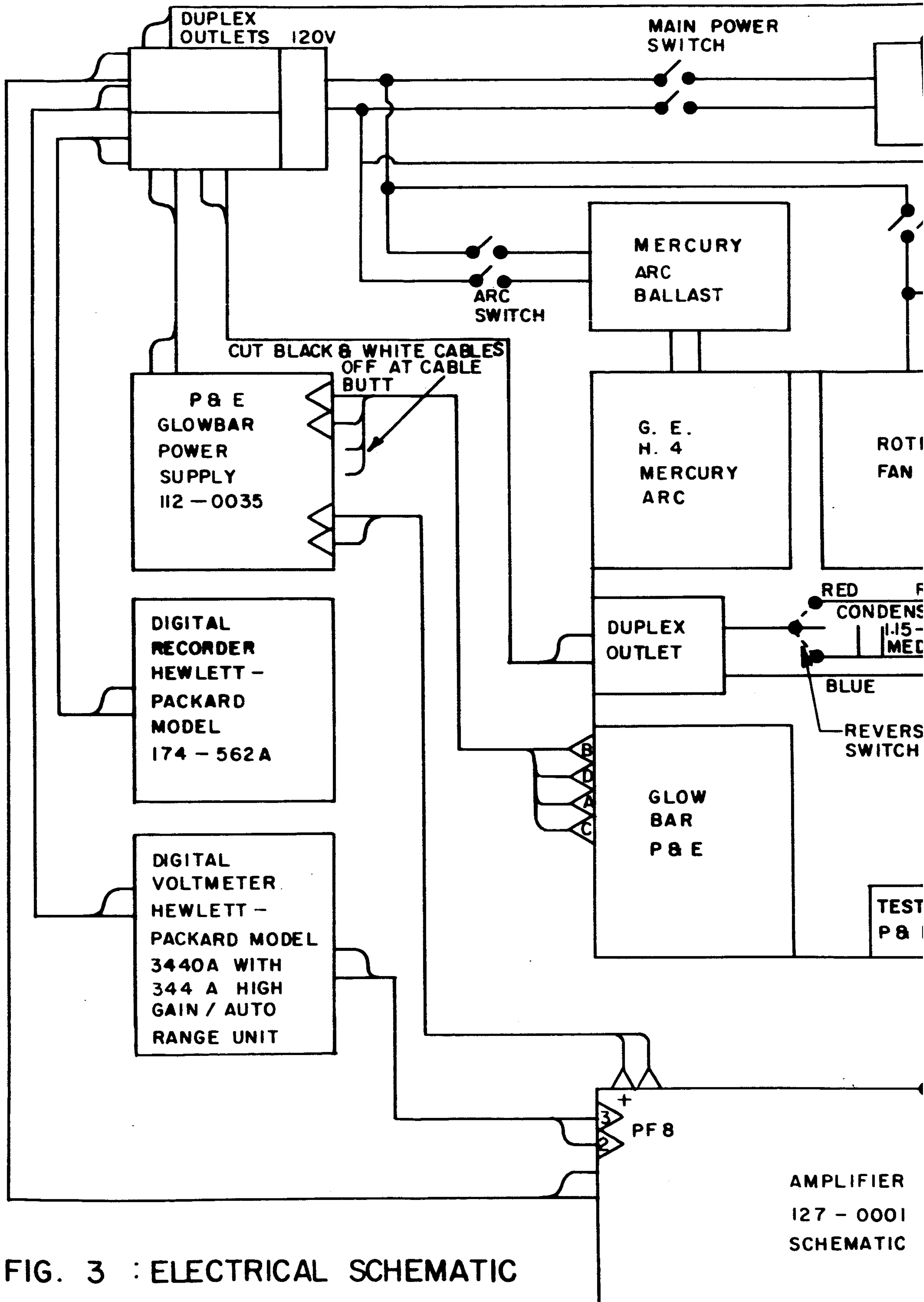
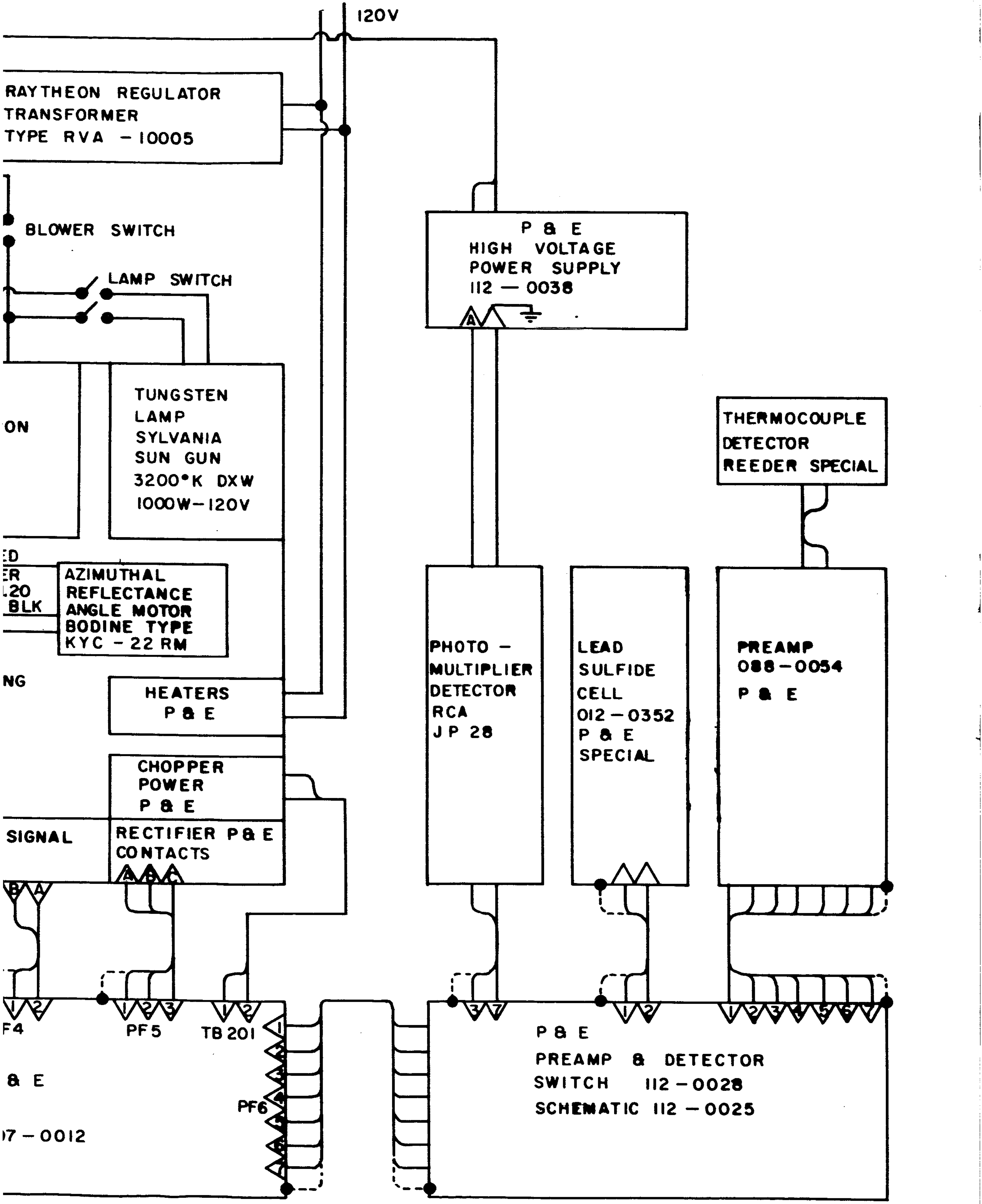


FIG. 3 : ELECTRICAL SCHEMATIC


120V
RAYTHEON REGULATOR TRANSFORMER TYPE RVA - 10005
BLOWER SWITCH
LAMP SWITCH
P & E HIGH VOLTAGE POWER SUPPLY 112 — 0038
A
ON
TUNGSTEN LAMP SYLVANIA SUN GUN 3200°K DXW 1000W-120V
THERMOCOUPLE DETECTOR REEDER SPECIAL
AZIMUTHAL REFLECTANCE ANGLE MOTOR BODINE TYPE KYC - 22 RM
BLK
NG
HEATERS P & E
PHOTO - MULTIPLIER DETECTOR RCA JP 28
LEAD SULFIDE CELL 012 - 0352 P & E SPECIAL
PREAMP 088 - 0054 P & E
CHOPPER POWER P & E
SIGNAL
RECTIFIER P & E CONTACTS
A B C
PF5
TB 201
PF6
P & E PREAMP & DETECTOR SWITCH 112 - 0028 SCHEMATIC 112 - 0025
& E
7 - 0012

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