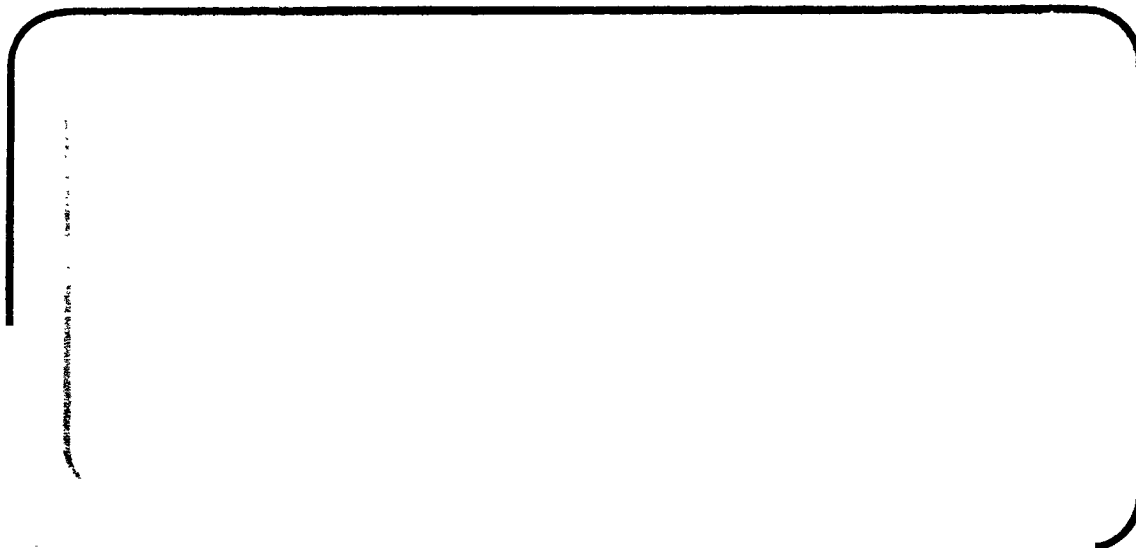


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FINAL REPORT
AN INVESTIGATION OF THE THERMAL RADIATION
PROPERTIES OF CERTAIN SPACECRAFT MATERIALS

by

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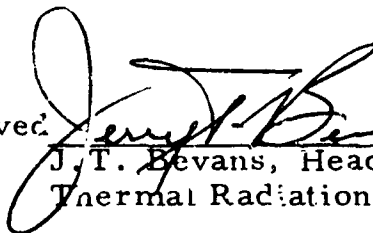
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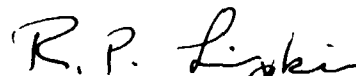
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I. INTRODUCTION

This is the final report for Contract NAS5-1102, "An Investigation of the Thermal Radiation Properties of Certain Spacecraft Materials." The study was originally conceived as an examination of thermal control materials with emphasis upon surfaces which have large or small values of the ratio of solar absorptance to ambient temperature (e. g., 80°F) emittance, i. e., α/ϵ ratio. The sulfuric anodizing process for aluminum was of special interest for the low α/ϵ surfaces and other electrochemical processes on other materials were to be studied for both large and small α/ϵ ratios. Approximately half-way through the original program period, the degradation effects of the ultraviolet and vacuum environment of space were found to be particularly severe for sulfuric anodized aluminum. After discussions with Mr. M. Schach of Goddard Space Flight Center, the contract period was extended to 31 December 1962, and emphasis was placed upon this degradation of sulfuric anodized aluminum.

As described above, the study of different anodizing processes was originally a significant portion of the program but emphasis later shifted to ultraviolet and vacuum degradation studies. Although the effects of the space environment can not be ignored in the selection of a thermal control material, circumstances have caused an arbitrary division between the material development and the degradation studies. This separation is retained in this report and these two aspects of the program will be presented in separate sections. The artificiality of this is recognized.

The first section of the report will briefly describe the instrumentation used for the reported thermal radiation property measurements. The results of the studies for developing controlled values of solar absorptance and emittance will then be presented. This is followed by a discussion of the ultraviolet and vacuum degradation program. The final section will give the results obtained from certain secondary studies, e. g., use of a CsI prism with the heated cavity reflectometer.

II. INSTRUMENTATION USED FOR THERMAL RADIATION PROPERTY MEASUREMENTS

An examination of the data reported in the literature for apparently identical materials will often reveal large differences. In the past, such variances were primarily a result of nonidentical samples and now proper sample description has become a paramount requirement in data reporting. With the development of the awareness for adequate sample description, the differences between various types of instrumentation and alternative experimental techniques have also become apparent. For this reason, the following description of the experimental apparatus is included. This discussion will also provide a means for commenting upon the advantages and disadvantages of this equipment.

The spectral reflectances of the sample materials between 2.0 and 25 microns were measured with a heated cavity reflectometer. This instrument is identical to that described by Dunkle, et al.* and eliminates the errors inherent in an earlier design by Gier and Dunkle.** Unless specified, all of the spectral reflectance data taken with this instrument and reported herein was obtained at an angle of 15° from the normal to the sample surface. The sample temperature was between 70 and 100°F but was not monitored for every sample.

The spectral reflectance in the wavelength region 0.3 to 2.5 microns was obtained with either an integrating sphere reflectometer of the type described by Edwards, et al.*** or a Beckman DK-2A modified reflectometer. The two instruments are identical in principle but the Edwards sphere utilizes a Perkin-Elmer Model 98 (or 99) monochromator with greater control of the spectral resolution. The modified reflectometer for the Beckman instrument was installed when the data obtained with the

* R. V. Dunkle, and others. "Heated Cavity Reflectometer for Angular Reflectance Measurements," Progress in International Research on Thermodynamic and Transport Properties, Am. Soc. Mech. Engrs., (1962) p. 541

** J. T. Gier, R. V. Dunkle, and J. T. Bevans, J. Opt. Soc. Am. 44:558 (1954)

*** D. K. Edwards and others, J. Opt. Soc. Am.

original Beckman integrating sphere was found to be seriously in error. In the conventional Beckman DK-2A reflectometer, part of the energy reflected from the sample may fall directly on the detector or pass out of the integrating sphere through one of the two large entrance ports. The errors that can occur are illustrated for a striated aluminum sample in Figure II-1. In this figure, the reflectance differences are a consequence of changing the azimuthal angle of incidence, not the polar angle. The new sphere utilizes one entrance port and places the detector directly below the sample so as to avoid any direct reflected energy. In addition, the sample holder is placed in the middle of the sphere and the angle of energy incidence can be varied from near normal to 65° (limited by sample size). The reference beam is directly incident upon the integrating sphere wall and the measurements can be termed "absolute." This modification permits an absolute spectral reflectance measurement to be made in 15 to 20 minutes. The major deficiency is the lack of control over the spectral slit widths of the monochromator and hence, no control of the resolution. The major advantage is the increased measurement speed and for the great majority of samples, the accuracy is adequate for design property values. The new attachment is readily removed from the system and the conventional integrating sphere attached for transmission measurements. The modified Beckman system and the Edwards integrating sphere have been compared and for samples which do not exhibit reflection characteristics requiring high resolution, the two are within ± 1 percent. Figure II-2 shows the instrument installed on the Beckman spectrometer.

Although not used for the data reported here, two total near normal emittance instruments are now available. These have been described in the Progress Report dated 16 April 1962. In addition, a total hemispherical emittance system was assembled for the contract but a combination of time and experimental difficulties have not allowed sufficient data to be collected and to be reported with confidence. This system consists of a vacuum system, a bell jar, and a liquid nitrogen cooled enclosure within the bell jar (Figure II-3.) This is the conventional cryogenic emittance system but the design is based on a study of the errors involved

in such measurements.* The sample consists of a wire wound heater sandwiched between thin metal sheets (e.g., brass shim stock) four inches square. The sample is suspended within the enclosure by the heater wires and the necessary thermocouple(s). The power supplied to the sample is monitored by the usual four terminal resistor technique and the temperature is determined with 40 ga. Cu-Co thermocouple(s). The coating or material to be measured is applied to the outside of the sample. The primary difficulty encountered to date is the fabrication of the sample heaters and the establishment of uniform sample temperature. At the time of this writing, these problems are still under attack.

The spectral reflectance data which is reported is all near normal (15°) incidence unless otherwise specified. No attempt has been made to reduce the near normal emittance values calculated from spectral reflectance measurements to hemispherical emittance. If hemispherical data is desired, the conversion can be performed with either Figure 4-10 of "Heat Transfer," Vol. I, by M. Jakob or Figure 13-15, of "Heat and Mass Transfer," by E. R. G. Eckert and R. M. Drake.

*K. E. Nelson and J. T. Bevans, "Errors in the Calorimetric Method of Total Emittance Measurement," STL Proposal 0709.00/4567 dated 30 Nov. 1961 to SSD, USAF, presented at the Symposium on Measurement of Thermal Properties of Solids, Dayton, Ohio, Sept. 5-7, 1962

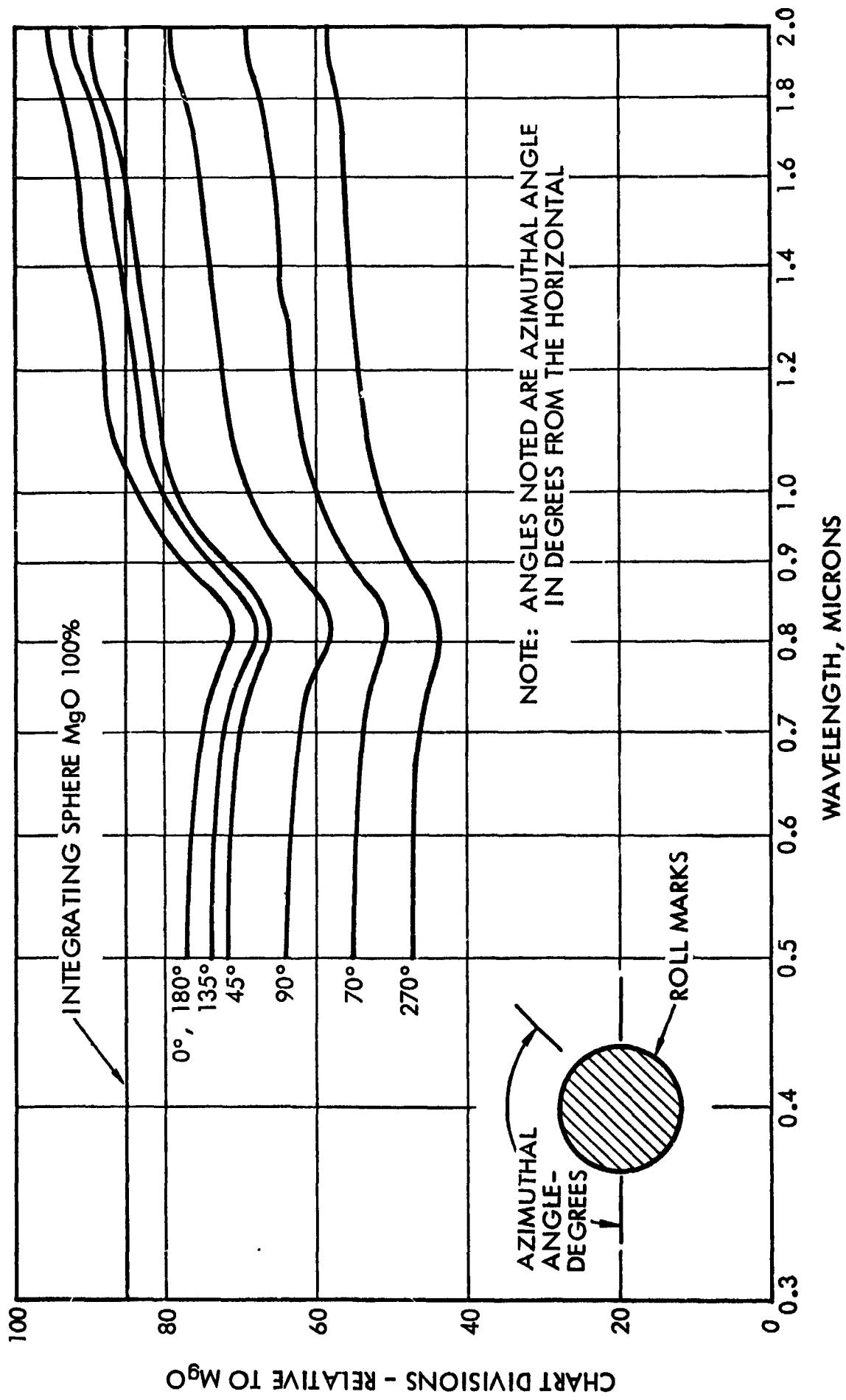


Figure II-1. Beckman DK-2A Integrating Sphere—Reflectance of Soft Anodized 1199 Aluminum Foil as a Function of Sample Position



Figure II-2. Integrating Sphere Attachment for the Beckman DK-2A Spectrometer

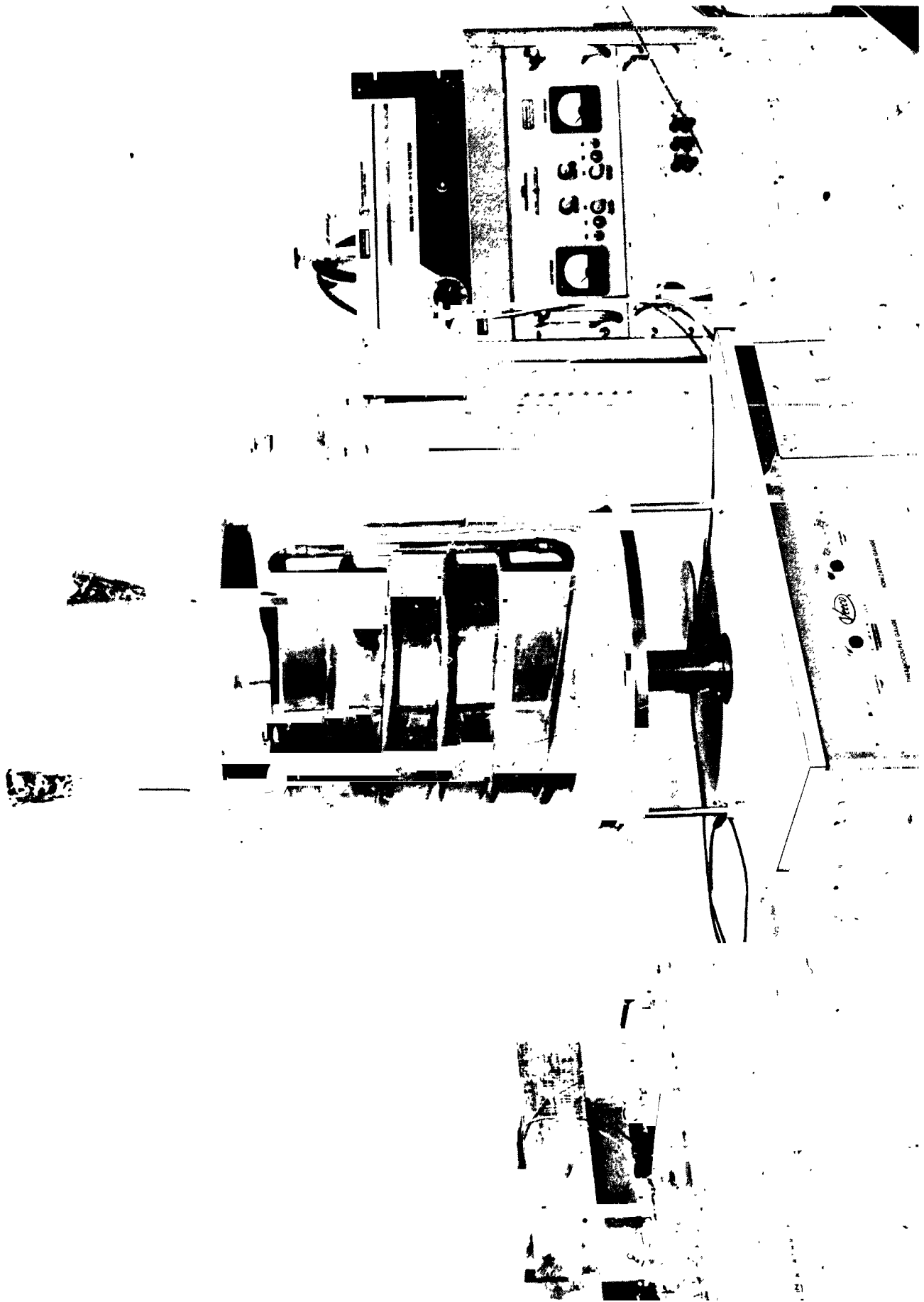


Figure II-3. Calorimetric Total Hemispherical Emittance

III. INVESTIGATION OF THERMAL RADIATION PROPERTIES

Aluminum is a primary material for the construction of spacecraft but in its natural condition, it is a poor material for thermal control; i. e., it has a large ratio of solar absorptance to emittance ($\alpha/\epsilon \gg 1$). Certain anodizing processes, however, can provide a coating for aluminum which has an α/ϵ ratio which is much less than unity. One phase of the program was the investigation of the anodizing processes to determine what values of solar absorptance and emittance could be obtained by various anodizing techniques. The sulfuric acid anodizing electrolyte is one of the most common processes used to obtain such a coating and was given primary attention. In the first portion of the following section, results are presented of a study to determine the thermal radiation property versus thickness characteristics of the sulfuric anodizing process for several alloys. The second part describes an examination of other aluminum anodizing processes in order to find possible alternative methods of securing properties comparable to the sulfuric acid method. The final portion describes several processes for metals other than aluminum. The ultraviolet and vacuum degradation characteristics of the various coatings will not be considered in this section. (See Section IV).

A. SULFURIC ACID ANODIZING PROCESS

The specific sulfuric acid processes examined are summarized in Table III-1. For increasing coating thicknesses, the measured ranges of the values of solar absorptance and normal emittance are shown in Table III-2 for the eight alloys used. As shown in the resumé, values of solar absorptance less than 0.25 are not difficult to obtain and can be secured with emittances greater than 0.8. For design purposes curves of property versus thickness are of primary importance and this information is shown in Figures III-1 through III-24.

The utility of the anodizing process for controlled thermal radiation properties is readily apparent from Figures III-1 through III-24. In general, the solar absorptance depends more strongly upon thickness than emittance. Hence, the approximate emittance can be selected by process and alloy and the absorptance can be obtained by selecting the thickness. This technique has certain limitations since not all processes and alloys

will give the desired range of absorptance. For example, the 7075 and 1199 alloys have quite large differences in solar absorptance but emittance ranges overlap.

The effect of process variables are also shown by these figures. The solar absorptance of type 6061 alloy with a "Martin Hardcote" anodize varies significantly with thickness (Figure III-18) whereas the 1199 soft anodize solar absorptance is relatively insensitive to thickness changes. The use of "sealed" anodized coatings does not appear to affect the properties significantly (Figures III-1, 2, 5, and 6). The electrolyte temperature is quite important in the soft anodizing process on type 1170 aluminum as shown by Figures III-7 and 8. These same figures also show the effect of changing the current density (amperes/square foot) which is one method for altering the rate of anodized coating formation.

The results which are presented indicate the value of the anodizing process for providing a thermal control material on aluminum. The use of these materials in a space environment depends upon the degradation that will occur. The section on ultraviolet and vacuum degradation discusses this problem.

B. NONSULFURIC ACID ANODIZING PROCESSES

The anodized layer on aluminum is not pure aluminum oxide but it consists of the oxide, aluminum salts of the electrolyte and hydrates of both the oxide and the salts. The compositional differences between electrolytes should result in differences in thermal radiation properties and this phase of the program was performed to determine these differences for as many electrolytes as feasible. The number of variables that can be considered in such a program is practically infinite. In order to examine as many processes as possible, only a limited study of each electrolyte was performed and the number of electrolytes restricted by the time available.

The electrolytes which were studied are summarized in Table III-3. The tests performed for each electrolyte along with the results obtained are given in Tables III-4 through III-11. Of the several processes examined, the oxalic and chromic acid electrolytes yielded solar absorptances in the range 0.3 to 0.6, emittances between 0.5 and 0.9 and values of α/ϵ

between 1/3 and 1/2. The zirconyl sulfate electrolyte appears to be competitive with the sulfuric acid electrolyte for small values of solar absorptance and large values of emittance. The oxalic-sulfuric acid electrolyte does not appear to give thermal radiation properties that are much different than the sulfuric acid process. The proof that not all anodizes are "the same" is well demonstrated by the results obtained for the citric acid electrolyte. This electrolyte is the barrier type and only a thin anodized coating is formed (coating insoluble in electrolyte). This could be a method for protecting aluminized surfaces in adverse environments prior to launch. The formation of an anodized layer on aluminum by boiling water is quite interesting and this material might be the best method for forming anodized layers for basic ultraviolet degradation mechanism studies.

None of these coatings have been subjected to ultraviolet and vacuum degradation tests. Such experimentation should be made a part of any future studies of these electrolytes. The compositional differences of the coatings obtained from the various electrolytes might accelerate or retard the degradation process and the results would be of basic interest. The spectral reflectance curves (Figures III-25 to 31) of examples of various anodizing processes are indicative of these differences.

C. ELECTROCHEMICAL COATINGS ON OTHER METALS

The emphasis in the development of thermal radiation materials has been upon aluminum as the substrate. However, other metals can be valuable in certain applications. One particular area is that of heat absorption (or rejection) for certain space missions. During the course of the program, coatings were tested which had large values of the ratio of solar absorptance to emittance ($\alpha/\epsilon > 5$). The first of these was a coating for type 430 stainless steel with a solar absorptance of 0.77 and an emittance of 0.15. This material was described on page 17 of the Progress Report, dated 16 April 1962. The vendor, Orion Products of Los Alamitos, California, subsequently furnished two more samples of this process. The two samples had solar absorptances of 0.77 and 0.86 and emittances of 0.12 and 0.18 respectively.

Another material with a large value of the α/ϵ ratio is obtained on copper with the Ebonol C process.* This technique requires careful control of the bath temperature and immersion time. The best results obtained to date were for a bath consisting of 100 grams/liter of the process chemical, a bath temperature of 217°F (boiling) and an immersion of three minutes. The copper was bright dipped prior to coating. The resulting solar absorptance was 0.84 and the emittance was 0.05.

A process for coating titanium was also reported in the Progress Report, dated 16 April 1962. This material was interesting since it duplicated the oxidized titanium used on the Able-5 vehicles ($\alpha/\epsilon = 0.71/0.12$). The vendor was the Hi-Shear Corporation of Torrance, California, and they exhibited a high degree of control over their process. An anodizing process for titanium was tested in the laboratory and it showed promise of duplicating the Hi-Shear material. However, the need for this duplication was not believed to be sufficient to continue the development.

* Ingredients available from the L. H. Butcher Co., Los Angeles.

Table III-1. Description of Sulfuric Acid Anodizing Processes

| <u>Bath</u> | <u>Description</u> |
|-------------|--|
| A | <p>Cleaned, deoxidized and bright dipped in Greater Mountain Chemicals 800 solution at 170°F for 10 minutes. Anodized in a 15 percent (by weight) sulfuric acid solution at 70°F ±2°F, current density 12 amperes per square foot, with either</p> <ol style="list-style-type: none"> 1. No seal. 2. Sealed in deionized water at 200°F for 20 minutes. 3. Sealed in a 0.5 percent nickel acetate solution at 200°F for five minutes. |
| B | <p>Same as A except 16% (by weight) sulfuric acid solution, voltage 11 to 18 vdc, no seal, bath temperature and current density as below:</p> <ol style="list-style-type: none"> 1. 60°F <ol style="list-style-type: none"> a. 6 amperes per square foot b. 8 amperes per square foot c. 12 amperes per square foot 2. 70°F <ol style="list-style-type: none"> a. 6 amperes per square foot b. 8 amperes per square foot c. 12 amperes per square foot 3. 80°F <ol style="list-style-type: none"> a. 6 amperes per square foot b. 8 amperes per square foot c. 12 amperes per square foot |
| C | <p>Same as A except 12 percent (by weight) sulfuric acid solution and current density 24 amperes per square foot, no seal.</p> |
| D | <p>No bright dip, anodized in 17 percent (by weight) sulfuric acid solution at 68°F ±2°F, current density 15 amperes per square foot, no seal.</p> |
| E | <p>Cleaned, deoxidized and bright dipped in Greater Mountain Chemicals 800 solution at 170°F for 10 minutes. Anodized in a 12 percent (by weight) sulfuric acid solution at 25°F ±2°F, current density 24 amperes per square foot, no seal.</p> |
| F | <p>Same as Bath E except 11 percent (by weight) sulfuric acid solution and current density either.</p> <ol style="list-style-type: none"> 1. 12 amperes per square foot or 2. 32 amperes per square foot |
| G | <p>Cleaned, deoxidized and bright dipped in Greater Mountain Chemicals 800 solution at 170°F for 10 minutes. Hard anodized by "Martin Hard Coating" standard process.</p> |

Table III-2. Observed Ranges of Solar Absorptance and Emittance for the Sulfuric Acid Anodizing Process and Various Alloys

| <u>Alloy and Bath</u> | <u>Range of Values</u> | |
|-----------------------------------|--------------------------|-------------------------|
| | <u>Solar Absorptance</u> | <u>Normal Emittance</u> |
| 1199 Alloy, Bath (A) | 0.12 - 0.20 | 0.56 - 0.89 |
| 1199 Alloy, Bath (F) | 0.18 - 0.60 | 0.72 - 0.79 |
| 1170 Alloy Clad on 2014, Bath (A) | 0.16 - 0.44 | 0.61 - 0.89 |
| 1170 Alloy Clad on 2014, Bath (C) | 0.18 - 0.52 | 0.45 - 0.82 |
| 1170 Alloy Clad on 2014, Bath (B) | 0.24 - 0.53 | 0.82 - 0.94 |
| 2014-T6 Alclad Alloy, Bath (D) | 0.32 - 0.54 | 0.76 - 0.95 |
| 2024-T3 Alclad Alloy, Bath (D) | 0.24 - 0.44 | 0.76 - 0.94 |
| 5052-H34 Alloy, Bath (D) | 0.26 - 0.41 | 0.79 - 0.93 |
| 5557 Alloy, Bath (D) | 0.15 - 0.23 | 0.76 - 0.87 |
| 5557 Alloy, Bath (E) | 0.22 - 0.66 | 0.73 - 0.83 |
| 6061 Alloy, Bath (D) | 0.35 - 0.60 | 0.78 - 0.95 |
| 6061 Alloy, Bath (G) | 0.32 - 0.92 | 0.72 - 0.85 |
| 7075 Alloy, Bath (D) | 0.40 - 0.51 | 0.79 - 0.94 |

Table III-3. Nonsulfuric Anodizing Electrolytes Tested

| | | |
|-----|--|---------|
| A. | Sulfamic Acid | |
| | Sulfamic acid (NH_3SO_3) | 100 g/l |
| B. | Zirconyl Sulfate | |
| (1) | Zirconyl sulfate ($\text{ZrOSO}_4 \cdot \text{H}_2\text{SO}_4 \cdot 3\text{H}_2\text{O}$) adjusted with distilled water to a density of 1.14 (32.8% ZrO_2) | |
| (2) | As above but a density of 1.07 | |
| (3) | As above but a density of 1.20 | |
| C. | Boric Acid | |
| | H_3BO_3 | 90 g |
| | $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ | 0.6 g |
| | H_2O | 1 l |
| D. | Oxalic acid | |
| (1) | $(\text{COOH})_2$ | 40 g |
| | H_2O | 1 l |
| (2) | $(\text{COOH})_2$, (Commercial Vendor) 3% (wt) solution | |
| E. | Chromic Acid | |
| (1) | Chromium trioxide | 100 g |
| | Phosphoric acid | 20 g |
| | Oxalic acid | 20 g |
| | H_2O | 1 l |
| (2) | Chromium trioxide | 50 g |
| | Oxalic acid | 5 g |
| | Boric acid | 3 g |
| | H_2O | 1 l |
| (3) | Chromic acid, 5% solution (commercial vendor) | |
| F. | Citric Acid | |
| (1) | Citric acid | 1 g |
| | Ammonium citrate | 1 g |
| | H_2O | 1 l |
| (2) | Citric acid | 2 g |
| | Ammonium citrate | 2 g |
| | H_2O | 1 l |
| (3) | Citric acid | 0.5 g |
| | Ammonium citrate | 0.5 g |
| | H_2O | 1 l |

Table III-3. Nonsulfuric Anodizing Electrolytes Tested (Continued)

| | | |
|-----|--|---------|
| (4) | Citric acid | 1 g |
| | Ammonium acetate | 1 g |
| | H ₂ O | 1 l |
| G. | Oxalic-sulfuric acid (commercial vendor) | |
| | H ₂ SO ₄ | 6% (wt) |
| | (COOH) ₂ | 6% (wt) |
| | H ₂ O | |
| H. | Water | |
| | Distilled water | |

Table III-4. Sulfamic Acid Anodized Aluminum

General Conditions: Bath temperature 20°C; lead (Pb) cathode; substrates were not given any pretreatment; substrate 1199 aluminum foil.

| <u>Sample No.</u> | <u>Specific Conditions</u> | <u>a</u> | <u>ε</u> |
|-------------------|---|----------|----------|
| 1 | 60 vdc, 0.5 - 0.7 a/90 cm ² , 20 min | 0.37 | 0.68 |
| 2 | 45 vdc, 0.2 a/90 cm ² , 20 min | 0.33 | 0.39 |
| 3 | 12 vac, 0.5 a/80 cm ² , 30 min | 0.27 | 0.05 |
| 4 | 15 vac, 1.8 a/80 cm ² , 30 min | 0.64 | 0.77 |
| 5 | 5.5 vac, 0.3 a/80 cm ² , 15 min | 0.27 | 0.03 |
| 6 | 16 vac, 1.0 a/80 cm ² , 120 min | 0.78 | 0.85 |

Table III-5. Zirconyl Sulfate Anodized Aluminum

| <u>Sample No.</u> | <u>Conditions</u> (See Table for bath description, no substrate pretreatment) | <u>α</u> | <u>ϵ</u> |
|-------------------|--|----------------------------|------------------------------|
| 1 | Bath B(1), type 1199 aluminum foil, 54 x 75 mm, 2 amps d-c, 33-36°C, 30 min, sealed in boiling H ₂ O for 10 min | 0.22 | 0.79 |
| 2 | Same as 1 but 1.0 amps d-c | 0.21 | 0.81 |
| 3 | Bath B(1), type 1170 clad on 2014 aluminum, 54 x 75 mm, 2 amps d-c, 33-35°C, 30 min | 0.36 | 0.85 |
| 4 | Bath B(2), type 1199 foil, 54 x 75 mm, 3 amps d-c, 70-58°C, 60 min | 0.18 | 0.89 |
| 5 | Same as 4 but 3.0 amps d-c, 40°C, 60 min | 0.24 | 0.90 |
| 6 | Bath B(2), type 1170 clad on 2014 aluminum, 54 x 75 mm, 0.8 amp d-c, 25-30°C, 150 min | 0.41 | 0.91 |
| 7 | Bath B(2), type 6061 aluminum, 54 x 75 mm, 2.0 amps d-c, 25-30°C, 120 min | 0.54 | 0.91 |
| 8 | Bath B(2), type 1199 sheet, 54 x 75 mm, 0.5-0.3 amp d-c, 25°C, 16 hours | 0.20 | 0.91 |
| 9 | Bath B(3), type 1199 aluminum foil 54 x 75 mm, 2.0 amps d-c, 27-32°C, 60 min | 0.20 | 0.87 |
| 10 | Bath B(3), type 5557 aluminum foil, 48 x 52 mm, 1.3 amps d-c, 40-42°C, 60 min | 0.26 | 0.91 |
| 11 | Same as 10 but 2.2 amps d-c, 27-41°C, 60 min | 0.35 | 0.93 |

Table III-6. Boric Acid Anodized Aluminum

| <u>Sample No.</u> | <u>Conditions</u> | <u>α</u> | <u>ϵ</u> |
|-------------------|---|----------------------------|------------------------------|
| 1 | Bath C, type 1199 aluminum foil 40 min, 250 vdc, 35°C | 0.36 | 0.06 |

Table III-7. Oxalic Acid Anodized Aluminum

| <u>Sample No.</u> | <u>Conditions</u> | <u>α</u> | <u>ϵ</u> |
|-------------------|---|----------------------------|------------------------------|
| 1 | Bath D(1), type 1199 aluminum foil 50 x 75 mm, 60 vdc, 50 min, 17-21°C | 0.35 | 0.81 |
| 2 | Bath D(1), type 1199 aluminum foil, 50 x 75 mm, 35 vdc, 25 min, 35°C | 0.28 | 0.71 |
| 3 | Bath D(1), type 6061 aluminum, 50 x 75 mm, 35 vdc, 35°C, 20 min | 0.36 | 0.76 |
| 4 | Bath D(1), type 6061 aluminum, 50 x 75 mm, 60 vdc, 17-19°C, 42 min | 0.46 | 0.75 |
| 5 | Bath D(1), type 6061 aluminum, 50 x 75 mm, 60 vdc, 17-19°C, sealed in boiling H ₂ O for 15 min | 0.46 | 0.80 |
| 6 | Bath D(1), type 6061 aluminum, 50 x 75 mm, 120 vdc, (max - steadily incr) 6-9°C | 0.61 | 0.87 |
| 7 | Bath D(1), type 1170 clad on 2014, 50 x 75 mm, 30 vdc, 35°C, 27 min | 0.26 | 0.72 |
| 8 | Bath D(1), type 2024 aluminum, 50 x 75 mm, 60 vdc, 17-19°C, 47 min | 0.42 | 0.78 |
| 9 | Bath D(2), type 1170 clad on 2014, deoxidized and GMC-800 bright dip at 170°F for 10 min, 7.5 amps/ft ² , 100°F, 45 min, 0.55 mil thick | 0.27 | 0.81 |
| 10 | Bath D(2), type 1170 clad on 2014, deoxidized and GMC-800 bright dip at 170°F for 10 min, 7.5 amps/ft ² , 100°F, 60 min, 0.8 mil thick | 0.31 | 0.86 |
| 11 | Bath D(2), type 1170 clad on 2014, deoxidized and GMC-800 bright dip at 170°F for 10 min, 7.5 amps/ft ² , 100°F 90 min, 1.2 mil thick | 0.34 | 0.90 |
| 12 | Bath D(2), type 5557 aluminum alloy, deoxidized and GMC-800 bright dip at 170°F for 10 min, 8.25 amps/ft ² , 100°F, 90 min, 0.8 mil thick | 0.30 | 0.84 |
| 13 | Bath D(2), type 5557 aluminum alloy, deoxidized and GMC-800 bright dip at 170°F for 10 min, 8.25 amps/ft ² , 100°F, 120 min, 1.0 mil thick | 0.32 | 0.88 |
| 14 | Bath D(2), type 5557 aluminum alloy, deoxidized and GMC-800 bright dip at 170°F for 10 min, 8.25 amps/ft ² , 100°F, 150 min, 1.2 mil thick | 0.34 | 0.91 |

Table III-7. Oxalic Acid Anodized Aluminum (Continued)

| <u>Sample No.</u> | <u>Conditions</u> | <u>α</u> | <u>ε</u> |
|-------------------|---|----------|----------|
| 15 | Bath D(2), type 6061 aluminum alloy, deoxidized and GMC-800 bright dip at 170°F for 10 min, amps/ft ² , 60 min, 0.4 mil thick | 0.42 | 0.81 |
| 16 | Bath D(2), type 6061 aluminum alloy, deoxidized and GMC-800 bright dip at 170°F for 10 min, amps/ft ² , 100 min, 0.8 mil thick | 0.50 | 0.90 |
| 17 | Bath D(2), type 6061 aluminum alloy, deoxidized and GMC-800 bright dip at 170°F for 10 min, amps/ft ² , 150 min, 1.0 mil thick | 0.52 | 0.92 |

Table III-8. Chromic Acid Anodized Aluminum

| <u>Sample No.</u> | <u>Conditions</u> | <u>α</u> | <u>ε</u> |
|-------------------|--|----------|----------|
| 1 | Bath E(1), type 1199 aluminum foil, 50 x 75 mm, 90 vdc (max), 60 min, 50°C | 0.31 | 0.70 |
| 2 | Bath E(1), type 1199 aluminum foil, 50 x 75 mm, 60 vdc, 90 min, 40°C | 0.31 | 0.72 |
| 3 | Bath E(1), type 6061 aluminum alloy, 50 x 75 mm, 100 vdc, 0.4 amp, 70 min, 50°C | 0.47 | 0.53 |
| 4 | Bath E(1), type 6061 aluminum alloy, 50 x 75 mm, 120 vdc, 0.3 amp, 90 min, 25°C | 0.48 | 0.10 |
| 5 | Bath E(2), type 1199 aluminum foil, 50 x 75 mm, 90 vdc (max), 0.5 amp, 60 min, 40°C | 0.43 | 0.66 |
| 6 | Bath E(2), type 6061 aluminum alloy, 60 vdc, 0.6 amp, 45°C | 0.56 | 0.73 |
| 7 | Bath E(3) type 1170 clad on 2014 aluminum alloy, deoxidized and GMC-800 bright dip at 170°F for 10 min, 40 vdc, 30 min, 95°F, 0.08 mil thick | 0.30 | 0.53 |
| 8 | Bath E(3), type 1170 clad on 2014 aluminum alloy, deoxidized and GMC-800 bright dip at 170°F for 10 min, 40 vdc, 60 min, 0.2 mil thick | 0.33 | 0.69 |
| 9 | Bath E(3), type 1170 clad on 2014 aluminum alloy, deoxidized and GMC-800 bright dip at 170°F for 10 min, 40 vdc, 120 min, 0.3 mil thick | 0.40 | 0.74 |

Table III-8. Chromic Acid Anodized Aluminum(Continued)

| <u>Sample No.</u> | <u>Conditions</u> | <u>a</u> | <u>ε</u> |
|-------------------|---|----------|----------|
| 10 | Bath E(3), type 5557 alloy, deoxidized and GMC-800 bright at 170°F for 10 min, 40 vdc, 30 min, 95°F, 0.1 mil thick | 0.30 | 0.62 |
| 11 | Bath E(3), type 5557 alloy, deoxidized and GMC-800 bright at 170°F for 10 min, 40 vdc, 60 min, 95°F, 0.2 mil thick | 0.38 | 0.69 |
| 12 | Bath E(3), type 5557 alloy, deoxidized and GMC-800 bright at 170°F for 10 min, 40 vdc, 120 min, 95°F, 0.4 mil thick | 0.56 | 0.78 |
| 13 | Bath E(3), type 6061 aluminum alloy deoxidized and GMC-800 bright dip at 170°F for 10 min, 40 vdc, 30 min, 95°F, 0.06 mil thick | 0.37 | 0.50 |
| 14 | Bath E(3), type 6061 aluminum alloy deoxidized and GMC-800 bright dip at 170°F for 10 min, 40 vdc, 60 min, 95°F, 0.2 mil thick | 0.41 | 0.72 |
| 15 | Bath E(3), type 6061 aluminum alloy deoxidized and GMC-800 bright dip at 170°F for 10 min, 40 vdc, 120 min, 95°F, 0.4 mil thick | 0.48 | 0.77 |

Table II-9. Citric Acid Anodized Aluminum

| <u>Sample No.</u> | <u>Conditions</u> | <u>a</u> | <u>ε</u> |
|-------------------|--|----------|----------|
| 1 | Bath F(1), type 1199 aluminum foil, 300 vdc, 60 min, lead (Pb) cathode | 0.27 | 0.08 |
| 2 | Same as 1 but bath F(2) and 40 min | 0.40 | 0.14 |
| 3 | Same as 1 but bath F(3) | 0.22 | 0.08 |
| 4 | Same as 1 but bath F(4), 45 min, 150-250 vdc | 0.37 | 0.07 |

Table III-10. Oxalic-Sulfuric Acid Anodized Aluminum

| <u>Sample No.</u> | <u>Conditions</u> | <u>α</u> | <u>ε</u> |
|-------------------|--|----------|----------|
| 1 | Bath B, type 1170 clad on 2014 aluminum alloy, deoxidized and GMC-800 bright dip at 170°F for 10 min, 12 amps/ft ² d-c, 70°F, 20 min, 0.5 mil thick | 0.25 | 0.78 |
| 2 | Same as 1 but 30 min, 0.7 mil thick | 0.30 | 0.80 |
| 3 | Same as 1 but 50 min, 1.1 mil thick | 0.42 | 0.82 |
| 4 | Same as 1 but type 5557 alloy, 24 min, 0.5 mil thick | 0.18 | 0.78 |
| 5 | Same as 4 but 36 min, 0.6 mil thick | 0.22 | 0.79 |
| 6 | Same as 4 but 60 min, 1.0 mil thick | 0.26 | 0.81 |
| 7 | Same as 1 but type 6061 aluminum alloy, 24 min, 0.4 mil thick | 0.42 | 0.81 |
| 8 | Same as 7 but 36 min, 0.6 mil thick | 0.52 | 0.82 |
| 9 | Same as 7 but 60 min, 1.0 mil thick | 0.58 | 0.82 |

Table III-11. Water Anodized Aluminum

| <u>Sample No.</u> | <u>Conditions</u> | <u>α</u> | <u>ε</u> |
|-------------------|--|----------|----------|
| 1 | Bath H, type 1199 aluminum alloy foil 22 hours at boiling point of distilled water/slightly acidic | 0.20 | 0.52 |

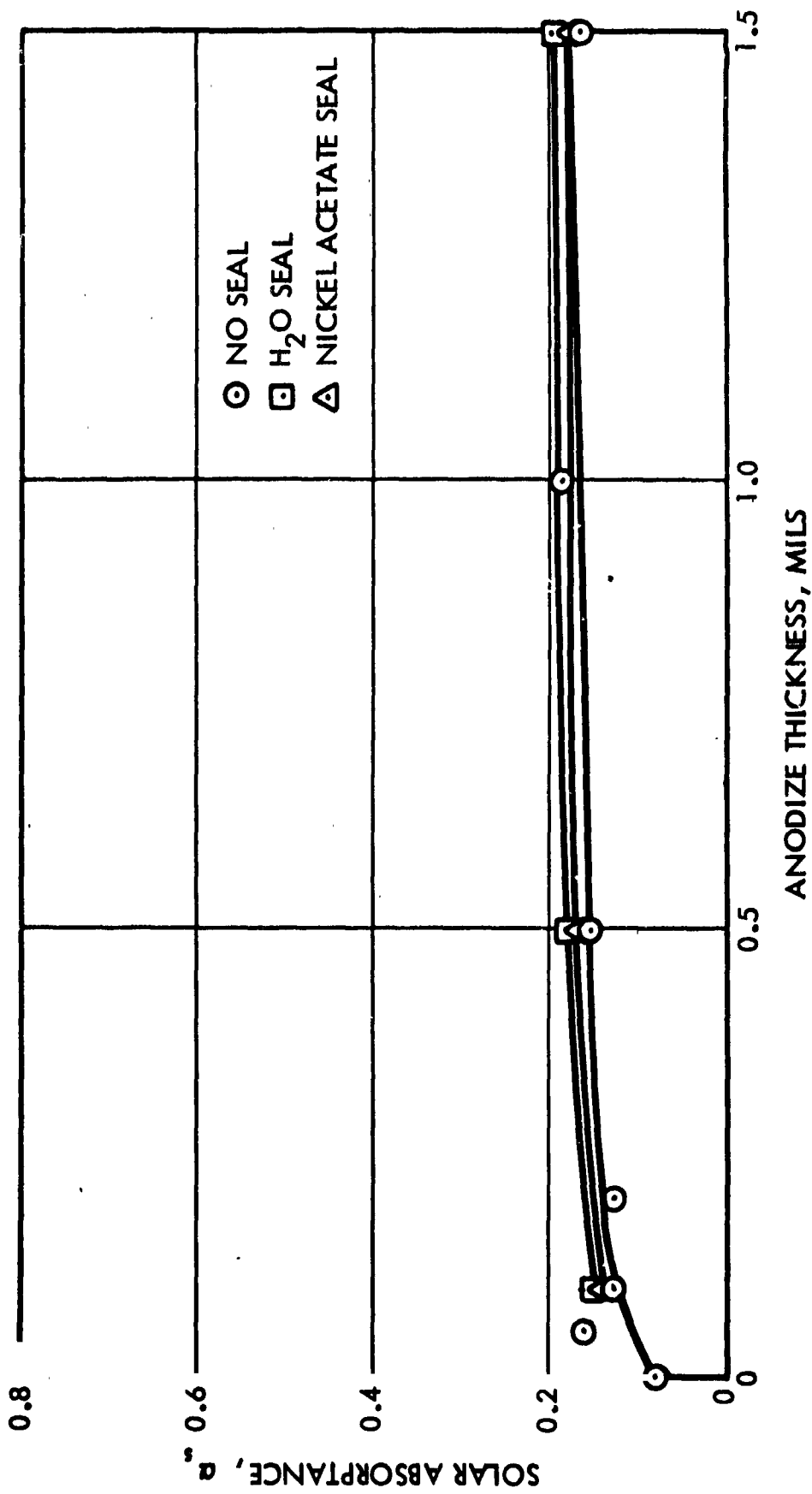


Figure III-1. 1199 Aluminum, Cleaned, Deoxidized and Bright Dipped in Greater Mountain Chemicals 800 Solution at 170°F for 10 Minutes, Anodized in a 15% (by weight) Sulfuric Acid Solution at 70°F ±2°F, Current Density 12 Amperes per Square Foot, Approximately 15 Volts dc

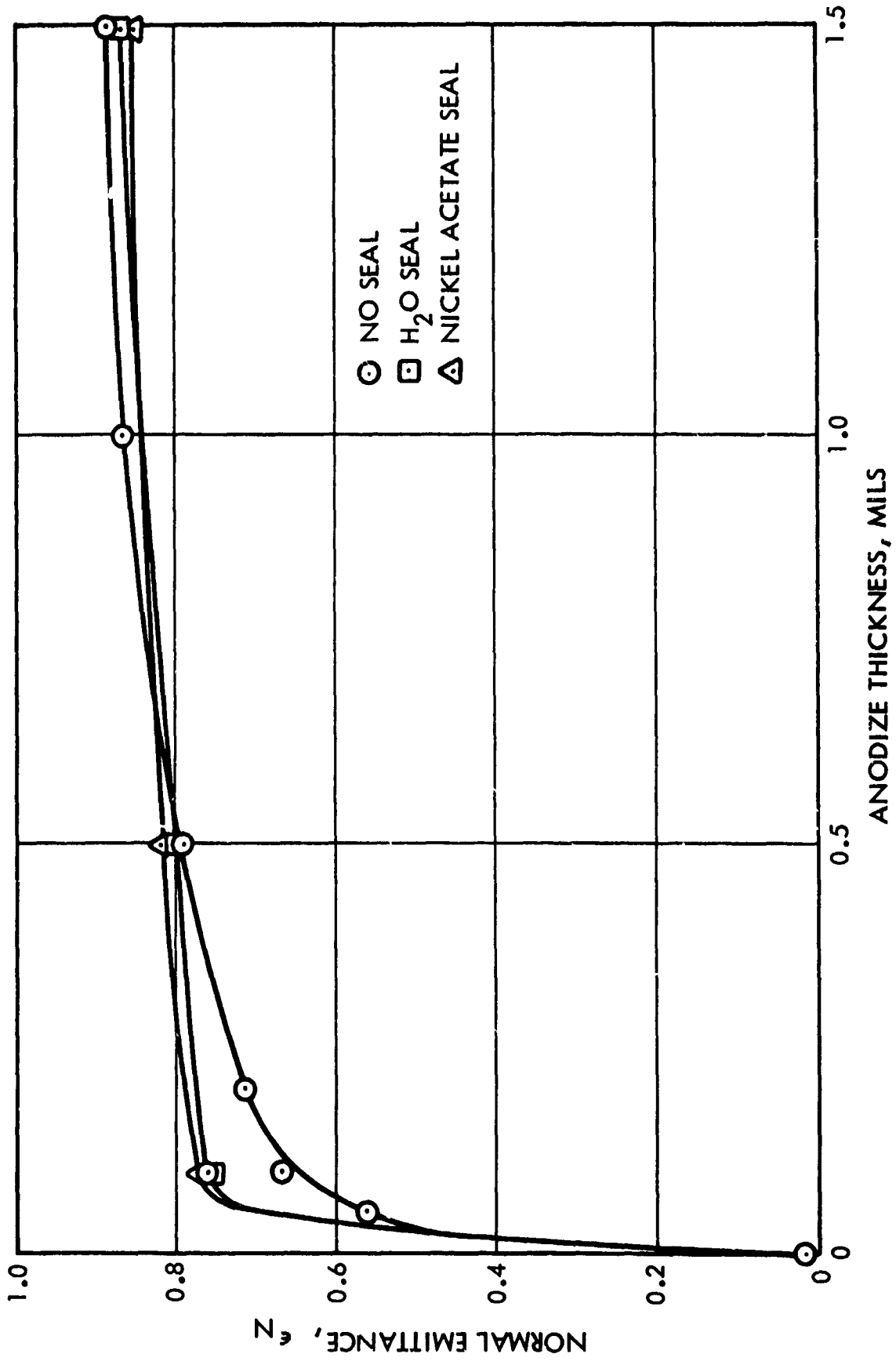


Figure III-2. 1199 Aluminum, Cleaned, Deoxidized and Bright Dipped in Greater Mountain Chemicals 800 Solution at 170°F for 10 Minutes, Anodized in a 15% (by weight) Sulfuric Acid Solution at 70°F ±2°F, Current Density 12 Amperes per Square Foot, Approximately 15 Volts dc

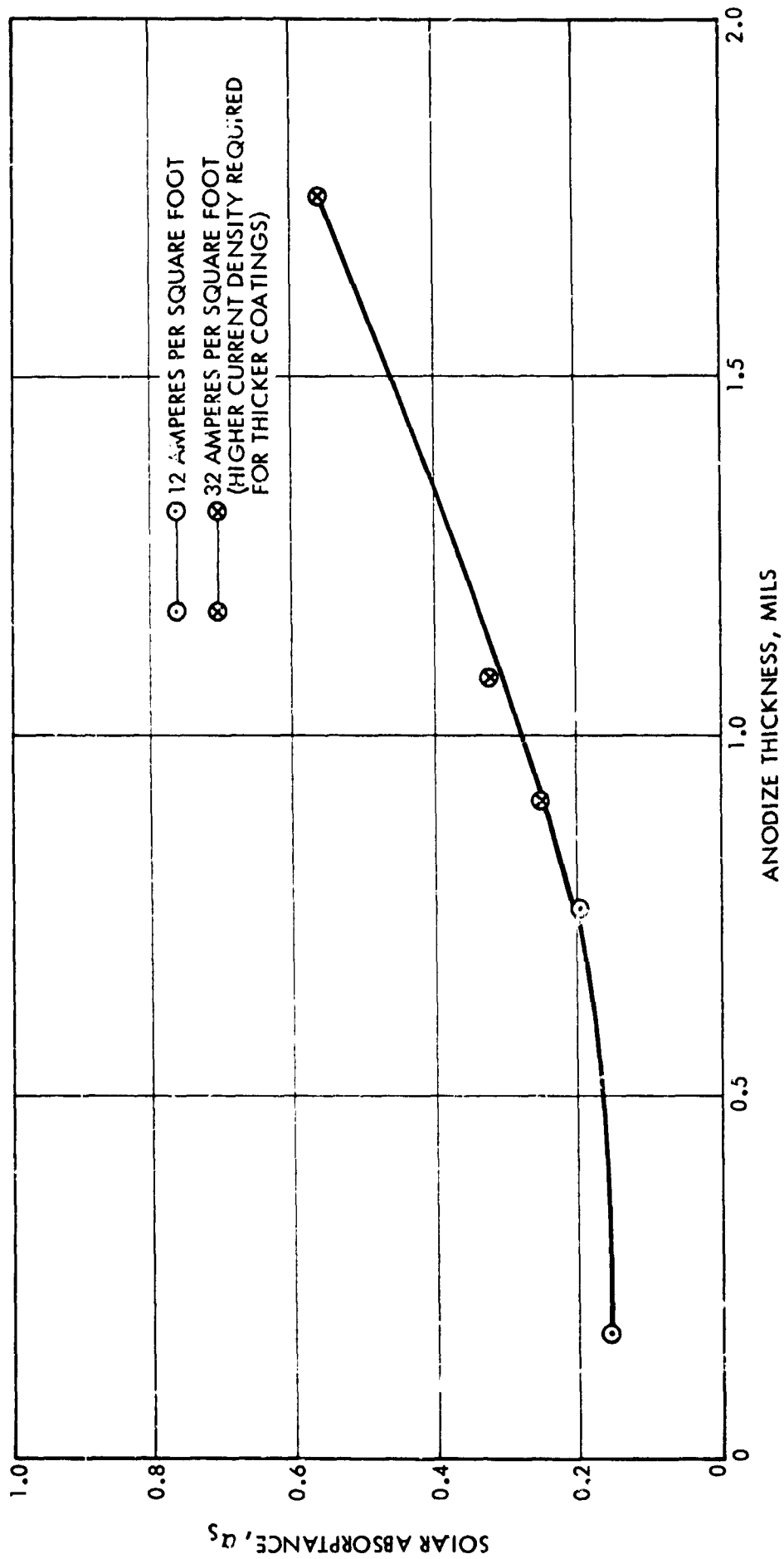


Figure III-3. 1199 Aluminum, Cleaned, Deoxidized and Bright Dipped in Greater Mountain Chemicals 800 Solution, Anodized in a 11% (by weight) Sulfuric Acid Solution at 25°F, No Seal

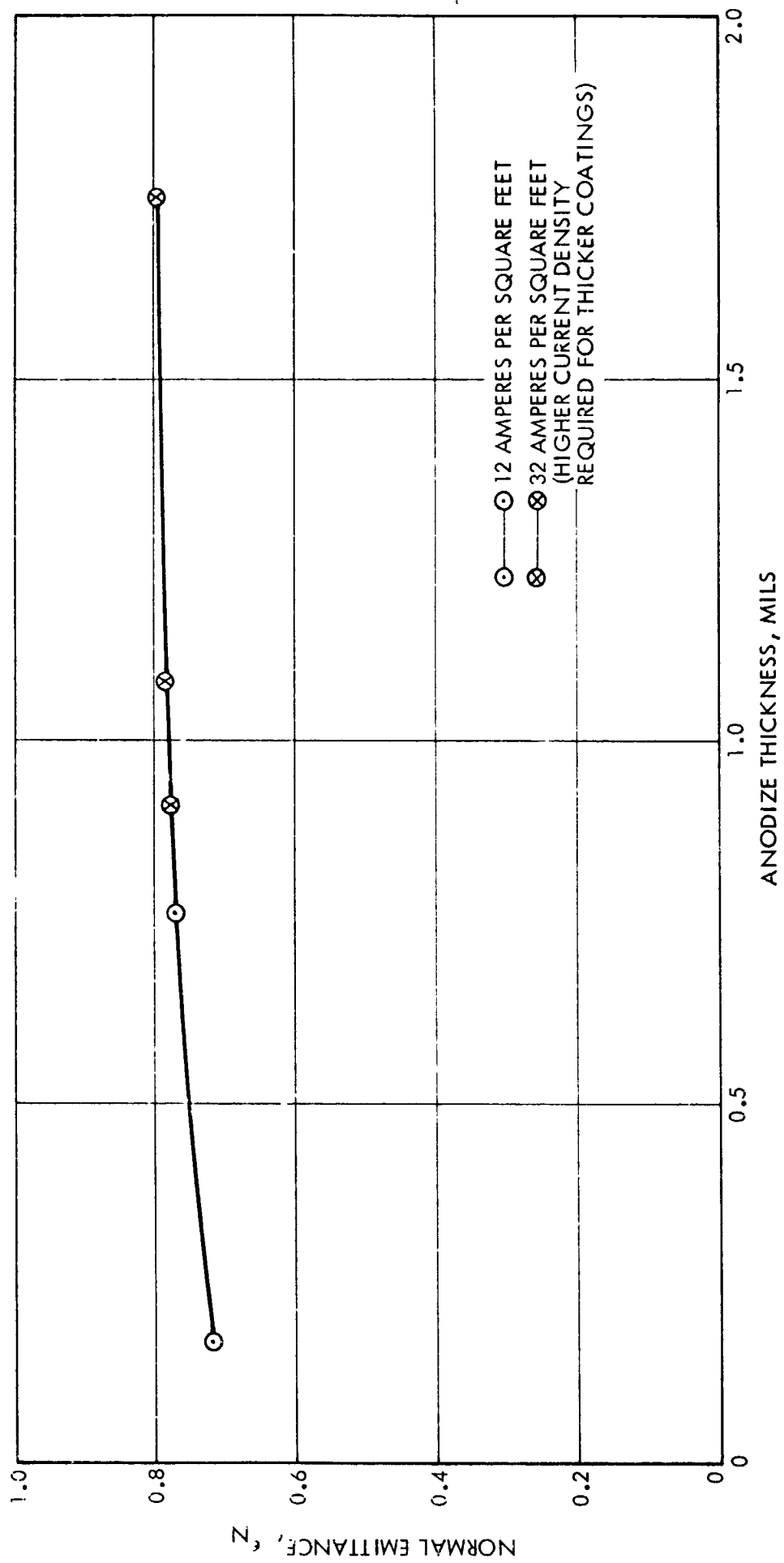


Figure III-4. 1199 Aluminum, Cleaned, Deoxidized and Bright Dipped in Greater Mountain Chemicals 800 Solution, Anodized in a 12% (by weight) Sulfuric Acid Solution at 25°F

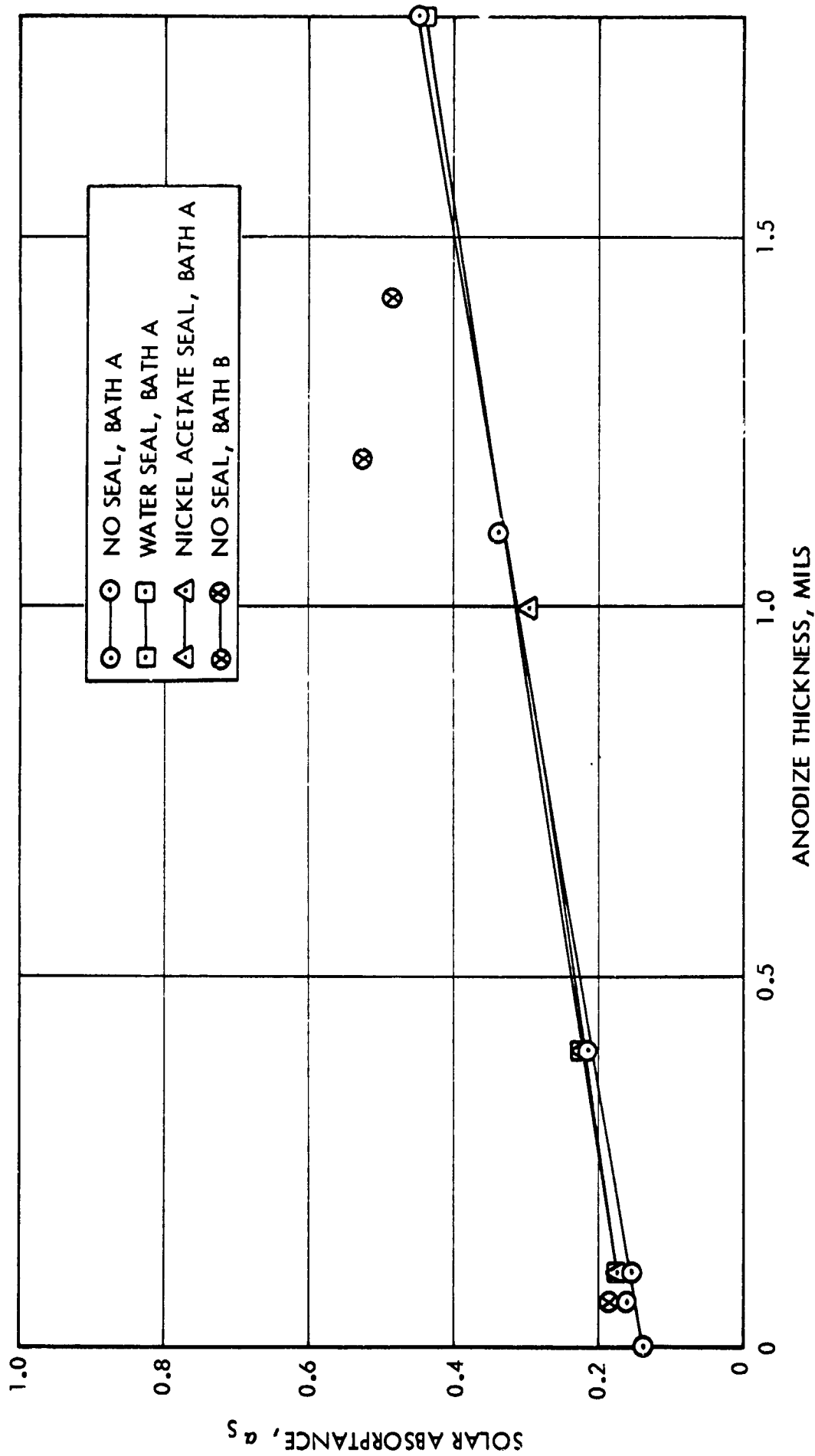


Figure III-5. 1170 Aluminum Alloy Clad on 2014, Cleaned, Deoxidized and Bright Dipped in Greater Mountain Chemicals 800 Solution at 170°F for 10 Minutes, Anodized in a 15% (by weight) Sulfuric Acid Solution at 70°F ±2°F, Current Density 12 Amperes per Square Foot (Bath A) or a 12% (by weight) Sulfuric Acid Solution at 70°F ±2°F, Current Density 24 Amperes per Square Foot (Bath B)

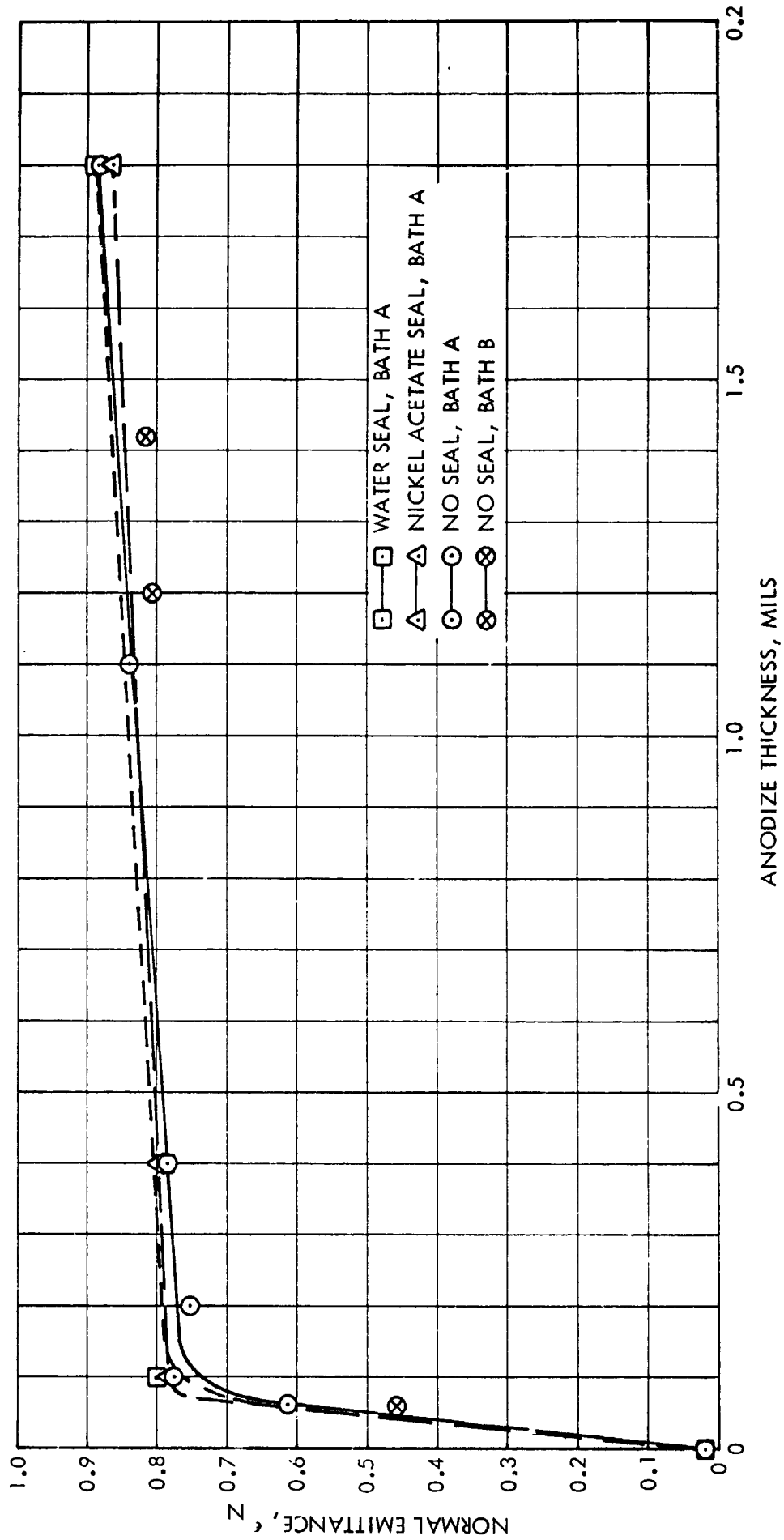


Figure III-6. 1170 Aluminum Alloy Clad on 2014, Cleaned, Deoxidized, and Bright Dipped in Greater Mountain Chemicals 800 Solution at 170°F for 10 minutes, Anodized in a 15% (by weight) Sulfuric Acid Solution at 70°F ± 2°F, Current Density 12 Amperes per Square Foot. (Bath A) or a 12% (by weight) Sulfuric Acid Solution at 70°F ± 2°F, Current Density 24 Amperes per Square Foot. (Bath B)

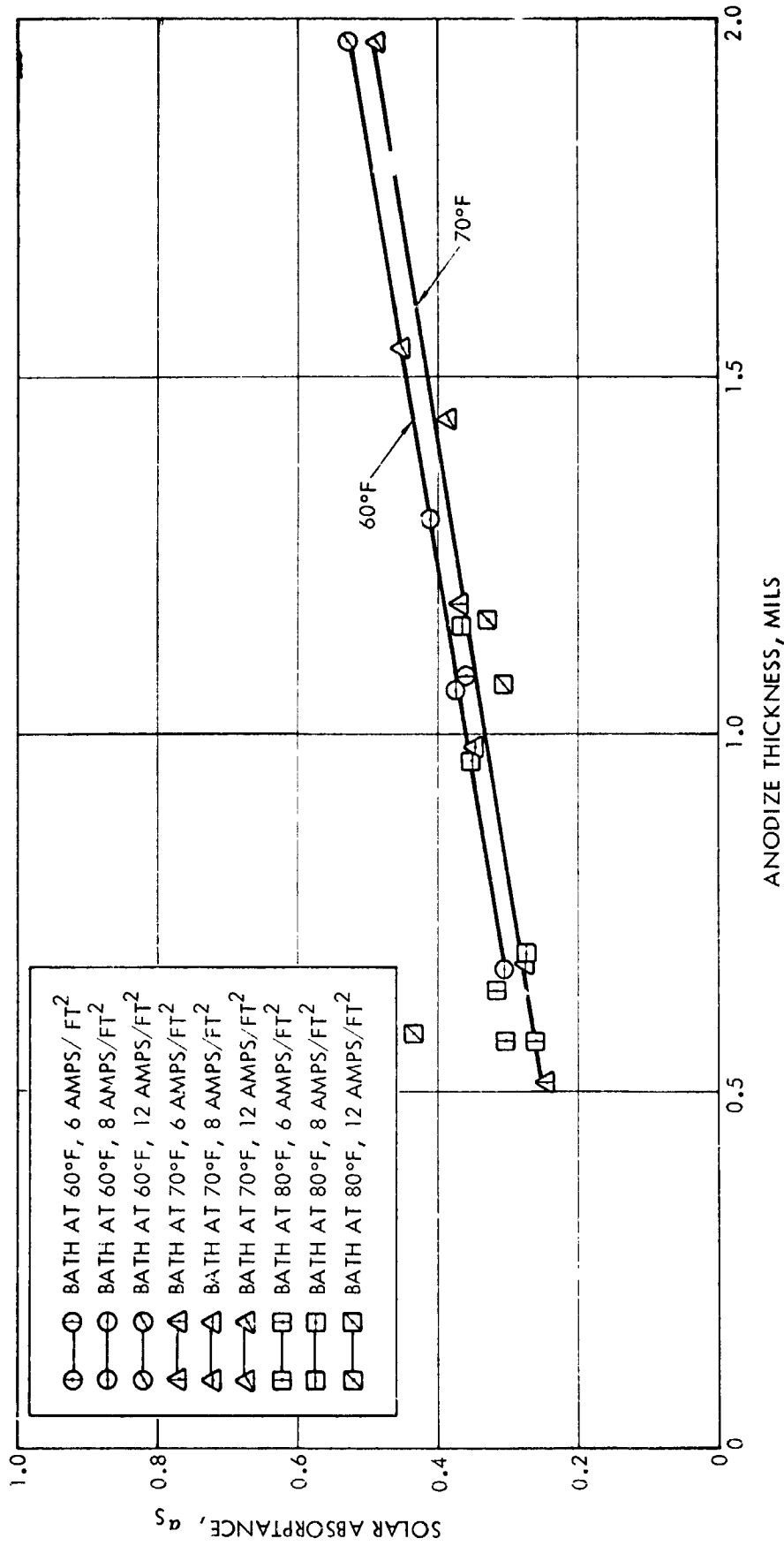


Figure III-7. 1170 Aluminum Alloy Clad on 2014, Cleaned, Deoxidized and Bright Dipped in Greater Mountain Chemicals 800 Solution at 170°F for 10 Minutes, Anodized in a 16% (by weight) Sulfuric Acid Solution, Current Density 6, 8 and 12 Amperes per Square Foot, Voltage 11 to 18 Volts dc, No Seal

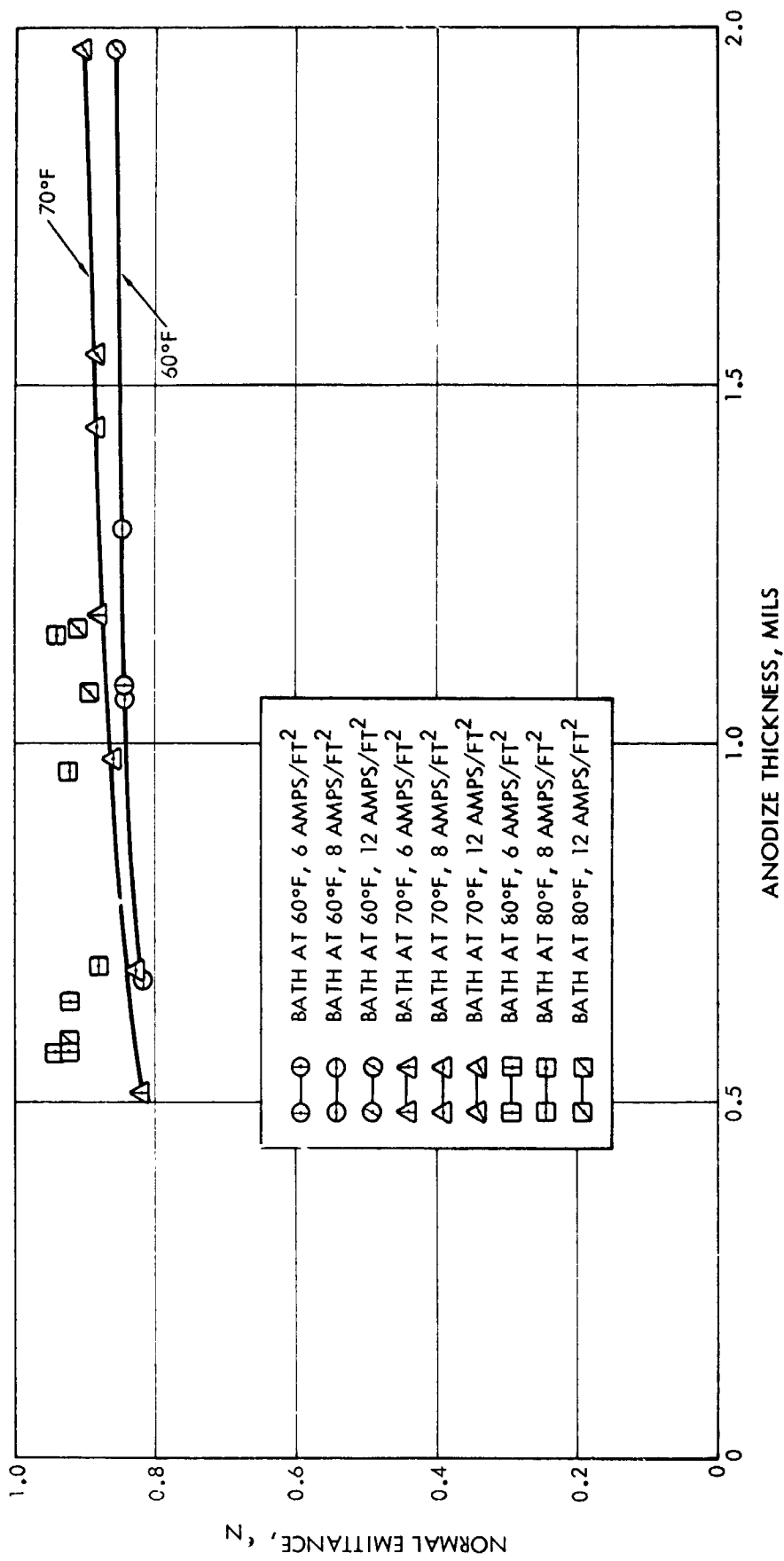


Figure III-8. 1170 Aluminum Alloy Clad on 2014, Cleaned, Deoxidized and Bright Dipped in Greater Mountain Chemicals 800 Solution at 170°F for 10 Minutes, Anodized in a 16% (by weight) Sulfuric Acid Solution, Voltage 11 to 18 Volts dc, No Seal

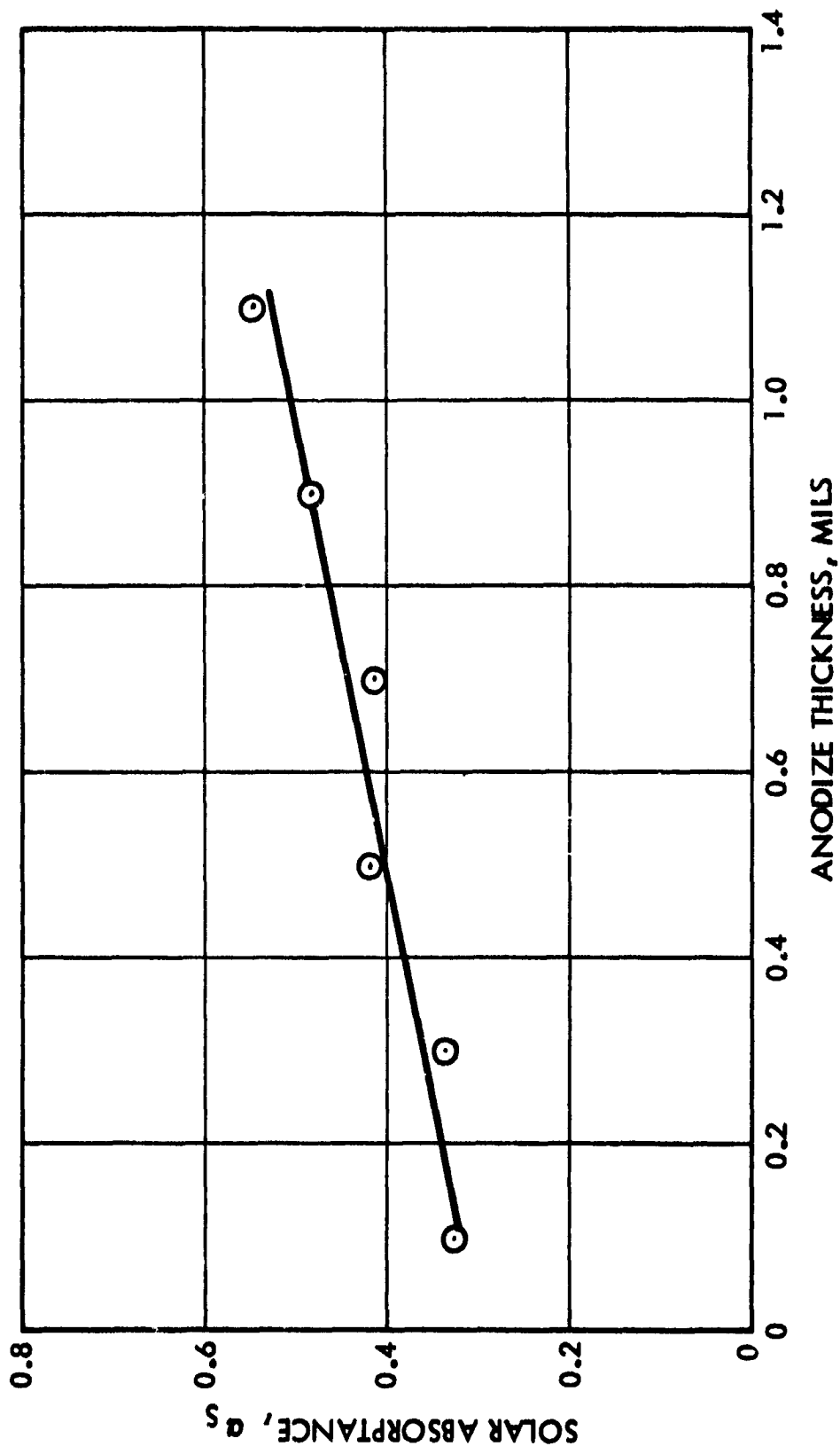


Figure III-9. 2014-T6 Alclad Aluminum Alloy, Anodized in a 17% (by weight) Sulfuric Acid Solution at 68° F ± 2° F, Current Density, 15 Amperes per Square Foot, No Seal

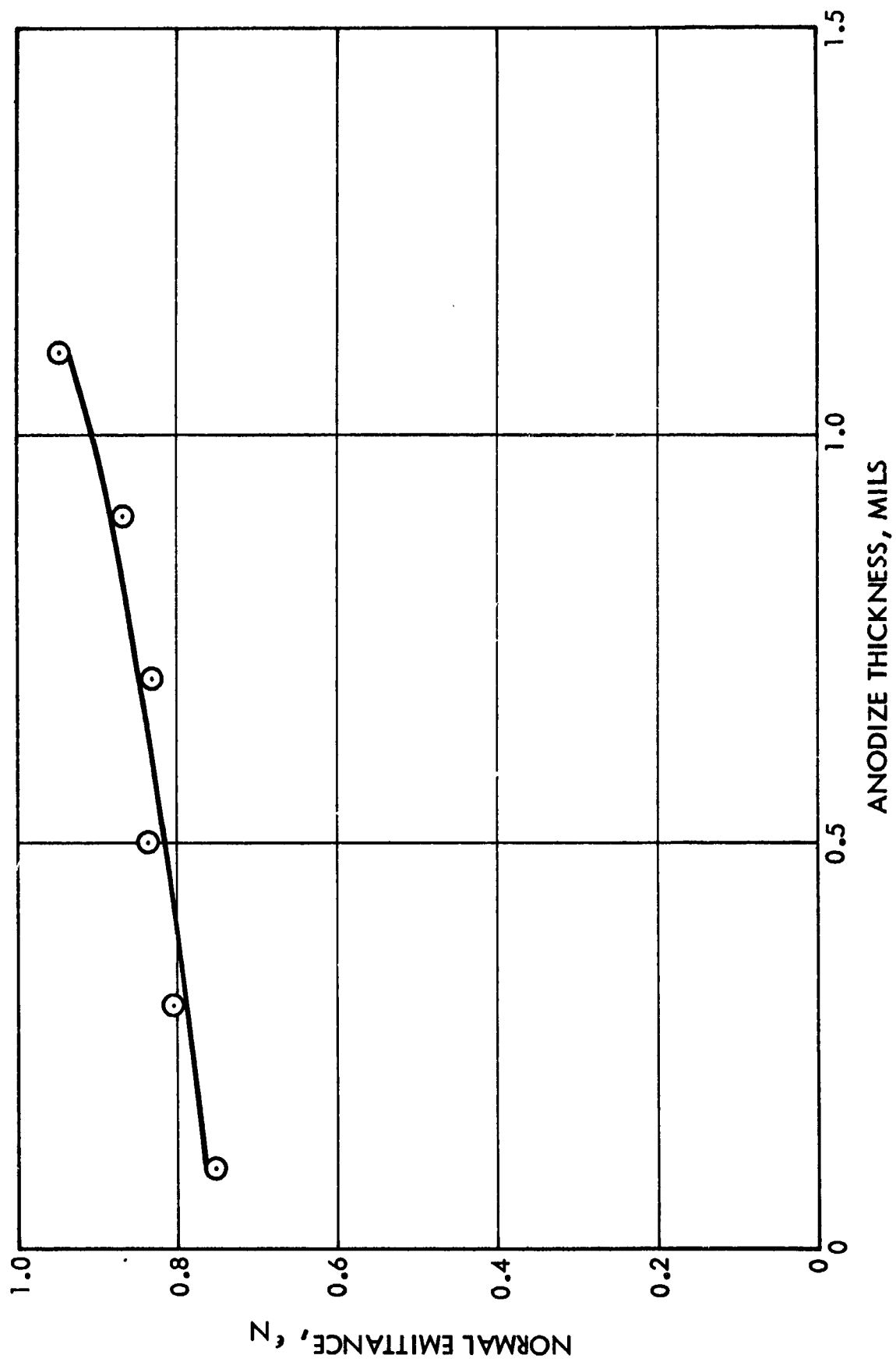


Figure III-10. 2014-T6 Alclad Aluminum Alloy, Anodized in a 17% (by weight) Sulfuric Acid Solution at 68°F ± 2°F, Current Density 15 Amperes per Square Foot, No Seal.

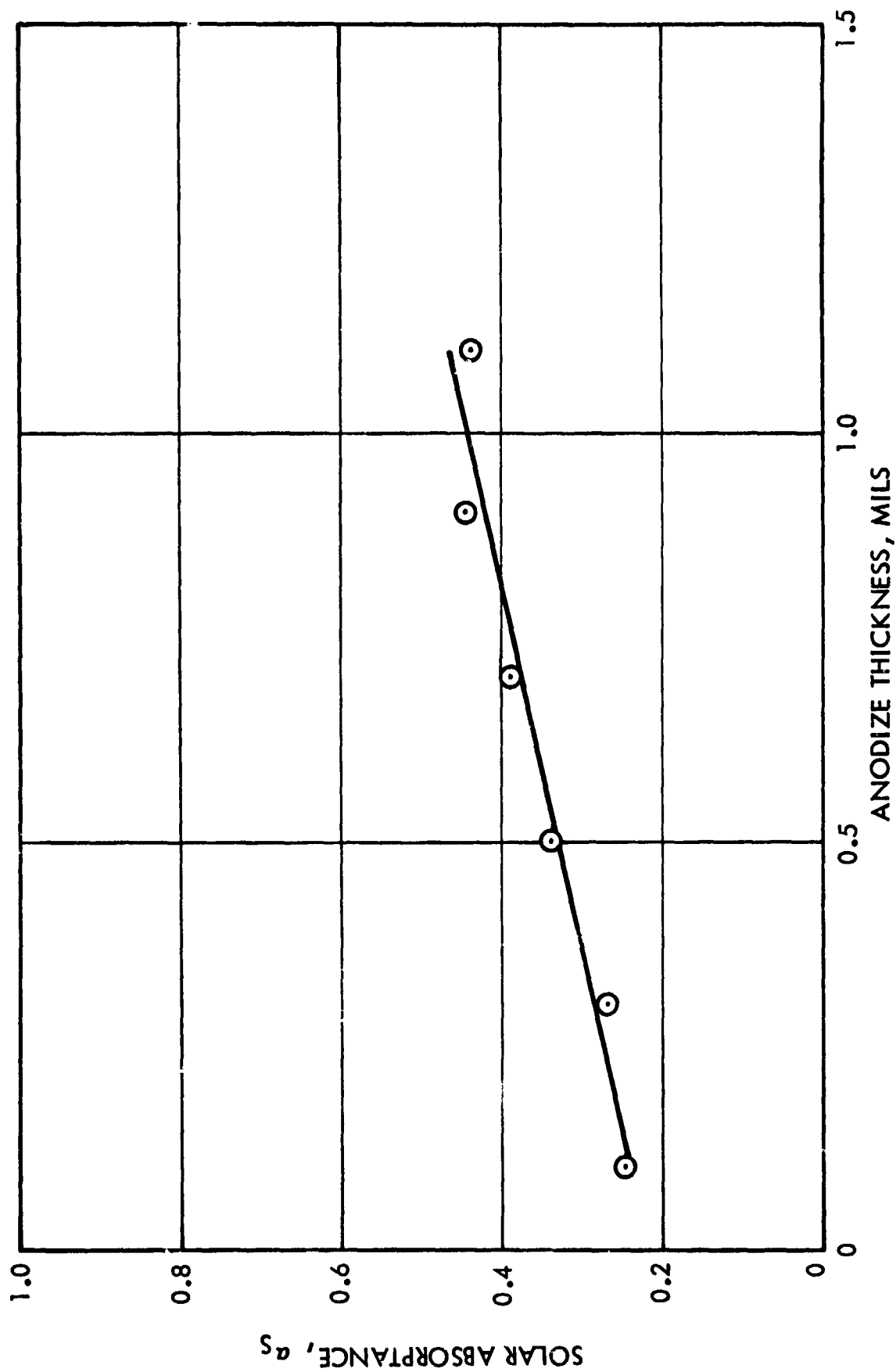


Figure III-11. 2024-T3 Alclad Aluminum Alloy, Anodized in a 17% (by weight) Sulfuric Acid Solution at $68^{\circ}\text{F} \pm 2^{\circ}\text{F}$, Current Density 15 Amperes per Square Foot, No Seal

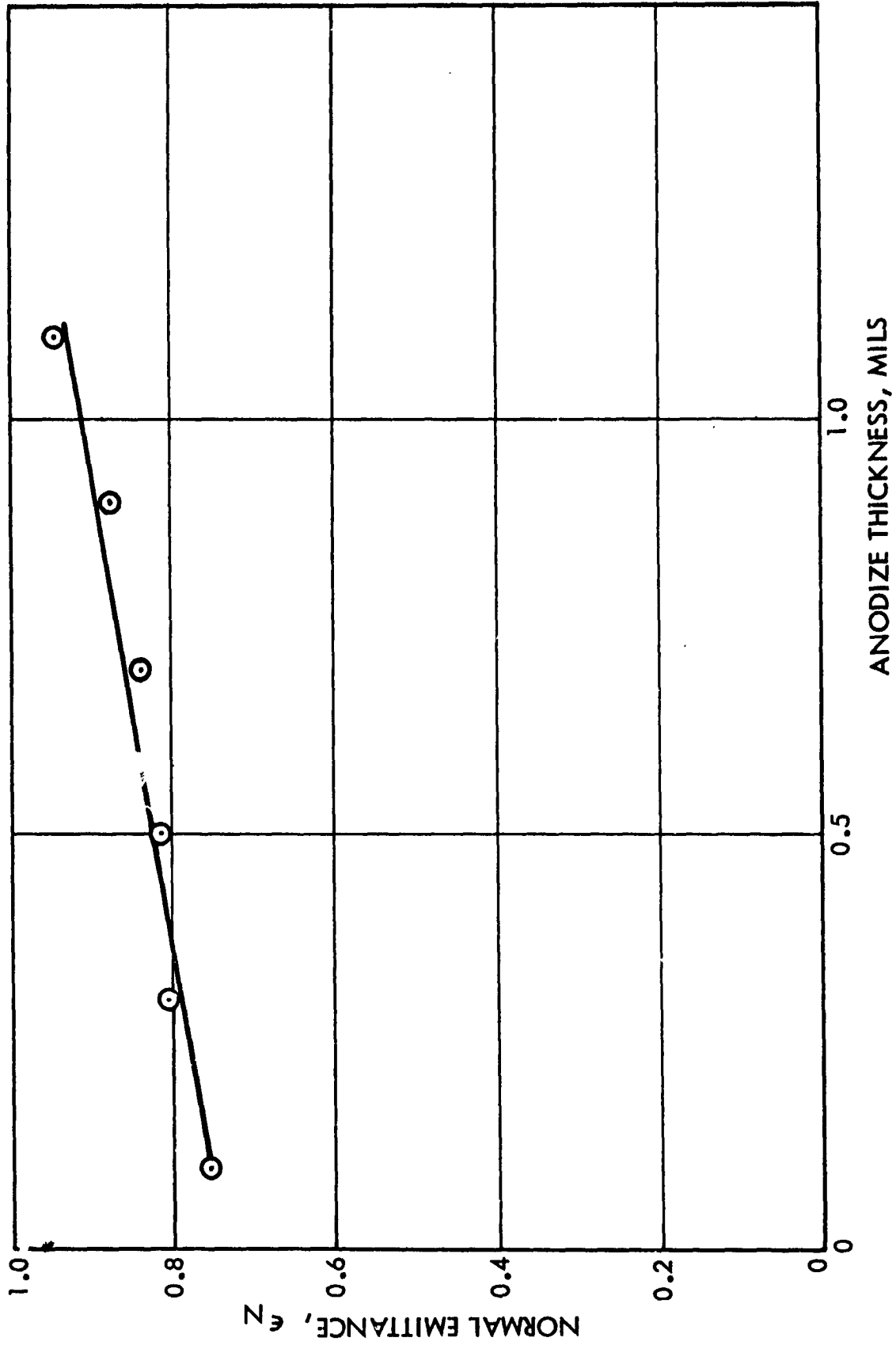


Figure III-12. 2024-T3 Alclad Aluminum Alloy, Anodized in a 17% (by weight) Sulfuric Acid Solution at 68° F ± 2° F, Current Density 15 Amperes per Square Foot, No Seal

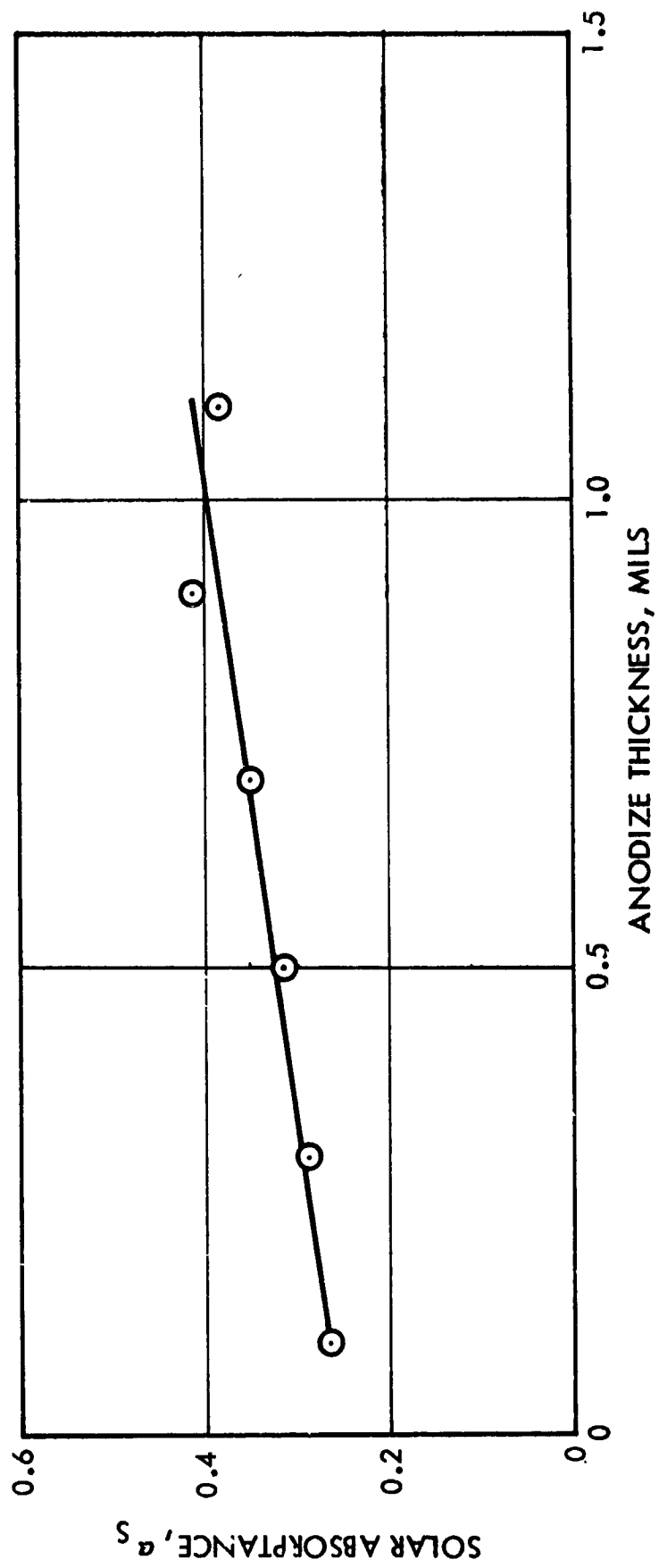


Figure III-13. 5052-H34 Aluminum Alloy, Anodized in a 17% (by weight) Sulfuric Acid Solution at 68° F ±2° F, Current Density 15 Amperes per Square Foot, No Seal

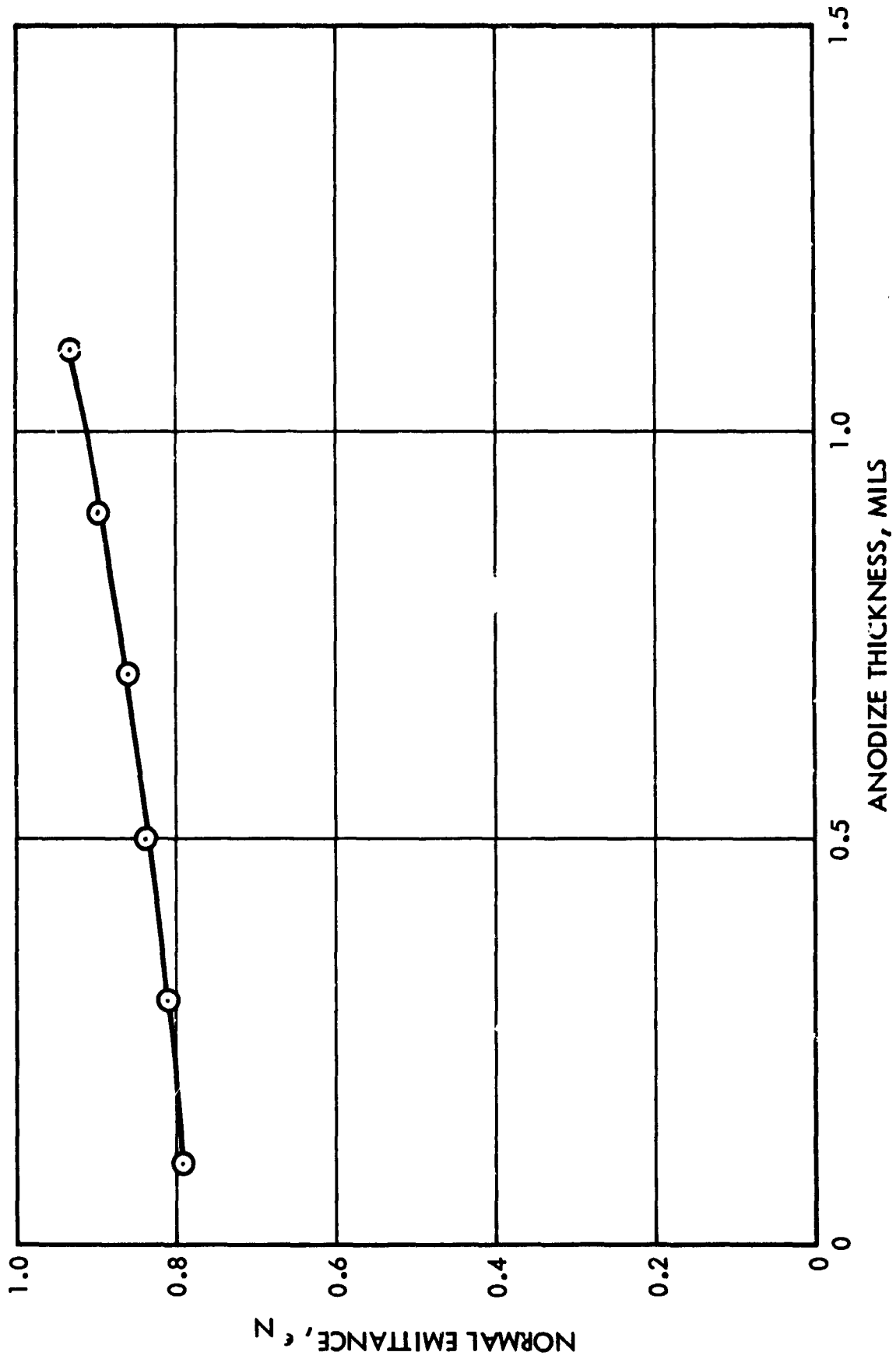


Figure III-14. 5052-H34 Aluminum Alloy, Anodized in a 17% (by weight) Sulfuric Acid Solution at 68°F ±2°F, Current Density 15 Amperes per Square Foot, No Seal

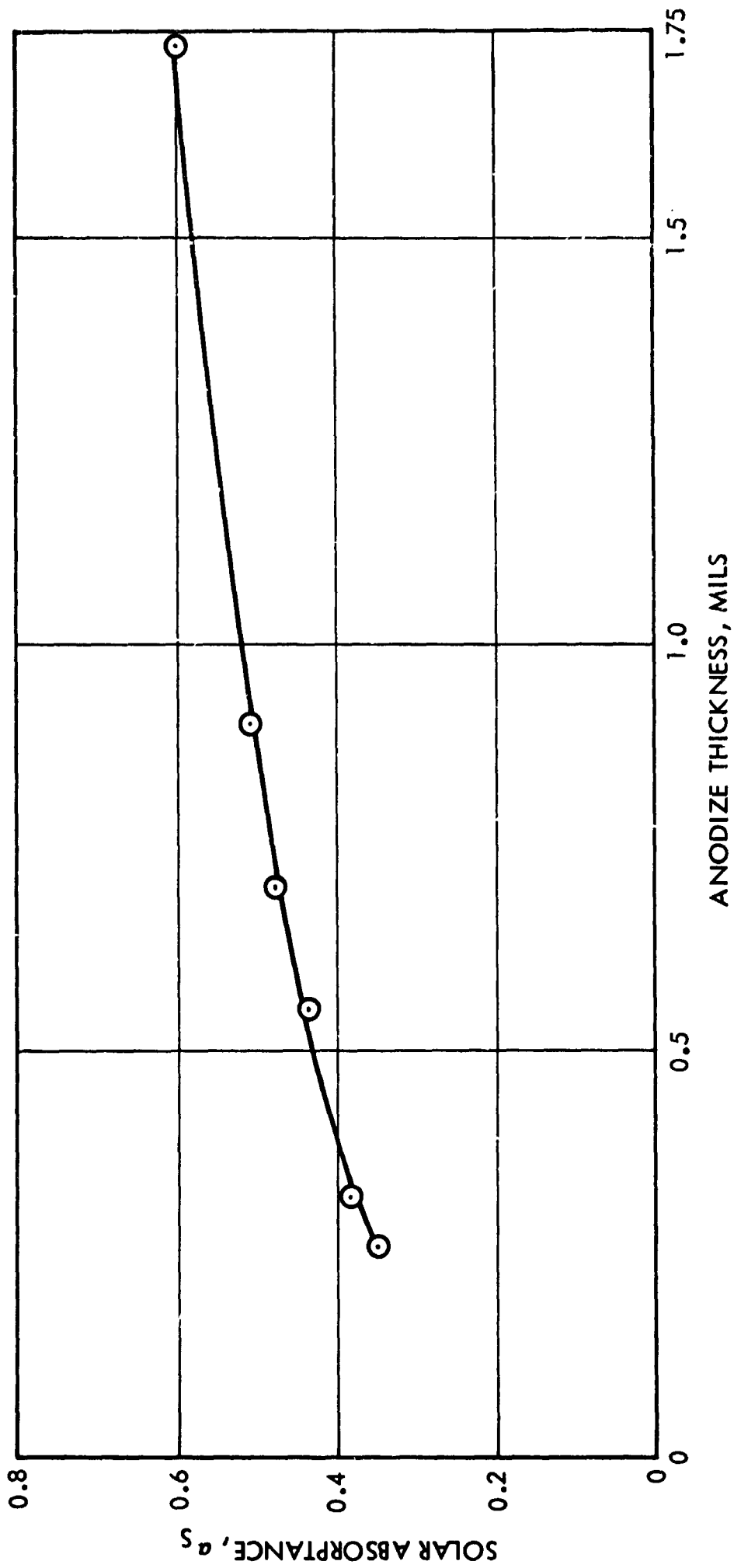


Figure III-15. 6061-T6 Aluminum Alloy, Anodized in a 17% (by weight) Sulfuric Acid Solution at $68^{\circ}\text{F} \pm 2^{\circ}\text{F}$, Current Density 15 Amperes per Square Foot, No Seal

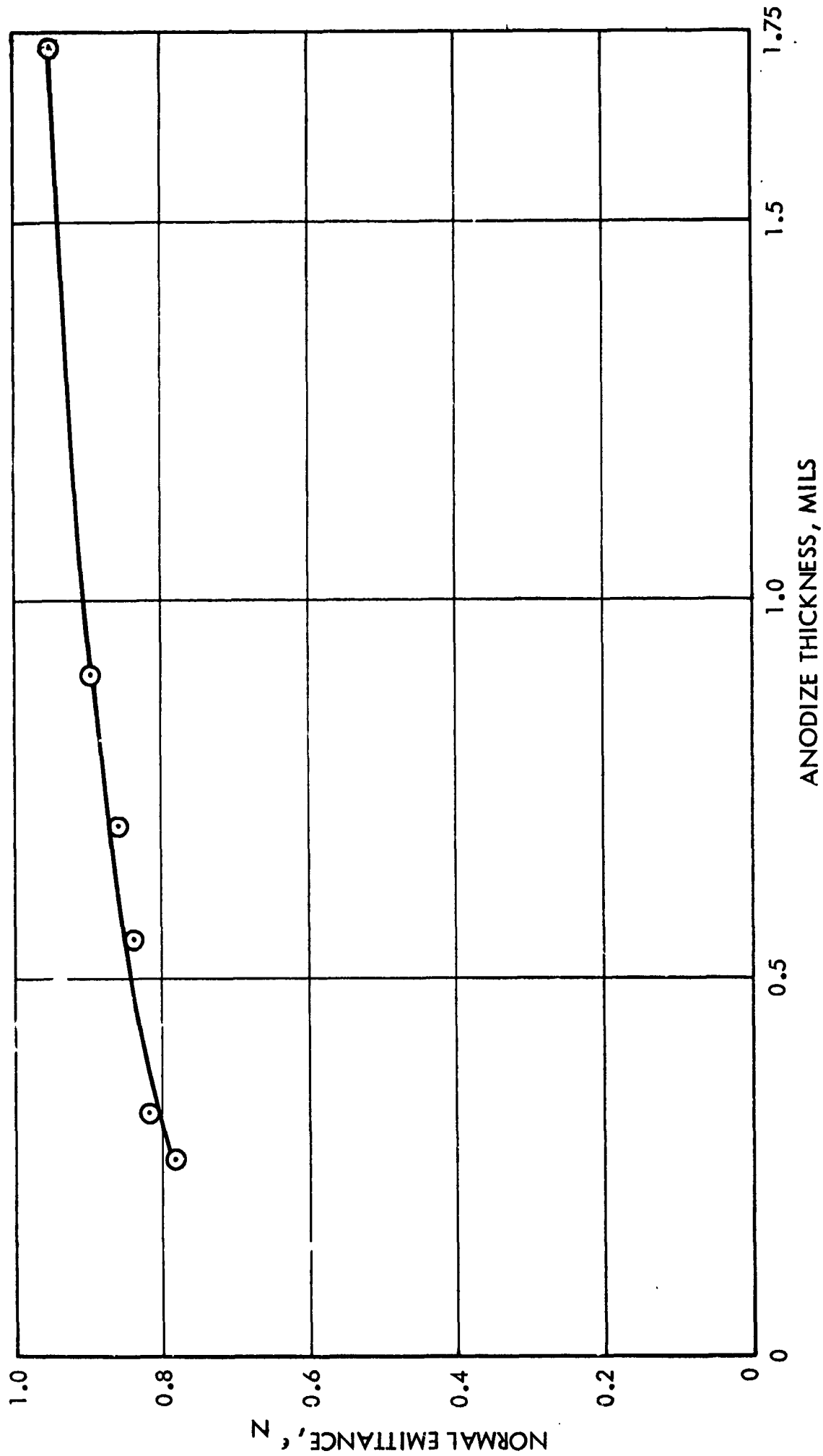


Figure III-16. 6061-T6 Aluminum Alloy, Anodized in a 17% (by weight) Sulfuric Acid Solution at $68^{\circ}\text{F} \pm 2^{\circ}\text{F}$, Current Density 15 Amperes per Square Foot, No Seal

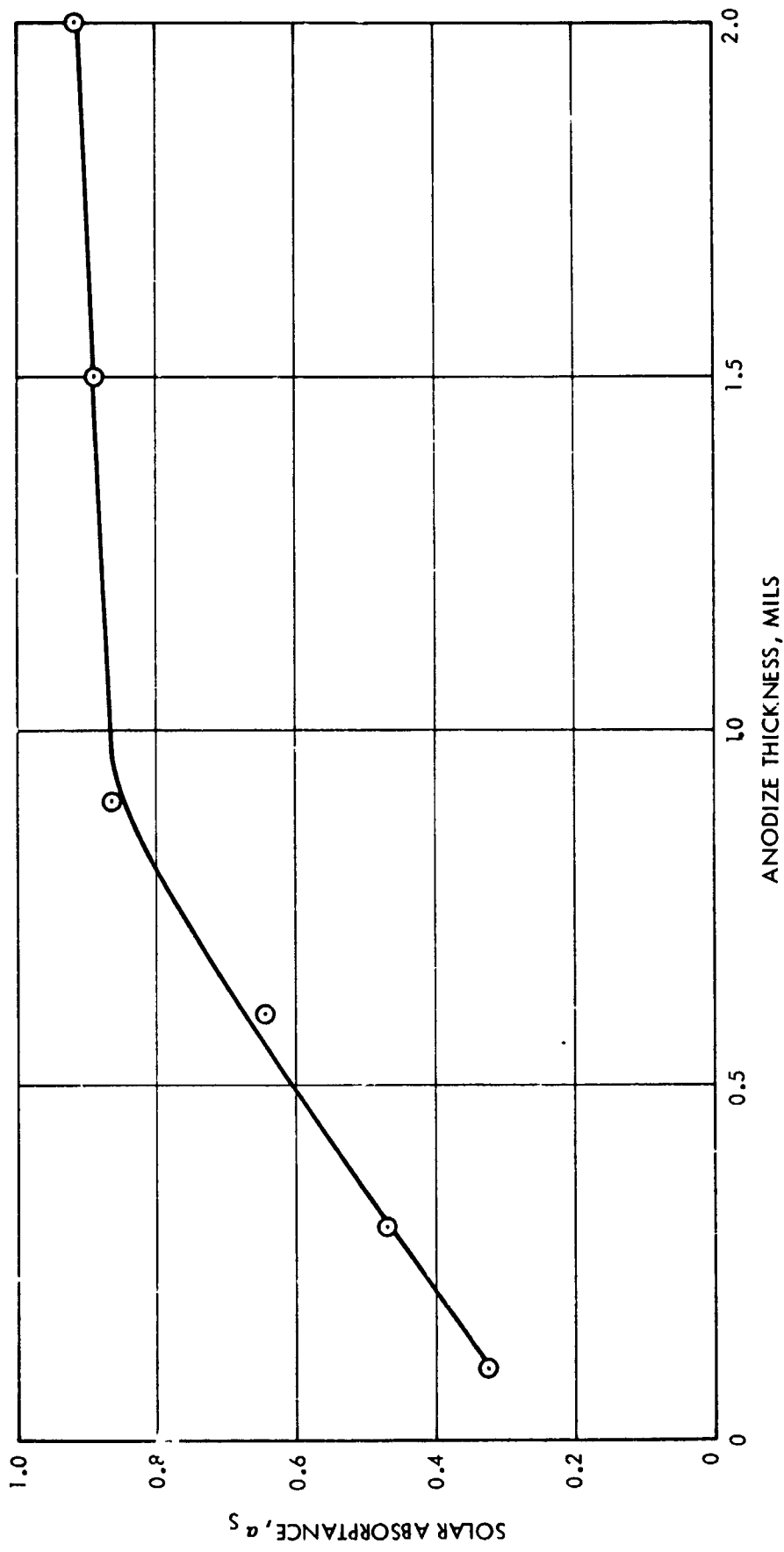


Figure III-17. 6061-T6 Aluminum Alloy, Anodized by "Martin Hard Coating" Standard Process

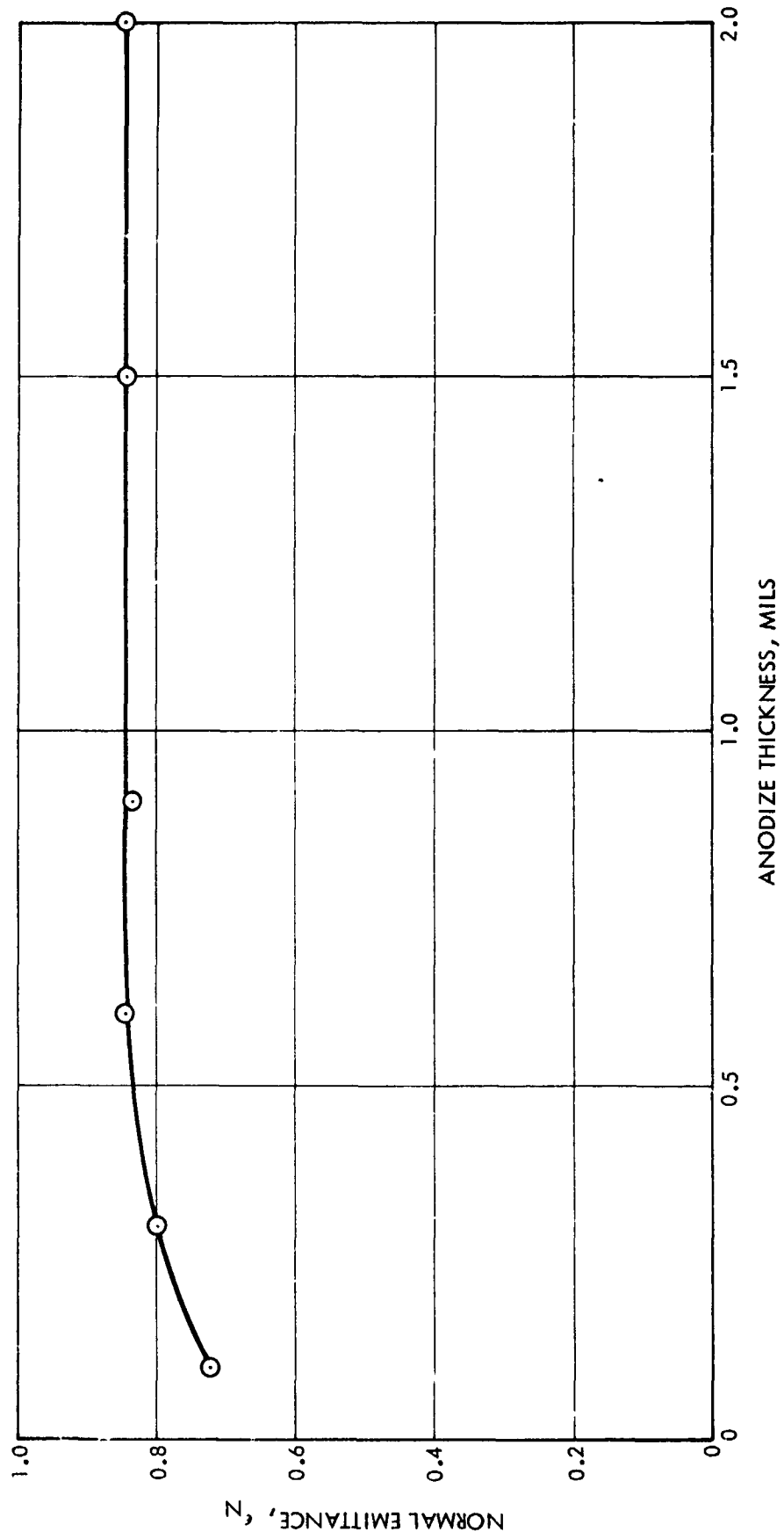


Figure III-18. 6061-T6 Aluminum Alloy, Anodized by "Martin Hard Coating" Standard Process

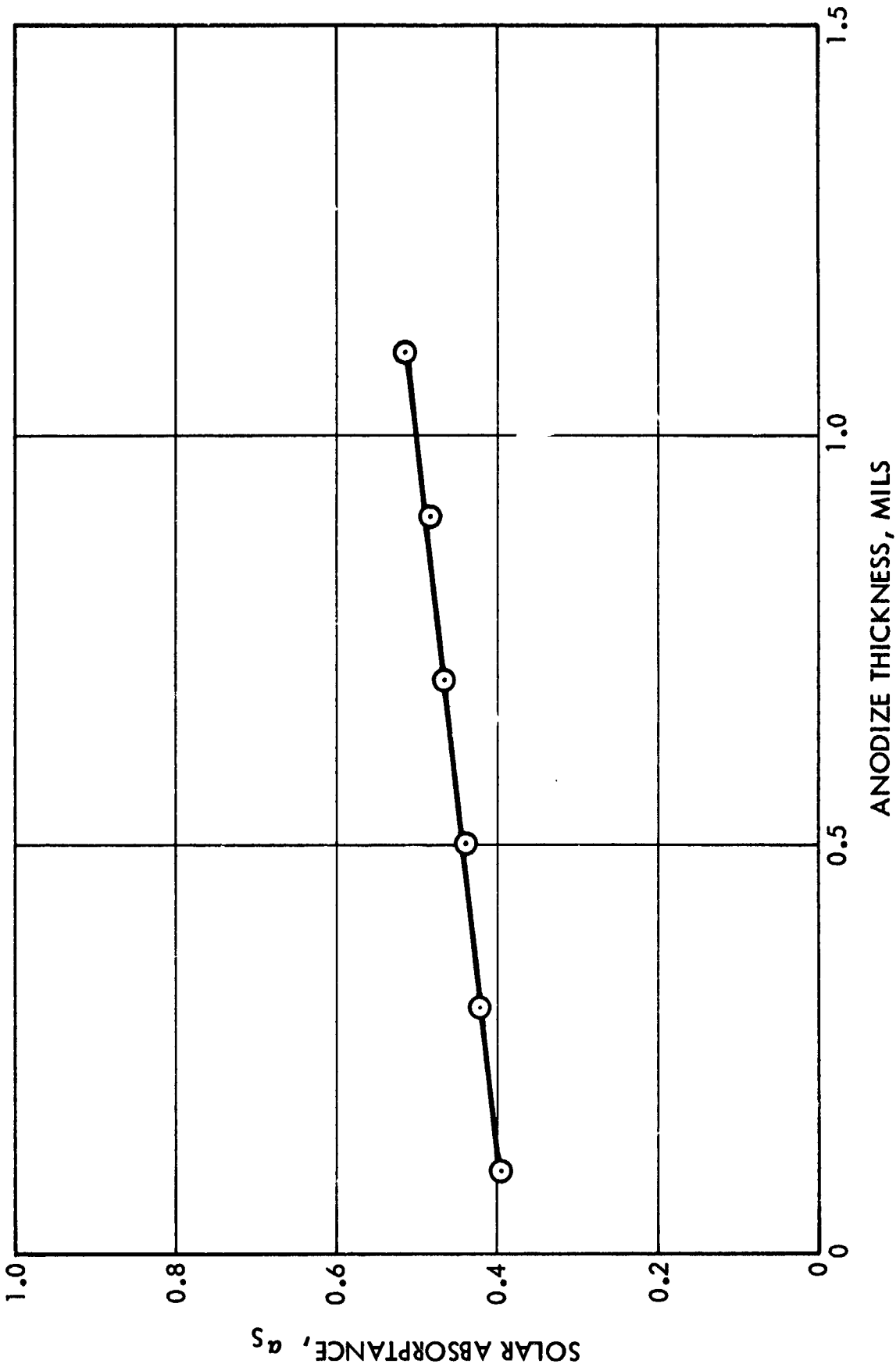


Figure III-19. 7075-T6 Aluminum Alloy, Anodized in a 17% (by weight) Sulfuric Acid Solution at $68^{\circ}\text{F} \pm 2^{\circ}\text{F}$, Current Density 15 Amperes per Square Foot, No Seal

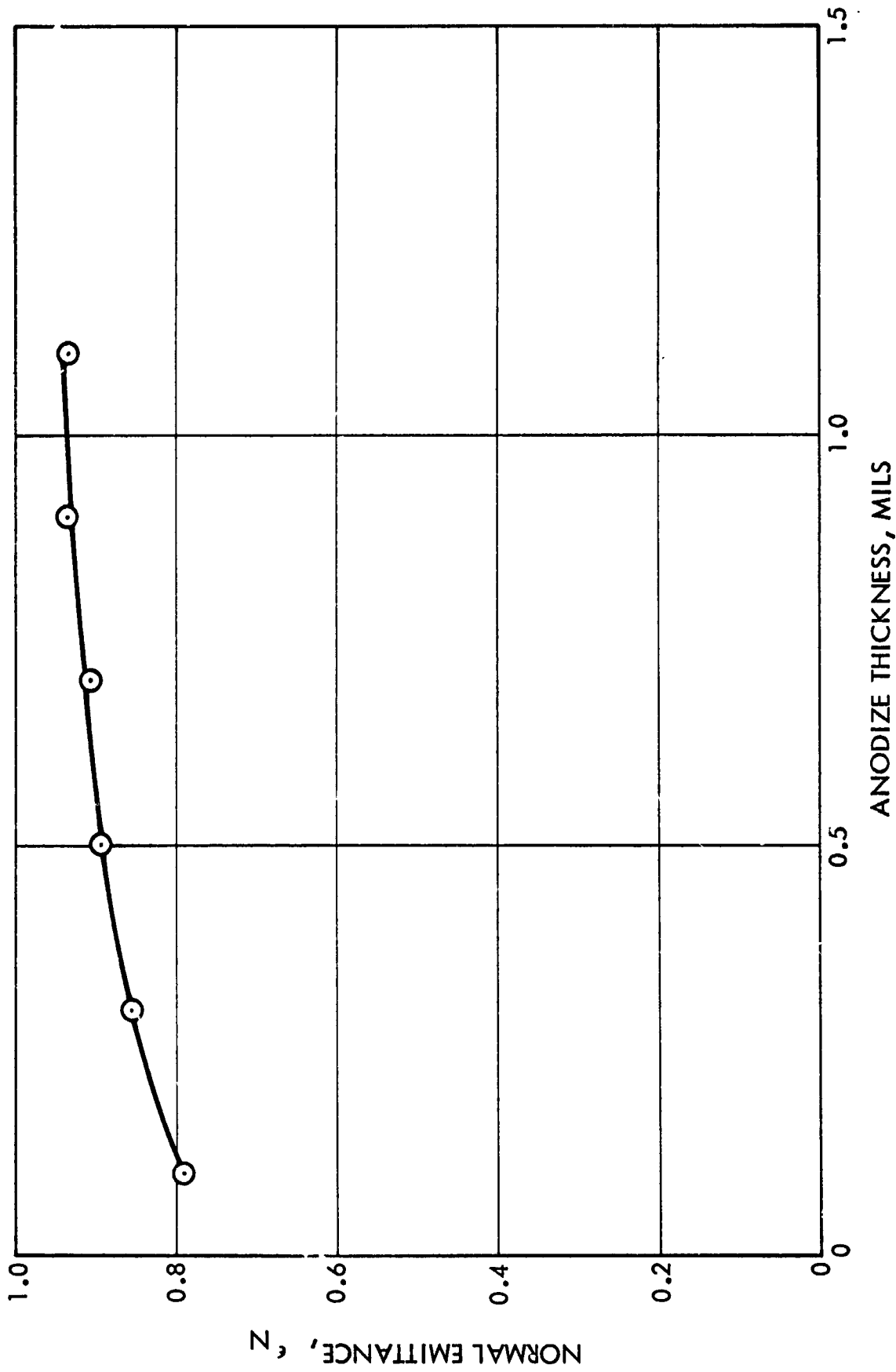


Figure III-20. 7075-T6 Aluminum Alloy, Anodized in a 17% (by weight) Sulfuric Acid Solution at $68^{\circ}\text{F} \pm 2^{\circ}\text{F}$, Current Density 15 Amperes per Square Foot, No Seal

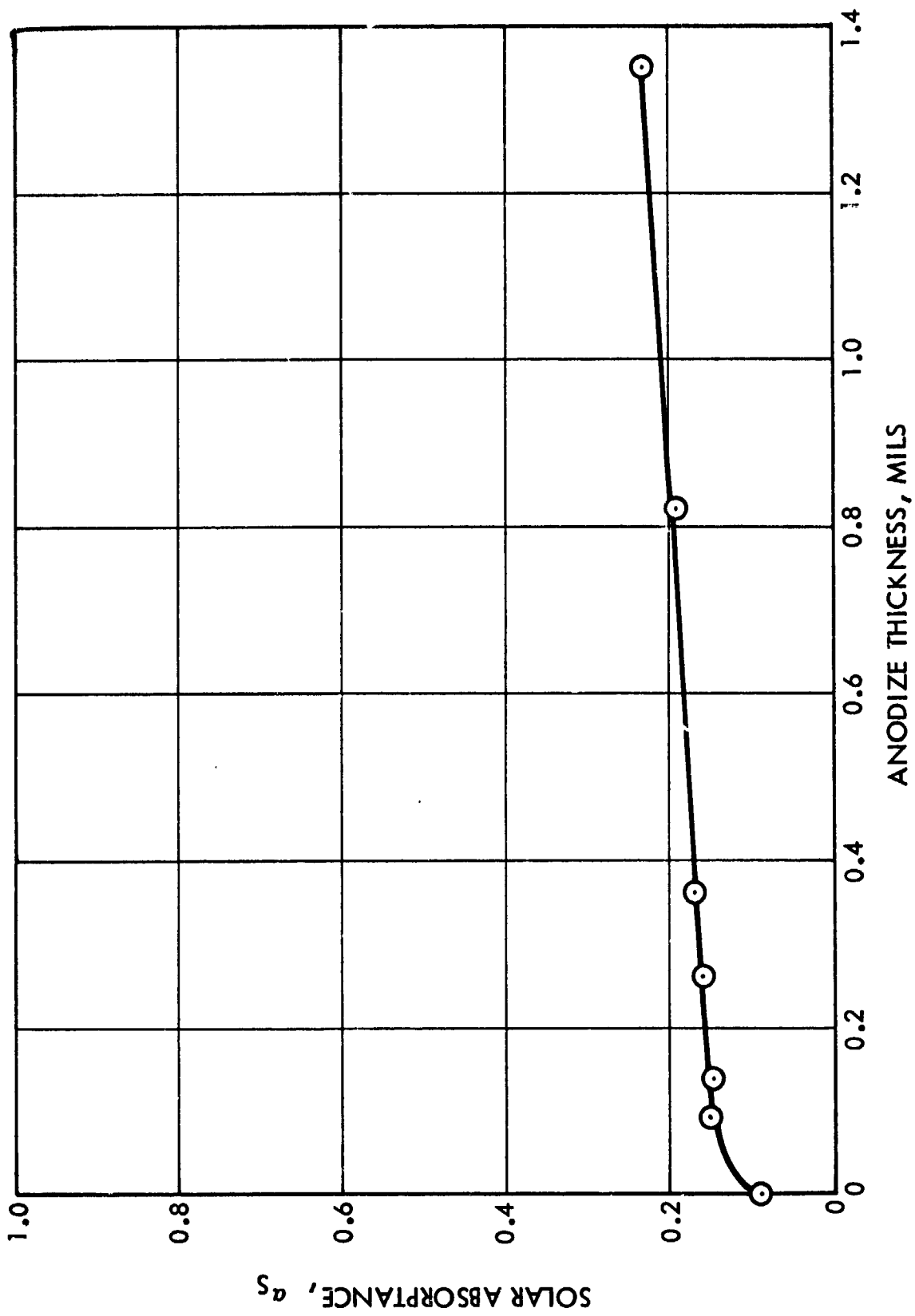


Figure III-21. 5557 Aluminum Alloy, Cleaned, Deoxidized and Bright Dipped in Greater Mountain Chemicals 800 Solution at 170°F for 10 Minutes, Anodized in a 15% (by weight) Sulfuric Acid Solution at 70°F ±2°F, Current Density 12 Amperes per Square Foot, No Seal

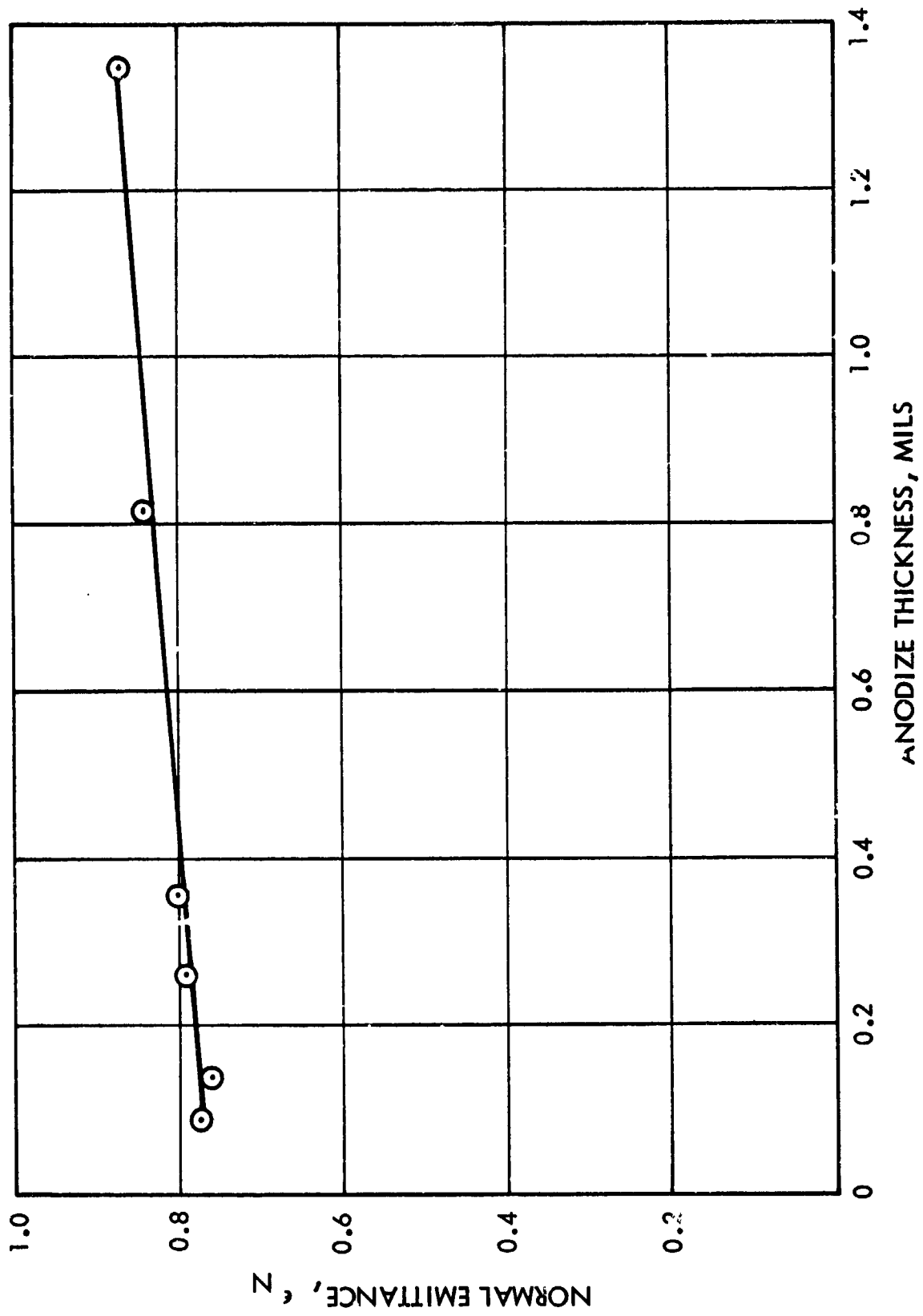


Figure III-22. 5557 Aluminum Alloy, Cleaned, Deoxidized, and Bright Dipped in Greater Mountain Chemicals 800 Solution and 170°F for 10 Minutes, Anodized in a 15% (by weight) Sulfuric Acid Solution at 70°F ±2°F, Current Density 12 Amperes per Square Foot, No Seal

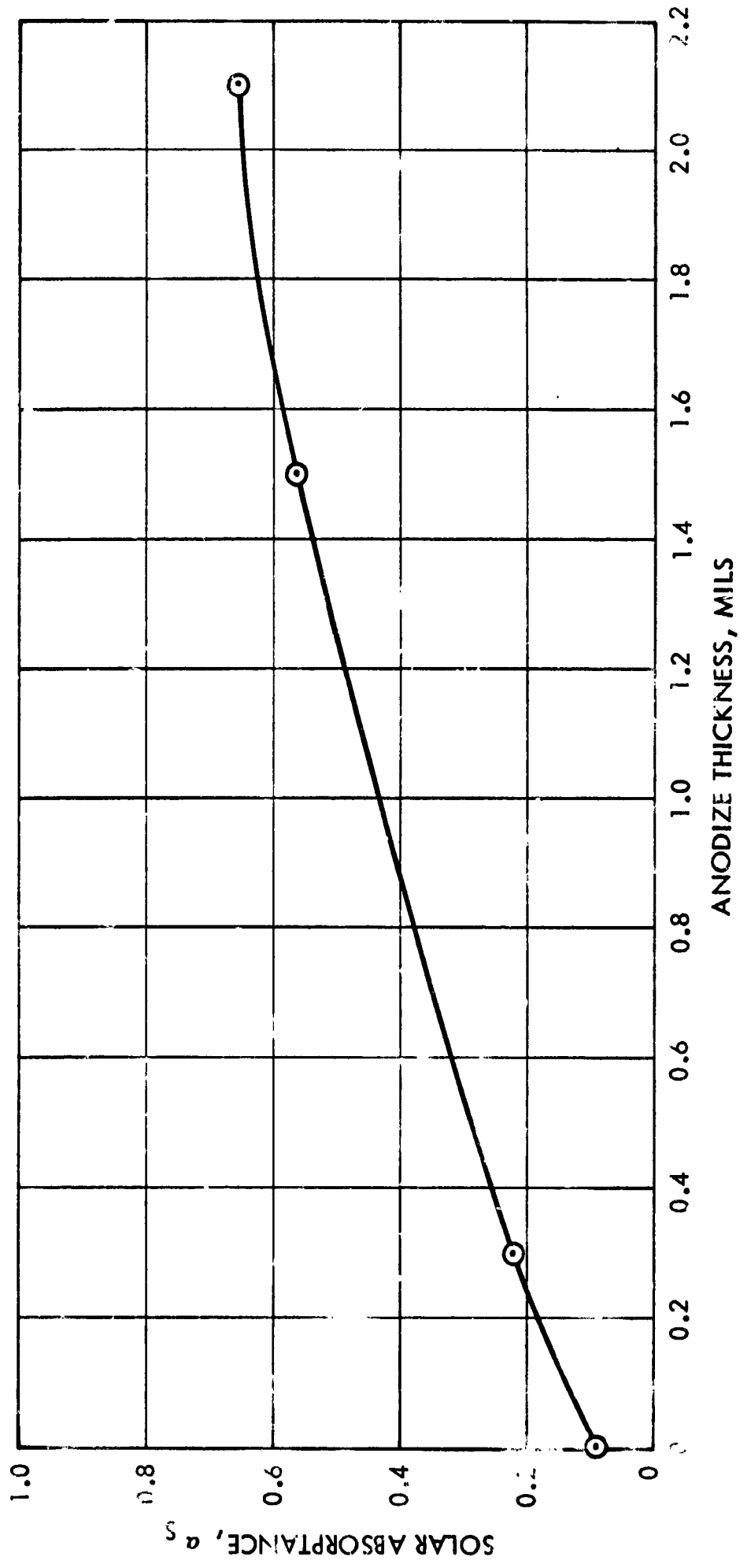


Figure III-23. 5557 Aluminum Alloy, Cleaned, Deoxidized, and Bright Dipped in Greater Mountain Chemicals 800 Solution at 170°F for 10 Minutes, Anodized in a 12% (by weight) Sulfuric Acid Solution at 25°F, Current Density 24 Amperes per Square Foot, No Seal

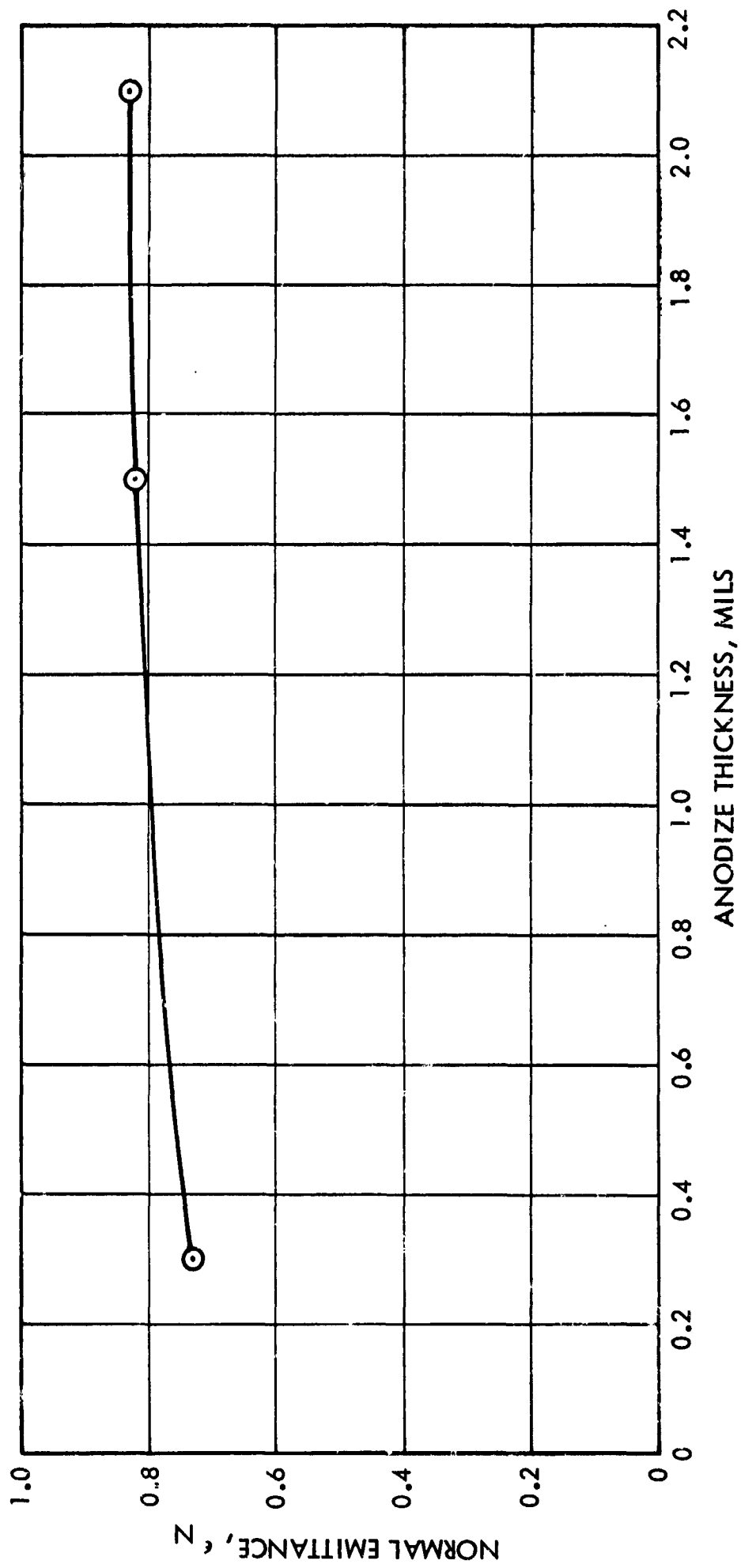


Figure III-24. 5557 Aluminum Alloy, Cleaned, Deoxidized, and Bright Dipped in Greater Mountain Chemicals 800 Solution at 170°F for 10 Minutes, Anodized in a 12% (by weight) Sulfuric Acid Solution at 25°F, Current Density 24 Amperes per Square Foot, No Seal

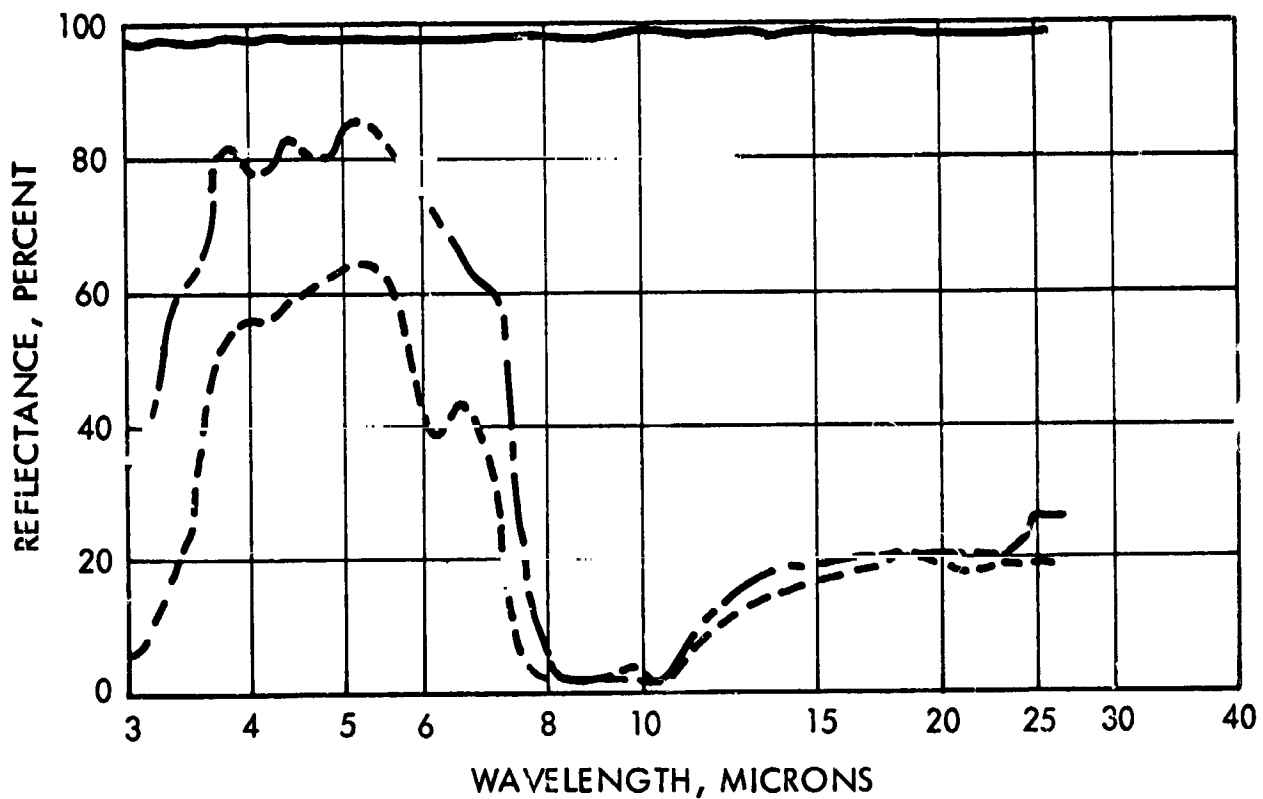
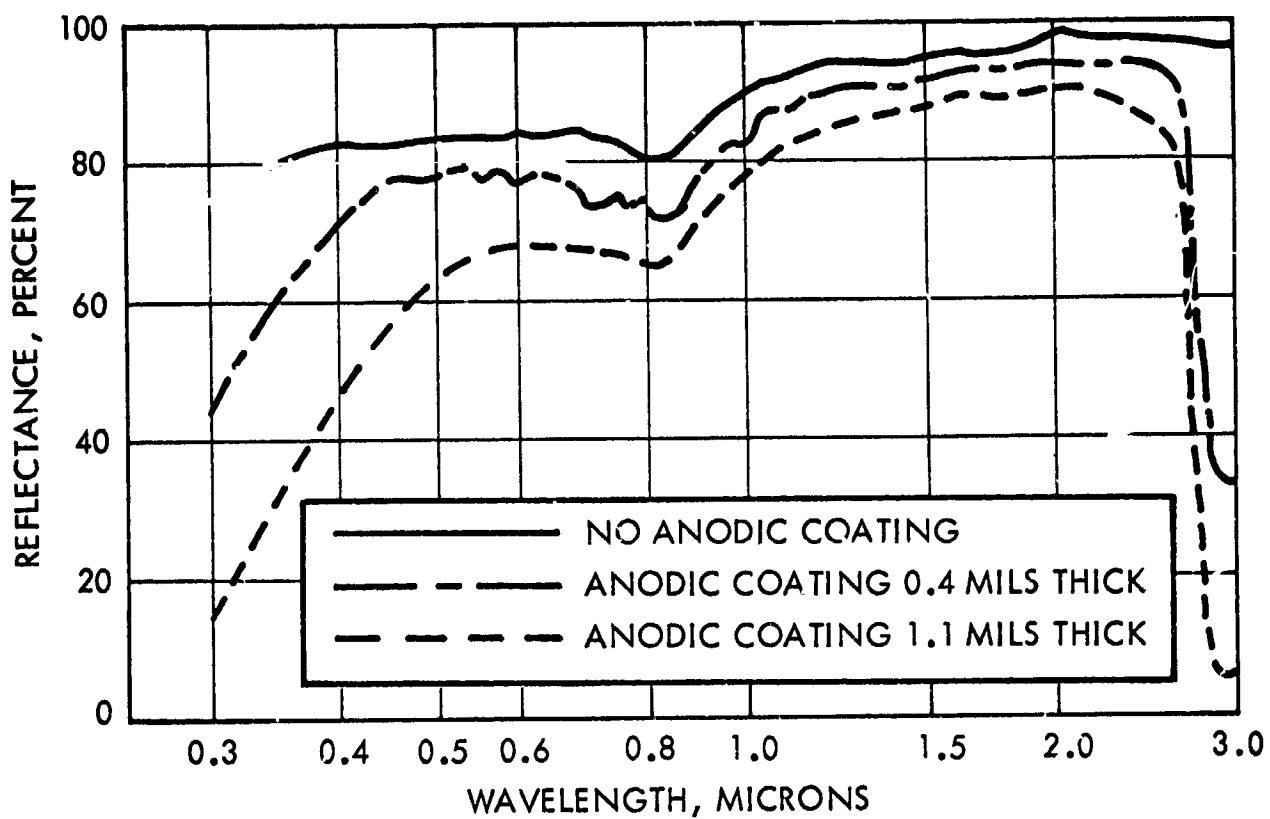


Figure III-25. 1170 Aluminum Alloy Clad on 2014, Bath A, No Seal (See Table III-1).

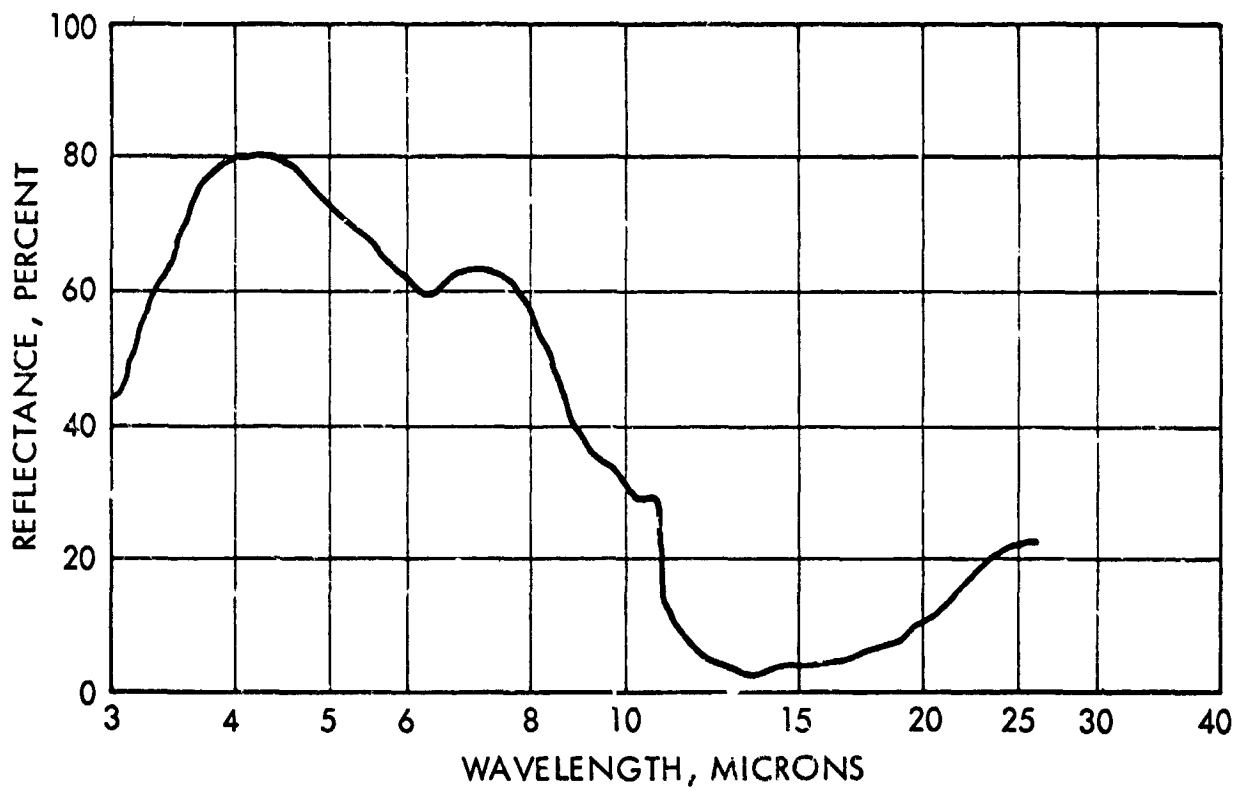
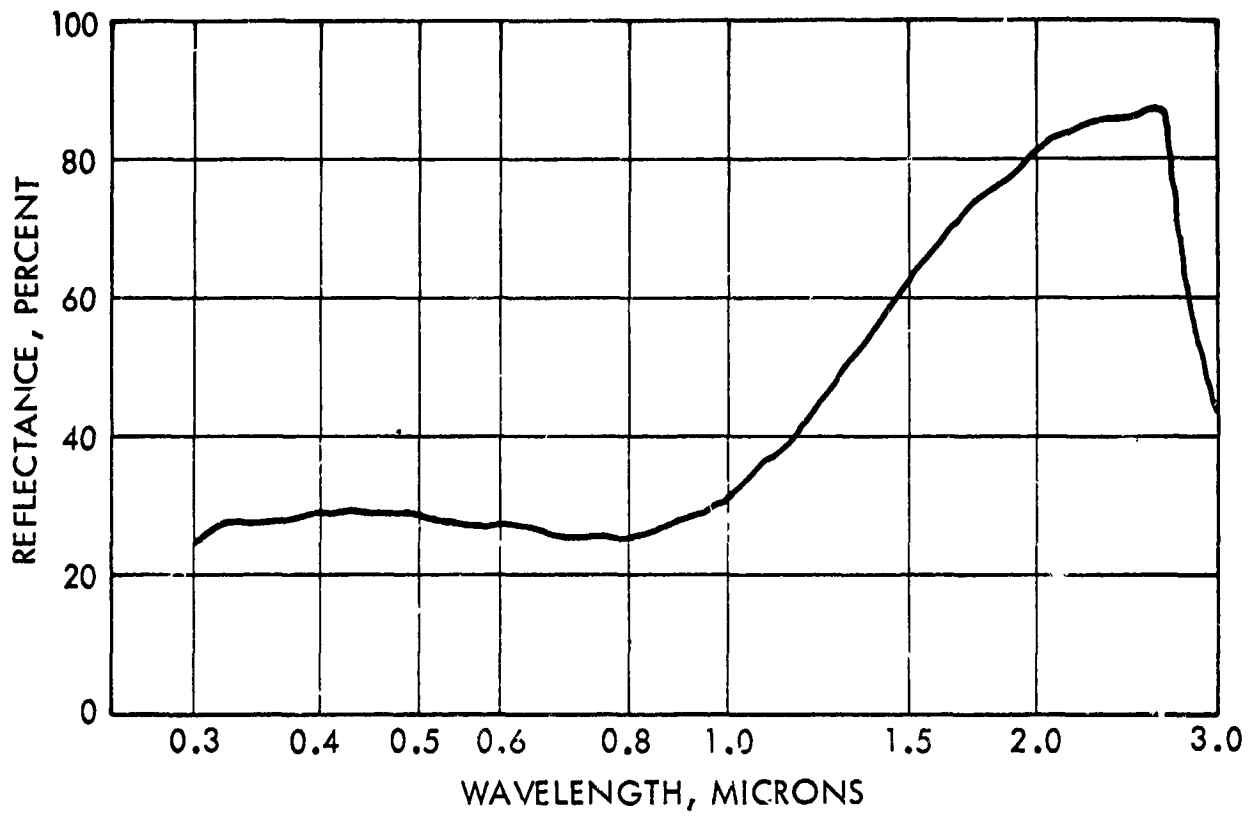


Figure III-26. 1199 Aluminum Foil, Sulfamic Acid Electrolyte
(Table III-4, Sample No. 4)

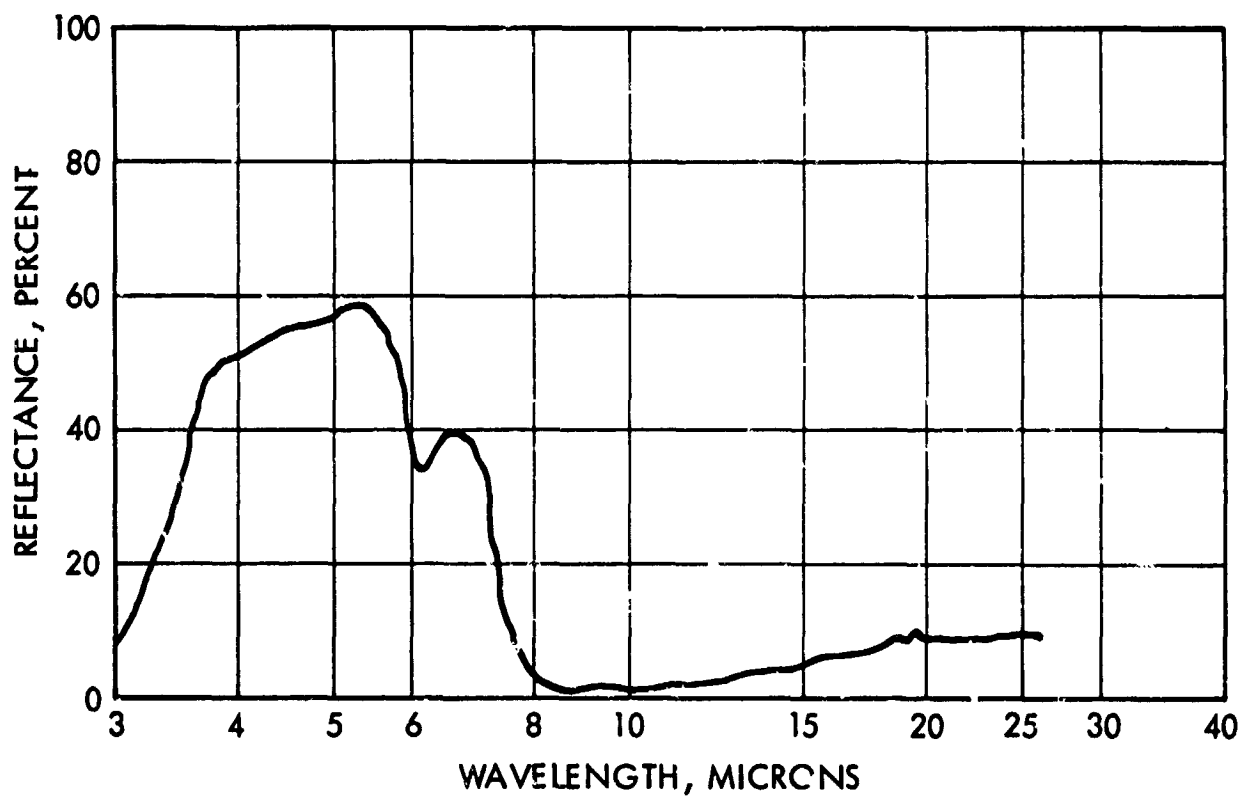
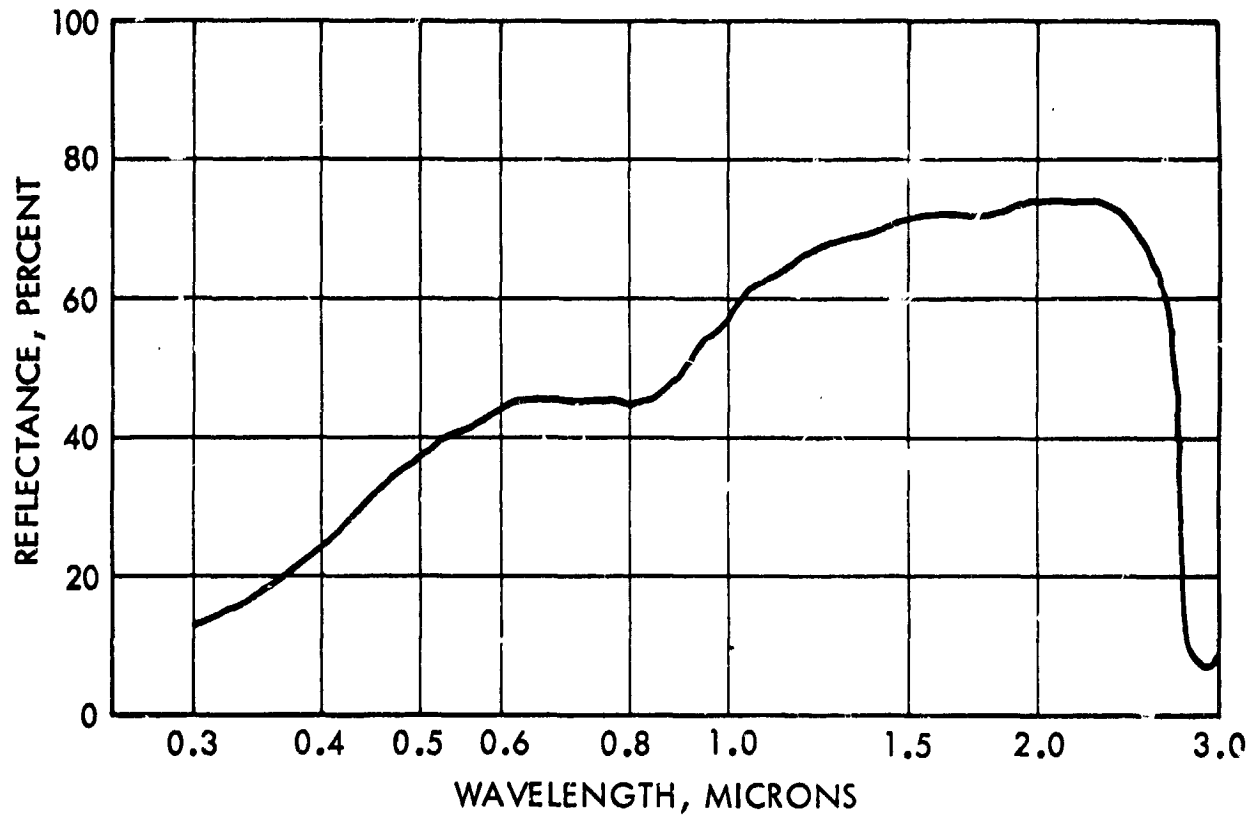


Figure III-27. 6061 Aluminum Alloy, Zirconium Oxide-Sulfuric Acid Electrolyte. (Table III-5, Sample No. 7)

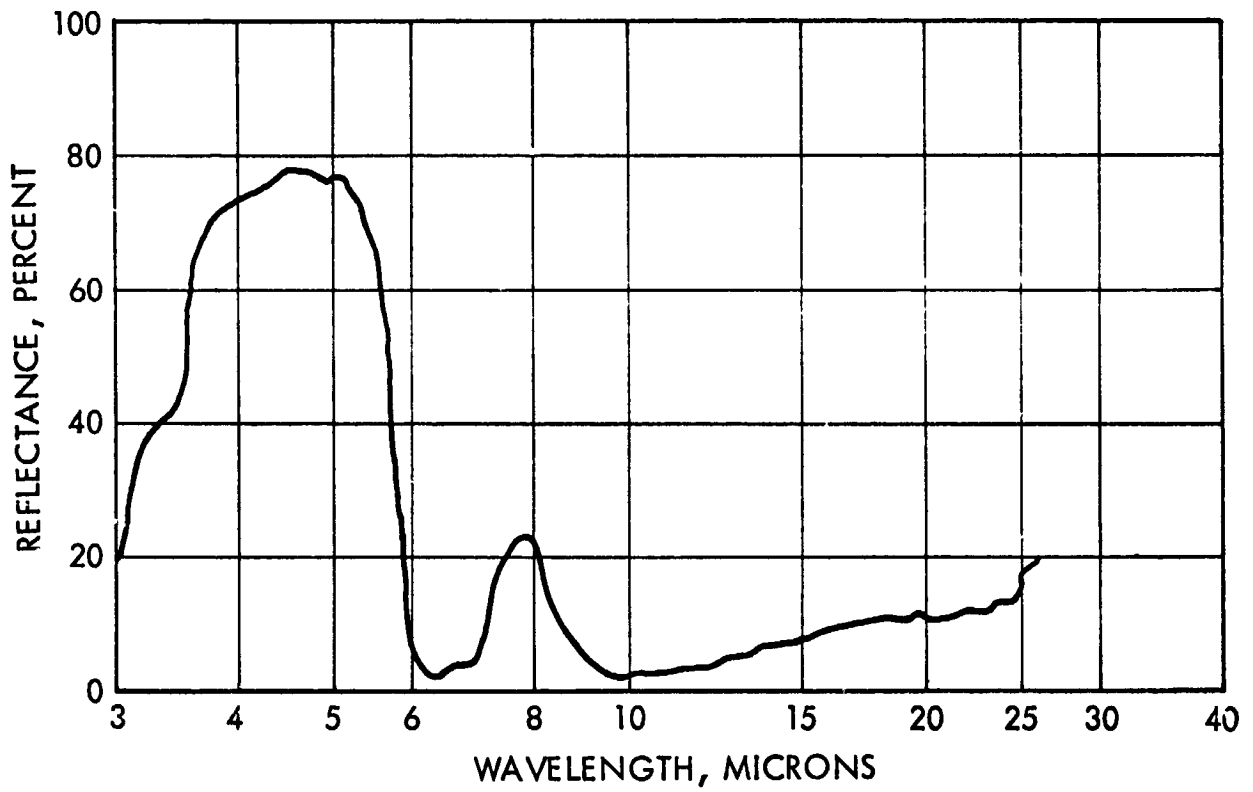
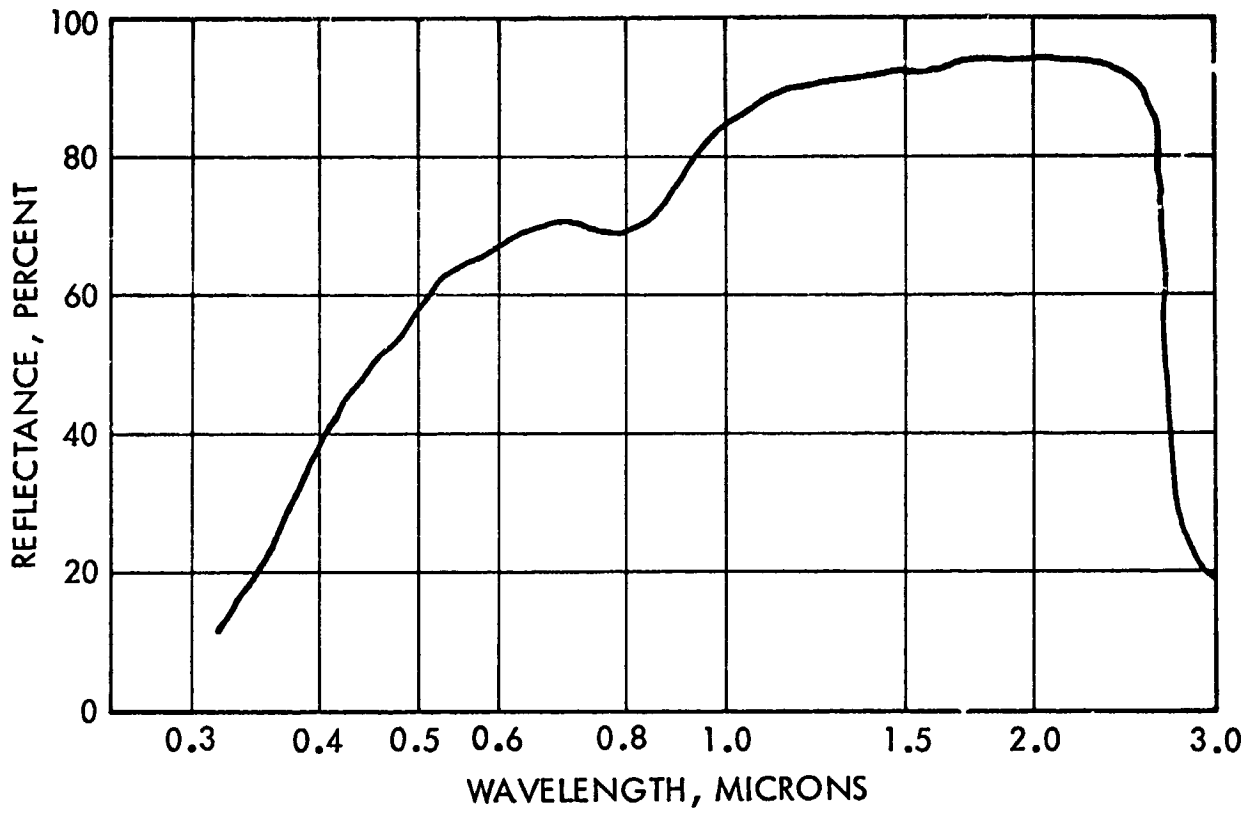


Figure III-28. 5557 Aluminum Alloy, Oxalic Acid Electrolyte
(Table III-7, Sample No. 13)

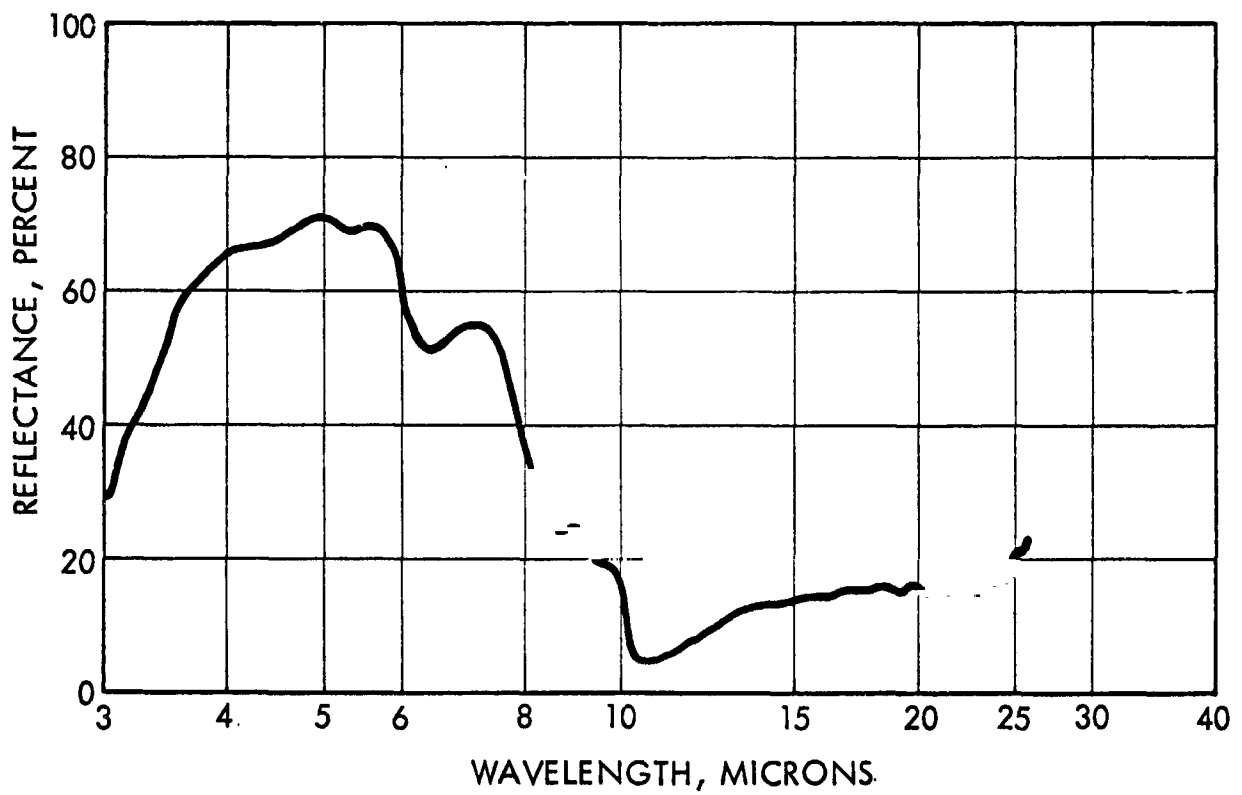
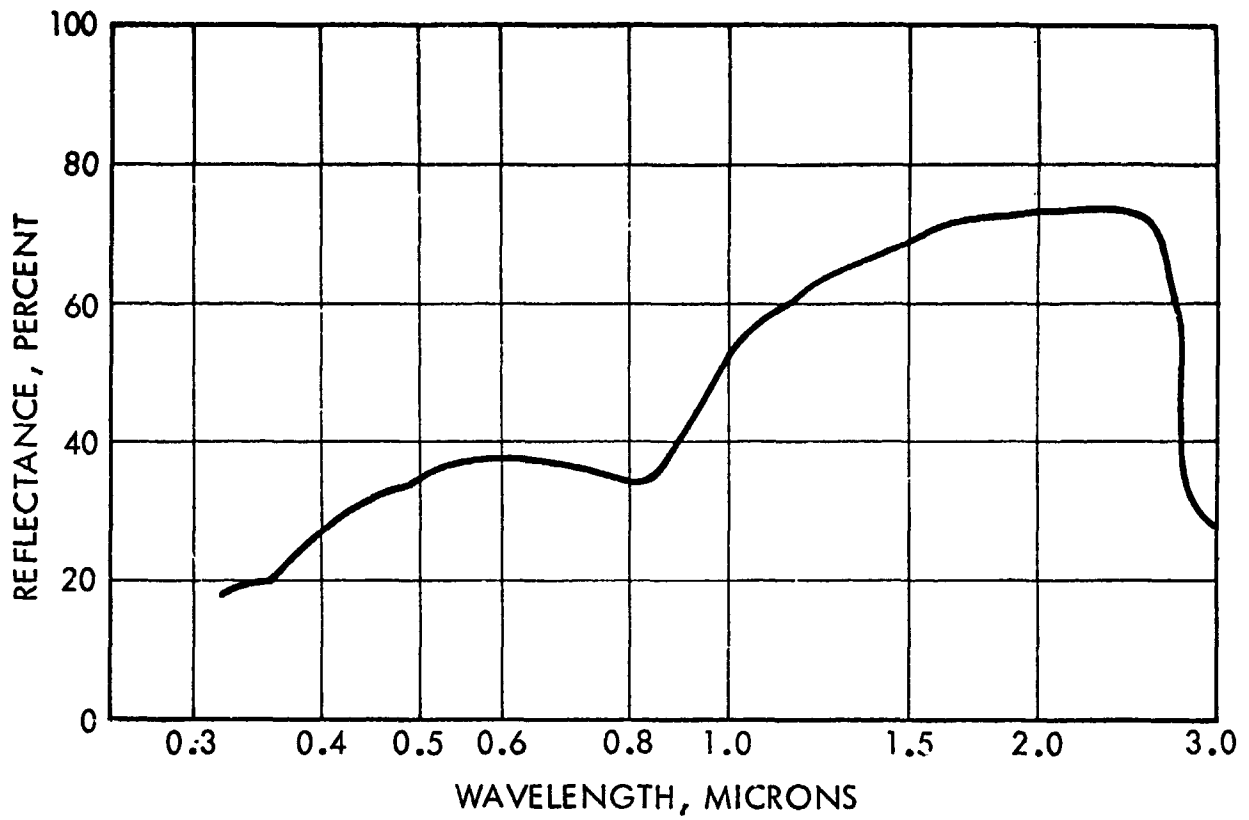


Figure III-29. 5557 Aluminum Alloy, Chromic Acid Electrolyte.
(Table III-8, Sample No. 12)

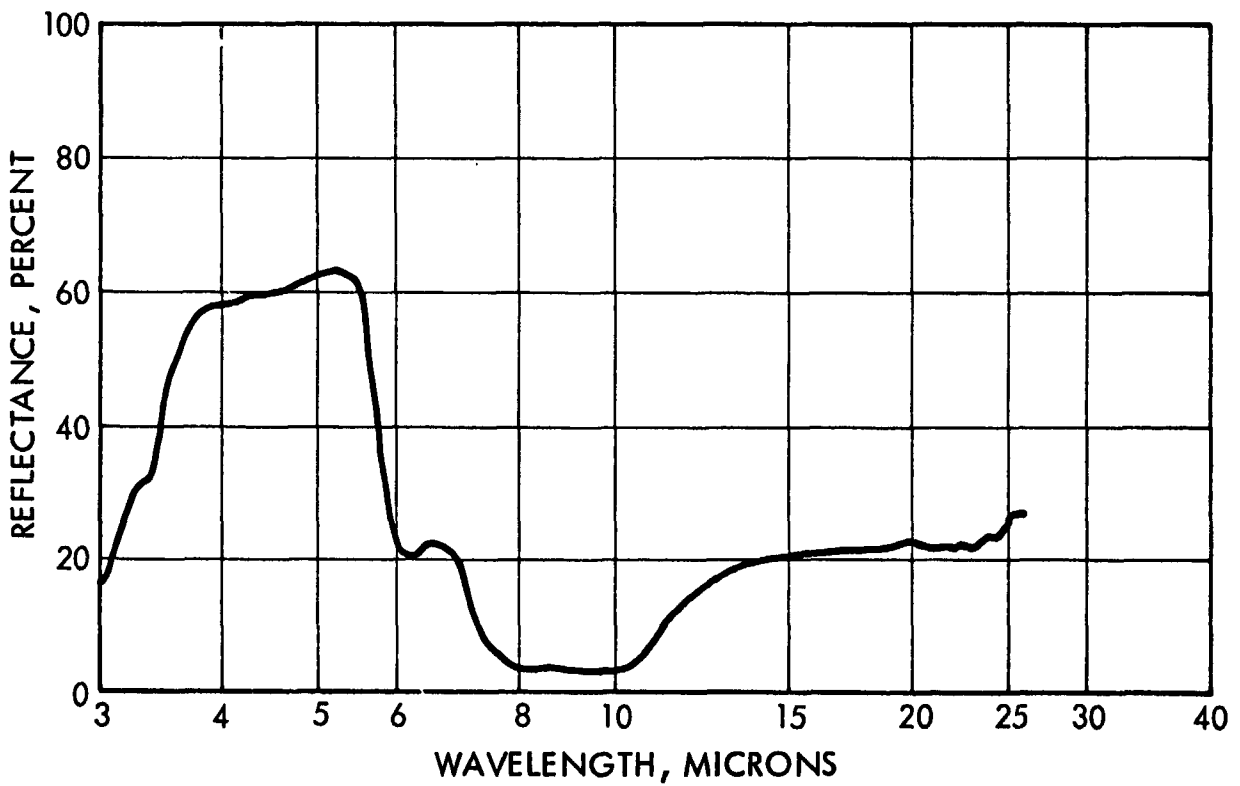
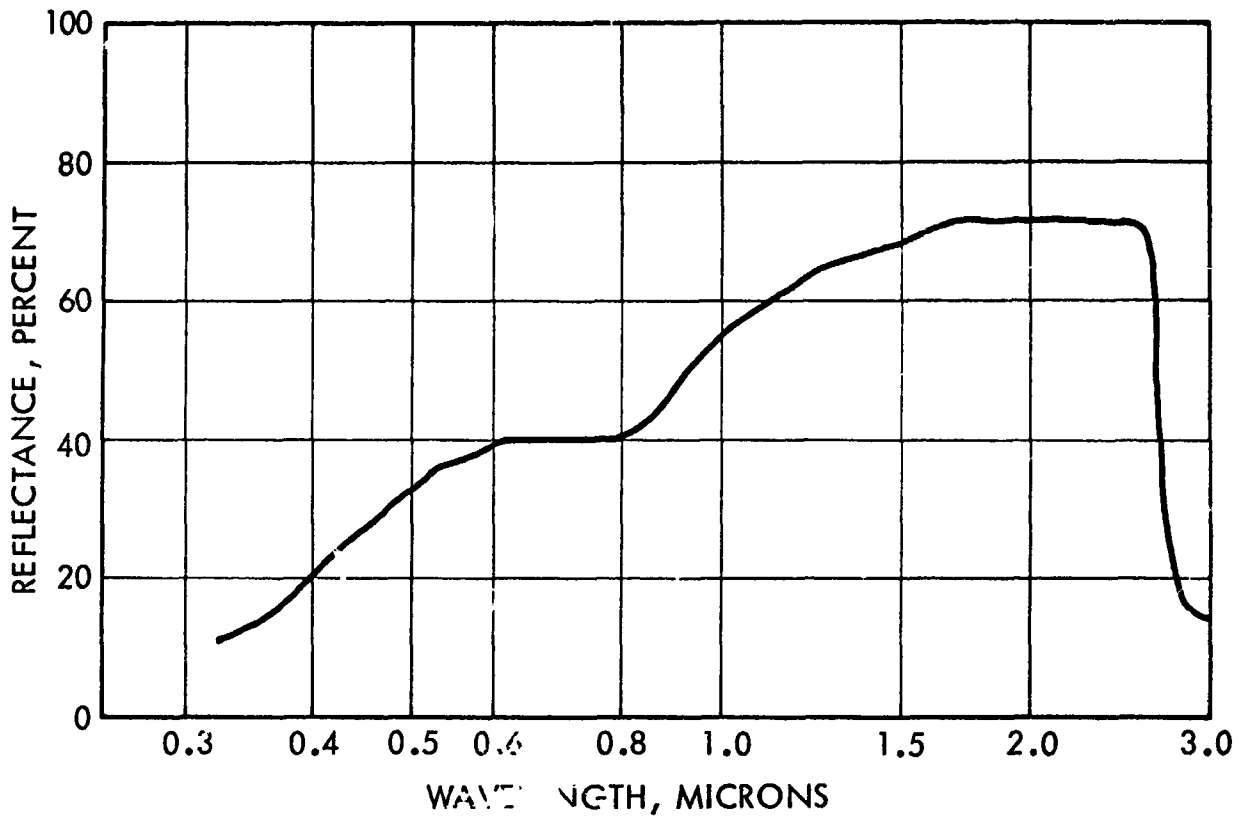


Figure III-30. 6061 Aluminum Alloy, Oxalic-Sulfuric Acid Electrolyte. (Table III-10, Sample No. 9)

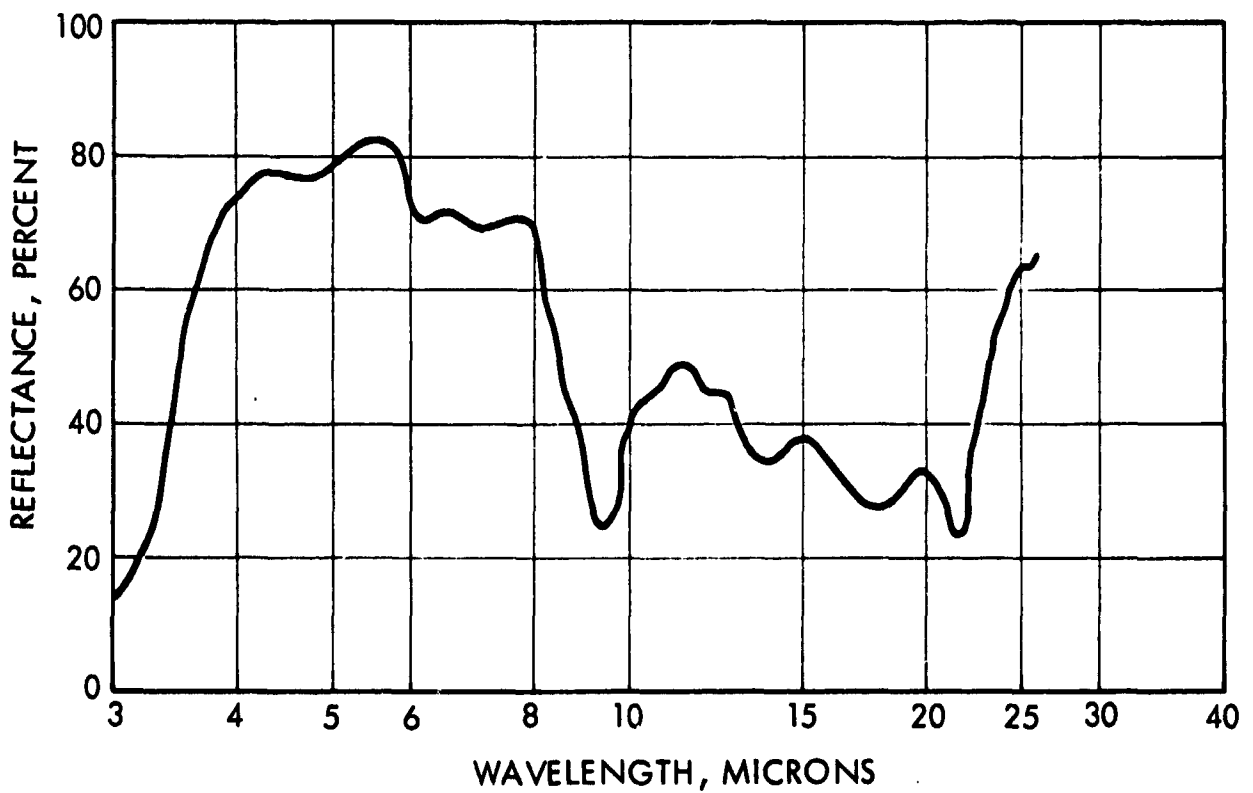
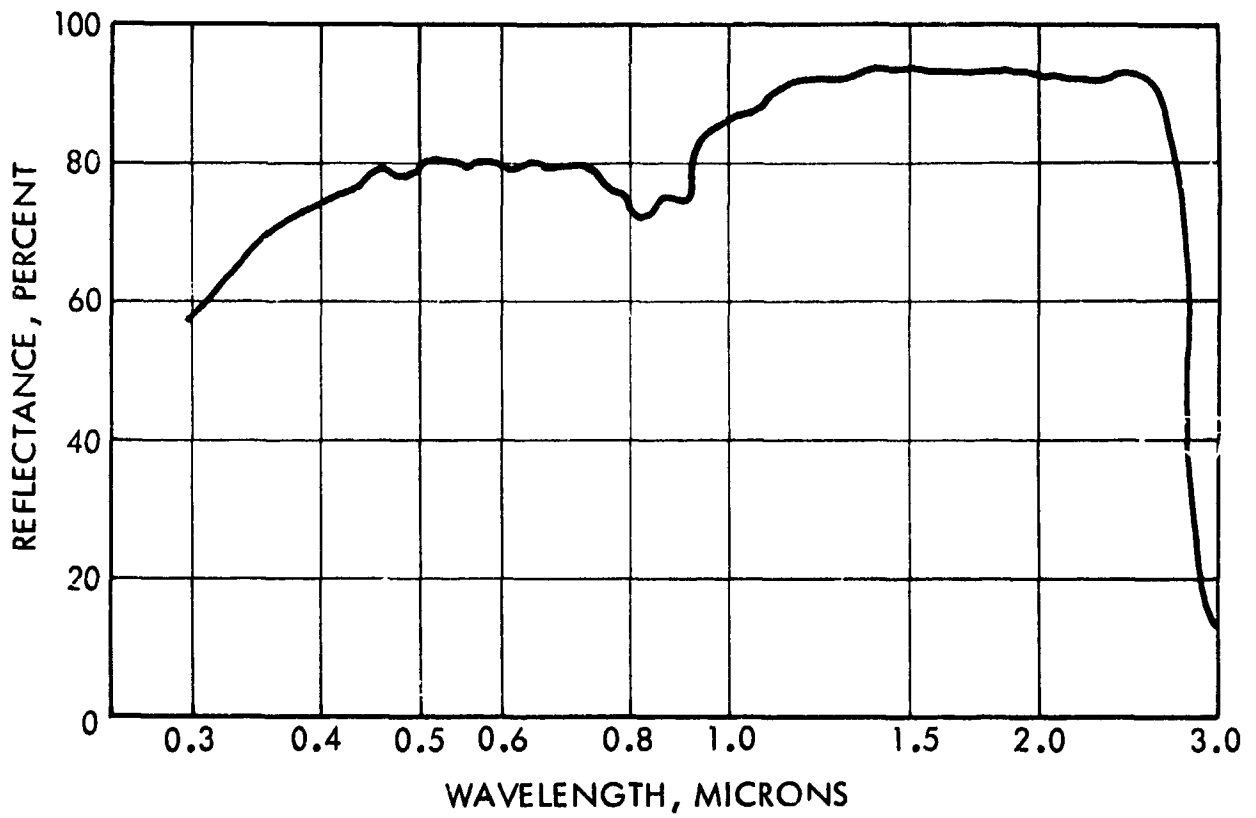


Figure III-31. 1199 Aluminum Foil, Distilled Water Electrolyte
(Table III-11 Sample No. 1)

IV. ULTRAVIOLET AND VACUUM DEGRADATION STUDIES

A. INTRODUCTION

This particular phase of the study is currently one of the most important technical problems in thermal control of spacecraft. When the study was first proposed and until approximately the mid-point of the original contract period, the significance of the ultraviolet and vacuum environmental factors upon thermal control materials was not fully recognized in the industry. Relative to this program, the rude awakening occurred with the first degraded sample of sulfuric anodized aluminum. The magnitude of the experimental problem which was found can be illustrated by Figure IV-1. In this photograph, eight samples of hard sulfuric anodized type 1199 aluminum are shown after the exposures indicated. All samples came from the same anodized sheet and the localized degradation has yet to be properly explained. To further complicate the problem, the undarkened areas became brighter (solar absorptance decreased).

The obvious experimental problems of vacuum and ultraviolet degradation testing have been discussed in the Progress Report dated 16 April 1962. The experience which has been gained since then has not reduced the problem. However, there are several conclusions which can be drawn from the results which have been obtained to date for anodized aluminum:

- 1) Accelerated testing (high irradiation level) is not valid; i. e., equal exposures but different irradiation levels do not yield equal degradation.
- 2) Correlation of degradation results is not easily performed with an engineering material which is subject to a large number of process variables, e. g., anodized aluminum.
- 3) The physical basis for correlating degradation results on a logarithmic or semi-logarithmic basis does not rest upon a valid foundation. The convenience of a logarithmic scale is the primary reason.
- 4) The wavelength region to be used for correlating results is quite arbitrary and will depend upon the wavelength sensitivity of the material.
- 5) The experimental results to date are quite preliminary and insufficient for any specific conclusions to be made, i. e., only general statements are possible.

The fact that many anodized aluminum materials darken (degrade) under vacuum and ultraviolet conditions has been the basis for many investigators abandoning the development of this material for space applications. However, some anodized samples did not darken, others lightened, and others appeared to reach an "equilibrium" condition without further change. To a designer of spacecraft, either the first or last condition is adequate and in many cases, a material that decreases in solar absorptance (lightens) is very useful. For these reasons, anodized aluminum is not believed to be a useless material. The following results are presented with this belief as a basis for the accompanying discussion.

B. EXPERIMENTAL APPARATUS

In anticipation of this laboratory program and for support of several major spacecraft programs, a vacuum-ultraviolet testing apparatus had just been designed when the study began. This equipment was constructed early in contract period and placed in operation at the beginning of 1962. The apparatus consisted of 12 Vac-Ion pumps with attached sample holders arranged about a central energy source (see Figure IV-2). The distance between the sample and source was fixed at 130 mm and with an Osram HBO-200 mercury arc source, a total irradiation power level of 700 watts/m^2 was obtained. This gave an irradiation very nearly equal to the energy of the sun at wavelengths less than 4000 \AA (125 watts/m^2) since 20 percent of the energy of the HBO-200 lamp is at wavelengths less than 4000 \AA . However, accelerated testing was not possible and an alternative design was developed.

The new design consists of a central source with 4 Vac-Ion pumps placed symmetrically about it. Each pump with attached sample chamber is mounted on a movable base and the distance between the source and sample is continuously variable from 75 to 215 mm (Figure IV-3). Two sources have been used, a G.E. B-H6 mercury arc (900 w) and a Hanovia Xenon arc (2.2 kw). With the B-H6 source, the total irradiation power level can be varied from 0.7 to 6 kw/m^2 ; the data supplied by the manufacturer and verified in the laboratory indicate 30 percent of the total energy of the lamp is at wavelengths less than 4000 \AA . The Hanovia Xenon lamp provides a total irradiation power level of 1.5 to 22 kw/m^2

but only five percent of this energy is at wavelengths less than 4000 Å. Table IV-1 gives a comparison of the energies emitted by the B-H6 and xenon sources compared to the Johnson solar spectrum.

Table IV-1. Comparison of Ultraviolet Energies of the Sun and Degradation Sources

| <u>Source</u> | <u>Fraction of Total Energy which is Below 0.4 μ</u> | <u>Fraction of Total Energy which is Below 0.3 μ</u> |
|--|--|--|
| Sun Outside Earth's Atmosphere | 0.10 | 0.02 |
| General Electric B-H6 | 0.30 | 0.10 |
| Hanovia 2.2 kw Xenon | 0.05 | 0.015 |
| <u>Output of B-H6</u> <u>Output of Sun</u> | 3.0 | 5.0 |
| <u>Output of Xenon</u> <u>Output of Sun</u> | 0.5 | 0.7 |

The energy emitted by the two sources is monitored with a thermopile detector in total energy and within wavelength bands by filter spectroscopy. The filters used and the wavelengths passed by each filter are given in Table IV-2. The B-H6 mercury source is subject to a more rapid degradation in output than the Xenon arc and requires greater attention by the operator to insure proper experimental conditions. The Xenon source is symmetrical in the horizontal plane and the angular positions of the samples and monitor are not important. The B-H6 mercury source is, however, not symmetrical in the horizontal plane and the pumps were positioned 30 degrees from the normal to the axis of the source. The B-H6 was monitored normal to the source axis and from experimental measurements, a correction was found for the difference in position of monitor and sample. As indicated in Figure IV-3, a water cooled housing was placed about each lamp. Since this will affect the energy incident upon the sample, each source-housing system was measured as a function of sample-source distance to determine the actual energy incident upon the samples.

Table IV-2. Filter Transmission Bands for Ultraviolet Energy Monitoring

| <u>Filter</u> | <u>Transmission</u> | |
|--------------------------|-----------------------------------|-----------------------------------|
| | <u>λ Lower</u> | <u>λ Upper</u> |
| Quartz | 0.22 | > 2.0 microns |
| 1 x 3 (Glass) | 0.310 | > 2.0 |
| (7 - 54) | 0.25 | 0.39** |
| - [(7 - 54) + (2 - 63)]* | | |
| (4 - 71) | 0.36 | 0.59** |
| - [(4 - 71) + (2 - 63)]* | | |
| (2 - 63) | 0.59 | > 2.0 |
| Solar Cell | 0.44 | > 2.0 |
| Filter Cover Glass | | |

* Corning Glass filter designations

** Transmission band obtained by difference of two measurements

The nominal life of the B-H6 mercury source is 200 hours but this is a direct function of the number of lamp starts. One lamp lasted 551 hours but the output had dropped to 50 percent of its original value. The B-H6 source normally starts at about 120 percent of nominal and decreases linearly at the approximate rate of 0.2 percent per hour for the first 200 hours. A constant voltage source is also required to eliminate fluctuations in lamp output resulting from line voltage changes. The Xenon source is more subject to control since the lamp current can be varied to offset lamp deterioration. Operation overnight was necessary for the long exposure times and to insure a true knowledge of the time, running time meters were installed in both source systems.

The two different energy sources were selected in an attempt to determine the effect of spectral energy distribution upon measured sample degradation. The B-H6 source is a high pressure mercury arc and has a spectrum with the characteristic mercury emission lines (broadened). The xenon source does not have strong emission lines in the ultraviolet and is

more representative of the continuum of the solar spectrum. The two source spectra as given by the source manufacturers are shown in Figures IV-4 and IV-5 and are compared in cumulative energy to the sun in Figure IV-6.

The important engineering measure of degradation is the change in solar absorptance; the Beckman DK-2A reflectometer was to be used for this purpose as discussed in the Instrumentation Section. The errors of the Beckman integrating sphere are quite significant and as a result, many spectral reflectance measurements had to be repeated with the new sphere. All of the results reported for the 0.25 to 2.0 μ wavelength range were measured with the new integrating attachment for the Beckman instrument. When required, the spectral reflectance between 2.0 and 25 microns was measured with the heated cavity reflectometer.

C. EXPERIMENTAL PROCEDURE

The sample materials were prepared by a commercial vendor (Anadite Corp., South Gate, Calif.). Sample discs, 19 mm in diameter were punched from the anodized sheets, marked, measured for spectral reflectance between 0.25 and 2.0 microns, and individually boxed. When a sample was to be tested, it was placed in the sample chamber on a water cooled holder and the chamber evacuated to 10^{-2} torr with a sorption pump to avoid hydrocarbon contamination. The Vac-Ion pump was started and a vacuum of at least 10^{-7} torr achieved before exposure of the sample to the source. The sample chamber was shielded to prevent heating by the source but the pressure would generally rise immediately as a result of the out-gassing of the irradiated sample. The energy was incident upon the sample through a quartz window mounted in the sample chamber wall and the transmission of each quartz window was measured at frequent intervals.

The pressure in the sample chamber and the energy from the source was recorded periodically during a test. The information accumulated during each test is illustrated by the typical data sheet shown in Figure IV-7. After the desired exposure had been obtained, the sample was removed from the chamber and the spectral reflectance in the region 0.25 to 2.0 μ measured. Generally, this measurement was made immediately after removal but no changes were noted in the measured values for samples stored in air for several days.

The use of individual sample chambers was found to have significant advantages. There was never any question of cross contamination from samples. Furthermore, the testing of one sample did not interfere with another and the exposure times or irradiation levels were freely chosen by the experimenter.

D. EXPERIMENTAL RESULTS

Four aluminum alloys were used in the tests:

Type 1199 (99.99 percent aluminum)

Type 2024

Type 5557

Type 6061

All alloys were tested with hard and soft anodized coatings as shown in Table IV-3. The results obtained are summarized in Tables IV-4 through IV-11. In order to report these results, certain specifications in nomenclature and units of exposure must be made. Exposure is defined as the product of irradiation and time and a convenient unit is joules per square meter. A surface exposed normal to the sun for 24 hours outside the atmosphere would be subjected to 1.2×10^8 joules/m². The irradiation is reported in terms of watts per square meter, time is in hours, and the ultraviolet region is taken to be wavelengths less than 0.4 microns (4000 Å). The solar energy level outside the atmosphere of the earth is considered to be 1.4×10^3 watts/m² and there are 0.125×10^3 watts/m² at the wavelengths less than 0.4 μ.

The difficulties encountered in explaining many of the results obtained can be illustrated by Figure IV-8 where the spectral reflectances of two different areas of the same sample of hard anodized type 1199 aluminum alloy are shown. For one area, the reflectance decreased, i. e., positive degradation (+Δα); for another area, the reflectance increased, i. e., the solar absorptance decreased. A similar situation was found with a second sample of type 1199 hard anodize. It did not exhibit this localized degradation but it did show a decreased solar absorptance (Figure IV-9). Hard anodized type 5557 aluminum alloy also had a negative change in solar absorptance but there were small streaked regions of a slightly brown

color. As the exposure of this material was increased, the change in solar absorptance appeared to be reversing, i. e., $\Delta \alpha$ negative but less negative with increased exposure. The decrease in solar absorptance was also noted for the hard anodized type 2024 and 6061 alloys. The initial values of solar absorptance were of the order of 0.8 for these latter materials. Other than to confirm the common behavior of the hard anodized material, the observed change was too small to be significant.

The soft anodized materials also exhibit a common behavior. All of the soft anodized alloys tested increased significantly in solar absorptance (see Figures IV-10 through IV-12). The worst case observed was a type 2024 aluminum soft anodized coating which started with a solar absorptance of 0.44 and after 96 hours at 5.6×10^3 watts/m², from a B-H6 lamp (an exposure of 65×10^7 watts/m², $\lambda < 0.4 \mu$), reached a value of 0.89. The soft anodized alloys have a higher emittance than the hard anodized alloys and for this reason have been favored as a thermal control material. The degradation characteristics, however, indicate the hard anodize process may be a better overall choice.

If the tabulated results (Tables IV-4 through IV-11) for the various test materials are examined, the practice of accelerated testing (high irradiation level, short time) is shown to be questionable. The results for each material have been arranged in increasing exposure ($\lambda < 0.4$ microns) and it is evident that the irradiation level is an equally important parameter. Similarly, the degradation caused by the mercury B-H6 source and the xenon arc is not significantly different for anodized aluminum and the two sources are apparently equally effective for energy at wavelengths less than 0.4 microns. A few tests were performed to establish the relative importance of energy at wavelengths less than 0.3 microns and the results indicated energy at these wavelengths to be more effective in degradation. However, the anodized material was still affected by energy transmitted through a glass filter ($\lambda > 0.3 \mu$). Consequently, the spectral dependency of the degradation has not been defined adequately.

A preliminary study of the effect of "sealing" the anodized layer was performed. These results did not indicate a seal with either water or nickel acetate had a significant effect. However, the tests were quite limited and were not complete.

The synergistic behavior of the ultraviolet and vacuum factors is shown by the several tests made with the test materials exposed to this irradiation while in air. In each case, the reflectance of the materials increased as shown by Tables IV-4, IV-5, IV-6, IV-7, IV-8, IV-9, IV-11, and Figure IV-13. As described in the Progress Report dated 16 April 1962, degraded anodized aluminum can be reverted by heating in air or by additional ultraviolet irradiation in air. This behavior may indicate the discoloration is diffusion controlled and account for the observation that the color is not confined to the surface of the anodize layers but is a bulk effect.

A number of graphical attempts have been made to correlate the results of the degradation studies. None have been satisfactory. Without a clear understanding of the degradation mechanism, no analytical basis for correlation can be established. The practice of using a logarithmic scale for exposure for correlation is a convenience since this factor can range over several orders of magnitude. A linear or logarithmic scale for solar absorptance (or changes in this quantity) has not been substantiated physically. The success obtained by other investigators in correlating their results may be a fortunate consequence of a constant irradiation level, limited number of samples, and materials tested.

There are certain general conclusions that can be made from the tests performed:

- 1) Hard anodized aluminum appears to degrade with a decrease in solar absorptance; soft anodize degrades with an increase in solar absorptance.
- 2) The irradiation level is an important parameter and exposure (product of irradiation and time) is not sufficient to determine degradation under the environment of space.
- 3) The effects of ultraviolet irradiation and vacuum are synergistic.
- 4) Correlation of degradation tests is an unresolved problem and must await a fundamental study of the mechanisms involved.
- 5) Anodized aluminum can not be eliminated from the list of materials useful in space and in fact, the hard anodizing process is quite promising.

Table IV-3. Initial Solar Absorbances of the Aluminum Alloys as Anodized

| <u>Aluminum Alloy Number</u> | <u>Anodize</u> | <u>Thickness Microns</u> | <u>τ_0</u> |
|------------------------------|----------------|--------------------------|----------------------------|
| 1199 | Soft | 30 | 0.14 |
| 2024 | Soft | 30 | 0.44 |
| 5557 | Soft | 50 | 0.18 |
| 6061 | Soft | 30 | 0.59 |
| 1199 | Hard | 2 | 0.14 |
| 1199 | Hard | 5 | 0.28 |
| 1199 | Hard | 20 | 0.47 |
| 1199 | Hard | 50 | 0.50 |
| 1199 | Hard | 60 | 0.60 |
| 2024 | Hard | 50 | 0.88 |
| 5557 | Hard | 25 | 0.50 |
| 6061 | Hard | 25 | 0.89 |

Soft Anodize = Bright dip, anodized in 17 percent (by weight) sulfuric acid solution at $68^{\circ}\text{F} \pm 2^{\circ}\text{F}$, current density 15 amperes per square foot no seal

Hard Anodize = Bright dip, Martin Hard Coat standard process

Bright Dip = Greater Mountain Chemical 800 solution at 170°F for 10 minutes

Table IV-4. Data Summary Type 1199 Aluminum Soft Anodize
30 Microns Thick $\alpha_0 = 0.14$

| Sample | Lamp | Irradiation | | | Exposure | | $\Delta \alpha$ |
|----------------|---------|-------------------------------|--|---------------|-------------------------------|--|-----------------|
| | | Total 10^3 W/M^2 | $\lambda < 0.4\mu$ 10^3 W/M^2 | Time Hours | Total 10^7 J/M^2 | $\lambda < 0.4\mu$ 10^7 J/M^2 | |
| 10^{-6} Torr | | | | | | | |
| S1-3 | B-H6 | 1.7 | 0.51 | 3.0 | 1.9 | 0.57 | +0.01 |
| B4-18 | B-H6 | 3.5 | 1.05 | 1.5 | 1.9 | 0.57 | +0.01 |
| S1-2 | B-H6 | 5.2 | 1.56 | 1.0 | 1.9 | 0.57 | +0.23 |
| S1-4 | B-H6 | 7.0 | 2.20 | 0.75 | 1.9 | 0.57 | +0.22 |
| S1-14 | Xenon | 21.2 | 1.06 | 1.5 | 11.5 | 0.57 | +0.36 |
| S1-13 | Xenon | 12.0 | 0.6 | 3.0 | 13.0 | 0.65 | +0.04 |
| B4-8 | B-H6 | 5.5 | 1.65 | 1.5 | 3.0 | 0.9 | +0.02 |
| S1-6 | B-H6 | 1.6 | 0.48 | 6.0 | 3.5 | 1.0 | +0.01 |
| S1-8 | B-H6 | 3.2 | 0.99 | 3.0 | 3.5 | 1.0 | +0.02 |
| S1-9 | B-H6 | 4.8 | 1.44 | 2.0 | 3.5 | 1.0 | +0.46 |
| S1-7 | B-H6 | 6.6 | 1.97 | 1.5 | 3.5 | 1.0 | +0.48 |
| S1-10 | B-H6 | 4.3 | 1.29 | 2.7 | 4.1 | 1.2 | +0.05 |
| S1-5 | B-H6 | 4.8 | 1.44 | 3.0 | 5.2 | 1.6 | +0.01 |
| B4-16 | B-H6 | 2.8 | 0.84 | 6.0 | 6.0 | 1.8 | +0.01 |
| B4-15 | B-H6 | 5.5 | 1.65 | 3.0 | 6.0 | 1.8 | +0.41 |
| B4-6 | B-H6 | 5.5 | 1.65 | 3.0 | 5.9 | 1.8 | +0.51 |
| B4-12 | B-H6 | 5.5 | 1.65 | 3.0 | 6.0 | 1.8 | +0.28 |
| B4-14 | B-H6 | 1.0 | 0.30 | 18.0 | 6.5 | 1.9 | +0.01 |
| B4-13 | B-H6 | 1.4 | 0.42 | 24.0 | 12.0 | 3.6 | +0.01 |
| B4-11 | B-H6 | 5.5 | 1.65 | 6.0 | 12.0 | 3.6 | +0.04 |
| B4-7 | B-H6 | 5.5 | 1.65 | 6.1 | 12.0 | 3.6 | +0.41 |
| B4-4 | HBO-200 | 0.7 | 0.14 | 96.0 | 23.0 | 4.6 | +0.04 |
| S1-16 | Xenon | 3.0 | 0.15 | 96.0 | 108.0 | 5.4 | +0.07 |
| B4-9 | B-H6 | 5.5 | 1.65 | 12.0 | 24.0 | 7.2 | +0.42 |
| B4-10 | B-H6 | 5.5 | 1.65 | 24.0 | 48.0 | 14.4 | +0.54 |
| B4-5 | B-H6 | 5.5 | 1.65 | 48.0 | 96.0 | 29.0 | +0.59 |
| No Vacuum | | | | | | | |
| S1-12 | B-H6 | 5.2 | | 6.1 | 11.5 | 3.5 | -0.01 |

Table IV-5. Data Summary Type 1199 Aluminum Hard Anodize
60 Microns Thick $\alpha_0 = 0.30$

| Sample | Lamp | Irradiation | | Time Hours | Exposure | | Light $\Delta\alpha$ | Dark $\Delta\alpha$ |
|----------------|---------|-------------------------------|--|---------------|-------------------------------|--|-------------------------|------------------------|
| | | Total 10^3 W/M^2 | $\lambda < 0.4\mu$ 10^3 W/M^2 | | Total 10^7 J/M^2 | $\lambda < 0.4\mu$ 10^7 J/M^2 | | |
| 10^{-6} torr | | | | | | | | |
| B1-5 | B-H6 | 5.5 | 1.65 | 1.5 | 3.0 | 0.9 | -0.01 | |
| J1-5 | HBO-200 | 0.7 | 0.14 | 24.0 | 6.0 | 1.2 | +0.03 | |
| B1-7 | B-H6 | 5.5 | 1.65 | 3.0 | 6.0 | 1.8 | -0.05 | |
| J1-4 | B-H6 | 3.3 | 0.99 | 6.0 | 7.0 | 2.1 | +0.03 | |
| J1-7 | B-H6 | 5.1 | 1.55 | 4.0 | 7.4 | 2.2 | -0.01 | +0.18 |
| B1-8 | B-H6 | 5.5 | 1.65 | 6.0 | 12.0 | 3.6 | -0.02 | +0.17 |
| J1-2 | Xenon | 10.2 | 0.51 | 24.0 | 88.0 | 4.4 | -0.03 | +0.24 |
| J1-6 | B-H6 | 6.7 | 2.01 | 6.0 | 14.5 | 4.4 | -0.05 | +0.35 |
| B1-10 | B-H6 | 5.5 | 1.65 | 12.0 | 24.0 | 7.2 | +0.08 | +0.28 |
| B1-13 | HBO-200 | 0.7 | 0.14 | 192.0 | 48.0 | 14.5 | -0.03 | |
| B1-4 | B-H6 | 5.5 | 1.65 | 96.0 | 190.0 | 57.0 | -0.10 | +0.01 |
| B1-1 | B-H6 | 5.5 | 1.65 | 192.0 | 380.0 | 114.0 | -0.05 | |
| No Vacuum | | | | | | | | |
| J1-8 | B-H6 | 5.0 | 1.5 | 24.0 | 43.0 | 13.0 | -0.02 | |
| J1-9 | B-H6 | 5.0 | 1.5 | 24.0 | 43.0 | 13.0 | -0.05 | |
| B1-9 | B-H6 | 5.0 | 1.5 | 40.0 | 80.0 | 24.0 | -0.03 | |

NOTE: Visually all samples appeared equally lightened; light $\Delta\alpha$ refers to light area except it was not always possible to measure clear area without some of dark spot; dark $\Delta\alpha$ refers to dark spots again with exception that it is not always possible to cover the dark area only. Therefore the measured $\Delta\alpha$ tends to be smaller than actual.

Table IV-6. Data Summary Type 2024 Aluminum Soft Anodize
30 Microns Thick $a_o = 0.44$

| Sample | Lamp | Irradiation | | Time Hours | Exposure | | Δa |
|----------------|---------|-------------------------------|--|---------------|-------------------------------|--|------------|
| | | Total 10^3 W/M^2 | $\lambda < 0.4\mu$ 10^3 W/M^2 | | Total 10^7 J/M^2 | $\lambda < 0.4\mu$ 10^7 J/M^2 | |
| 10^{-6} torr | | | | | | | |
| A4-3 | B-H6 | 5.5 | 1.65 | 1.5 | 3.0 | 0.9 | +0.36 |
| S2-13 | Xenon | 21.2 | 1.06 | 3.0 | 22.8 | 1.1 | +0.11 |
| S2-14 | Xenon | 3.0 | 0.15 | 24.0 | 26.0 | 1.3 | +0.03 |
| A4-8 | B-H6 | 6.0 | 1.80 | 3.0 | 6.5 | 1.9 | +0.02 |
| S2-6 | B-H6 | 1.5 | 0.45 | 12.0 | 6.5 | 2.0 | +0.04 |
| S2-2 | B-H6 | 3.2 | 0.96 | 6.0 | 6.8 | 2.0 | +0.18 |
| S2-4 | B-H6 | 4.6 | 1.38 | 4.0 | 6.6 | 2.0 | +0.21 |
| S2-7 | B-H6 | 6.2 | 1.86 | 3.1 | 7.0 | 2.1 | +0.04 |
| S2-3 | B-H6 | 6.4 | 1.92 | 3.0 | 7.0 | 2.1 | +0.21 |
| S2-5 | B-H6 | 4.9 | 1.47 | 4.4 | 7.5 | 2.2 | +0.19 |
| A4-10 | B-H6 | 1.2 | 0.36 | 18.7 | 8.0 | 2.4 | +0.04 |
| S2-10 | B-H6 | 1.3 | 0.39 | 24.0 | 11.2 | 3.4 | +0.01 |
| S2-12 | Xenon | 8.4 | 0.42 | 24.0 | 72.0 | 3.6 | +0.10 |
| S2-11 | Xenon | 10.2 | 0.51 | 24.0 | 88.0 | 4.4 | +0.12 |
| A4-5 | HBO-200 | 0.7 | 0.14 | 96.7 | 24.0 | 4.9 | +0.03 |
| A4-9 | B-H6 | 2.0 | 0.60 | 24.0 | 17.0 | 5.2 | +0.27 |
| A4-7 | B-H6 | 5.5 | 1.65 | 12.0 | 24.0 | 7.1 | +0.39 |
| A4-2 | B-H6 | 5.5 | 1.65 | 21.0 | 42.0 | 12.5 | +0.08 |
| A4-6 | B-H6 | 5.5 | 1.65 | 24.0 | 48.0 | 14.0 | +0.12 |
| A4-4 | B-H6 | 5.5 | 1.65 | 48.0 | 95.0 | 29.0 | +0.17 |
| S2-16 | B-H6 | 6.2 | 1.86 | 97.0 | 214.0 | 65.0 | +0.45 |
| No Vacuum | | | | | | | |
| S2-15 | B-H6 | 5.5 | 1.65 | 211.5 | 418.0 | 64.0 | -0.03 |

Table IV-7. Data Summary Type 2024 Aluminum Hard Anodize
 50 Microns Thick $\alpha_0 = 0.87$

| Sample | Lamp | Irradiation | | Time Hours | Exposure | | $\Delta\alpha$ |
|----------------|-------|-------------------------------|--|---------------|-------------------------------|--|----------------|
| | | Total 10^2 W/M^2 | $\lambda < 0.4\mu$ 10^3 W/M^2 | | Total 10^7 J/M^2 | $\lambda < 0.4\mu$ 10^7 J/M^2 | |
| 10^{-6} torr | | | | | | | |
| H2-9 | B-H6 | 1.6 | 0.48 | 24.0 | 14.1 | 4.2 | ≤ 0.01 |
| H2-4 | B-H6 | 3.0 | 0.90 | 12.8 | 13.8 | 4.1 | +0.005 |
| H2-12 | B-H6 | 4.8 | 1.44 | 8.0 | 13.7 | 4.1 | -0.005 |
| H2-5 | B-H6 | 6.4 | 1.92 | 6.0 | 13.9 | 4.2 | -0.005 |
| H2-16 | Xenon | 3.0 | 0.15 | 96.0 | 104.0 | 5.2 | +0.00 |
| H2-7 | B-H6 | 1.4 | 0.42 | 48.0 | 24.6 | 7.4 | ≤ 0.01 |
| H2-6 | B-H6 | 2.9 | 0.87 | 24.0 | 24.9 | 7.5 | ≤ 0.01 |
| H2-15 | B-H6 | 3.9 | 1.17 | 16.0 | 22.2 | 6.7 | -0.005 |
| H2-8 | B-H6 | 5.3 | 1.59 | 12.0 | 22.7 | 6.8 | +0.01 |
| H2-2 | B-H6 | 4.6 | 1.38 | 16.0 | 26.6 | 8.0 | -0.005 |
| H2-11 | Xenon | 8.4 | 0.42 | 96.0 | 292.0 | 14.6 | -0.00 |
| A1-10 | B-H6 | 5.5 | 1.65 | 192.0 | 380.0 | 114.0 | +0.01 |

Table IV-8. Data Summary Type 5557 Aluminum Soft Anodize
50 Microns Thick $\alpha_0 = 0.18$

| Sample | Lamp | Irradiation | | Time Hours | Exposure | | $\Delta\alpha$ |
|----------------|-------|-------------------------------|--|---------------|-------------------------------|--|----------------|
| | | Total 10^3 W/M^2 | $\lambda < 0.4\mu$ 10^3 W/M^2 | | Total 10^7 J/M^2 | $\lambda < 0.4\mu$ 10^7 W/M^2 | |
| 10^{-6} torr | | | | | | | |
| B2-9 | B-H6 | 5.5 | 1.65 | 1.5 | 3.0 | 0.9 | +0.02 |
| B2-5 | B-H6 | 5.5 | 1.65 | 3.0 | 6.0 | 1.8 | +0.03 |
| B2-3 | B-H6 | 5.5 | 1.65 | 6.0 | 11.9 | 3.6 | +0.05 |
| S5-4 | Xenon | 8.3 | 0.42 | 24.0 | 72.0 | 3.6 | +0.05 |
| B2-6 | B-H6 | 5.5 | 1.65 | 6.1 | 12.0 | 3.6 | +0.04 |
| S5-3 | B-H6 | 3.2 | 0.96 | 12.0 | 13.7 | 4.1 | +0.02 |
| S5-5 | B-H6 | 4.8 | 1.44 | 8.0 | 13.8 | 4.1 | +0.10 |
| S5-2 | B-H6 | 6.4 | 1.92 | 6.0 | 14.0 | 4.2 | +0.06 |
| S5-1 | B-H6 | 1.6 | 0.48 | 24.0 | 14.1 | 4.2 | +0.06 |
| S5-6 | Xenon | 2.8 | 0.14 | 96.0 | 98.0 | 4.9 | +0.08 |
| B2-7 | B-H6 | 5.5 | 1.65 | 24.0 | 47.5 | 14.0 | +0.08 |
| B2-10 | B-H6 | 5.5 | 1.65 | 24.0 | 47.5 | 14.0 | +0.15 |
| B2-4 | B-H6 | 5.5 | 1.65 | 96.0 | 190.0 | 57.0 | +0.20 |
| B2-2 | B-H6 | 5.5 | 1.65 | 192.0 | 380.0 | 114.0 | +0.20 |
| No Vacuum | | | | | | | |
| S5-7 | B-H6 | 5.5 | 1.65 | 165.7 | 327.0 | 98.0 | 0.00 |

Table IV-9. Data Summary Type 5557 Aluminum Hard Anodize
25 Microns Thick $\alpha_0 = 0.51$

| Sample | Lamp | Irradiation | | Time Hours | Exposure | | $\Delta \alpha$ |
|----------------------------------|-------|-------------------------------|--|---------------|-------------------------------|--|-----------------|
| | | Total 10^3 W/M^2 | $\lambda < 0.4\mu$ 10^3 W/M^2 | | Total 10^7 J/M^2 | $\lambda < 0.4\mu$ 10^7 J/M^2 | |
| 10 ⁻⁶ torr | | | | | | | |
| H5-6 | Xenon | 21.2 | 1.06 | 1.5 | 11.5 | 0.57 | -0.08 |
| H5-7 | Xenon | 12.0 | 0.60 | 3.0 | 13.0 | 0.65 | -0.05 |
| H5-8 | Xenon | 8.3 | 0.42 | 24.0 | 76.0 | 3.8 | -0.05 |
| H5-3 | B-H6 | 3.2 | 0.96 | 12.0 | 13.7 | 4.1 | -0.12 |
| H5-2 | B-H6 | 6.4 | 1.92 | 6.0 | 14.0 | 4.2 | -0.13 |
| H5-1 | B-H6 | 1.6 | 0.48 | 24.0 | 14.1 | 4.2 | -0.09 |
| H5-9 | Xenon | 3.0 | 0.15 | 96.0 | 104.0 | 5.2 | -0.07 |
| H5-10 | B-H6 | 2.3 | 0.69 | 24.2 | 19.8 | 6.0 | -0.06 |
| A2-3 | B-H6 | 5.5 | 1.65 | 48.0 | 95.0 | 29.0 | -0.10 |
| Multiple Exposures on one sample | | | | | Accumulated | | |
| H5-4 | B-H6 | 2.8 | 0.84 | 24.0 | 24.2 | 7.2 | -0.09 |
| | B-H6 | 2.7 | 0.81 | 24.0 | 48.4 | 14.4 | -0.08 |
| | B-H6 | 2.7 | 0.81 | 96.0 | 140.4 | 42.0 | -0.06 |
| | B-H6 | 2.5 | 0.75 | 192.0 | 310.4 | 93.0 | -0.07 |
| No Vacuum | | | | | | | |
| A2-4 | B-H6 | 4.0 | 1.20 | 223.0 | 320.0 | 96.0 | -0.12 |

Table IV-10. Data Summary Type 6061 Aluminum Soft Anodize
30 Microns Thick $\alpha_0 = 0.59$

| Sample | Lamp | Irradiation | | Time Hours | Exposure | | $\Delta\alpha$ |
|----------------|-------|-------------------------------|--|---------------|-------------------------------|---|----------------|
| | | Total 10^3 W/M^2 | $\lambda < 0.4\mu$ 10^3 W/M^2 | | Total 10^7 J/M^2 | $\lambda < 0.4$ 10^7 J/M^2 | |
| 10^{-6} torr | | | | | | | |
| S6-13 | Xenon | 21.2 | 1.06 | 1.6 | 12.2 | 0.61 | +0.14 |
| B3-6 | B-H6 | 5.5 | 1.65 | 1.5 | 3.0 | 0.9 | +0.22 |
| S6-5 | B-H6 | 6.2 | 1.86 | 1.5 | 3.3 | 1.0 | +0.17 |
| S6-4 | B-H6 | 1.6 | 0.48 | 6.0 | 3.6 | 1.1 | +0.02 |
| S6-3 | B-H6 | 5.0 | 1.50 | 2.0 | 3.6 | 1.1 | +0.15 |
| S6-12 | Xenon | 21.2 | 1.06 | 3.0 | 22.3 | 1.1 | +0.17 |
| S6-9 | B-H6 | 4.2 | 1.26 | 2.6 | 3.9 | 1.2 | +0.16 |
| S6-18 | Xenon | 3.0 | 0.15 | 24.0 | 26.0 | 1.3 | +0.00 |
| S6-19 | Xenon | 13.8 | 0.69 | 6.0 | 30.0 | 1.5 | +0.00 |
| S6-2 | B-H6 | 3.2 | 0.96 | 3.1 | 6.3 | 1.9 | +0.005 |
| S6-6 | B-H6 | 4.6 | 1.38 | 4.0 | 6.6 | 2.0 | +0.03 |
| S6-7 | B-H6 | 6.4 | 1.92 | 3.0 | 7.0 | 2.1 | +0.19 |
| S6-8 | B-H6 | 6.5 | 1.95 | 3.0 | 7.0 | 2.1 | +0.15 |
| B3-8 | B-H6 | 5.5 | 1.65 | 6.0 | 12.0 | 3.6 | +0.18 |
| B3-9 | B-H6 | 5.5 | 1.65 | 6.0 | 12.0 | 3.6 | +0.26 |
| S6-15 | Xenon | 8.3 | 0.42 | 24.0 | 76.0 | 3.8 | +0.02 |
| S6-14 | Xenon | 10.4 | 0.52 | 24.0 | 88.0 | 4.4 | +0.06 |
| B3-7 | B-H' | 5.5 | 1.65 | 12.0 | 24.0 | 7.2 | +0.02 |
| B3-5 | B-H6 | 5.5 | 1.65 | 24.0 | 48.0 | 14.4 | +0.07 |

Table IV-11. Data Summary Type 6061 Aluminum Hard Anodized
 Aluminum 25 Microns Thick $\alpha_0 = 0.90$

| Sample | Lamp | Irradiation | | Time Hour | Exposure | | $\Delta\alpha$ |
|----------------|-------|-------------------------------|--|--------------|-------------------------------|--|----------------|
| | | Total 10^3 W/M^2 | $\lambda < 0.4\mu$ 10^3 W/M^2 | | Total 10^7 J/M^2 | $\lambda < 0.4\mu$ 10^7 J/M^2 | |
| 10^{-6} torr | | | | | | | |
| A3-3 | B-H6 | 5.5 | 1.65 | 3.0 | 4.2 | 1.2 | +0.01 |
| H6-16 | Xenon | 3.0 | 0.15 | 24.0 | 26.0 | 1.3 | -0.00 |
| H6-14 | Xenon | 3.9 | 0.20 | 27.0 | 38.0 | 1.9 | +0.00 |
| H6-2 | B-H6 | 1.6 | 0.48 | 24.0 | 14.1 | 4.2 | -0.005 |
| H6-4 | B-H6 | 3.2 | 0.96 | 12.0 | 13.9 | 4.2 | -0.005 |
| H6-5 | B-H6 | 4.8 | 1.44 | 8.0 | 13.9 | 4.2 | -0.005 |
| H6-3 | B-H6 | 6.3 | 1.89 | 6.2 | 14.1 | 4.2 | -0.005 |
| H6-7 | B-H6 | 1.4 | 0.42 | 48.0 | 24.6 | 7.4 | -0.005 |
| H6-6 | B-H6 | 2.9 | 0.87 | 24.0 | 24.7 | 7.4 | -0.005 |
| H6-13 | B-H6 | 4.6 | 1.38 | 16.0 | 26.5 | 8.0 | +0.01 |
| H6-9 | B-H6 | 4.2 | 1.26 | 18.0 | 27.2 | 8.2 | -0.01 |
| A3-2 | B-H6 | 5.5 | 1.65 | 24.0 | 33.0 | 9.9 | +0.01 |
| H6-11 | Xenon | 8.3 | 0.42 | 96.0 | 290.0 | 14.5 | -0.005 |
| A3-4 | B-H6 | 5.5 | 1.65 | 96.0 | 137.0 | 41.0 | +0.01 |
| A3-5 | B-H6 | 5.5 | 1.65 | 192.0 | 274.0 | 82.0 | -0.01 |
| No Vacuum | | | | | | | |
| H6-12 | B-H6 | 5.5 | 1.65 | 114.0 | 226.0 | 11.3 | -0.00 |

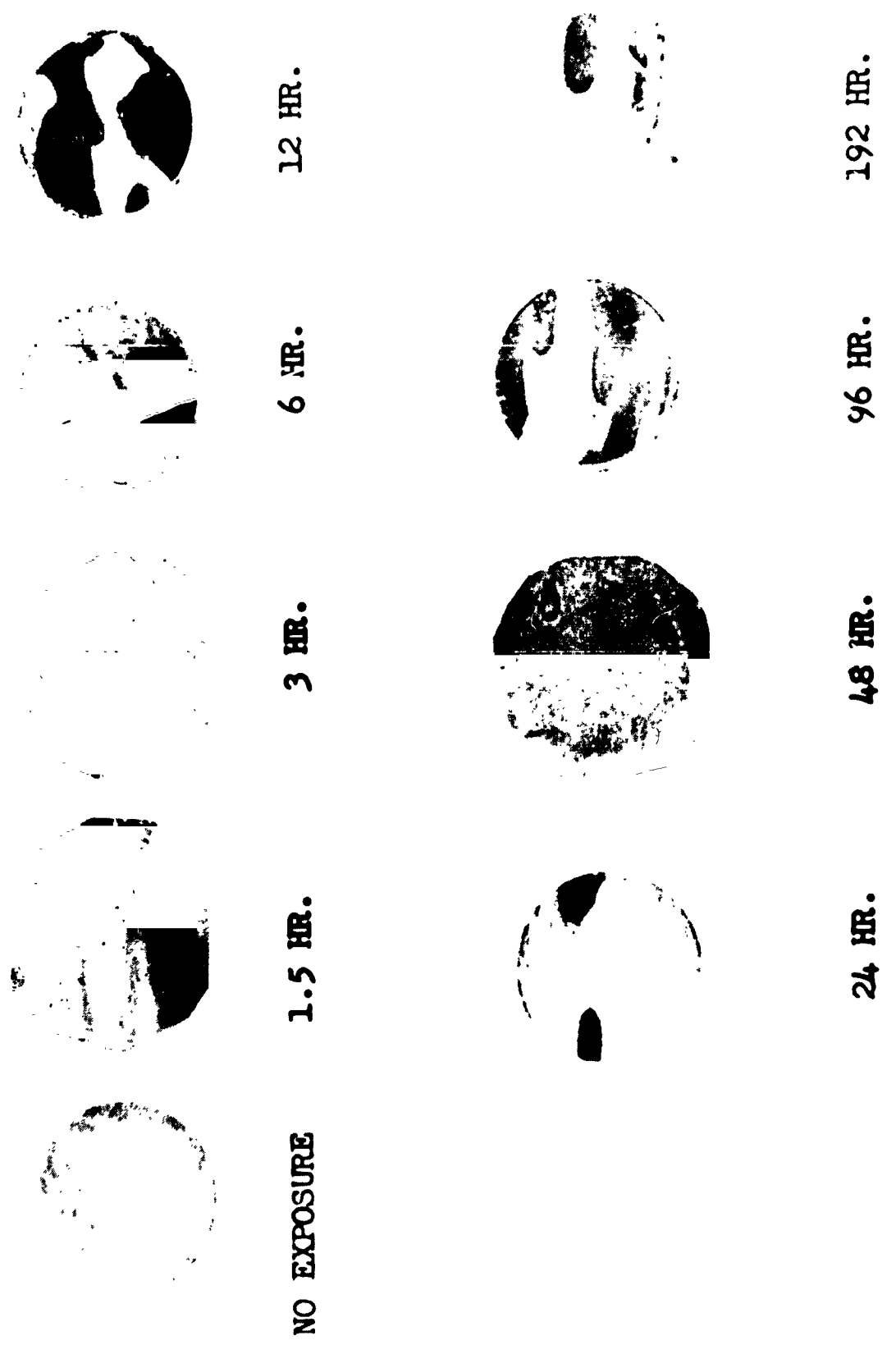


Figure IV-1. Samples of Type 1199 Aluminum Hard Anodized 60 Microns Thick Exposed to a GE B-H6 Mercury Arc Lamp at an Irradiation Level of 5.5×10^3 Watt/ Meter² for the Times as Indicated

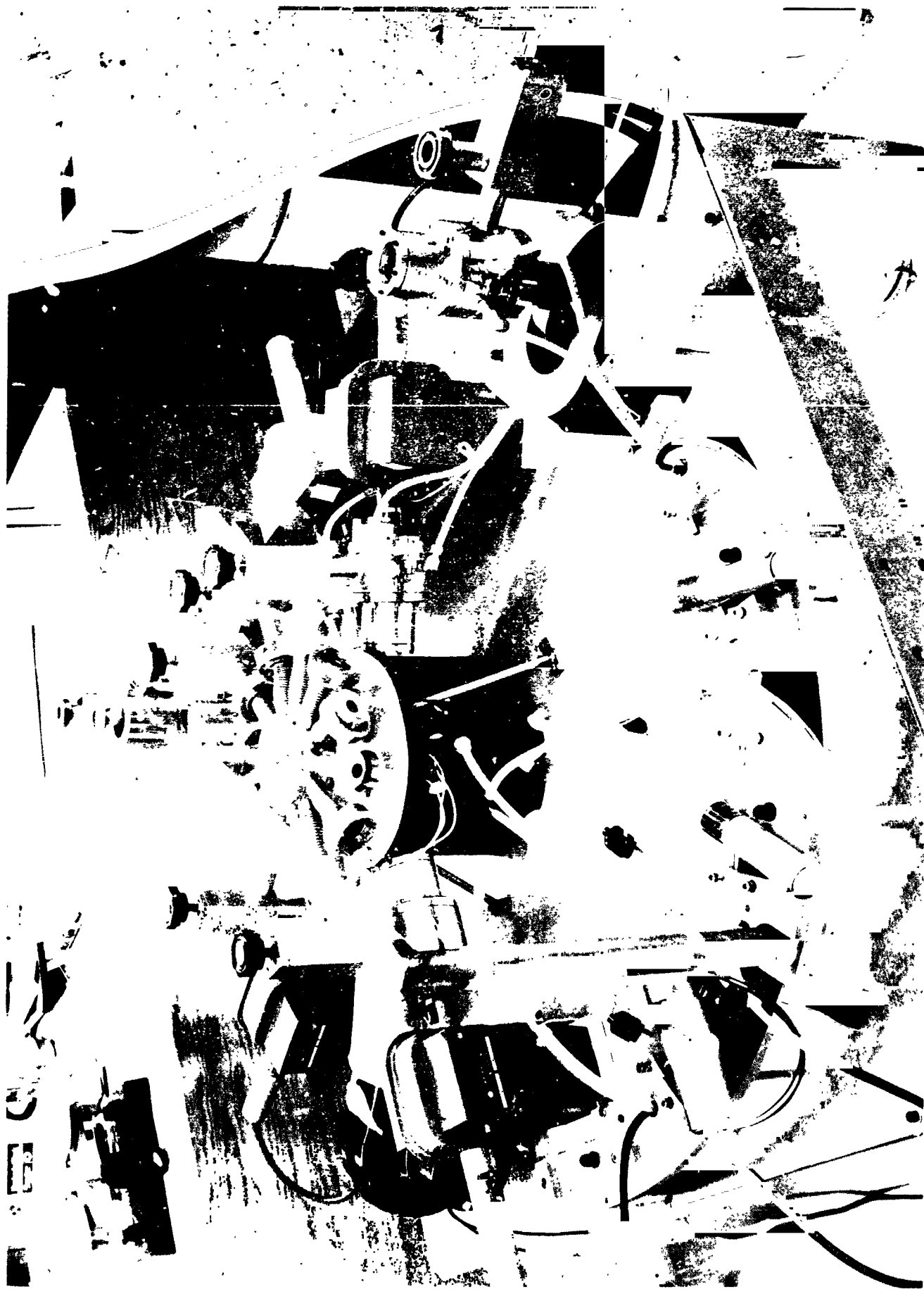


Figure IV-2. Original Multichamber System Fixed Distance

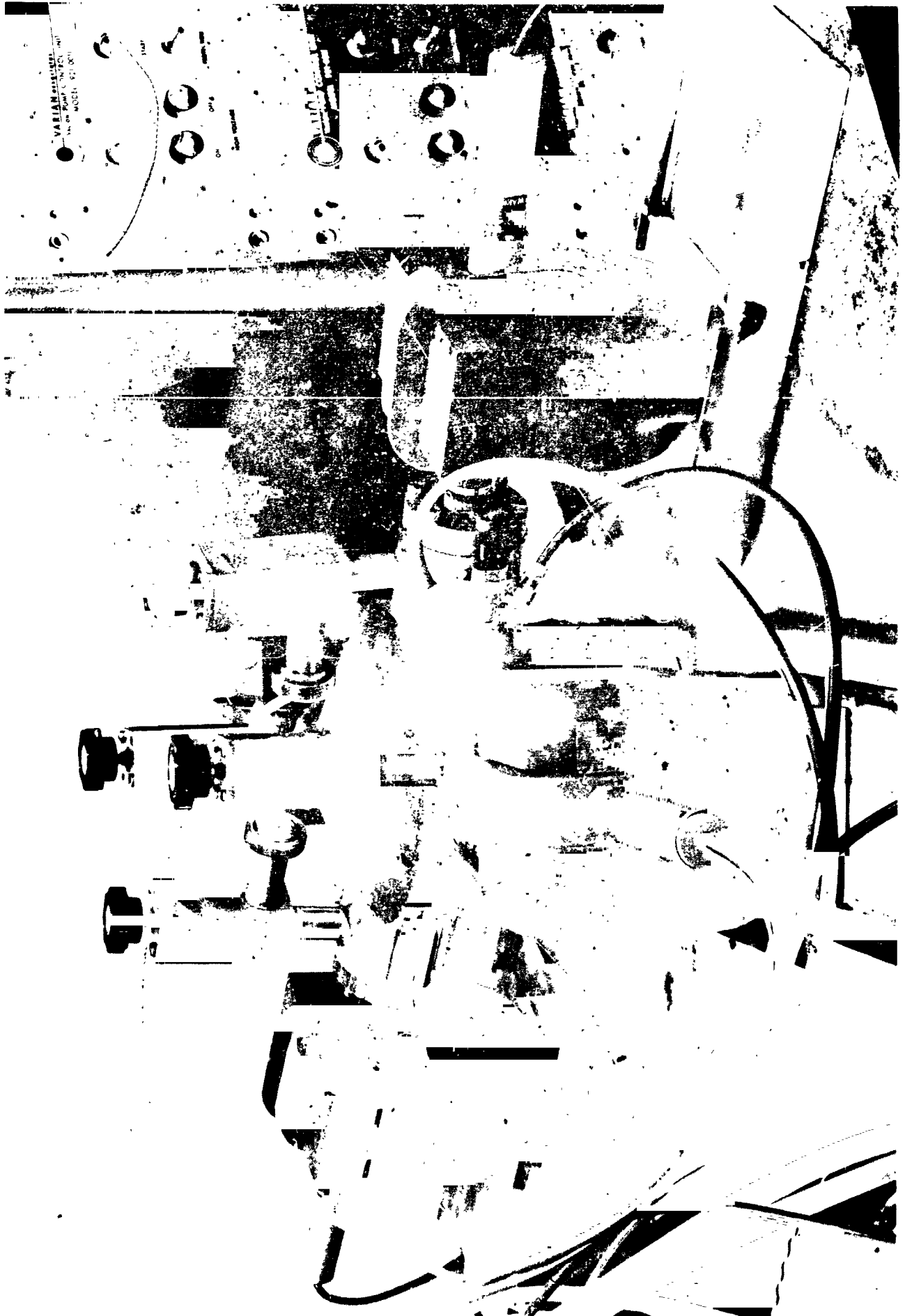


Figure IV-3. Second Multichamber System with Lamp to Sample Distance Variable

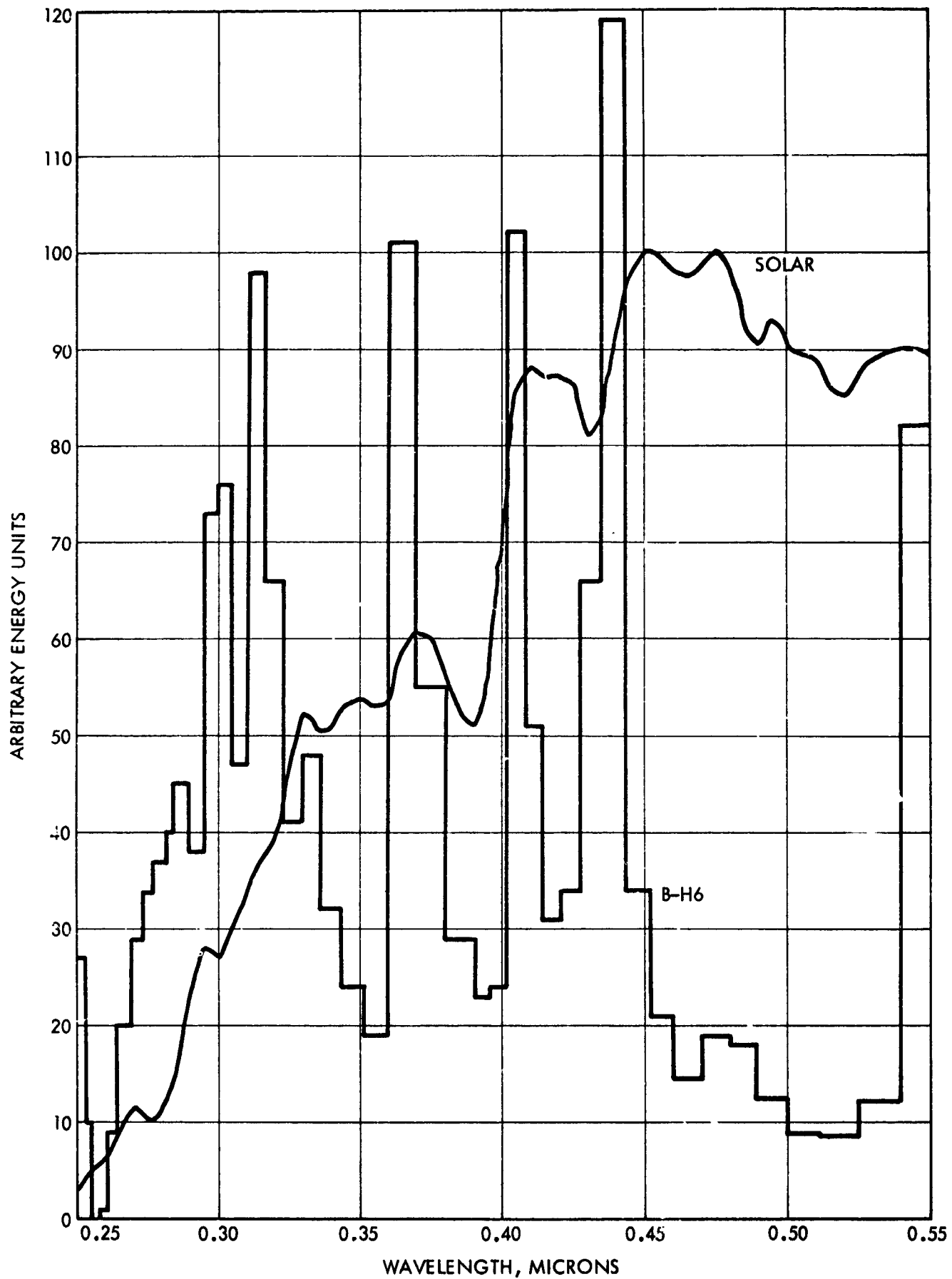


Figure IV-4. GE B-H6 Mercury Arc Spectrum and Solar Energy Spectrum

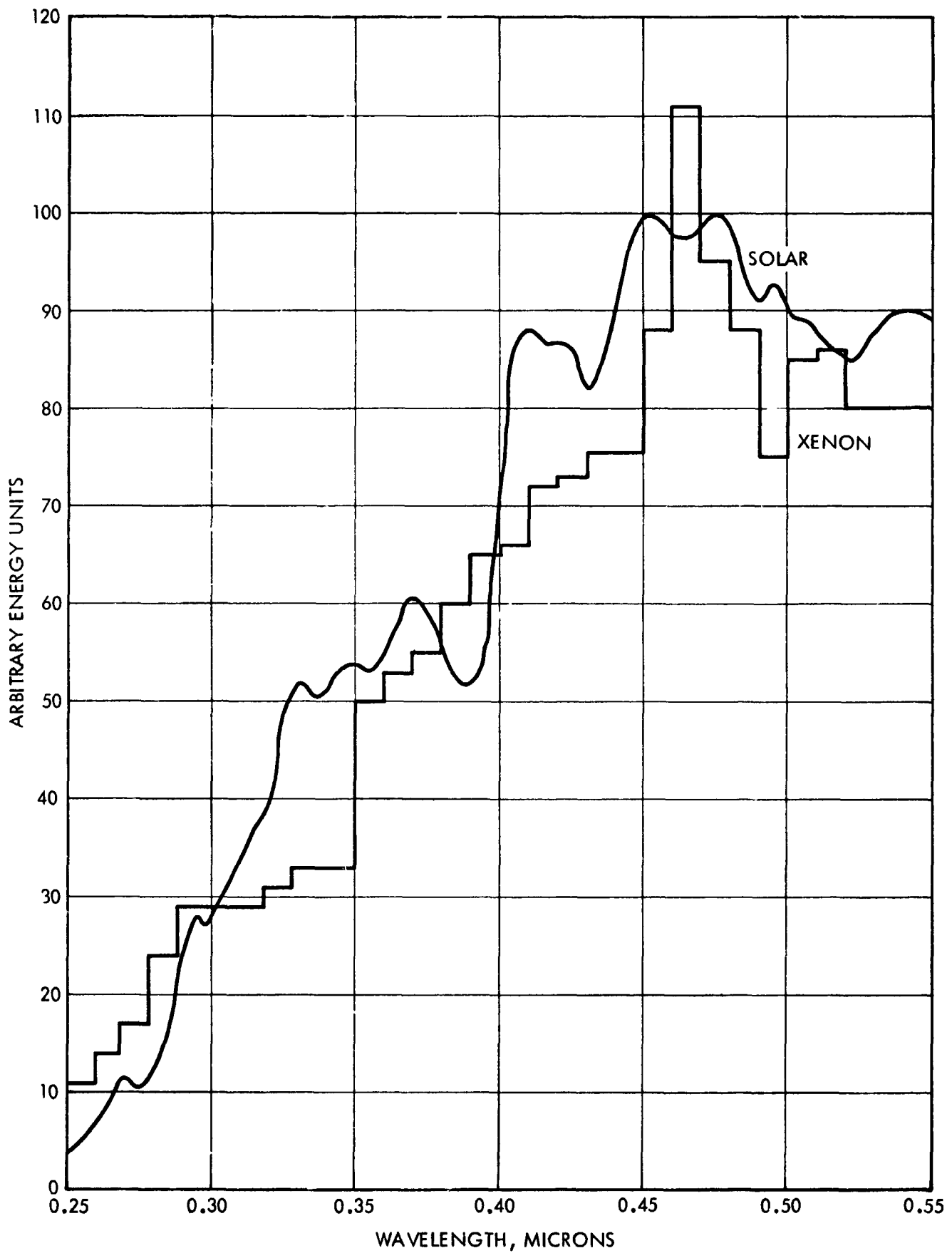


Figure IV-5. Hanovia 2.2 KW Xerion Arc Spectrum and Solar Energy Spectrum

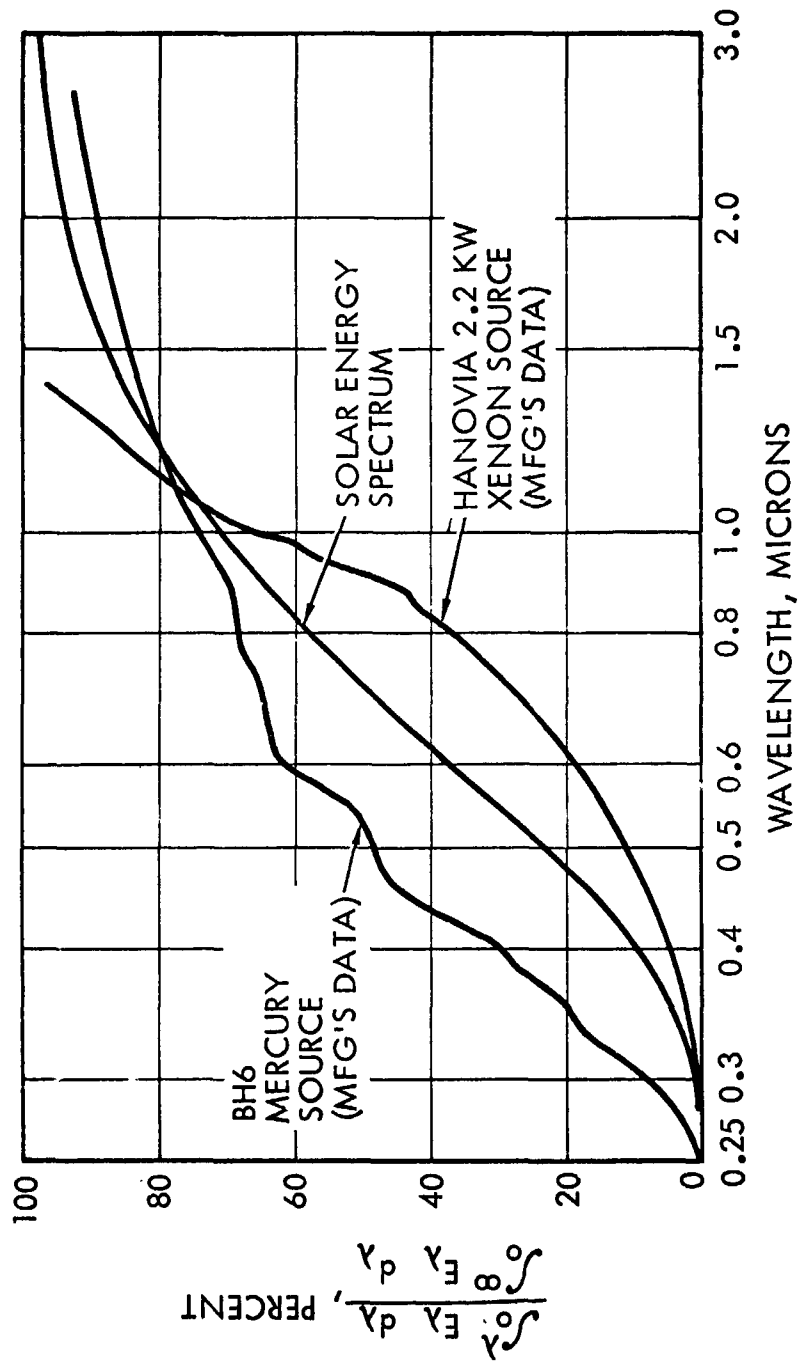


Figure IV -6. Cumulative Spectrums of GE B-H6 Mercury Arc Lamp, Johnson Solar and Hanovia 2.2 KW Xenon Arc Lamp

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ULTRAVIOLET DEGRADATION

SAMPLE DATA SHEET

SERIES UVD 10

SAMPLE DESIGNATION UVD-H02-16

MATERIAL 2024 ALUM

FINISH HARD ANODIZE

EXPOSURE 104 KJ/cm²

5.2 KJ/cm² < .4 μ

SUNHR < .4

HOURS

IRRADIATION .30 watt/cm²

LAMP 2.2 KW XENON

DISTANCE 175 mm S

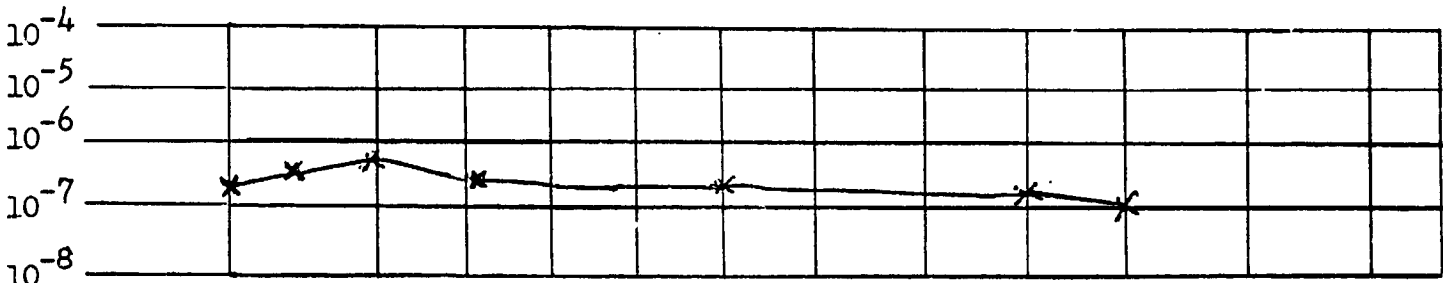
Hours on Lamp 325

Current 60 AMP

INITIAL α

CHANGE IN α

TORR



HOURS

0 .1 .5 1.0 2 4 8 12 24 48 96 144 192

DAYS

.5 1 2 4 6 8

TP READING 6.0

6.0

6.0

6.0

DATE

CIVIL TIME

RUNNING TIME METER

START: 15 Nov 62

1525

325.5

FINISH: 19 Nov 62

1525

421.5

HOUSING 7

PUMP 16

NOSE 4

POSITION 4

WINDOW 61

LAMP START RTM 000.5

Figure IV-7. Typical Data Sheet for Ultraviolet and Vacuum Degradation

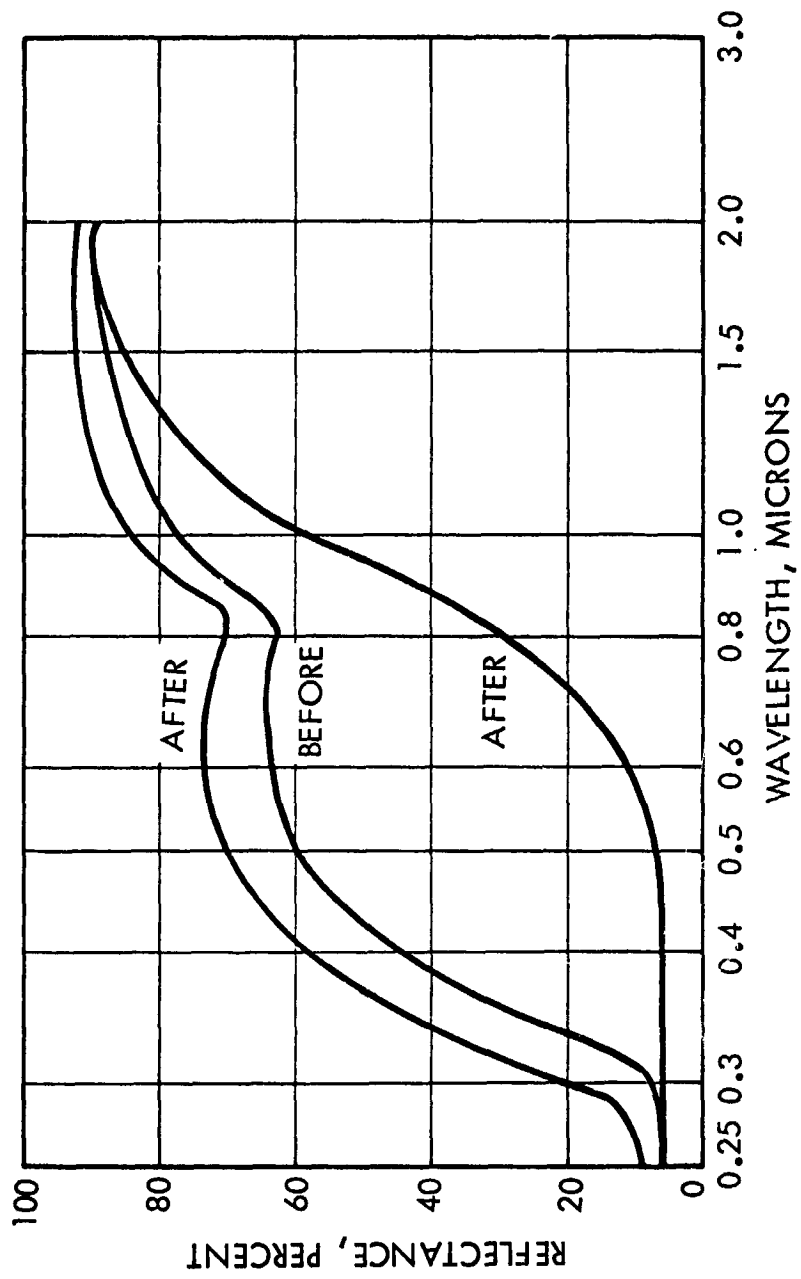


Figure IV-8. Type 1199 Aluminum Hard Sulfuric Acid Anodize 60 Microns Thick
 Irradiation: GE B-H6 Mercury Arc Lamp 6.7 x 10³ Watt/M²
 6 Hours 10⁻⁶ Torr Δa = -0.08, +0.15
 Different Areas on Same Sample

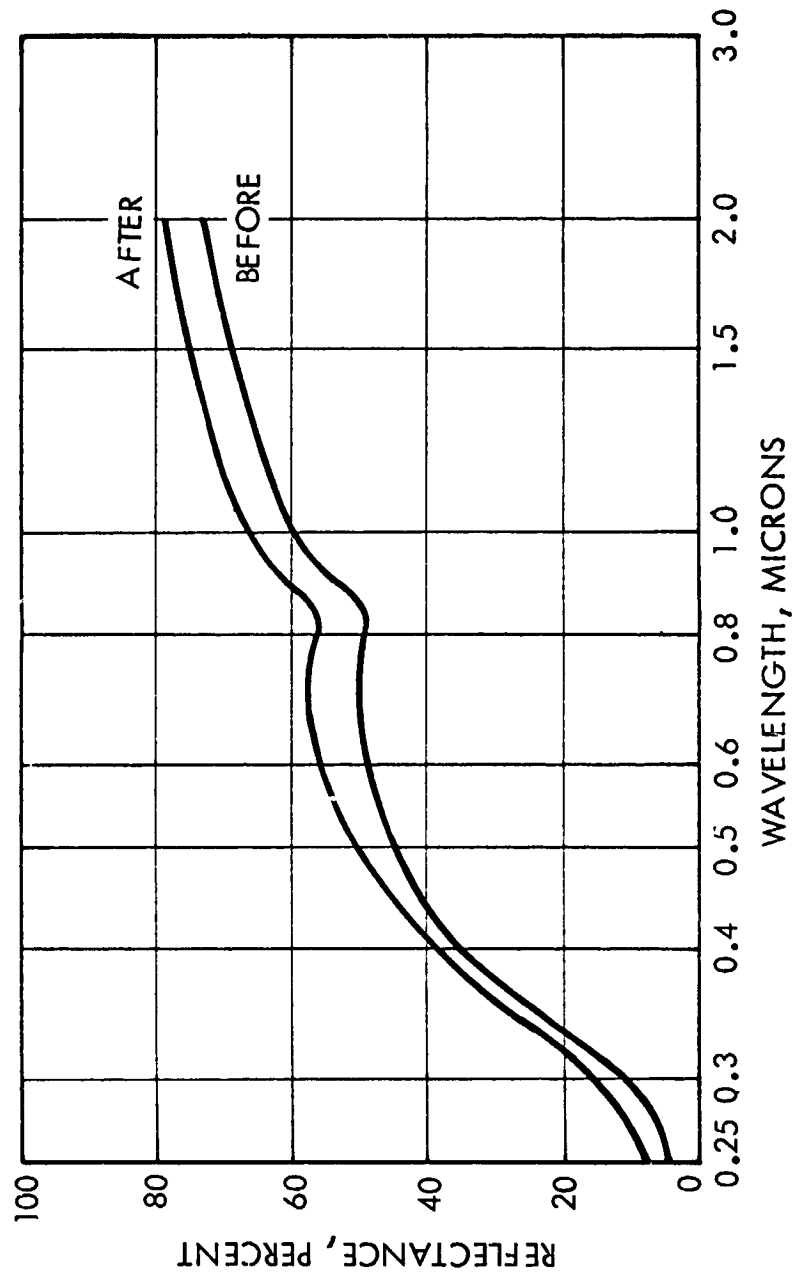


Figure IV-9. Type 1199 Aluminum Hard Sulfuric Acid Anodize
 50 Microns Thick Irradiation: Hanovia 2.2 KW
 Xenon Arc Lamp 3.9×10^3 watt/meter² 27 Hours
 10^{-6} Torr $\Delta\alpha = -0.06$

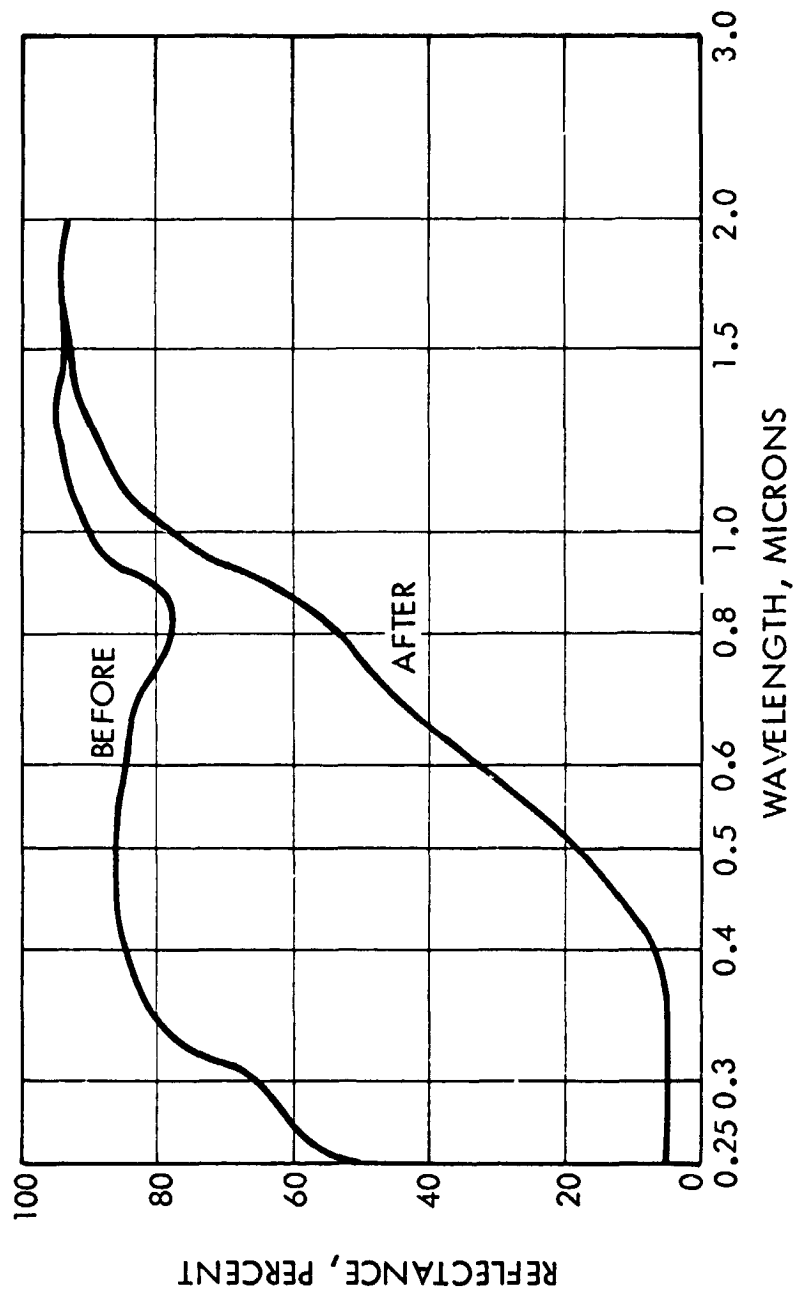


Figure IV-10. Type 1199 Aluminum Soft Sulfuric Acid Anodize
 30 Microns Thick Irradiation: Hanovia 2.2 KW
 Xenon Arc Lamp 2.1×10^4 Watt/Meter²
 1.5 Hours 10-6 Torr $\Delta\alpha = +0.37$

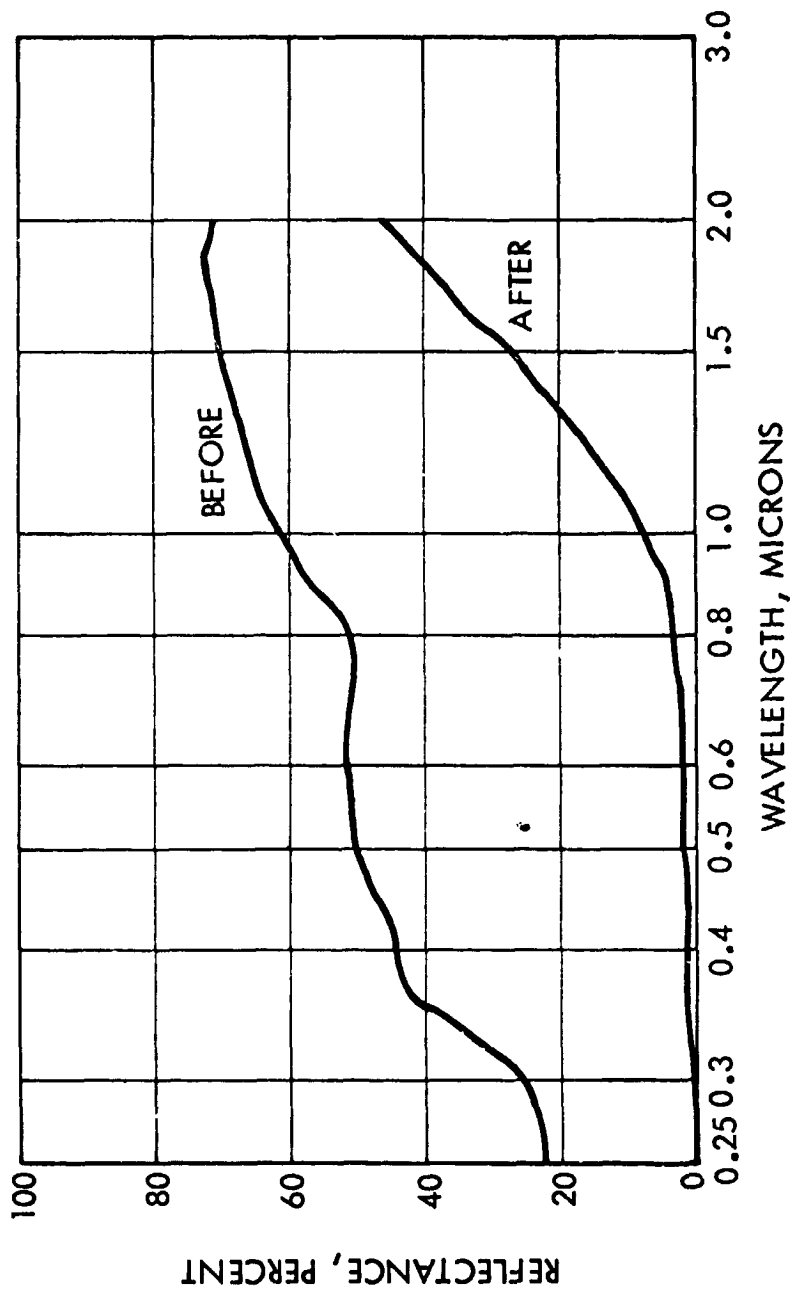


Figure IV-11. Type 2024 Aluminum Soft Sulfuric Acid Anodize
 30 Microns Thick Irradiation: G.E. B-H6 Mercury
 Arc Lamp 5.5×10^3 Watt/Meter² 96 hours 10^{-6} Torr
 $\Delta\alpha = +0.45$

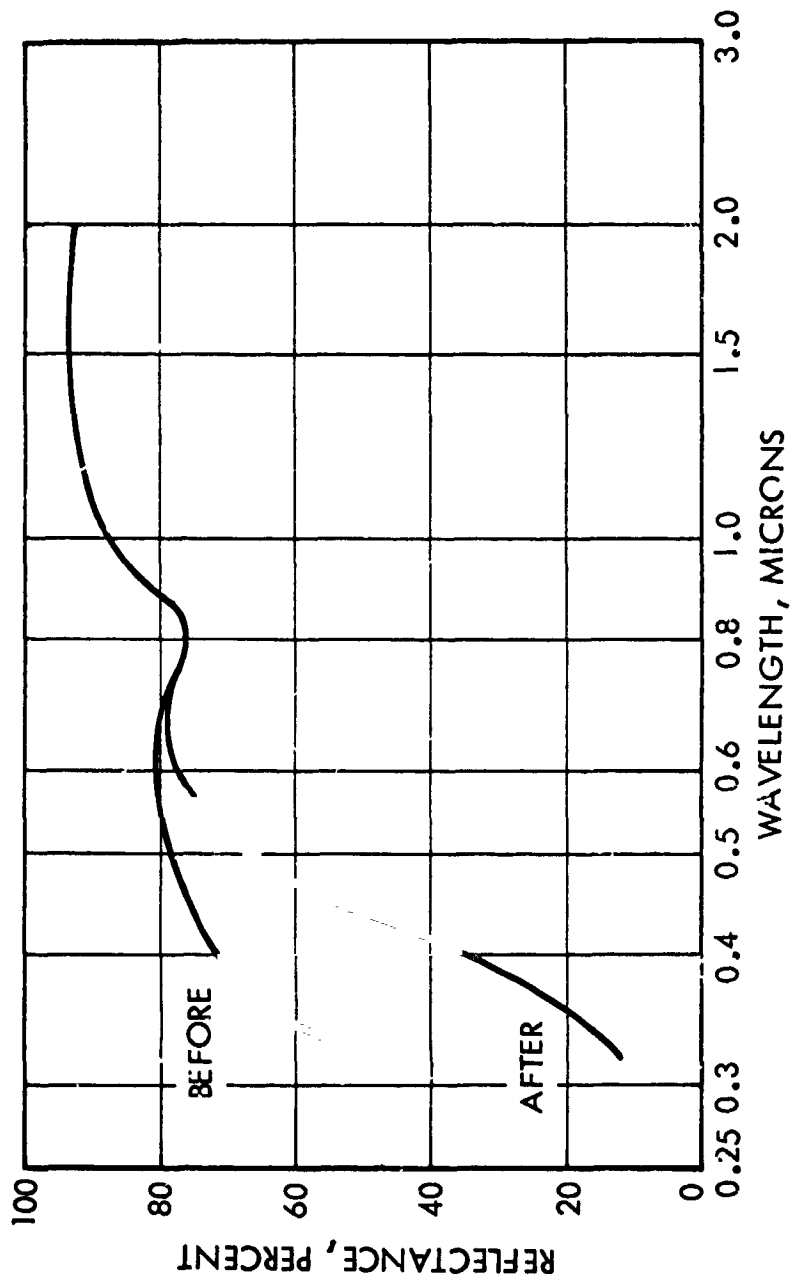


Figure IV-12. Type 5557 Aluminum Soft Sulfuric Acid Anodize
 50 Microns Thick Irradiation: Hanovia 2.2 KW
 Xenon Arc 2.8×10^3 Watt/Meter² 96 Hours 10⁻⁶
 Torr $\Delta a = +0.08$

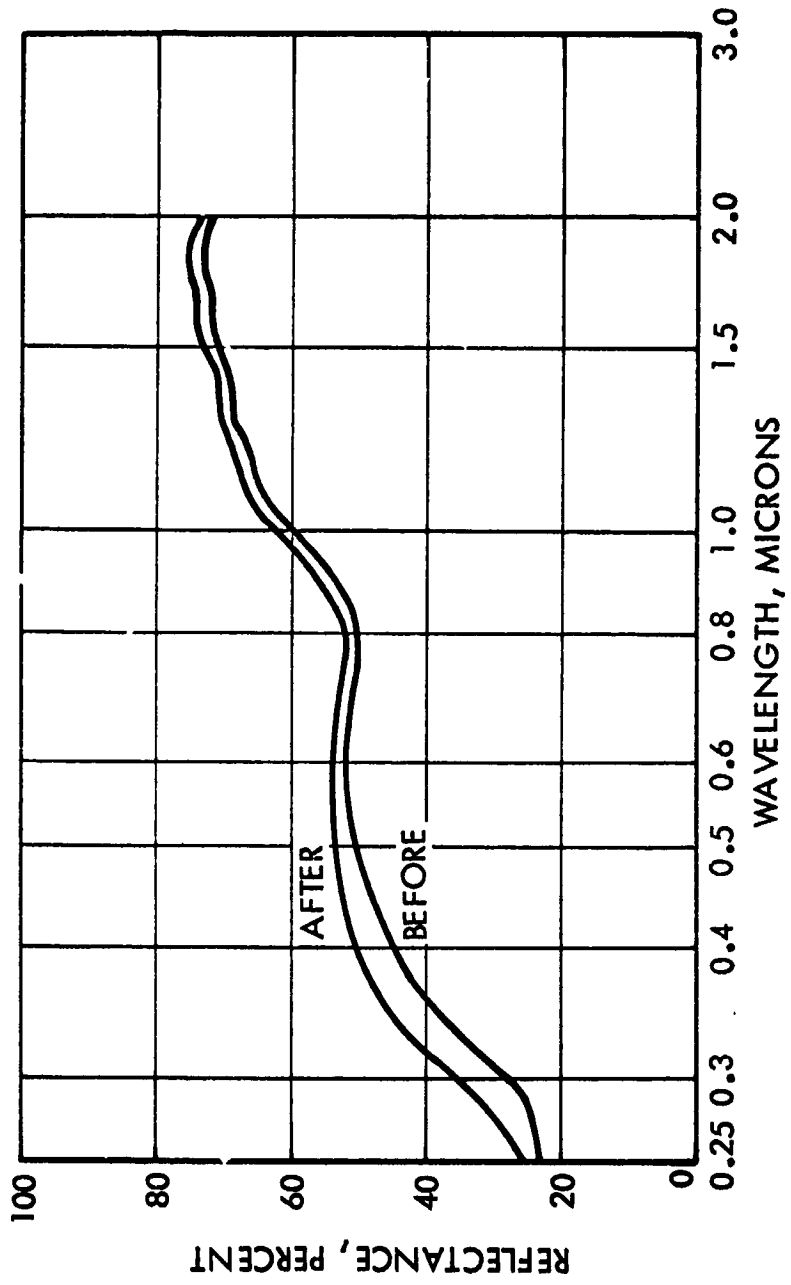


Figure IV -13. Type 2024 Aluminum Soft Sulfuric Acid Anodize
 30 Microns Thick Irradiation: G.E. B-H6 Mercury
 Arc Lamp 5.5×10^3 Watt/Meter² 2.2 Hours $\Delta\alpha = -0.03$
 Atmospheric Pressure

great deal of consideration in the above decisions. Micrometeorite studies were costly and this degradation could not be simulated properly; charged particle studies were also too costly in time and money. Ultraviolet and vacuum conditions could be simulated and the problem was vital to the design of thermal control systems, i.e., it was not as unknown a factor, as micrometeorites and charged particles were. This left only the illumination of the solar cell and the CsI prism test as additional studies.

A. SOLAR CELL PROPERTIES UNDER ILLUMINATION

In order to determine whether the reflectance of a solar cell was affected by illumination, a Hoffman shallow diffused cell with a 6 mil cover glass (blue filter) was placed in an integrating sphere reflectometer. A 100 ohm resistor was connected across the cell to simulate a load condition. An external energy source was focused upon the cell through an inspection port in the sphere wall (see Figure V-1).

Reflectance measurements proved to be quite difficult. The system was subject to a large amount of "noise" in the detector system. Several attempts were made to locate and eliminate this noise, e.g. d-c power supply for the external energy source, but all were unsuccessful. Consequently a complete spectral reflectance curve was not obtained. However, measurements at 0.54 and 0.80 micron showed no noticeable changes in the reflectance of the cell when illuminated and the conclusion was that there was no change.

B. CsI PRISM IN THE HEATED CAVITY REFLECTOMETER

The heated cavity reflectometer is limited to wavelengths less than 25 microns and in many cases measurements are desired beyond this. In an effort to extend the range, a CsI prism was mounted in the Perkin-Elmer Model 98 monochromator of the heated cavity reflectometer. A wavelength calibration of the system using the heated cavity as a source proved to be unsuccessful. The basic difficulty was found to be the lack of energy acting in combination with the low dispersion of this prism in the calibration region. Atmospheric absorption by water vapor and carbon dioxide did not

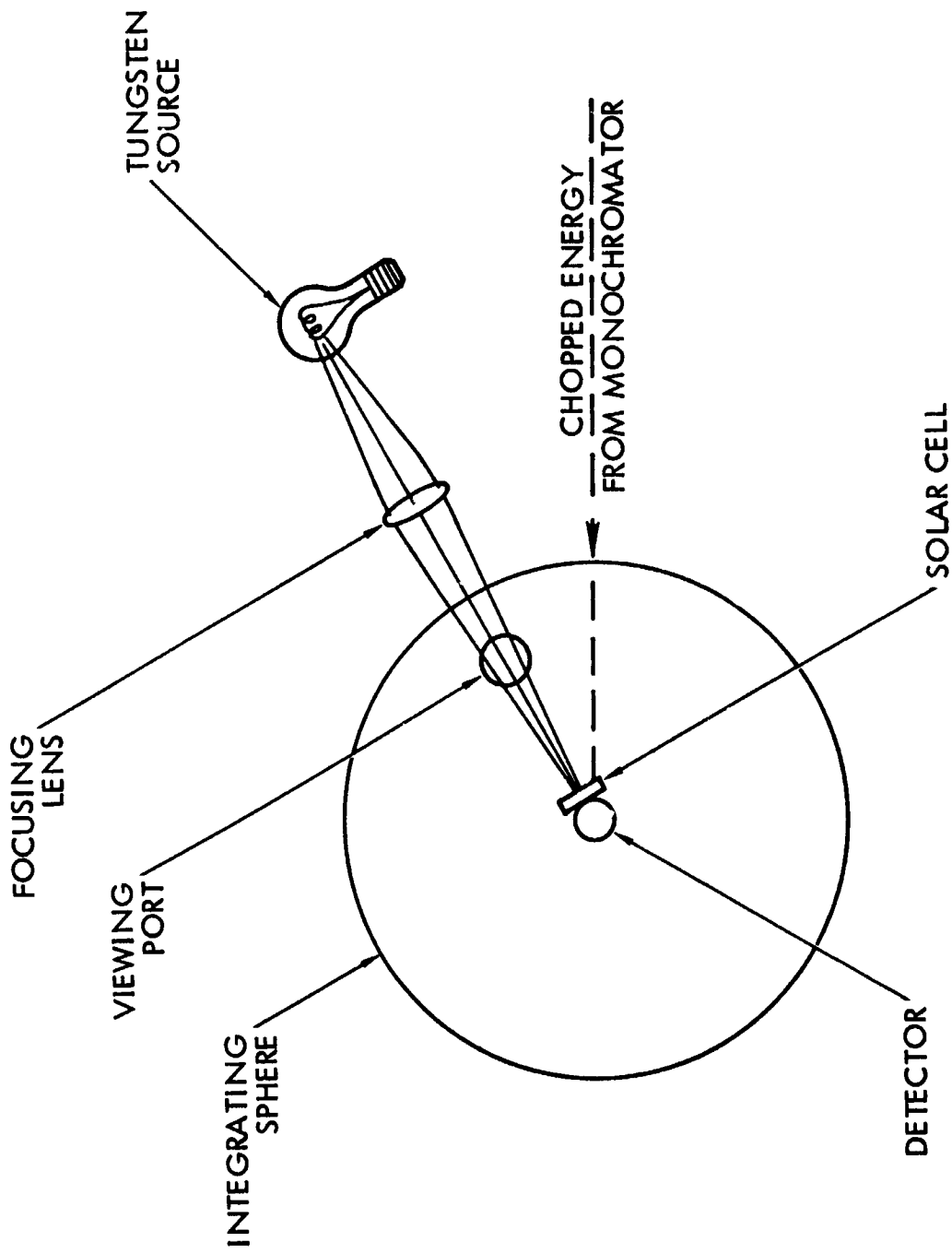


Figure V-1. Schematic of Solar Cell Illumination Experiment

V. OTHER STUDIES

The proposed development program called for several studies related to thermal control materials. A major area was the effect of the space environment upon these materials, i.e., micrometeorites, charged particle radiation, ultraviolet radiation and vacuum. An examination was to be made of the effect of illumination upon the spectral reflectance of a solar cell. A third study was the measurement of the transmission of plastics in an effort to obtain an alternative to solar cell cover glasses. The final area of examination was the use of a CsI prism in the heated cavity reflectometer in an attempt to extend the measurement range to 40 microns. The reorientation of the program in April 1962 necessitated an examination of these problems and a decision as to the merits of retaining them. For example, a UCLA report* was known to be ready for issue which contained spectral transmissions of a number of the plastics which would have been considered. This, plus the known ultraviolet instability of these materials, was believed to warrant dropping this part of the work.

The problem of the space environmental factors was more difficult, however. The simulation of micrometeorite bombardment is still in a very crude stage. A local concern, Rhodes and Bloxson, could provide simulated micrometeorite exposure with very limited control of particle size and velocity. The cost was approximately \$10,000 per run. STL has an electrostatic system with excellent control of size and velocity but the device did not provide the fluxes desired at that time. In view of the urgency and availability of the ultraviolet degradation apparatus, the micrometeorite studies were abandoned. It should be noted however, that anodized aluminum is a brittle material upon a ductile substrate. Consequently, it should be an interesting test material for micrometeorite studies. Charged particle studies were eliminated for essentially the same reasons as the micrometeorite studies, i.e., cost, time required, and urgency of the ultraviolet studies. The ultraviolet and vacuum degradation studies were given a

* D. K. Edwards and R. D. Roddick. "Basic Studies on the Use and Control of Solar Energy," Report 62-27, July 1962

produce any specific absorption lines that could be definitely described as originating with these gases. A similar problem existed with the 2, 2, 4 trichlorobenzene absorption cell.

If the experiment should be tried again, the heated cavity should be operated at as high a temperature as possible. In addition, a double pass monochromator (P-E model 99) should be used. The problem of stray energy is not difficult to overcome by the use of selective filters if sufficient energy is available.

VI. CONCLUSIONS

The examination and development of techniques for ultraviolet and vacuum degradation testing of thermal control materials is believed to be the most important consequence of this program. The observed irradiation level dependence of this degradation for anodized aluminum has made accelerated testing a questionable practice. Other questions which have been raised but not answered include the spectral dependency of degradation, the method of correlating results, and the level of vacuum required in the testing process. The test material has been limited to anodized aluminum and any specific conclusions must be restricted to this material. A broader program is needed to examine "types" of materials and to establish reliable testing procedures which are more general. With the limited number of spacecraft thermal control materials available, testing cannot be tolerated which rejects materials through incorrect procedures.

The study of anodized aluminum coatings, exclusive of the degradation effects, has shown that this process can provide materials with a wide range of thermal properties. The designer is thus provided an important means for securing the properties required by analysis. The degradation studies indicate that for hard anodized aluminum, these properties may be secured with satisfactory resistance to the ultraviolet and vacuum degradation of space.

As a result of this program, anodized aluminum has been shown to be a promising material for spacecraft thermal control. A more extensive examination of the material is needed, however, to establish the variables involved in processing and in degradation.