5331-FR

FINAL REPORT

FOR

PELLICLE BEAM SPLITTER

(15 February 1965 to 31 December 1965)

Contract No. NAS5-9555

Prepared for

Goddard Space Flight Center
Greenbelt, Maryland

BAIRD-ATOMIC 33 UNIVERSITY ROAD, CAMBRIDGE, MASSACHUSETTS 02138

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Submitted by: William Vaughan
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ABSTRACT

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This report describes the work done by Baird-Atomic, Inc., on NASA Contract NAS5-9555 for the development and fabrication of a Pellicle Beam Splitter. The report covers the contract period from 15 February 1965 to 31 December 1965. It describes materials and methods used and results obtained in each attempt to produce a pellicle-type beam splitter that operates efficiently in the spectral region from 5 to 30 microns.

The work consisted of a literature survey to determine state of the art on pellicle materials and coating design, a computer analysis of the suggested metal and dielectric coatings, and the selection of the most promising candidate materials for trial. Methods were then developed for producing thin pellicles of the candidate materials and for applying coatings to them. The reflectance and transmittance of bare and coated materials were measured. Candidate materials were tested for mechanical strength and effects of humidity, temperature, and vibration. A strain-free mount was designed and fabricated, and the final pellicle beam splitter was fabricated.

The conclusions are that two materials examined would make good infrared beam splitter pellicles, and a third has possibilities for further development. Cellulose nitrate and Parylene N can both be formed to a thickness of as little as 0.1 micron, show a minimum of absorption in the infrared region, and are mechanically strong enough to withstand fairly severe environmental conditions. Propylene has excellent absorption and mechanical properties and can be formed to the thickness desired, but it presents problems of nonuniformity in thickness and flatness. The satisfactory coating developed was a trilayer coating of zinc sulfide and germanium. Special techniques were designed to permit deposition of this coating on cellulose nitrate and Parylene N.

If further work is done it is recommended that Parylene N substrate with three or more layers of zinc sulfide and germanium be developed. In addition, methods should be investigated for producing a uniformly thin pellicle of polypropylene.

AUTHOR

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PELLICLE BEAM SPLITTER

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1. INTRODUCTION

This final report covers the work done by Baird-Atomic, Inc. on NASA Contract NAS5-9555 for the development and fabrication of a Pellicle Beam Splitter during the contract period of 15 February 1965 to 31 December 1965. The report describes the materials, methods, and the results obtained in each attempt to produce a pellicle-type beam splitter that operates efficiently in the spectral region from 5 to 30 microns.

2. SCOPE OF WORK

The work consisted of:

a. Literature survey to determine latest state of the art on coating design and pellicle materials

b. Computer analysis of suggested metal and dielectric coatings

c. Selection of most promising candidate materials for trial

d. Development of methods for producing thin pellicles of candidate materials

e. Development of methods for applying coatings to various materials

f. Measurement of reflectance and transmittance of bare and coated materials

g. Environmental testing of candidate materials

h. Design and fabrication of a strain-free mount

i. Fabrication of a final pellicle beam splitter

3. LITERATURE SEARCH

The Journal of the Optical Society of America and Applied Optics publications were searched (ten years of past issues) for information on thin films, their optical properties in the infrared region, and their uses as beam splitters. Appendix I contains a list of references. The literature search showed that recent techniques in beam splitter coating design include many multilayer dielectric combinations and graded index coatings.

4. SURVEY OF MATERIALS

4.1 List of Materials Considered

A search for likely candidate materials covered suppliers and manufacturers of plastics available in the form of a castable liquid or commercially available in thin sheeting.

The castable group included:

Cellulose nitrate
Cellulose nitrate and Glyptal
Cellulose acetate
Cellulose acetate butyrate
Polyurethane

The second group, plastics commercially available in thin sheeting, included:

Polyethylene
Polypropylene
Mylar
Saran
Polycarbonate
Parylene
Teflon

4.2 Research on Fabrication of Unsupported Film

Consulting assistance was obtained from the Moleculon Research Corporation, 139 Main Street, Cambridge, Massachusetts. A copy of their report is attached (appendix II).

4.3 Basic Methods of Forming Thin Film

The fabrication techniques of thin films were reviewed in the following technical papers.

Blodgett, "Properties of Built-up Films of Barium Stearate"; J. Phys. Chem.; Vol. 41, p. 975-984 (1937).

Cornell and Cassidy, "The Preparation of Membranes"; J. Polymer Sci.; Vol. 55, p. 233-249 (1961).

Harris and Jolinson, "The Production of Strong Cellulose Accetate Films"; Rev. of Sci. Instr.; Vol. 4, p. 454-455 (1933).

Pate and Yaffe, "A New Material and Techniques for the Fabrication and Measurement of Very Thin Films for Use in 4 π - Counting"; Can. J. Chem.; Vol. 33, p. 15-23 (1955).

Schaefer and Harker, "Surface Replicas for Use in the Electron Microscope"; J. Ap. Phys.; Vol. 13, p. 427-433 (1942).

Based on this review, the following basic methods of forming thin films were used.

4.3.1 Liquid Casting

The material is first dissolved in a suitable solvent. A small amount is gently flowed on to a liquid surface. As the solvent evaporates, a thin film is left which can be picked up and transferred to a suitable mount.

4.3.2 Stretching

Some plastic materials can be pulled under tension well beyond their elastic limit. As the material stretches, it becomes thinner. Stretching can be done in a number of ways. The material can be held in a ring and blown by compressed air like half of a balloon (figure 1). It can be pulled radially, or it can be pulled first in one direction and then in another (figure 2).

Once the desired thickness is reached, the pulling force can be released and, since the material has exceeded its elastic limit, it will not go back to its original size and thickness.

4.3.3 Condensation

Vapor phase deposition, or condensation, is accomplished by depositing the material on a substrate. To obtain a free film, the material is either stripped off the substrate, or the substrate is chemically removed by an etchant which attacks the substrate, but does not damage the deposited film.

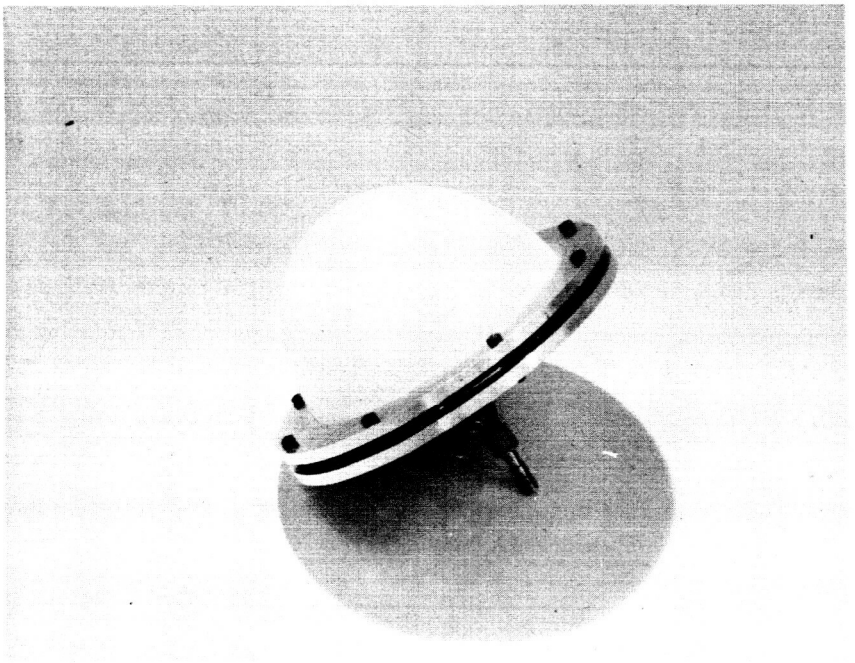

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Figure 1. Blow Stretcher

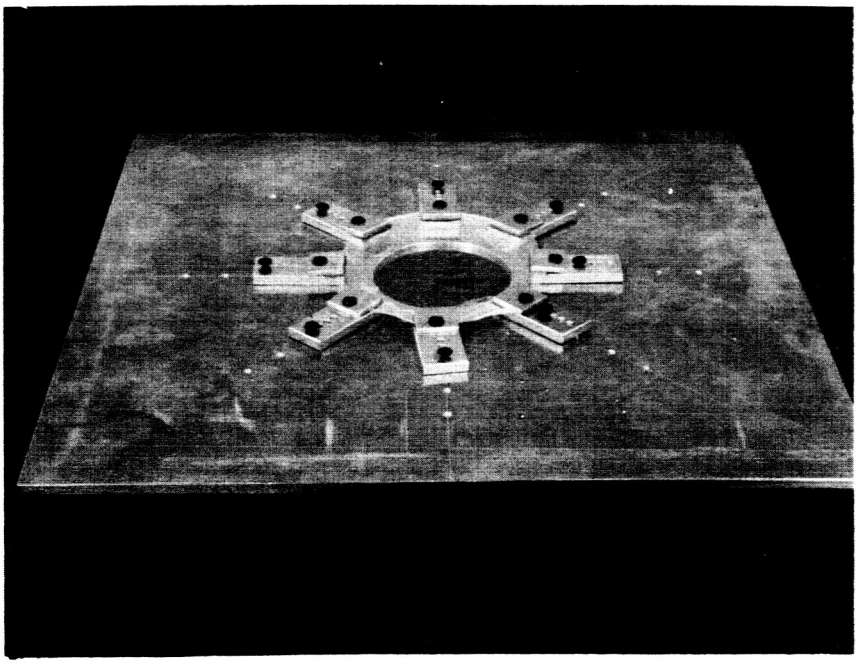

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Figure 2. Mechanical Stretcher

4.4 Evaluation of Survey

4.4.1 Castable Group

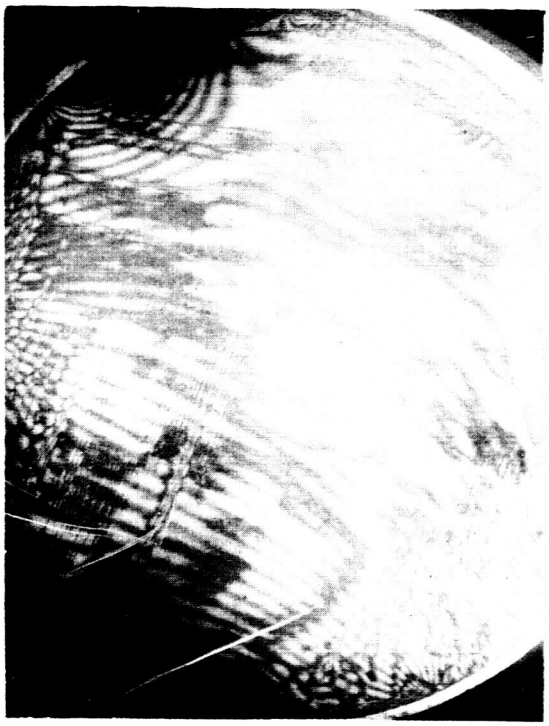
In the castable group, the following results were obtained:

Polyurethane	Film was rough and did not stay tight on the ring
Cellulose Acetate Butyrate	Film wrinkled badly when spread on water and could not be stretched flat
Cellulose Acetate	Same as cellulose acetate butyrate
Cellulose Nitrate	Flat, uniform films were obtained up to three inches in diameter
Cellulose Nitrate and Glyptal	No increase in strength over cellulose nitrate

4.4.2 Thin Sheeting Group

In this group, the following results were obtained:

Polyethylene	Could not dissolve in solvent. Limited stretch before fracture
Polypropylene	Could not dissolve in solvent. Could be stretched to the point where interference colors were visible over areas greater than three inches in diameter. The colors and hence the thickness were not uniform (figure 3). Spectral transmittance in the region from 2 to 16 microns showed very little absorption. Mechanical strength was excellent. During coating, film was easily destroyed by high temperature
Mylar and Saran	Could not be dissolved in any solvent tried. Stretching produced rupture before elongation was very great
Polycarbonate	Spectral transmittance showed many severe absorption bands in the 2 to 16 micron region
Parylene	Could be deposited on a glass or aluminum substrate in thicknesses as little as 0.1 micron. Could be stripped from suitably prepared glass substrate. Aluminum substrate was etched away in caustic without visible damage to the film. Absorption in infrared is low.



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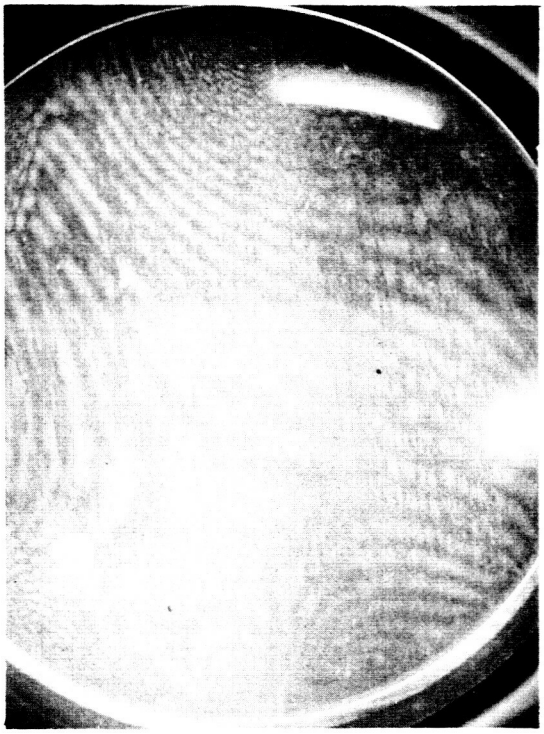

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Figure 3. Hand Stretched Polypropylene on 3-Inch Polished Ring

4.4.3 Transmittance Measurements

Transmittance and reflectance measurements up to 16 microns wavelength were made on a Baird-Atomic Infrared Recording Spectrophotometer, Model 4-55. Measurements in the spectral region from 16 to 30 microns were made on a Baird-Atomic NK-1 Recording Spectrophotometer using a cesium bromide prism and bolometer detector with a cesium bromide window. Some measurements in this region were also made on a Perkin-Elmer Infrared Recording Spectrophotometer.

The spectral transmittance measurements made on various uncoated materials are given in table 1. Spectrophotometer graphs are included.

Table 1
Spectral Transmittance Measurements on
Various Uncoated Materials

Material	Thickness (microns)	Incidence (degrees)	Range (microns)	Figure Number
Cellulose nitrate	0.25	Normal	2-16	4
Cellulose nitrate	0.25	45°	2-16	5
Cellulose nitrate	2.5	Normal	2-16	6
Cellulose nitrate	0.25	Normal	5-30	7
Defensite (polyurethane)	1.0	Normal	2-16	8
Polypropylene	0.9	Normal	2-16	9
Polypropylene	0.9	45°	2-16	10
Polypropylene	0.9	Normal	14-30	11
Mylar	6	Normal	2-16	12
Mylar	12	Normal	2-16	12
Saran	25	Normal	2-16	13
Polycarbonate	2	Normal	2-16	14
Parylene	0.5	Normal	2-16	15
Parylene	0.5	Normal	14-30	16
Teflon	12	Normal	2-16	17
Polyvinyl acetate	2.5	Normal	2-16	18

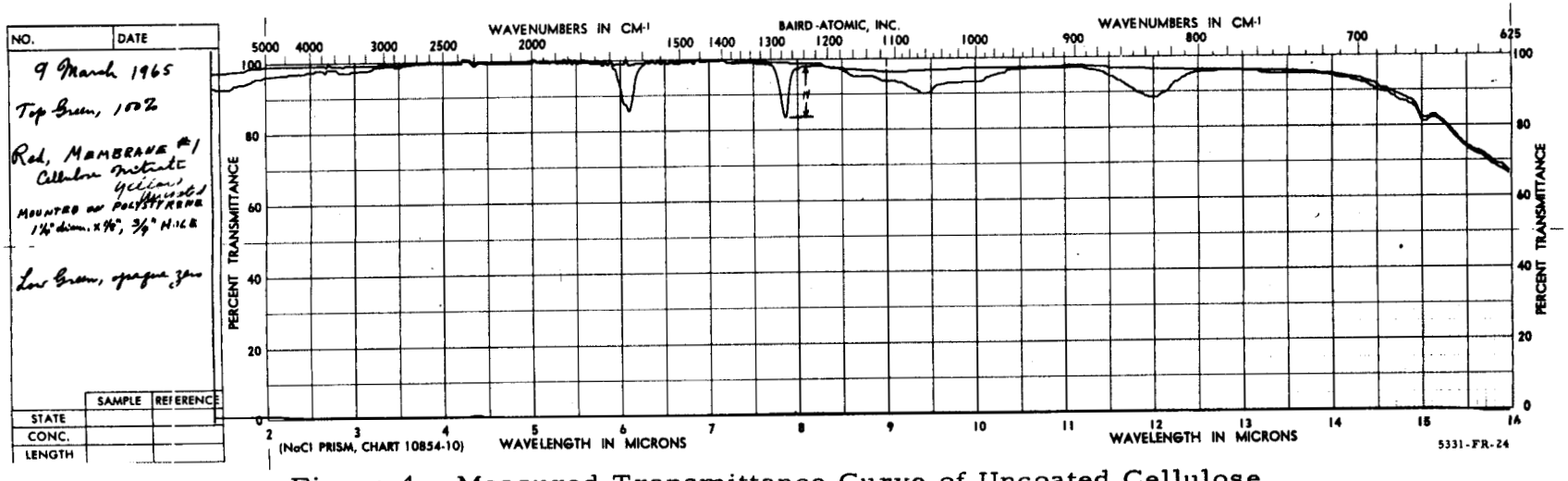


Figure 4. Measured Transmittance Curve of Uncoated Cellulose Nitrate Membrane, 0.25-Micron Thickness, 2 to 16 Microns

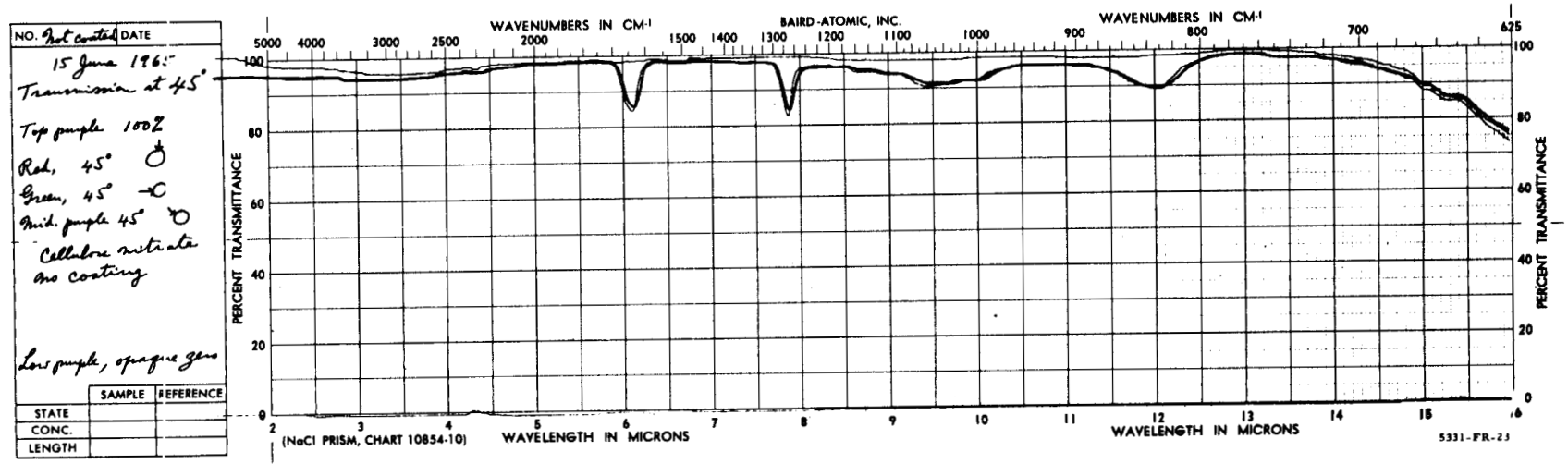


Figure 5. Measured Transmittance Curve of Uncoated Cellulose Nitrate Membrane, 0.25-Micron Thickness, 2 to 16 Microns

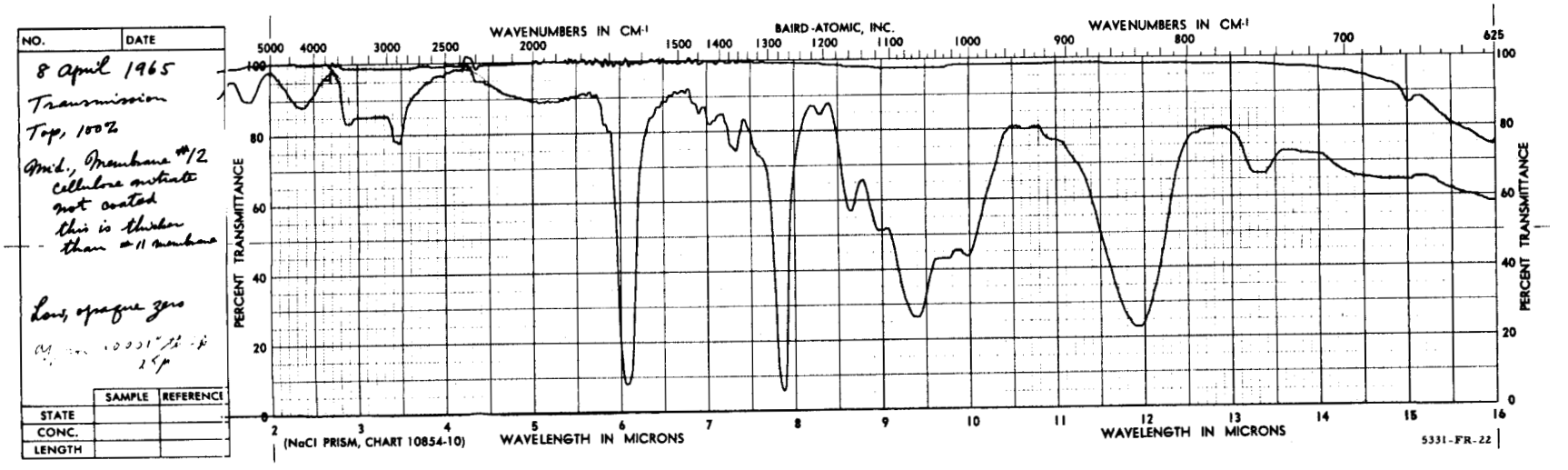


Figure 6. Measured Transmittance Curve of Uncoated Cellulose Nitrate Membrane, 2.5-Micron Thickness, Normal Incidence, 2 to 16 Microns

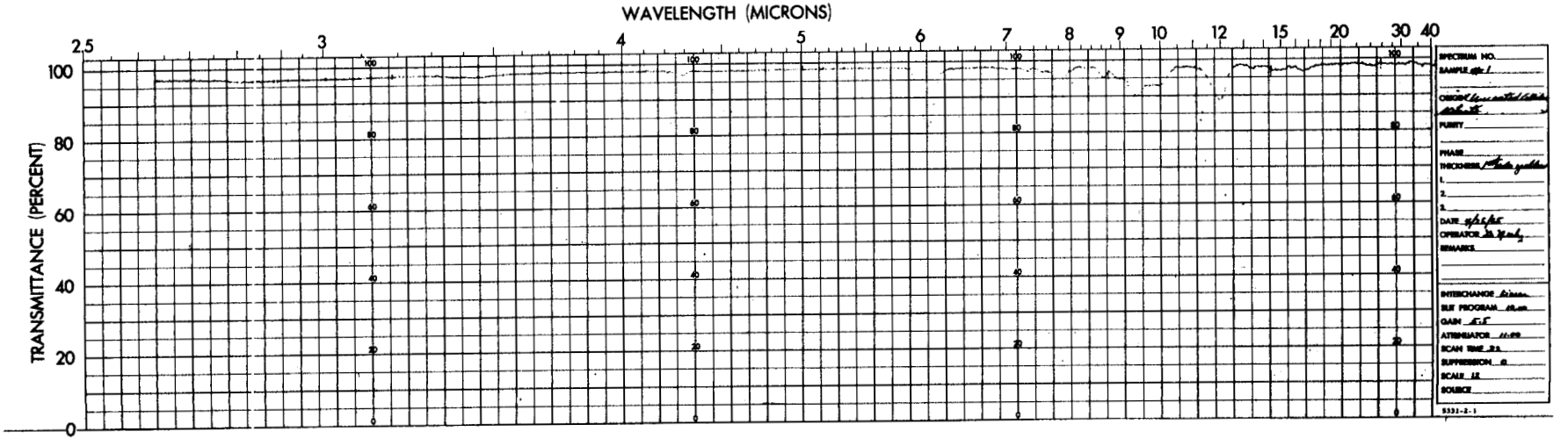


Figure 7. Measured Transmittance Curve of Uncoated Cellulose Nitrate Membrane, 0.25-Micron Thickness, Normal Incidence, 2 to 16 Microns

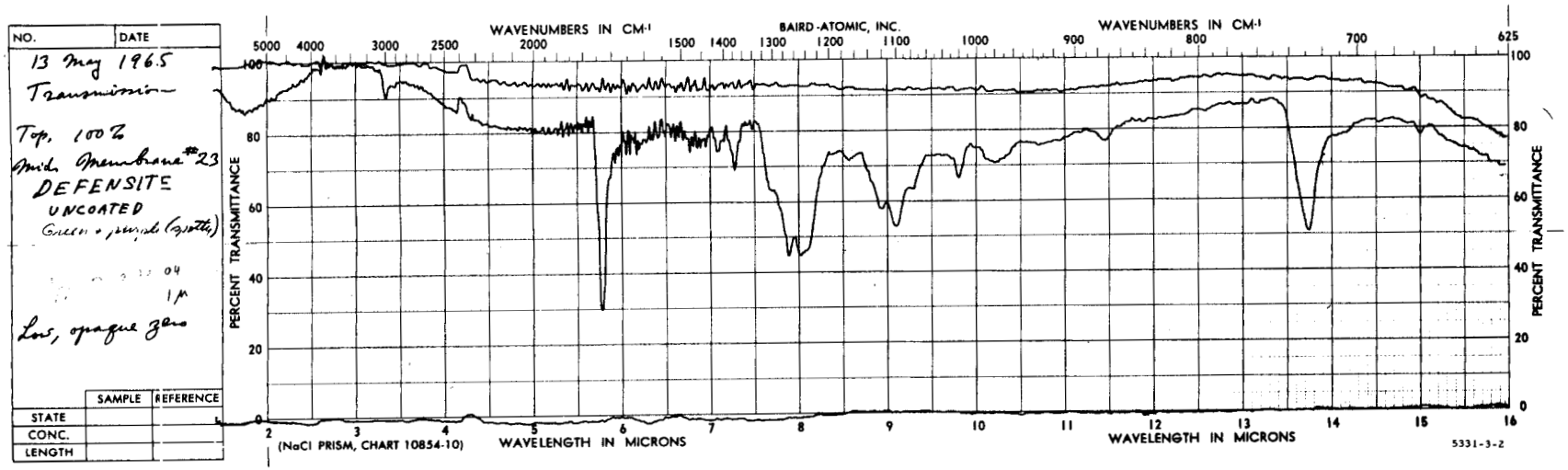


Figure 8. Measured Transmittance Curve of Uncoated Defensite Membrane, 2 to 16 Microns

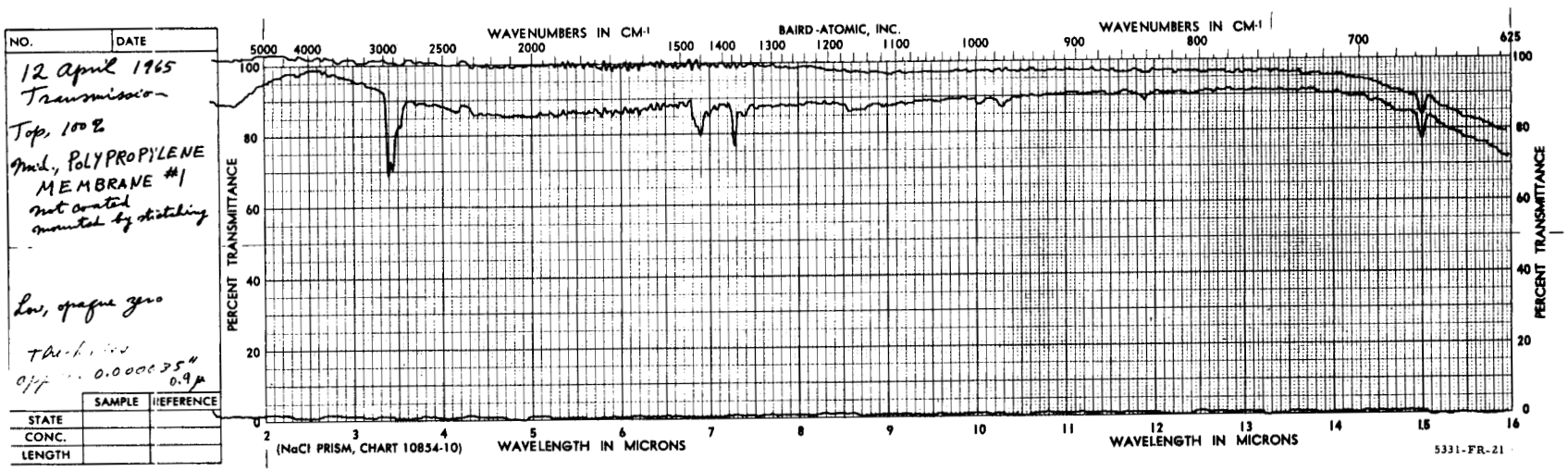


Figure 9. Measured Transmittance Curve of Uncoated Polypropylene Membrane, Normal Incidence, 2 to 16 Microns

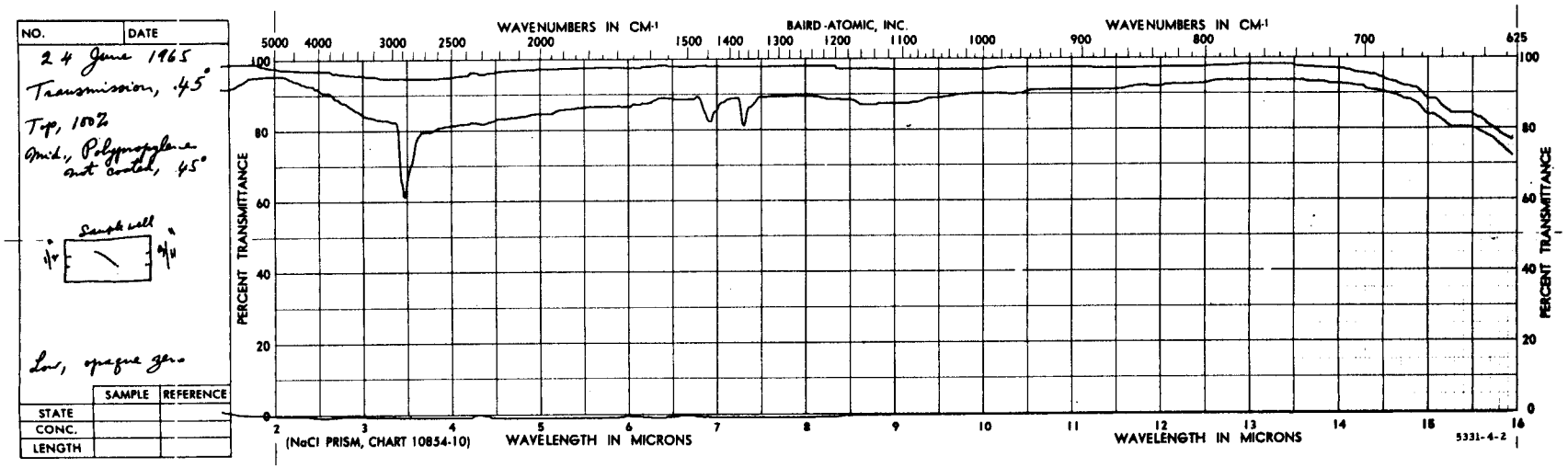


Figure 10. Measured Transmittance Curve of Uncoated Polypropylene Membrane, 45-Degree Incidence, 2 to 16 Microns

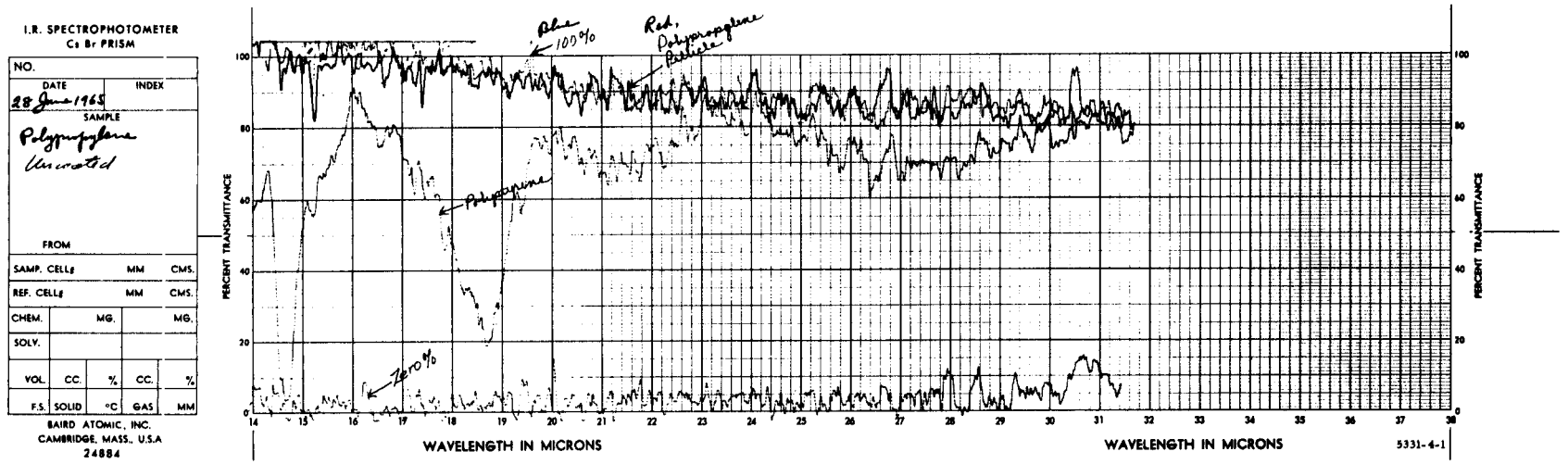


Figure 11. Measured Transmittance Curve of Uncoated Polypropylene Membrane, 14 to 30 Microns

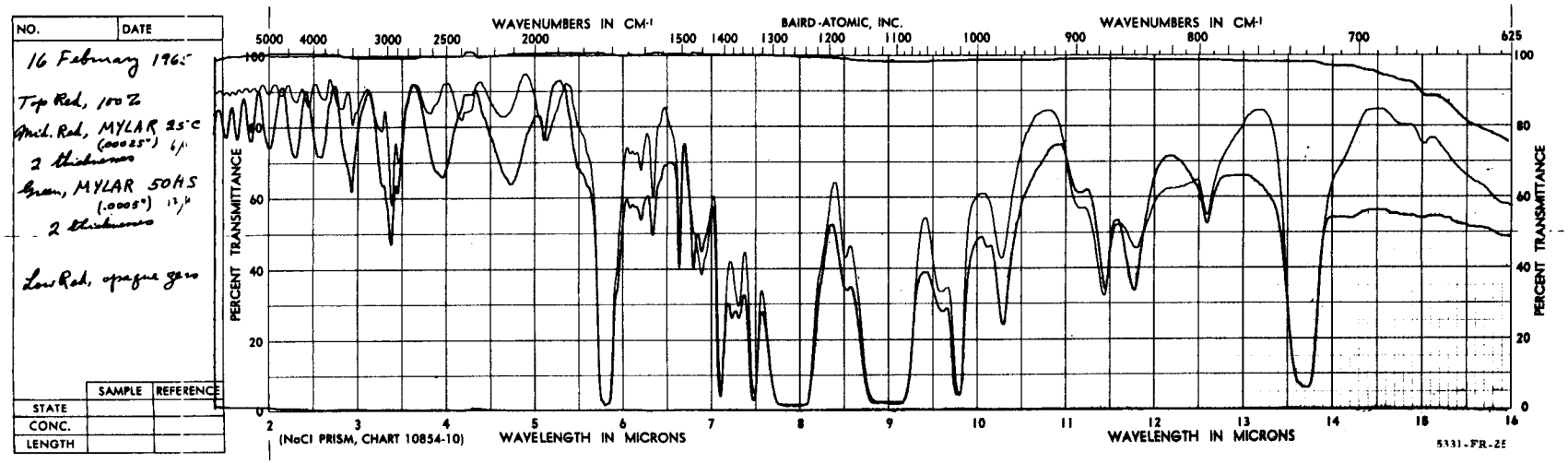


Figure 12. Measured Transmittance Curve of Uncoated Mylar Membrane, 2 to 16 Microns

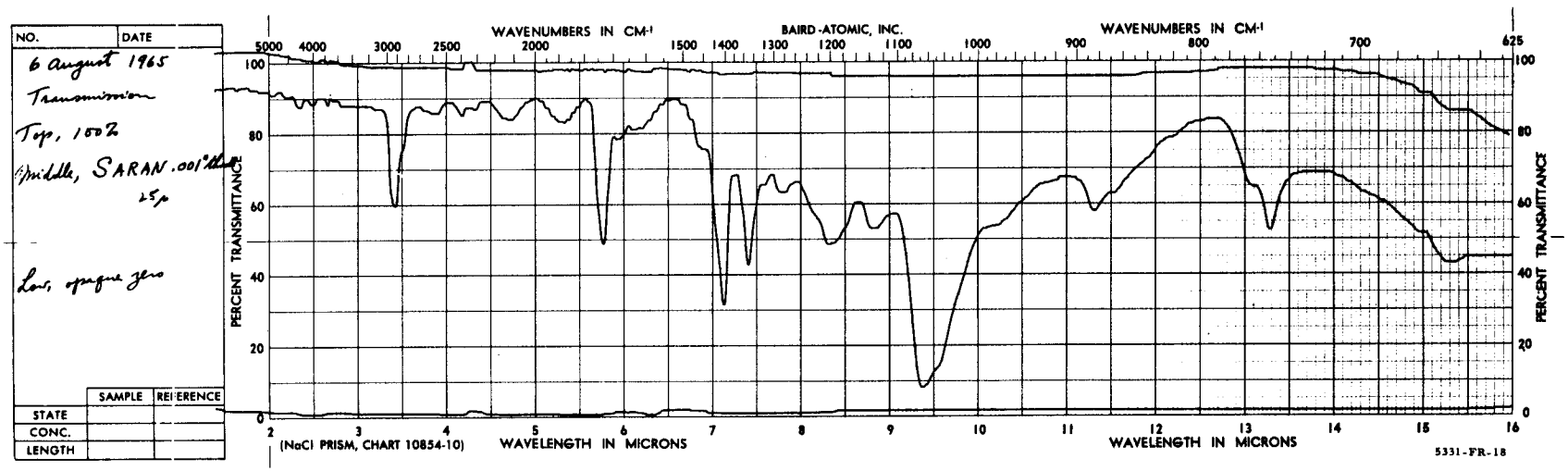


Figure 13. Measured Transmittance Curve of Uncoated Saran Membrane, 2 to 16 Microns

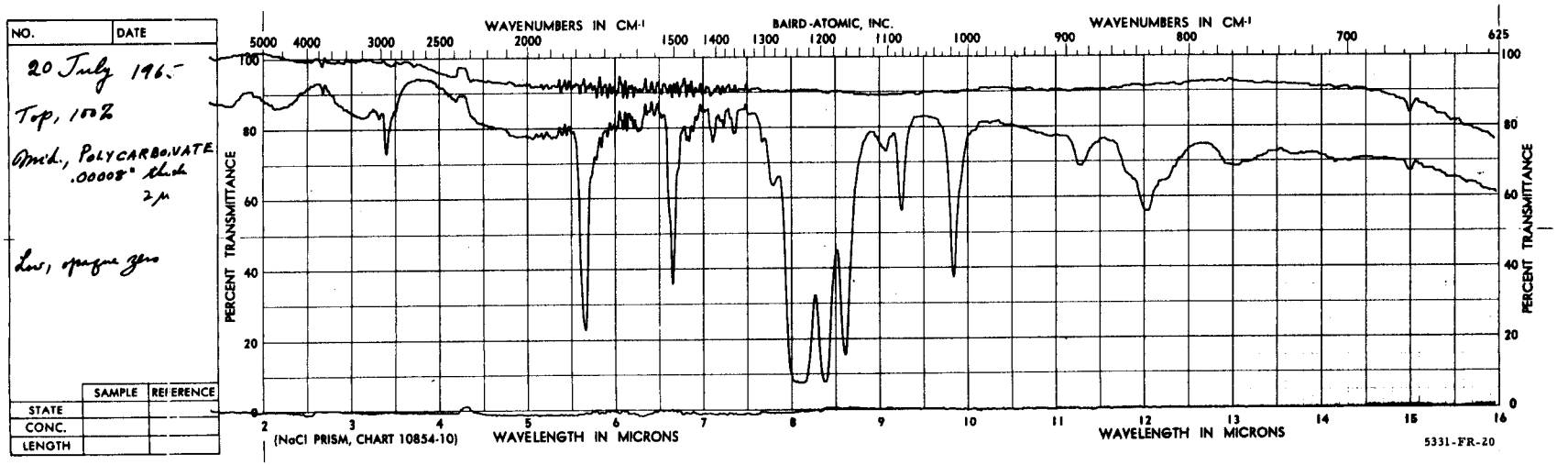


Figure 14. Measured Transmittance Curve of Uncoated Polycarbonate Membrane, 2 to 16 Microns

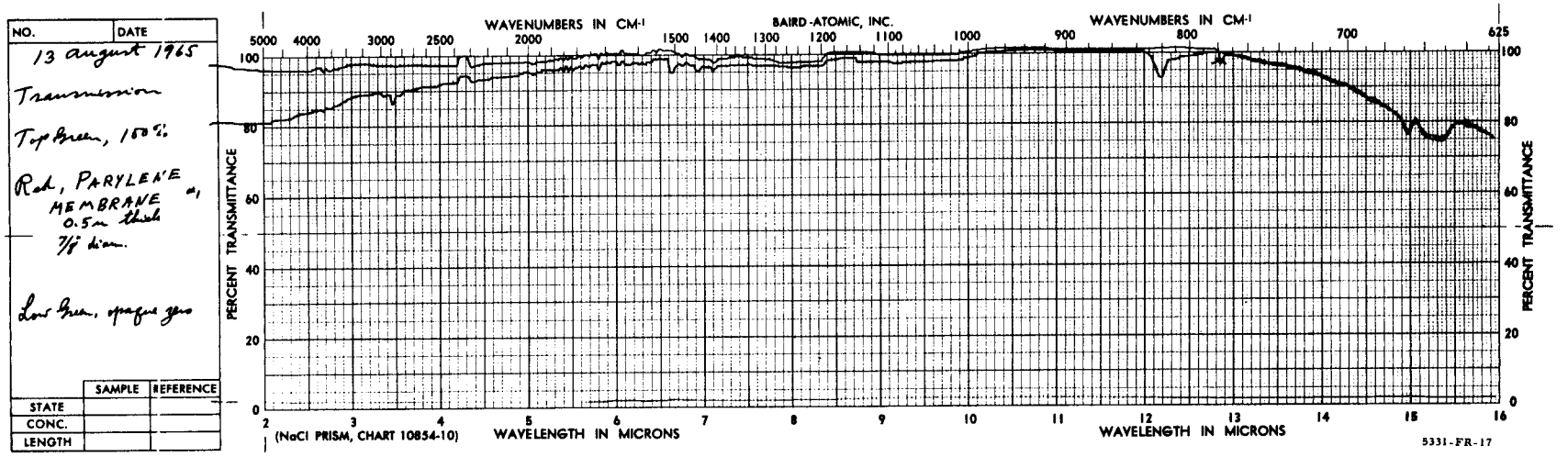


Figure 15. Measured Transmittance Curve of Uncoated Parylene Membrane, 2 to 16 Microns

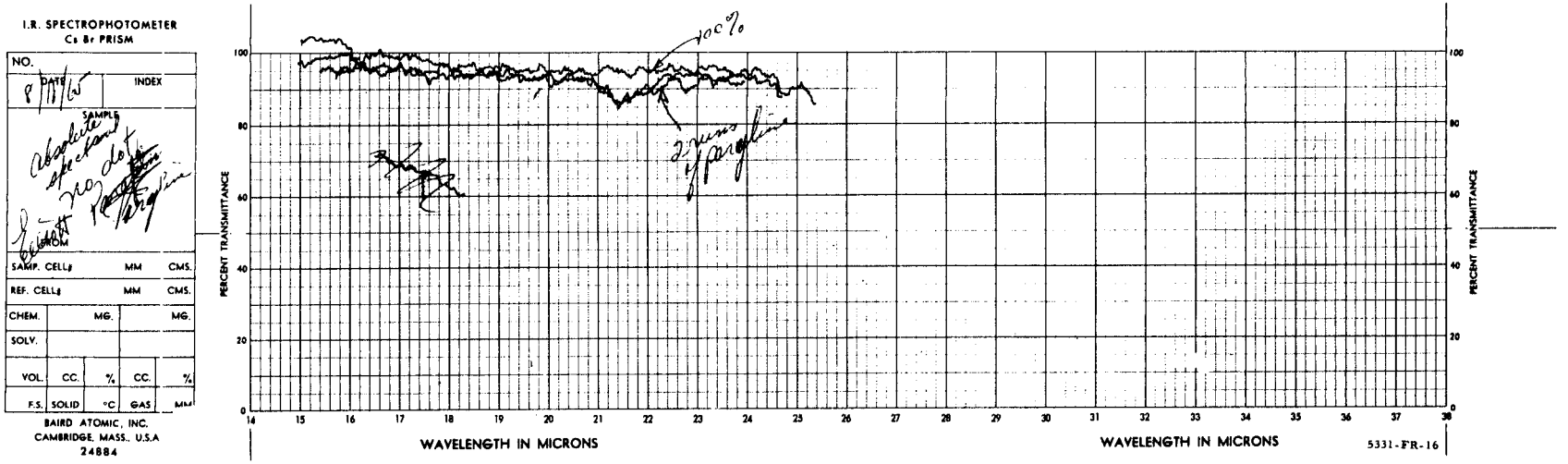


Figure 16. Measured Transmittance Curve of Uncoated Parylene Membrane, 14 to 30 Microns

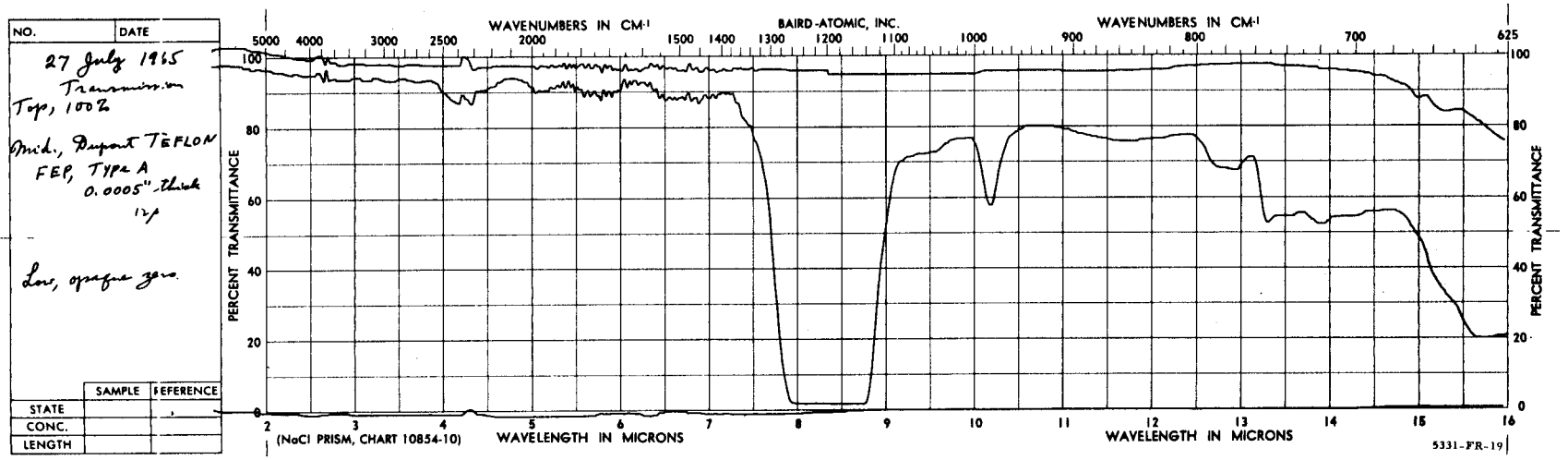


Figure 17. Measured Transmittance Curve of Uncoated Teflon Membrane, 2 to 16 Microns

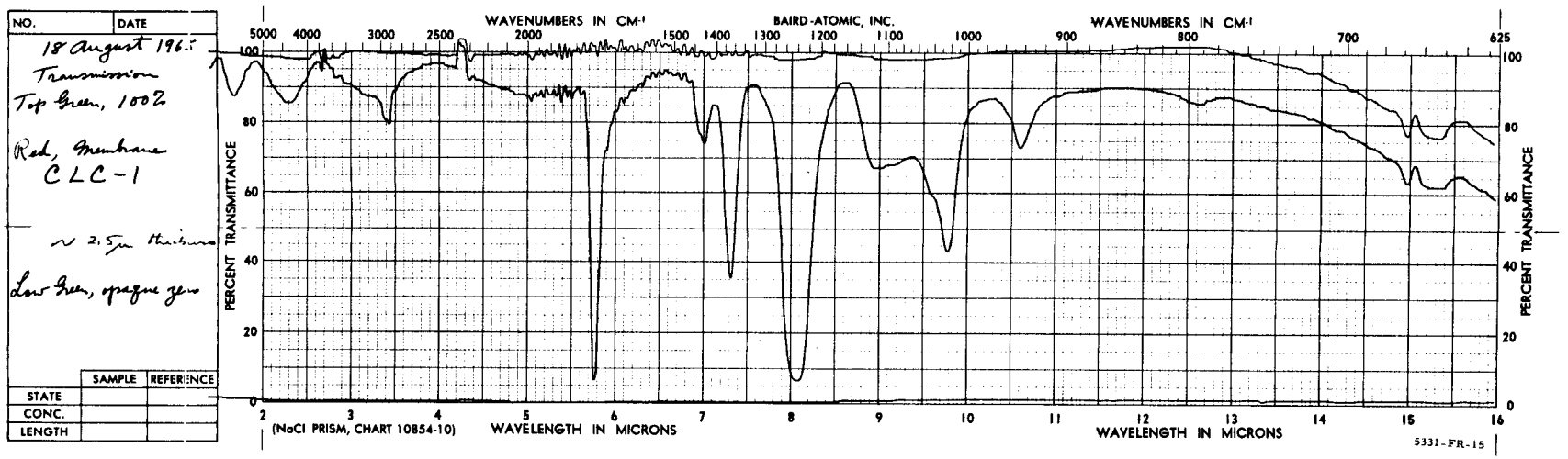


Figure 18. Measured Transmittance Curve of Uncoated Polyvinyl Acetate Membrane, 2 to 16 Microns

5. COMPUTER PROGRAM

5.1 Metallic Films and Coating Constructions Considered and Results

Metallic films which were investigated theoretically were aluminum, silver, germanium, cobalt, antimony, and bismuth (figures 19 and 20). In the case of metallic layers, electromagnetic theory provides a means for calculating reflectance R, transmittance T, and absorption A.

Three general types of coating construction were investigated:

Metallic
Two- and three-layer dielectric on single substrate
Three-layer dielectric between two layers of substrate

Curves are shown for germanium-chiolite (figure 21) and zinc sulfide-germanium-zinc sulfide (figure 22). Also, the same curves are plotted against the limits imposed by the requirements of the specification (figure 23).

The germanium-chiolite combination performed better theoretically than the zinc sulfide-germanium-zinc sulfide. However, chiolite, which is a sodium-aluminum fluoride compound, is attacked by moisture. For this reason, the sandwich type of construction (the third type mentioned above) was conceived so that the substrate material would serve to protect the coating. In all cases, the calculations showed that the addition of the second layer of substrate material resulted in a loss of beam splitter efficiency (figures 24 through 29).

5.2 Sources of Data

Sources of data used in computing were

a. American Institute of Physics Handbook

b. JOSA, Vol. 53 (1963) Harris and Piper; "Optical and Electrical Properties of Bismuth Deposits"

c. JOSA, Vol. 54 (1964) Harris and Corrigan "Optical and Electrical Properties of Antimony"

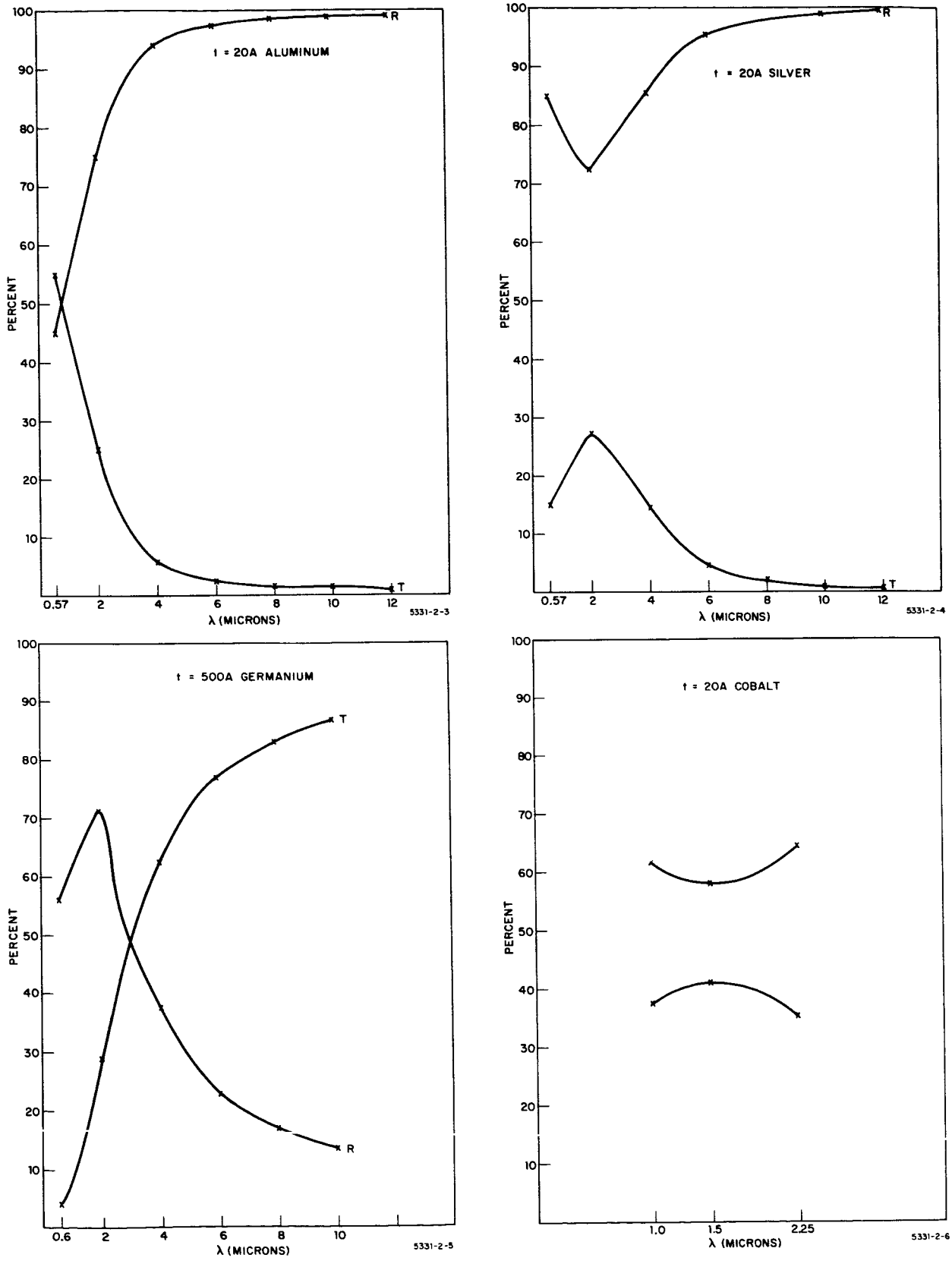
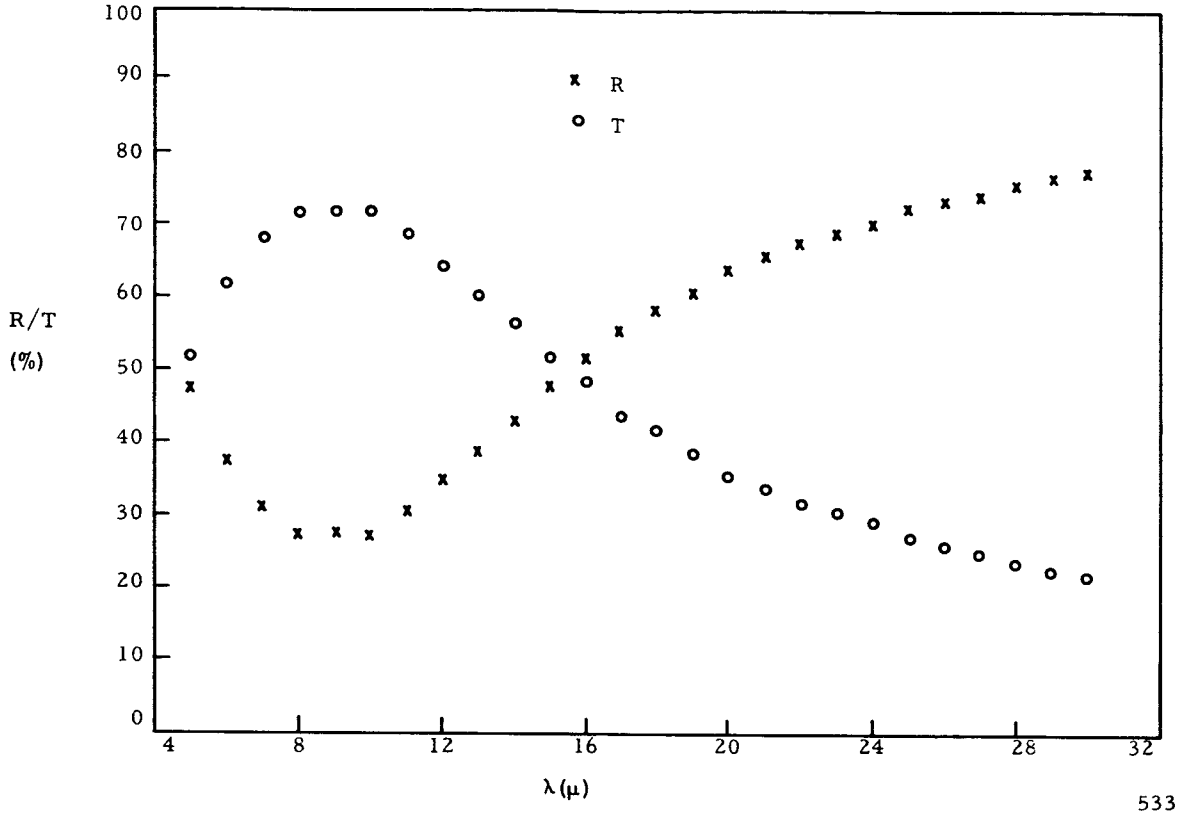
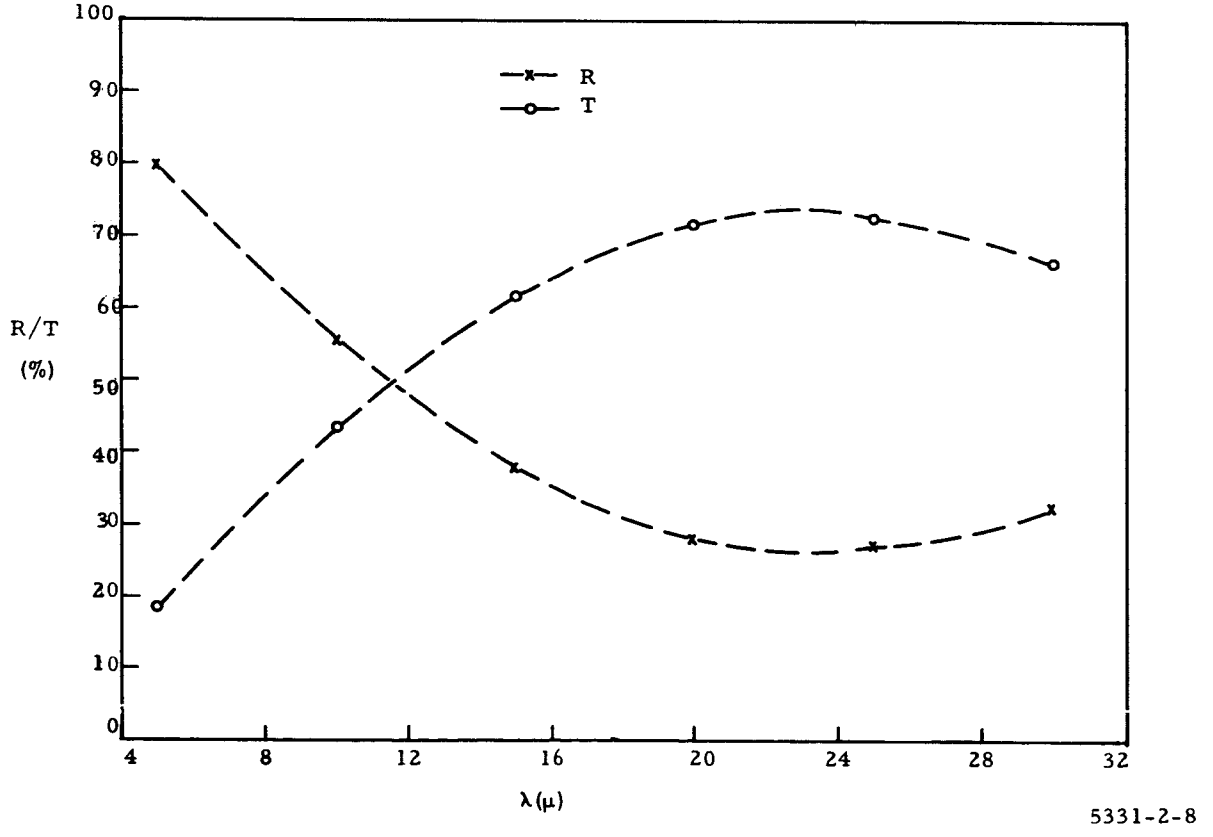


Figure 19. Calculated Reflectance and Transmittance of Aluminum, Silver, Germanium, and Cobalt



A. 140A Layer of Antimony



B. 500A Layer of Bismuth

Figure 20. Calculated Reflectance and Transmittance of Antimony and Bismuth

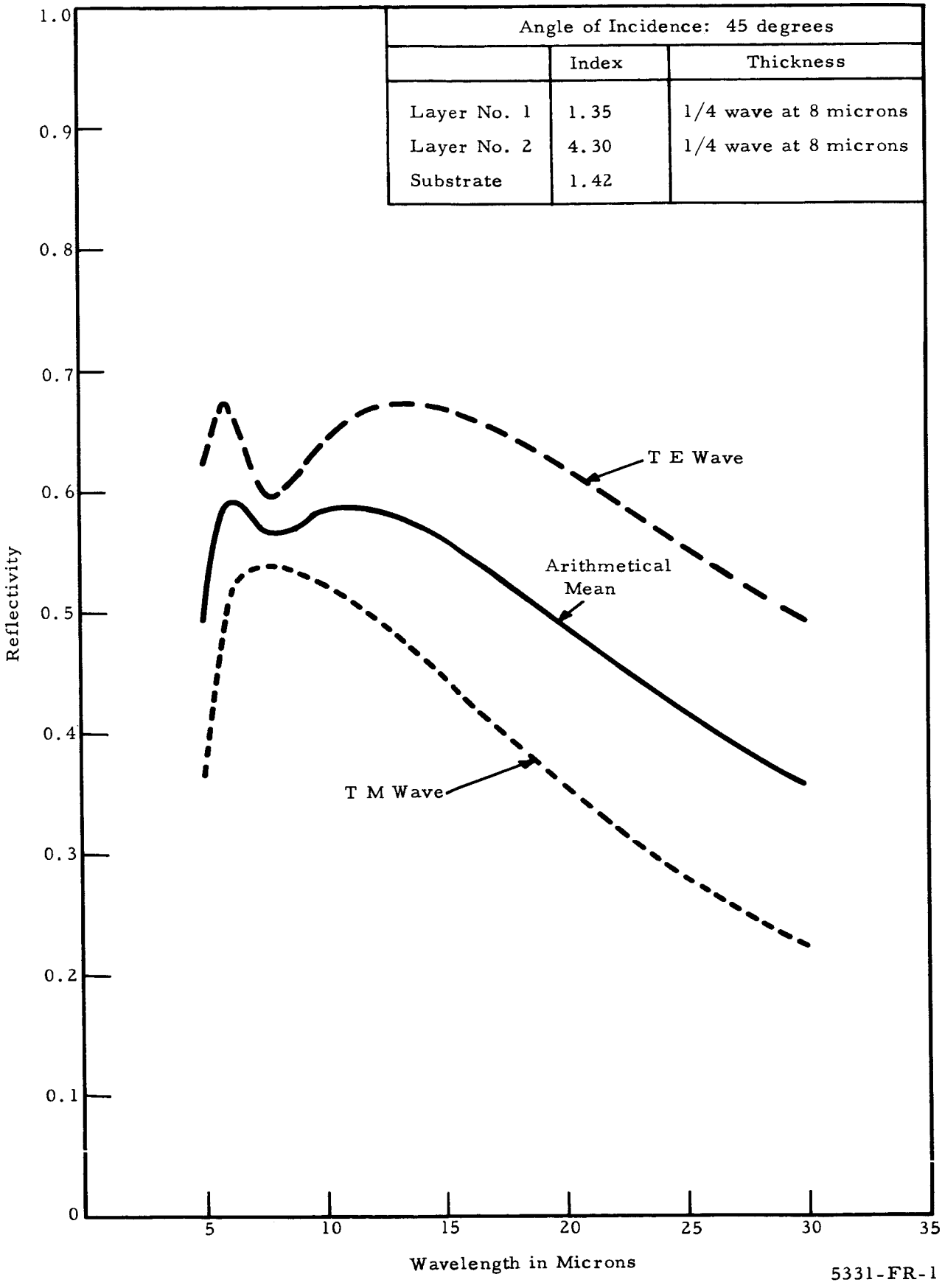


Figure 21. Calculated Reflectance of 2-Layer Dielectric Film, One-Quarter Wave Each of Germanium and Chiolite

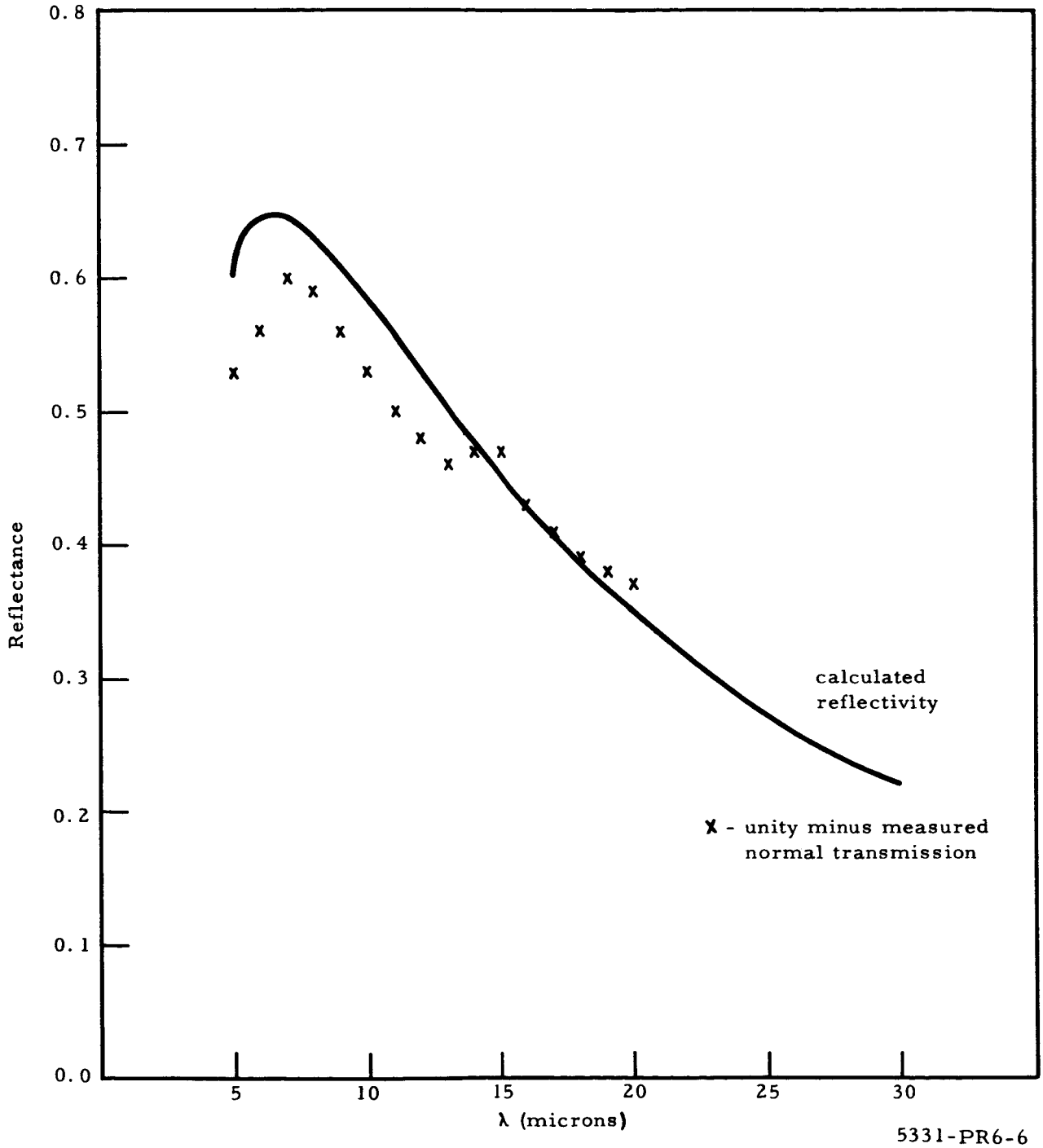


Figure 22. Calculated and Measured Performance of Zinc Sulfide-Germanium-Zinc Sulfide Coating

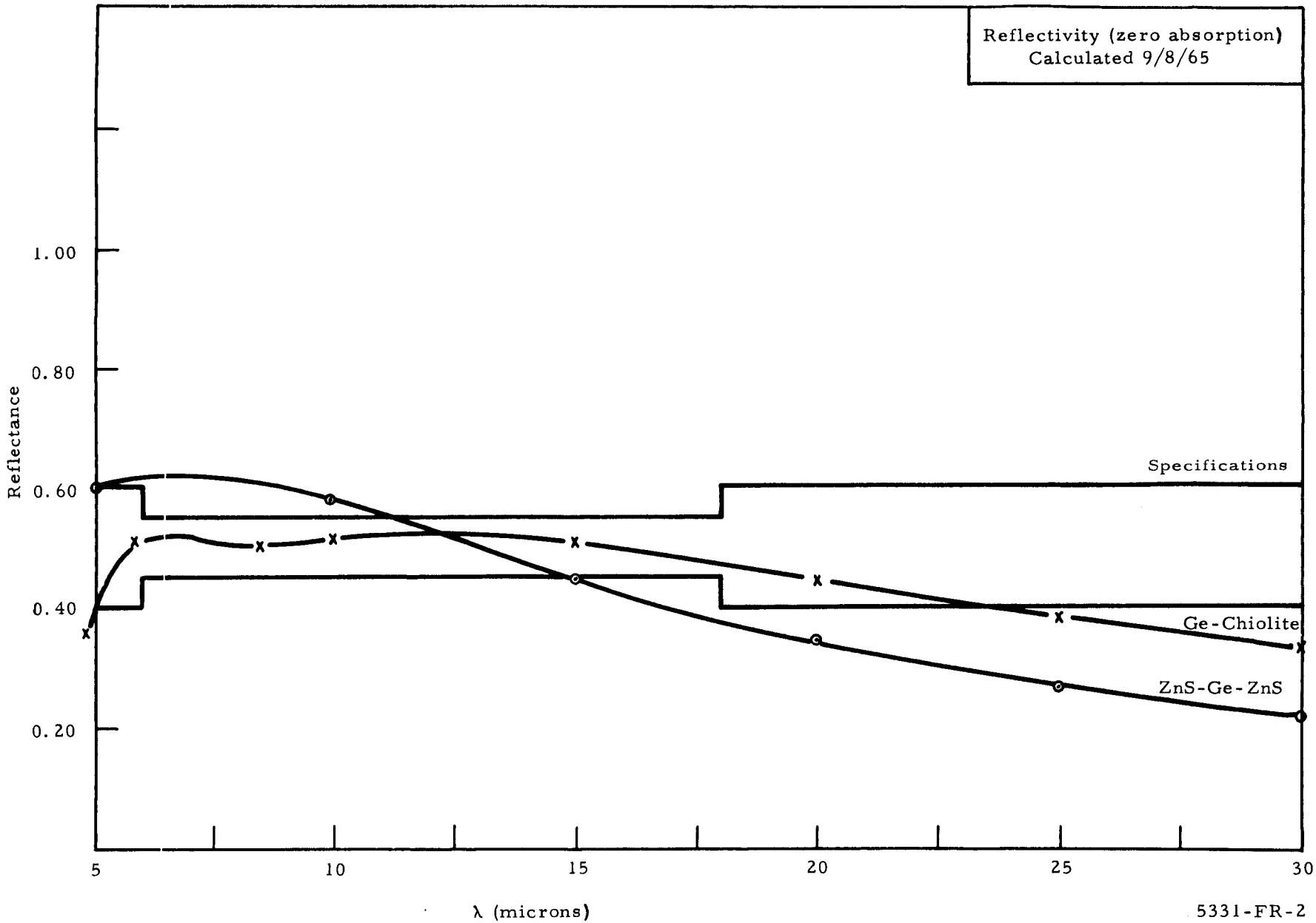


Figure 23. Calculated Reflectance of the 2-Layer and 3-Layer Dielectric Films Against Specification Limits

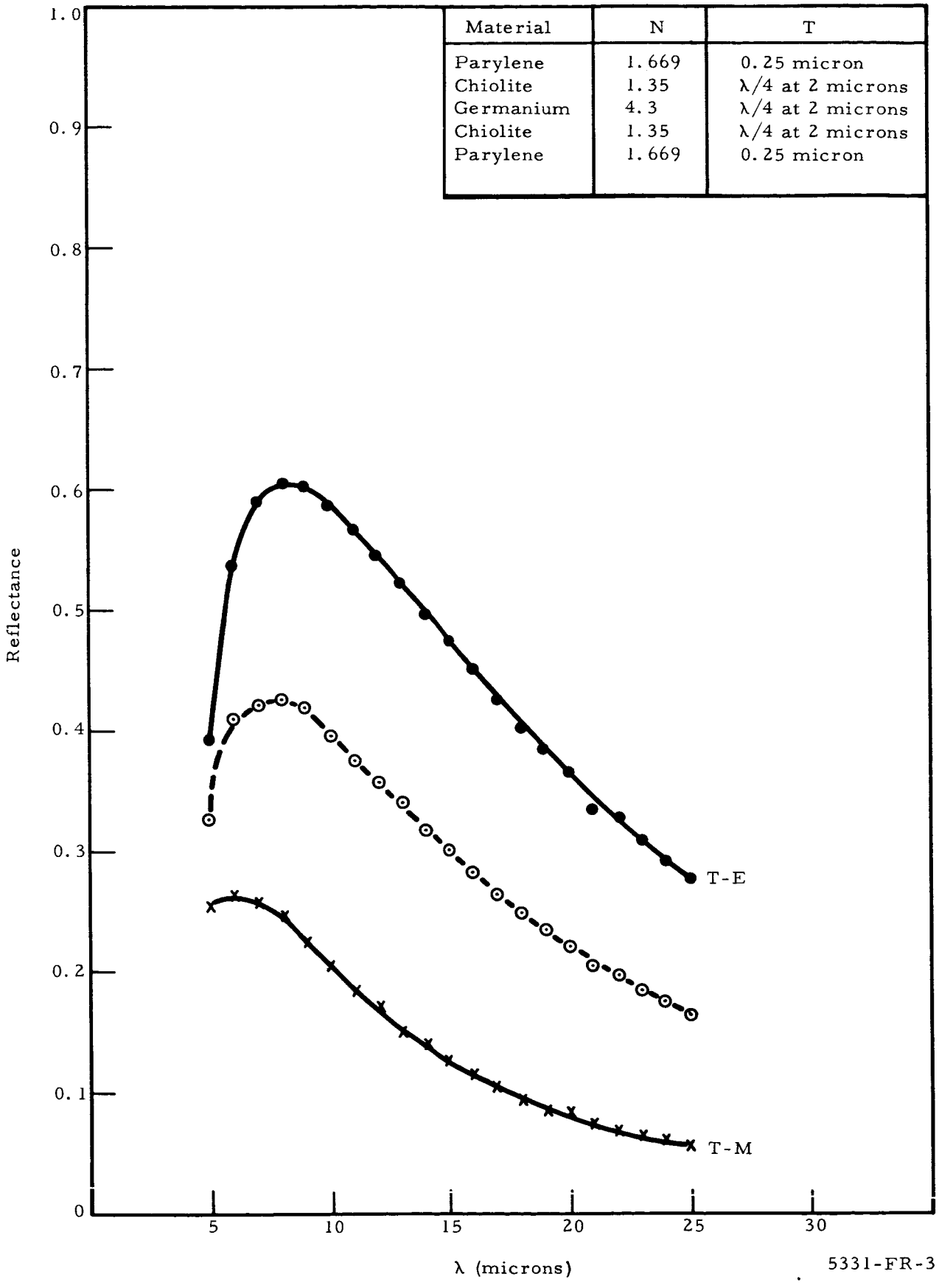


Figure 24. Calculated Reflectance of 3-Layer Dielectric Film, One-Quarter Wavelength at 2 Microns Each of Chiolite-Germanium-Chiolite, Between Layers of Parylene

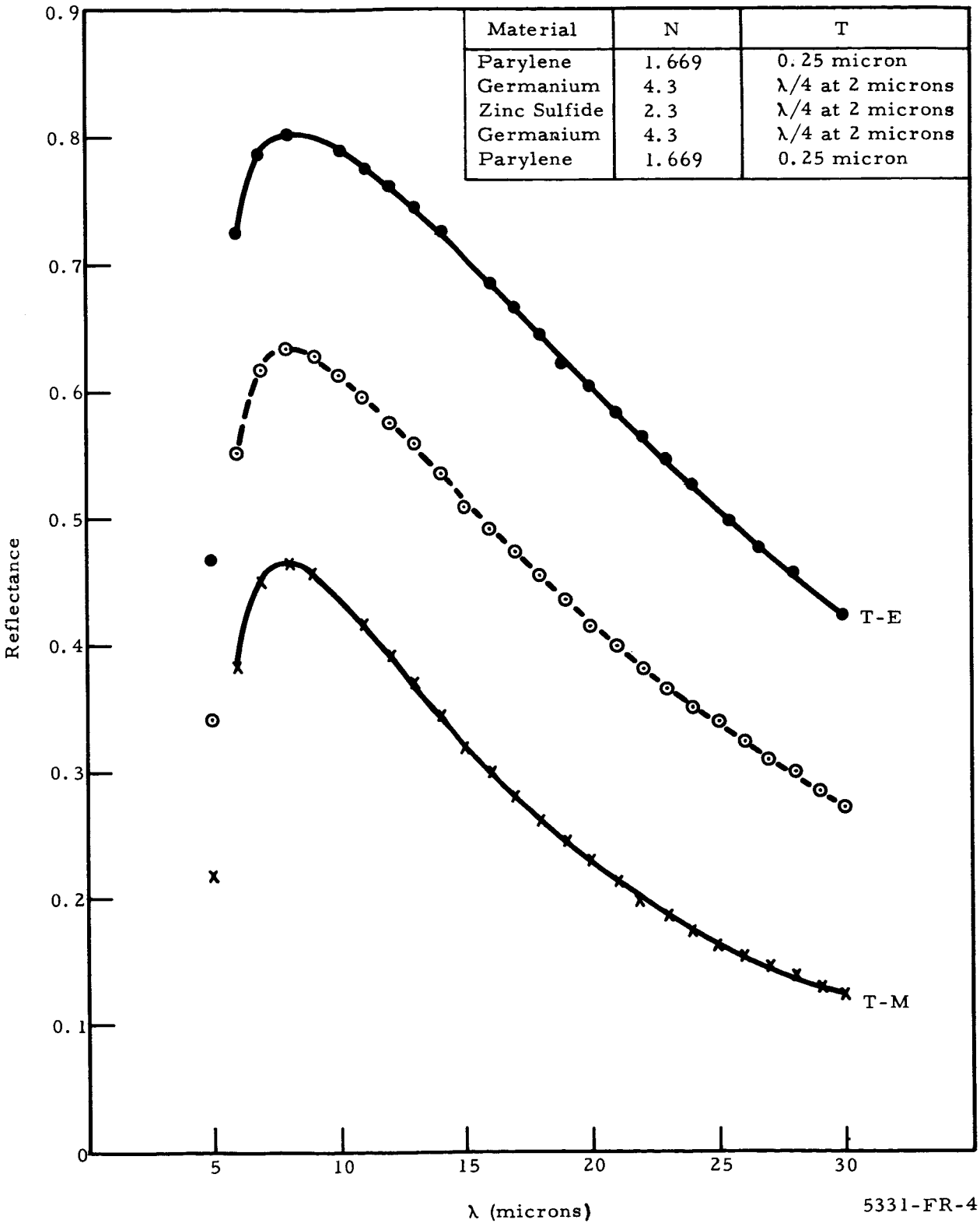


Figure 25. Calculated Reflectance of 3-Layer Dielectric Film, One-Quarter Wavelength at 2 Microns Each of Germanium-Zinc Sulfide-Germanium, Between Layers of Parylene

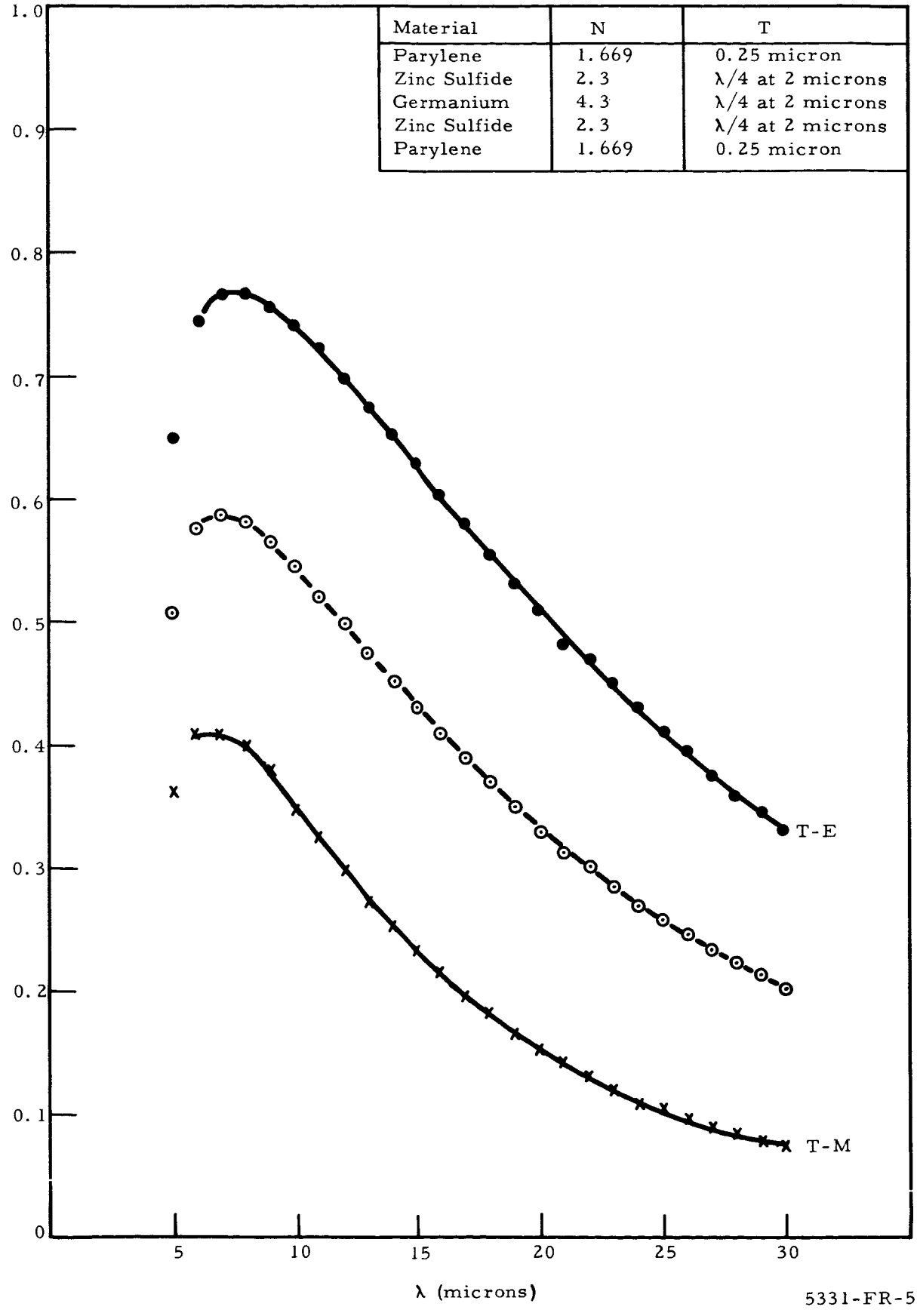


Figure 26. Calculated Reflectance of 3-Layer Dielectric Film, One-Quarter Wavelength at 2 Microns Each of Zinc Sulfide-Germanium-Zinc Sulfide, Between Layers of Parylene

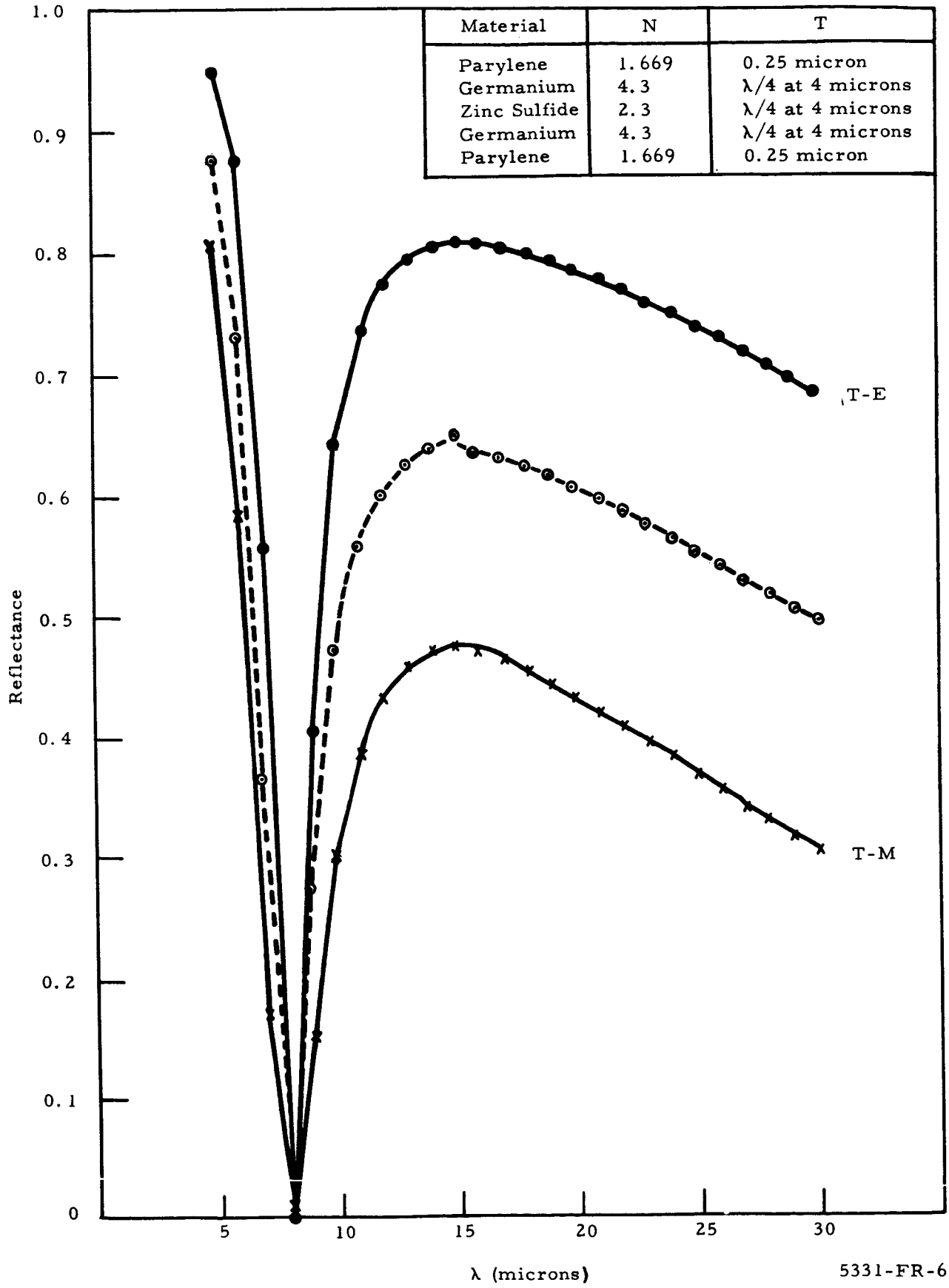


Figure 27. Calculated Reflectance of 3-Layer Dielectric Film, One-Quarter Wavelength at 4 Microns Each of Germanium-Zinc Sulfide-Germanium, Between Layers of Parylene

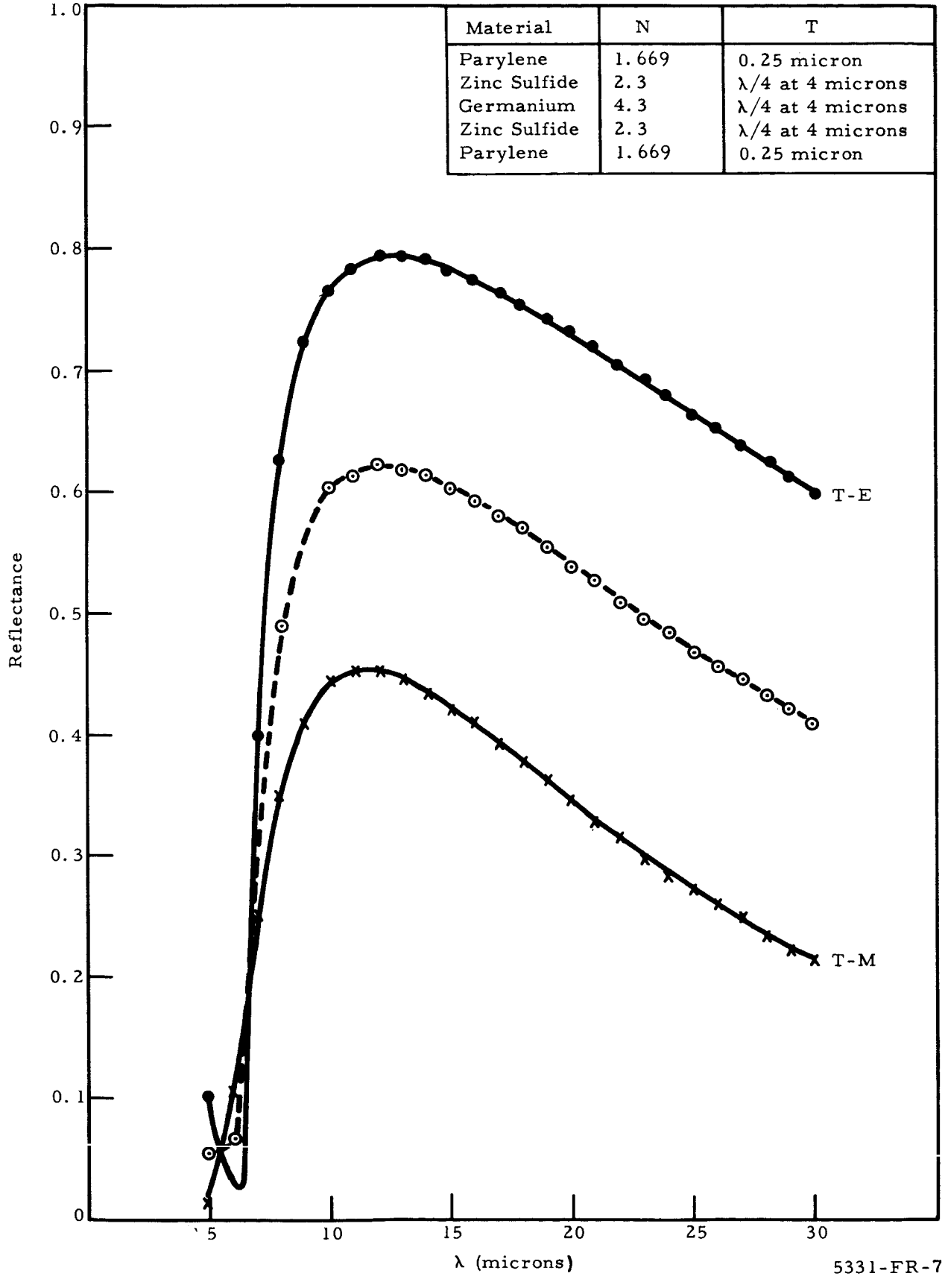


Figure 28. Calculated Reflectance of 3-Layer Dielectric Film, One-Quarter Wavelength at 4 Microns Each of Zinc Sulfide-Germanium-Zinc Sulfide, Between Layers of Parylene

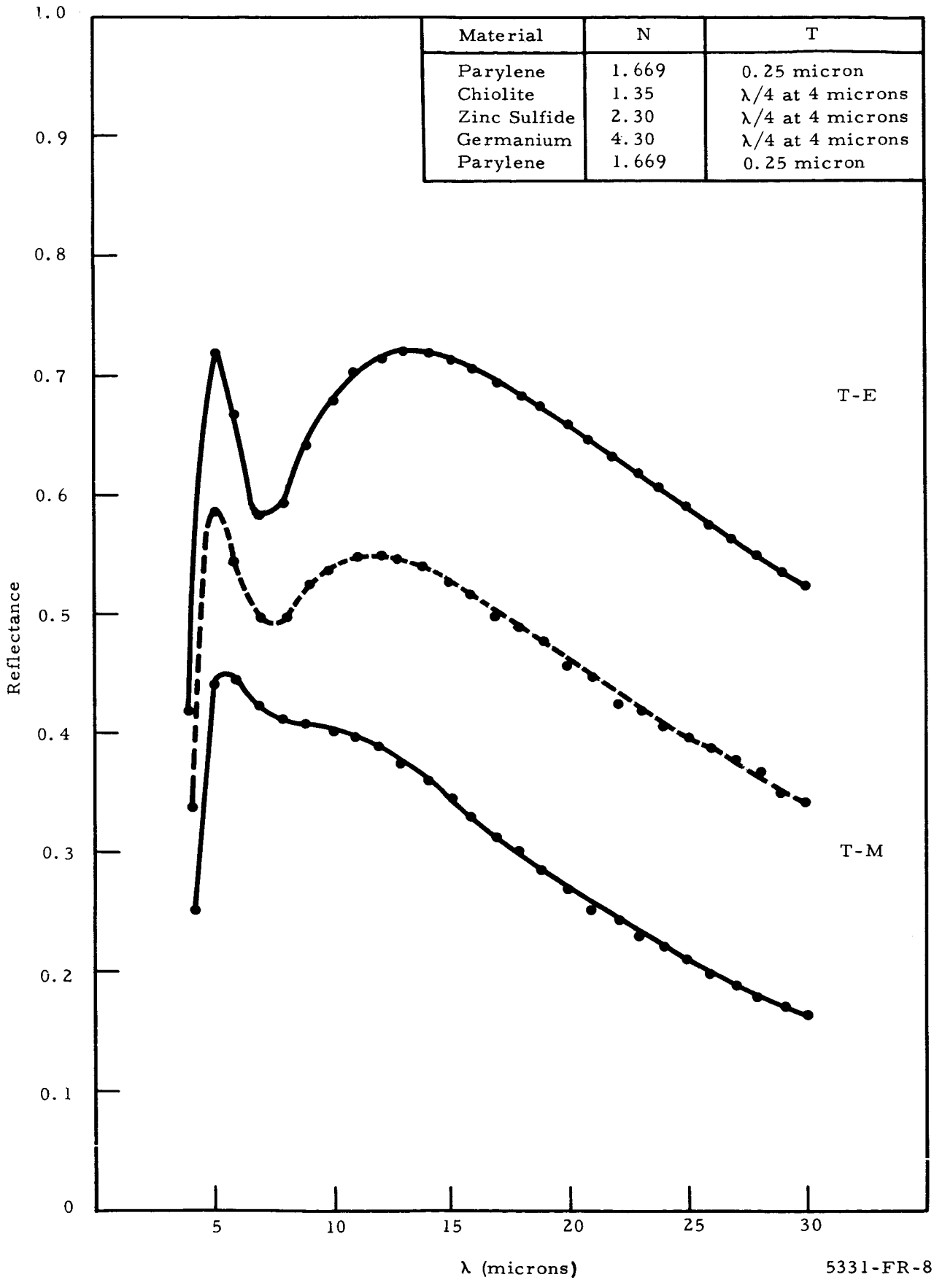


Figure 29. Calculated Reflectance of 3-Layer Dielectric Film, One-Quarter Wavelength at 4 Microns each of Chiolite-Zinc Sulfide-Germanium

5.3 Formulae

5.3.1 Dielectric Films

The reflection and transmission coefficients for a dielectric multilayer film can be found in a variety of ways. From a computational point of view, it is advantageous to choose a method of solution which gives the answer in the form of a recursion formula that is, the coefficients for n + 1 layers are written as functions of the coefficients for n layers and the characteristics of the (n + 1)st layer. A program has been written for the IBM 1620 computer that can handle films with a maximum of 62 layers. The input consists of one card specifying the number of layers, angle of incidence, and mixed and final wavelengths as well as the wavelength increment which are desired. This is followed by as many cards as there are media, each card containing the index and thickness of one layer, with the exception of the cards corresponding to the medium from which the ray originates and the substrate, which contain zeros as thicknesses. The output is punched on cards which are subsequently listed on an off-line printer. For films containing some five or six layers, the computations at one wavelength are executed in about 8 seconds. If the number of layers is increased to about 12, the corresponding time is tripled.

The equations used to solve the problem are:

a. Define a complex reflection coefficient Υ which equals the ratio of reflected to incident electric vectors

$$\Upsilon = \frac{1 - t_1}{1 + t_1}$$

b. The quantities t_i are given by the recursion relations

$$t_n = \frac{\Upsilon_{n+1} \cos \delta_n - i \sin \delta_n}{\cos \delta_n - i \Upsilon_{n+1} \sin \delta_n}$$

where

$$\left(\tau_j\right)_{TE} = \frac{n_j \cos \theta_j}{n_{j-1} \cos \theta_{j-1}}$$

$$\left(\tau_j\right)_{TM} = \frac{n_{j-1} \cos \theta_j}{n_j \cos \theta_{j-1}}$$

$$\delta_j = \left(\frac{2\pi}{\lambda}\right) n_j d_j \cos \theta_j$$

and

d_j = thickness of the j-th layer

λ = wavelength (in vacuum) of incident light

n_j = index of the j-th layer

θ_j = angle of refraction in j-th layer

TE and TM denote transverse electric and transverse magnetic, respectively.

To determine Υ, start from the known values of t_{n+1} and δ_n [substrate] and then, by successive application of the recursion relation, eventually determine t_1. The recursion formula is used as many times as there are layers in the film.

c. The reflection coefficient for a particular wavelength is found by multiplying Υ and its complex conjugate,

$$\mathcal{R} = \Upsilon\Upsilon^*$$

d. The fraction of the incident intensity that is transmitted is obtained by subtracting $\mathcal{R}$ from unity.

5.3.2 Absorbing Layers

The situation is quite different when the layers have appreciable absorption. The complexity of the problem is increased manyfold and a computer program capable of handling many layers can yield results in a reasonable time only for the very large machines. With Baird-Atomic's small computer it is not possible to go much beyond two or three layers. However, since it was felt that the attractiveness of the absorbing layers was the possibility of making a beam splitter of maximum simplicity, only the case corresponding to an absorbing thin film deposited on a nonabsorbing substrate was programmed. The steps needed to find $\mathcal{R}$ and T, the reflection and transmission coefficients, are summarized below.

a.

$$\mathcal{R} = \Upsilon\Upsilon^*$$

$$T = \left(\frac{n_3 \cos \theta_3}{n_1 \cos \theta_1}\right) tt^*$$

b.

$$\Upsilon = \frac{\Upsilon_1 + \Upsilon_2 t_M \tau_M}{1 - \Upsilon_2 \rho_1 t_M \tau_M}$$

$$t = \frac{t_1 t_2 t_M}{1 - \Upsilon_2 \rho_2 t_M \tau_M}$$

$$\frac{\sin \theta_3}{\sin \theta_1} = \frac{n_1}{n_3}$$

c. For a TE wave, for example,

$$t_1 = \frac{2n_1 \cos \theta_1}{n_2 p + n_1 \cos \theta_1 + i n_2 q}$$

$$t_2 = \frac{2n_2 (p + i q)}{n_2 p + n_3 \cos \theta_3 + i n_2 q}$$

$$t_M = \tau_M = e^{-\frac{2\pi\rho d}{\lambda}(n_2 \sin j + n_2 k_2 \cos j)} e^{i\frac{2\pi\rho d}{\lambda}(n_2 k_2 \sin j - n_2 \cos j)}$$

$$\Upsilon_1 = -\rho_1 = -\frac{n_2 p - n_1 \cos \theta_1 + i n_2 q}{n_2 p + n_1 \cos \theta_1 + i n_2 q}$$

$$\Upsilon_2 = -\rho_2 = \frac{n_2 p - n_3 \cos \theta_3 + i n_2 q}{n_2 p + n_3 \cos \theta_3 + i n_2 q}$$

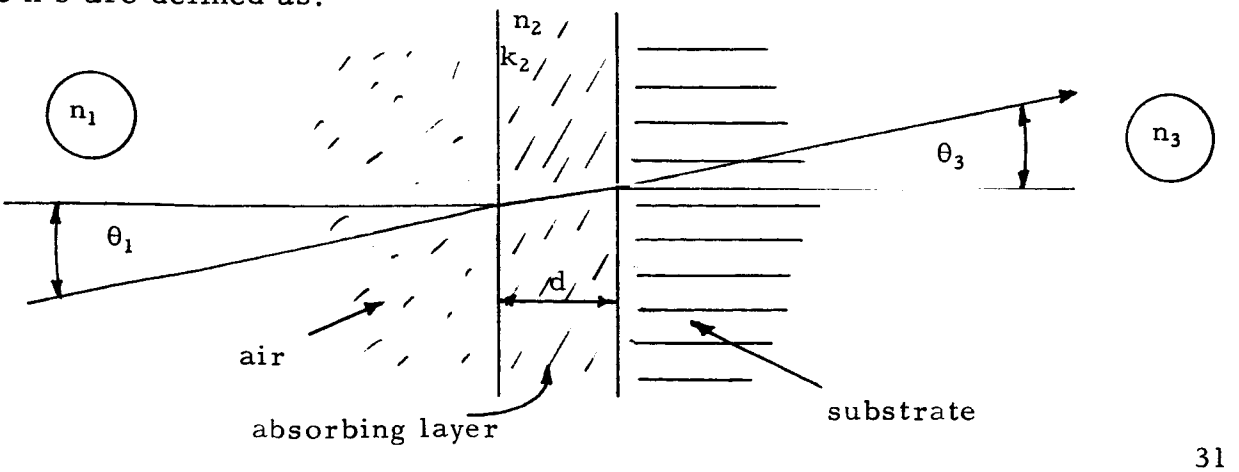
$$p = \rho (\cos j - k_2 \sin j)$$

$$q = \rho (\sin j + k_2 \cos j)$$

$$\rho^4 = \left[1 - \frac{n_1^2 \sin^2 \theta_1}{n_2^2 (1+k_2^2)}\right]^2 + \frac{4n_1^2 k_2^2 \sin^2 \theta_1}{n_2^2 (1+k_2^2)^2}$$

$$\tan 2j = \frac{2n_1^2 k_2 \sin^2 \theta_1}{n_2^2 (1+k_2^2)^2 - n_1^2 (1-k_2^2) \sin^2 \theta_1}$$

The n's are defined as:



6. FABRICATION AND TESTING OF COATED PELLICLES

6.1 Methods

In order to facilitate handling in the coating and measurement processes, a number of two-inch diameter aluminum rings were made for trial purposes. These were used to develop coating techniques and to measure transmittance and reflectance of experimental coatings.

6.2 Results

Coatings applied and measured were:

Aluminum on cellulose nitrate (figures 30, 31 and 32)

Antimony on cellulose nitrate (figures 33, 34, 35, and 36)

Bismuth on cellulose nitrate (figures 36 and 37)

Antimony and bismuth on cellulose nitrate (figures 38 and 39)

Germanium on cellulose nitrate (figures 40 and 41)

Germanium and cryolite on calcium fluoride (figures 42 and 43)

Germanium and zinc sulfide on cellulose nitrate (figures 44 and 45)

Zinc Sulfide-Germanium-Zinc Sulfide on cellulose nitrate (figures 46 and 47)

Zinc Sulfide-Germanium-Zinc Sulfide on Parylene (figures 48 and 49)

Evaporation of aluminum, antimony, and bismuth was done from a tungsten spiral. Evaporation rates and shuttering of the source were adjusted to prevent breakage of the pellicle. Density of the coating was monitored with a photocell and galvanometer with scale reading corrections for the infrared region.

A series of experiments was run on cellulose nitrate pellicles to determine optimum deposition rates for germanium, cryolite, and zinc sulfide. Germanium is troublesome in that it deposits in a state of compression. Zinc sulfide deposits in a state of tension*. It was hoped that the combination of germanium

*Ref. B & L Quarterly Technical Report No. 1, Contract DA-44-009-eng-2117, "Studies of Semiconductor Films"

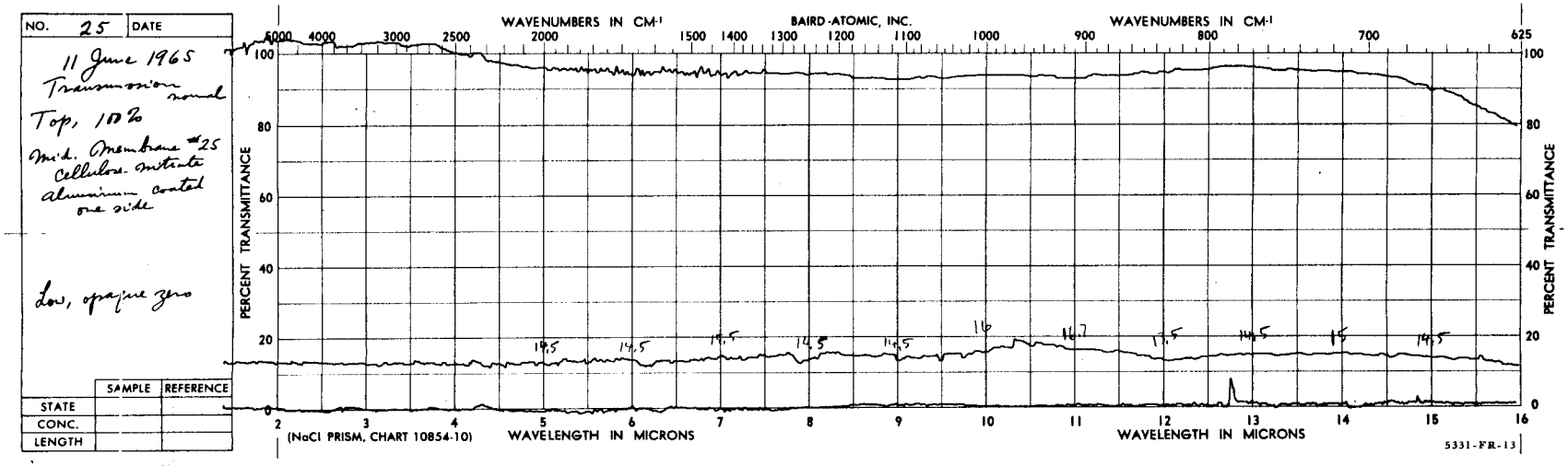


Figure 30. Measured Transmittance Curve of Cellulose Nitrate Membrane, Aluminum Coated, 2 to 16 Microns

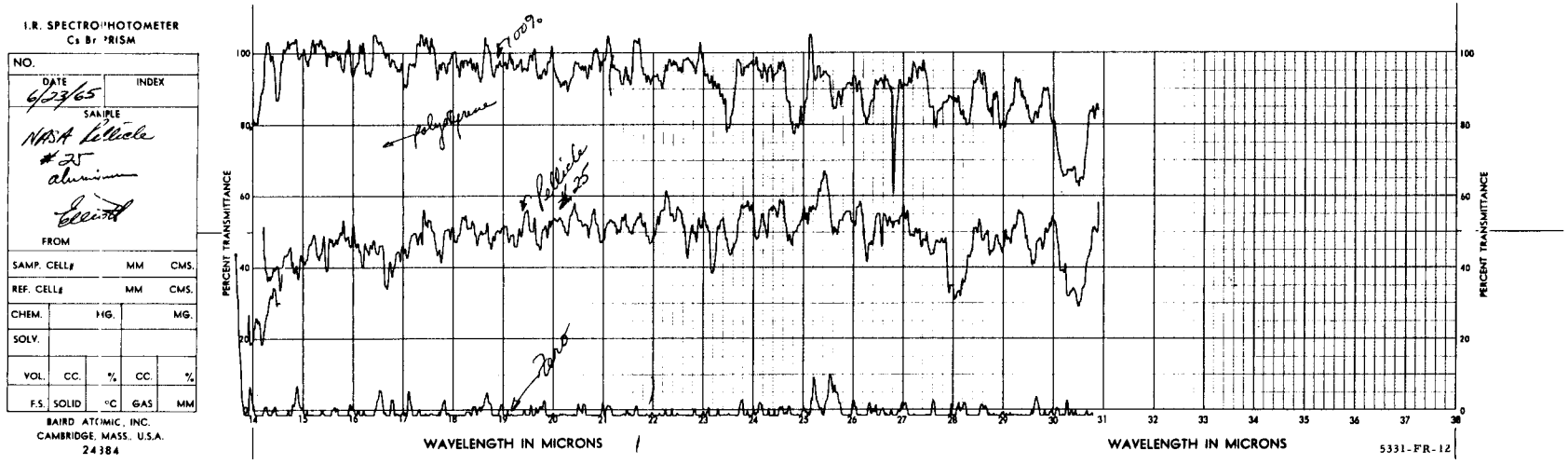


Figure 31. Measured Transmittance Curve of Cellulose Nitrate Membrane, Aluminum Coated, 14 to 30 Microns

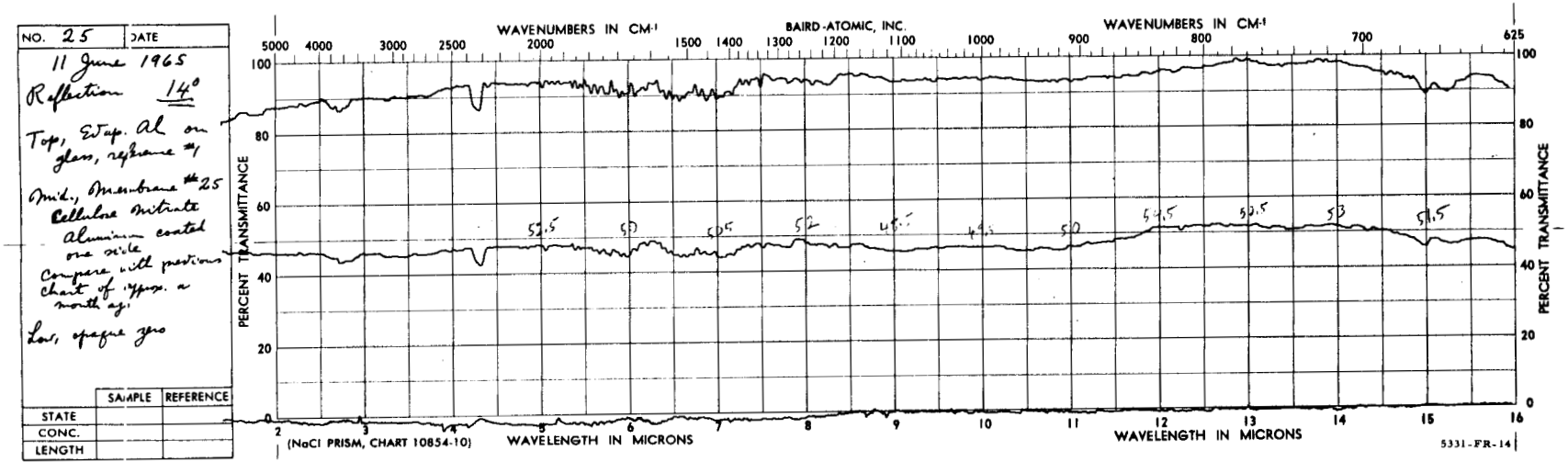


Figure 32. Measured Reflectance Curve of Cellulose Nitrate Membrane, Aluminum Coated, 2 to 16 Microns

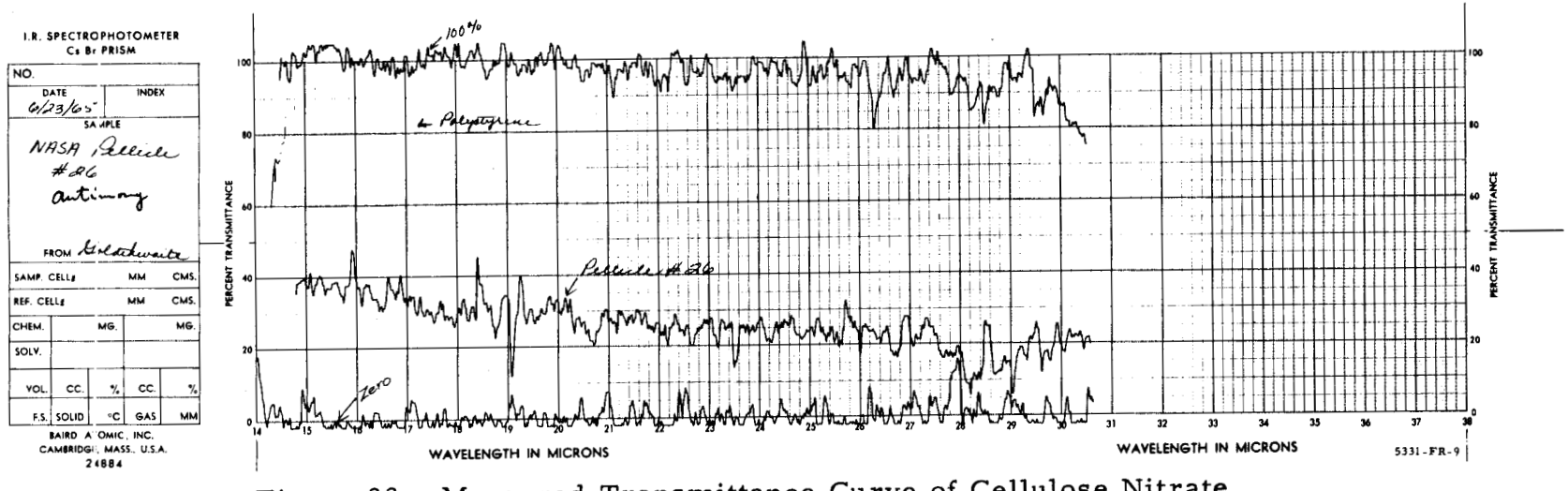


Figure 33. Measured Transmittance Curve of Cellulose Nitrate Membrane, Antimony Coated, 14 to 30 Microns

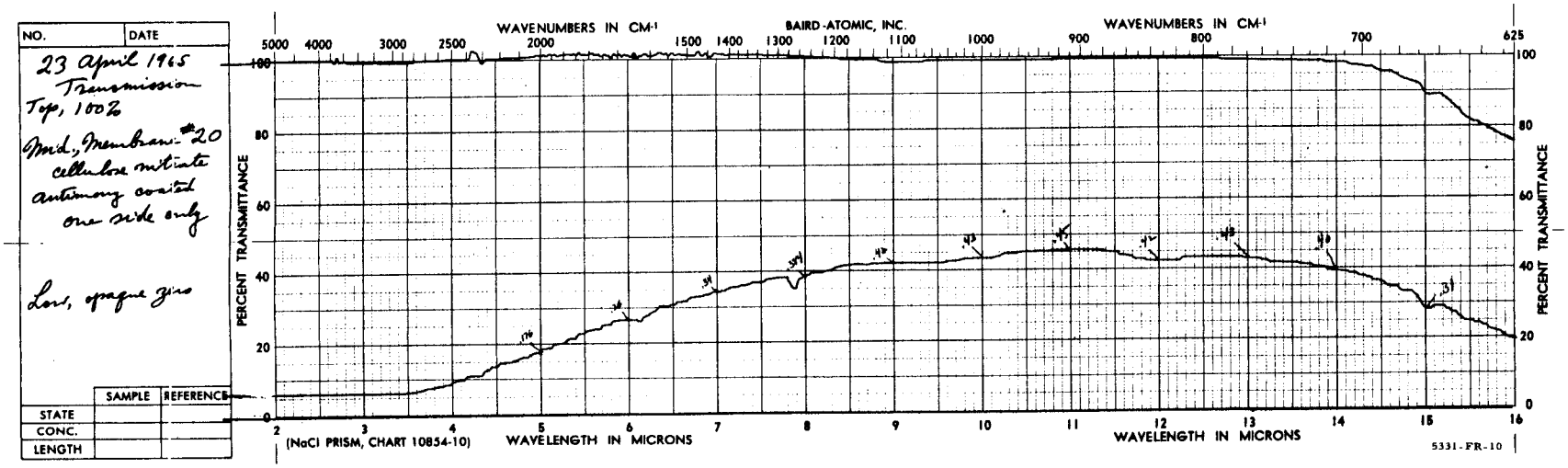


Figure 34. Measured Transmittance Curve of Cellulose Nitrate Membrane, Antimony Coated, 2 to 16 Microns

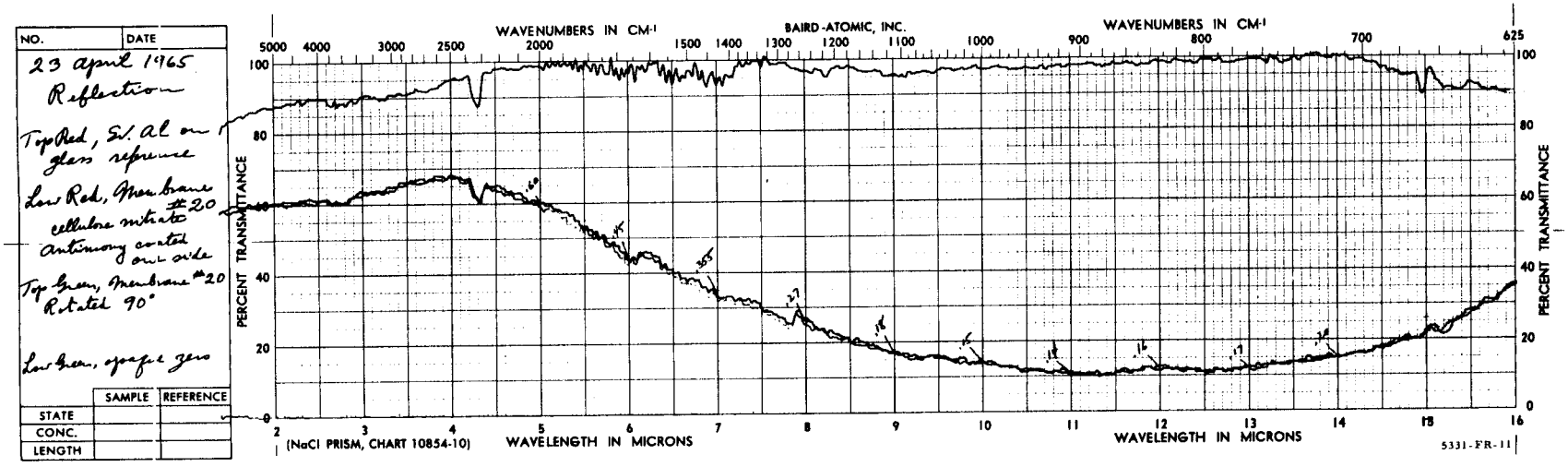


Figure 35. Measured Reflectance Curve of Cellulose Nitrate Membrane, Antimony Coated, 2 to 16 Microns

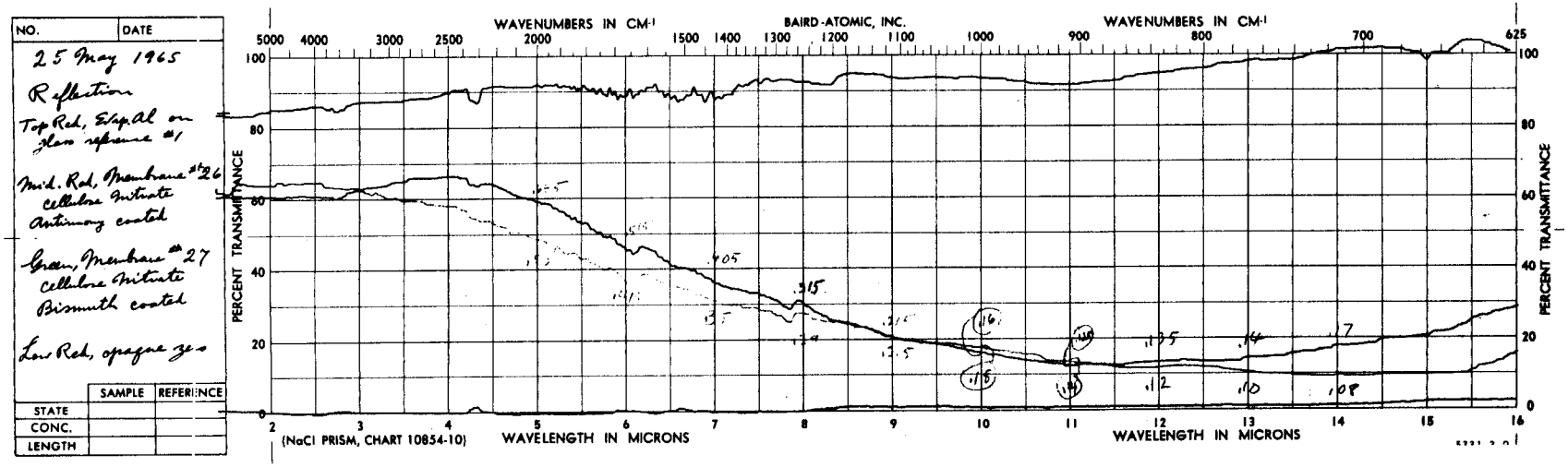


Figure 36. Measured Reflectance Curve of Cellulose Nitrate Membrane, Antimony Coated (No. 26) and Bismuth Coated (No. 27), 2 to 16 Microns

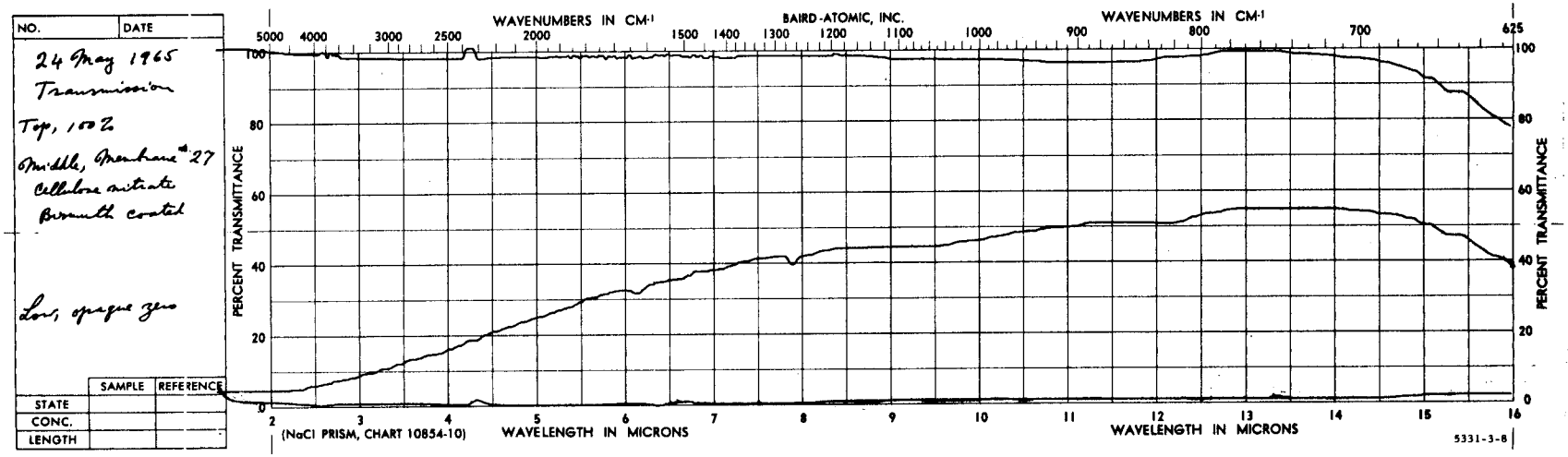


Figure 37. Measured Transmittance Curve of Cellulose Nitrate Membrane, Bismuth Coated, 2 to 16 Microns

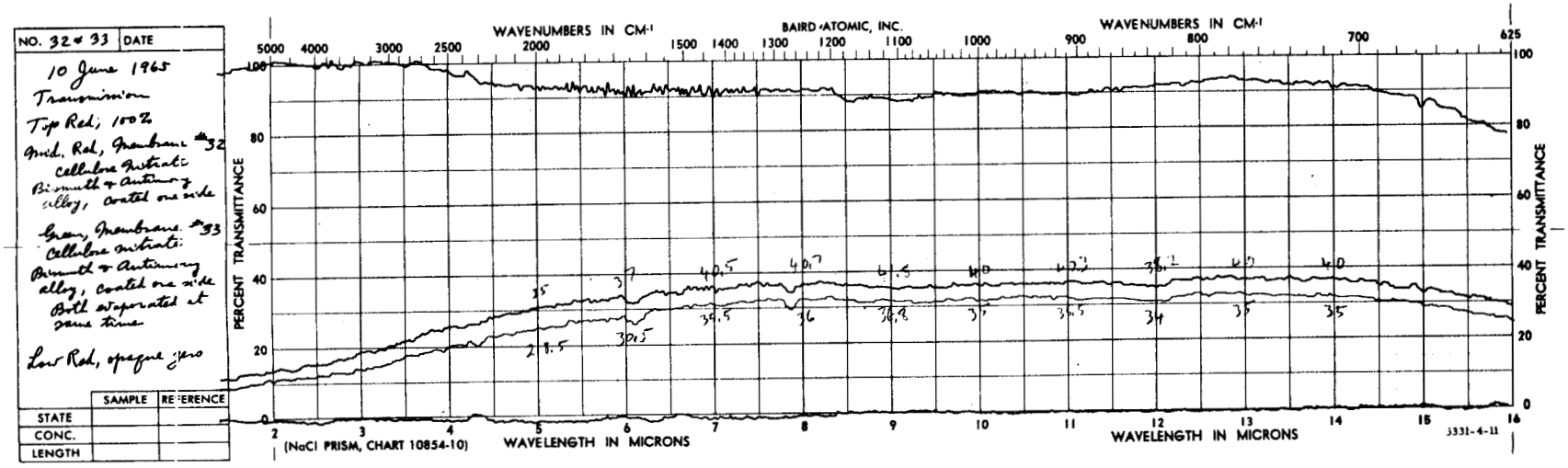


Figure 38. Measured Transmittance Curve of Cellulose Nitrate Membrane, Bismuth and Antimony Coated, 45-Degree Incidence, 2 to 16 Microns

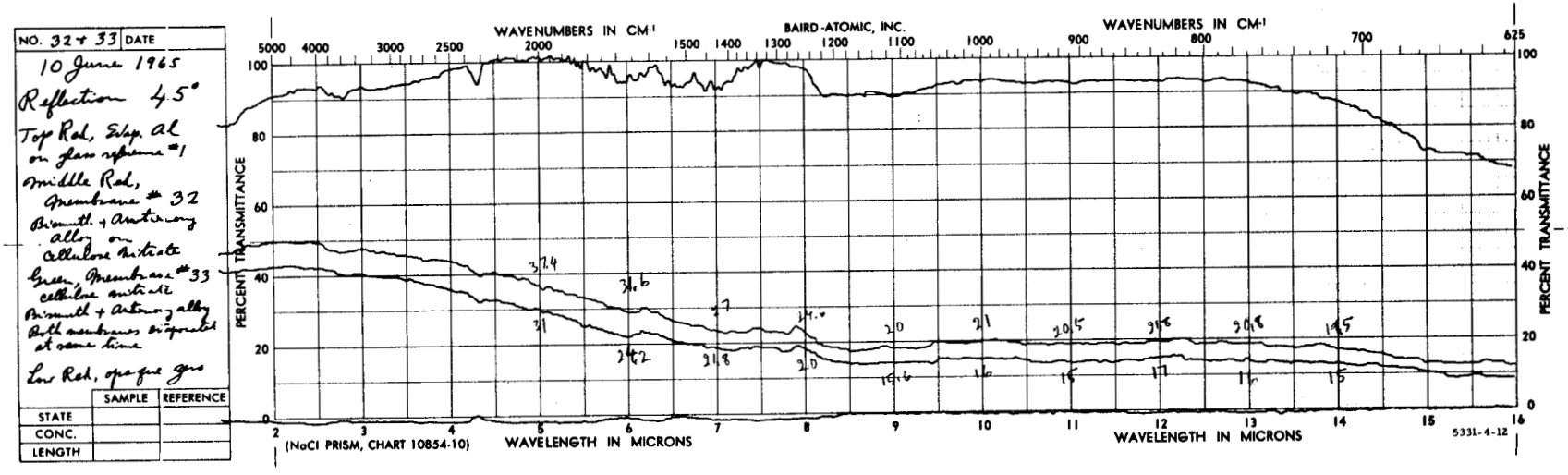


Figure 39. Measured Reflectance Curve of Cellulose Nitrate Membrane, Bismuth and Antimony Coated, 45-Degree Incidence, 2 to 16 Microns

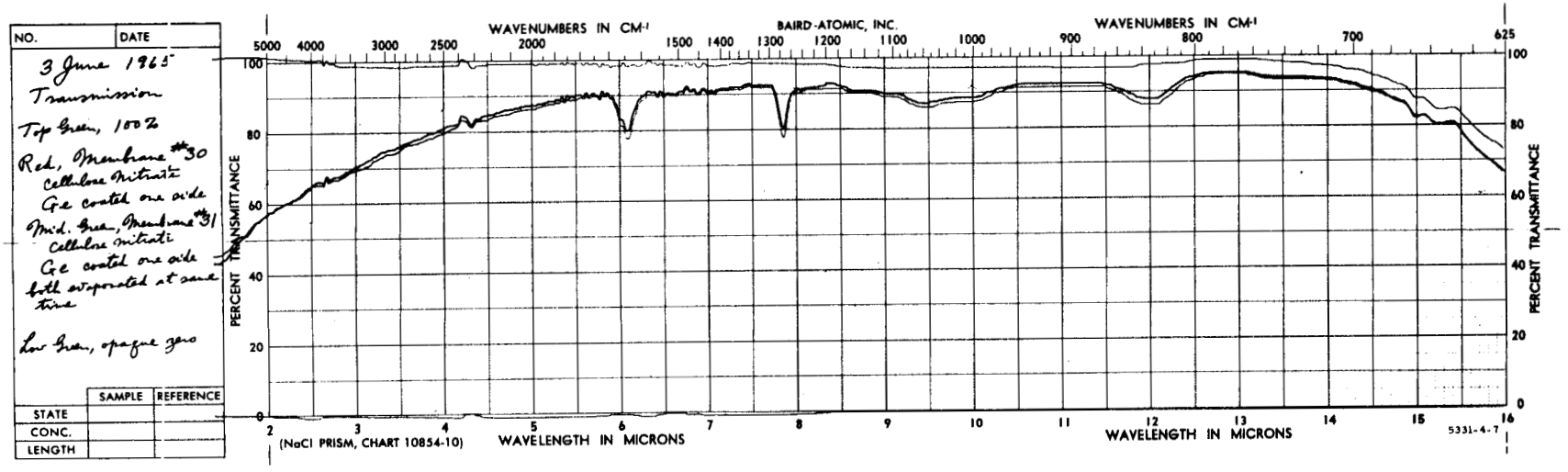


Figure 40. Measured Transmittance Curve of Cellulose Nitrate Membrane, Germanium Coated, 2 to 16 Microns

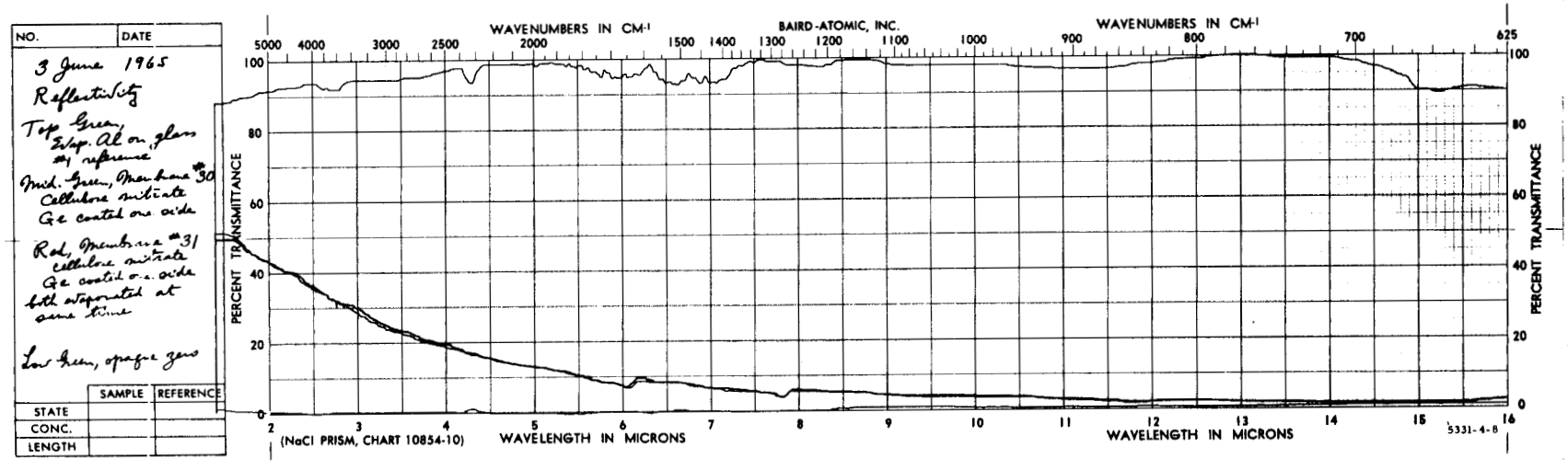


Figure 41. Measured Reflectance Curve of Cellulose Nitrate Membrane, Germanium Coated, 2 to 16 Microns

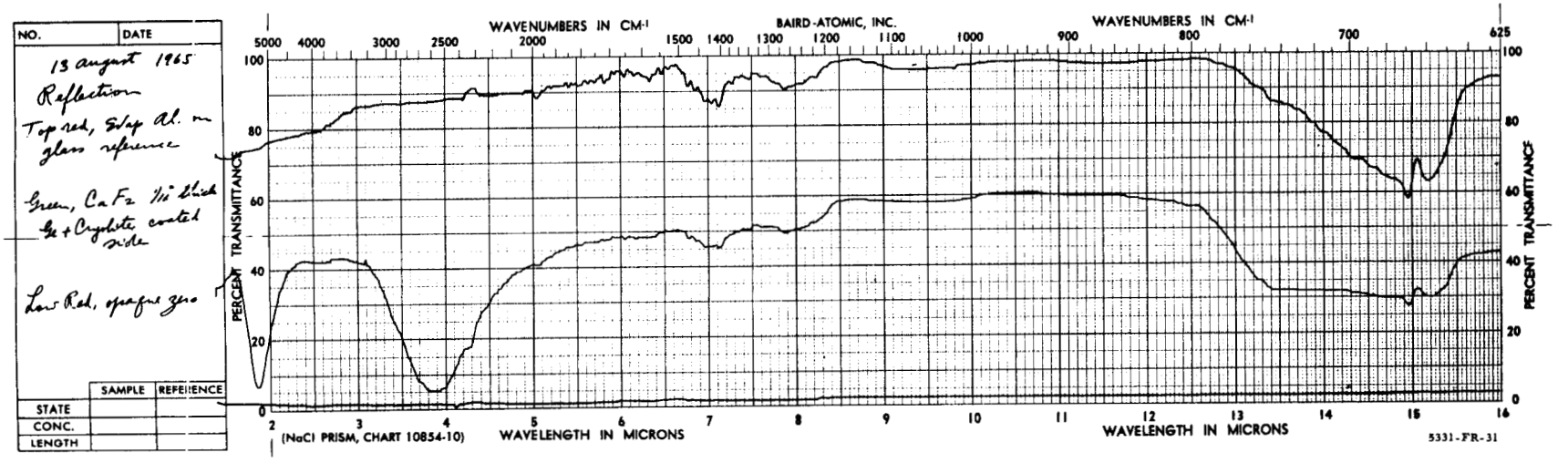


Figure 42. Measured Transmittance Curve of Calcium Fluoride, Germanium and Cryolite Coated, 2 to 16 Microns

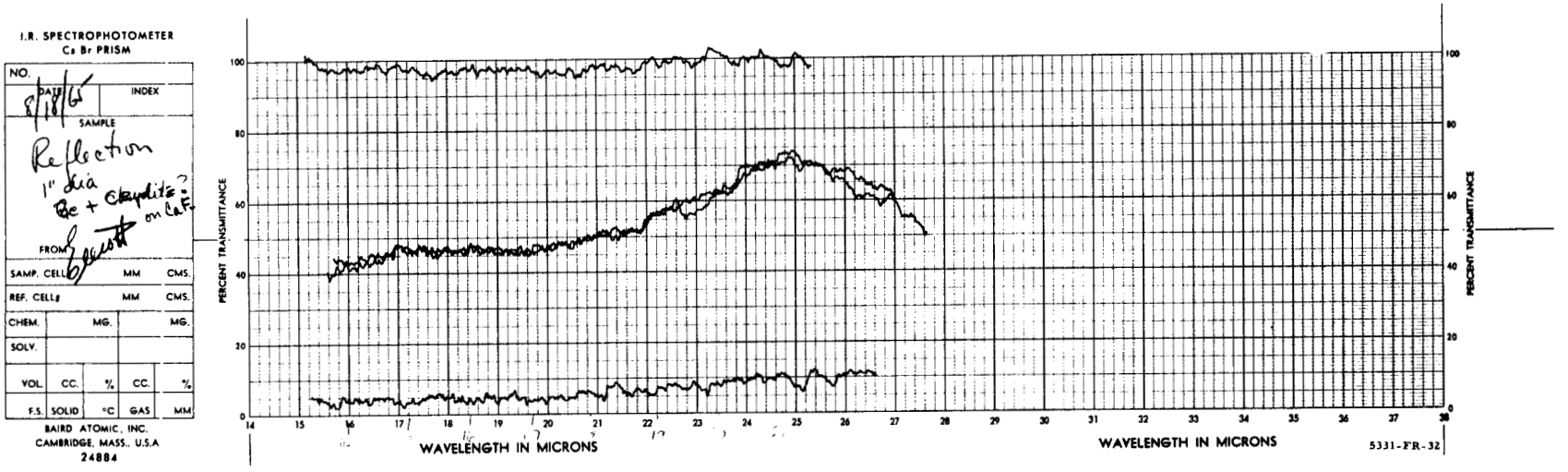


Figure 43. Measured Reflectance Curve of Calcium Fluoride, Germanium, and Cryolite Coated, 2 to 16 Microns

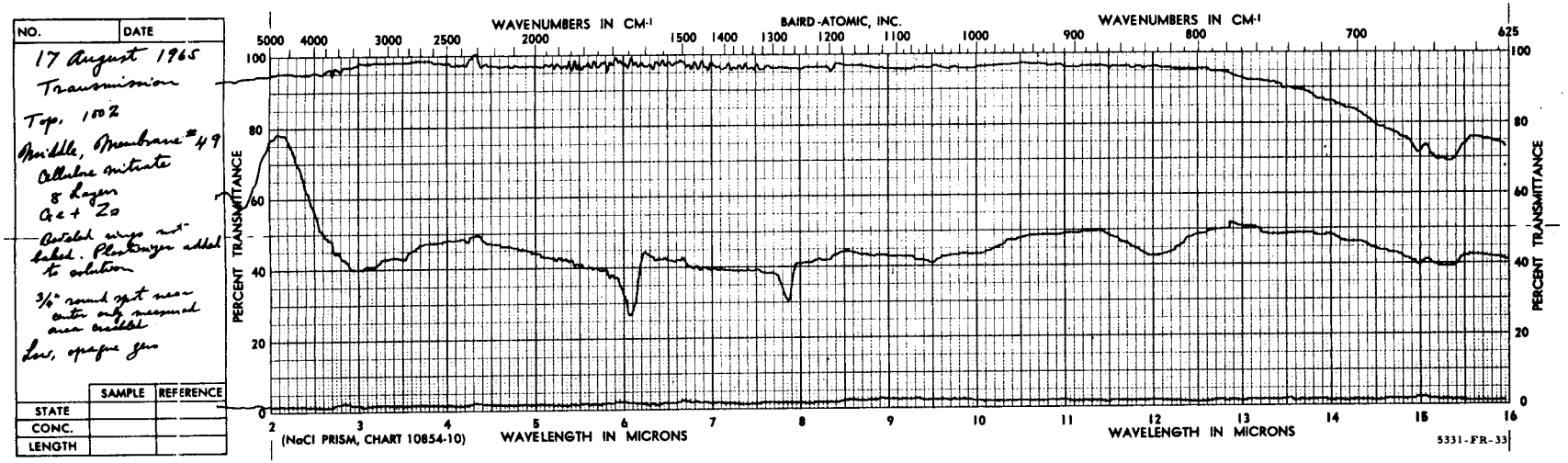


Figure 44. Measured Transmittance Curve of Cellulose Nitrate Membrane, Germanium and Zinc Sulfide Coated, 2 to 16 Microns

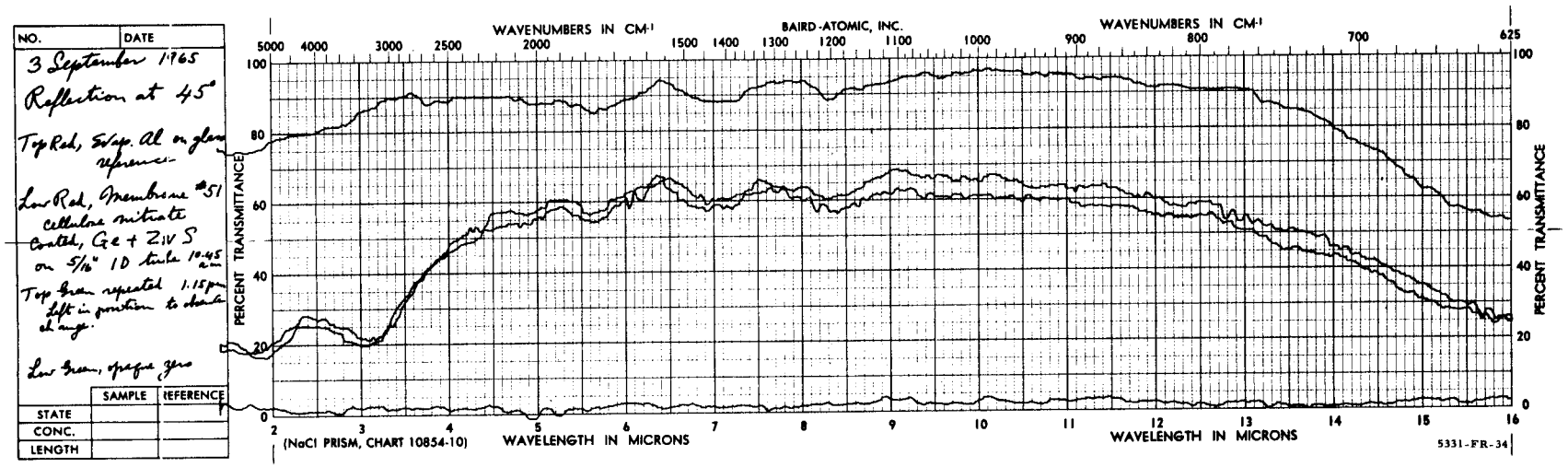


Figure 45. Measured Reflectance Curve of Cellulose Nitrate Membrane, Germanium and Zinc Sulfide Coated, 2 to 16 Microns

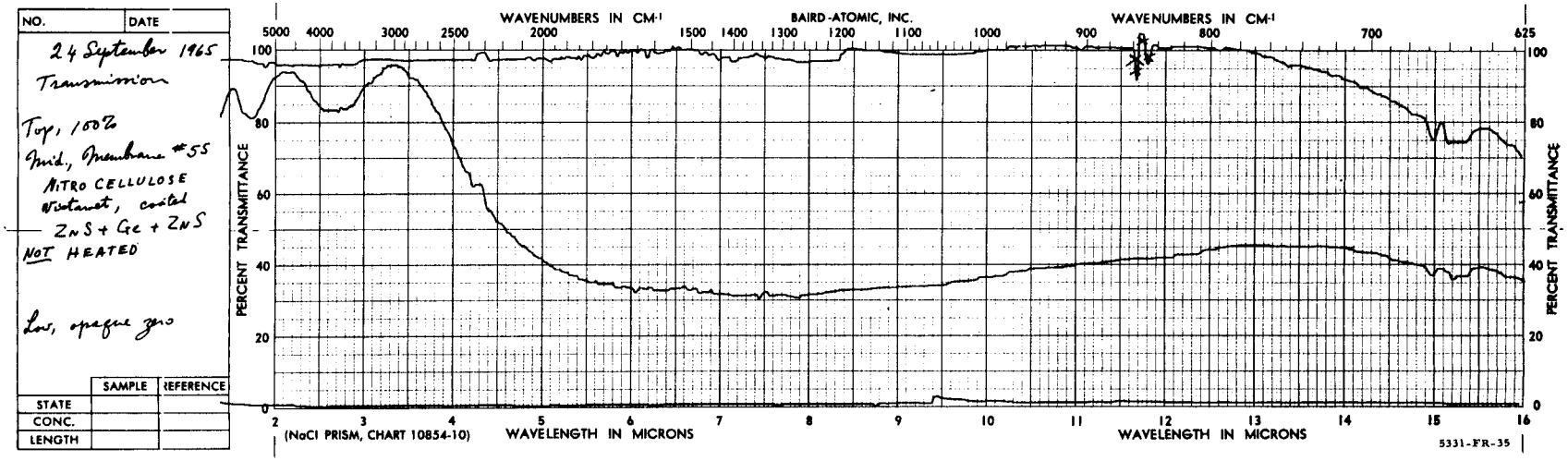


Figure 46. Measured Transmittance Curve of Cellulose Nitrate Membrane, Zinc Sulfide-Germanium-Zinc Sulfide Coated, 2 to 16 Microns

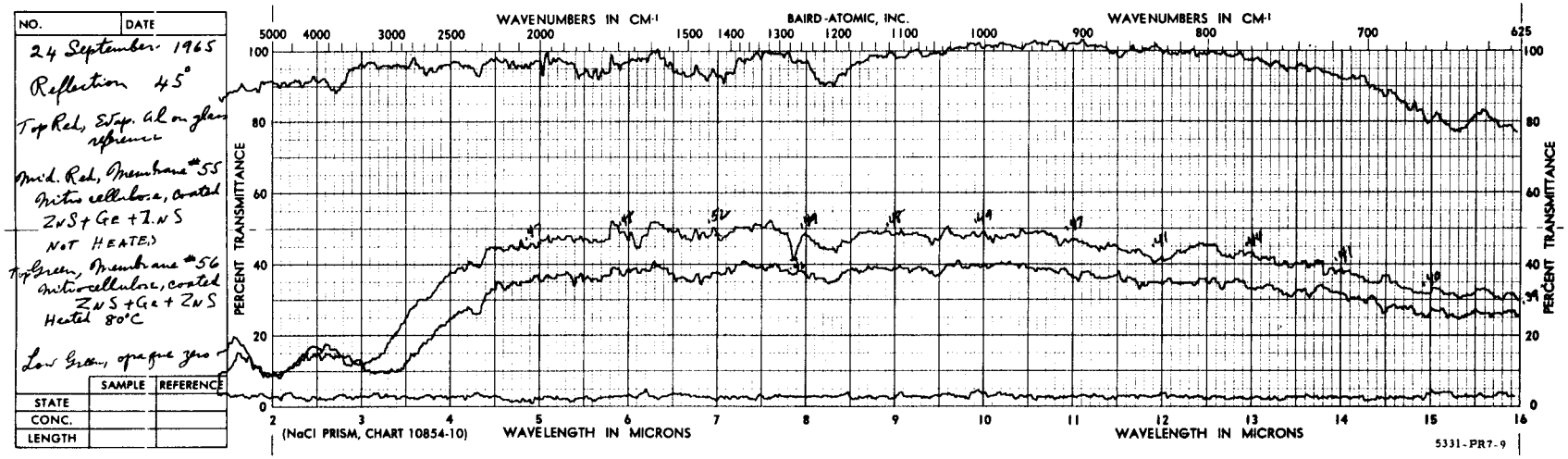


Figure 47. Measured Reflectance Curve of Cellulose Nitrate Membrane, Zinc Sulfide-Germanium-Zinc Sulfide Coated, 2 to 16 Microns

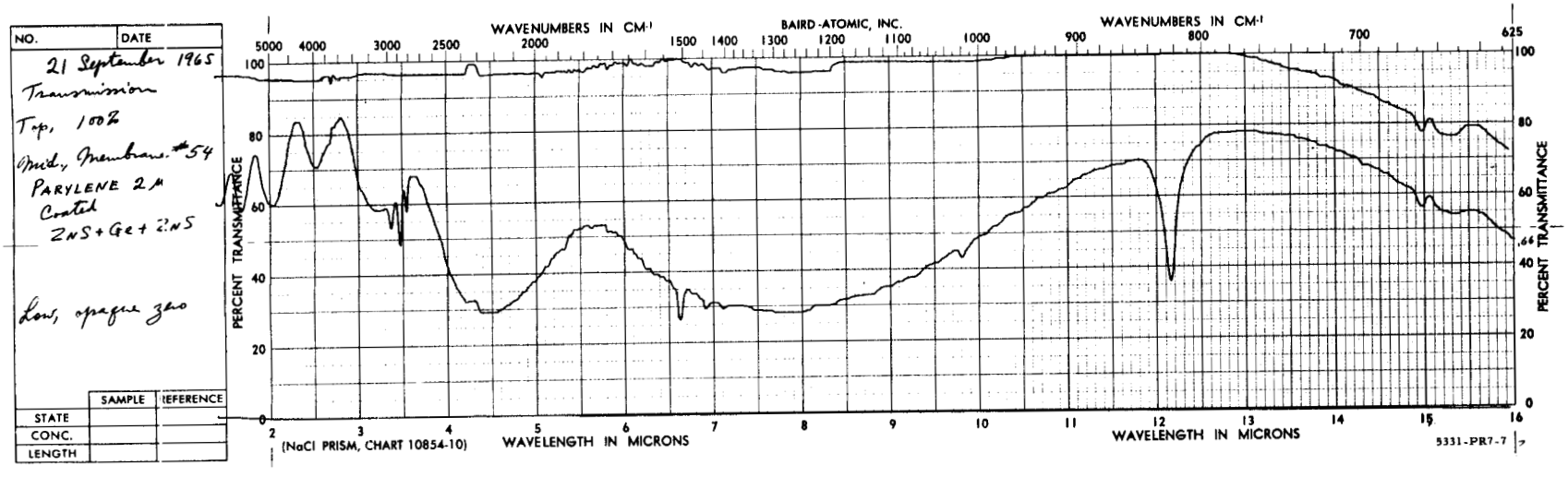


Figure 48. Measured Transmittance Curve of Parylene, Zinc Sulfide-Germanium-Zinc Sulfide Coated, 2 to 16 Microns

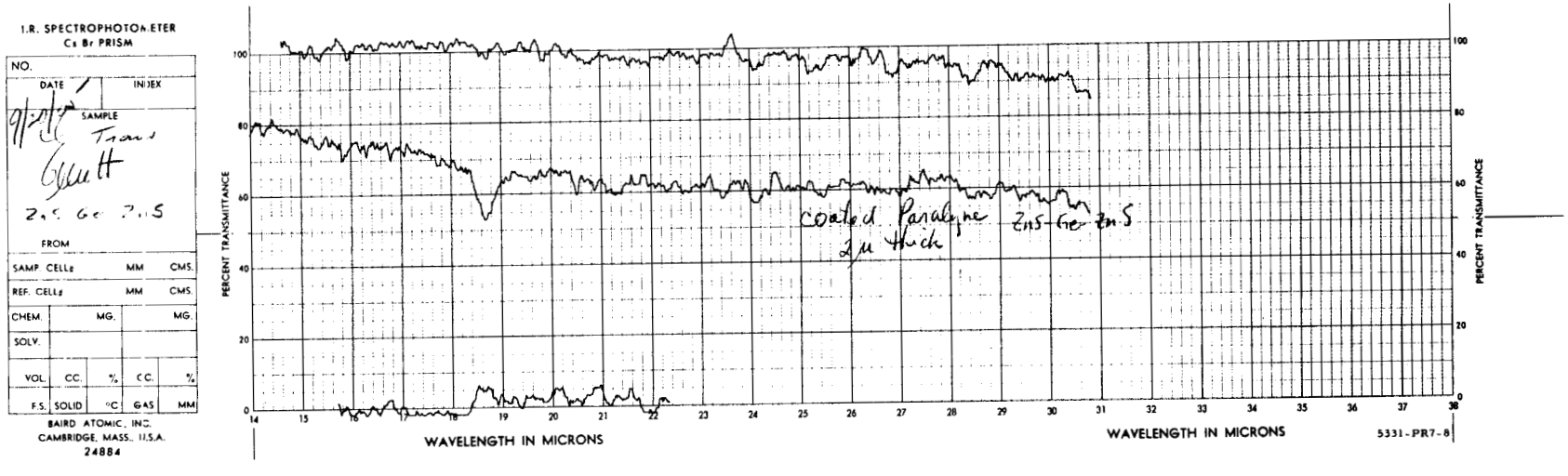


Figure 49. Measured Transmittance Curve of Parylene, Zinc Sulfide-Germanium-Zinc Sulfide Coated, 14 to 30 Microns

and zinc sulfide would balance out the stresses. Wrinkling was initially a problem until a technique was developed for supporting the bare pellicle on a piece of flat glass during coating, and then stripping the coated pellicle from the glass. Stripping was accomplished by placing the glass with coated pellicle in a shallow dish with one side blocked up so that the plate rests at an angle of about 10 degrees. Distilled water is poured slowly into the dish until the lower edge of the glass plate is just covered. The water will soon begin to work its way between the pellicle and the glass. As it does, more water is added to the dish raising the level until the entire pellicle has become detached from the glass and is floating on the surface of the water. This procedure was made feasible by initially treating the glass with a releasing agent (Victawet)*. All evaporting was done at a vacuum of 10^{-5} Torr.

6.3 Environmental Testing

A three-inch diameter cellulose nitrate pellicle, uncoated, was placed in a sealed container containing water at room temperature. The pellicle was supported directly over the water surface. The pellicle remained in the chamber for 12 hours. Condensation on the chamber walls indicated that 100-percent humidity existed. The pellicle showed no visible signs of wrinkling or other damage.

A three-inch diameter cellulose nitrate pellicle, uncoated, was subjected to a vibration test from 5 to 2000 cps up to ± 10 g. Vibration was applied in a direction normal to the surface of the pellicle (thrust axis). The vibration test fixture included a protective cover over the pellicle. The protective cover was approximately 3-1/2 x 3-1/2 x 3-1/2 inches. The pellicle survived this vibration test (appendix III, Test Report NT-2256-11 from Associated Testing Laboratories).

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*Manufactured by Stauffer Chemical Co., Victor Division, 380 Madison Ave., New York, New York.

A three-inch diameter cellulose nitrate pellicle, uncoated, was subjected to a temperature of + 55°C and -25°C for 12 hours each. No visible damage was noted.

A three-inch diameter polypropylene pellicle, uncoated, was subjected to the same high and low temperature. No damage was noted.

Relative mechanical strength was measured by dropping a 3/8-inch diameter cork ball on to a two-inch diameter sample. The height from which the ball dropped was increased in one-inch increments until the pellicle ruptured. The following results were obtained:

	Thickness of Membrane (microns)	Height (inches)
Cellulose nitrate	1/3	4
Polypropylene	1/2	27
Parylene	2	20

6.4 Final Item

Modification no. 5 dated 18 November 1965, authorized a reduction in requirements permitting the delivery of an aluminum coating on cellulose nitrate and elimination of environmental testing program.

Cellulose nitrate substrate, 0.8 micron in thickness, was deposited over the final 3-inch ring. Coating thickness was monitored in the visible region and sample pellicles on 2-inch rings were coated and then measured for T and R from 6 to 30 microns until the best thickness for equal R and T was obtained. Then, the final 3-inch pellicle was coated.

Difficulty in obtaining repeatable results was encountered due to the fact that a small percentage variation in the visible region results in a much larger change in the region from 4 microns out.

Transmittance and reflectance curves (figures 50 and 51) are shown for the 6 to 16-micron region and transmittance for the 16- to 30-micron region.

It was impossible to obtain reflectance measurement for 16 to 30 microns due to difficulties with the reflectance attachment for the modified NK-1 Spectrophotometer. However, experience has shown that in this region of the spectrum, both reflectance and transmittance are always nearly uniform.

Table 2 shows measured values taken from the curves. Curves have been normalized for zero and 100 percent.

Flatness measured on the Fizeau interferometer is two fringes or 0.5 micron/2.5 inches.

Table 2
Measured Values taken from Transmittance and Reflectance Curves of Cellulose Nitrate Membrane

λ (microns)	R	T	R·T	(T-R)
6	0.35	0.22	0.077	0.13
7	0.36	0.26	0.094	0.10
8	0.35	0.29	0.102	0.06
9	0.26	0.31	0.081	0.05
10	0.20	0.37	0.074	0.17
11	0.11	0.40	0.044	0.29
12	0.16	0.30	0.048	0.14
13	0.16	0.29	0.046	0.13
14	0.21	0.26	0.055	0.05
15	0.22	0.24	0.026	0.02
16	0.21	0.22	0.023	0.01
17	0.22			
18	0.23			
19	0.22			
20	0.23			
21	0.25			
22	0.30			
23	0.26			
24	0.26			
25	0.26			
26	0.26			
27	0.27			
28	0.28			
29	0.31			
30	0.27			

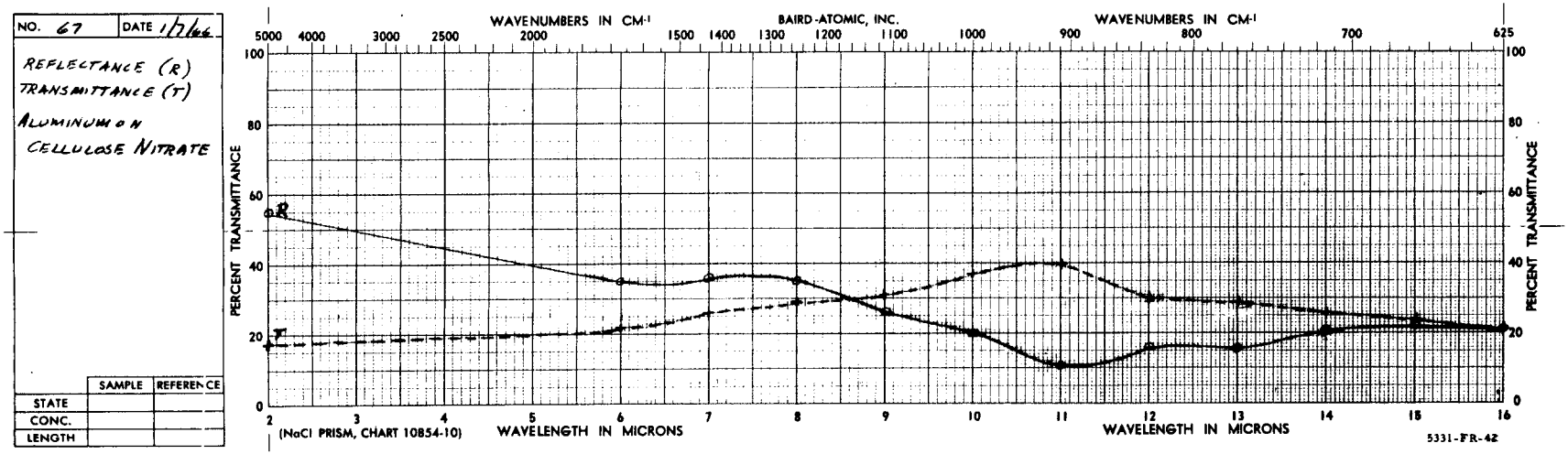


Figure 50. Normalized Transmittance and Reflectance Curves of Cellulose Nitrate Membrane, Aluminum Coated, 2 to 16 Microns

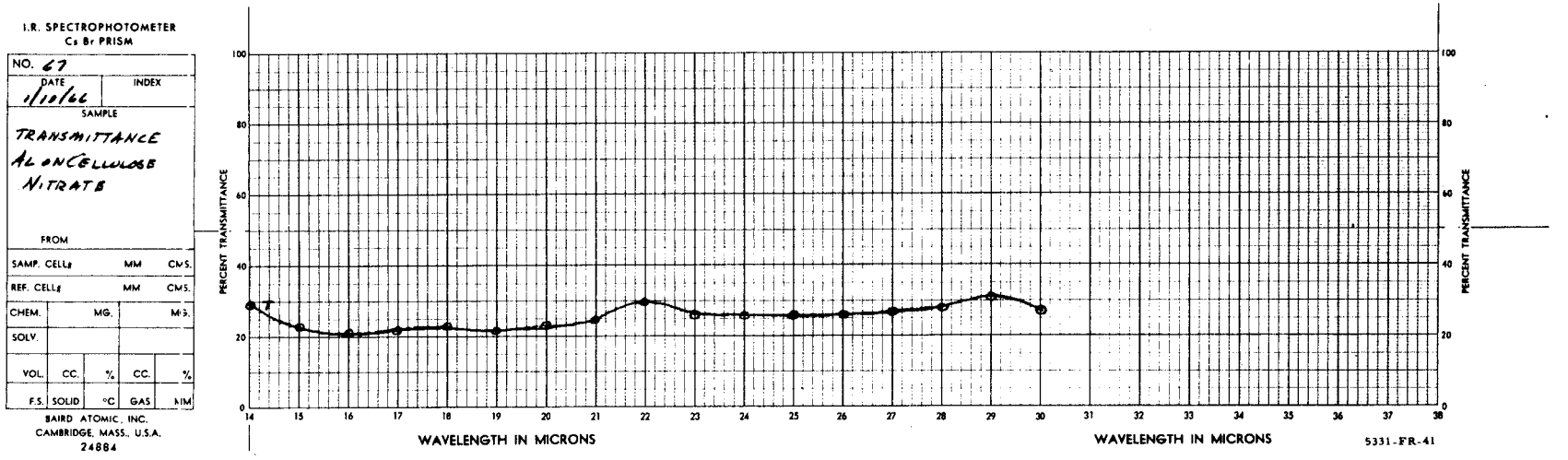


Figure 51. Normalized Transmittance Curve of Cellulose Nitrate Membrane, Aluminum Coated, 14 to 30 Microns

7. MECHANICAL DESIGN OF MOUNT

Figure 52 shows the assembly of the Pellicle Beam Splitter mount. This design features a mounting ring (figure 53) with a lip 0.060 inch in width which is polished to optical flatness. Figure 54 shows the pellicle material stretched over this lip. The flatness then achieved is shown in figure 55. The membrane and holder assembly (figure 54) is supported in the system mount (figure 56) by a kinematic support consisting of three polished steel balls and three springs which hold the membrane and holder assembly up against the steel balls, which in turn seat into shallow conical holes in the system mount. A cover (figure 57) is screwed on (figure 58) and holds everything in place. Stresses which may be applied to the system mount in attaching it to the interferometer are isolated from the pellicle supporting ring so that the pellicle will maintain its flatness (figure 59).

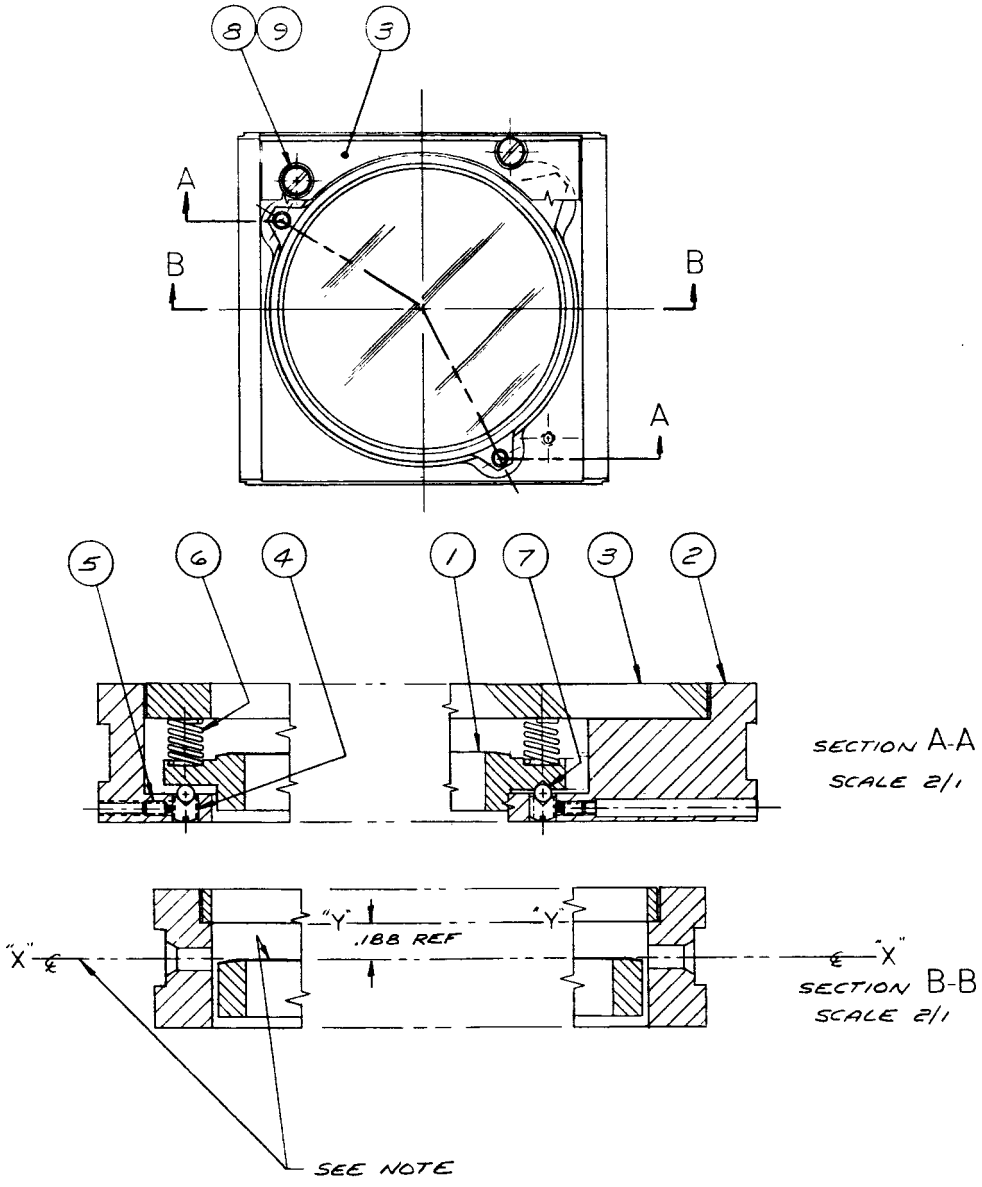
8. CONCLUSIONS

There are at least two materials which would make good infrared beam splitter pellicles. These are cellulose nitrate and Parylene N (trade name for thermoplastic polymer made by Union Carbide Corporation). These materials show a minimum of absorption in the infrared region, can be formed in thicknesses of as little as 0.1 micron, and are sufficiently strong mechanically to withstand fairly severe environmental conditions.

A third material, polypropylene, has excellent mechanical and absorption properties and can be formed down to the approximate thickness desired. Nonuniformity in thickness and flatness are its greatest drawbacks.

Metallic coatings inherently exhibit absorption throughout the infrared region. Multilayer dielectric coatings are nonabsorbing, but their reflectivity and transmittance are wavelength dependent.

A satisfactory trilayer coating of zinc sulfide and germanium has been designed. Special techniques have been devised to permit deposition of this coating on cellulose nitrate and Parylene N.



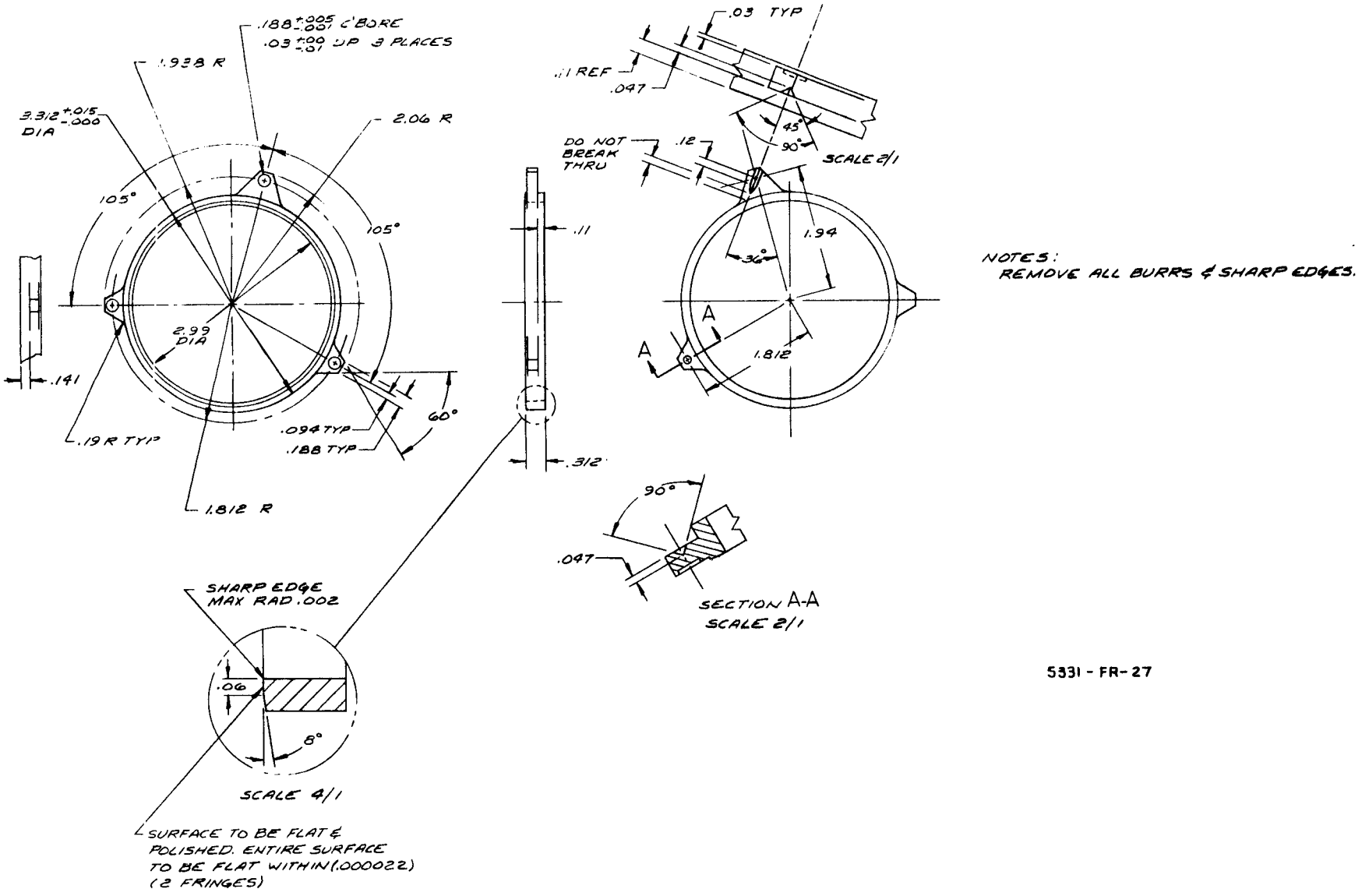
MEMBRANE PLANE (ITEM 1) TO BE PARALLEL TO, & PASS THROUGH, ℄ X-X (℄ ESTABLISHED BY CSKS) WITHIN .0005. POSITION OF MEMBRANE RELATIVE TO ℄ CAN BE ESTABLISHED BY MEASURING FROM SURFACE Y-Y (OF ITEM 2).

QTY REQD	PART NO. IDENTIFYING NO.	CODE IDENT	NOMENCLATURE OR DESCRIPTION	SPECIFICATION	MATERIAL	ITEM
3	#6		WASHER, FLAT (.391 O.D.)		CRES	9
3	#6-32 x.3814		SCREW, PAN HD		CRES	8
3	AK-2		BALL (PIC)		SST	7
3	.1870 D. x .375 LG x .023 DIA		SPRING, COMP HARDWARE PRODUCTS CO.			6
3	CS-8		SCREW, SET (NO-MAR - PIC)		SST & NYLON	5
3	5331-1004		SCREW, SET - REWORK			4
1	5331-1003		COVER			3
1	5331-1002		SYSTEM MOUNT			2
1	5331-0101		MEMBRANE & HOLDER ASSY			1

LIST OF MATERIALS OR PARTS LIST

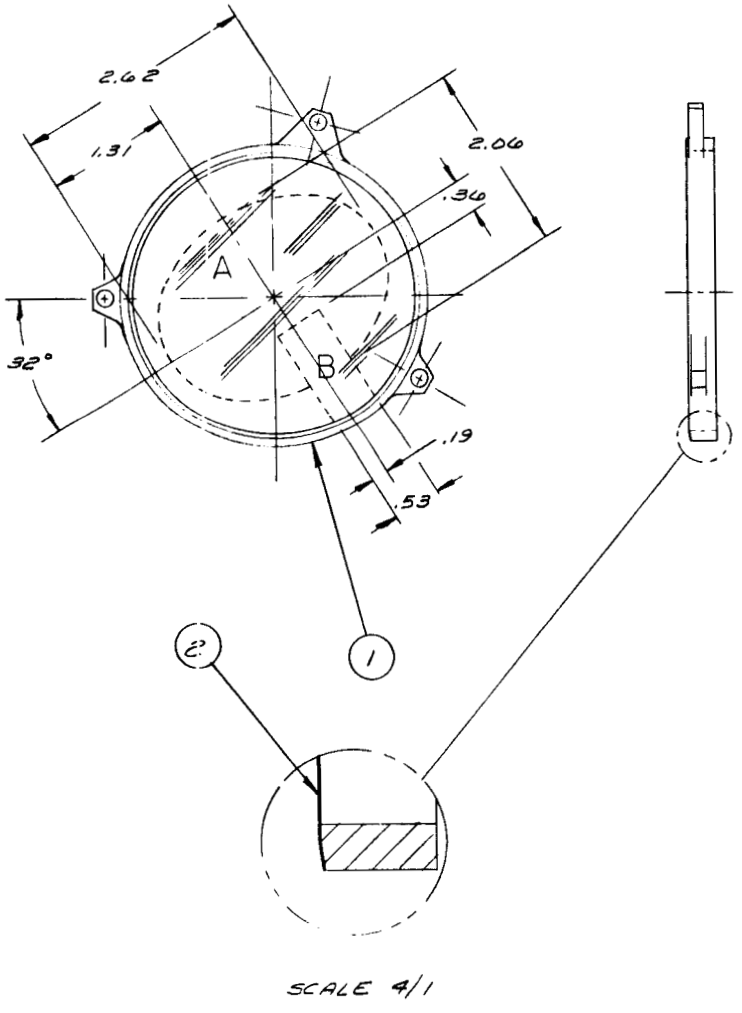
5331 - FR-26

Figure 52. Beam Splitter Assembly



5331 - FR-27

Figure 53. Ring, Pellicle



NOTES:

MATERIAL ITEM 2: CELLULOSE NITRATE 0.2–0.3 MICRONS THICK.

BAKE AT 200°C BEFORE COATING.

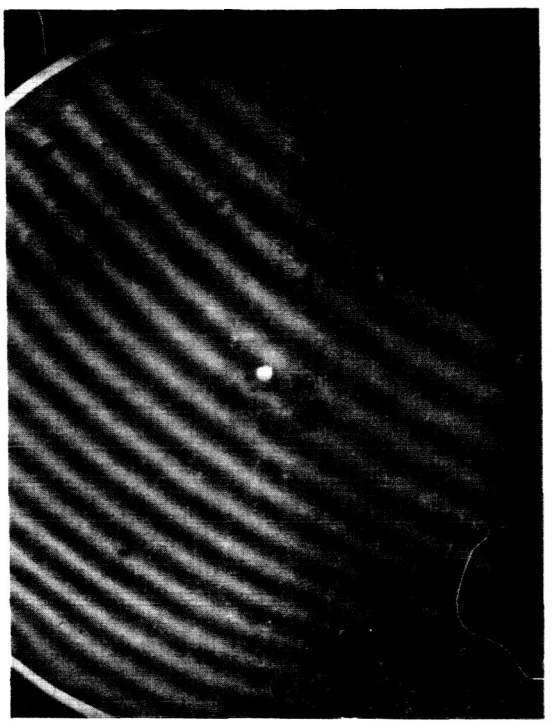
BOTH SURFACES TO BE EQUALLY COATED WITH ALUMINUM SUCH THAT THE FOLLOWING TOLERANCES HOLD OVER AREA "A".

λ	R x T	T - R
5-6 MICRONS	≥ .10	≤ .20
6-18	≥ .16	≤ .10
18-30	≥ .10	≤ .20

AREA "B" TO BE COATED FOR MINIMUM REFLECTION & TRANSMISSION OF 35% AT .5852 MICRONS.

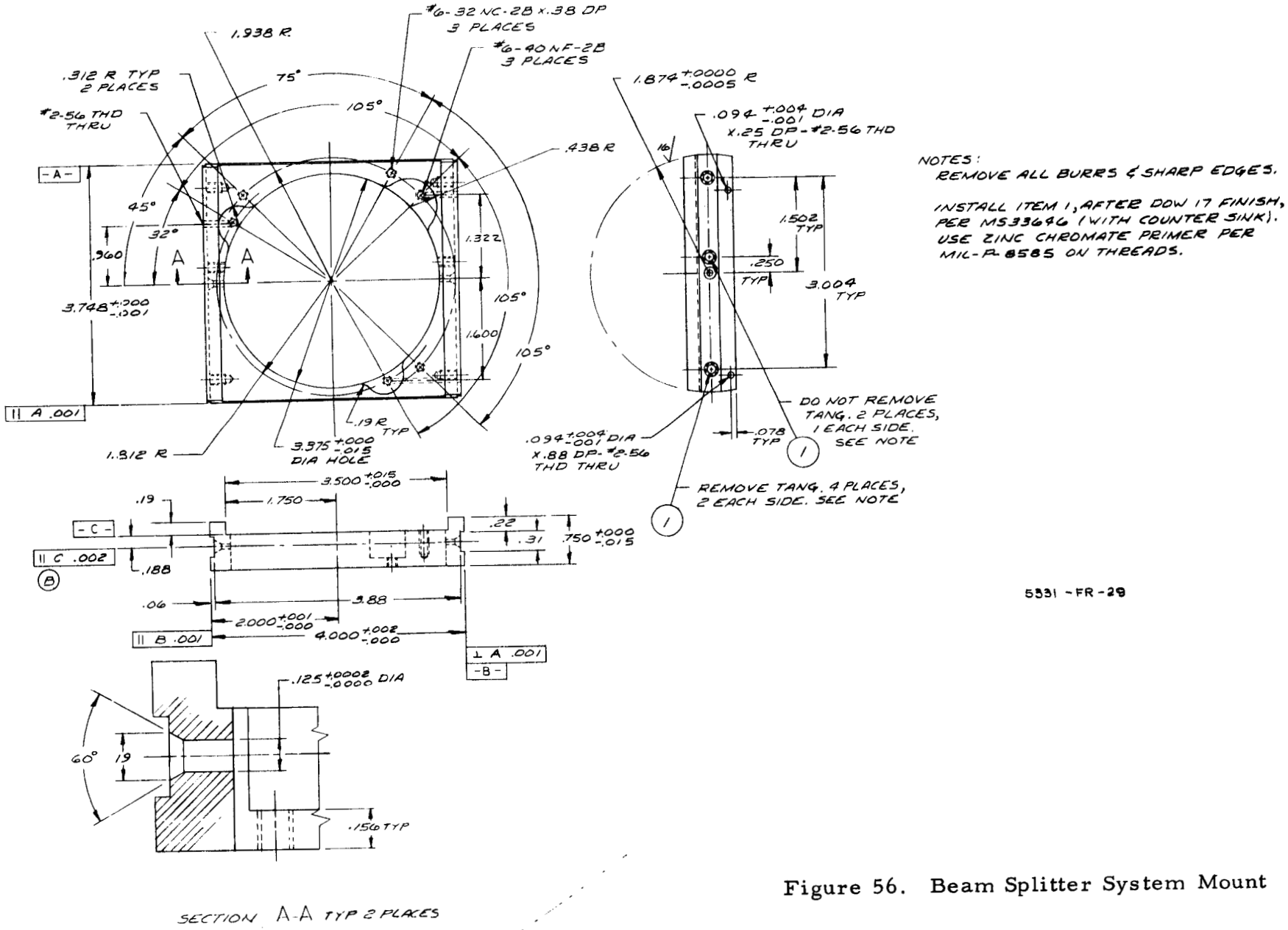
5331 - FR-28

Figure 54. Membrane and Holder Assembly



5331-3-1

Figure 55. Fringe Pattern Showing Flatness of Cellulose Nitrate Membrane Mounted on a 3-inch Aluminum Ring (X1.25)

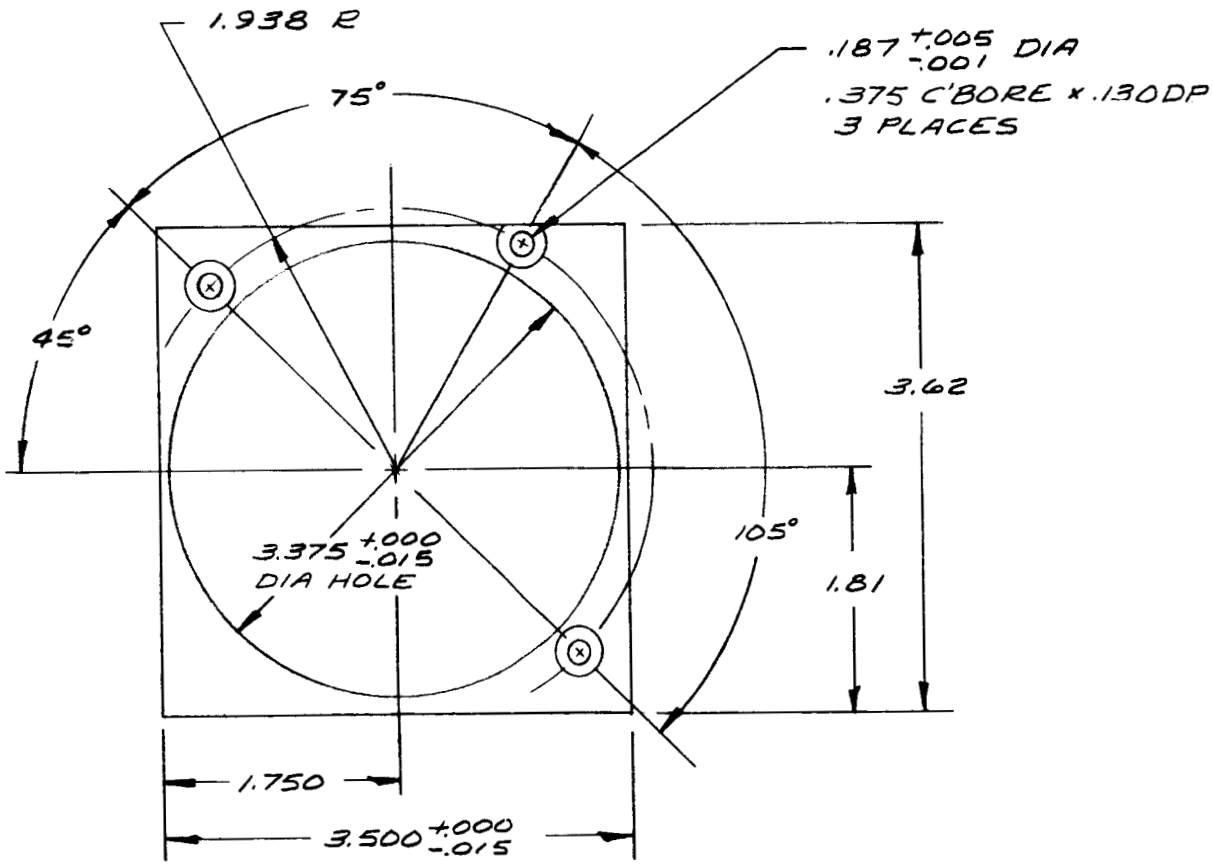


NOTES:
REMOVE ALL BURRS & SHARP EDGES.

INSTALL ITEM 1, AFTER DOW 17 FINISH, PER MS33646 (WITH COUNTER SINK). USE ZINC CHROMATE PRIMER PER MIL-P-8585 ON THREADS.

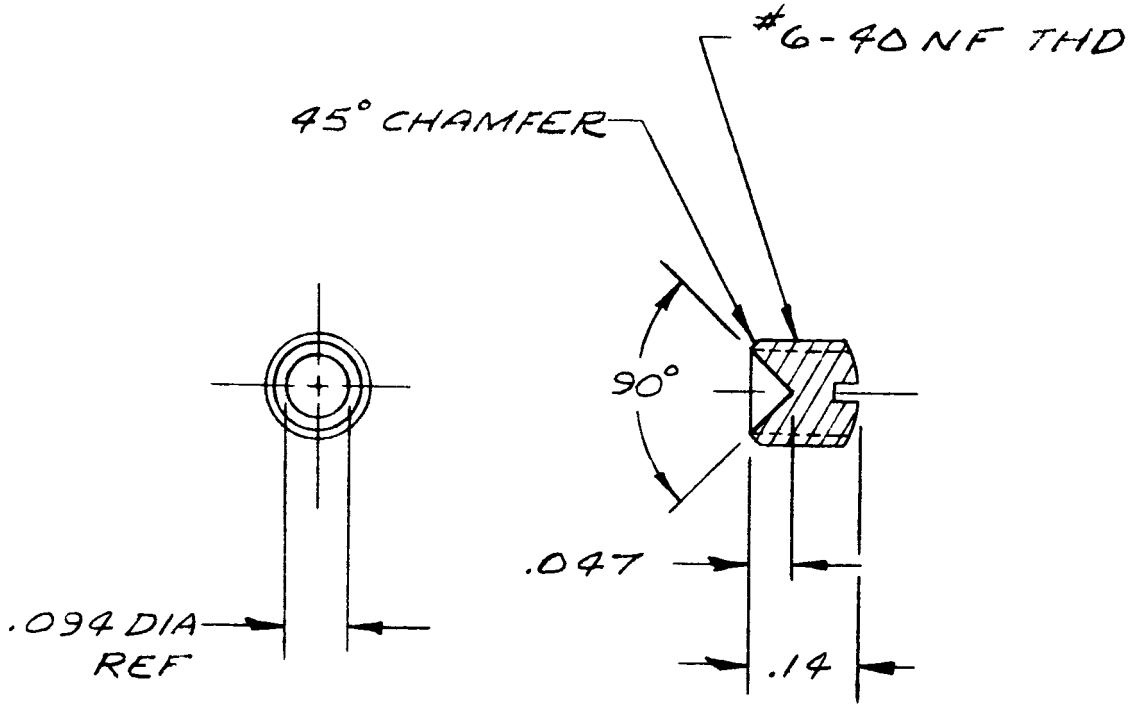
5331-FR-29

Figure 56. Beam Splitter System Mount



NOTE:
REMOVE ALL BURRS & SHARP EDGES.

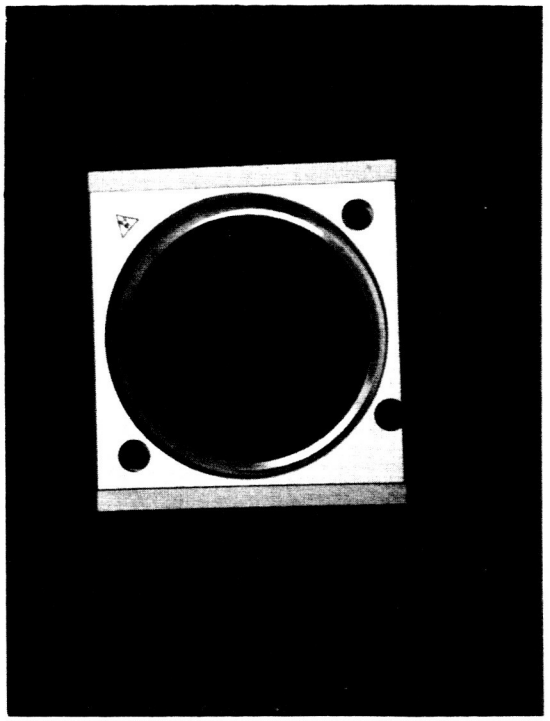
Figure 57. Cover



NOTE:

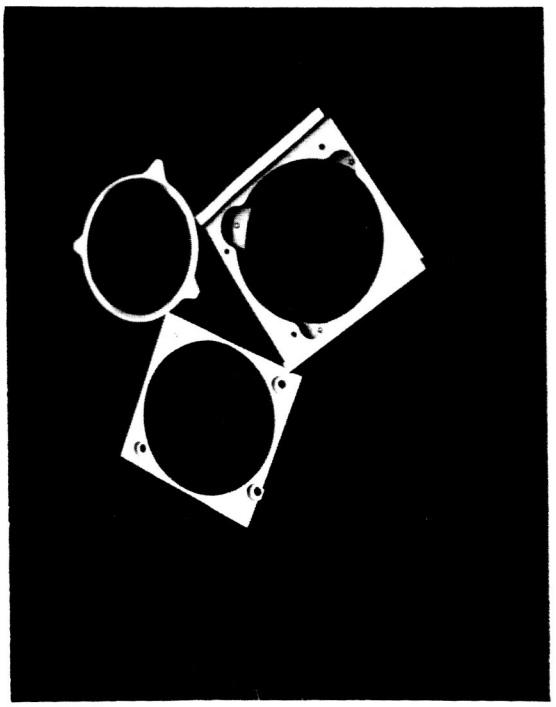
MATERIAL: MAKE FROM STANDARD SET SCREW #6-40NF THD x .25 MIN LENGTH. SLOTTED WITH FLAT OR CUP POINT. CORROSION RESISTING STEEL, PASSIVATED

Figure 58. Setscrew



5331-PR6-8

A. Assembled



5331-PR6-9

B. Disassembled

Figure 59. Beam Splitter Mount

The steps necessary to transfer to coated pellicle to a flat mounting ring and stretch it taut have been thought out, but not actually performed.

9. RECOMMENDATIONS

If further development of an infrared beam splitter of the pellicle type is done, it is recommended that this work be promulgated in the following areas.

a. Parylene N substrate with three or more layers of zinc sulfide and germanium

b. Investigate methods for producing a uniformly thin pellicle of polypropylene

c. Symmetrical sandwich construction with multiple layers of zinc sulfide and germanium between two layers of Parylene N.

APPENDIX I

THIN FILM REFERENCES

J. OPT. SOC. AM.

1. 54, 422 Jacobsson, R.
Matching a Multilayer Stack to a High-Refractive Index Substrate by means of an Inhomogeneous Layer
(Possible application to beam splitter design)

2. 54, 342 Seeley, J.S.
Resolving Power of Multilayer Filter
(Matrix formula applicable to design of multiple half-wave coatings)

3. 54, 198 Hacskaylo, M.
Determination of the Refractive Index of Thin Dielectric Films
(Includes data on n of CaF_2, SiO, SiO_2, Al_2O_3 and ZnS in the visible)

4. 53, 1317 and 53, 880 Fujiwara, S.
Refractive Indices of Evaporated Cerium Fluoride-Zinc Sulfide Films and Refractive Indices of Evaporated Cerium Dioxide-Cerium Fluoride Films
(Describes techniques for simultaneous evaporation of each pair of materials to get a layer with any index between that of the pure materials; CeF_3, n = 1.60; CeO_2, n = 2.13; ZnS, n = 2.40)

5. 53, 1266 Thelen, A.
Multilayer Filters with Wide Transmittance Bands
(Describes designs and design methods for suppressing high-reflectivity bands in multilayer filters by use of basic stack elements such as ABCBA instead of ABA).

6. 52, 1149 Baumeister, P.
Methods of Altering the Characteristics of a Multilayer Stack
(Mathematical basis for multilayer design refinement by a technique similar to automatic lens design, especially in that a computer is needed).

7. 52, 753 Young, L.
Prediction of Absorption Losses in Multilayer Interference Filters
(Method for calculating absorption losses to be expected with real materials, after design has been done with the usual assumption of lossless materials. Formulation is based on transmission line analogy).

8. 52, 431 Berning, P.
Use of Equivalent Films in the Design of Infrared Multilayer Antireflection Coatings
(Design of multilayers with each individual layer much less than the wavelength of interest. The equivalent index of such a multilayer can be designed to be any value within the range of the individual layers).

9. 51, 1406 Cox, J. T.
Special Type of Double-Layer Antireflection Coating for Infrared Optical Materials with High Refractive Indices
(Design of Double-layers with a total thickness less than one quarter-wave)

10. 51, 855 and 51, 280 Monaco, S.
Homogeneous-Inhomogeneous Thin-Film Calculations and Reflectance of an Inhomogeneous Thin Film
(Calculation techniques for a thin film of varying index)

11. 49, 116 Hass, et al
Optical Properties of Various Evaporated Rare Earth Oxides and Fluorides
(Complex index of refraction for several materials for 0.2 to 2.0 μ)

12. 46, 228 Baumeister, P. and Stone, J.
Broad-Band Multilayer Film for Fabry-Perot Interferometers
(Describes beneficial effects of varying from quarter-wave thicknesses in a 5-layer zinc sulfide-cryolite stack to broaden high reflectivity region. Achieved almost one octave)

13. 44, 357 and 362 Schulz. L.
Optical Constants of Silver, Gold, Copper and Aluminum I and II
(Gives data on the complex index of refraction of these materials over the range 0.4 to 0.95 μ)

APPLIED OPTICS

1. 3, 867 Plass, G.
Mie Scattering and Absorption Cross Section for Aluminum Oxide and Magnesium Oxide
(Includes best estimates, based on literature survey of complex index of Al_2O_3 and MgO for the region 0.5 to 10 μ)

APPENDIX II

REPORT FROM MOLECULON RESEARCH CORP.

MOLECULON

139 MAIN STREET CAMBRIDGE MASSACHUSETTS 02142

TEL. Kirkland 7-2353 (AREA CODE 617)
PILOT PLANT 181 PORTLAND ST., CAMBRIDGE

RESEARCH CORP.

March 12, 1965

Mr. William Vaughan
Baird-Atomic, Inc.
95 Second Avenue
Waltham, Massachusetts 02154

Dear Mr. Vaughan,

This letter contains our findings regarding the problem of fabricating an unsupported film 0.5 μ thick x 3 inch diameter having high transparency in the infrared region of 5-30 μ. The film must also be capable of withstanding 95% relative humidity, vibration from 5 to 2,000 cps, vacuum, and temperature in the range -25 to +25°C.

The material showing most promise of success is polypropylene. This material forms exceptionally strong films. The second most promising material is polyethylene. The infrared spectrum of both of these materials is similar and is characterized by a few strongly absorbing peaks on a completely transparent background. In the range 5-16 μ, these peaks occur at approximately 6.8, 7.3, and 13.8 μ. Figure 1 shows the amplitudes of these peaks for polyethylene film 20 μ thick. Applying Lambert's law (absorption proportional to thickness) the absorbance of these peaks for a 0.5 μ film of polyethylene would be:

Wavelength (μ)	Approximate Absorbance for 0.5 μ film
6.8	.025
7.3	.004
13.8	.012

A third possible material is polystyrene. Its infrared spectrum shows many absorption peaks, Figure 2 shows the spectrum of polystyrene 70 μ thick. In a thinner film these peaks would be proportionately lower and would present a more or less continuous background absorption as opposed to the few sharp peaks of polypropylene and polyethylene.

A fourth possibility is Mylar. Although it forms exceptionally strong films, it also exhibits strong infrared absorption in the region 6-10 μ and at 13.7 μ. In spite of its absorption, Mylar may still be usable in very thin films.

There are three methods of forming a 0.5 μ film:

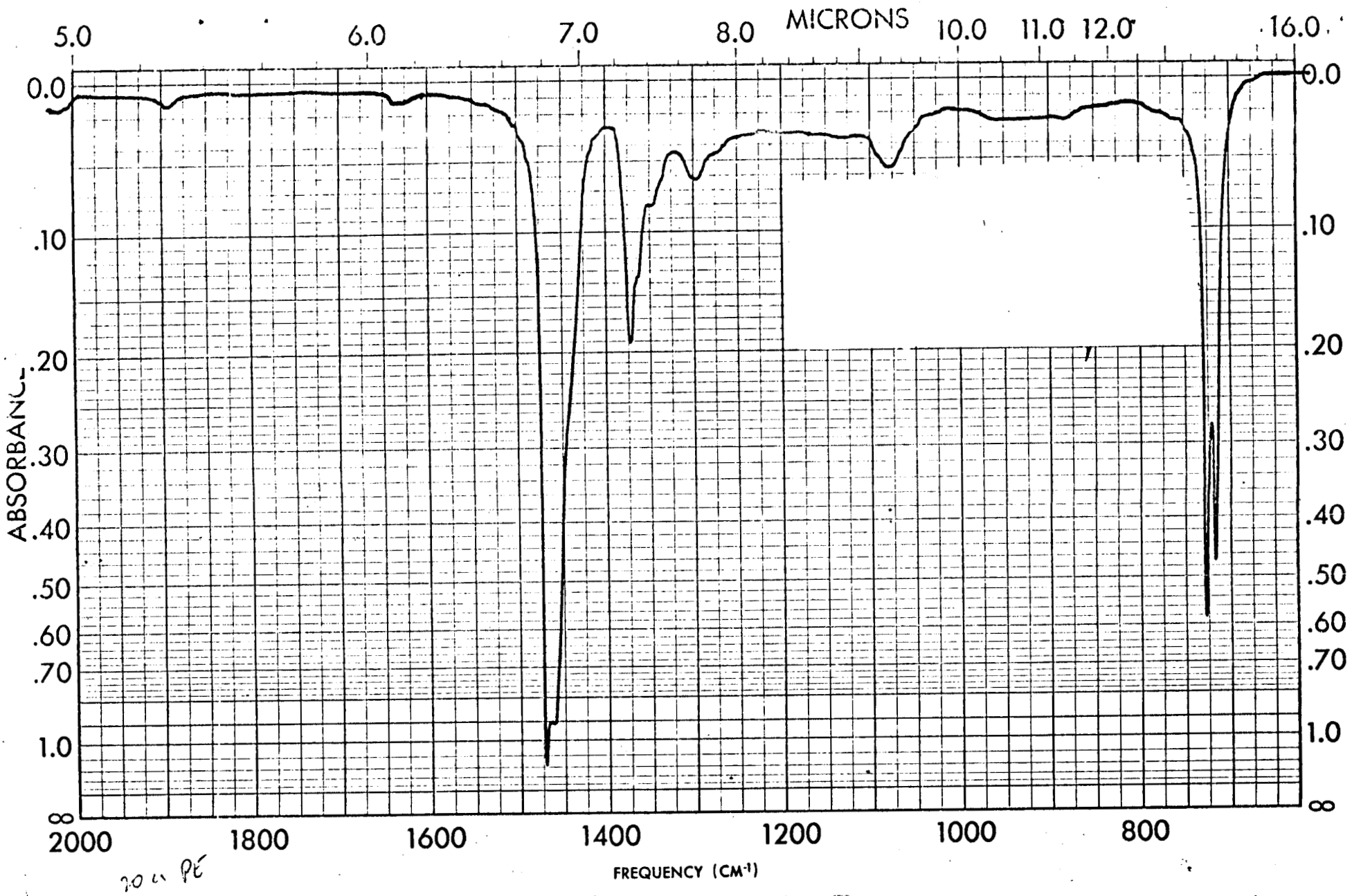


Figure 1. Polyethylene - 20 μ

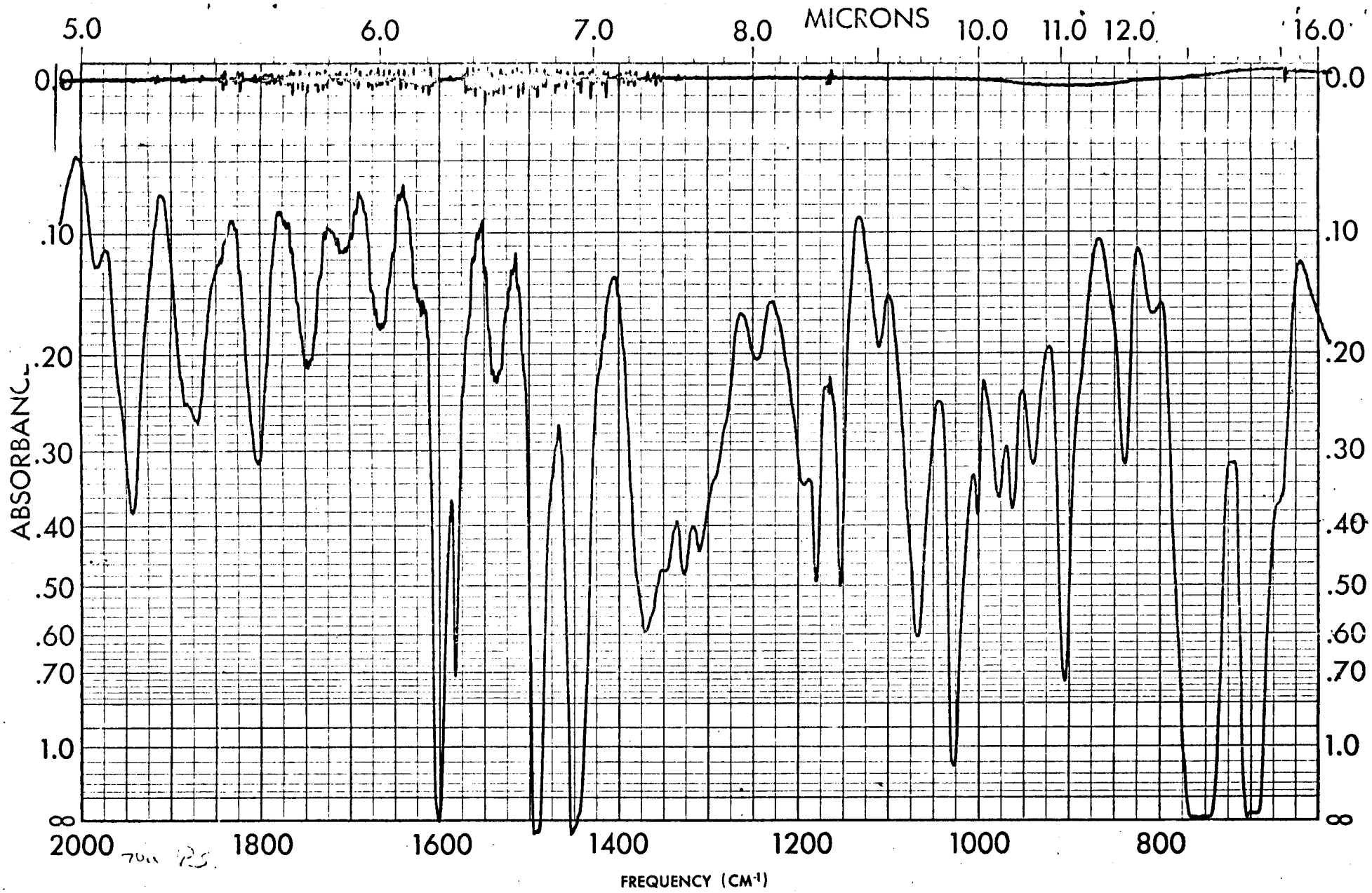


Figure 2. Polystyrene - 70 μ

a) casting from dilute solution onto a mercury or water surface.

b) blowing an already thin film to a still thinner one after heating it.

c) mechanically stretching biaxially, an already thin, heated film until the desired thickness is obtained.

The casting process gives the weakest films. Mechanical deformation, either by blowing or by stretching, results in at least twice the tensile strength, compared to casting, due to molecular and crystallite orientation effects. Biaxial mechanical stretching, because it permits of greater control than does blowing, is the method which gives the greatest promise of succeeding. Figure 3 shows the technique recommended:

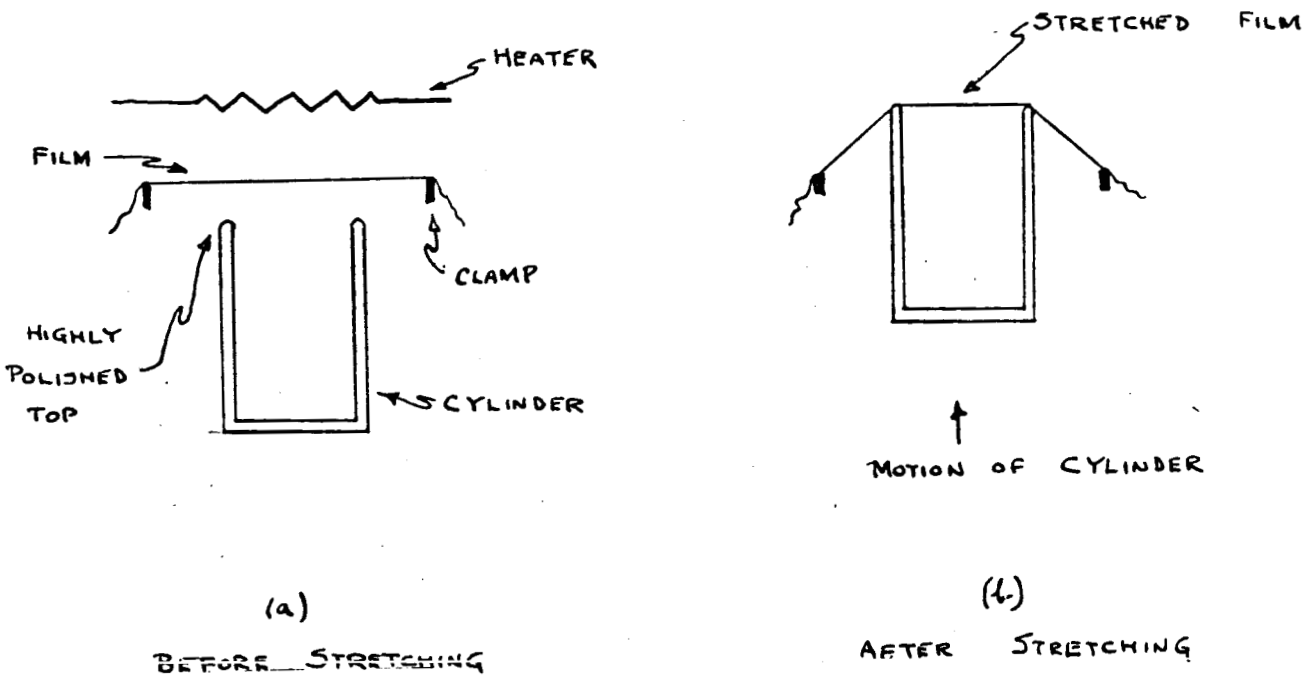


FIG. 3

BIAXIAL STRETCHING TECHNIQUE

A thin circular film of plastic is secured in a ring clamp and carefully heated. A cylinder with rounded and polished end is then slowly pushed up into the film. Starting with 10μ film, the diameter of the original circle of film must be increased 4.5 times to yield a product 0.5μ thick. Both polypropylene and polyethylene are available in 10μ film.

We recommend that the stretching technique be given a trial on a limited attempt basis. Moleculon Research has considerable experience in thin plastic films. One of our projects has been the study by infrared analysis of chemical alterations in thin plastic films due to high energy radiation. Our proposed effort would require the services of one senior scientist at $16 per hour for five days.

We anticipate that the apparatus can be fabricated for approximately $300. If it is made by an outside shop, we will bill you at cost to us; if we elect to make it in our own shop, you will be charged our standard labor rates, plus cost of materials. The total cost to you for this entire exploratory effort will not exceed $940. Our effort would be limited to polypropylene and polyethylene.

Although we have every reason to believe our work will be successful, we cannot, of course guarantee a result. If our work showed little chance of success in achieving our objective, we would then propose to you a similar effort based on the blowing technique.

Very truly yours,

MOLECULON RESEARCH

Warren A. Salmon

Warren A. Salmon
Senior Chemist

WAS/rac

APPENDIX III

TEST REPORT NT-2256-11 FROM ASSOCIATED TESTING LABORATORIES

Test Report No. NT-2256-11 No. of Pages 3

Report of Test on

PELLICLE BEAM SPLITTER

Sinusoidal Vibration Test

for

Baird-Atomic, Inc.

Associated Testing Laboratories, Inc.

Burlington, Massachusetts

Date June 14, 1965

	Prepared	Checked	Approved
By	D. Schuster	R.E. Borghetti	D.C. Jensen
Signed	D. Schuster	R.E. Borghetti	D.C. Jensen
Date	6/14/65	6/14/65	6/14/65

Administrative Data

1.0 Purpose of Test:

To subject the Pellicle Beam Splitter to a Sinusoidal Vibration Test for Engineering evaluation purposes.

2.0 Manufacturer: Baird-Atomic, Inc.
95 Second Avenue
Waltham, Massachusetts

3.0 Manufacturer's Type or Model No.: Pellicle Beam Splitter

4.0 Drawing, Specification or Exhibit: In accordance with written instructions from an Engineering Representative of Baird-Atomic, Inc.

5.0 Quantity of Items Tested: One (1)

6.0 Security Classification of Items: Unclassified

7.0 Date Test Completed: June 10, 1965

8.0 Test Conducted By: **Associated Testing Laboratories, Inc.**
NEW ENGLAND DIVISION

9.0 Disposition of Specimens: Returned to Baird-Atomic, Inc.

10.0 Abstract:

The Pellicle Beam Splitter was subjected to Sinusoidal Vibration over the frequency range from 5 to 2000 cps at vibratory levels up to ±10g. Vibration was applied in a direction normal to the surface of the Pellicle, (thrust axis). The unit was visually examined upon completion of the Vibration Test, and there was no evidence of mechanical damage noted.

TEST PROCEDURE

The Pellicle Beam Splitter was subjected to a Sinusoidal Vibration Test in accordance with written instructions from an Engineering Representative of Baird-Atomic, Inc. The following is a description of the Test Procedure as it was performed.

The Pellicle Beam Splitter was mounted to a Vibration Test fixture which was supplied by Baird-Atomic, Inc. The fixture was, in turn, securely fastened to the Vibration Exciter. The unit was then subjected to Sinusoidal Vibration over the frequency range from 5 to 2000 cps at vibratory levels of 0.5 inch double amplitude of ±10g's, whichever was the limiting value. The entire frequency range from 5 to 2000 cps and return to 5 cps was traversed logarithmically at a rate of 2 octaves/minute. Vibration was applied in a direction perpendicular to the Pellicle (thrust axis) only.

Upon completion of the Vibration Test, the Pellicle Beam Splitter was visually examined for evidence of mechanical damage and was returned to Baird-Atomic, Inc. for further evaluation.

LIST OF APPARATUS

1. Vibration Test System consisting of the following equipment:

   a. Vibration Exciter, MB Electronics, Model C-50.

   b. Control Console, MB Electronics, Model T130MC.

   c. Power Amplifier, MB Electronics, Model 4150MB.

   d. Accelerometer, Endevco Corp., Model 2215-C.

TEST RESULTS

There was no evidence of mechanical damage noted. Further evaluation was performed at the facilities of Baird-Atomic, Inc.