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INVESTIGATION OF LIGHT SCATTERING IN HIGHLY REFLECTING PIGMENTED COATINGS

National Aeronautics and Space Administration
Office of Advanced Research and Technology

Gene A. Zerlaut [1963]

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INVESTIGATION OF LIGHT SCATTERING IN HIGHLY REFLECTING PIGMENTED COATINGS

August 1 through September 1, 1963

National Aeronautics and Space Administration
Office of Advanced Research and Technology

Contract NASr-65(07)
IITRI Project C6018

This is the ~~second~~ first monthly progress report under the subject contract for the National Aeronautics and Space Administration. The period covered is August 1 through September 1, 1963.

DARK ROOM PREPARATION OF MONODISPERSE CRYSTALS IN GELATIN SOLUTION

Initially, crystals of silver bromide were prepared after the method of Berry.[1,2] However, contrary to the information in Berry's paper, silver bromide was found to be insufficiently stable to permit reproducible reflectance and transmittance measurements. The pale, creamy yellow color of silver bromide makes it somewhat undersirable as a model for scattering studies since the absence of absorption in the region of interest is implicit in the maximization of light scattering.

Silver chloride was used as a white substitute for silver bromide. Silver chloride is sensitive mainly to ultraviolet light, and less sensitive to the visible wavelengths.

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[1] Berry, C. R., J. Optical Soc. America, 52, 888, 1962.

[2] Berry, C. R., Photo. Sci. and Engr., 5, 332, 1961.

Therefore, it was thought that proper protection of the crystal suspensions coupled with the addition of desensitizers would give a whiter, more stable film than silver bromide.

Silver chloride suspensions were prepared in the darkroom, under a red safety light. The procedure was identical to that used for silver bromide. However, since stock gelatin produced fern-like irregularities in dried films, photographic emulsion grade gelatin was substituted in the reaction vessel. Stoichiometric quantities of silver nitrate and potassium chloride were simultaneously injected at equal rates into 100 ml of 3% gelatin solution at 48°C.

INHIBITION OF LIGHT SENSITIVITY

Unfortunately, silver chloride prepared in emulsion gelatin was found to be just as sensitive as silver chloride prepared in stock gelatin. Excess potassium bromide is a potent desensitizer but has two drawbacks: 1) the surface of the silver chloride crystals is changed to the pale yellow bromide, and 2) potassium bromide does not prevent the graying of the silver chloride suspension when exposed to light (graying takes place in about 30 seconds). Potassium bromide only retards the formation of free silver. Excess quantities of potassium bromide small enough so as not to cause the yellow cast were also too small to effectively inhibit light sensitivity. However, this small excess of potassium bromide when added together with a large excess of potassium chloride proved to be as potent an inhibitor as a large excess of potassium bromide alone, without bringing about the yellow cast that potassium bromide would cause. Excess potassium chloride alone showed only minor inhibitory effect.

Gelatin films dried in the dark before exposure to room light were more stable to light than were wet films. Also, photographic emulsion gelatin gave much more uniform films than did stock gelatin. Crystals did not agglomerate during drying, and the dried gelatin showed a greatly reduced formation of structural irregularities.

SIZING OF SILVER CHLORIDE CRYSTALS IN THE ELECTRON MICROSCOPE

In the dark room an aliquot of the freshly prepared silver chloride suspension is diluted 1:100. Stainless steel sample screens bearing a Formvar film are dipped once into this dilute suspension. The excess adhering fluid is washed off with a stream of distilled water, and the screens are allowed to dry on a glass slide. Then a small amount of Duco cement at the edge secures each screen to the glass. The slide is put on a protractor mount and is shadow cast at 18° with a carbon-platinum pellet. Next it is uniformly cast with carbon from 0 to 180°. The mounted crystals are now ready for viewing with the electron microscope Figure 1 shows a photomicrograph of screen #2 after shadow cast. Contrary to Berry[2], it was not found that silver halide crystals treated by the above procedure were altered by the electron beam, yielding masses of what is presumably free silver. Berry suggests further treatment of the shadow cast crystals to prevent this reduction to silver. Bathing the silver chloride in photographic acid fix (Kodak Formula F-5) dissolves out all unreacted silver chloride, leaving behind sharply defined, clear ghosts of the crystals. Figure 2 shows screen #2 after 3 hours in fix bath. At 10,000X, the crystals appear 2-3 mm on an edge corresponding to 0.2 μ high, calculated from

$$\tan 18^\circ = 0.325 = \frac{\text{height of crystal}}{\text{length of shadow}}$$

The crystals prepared by the above procedure are therefore cubic, being about 0.2 μ on each edge.

Figure 1

SILVER CHLORDIE CRYSTALS AFTER SHADOW CASTING (15,000X)

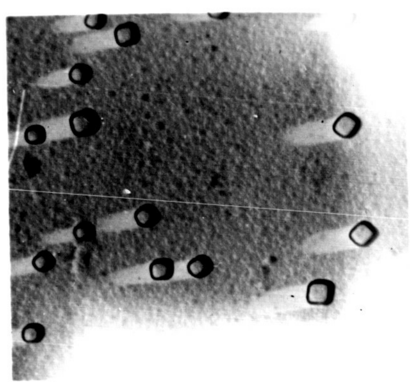

Figure 2

SILVER CHLORIDE CRYSTALS AFTER 3 HOURS IN ACID FIX BATH

REFLECTANCE OF SILVER CHLORIDE CRYSTAL FILMS

Silver chloride films frozen on aluminum substrates show the same unstable reflectance as silver bromide, due to formation of ice crystals. When dried on the freshly cleaned aluminum surface, both silver chloride and silver bromide form dirty black, uneven films. If the aluminum is coated with Collodion before the silver chloride suspension is applied to it, then the silver chloride does not turn black. See Figure 3 for reflectance of dried silver chloride films.

TRANSMITTANCE OF THIN SILVER CHLORIDE FILMS

A Collodion film is stretched over an aperture of known area to form a shallow boat with a transparent bottom. A measured volume of undiluted silver chloride suspension is spread in the bottom of the boat and allowed to dry. From the concentration of silver chloride in the original suspension the amount of silver chloride per unit area of dried film can be calculated. See Figure 4 for the transmittance of a thin silver chloride film.

This method for preparing and determining the density of the final film has met with only limited success. Because the Collodion films are so fragile, the procedure is tedious and not always successful. Materials that are stiffer and more manageable than Collodion should be sought. They should nevertheless be as transparent as Collodion to light of wavelengths from 0.32 to 2.0 μ. Saran Wrap may be a suitably tough substitute for Collodion. Its transmittance (Figure 5) is only about 90% that of Collodion

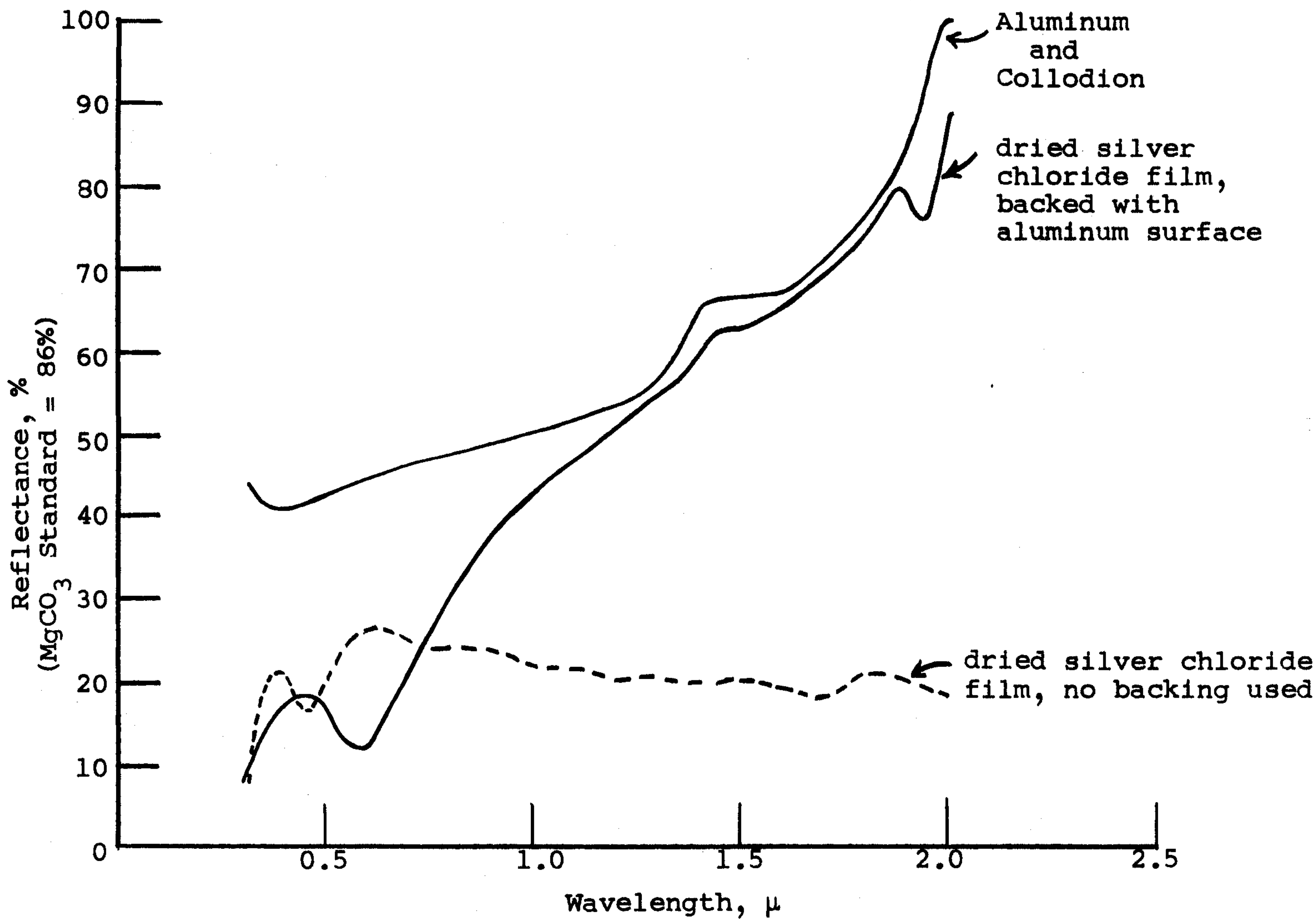

Figure 3

REFLECTANCE OF DRIED SILVER CHLORIDE FILM

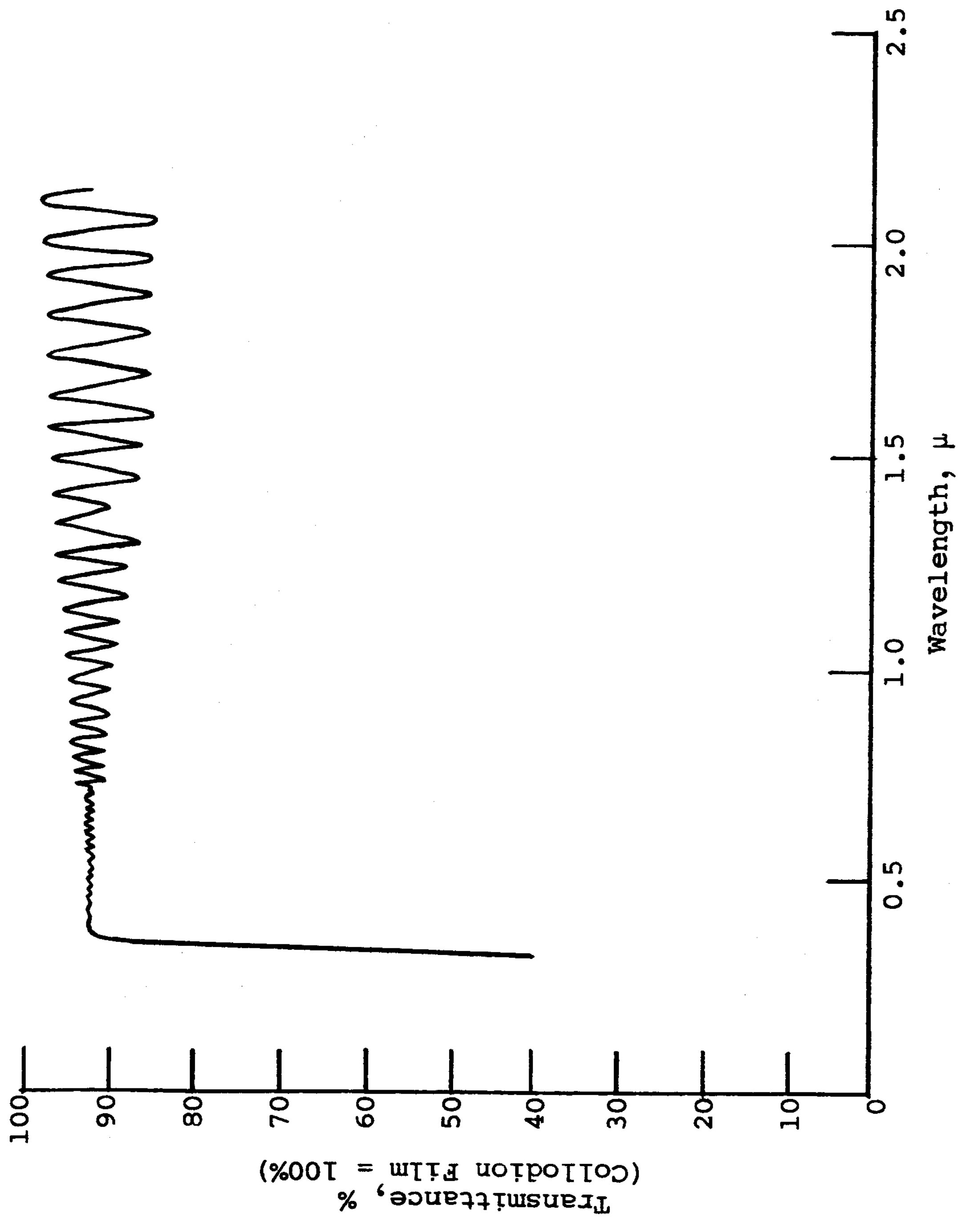

Figure 5

TRANSMITTANCE OF SARAN WRAP

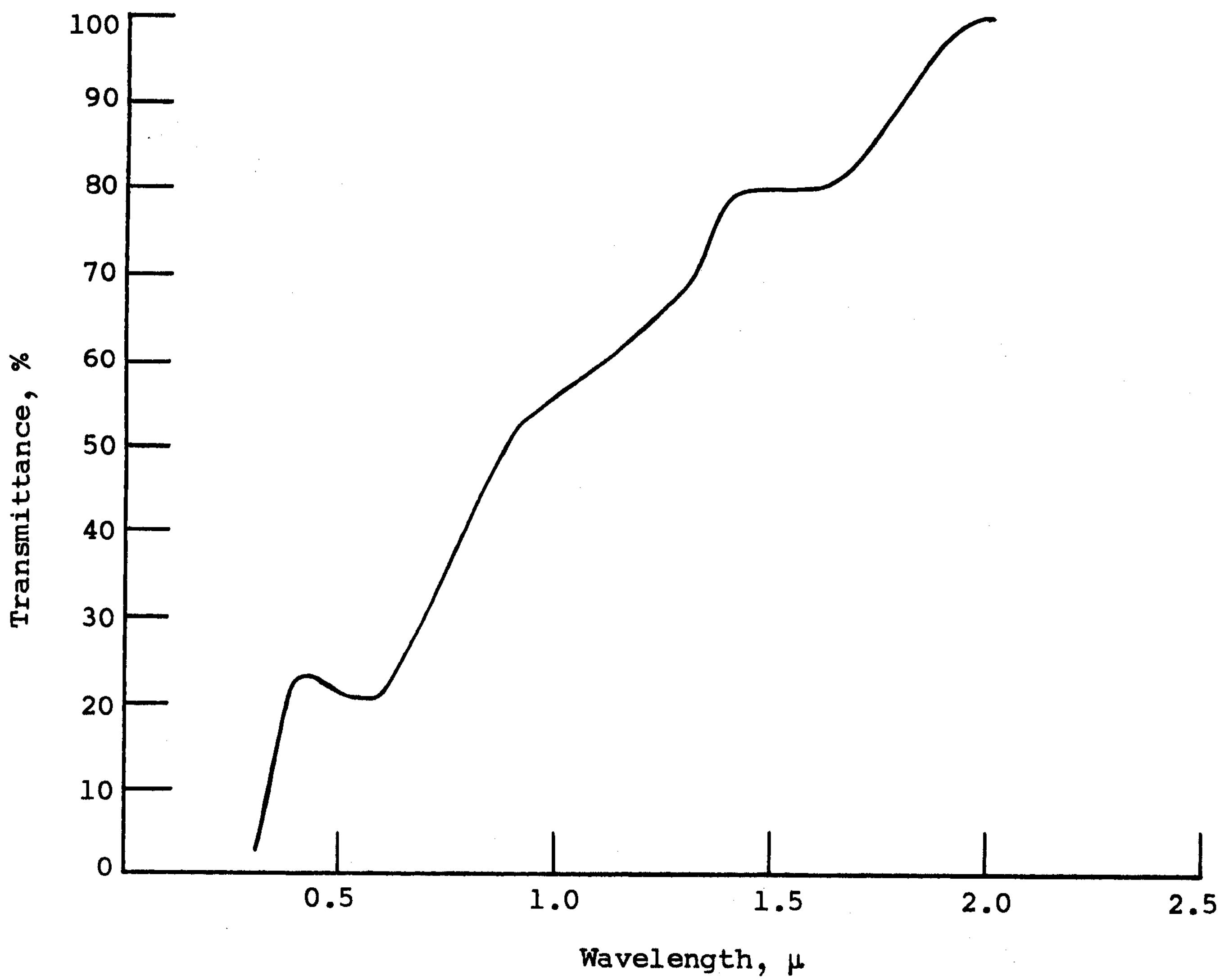

Figure 4

TRANSMITTANCE OF THIN SILVER CHLORIDE FILMS

films, but it does form a much stronger support.

Cleared photographic film may be another alternative support film and has in fact been used as a backing for silver bromide suspension layers on which scattering measurements have been made at shorter wavelengths.[3]

LOGBOOKS AND PERSONNEL

Experimental data are recorded in IITRI Logbooks C 13738 and C 13906. Dr. S. Katz is performing the theoretical analyses, J. Stockham is directing the laboratory studies, P. Schossberger prepared the silver halide specimens, and D. Vance performed the reflectance and transmittance studies.

Respectfully submitted,

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[3] Personal communication with C. R. Berry, Eastman Kodak.

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