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(NASA-CR-141777) BLACK-AND-WHITE SEPARATIONS OF SPACECRAFT ORIGINAL FILM (Technicolor Graphic Services, Inc.) 30 p HC $3.75 CSCL 14E

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TECHNICAL REPORT

Black-and-White Separations of Spacecraft Original Film (Preliminary)

Prepared Under

Contract NAS 9-11500
Task Order HT-133

Prepared By

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Technicolor Graphic Services, Inc.

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LYNDON B. JOHNSON SPACE CENTER
Houston, Texas

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March 1975

Photographic Technology Division
National Aeronautics and Space Administration
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BLACK-AND-WHITE SEPARATIONS OF SPACECRAFT ORIGINAL FILM

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INTRODUCTION

During the space program a large amount of photography has been acquired in both color and black-and-white. Much of this photography is of great historical and/or scientific importance and should be preserved with the best techniques available.

Unfortunately, most present day color materials employ dyes which fade over extended periods of time. While proper storage conditions can greatly reduce the rate of fading, adequate archival stability over periods of 50 to 100 years cannot be assured.

In order to best preserve these valuable color originals, silver separation negatives will be produced. These separation negatives have excellent stability, and meet archival storage requirements.

This preliminary report discusses several separation systems and gives a detailed procedure for the production and evaluation of separation negatives.

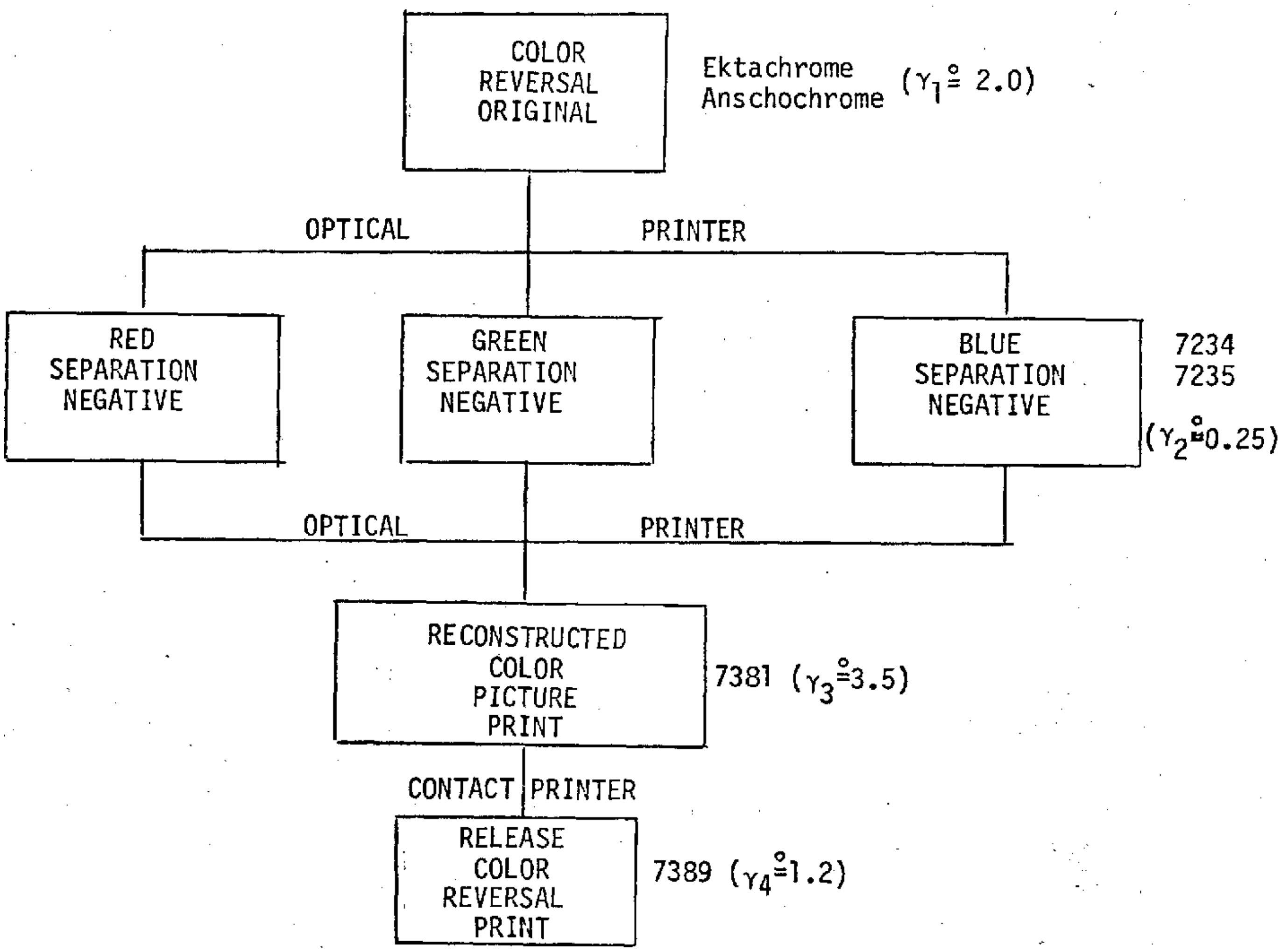
SEPARATION SYSTEM ALTERNATIVES

Several procedures for making black-and-white silver separations from spaceflight film were examined. Although all methods will yield a product capable of recombination into a full color image, they vary in the degree of complexity.

In choosing a separation system, some assumption has to be made about the mode of duplication. This will determine the choice of separation film and the tone reproduction criteria. Figures 1, 2, and 3 give three possible separation and duplication systems. A system gamma of 2.1 was chosen since this is in common usage in many photographic systems today. The product of the gammas of the individual steps including the original, must equal 2.1.

FIGURE 1

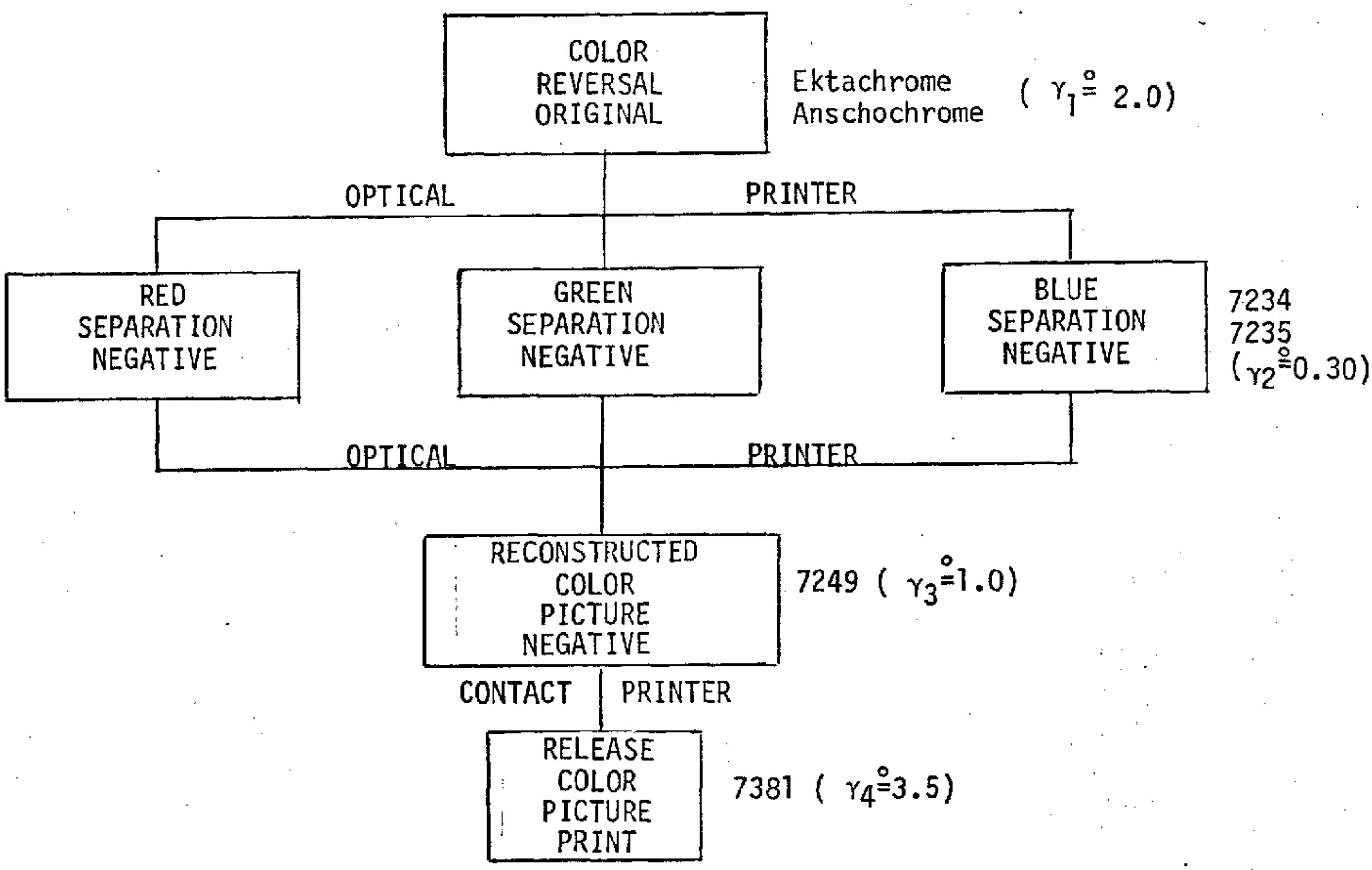
System I



This system would utilize totally in-house capabilities at the present time. Achieving a 0.25 gamma on the separation negatives could be a problem. Special chemistry would be required and curve linearity might be poor.

FIGURE 2

System II

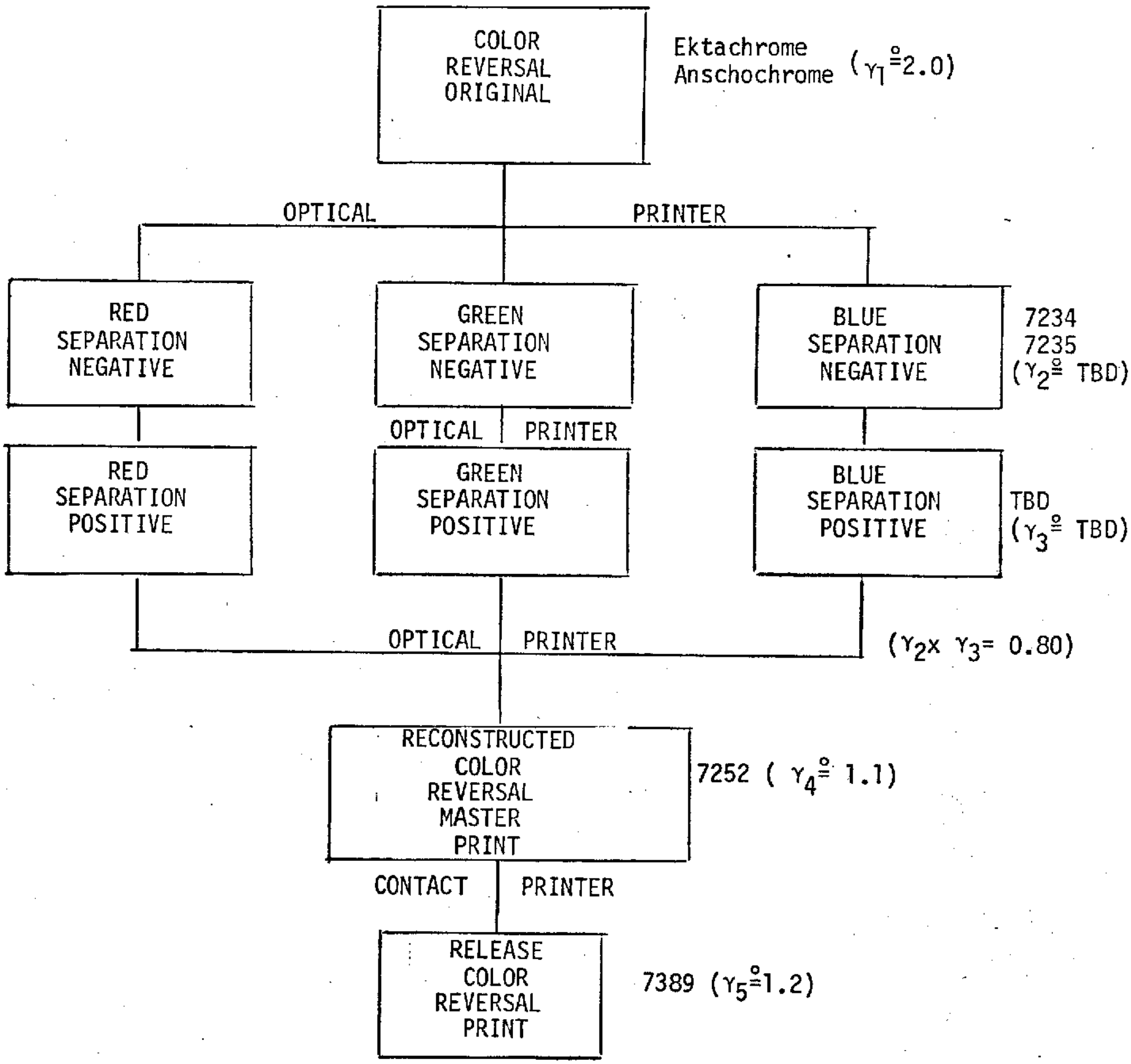


This system would use the Eastman Color negative/positive system exclusively. Use of the 7249 Color Reversal Intermediate film would require the CRI-1 process, which is not currently available at PTD.

As in system I, a very low gamma is required for the separation negatives due to the high gamma of the original and the 7381 print film.

FIGURE 3

System III



Choice of the film to make the separation positives will determine γ_2 and γ_3. Their product must equal 0.80 for the system above in order to have a system gamma of 2.1.

The separation positive film should be an Estar base material for best archival storage qualities. Although this method involves an additional step, it offers some advantages.

> Since the separation negative has already been made, the separation positive film need not be panchromatic, thus allowing a wider choice of films.

Consideration must be given to image orientation. One of the optical steps would have to be base to emulsion.

Film Selection Criteria

The film chosen for the final separations to be stored must have the following properties.

1. Contrast - the contrast dictated by the reproduction system must be easily achievable in standard developers.

2. Resolution - must be sufficiently high that it does not contribute significantly to image degradation in the duplication process.

3. Linearity - must have excellent straight line characteristics over a log E range of at least 2.0. This is necessary in order to avoid color balance problems in the subsequent duplication.

4. Granularity - must be extremely low, since most of the subsequent duplication stages result in contrast increases, especially when using the color print films.

5. Base - a clear polyester (Estar, Cronar) base is highly desirable for best dimensional stability. Non-uniform shrinkage or expansions would make reconstruction extremely difficult. The base must be clear, since any dyes used are subject to fading over extended periods of time.

6. Spectral Sensitivity - the only requirement here is that the film used to make the first separations from the original color transparency be panchromatic. Any subsequent duplicating films need not be panchromatic. This would open up a much wider range of films for the separations to be archived.

Other criteria such as compatibility of emulsion surface with other films, variation of gamma with wavelength, and type of backing must be considered.

Method of Separation Production

The method of separation used will depend on the film size. Since the 16mm film is generally intended for projection, the separation and reconstruction must be done for a full roll on a registering optical printer.

The 35mm and 70mm films do not have the consistent frame to frame spacing of a motion picture film. Thus, these rolls can be separated on a full roll basis, but will have to be recombined on a frame-to-frame basis. unless special registering equipment is used for the separations.

Filtration-Filters used for separation should be matched closely to the wavelengths on the dye absorption curves (Figure 4 and 5) where there is the least interlayer cross-talk. A suitable set for Ektachrome and Anscochrome dyes is Wratten 98, 74, and 70 (Figure 6, 7, and 8) for the blue, green, and red bands, respectively. While not as narrow-band as interference filters, these gelatin filters are very well matched to the dye absorptions and are easily inserted into the printer's optical system.

Exposure-All separations should be exposed to place the imagery on the straight line portion of the characteristic curve. Special care should be taken to insure that the highlights of the original are not exposed on any part of the shoulder, since this will make it nearly impossible to reconstruct with good color balance.

Some density analysis of the originals will aid in determining proper exposure.

Process Control-Equal gamma separations are necessary for proper color reproduction. This will probably necessitate a different process control for the red, green, and blue records.

Washing-An area of prime importance in archival separations is the elimination of residual processing chemicals, primarily thiosulfates and polythionates. The ANSI standard for archival storage (PH 1.41-1973) sets the maximum residual thiosulfate level at 0.7 micrograms/cm^2 (or 5 micrograms/$inch^2$), expressed as thiosulfate ion. This amount is permitted on each side of the film. Thus a film with a gelatin backing would be permitted a higher level of thiosulfate.

Rolls of archival separations will be tested for residual thiosulfate, using the methylene blue method[1]. Rewashing will be required if the thiosulfate level exceeds 0.7 micrograms/cm^2.

[1] Based on the work of Warburton and Przyblowicz, Photographic Science and Engineering, 10:86, March-April 1966. A detailed procedure is given in the Eastman Kodak processing manuals for the ME-4/ECO-3 process as method 1330A.

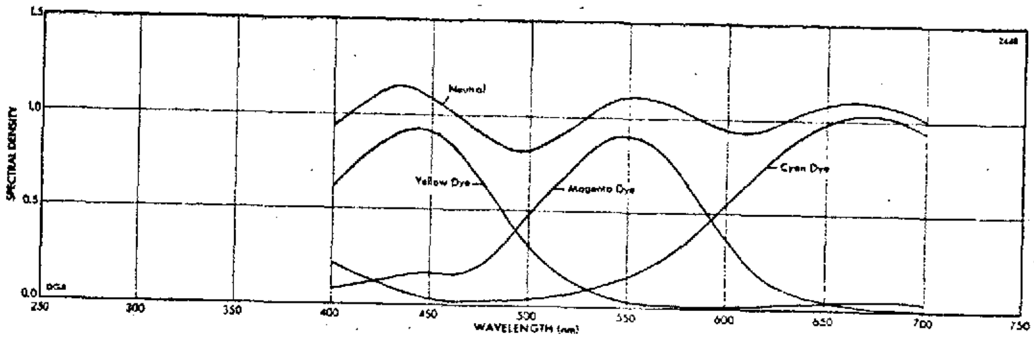


FIGURE 4. Ektachrome Dye Absorptions

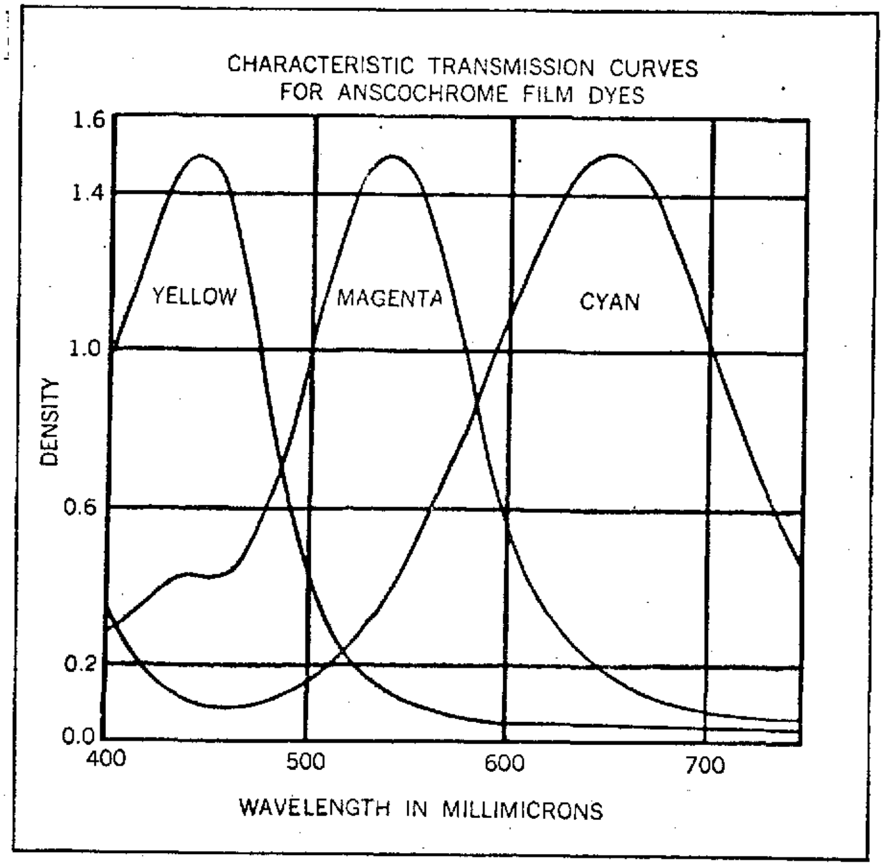


FIGURE 5. Anschochrome Dye Absorption

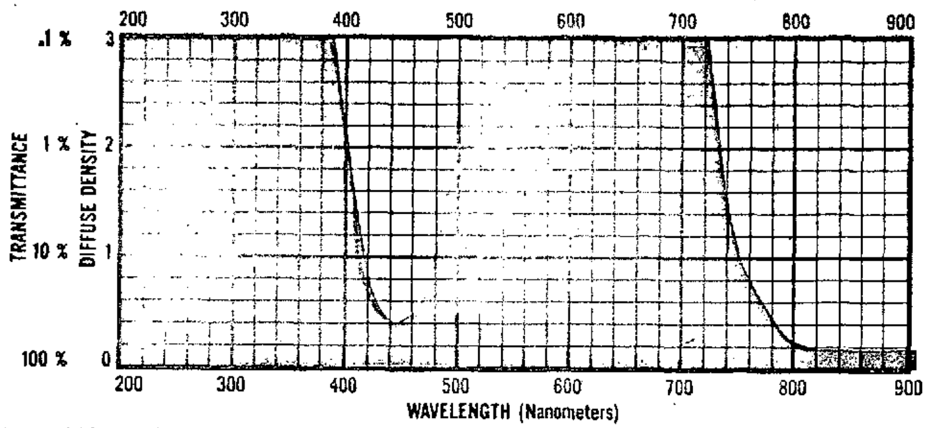


FIGURE 6. Wratten 98

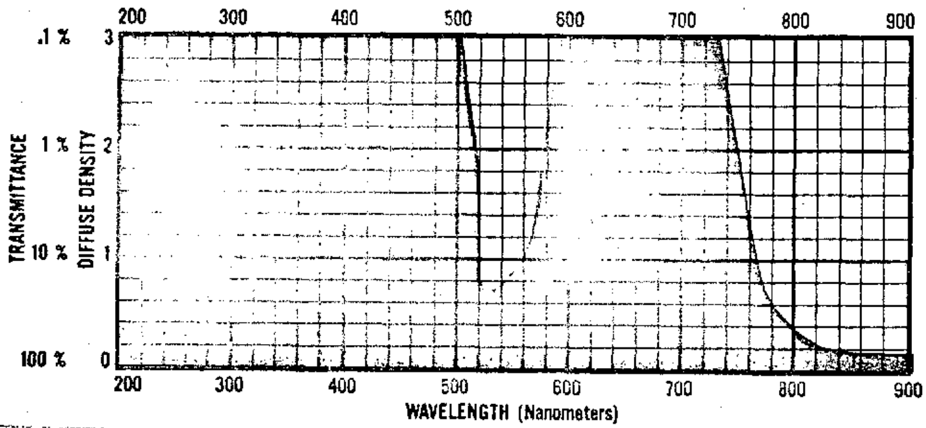


FIGURE 7. Wratten 74

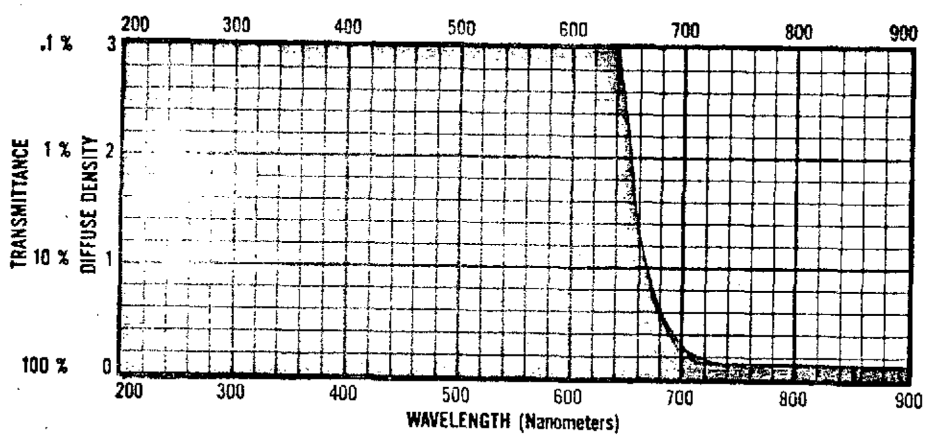


FIGURE 8. Wratten 70

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OF POOR QUALITY

Construction of Test Roll
A test roll will be constructed in 16mm, 35mm, and 70mm in order to evaluate the separation production system and familiarize laboratory personnel with the procedures involved in making color separations. This is essential if the actual spaceflight imagery is to be handled in an orderly manner. The test roll will also be used for setting up the printer and process before running the archival separations.

The roll will consist of the following:

1. Imagery on the same or similar film types as the spaceflight film.

2. Resolution targets in red, green, and blue to be used to evaluate resolution retention.

3. Sensitometric step tablets on the same film type as the originals.

4. A color coded indicator to verify the color of each record. This can simply be the words RED, GREEN, AND BLUE, each recorded in its own color. Thus, when exposed onto the separation film, only the color corresponding to the separation filter will print through.

This test roll should be spliced into every roll of original, since it forms part of the quality control system.

Quality Control Program

Careful monitoring of the separations during and after production is necessary in order to assure that the correct high-quality products are sent to the archives. Checks must be made of the physical and sensitometric quality plus the level of residual thiosulfate remaining in the film.

The following checks must be made on every roll of archival separation film.

1. Density- A print through density step must be checked. The criterion is that the maximum useful density of the original should print back to a density at the start of the separation film straight line, generally around 0.40. Readings must be made with a status A blue densitometer filter.

2. Gamma - The specified gamma will be checked and should be the same for each record. This will be determined by taking the density difference between two steps printed on the straight line of the separation film.

3. Record Color Verification- A check of the color indicator will be made to determine that all three records are present from each roll of the original.

4. Physical Inspection- An examination will be made of each record to check for scratches, streaks, fog, etc. A report will be made of these conditions and a decision made to accept or reject the film based on the effect of these anomalies on the imagery.

5. Resolution- The three-bar targets will be read, and must exceed a minimum specified value.

   The following checks will be made on sample rolls with a sampling frequency to be determined.

6. Residual chemicals- A methylene blue thiosulfate test will be performed on each roll. The residual thiosulfate level must be less than 0.7 micrograms/cm^2. Results greater than this amount will require rewashing of the roll and retesting until the specification is met.

7. Registration Accuracy - A check of the accurate registration of the records will be made with a device which superimposes the records, either optically or physically.

Conclusions

Of the three systems examined, System III is considered the most practical for our needs. The very low separation negative gamma required in the other two systems would be difficult to achieve. It would also be very difficult to work with, since the gamma differences between records would have to be extremely small, around 0.03, in order to achieve good color balance in reproduction.

In using System III, we would only proceed as far as the separation negative. The future user would then be required to make the separation positives, color reversal master, and color reversal release print. Of course, it would also be possible, by means of an additional step, to make a duplicate negative at the 0.25 or 0.30 gamma required for System I or II.

Another advantage of system three is that equal gammas of the three separations are not essential. Since separation positives must be made for reconstruction, the product gammas can be equalized at that point. This will greatly simplify process control and speed the production schedule.

APPENDIX A

PROCEDURE FOR PRODUCTION OF ARCHIVAL SEPARATIONS

1. Obtain original film from vault.

   1.1 Films will generally be withdrawn in chronological order.

   1.2 Production will start with the 16mm films.

2. Inspect and catalog original film.

   2.1 Fill out all information required on 5 x 7-inch index card.

      2.1.1 Mission - e.g. Gemini 12, Mercury 2, etc.

      2.1.2 Roll or Magazine designation - number or letter.

      2.1.3 Film Type - manufacturer, number, name, e.g. EK SO-168 (EF Ektachrome)

      2.1.4 Size - width in millimeters

      2.1.5 Sensitometry - present, yes or no: and type - e.g. 21 step I-B

      2.1.6 Roll length - in feet

      2.1.7 Description - general description of subject matter - e.g. Earth photography, EVA, moon walk, etc.

      2.1.8 Physical condition - document any large anomalies, e.g. bad scratches, fog in imagery, splices in roll, etc.

      2.1.9 Color balance - overall color balance and estimate of whether printable or not.

3. Splice in test sections - head and tail

   3.1 Test sections will include the following:

      3.1.1 Resolution targets - red, green, and blue

      3.1.2 Color-coded indicator

      3.1.3 Sensitometric step tablet - Ektachrome MS or EF

      3.1.4 Standard Imagery - Ektachrome MS or EF

4. Clean original film.

5. Print test roll

5.1 Exposure to be determined by initial testing with later results used to update.

6. Process test roll

6.1 Process to achieve the specified gamma

6.1.1 All density readings must be made with a status A Blue filter This is done in order to read printing density rather than visual density, since the separation negative is most likely to be printed onto a blue-sensitive black-and-white duplicating film.

6.1.2 The processing configuration will be determined for each color record.

7. Check control parameters

7.1 Gamma - specified value ± 5%

7.2 Density - return specified step to a density + tolerance TBD.

7.3 Resolution - a minimum specified print-through resolution must be maintained.

7.3.1 This value will be recorded for each color record.

7.4 If any of these parameters are not within tolerance, corrections will be made, and the sequence returns to step 5.

8. Print original

8.1 Use printer setting determined with test roll.

9. Process print of original

9.1 Use processor configuration determined with test roll.

10. Re-Wash

10.1 Separation film will be rewashed with water and one tank of hypo clearing bath if required to meet archival chemical retention standards.

11. Check control parameters

11.1 Same tolerances as in step 7. apply.

11.1.1 Density

11.1.2 Gamma

11.1.3 Resolution

11.2 Record all readings

11.2.1 Final acceptable readings must be recorded on the 5 x 7-inch index card for each roll of original.

11.3 Inspect for physical condition.

11.4 If any parameters are out of tolerance, make corrections and return to step 8.

12. Check residual thiosulfate content

12.1 Must be less than 0.7 micrograms/cm2 of thiosulfate ion

12.2 Use methylene blue method (ME-4 method 1330A - based on ANSI standard PH 4.8-1971)

12.3 Unacceptably high thiosulfate content will require rewashing.

12.4 Record final acceptable readings.

13. Steps 5 through 12 must be repeated for the red, green, and blue records.

14. Verify that all three color records are present, and check their registration.

14.1 Check color-coded indicator.

14.1.1 On each record, only one of the words red, green, or blue will be printed through by its own color.

15. Condition all three separation rolls.

15.1 Temperature - TBD

15.2 Relative humidity - TBD

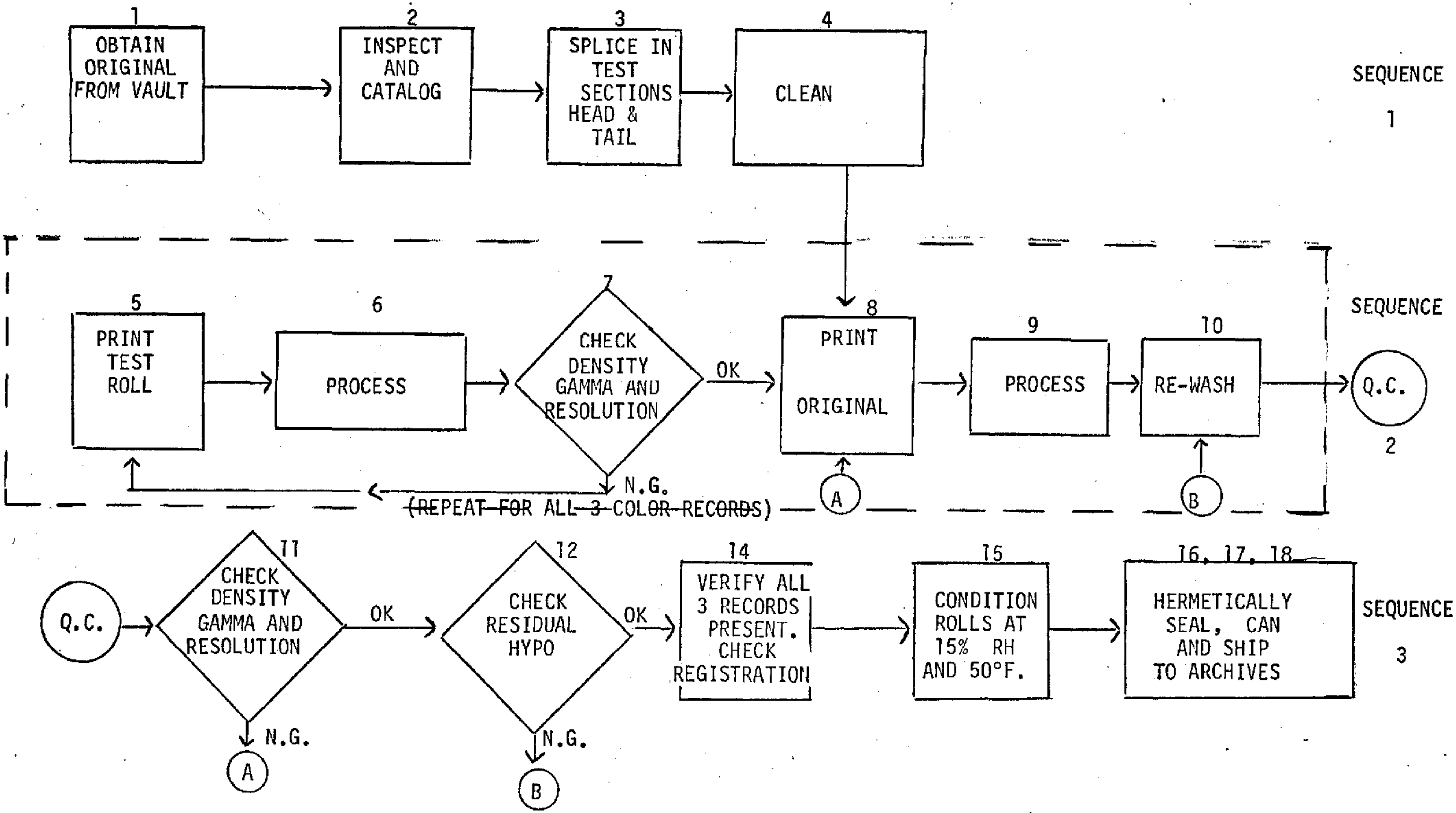
15.3 Conditioning period - TBD

16. Hermetically seal all three rolls into a single package in the conditioning environment.

17. Place package in plastic light-proof film can and seal with black vinyl tape.

18. Ship to archives

18.1 Record date of shipment on 5 x 7-inch index card.



NOTE: Sequences 1, 2, and 3 can be going on simultaneously, on the same or different rolls. Numbers above blocks refer to steps in procedure outline.

ARCHIVAL SEPARATION FLOW CHART

APPENDIX B

American National Standard Specifications for Photographic Film for Archival Records, Silver-Gelatin Type, on Polyester Base

1. Scope

1.1 This standard applies only to safety polyester-type films having silver-gelatin type emulsions processed to produce a black-and-white photographic image. Color films are excluded.

1.2 Silver images that have been chemically altered by treatments such as toning, reduction, or intensification are excluded. However, a gold protection treatment is not excluded.

1.3 This standard applies to microfilm, motion-picture film, roll film, and sheet film.

1.4 This standard applies only to photographic films intended for archival record films.

1.5 Lacquers and lubricants have been applied to processed film for fungus and scratch protection without any known detrimental effect on permanence. However, the wide variety of possible materials does not permit their specification in this standard.

2. Definitions

archival record film. A photographic film composed and treated so that under archival storage conditions (see American National Standard Practice for Storage of Processed Silver-Gelatin Microfilm, PH5.4-1970, and American National Standard Practice for Storage of Processed Safety Photographic Film Other Than Microfilm, PH1.43-1971) it is suitable for the preservation of records having permanent value (see Appendix D).

emulsion layer. The silver-gelatin image layer of the processed film.

film base. The plastic support for the emulsion and backing layers.

noncurl backing layer. A layer usually made of gelatin, applied to the side of the film base opposite that of the emulsion layer, for the purpose of preventing curl. It is comparable to the emulsion layer in thickness and is not removed in processing. (Antihalation or other layers removed in processing are excluded from this definition.)

safety poly(ethylene terephthalate) base. Film base composed mainly of a polymer of ethylene glycol and terephthalic acid.

3. Film Base Requirements

3.1 Film Base Type. The base used for archival record film, as specified in this standard, shall be of a safety poly(ethylene terephthalate) type and can be identified by the method described in 7.1.

3.2 Viscosity Retention. The relative viscosity of a solution of film base obtained from processed film shall not show a loss which exceeds 5% as the result of accelerated aging of the processed film. The accelerated aging shall be accomplished as described in 7.2 and the viscosity determined as described in 7.3.

4. Processed Film Requirements

4.1 Ignition Time. The film shall meet the requirements for ignition time specified in American National Standard Specifications for Safety Photographic Film, PH1.25-1969 (R-1965).

4.2 Burning Time. The film shall meet the requirements for burning time specified in American National Standard PH1.25-1969 (R-1965).

4.3 Nitrate Nitrogen Content. The film shall not contain over 0.15% nitrate nitrogen, as determined by the method described in American National Standard PH1.25-1969 (R-1965).

4.4 Free Acidity and Acidity Increase. The film shall not have a free acidity greater than the equivalent of 1.0 ml of 1/10 normal sodium hydroxide solution per gram of film when measured as described in 7.4. The volume of 1/10 normal sodium hydroxide equivalent to the free acidity of the processed film shall not in-

Table 1
Limits for Tensile Properties and Tensile Properties Loss on Aging for Poly(ethylene Terephthalate) Films

	Tensile Strength at Break	Elongation at Break
1. Unheated Film: Minimum permissible tensile properties	20 000 psi	75%
2. Film after Accerated Aging: Maximum permissible loss in tensile properties compared with unheated film	10%	10%

Table 2
Limits for Mushiness Value Loss

	Original Mushiness Value	
	50 Grams or Less	Greater than 50 Grams
Maximum permissible mushiness value loss as a result of accelerated aging (see Note)	50%	25%

NOTE: The increase in the limits for percent loss in mushiness after accelerated aging with decrease in original mushiness level is necessary because the test is not sufficiently accurate to distinguish small differences.

crease more than 0.5 ml over its original value after accelerated aging as described in 7.2.

4.5 Tensile Properties and Tensile Properties Loss. The film samples shall be processed and dried under the conditions used for the film records. Processed film shall have a tensile strength and elongation at break as specified in line 1 of Table 1 when tested as described in 7.5. The loss in tensile properties after accelerated aging, as described in 7.2, shall not exceed the percentage specified in line 2 of Table 1.

5. Processed Emulsion and Backing Layer Requirements

5.1 Layer Adhesion

5.1.1 Tape-Stripping Adhesion. The processed film shall not show any removal of the emulsion layer or backing layer when tested as described in 7.6.

5.1.2 Humidity-Cycling Adhesion. The emulsion layer or backing layer of the processed film shall not show separation or cracking which would possibly impair its intended use, when tested as described in 7.7 (see Appendix D).

5.2 Mushiness Loss.[1] The processed film shall not show a loss in the mushiness value of the emulsion and backing layer which exceeds the percentage specified in Table 2 as the result of accelerated aging of the processed film. The mushiness value shall be determined as described in 7.8 when the accelerated aging is performed as described in 7.2.

5.3 Blocking. Processed film shall show no evidence of blocking (sticking), delamination, or surface damage when tested as described in 7.9. A slight sticking of the film samples which does not result in physical damage or a change in the gloss of the surface shall be acceptable.

5.4 Thiosulfate Concentration. Films may be fixed in solutions containing either sodium thiosulfate or ammonium thiosulfate [4]. After processing, the film shall not contain a greater concentration of residual thiosulfate calculated as thiosulfate ion ($S_2O_3^{=}$) than specified in Table 3, when determined by the methylene blue test method described in American National Standard Methylene Blue Method for Measuring Thiosulfate and Silver Densitometric Method for Measuring Residual Chemicals in Films, Plates, and Papers, PH4.8-1971 [5].[2]

The analysis for thiosulfate shall be made on a film sample from a clear area and shall be made within two weeks after processing. (See Appendix A.) The test method does not measure any change in the sample between the time of processing and the time of analysis but is used to judge the keeping of the film following the time of the test.

Films having photographic layers on both sides of the base, or films having a photographic layer on one side and a noncurl backing layer on the reverse side, are permitted the specified concentration of residual thiosulfate in each side of the film.

Films are classified according to two broad classes of graininess in Table 3. If the graininess of the film is not known, a sample of the exposed and processed

[1] High mushiness values denote a high wet strength of the emulsion and backing layers, and the loss in the mushiness value on aging is a measure of degradation.

[2] Two methods for measuring residual chemicals in film are described in American National Standard PH4.8-1971. At this time, only the methylene blue method is considered sufficiently reliable to report thiosulfate concentrations at the archival level of 0.7 μg (microgram) $S_2O_3^{=}$ per cm^2. Unlike the methylene blue test method, the silver densitometric test method measures polythionate decomposition products and other residual chemicals in addition to thiosulfate. It has been used to indicate archival quality if the optical density produced in the test is less than 0.02.

Table 3
Limits for Thiosulfate Concentration

Classification of Films According to Graininess of Developed Image	Maximum Permissible Concentration of Thiosulfate Ion, $S_2O_3^{=}$ in Micrograms per cm^2 (See Note 1)
Class 1 Fine-grain copying, duplicating, and printing films (includes ordinary microfilms) (See Note 2)	0.7
Class 2 Medium-grain continuous-tone camera films (negative and reversal) and coarse-grain x-ray films	2

NOTE 1: For films having photographic layers on both sides, or a noncurl backing layer, value are for each side of film.

NOTE 2: For films having finer grain structure than those types covered by Class 1 (that is, high-resolution films), it is desirable to have a residual thiosulfate concentration less than 0.7 $\mu g/cm^2$ (0.004 mg/in^2).

film should be examined under a comparison microscope at 20X magnification or greater, along with samples whose classifications according to graininess are known, and classified accordingly. Any film which cannot be classified definitely according to graininess shall be considered as Class 1. (See Appendix A.)

6. Image Stability

The film image shall show no degradation which would impair the film for its intended use, when tested as described in 7.10.

7. Test Methods

7.1 Identification of Film Base. All emulsion and backing layers shall be removed from a sample of the unknown film, either by scraping or by the use of an enzyme solution. All sublayers shall then be removed by scraping. A sample of the base material shall then be prepared by scuffing the surface with a suitable tool. It has been found that a hardened steel file or a section of file approximately 2.5 X 2.5 cm (1 X 1 inch) and having a No. 0 surface is satisfactory.

The general procedure is to move the scuffing device back and forth over the sample manually while exerting a very slight pressure. This removes the top layer of the base as a very fine dust, which is carefully brushed into a mortar. The sample shall be mixed with about 100 times its weight of potassium bromide previously ground to about 200 mesh. A strip or pellet shall be prepared as described in the literature [6]. An infrared absorption curve shall be obtained from the pellet so prepared, by means of an infrared spectrophotometer. By comparing the infrared absorption curve for the unknown with curves for known polymers, the identity of the unknown can be established [7].

7.2 Accelerated Aging Conditions.[3] The test samples shall be mounted in a sample rack so they are freely exposed to surrounding air. The rack shall then be placed in a glass laboratory desiccator jar. Care shall be taken to prevent contact of the samples with each other or with the walls of the jar. The jar shall be heated in a forced air circulating oven for 72 hours at 100°C ± 2°C (212°F ± 4°F). The atmosphere within the jar shall be maintained at 20% ± 2% relative humidity. A relative humidity of approximately 20% can be obtained by keeping a saturated solution of potassium acetate in water at the bottom of the jar. Care must be exercised so that the saturated solution contains an excess of undissolved crystals at 100°C. The undissolved crystals shall be completely covered by a layer of saturated salt solution and the surface area of the solution should be as large as practical. The jar and salt solution shall be at 100°C for at least 20 hours prior to use to ensure adequate equilibrium.

Alternatively, exposure to these temperature and humidity conditions may be provided by means of a conditioning air cabinet in which the samples are suspended to keep them separated from each other. No other materials shall be in the same environment as the test samples during the heating period.

[3] Processed film must be subjected to the accelerated aging conditions to meet the requirements for viscosity retention, acidity increase, tensile loss, and mushiness loss.

In subsequent sections, samples subjected to these accelerated aging conditions are designated as "heated samples."

7.3 Relative Viscosity Test. Measurements shall be made on two unheated and two heated samples of processed film that have the same average silver density and weighing 1.000 gram each. Each sample shall be immersed in a separate 100-ml volumetric flask containing approximately 15 ml of a mixture of 60% phenol and 40% chlorobenzene by weight.[4] The base may be dissolved by repeated shakings for 15 minutes in a 140°C ± 1°C (284°F ± 2°F) oil bath.

The flasks shall be immersed in a water bath maintained at 25°C ± 0.1°C (77°F ± 0.2°F) until temperature equilibrium has been reached. Solvent shall be added to adjust the volume of the solution to 100 ml and the contents thoroughly mixed. Nonsupport layers shall be allowed to settle to the bottom of the flask. A portion of the liquid shall be transferred, with filtration[5] if necessary, to an Ostwald pipette or an equally suitable capillary viscometer[6] immersed in a constant temperature bath at the same temperature. The pipette chosen should have a flow time between 70 and 110 seconds for the solvent. The volume taken should be sufficient to half fill the lower bulb of the pipette. The time of flow of the solution through the capillary of the pipette shall be measured to the nearest 1/5 of a second. The time of flow shall also be measured for the same volume of the pure solvent. Not less than three readings shall be made for each portion and the results averaged. The relative viscosity is the ratio of the time of flow of the solution to the time of flow of the solvent. Duplicate determinations shall be made on both the unheated and heated samples and the duplicates should agree within 1/2 second. The averages shall be calculated separately for the unheated and heated film.

7.4 Free-Acidity Test. Measurements shall be made on two unheated and on two heated samples of processed film that have the same average silver density and weigh approximately 1 gram each. Each sample shall be accurately weighted and shall be immersed in approximately 10 ml of benzyl alcohol. The polyester support shall be dissolved by heating,[7] leaving the emulsion layer. The solution shall be cooled and diluted by the addition of 80 ml of chloroform and 80 ml of ethyl alcohol denatured by the 2B or 3A formula. Acidity shall be determined by direct titration of the mixture with 1/10 normal sodium hydroxide, using cresol purple as the indicator. A blank titration shall also be made on the solvent mixture. The acidity shall be reported as the difference between the two titrations in milliliters of 1/10 normal sodium hydroxide per gram of film. The averages shall be calculated separately for the unheated and heated film.

7.5 Tensile Strength and Elongation Test

7.5.1 Sample Preparation. Processed film already in 16mm form may be tested in this width. In the case of perforated 16mm film, samples shall be cut from between the perforations. Films in other sizes shall be cut 15–16 mm (0.59–0.63 inch) wide and at least 15 cm (6 inches) long, using a sharp tool which does not nick the edges of the sample. Five samples are required for the unheated film and five samples for the heated film. The samples to be heated and the control samples shall be cut alternately and contiguously from a single piece of film. The thickness of each sample shall be measured with a suitable gage to the nearest 0.002 mm (0.0001 inch) and the width to the nearest 0.1 mm (0.005 inch).

7.5.2 Accelerated Aging. Five samples shall be subjected to accelerated aging as described in 7.2.

7.5.3 Conditioning. All samples, both unheated and heated, shall be conditioned at 21°C–24°C (70°F–75°F) and 50% ± 2% relative humidity for at least 15 hours. This may be accomplished by means of an air-conditioned room or a conditioning air cabinet. The samples shall be supported in such a way as to permit free circulation of air around the film and the linear air velocity shall be at least 15 cm (0.5 foot) per second.

7.5.4 Procedure. The film samples shall not be removed from the conditioning atmosphere for testing. The tensile strength and percent elongation at break of unheated and heated film samples shall be tested alternately by means of a tensile machine, as specified in Method A, Static Weighing – Constant-Rate-of-Grip Separation Test, of American National Standard Method of Test for Tensile Properties of Thin Plastic Sheeting, K65.76-1971 (ASTM D 882-67 (1972)). The initial grip separation shall be 10 cm (4 inches) and the rate of grip separation shall be 5 cm (2 inches) per minute. The tensile strength and elongation at break shall be calculated separately for the unheated and heated film.

[4] Caution should be taken with the mixture of phenol and chlorobenzene to avoid inhalation of the vapor or contact with the skin. It may be rapidly absorbed through the skin and can cause severe burns. In case of contact, area should be washed with water for at least 15 minutes. In case of contact with eyes, medical attention should be obtained. Solvent mixture should be used only with adequate ventilation and hot solutions should be used only in a hood.

[5] Filtration must be rapid to avoid solvent loss. This may be accomplished by filtering through a porous glass wool pad.

[6] These solutions are hazardous and must not be drawn into pipettes by mouth. Vacuum or water pumps should be used.

[7] Caution should be taken to avoid inhalation of the vapor or prolonged or repeated contact with the skin. Heating should take place in a hood.

7.6 Tape-Stripping Adhesion Test

7.6.1 Sample Preparation. Although the dimensions of the processed film sample are not critical, one dimension shall be at least 15 cm (6 inches). Four samples are required for the emulsion surface and four samples for the backing layer, if present.

7.6.2 Conditioning. Samples shall be conditioned as described in 7.5.3.

7.6.3 Procedure. The film samples shall not be removed from the conditioning atmosphere for testing. A strip of pressure-sensitive plastic base adhesive tape about 15 cm (6 inches) long shall be applied to the surface of the processed film. The tape shall be pressed down with thumb pressure to assure adequate contact, leaving enough tape at one end to grasp. No portion of the tape shall extend to the edges of the film samples or extend to film perforations. The sample shall be held firmly on a flat surface and the tape rapidly removed from the film surface. This shall be accomplished by peeling the tape back on itself and pulling the end so that it is removed from the film at an angle of approximately 180 degrees. Removal by the tape of any portion of the surface layer on any of the samples shall be considered failure.

The results of the tape-stripping test may be very dependent upon the adhesive tape used if the bonding force between it and the particular film surface under test is not sufficiently high. For this reason a minimum bonding force is specified for this test. This bonding force shall be determined by applying the adhesive tape to the film surface in the same manner as described in the tape-stripping test. The tape shall be rapidly peeled back from the film surface at an angle of approximately 180 degrees. The peelback force required to separate the tape from the film shall be measured by a suitable device such as a strain gage or maximum reading spring scale. A bonding force of at least 900 grams per cm of tape width (5 pounds per inch of width) is required.

7.7 Humidity-Cycling Adhesion Test

7.7.1 Sample Preparation. A sample 5 cm (2 inches) square or 5 cm by the film width is convenient, but the dimensions are not critical. Two samples of processed film shall be selected from a high silver density area.

7.7.2 Procedure. The test samples shall be mounted in a sample rack and placed in a glass laboratory desiccator jar in such a way that they are freely exposed to the required conditioning atmospheres. The jar shall be put into a forced air circulating oven for 8 hours at 50°C ± 2°C (122°F ± 4°F). The atmosphere within the jar shall be maintained at 96% ± 2% relative humidity, which can be obtained by keeping a saturated solution of potassium sulfate in water [8] in the bottom of the jar. After the 8-hour time period, the samples and sample rack shall be placed for 16 hours in a second desiccator jar which is also in the same oven. The atmosphere within this second jar shall be maintained at 11% ± 2% relative humidity, which can be obtained by keeping a saturated solution of lithium chloride in water [8] in the bottom of the jar. The precautions described in 7.2 shall be taken to ensure that in both cases the proper humidity is obtained.

A time period of 8 hours at the high humidity and 16 hours at the low humidity shall constitute one cycle.[8] The film samples shall be subjected to twelve humidity cycles, after which the film samples shall be removed from the sample rack and the emulsion and any backing layer examined for any evidence of peeling, flaking, or cracking produced as a result of the humidity cycling treatment.[9] The film shall be examined under the same magnification and lighting conditions as normal for the end use of the product. During a weekend interruption in the cycling procedure, the film samples shall be kept at 50°C ± 2°C (122°F ± 4°F) and 11% ± 2% relative humidity.

7.8 Emulsion Mushiness Test. Measurements shall be made on two unheated and on two heated samples of processed film. The emulsion mushiness value shall be determined as described in the literature [9]. This involves the application of a gradually increasing load on a 0.015-inch (0.3811-mm) radius spherical sapphire stylus as it travels against the emulsion surface. Processed film samples shall be immersed in 21°C (70°F) water and then placed horizontally, emulsion-surface up, in the tray of the apparatus containing 21°C (70°F) water. After 15 minutes total immersion time, the sapphire stylus shall be placed on the emulsion surface under zero load. The stylus load shall be gradually increased. The mushiness value shall be taken as the stylus load at which the stylus makes the first rupture or plowing of the emulsion surface. This rating may be made by reflected or transmitted light. Three measurements shall be made on each film sample. The averages shall be calculated separately for the unheated and heated film.

7.9 Blocking Test. At least five samples of processed film shall be conditioned to 62% ± 2% relative humid-

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[8] This can be most easily accomplished by placing the samples in the 96% relative humidity jar in the morning and in the 11% relative humidity jar in the evening.

[9] Films may sometimes exhibit small pinholes in the image after processing. These can be caused by dirt or dust particles on the emulsion surface at the time the raw film is exposed and should not be confused with holes or cracks in the emulsion layer. The existence of such pinholes in the image prior to humidity cycling should be noted so that their presence does not lead to the false interpretation of an adhesion weakness.

ity at 40°C ± 2°C (104°F ± 4°F). A sample size of 5 cm (2 inches) square is convenient where the size of the film permits, but the dimensions are not critical provided all samples are of uniform size. The samples shall be placed in a glass laboratory desiccator jar so that they are freely exposed to the required conditioning atmosphere for at least 15 hours. The jar containing the samples shall be put into a forced air circulating oven at 40°C ± 2°C (104°F ± 4°F). A relative humidity of approximately 62% can be obtained by keeping a saturated solution of sodium nitrite [10] in water at the bottom of the jar. The precautions described in 7.2 shall be taken to ensure that the proper humidity is obtained.

After moisture equilibrium is attained, the jar shall be removed from the oven. Without removing the film samples from the jar, at least five film samples shall be stacked so that the emulsion surface of one sample is against the back surface of the adjacent sample. The stack shall be placed under a uniform pressure of 0.35 kg/cm^2 (5 psi). This may be accomplished by placing a weight on the film stack, the dimensions of the weight being greater than those of the film samples. The jar containing the weighted stack shall be put back into the forced air circulating oven for 3 days at 40°C (104°F). Alternatively, exposure to the conditions of temperature and humidity may be provided by means of air conditioning cabinets or rooms.

The film stack shall be removed from the oven and allowed to cool. The film samples shall then be individually removed from the stack and observed for evidence of film blocking (sticking).

7.10 Image Stability Test. Three samples of processed film which contain similar and representative image areas shall be prepared. A sample size of 5 cm (2 inches) square is convenient for sheet film products or 5 cm (2 inches) in length by the film width for roll film products. These sample dimensions are not critical. Two samples shall be mounted so that they are freely exposed to surrounding air and shall be incubated for 30 days at 60°C ± 2°C (140°F ± 4°F) and 70% ± 2% relative humidity. This may be accomplished best by means of an air conditioning cabinet. After the incubation period, the film samples shall be compared visually with the unincubated comparison sample which has been stored at room temperature and at a relative humidity not exceeding 60%.

8. Revision of American National Standards Referred to in This Document

When the following American National Standards referred to in this document are superseded by a revision approved by the American National Standards Institute, Inc, the revision shall apply:

American National Standard Method of Test for Tensile Properties of Thin Plastic Sheeting, K65.76-1971 (ASTM D 882-67 (1972))

American National Standard Specifications for Safety Photographic Film, PH1.25-1969 (R-1965)

American National Standard Practice for Storage of Processed Safety Photographic Film Other Than Microfilm, PH1.43-1971

American National Standard Methylene Blue Method for Measuring Thiosulfate and Silver Densitometric Method for Measuring Residual Chemicals in Films, Plates, and Papers, PH4.8-1971

American National Standard Practice for Storage of Processed Silver-Gelatin Microfilm, PH5.4-1970

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