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EVALUATION OF MOTION-PICTURE FILM FOR PERMANENT RECORDS

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ABSTRACT

In previous publications, the results have been reported of studies on the stability of motion-picture film and its care in libraries. To assist the user of film in securing a material of high quality, this paper gives detailed test methods for determining folding endurance, pH, copper number, and relative viscosity. It is suggested that for record purposes film having a base of cellulose acetate that is free from cellulose nitrate be required. The cellulose acetate film should have the following resistance to an aging test consisting in heating 72 hours at 100° C: Retention of folding endurance, not less than 75 percent; decrease in relative viscosity, not over 3.0 percent; increase in copper number, not more than 0.2; a change in pH of not over 0.5. Developed film should be free from sodium thiosulphate (hypo).

CONTENTS

I. INTRODUCTION

Motion-picture film of the cellulose acetate type appears to be destined for very wide use for record purposes. Its use facilitates the rapid and inexpensive copying of books and manuscripts. Records on this medium require only a fraction of the storage space required for the same on paper, and accelerated aging tests at the National Bureau of Standards [1] have shown the acetate film, if well made and properly developed and fixed, to be as stable as paper used for permanent records. The preservation of acetate film has been described in a previous publication.[2] The purpose of this paper is to assist the user in selecting film for record purposes, by describing in detail tests that are applicable, and by suggesting limiting values of quality.

[1] J. Research NBS 17, 871 (1936) RP950.
[2] J. Research NBS 17, 753 (1936) RP942.

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II. TESTING

The stability of film was determined by measuring the effects of accelerated aging on some of its properties. The properties tested were flexibility, which is the physical property most closely related to use requirements, was measured by a folding test; acidity, which is a measure of the harmful acid present, was expresed as pH value; the copper number, a measure of the degraded cellulose present, was determined; and the relative viscosity in acetone solution was measured. A lowering of viscosity is considered an indication of breakdown in the molecular structure of a cellulose compound. These tests were made on samples before and after accelerating aging, and the results compared to find the changes occurring.

1. ACCELERATED AGING

The aging treatment used at the Bureau consists in oven-aging at 100° C in dry air for a period of 72 hours. This test of stability was chosen because it was expected that film, having a cellulose compound as a base, might behave similarly to paper, for which the results of such heat treatment are in accord with the general experience in the use of papers. The results of the accelerated aging of papers also agree well with those obtained by exposure to sunlight,[3] exposure to air at more nearly normal temperatures,[3] and a 4-year period of natural aging.[4]

2. FOLDING ENDURANCE

For determination of the folding endurance of film, the Bureau uses a folding tester of the Pfund type [5] shown in figure 1. It consists essentially of a fixed jaw *A*, and a movable jaw *B*, which are opened and closed by the screws *C*. In operation, a test specimen is clamped in place with the jaws at the maximum distance apart, and folded by moving jaw *B* up against jaw *A*, then back against plate *D*. The folds are caused to form alternately on opposite sides of the jaws by directing the film with a blunt-pointed instrument. The testing length is controlled by the position of plate *D* which regulates the maximum distance between the jaws. The number of folds sustained by the film before breaking is recorded as the folding endurance. The instrument used tests specimens 15 mm wide, and these strips should be cut approximately 50 mm long. The 15 mm width is taken from the center of the film by trimming off the edges.

Although operated manually, this tester gave more uniform results than were obtained with the testers commonly used for paper. Difficulty was encountered in the use of the latter because slight distortion of the film on aging caused edge cracks when folded over the jaw edges. The Pfund instrument folds without tension, forming a small loop not in contact with any metal edge. Table 1 shows some results obtained by different operators over a period of several months on the same brand of film. Each value is an average for 10 strips of film. The testing length was 19 mm.

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[3] R. H. Rasch, *Accelerated aging test for paper*, BS J. Research **7**, 465 (1931) RP352.

[4] R. H. Rasch, and B. W. Scribner, *Comparison of natural aging of paper with accelerated aging*, BS J. Research **11**, 727 (1933) RP620.

[5] This instrument was developed by A. H. Pfund, Johns Hopkins University, for the Du Pont Film Manufacturing Corporation.

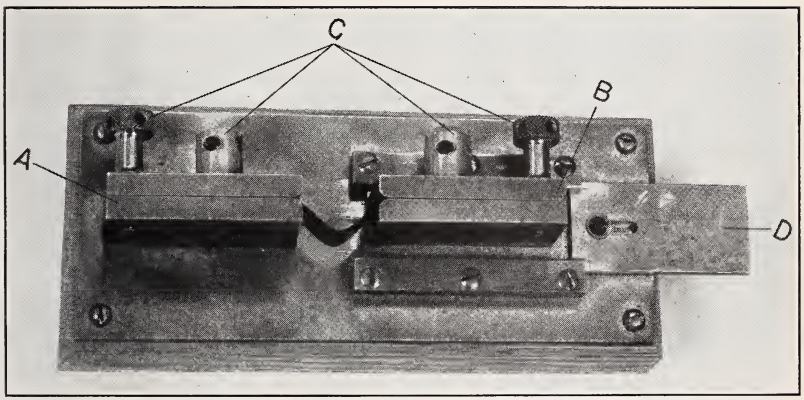

FIGURE 1.—*Pfund type of folding endurance tester used for determining the flexibility of motion-picture films.*

TABLE 1.—*Test data on cellulose acetate film, showing reproducibility of results with Pfund tester*

Series	Number of single folds (average of 10 strips of film)		
	Operator *A*	Operator *B*	Operator *C*
1	17	18	17
2	19	18	20
3	18	18	21
Average	18	18	19
Standard deviation of individual values	1.71	1.68	2.05

3. pH VALUE

The pH value of film was determined in the following manner. A 1-g sample of film, including both base and emulsion, is transferred to an Erlenmeyer flask and 100 ml of acetone, containing 10 percent by volume of water, is added. After complete dispersion of the film base, the pH of the solution is determined by means of a glass electrode. A convenient apparatus for this purpose has been described by Wallace [6] for the measurement of the pH of leather extracts. The water and acetone should be purified by distillation, and the combined solvent should have a pH of 7 ± 0.4. Duplicate determinations for the film should agree within 0.1 pH unit.

4. COPPER NUMBER

For use in the determination of copper number, the film is made into a thin solution by dissolving 1 g of the film base in 20 g of acetone. The strips of gelatin remaining are removed by decantation. Approximately 45 ml of the acetone solution is poured in a fine stream into a liter of water, heated to approximately 85° C. The water is stirred vigorously during the addition to precipitate the cellulose acetate in a convenient form. The precipitated cellulose acetate is filtered and washed five or six times with ether to remove water and plasticizer. After drying 1 hour at 65° C, samples of the precipitated acetate are weighed for the copper-number determination. The determination is made exactly as prescribed for paper in the standard methods of the Technical Association of the Pulp and Paper Industry.[7]

The test is based upon the quantitative reduction of an alkaline copper solution by the degraded celluloses in the sample. The copper number is the weight in grams of copper reduced from the cupric to the cuprous state, by 100 g of the sample.

5. RELATIVE VISCOSITY

A solution, consisting of approximately 1 percent of cellulose ester dissolved in acetone, is used in determining the relative viscosity. This solution is made from a sample of film (base and emulsion) weighing 1.000 g. The sample is cut into pieces small enough to drop into a 100-ml flask, and the flask is partially filled with acetone. After solution of the film base is complete and the emulsion has settled to

[6] J. Research NBS 15, 5 (1935) RP805.
[7] Tech. Assn. Pulp and Paper Ind. T430m (1936). Copies can be secured from the Association, 122 East 42d St., New York, N. Y., for 25 cents each.

the bottom, the flask is immersed in a bath maintained at $30 \pm 0.02°$ C until temperature equilibrium is reached, the solution diluted to 100 ml with acetone, and 5 ml is then transferred to an Ostwald pipet which is also immersed in the constant-temperature bath. The time of flow of the solution through the capillary of the pipet is measured with a stop watch which can be read to at least one-fifth second. The time of flow is also measured for the pure solvent. Not less than three or four determinations are made for each solution, and the values obtained should agree within two or three tenths of a second. The relative viscosity is then calculated as the ratio of the time of flow of the solution to the time of flow of the solvent.

6. CONDITIONING

Since cellulose esters and gelatin are hygroscopic substances, the physical properties of the film will be affected by the relative humidity of the surrounding atmosphere. Consequently, in order to obtain as uniform results as possible, all samples of film are conditioned 24 hours at 65 percent relative humidity and 70° F before testing them, and all weighings and physical tests are made under those conditions, which are standard for paper and textiles.

In the determination of folding endurance, the strips of film reserved for control tests are kept in an atmosphere with relative humidity below 65 percent until the heated strips are ready for test, then all strips are conditioned and tested together. When this procedure is followed, both control and heated strips approach equilibrium from the same direction, and hence have comparable moisture contents.

7. RESIDUAL HYPO

The method used for detecting the presence of sodium thiosulphate in films is that recommended by Crabtree and Ross.[8] This test consists in placing a strip of film in a solution of mercuric chloride in a test tube or glass cylinder and observing any turbidity which develops in the solution. If sodium thiosulphate is present it reduces mercuric ion, and an insoluble mercurous compound is formed which causes the turbidity. If no thiosu.phate or other reducing agent is present, the solution remains clear although the silver image is bleached white. The test solution contains 25 g of mercuric chloride, 25 g of potassium bromide, and water to make 1 liter. A 1.5-inch length of 35-mm motion-picture film, cut into small pieces and immersed in 10 ml of solution, was found to be a satisfactory amount for this test. The solution and film are allowed to stand 15 minutes, with occasional stirring, in order to give sufficient time for complete reaction.

8. CELLULOSE NITRATE

Traces of cellulose nitrate, the presence of which is undesirable, can be detected by means of a diphenylamine indicator. The indicator is a solution of diphenylamine which may be easily prepared by mixing 20 ml of concentrated sulphuric acid, 10 ml of glacial acetic acid, and 0.3 g of diphenylamine. A few drops of the indicator on the uncoated surface of the film results in a blue coloration if any nitrate is present.

[8] J. Soc. Motion Picture Engrs. **14**, 419 (1930).

III. PROPERTIES OF FILM FOR PERMANENT RECORDS

Table 2 contains some data obtained on cellulose acetate films from three different sources. These films are considered to have excellent stability.

TABLE 2.—*Properties of cellulose acetate film*

Sample	Folding endurance		Acidity		Copper number		Relative viscosity at 30° C	
	Original	Loss on aging [2]	pH value of original	Change after aging [2]	g of Cu per 100 g of Cellulose acetate original	Change after aging [2]	Original	Change after aging [2]
	Single folds [1]	*Percent*						*Percent*
1	17.4	18	6.2	−0.3	2.77	+0.07	2.65_5	−1.1
2	20.7	18	5.6	−.1	3.95	+.03	2.65_7	−2.1
3	17.7	21	6.5	+.2	2.72	−.01	2.78_3	−1.3

[1] Average values for 20 strips.
[2] Aging period 72 hours.

From a consideration of these results, it appears that cellulose acetate film having the following properties will be suitable for records of permanent value.

1. A folding endurance of not less than 16 single folds, with not less than 75 percent retention of folding endurance after heating 72 hours at 100° C.
2. A pH value of not less than 5.5, with a decrease of not more than 0.5 after heating 72 hours at 100° C.
3. An increase in copper number after heating 72 hours at 100° C of not more than 0.2.
4. A decrease in relative viscosity after heating 72 hours at 100° C of not more than 3.0 percent.
5. Developed film shall be free from sodium thiosulphate (hypo) as indicated by the mercuric chloride test, and the base should be free from cellulose nitrate as indicated by the diphenylamine test.

WASHINGTON, May 11, 1937.

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