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# INTERNATIONAL STANDARD



# 4331

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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## Photography — Processed photographic film for archival records — Silver-gelatin type on cellulose ester base — Specifications

*Photographie — Film photographique pour la conservation d'archives — Type gélatino-argentique sur support en ester de cellulose — Spécifications*

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# Photography — Processed photographic film for archival records — Silver-gelatin type on cellulose ester base — Specifications

## 0 INTRODUCTION

Since 1930, great advances have been made in the use of photographic films for the preservation of records. The preservation of film records by governments, banks, insurance companies, industry and other enterprises has been stimulated by a recognition of the economies in storage space, organization, accessibility, and ease of reproduction that result from the use of film records.

During the early development period of the art of copying documents, 35 mm nitrate motion-picture film was sometimes used. This material is highly flammable and deteriorates rapidly under unfavourable storage conditions. Nitrate film is not acceptable for any archival record use. The manufacture of nitrate film declined after World War II and was discontinued in most countries during the 1950s.

For many years, the only films suitable for archival storage were made on some type of cellulose ester base. Various synthetic polymer film bases have since appeared. The useful life of cellulose ester type material extends back only to about 1908. Experience with the film during that time and the results of accelerated ageing tests and other studies predict, however, that the material is capable of enduring as long as rag paper under normal storage conditions.

This International Standard is intended to eliminate possible hazards to permanence attributable to the chemical or physical characteristics of the processed film. Some of those characteristics are the responsibility of the film manufacturer, some of the film processor, and some are influenced by both. However, specifying the chemical and physical characteristics of the material does not, by itself, ensure satisfactory archival behaviour. It is essential to provide proper storage temperature and humidity and protection from the hazards of fire, water, fungus and certain atmospheric pollutants.

## 1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard establishes the specifications for safety cellulose ester type films having silver-gelatin type emulsions processed to produce a stable black-and-white photographic image.

1.2 Silver images produced by full reversal processing are included. Silver images that have been chemically altered by treatments such as toning, reduction, or intensification are excluded. Colour films are excluded. However, a gold protection treatment is not excluded. Silver images produced by dry processing, diffusion transfer, monobath processing and halide reversal processing are also excluded.

1.3 This International Standard is applicable to microfilm, motion-picture film roll film, and sheet film intended for archival record films.

1.4 Lacquers and lubricants have been applied to processed film for fungus and scratch protection without any known detrimental effect on permanence. However, because of the wide variety of possible materials, lacquered and lubricated film are excluded from this International Standard.

## 2 REFERENCES

ISO 417, *Photography — Determination of thiosulphate and other residual chemicals in processed photographic films, plates and papers — Methylene blue photometric method and silver sulphide densitometric method.*<sup>1)</sup>

ISO 543, *Cinematography — Motion-picture safety film — Definition, testing and marking.*

ISO 5466, *Photography — Practice for the storage of processed safety photographic film.*<sup>2)</sup>

## 3 DEFINITIONS

For the purposes of this International Standard, the following definitions apply.

3.1 **archival record film**: A photographic film composed and treated so that under optimum storage conditions it is suitable for the preservation of records having permanent value.

(See ISO 5466.)

1) At present at the stage of draft. (Revision of ISO/R 417.)

2) In preparation.

5.1.2 Films greater than 0,18 mm in thickness

Unheated processed film and processed film after accelerated ageing, as specified in 7.2, shall not break or crack when tested for flexibility as specified in 7.8.

5.2 Mushiness loss

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 ageing of the processed film. The mushiness value shall be determined as specified in 7.9 and the accelerated ageing shall be determined as specified in 7.2.

TABLE 2 – Limits for mushiness value loss after accelerated ageing

Characteristic	Original mushiness value	
	≤ 0,5 N	> 0,5 N
Maximum permissible mushiness value loss*	50 %	25 %

\* The increase in the limits with decrease in original mushiness level is necessary because the test is not sufficiently accurate to distinguish small differences.

5.3 Blocking

Processed film shall show no evidence of blocking (sticking), delamination, or surface damage when tested as specified in 7.10. 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 Image stability

The film image shall show no degradation which would impair the film for its intended use, when tested as specified in 7.11.

6 THIOSULPHATE CONCENTRATION

This characteristic is determined by the processing conditions. (See annex A.)

Films may be fixed in solutions containing either sodium thiosulphate or ammonium thiosulphate.<sup>[3]</sup> After processing, the film shall not contain a greater concentration of residual thiosulphate calculated as thiosulphate ions, S<sub>2</sub>O<sub>3</sub><sup>2-</sup>, than that specified in table 3, when determined as specified in ISO 417.<sup>[9]</sup>

TABLE 3 – Limits for thiosulphate concentration

Classification of films	Maximum permissible concentration of thiosulphate ion, S <sub>2</sub> O <sub>3</sub> <sup>2-</sup> , µg/cm <sup>2</sup>
Fine-grain copying, duplicating, and printing films	0,7
Medium-grain continuous-tone camera films and coarse-grain X-ray films	2

The analysis for thiosulphate shall be made on a film sample from a clear area and shall be made within 2 weeks after processing. (See annex B.) 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 a photographic layer on both sides of the base, or films having a photographic layer on one side and a non-curl backing layer on the reverse side, are permitted the specified concentration of residual thiosulphate 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 film should be examined under a comparison microscope at 20 X 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 fine grain, with the lower limit of thiosulphate concentration of table 3 applying.

7 TEST METHODS

7.1 Identification of film base

All emulsion and backing layers shall be removed from a specimen of the unknown film either by scraping or by the use of an enzyme solution. All sublayers shall then be removed by scraping. Base material shall then be prepared by scuffing the surface with a suitable tool to produce a very fine powder. This powder shall be mixed in a mortar with about 100 times its mass of potassium bromide previously ground to about 200 mesh. A strip or pellet shall be prepared as described in the literature.<sup>[1]</sup> An infra-red absorption curve shall be obtained from this pellet by means of an infra-red absorption spectrophotometer. The identity of the unknown base can be established<sup>1)</sup> by comparison with curves for known polymers.<sup>[13]</sup>

1) It is difficult, although not impossible, to distinguish between cellulose acetate, cellulose acetate propionate, and cellulose acetate butyrate base by this method, but such separation is not necessary for the purpose of this International Standard.

the film or extend to film perforations. The film 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°. Removal by the tape of any portion of the surface layer on any of the specimens 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 specified in the tape-stripping test. The tape shall be peeled back rapidly from the film surface at an angle of approximately 180°. The peel-back force required to separate the tape from the film shall be measured by a strain gauge or maximum reading spring scale. A bonding force of at least 9 N per centimetre of tape width is required.

## 7.6 Humidity cycling adhesion test

### 7.6.1 Specimen preparation

A specimen 5 cm square or 5 cm by the film width is convenient, but the dimensions are not critical. Two specimens of processed film shall be selected from a high silver density area.

### 7.6.2 Procedure

The specimens shall be mounted in a rack and placed in a glass laboratory desiccator jar so that they are freely exposed to the atmosphere. The jar shall be placed in a forced air circulating oven for 8 h at  $50 \pm 2^\circ\text{C}$ . The atmosphere within the jar shall be maintained at  $96 \pm 2\%$  relative humidity, which can be obtained by keeping a saturated solution of potassium sulphate in water<sup>[1]</sup> in the bottom of the jar. After the 8 h time period, the rack shall be placed for 16 h in a second desiccator which is also in the same oven. The atmosphere within this second jar shall be maintained at  $11 \pm 2\%$  relative humidity, which can be obtained by keeping a saturated solution of lithium chloride in water<sup>[1]</sup> in the bottom of the jar. The precautions specified in 7.2 shall be taken to ensure that the proper humidity is obtained.

A time period of 8 h at the high humidity and 16 h at the low humidity shall constitute one cycle.<sup>1)</sup> The film shall be subjected to 12 humidity cycles, after which it shall be removed from the rack and the emulsion and any backing layer examined for peeling, flaking, skidding or cracking.<sup>2)</sup>

The film shall be examined under the same magnification and lighting as normal for product use. During a weekend interruption, the film shall be kept at  $50 \pm 2^\circ\text{C}$  and  $11 \pm 2\%$  relative humidity.

1) This can be most easily done by placing the samples in the high humidity jar in the morning and in the low humidity jar in the evening.

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

## 7.7 Flexibility test for films 0,18 mm and less in thickness

### 7.7.1 Specimen preparation

Processed film in 16 mm form may be tested in this width. Films in other sizes shall be cut 15 to 16 mm wide and at least 12 cm long, using a sharp tool which does not nick the edges. Ten specimens are required for the unheated film and ten specimens for the heated film. The specimens to be heated and the control specimens shall be cut alternately and continuously from a single piece of film.

### 7.7.2 Accelerated ageing

Ten specimens shall be subjected to accelerated ageing as specified in 7.2.

### 7.7.3 Conditioning

Specimens shall be conditioned as specified in 7.5.2.

### 7.7.4 Procedure

The film shall not be removed from the conditioning atmosphere for testing. Folding endurance shall be determined using the MIT folding tester as described in the literature<sup>[1]</sup>. The unheated and heated specimens shall be tested alternately. The averages shall be calculated separately for the unheated and heated film.

## 7.8 Flexibility test for films greater than 0,18 mm in thickness

### 7.8.1 Specimen preparation

Specimens shall be prepared as specified in 7.7.1 except that only four specimens are required for the unheated and four for the heated film.

### 7.8.2 Accelerated ageing

Four specimens shall be subjected to accelerated ageing as specified in 7.2.

### 7.8.3 Conditioning

Specimens shall be conditioned as specified in 7.5.2.

### 7.8.4 Procedure

The film shall not be removed from the conditioning atmosphere for testing. The test apparatus shall be a 3 mm diameter mandrel made of polished metal, rigidly mounted on a stand. The film shall be slowly bent 180° around the mandrel, after which it shall be visually examined for cracks or breaks. Two specimens shall be tested with the film bent emulsion side out, and two with emulsion side in.

## ANNEX A

## FILM PROCESSING

The preservation of silver-gelatin type photographic film is dependent on the products which are still present after washing. The maximum content of residual thiosulphate is specified in clause 6 and discussed in annex B. Additional precautions required for optimum processing of archival records are described in this annex.

The fixing bath shall have been used as little as possible. As an indication of its suitability, it is recommended that the amount of silver be less than 0,5 % (*m/m*) in the case of a bath containing crystallized thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) or less than 0,8 % (*m/m*) in the case of a bath containing anhydrous ammonium thiosulphate [ $(\text{NH}_4)_2\text{S}_2\text{O}_3$ ]. In order to minimize defects in microfilms known as "ageing belemish microspots", the iodide content in the fixing bath for such products shall be between 0,1 and 0,5 g/l (expressed as KI).

Water used for washing shall be colourless and free from substances in suspension. Drinking water is generally pure enough for washing purposes. A washing water temperature maintained at a level between 15 and 25 °C is generally suitable and minimizes the danger of swelling introduced by higher temperatures and the decrease in the rate of solubility of the salts being removed at lower temperatures. Some films can tolerate higher temperatures. Fifteen minutes washing in well-agitated running water at a temperature of 20 °C is generally satisfactory, provided that the films are not in contact with one another and that water is renewed at their surface. For most automatic machines (droplet jets, etc.), the washing time is shorter and the water temperature is higher (35 °C).

Ion-exchange washing accelerators may be used. For instance, films may be soaked in a 20 g/l anhydrous sodium sulphite bath before washing, but thiosulphate-destructive washing accelerators such as oxidizing agents should not be used.

## iTeh STANDARD PREVIEW

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ANNEX B

## EFFECT OF RESIDUAL CHEMICALS ON THE DEVELOPED SILVER IMAGE

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During ageing of the developed photographic film, particularly in a humid atmosphere, the chemical reaction between residual thiosulphate<sup>1)</sup> and the grains of metallic silver causes discoloration and fading of the image. This effect increases in seriousness as the state of subdivision of the metallic silver in the image (referred to as "grain size") becomes smaller. It would be logical to specify the permissible residual thiosulphate as a continuous function of grain size. However, an exact measure of the grain size range in a film is not necessary for the purpose of this specification.

Excessive thiosulphate retained by the film also produces a coloured degradation in the low density areas of the film when subject to ageing in a humid atmosphere. In fact, this is the first place where this type of degradation is noted. The test for residual thiosulphate is specified to be made on non-image or clear areas of the film because it is in these areas that staining is most objectionable and that the thiosulphate concentration will not change appreciably over a period of 2 weeks.

The density or colour change of the image caused by residual thiosulphate is greatly accelerated at high temperature and humidity, allowing potential image degradation to be approximated in a matter of days. The limits for residual thiosulphate permitted in this specification have been selected on the basis of a thiosulphate concentration sufficiently low to cause no practical impairment in the image or clear areas when the film is subjected to a moist incubation test (see 7.11). Although any accelerated test involves some uncertainty, the validity of this test has been verified by results obtained after normal ageing conditions. The amount of thiosulphate retained in a processed film is dependent to a considerable degree on the composition of the fixing bath. Acid hardening fixing baths (which contain aluminium salts) condition the gelatin in the emulsion so that thiosulphate ion is retained more tenaciously than in a non-hardening bath. However, a non-hardening fixing bath should be used with caution because the gelatin may become soft. Ammonia or washing aids which contain oxidizing agents (such as hypochlorite and peroxides) should not be used.

Residual silver compounds and complexes are an important cause of image layer degradation in aged processed photographic films. The silver of the complex ion can combine with sulphur produced by the decomposition of the thiosulphate or can react with atmospheric contaminants to produce discoloration. High concentration of silver complexes in the fixing bath, oxidants in the wash water, and film drying conditions that provide an unclean atmosphere or impart a case-hardening effect on the emulsion are considered undesirable factors in the preparation of archival film.

1) The term thiosulphate is used generically to include its decomposition products, the polythionates and their silver complexes.

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