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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 black-and-white film for archival records — Silver-gelatin type on cellulose ester base — Specifications

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

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# Photography — Processed photographic black-and-white 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 in 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 these 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. Archival record films shall be stored under the conditions prescribed in ISO 5466.

## 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** It is applicable to silver images produced by negative and full reversal processing<sup>1)</sup>, positive working films and gold toning. It does not, however, apply to other silver images that have been chemically altered by treatments such as toning, reduction, or intensification.

This International Standard does not apply to colour films, films fixed in other than thiosulfate solutions<sup>[3]</sup>, and silver images produced by dry or thermal processing, diffusion transfer and halide reversal processing.

**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 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 film is not within the scope of 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.*

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

ISO 5466, *Photography — Processed safety photographic film — Storage practices.*

ISO 5626, *Paper — Determination of folding endurance.*

ISO 7830, *Photography — Safety photographic films other than motion-picture films — Material specifications.*

1) Full reversal processing includes a final fix and wash after the second development step.

Table 1 — Limits for flexibility and flexibility loss

Characteristic	Total film thickness	
	under 0,13 mm	0,13 to 0,18 mm
1 Unheated film minimum permissible average number of double MIT folds	20	10
2 Film after accelerated ageing maximum permissible average loss in folds	25 %	35 %

## 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 carried out 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 Properties controlled by the processor

These characteristics are determined by the processing conditions (see annex A).

### 6.1 Thiosulfate concentration

The thiosulfate concentration in processed films depends on processing conditions. Washing aids containing oxidizing agents such as peroxides or hypochlorites shall not be used. Processed film shall not contain a greater concentration of residual thiosulfate calculated as thiosulfate ions,  $S_2O_3^{2-}$ , than that specified in table 3, when determined as specified in ISO 417<sup>[9]</sup>.

Table 3 — Limits for thiosulfate concentrations

Classification of films	Maximum permissible concentration of thiosulfate ion $S_2O_3^{2-}$ g/m <sup>2</sup>
Fine-grain copying, duplicating and printing films	0,007
Medium-grain continuous-tone camera films and coarse-grain X-ray films	0,02

The analysis for thiosulfate 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 properties 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 thiosulfate on 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 X 20 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 thiosulfate concentration of table 3 applying.

### 6.2 Residual silver compounds

The area treated with sodium sulfide shall show no more than a barely perceptible tint when tested in accordance with 7.12.

## 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 sub-layers 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

specimens are required for the emulsion surface and four specimens for the backing layer, if present.

### 7.5.2 Conditioning

Specimens shall be conditioned at  $23 \pm 2$  °C and  $50 \pm 2$  % relative humidity for at least 15 h. This may be accomplished by means of an air-conditioned room or a conditioning air cabinet fitted with sealed arm access ports. The specimens 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/s.

### 7.5.3 Procedure

The film specimens shall not be removed from the conditioning atmosphere for testing. A strip of pressure-sensitive plastic base adhesive tape about 15 cm long shall be applied to the surface of the processed film. The tape shall be pressed down with thumb pressure to ensure adequate contact, leaving enough tape at one end to grasp. No portion of the tape shall extend to the edges of 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  $50 \pm 2$  °C. The atmosphere within the jar shall be maintained at 96 % relative humidity, which can be obtained by keeping a saturated solution of potassium sulfate in water<sup>[10]</sup> in the bottom of the jar.<sup>1)</sup> After the 8 h time period, the rack shall be placed for 16 h in a second desiccator jar which is also in the same oven. The atmosphere within this second jar shall be maintained at 11 % relative humidity, which can be obtained by keeping a saturated solution of lithium chloride in water<sup>[10]</sup> in the bottom of the jar.<sup>1)</sup> 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>2)</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, cracking, or differential emulsion-base shrinkage.<sup>3)</sup>

The film shall be examined under the same magnification and lighting as normal for product use. During any interruption in the cycling procedure, the film shall be kept at  $50 \pm 2$  °C and 11 % relative humidity.

## 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 contiguously 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 ISO 5626. The unheated and heated specimens shall be tested alternately. The averages shall be calculated separately for the unheated and heated film.

- 1) The relative humidity is based on the nominal vapour pressure of the salt solution but the relative humidity tolerances cannot be specified.
- 2) This can be most easily accomplished by placing the samples in the high humidity jar in the morning and in the low humidity jar in the evening.
- 3) 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 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 false interpretation of an adhesion weakness.

## Annex A

### Film processing

(This annex does not form part of the standard.)

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

To ensure that the fixing bath shall be suitable, it is recommended that the amount of silver be less than 0,5 % (*m/m*) in the case of a bath containing crystallized sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) or less than 0,8 % (*m/m*) in the case of a bath containing anhydrous ammonium thiosulfate [ $(\text{NH}_4)_2\text{S}_2\text{O}_3$ ]. In order to minimize defects in microfilms known as "ageing blemish 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 strikes a balance between 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. The wash water temperature should be within 3 °C of the developer temperature.

Ion-exchange washing accelerators may be used. For instance, films may be soaked in a bath containing 20 g of anhydrous sodium sulfite per litre before washing.

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## Annex C

### Microscopic blemishes

(This annex does not form part of the standard.)

Some processed negative microfilms in storage from 2 to 20 years have developed microscopically small coloured spots or blemishes. These spots were first discovered in 1961. The fogged leader at the outside of the roll is most frequently affected by the blemishes, which are generally red or yellow in colour and are smaller in size than the image characters (for example, a type-written cipher reduced 20 times) on the microfilm. Occasionally, these spots are observed farther into the roll and appear in image areas. A more detailed description of the blemishes and of the techniques used in inspecting microfilm is given in the literature<sup>[7]</sup>.

The spots are believed to be caused by the oxidation of the image silver and the migration of the silver ion to localized areas. The colour may be due to colloidal silver<sup>[4][11]</sup>. Oxidizing agents which can cause image degradation are aerial oxygen, whose action on the film is strongly accelerated by moisture, and atmospheric contaminants, such as peroxides, ozone, sulfur dioxide, hydrogen sulfide, and nitrogen oxides, all occurring in industrial atmospheres. Peroxides may be present in some varieties of wood and may also be formed as the result of ageing of paper inserts, etc., and cardboard containers commonly used in storing film. The latter appear to be a likely source of oxidants associated with blemish formation.

The conditions under which microfilm is processed have been found to play a role in the development of the blemishes when the film has been improperly stored in a humid atmosphere. Good processing techniques are essential so that the film emerges from the process free of water and chemical spots, dirt, dust, scratches, and other blemishes. Washing procedures should provide uniform removal of the thiosulfate and the associated silver compounds. Drying should be uniform and extremes of temperature should be avoided. A small quantity of potassium iodide in the fixing bath, combined with proper storage conditions, has been found to provide some protection against these blemishes in microfilm<sup>[5]</sup>.

It must be emphasized that the conditions under which microfilm is stored play a vital role in the development of these defects. Storage in cool, dry air that is free of the oxidizing gases or vapours mentioned in paragraph two above is important. Storage in suitable metal or plastic cans is recommended rather than in paper or in cardboard cartons, which, with age, produce peroxides. (See ISO 5466.)

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