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# INTERNATIONAL STANDARD



# 4332

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## Photography — Processed photographic film for archival records — Silver-gelatin type on poly(ethylene terephthalate) base — Specifications

*Photographie — Film photographique pour la conservation d'archives — Type gélatino-argentique sur support en polyéthylène téréphthalate — Spécifications*

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## FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4332 was drawn up by Technical Committee ISO/TC 42, *Photography*, and was circulated to the Member Bodies in November 1975.

It has been approved by the Member Bodies of the following countries :

Australia	Italy	Spain
Austria	Japan	Turkey
Belgium	Mexico	United Kingdom
Canada	Poland	U.S.A.
France	Romania	U.S.S.R.
Germany	South Africa, Rep. of	Yugoslavia

No Member Body expressed disapproval of the document.

# Photography — Processed photographic film for archival records — Silver-gelatin type on poly(ethylene terephthalate) 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. An ISO specification for archival photographic film on this base has been drawn up (ISO 4331, *Photography — Processed photographic film for archival records — Silver-gelatin type on cellulose ester base — Specifications*). In 1956, a synthetic polymer film base belonging to the polyester class was introduced. This particular polyester is known chemically as poly(ethylene terephthalate). It has several advantages over cellulose ester base, including superior mechanical properties and dimensional stability. Practical experience with this polyester film is considerably shorter than that with cellulose ester film. However, experience to date and accelerated ageing tests indicate that this film support is just as stable, if not more so, than safety cellulose ester base. This International Standard for archival film on poly(ethylene terephthalate) base parallels ISO 4331 for cellulose ester base.

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.

## 1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard establishes the specifications for safety poly(ethylene terephthalate) 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 micro-film, 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 film, plates and paper — Methylene blue photometric method and silver sulphide densitometric method.*<sup>1)</sup>

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

ISO/R 1184, *Plastics — Determination of tensile properties of films.*

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.)

**3.2 film base :** The plastics support for the emulsion and backing layers.

**3.3 safety poly(ethylene terephthalate) base :** A film base composed mainly of a polymer of ethylene glycol and terephthalic acid.

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

**3.5 non-curl 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.)

**3.6 mushiness; wet penetration strength :** A term used to indicate the wet strength of emulsion and backing layers. It is the minimum force on a stylus required to penetrate the emulsion surface. A high mushiness value denotes a high wet strength.

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

2) In preparation.

3) Poly(ethylene terephthalate) base films are characterized by excellent dimensional stability. The humidity and thermal coefficients of expansion are approximately 1/3 to 1/2 those of a comparable thickness film on cellulose ester base. Size change due to room temperature ageing is generally less than 0,05 % after 3 years.

## 4 PHYSICAL REQUIREMENTS CONTROLLED BY THE FILM MANUFACTURER

### 4.1 Film base type

The base used for archival record film shall be of a safety poly(ethylene terephthalate) type and can be identified by the method specified in 7.1.3)

### 4.2 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 ageing of the processed film. The accelerated ageing shall be accomplished as specified in 7.2 and the viscosity determined as specified in 7.3.

### 4.3 Ignition time

The film shall meet the requirements for ignition time specified in ISO 543.

### 4.4 Burning time

The film shall meet the requirements for burning time specified in ISO 543.

### 4.5 Nitrate nitrogen content

The film shall not contain more than 0,15 % nitrate nitrogen, as determined by the method specified in ISO 543.

### 4.6 Free acidity and acidity increase

The film shall not have a free acidity greater than the equivalent of 1,0 ml of 0,1 N sodium hydroxide solution per gram of film when measured as specified in 7.4. The volume of 0,1 N sodium hydroxide solution equivalent to the free acidity of the processed film shall not increase by more than 0,5 ml over its original value after accelerated ageing as specified in 7.2.

### 4.7 Layer adhesion

#### 4.7.1 Tape-stripping adhesion

The processed film shall not show any removal of the emulsion layer or backing layer when tested as specified in 7.5.

#### 4.7.2 Humidity-cycling adhesion

The emulsion layer or backing layer of the processed film shall not show separation or cracking which would impair its intended use, when tested as specified in 7.6. (See annex D.)

## 5 PHYSICAL REQUIREMENTS CONTROLLED BY THE FILM MANUFACTURER AND BY THE PROCESSOR

### 5.1 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 specified in 7.7. The loss in tensile properties after accelerated ageing, as specified in 7.2, shall not exceed the percentage specified in line 2 of table 1.

TABLE 1 — Limits for tensile properties and tensile properties loss

Characteristic	Tensile strength at break	Elongation at break
1 <i>Unheated film</i> Minimum permissible tensile properties	138 MPa*	75 %
2 <i>Film after accelerated ageing</i> Maximum permissible loss in tensile properties	10 %	10 %

\* 1 MPa = 10<sup>6</sup> N/m<sup>2</sup>

### 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 7.8 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 7.9. 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 Image stability

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

## 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 ion, S<sub>2</sub>O<sub>3</sub><sup>2-</sup>, than that specified in table 3 when determined by the test method specified in ISO 417.<sup>[8]</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 on each side of the film.

Films are classified according to the 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 shall 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 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 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 by comparison with curves for known polymers.<sup>[11]</sup>

## 7.2 Accelerated ageing conditions

The test samples shall be mounted in a sample rack so that they are freely exposed to the surrounding air. The rack shall then be placed in a glass laboratory desiccator jar. The jar shall be heated in a forced air circulating oven for 72 h at  $100 \pm 2$  °C. The atmosphere within the jar shall be maintained at  $20 \pm 2$  % relative humidity. This relative humidity can be obtained by keeping a saturated solution of potassium acetate in water at the bottom of the jar. Care must be exercised to ensure that the saturated solution contains an excess of undissolved crystals at 100 °C. The undissolved crystals shall be completely covered by the saturated salt solution and the surface area of the solution should be as large as practical. The jar and the salt solution shall be maintained at 100 °C for at least 20 h 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. The samples must be 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.

In subsequent sub-clauses, specimens subjected to these accelerated ageing conditions are designated "heat specimens".

## 7.3 Relative viscosity test

Measurements shall be made on two unheated and two heated specimens of processed film having the same average silver density and a mass of 1,000 g each after conditioning as specified in 7.5.2. Each specimen shall be immersed in a separate 100 ml volumetric flask containing approximately 15 ml of a mixture of 60 % phenol and 40 % chlorobenzene by mass.<sup>1)</sup> The base may be dissolved by repeated shakings for 15 min in an oil bath at  $140 \pm 1$  °C.

The flasks shall be immersed in a water bath maintained at  $25 \pm 0,1$  °C until temperature equilibrium has been reached. Solvent shall be added to adjust the volume to 100 ml and the contents thoroughly mixed. Non-support layers shall be allowed to settle to the bottom of the flask. A portion of the liquid shall be transferred, with filtration<sup>2)</sup>, to an Ostwald pipette or an equally suitable capillary viscometer<sup>3)</sup> immersed in a constant-temperature bath at the same temperature. The pipette chosen shall have a flow time between 70 and 100 s for the solvent. The volume taken shall 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 0,2 s. The time of flow shall also be measured for the same volume of the pure solvent. Three readings shall be made for each portion. The relative viscosity is the ratio of the average flow time of the solution to that of the solvent. Duplicate determinations shall be made on both the unheated and heated samples and the averages shall be calculated separately.

## 7.4 Free acidity test

Measurements shall be made on two unheated and on two heated specimens of processed film having the same average silver density and a mass of approximately 1 g each.

Each specimen shall be accurately weighed and shall be immersed in approximately 15 ml of benzyl alcohol. The polyester support shall be dissolved by heating<sup>4)</sup> for 90 s at 200 °C, leaving the emulsion layer. The solution shall be cooled and diluted by the addition of 80 ml of chloroform and subsequently 80 ml of ethyl alcohol. Acidity shall be determined by direct titration of the mixture with 0,1 N sodium hydroxide solution, 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 volumes, in millilitres, of 0,1 N sodium hydroxide solution used in the two titrations, per gram of film. The averages shall be calculated separately for the unheated and heated film.

## 7.5 Tape-stripping adhesion test

### 7.5.1 Specimen preparation

Although the dimensions of the processed film specimen are not critical, one dimension shall be at least 15 cm. Four 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. The specimens shall be supported in such a way as to permit free circulation of air round the film and the linear air velocity shall be at least 15 cm/s.

1) Care shall be taken with the mixture of phenol and chlorobenzene to avoid inhalation of the vapour or contact with the skin or eyes. It may be rapidly absorbed through the skin and can cause severe burns. Solvent mixture should be used only with adequate ventilation and hot solutions should be used only in a hood.

2) Filtration must be rapid to avoid solvent loss. This may be accomplished by filtration through a porous glass wool pad.

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

4) Care shall be taken to avoid inhalation of the vapour or prolonged or repeated contact with the skin or eyes. Heating should take place in a hood.

### 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 the 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  $180^\circ$ . 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  $180^\circ$ . 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 for 8 h in a forced air circulating oven maintained 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>[9]</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>[9]</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 conditions as normal for the end use of the product. During a weekend interruption in the cycling procedure, the film shall be kept at  $50 \pm 2^\circ\text{C}$  and  $11 \pm 2\%$  relative humidity.

### 7.7 Tensile strength and elongation test

#### 7.7.1 Specimen preparation

Unperforated 16 mm film may be tested in this width. In the case of perforated 16 mm film, specimens shall be cut from between the perforations. Films in other sizes shall be cut 15 to 16 mm wide and at least 15 cm long, using a sharp tool which does not nick the edges. Five specimens are required for the unheated film and five specimens for heated film. The specimens to be heated and the control specimens shall be cut alternatively and continuously from a single piece of film. The thickness of each specimen shall be measured to the nearest 0,002 mm and the width to the nearest 0,1 mm.

#### 7.7.2 Accelerated ageing

Five 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 be removed from the conditioning atmosphere for testing. The tensile strength and percentage elongation at break of unheated and heated film specimens shall be tested alternatively as specified in ISO/R 1184. The tensile strength and elongation at break shall be calculated separately for the unheated and heated film.

### 7.8 Mushiness test

Measurements shall be made on two unheated and on two heated specimens of processed film.

The mushiness value shall be determined as described in the literature.<sup>[7]</sup> This involves the application of a gradually increasing load on a 0,38 mm radius spherical sapphire stylus as it travels against the emulsion or the backing surface. Processed film specimens shall be placed horizontally, with the tested surface up, in the tray of the apparatus containing water at  $21^\circ\text{C}$ . After 15 min total

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

2) Films may sometimes exhibit small pinholes in the image processing. These can be caused by dirt or dust particles 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.

immersion time, the sapphire stylus shall be placed on the surface under zero load. The stylus load shall be gradually increased. The mushiness value shall be taken as the stylus load (in newtons) at which the stylus makes the first rupture or ploughing of the surface. This rating may be reflected or transmitted light. Three measurements shall be made for each surface. The averages shall be calculated separately for the unheated and heated film.

### 7.9 Blocking test

At least five specimens of processed film shall be conditioned to  $60 \pm 2$  % relative humidity at  $40 \pm 2$  °C. A specimen size of 5 cm square is convenient where the size of the film permits, but the dimensions are not critical, provided that all specimens are of uniform size.

The specimens shall be placed in a glass laboratory desiccator jar so that they are freely exposed to the required conditioning atmosphere for at least 15 h. The jar shall be placed in a forced air circulating oven at  $40 \pm 2$  °C. A relative humidity of approximately 62 % can be obtained by keeping a saturated solution of sodium nitrate<sup>[2]</sup> in water at the bottom of the jar. The precautions specified 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 specimens from the jar, at least five specimens shall be stacked so that the emulsion surface of one film is against the back surface of the adjacent one. The stack shall be placed under a uniform pressure of 35 kPa. This may be accomplished by

placing a weight on the film stack, the dimensions of the weight being greater than those of the film specimens. The jar containing the weighed stack shall be put back into the forced air circulating oven for 3 days at 40 °C. 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 specimens shall then be individually removed from the stack and observed for evidence of film blocking (sticking).

### 7.10 Image stability test

Three specimens of processed film which contain similar and representative image areas shall be prepared. A specimen size of 5 cm square is convenient for sheet film products or 5 cm in length by the film width for roll film products.

These specimens shall be mounted so that they are freely exposed to the surrounding air and shall be incubated for 30 days at  $60 \pm 2$  °C and  $70 \pm 2$  % relative humidity. This may be accomplished by means of an air-conditioning cabinet. After the incubation period, the film shall be compared visually with the unincubated comparison specimen which has been stored at room temperature and at a relative humidity not exceeding 60 %. It is recommended that the support and emulsion surfaces be observed from different angles of incidence in the manner in which transparencies are viewed.

## 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 sodium 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 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 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, film 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.

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