
International Standard



4332

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

Photographie — Film photographique noir et blanc traité pour la conservation d'archives — Type gélatino-argentique sur support en polyéthylène téréphtalate — Spécifications

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4332 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 4332-1977), of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Photography — Processed photographic black-and-white 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 in 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 (see ISO 4331). 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. 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 poly(ethylene terephthalate) 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 processing¹⁾, 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^[3], 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 1184, *Plastics — Determination of tensile properties of films.*

ISO 4331, *Photography — Processed photographic black-and-white film for archival records — Silver-gelatin type on cellulose ester base — Specifications.*

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

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

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

3 Definitions

For the purpose 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.

3.2 film base : The plastic 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.

NOTE — 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.

4 Physical requirements controlled by the film manufacturer

4.1 Film base type

The base used for archival record film shall be of safety poly(ethylene terephthalate) type and can be identified by the method specified in 7.1.¹⁾

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 Safety requirements

The polyester film shall comply with the requirements of the safety film specified in ISO 543 and ISO 7830.

4.4 Free acidity and acidity increase

The film shall not have a free acidity greater than the equivalent of 1,0 ml of 0,1 mol/l sodium hydroxide solution per gram of film when measured as specified in 7.4. The volume of 0,1 mol/l 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.

4.5 Layer adhesion

4.5.1 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.

4.5.2 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 properties 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.

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.

1) Poly(ethylene terephthalate) base films are characterized by excellent dimensional stability. The humidity and thermal coefficients of expansion are approximately 30 to 50 % 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.

Table 1 — Limits for tensile properties and tensile properties loss

Characteristic	Tensile strength at break	Elongation at break
1 Unheated film minimum permissible tensile properties	138 MPa*	75 %
2 Film after accelerated ageing maximum permissible loss in tensile properties	10 %	10 %

* 1 MPa = 10⁶ N/m²

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

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 ion, $S_2O_3^{2-}$, than that specified in table 3, when determined by the test method specified in ISO 417⁽⁸⁾.

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.

Table 3 — Limits for thiosulfate concentration

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

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 as described in 7.11.

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 powder shall be mixed in a mortar with about 100 times its mass of potassium bromide previously ground to about 75 μ m. A strip or pellet shall be prepared as described in the literature⁽¹⁾. 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⁽¹¹⁾.

7.2 Accelerated ageing conditions^[6]

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 ± 2 °C. The atmosphere within the jar shall be maintained at 20 % relative humidity. This relative humidity can be obtained by keeping a saturated solution of potassium acetate in water at the bottom of the jar.¹⁾ Care shall 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 at 100 °C for at least 20 h prior to use to ensure adequate equilibrium.

Alternatively, exposure to these temperatures and humidity conditions may be provided by means of a conditioning air cabinet. The samples shall 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 "heated specimens". Comparison specimens kept at room conditions are designated "unheated specimens".

7.3 Relative viscosity test

Measurements shall be made on the bases of two unheated and two heated specimens of processed film that have the same average silver density. Samples of the processed film shall be heated as described in 7.2. The emulsion and any backing layers shall be subsequently removed by the use of a 5 % solution of sodium hypochlorite, by enzymes, or by scraping before proceeding with the relative viscosity determination.

The sample of base without coatings should weigh 1,00 g. Each sample shall be immersed in a separate 100 ml volumetric flask containing approximately 95 ml of a mixture of 60 % (m/m) phenol and 40 % (m/m) chlorobenzene.

CAUTION — 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.

The base may be dissolved by repeated shaking for 30 min in a 140 ± 1 °C oil bath.

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. A portion of the liquid shall be

transferred, by filtration²⁾ to an Ostwald pipette or an equally suitable capillary viscometer immersed in a constant-temperature bath at the same temperature.

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

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 for 90 s at 200 °C, leaving the emulsion layer.

CAUTION — 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.

The solution shall be cooled and diluted by the addition of 80 ml of chloroform and subsequently 80 ml of ethyl alcohol.

CAUTION — Care shall be taken to avoid inhalation of the chloroform.

Acidity shall be determined by direct titration of the mixture with 0,1 mol/l 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 mol/l sodium hydroxide solution used in the two titrations, per gram of film. The averages shall be calculated separately for the unheated film 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.

1) The relative humidity is based on the nominal vapour pressure of the salt solution but the relative humidity tolerances cannot be specified.

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

7.5.2 Conditioning

Specimens shall be conditioned at 23 ± 2 °C and 50 ± 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 ± 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^[9] in the bottom of the jar.¹⁾ 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^[9] in the bottom of the jar.¹⁾ 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.²⁾ 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.³⁾

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 ± 2 °C and 11 % 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 contiguously 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 not 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 1184. The tensile strength and elongation at break 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 96 % relative humidity jar in the morning and 11 % relative 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 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.

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^[7]. 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 °C. After 15 min total 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 made by 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 prior to testing to 60 % relative humidity at 40 ± 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 ± 2 °C. A relative humidity of approximately 62 % can be obtained within the jar by keeping a saturated solution of sodium nitrite^[2] 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 72 h 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 ± 2 °C and 70 ± 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.

7.11 Residual silver compound test

Two samples of processed film of any convenient size shall be tested on both sides. The film shall be blotted dry, if necessary, and a clean area shall be spotted with a freshly prepared 0,2 % sodium sulfide solution. The reagent shall be washed away or blotted up after 3 min.

If there is more than a barely perceptible tint, a reference sample shall be prepared. The reference sample should be of the same film type, preferably the same lot. It shall be fixed successively for 5 min in each of two duplicate fresh fixing baths containing 240 g of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) per litre and washed for 30 min in running water flowing at a rate that will fill the vessel once every 5 min. The film shall be air dried and tested with the sodium sulfide solution.

Any stain in the test sample greater than that of the reference sample is an indication of residual silver salts.

* 1 kPa = 10^3 N/m²

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 bluish 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 B

Effect of residual chemicals on the developed silver image

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

During ageing of the developed photographic film, particularly in a humid atmosphere, the chemical reaction between residual thiosulfate¹⁾ and the grains of metallic silver causes discolouration 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 thiosulfate 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 thiosulfate 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 thiosulfate 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 thiosulfate concentration will not change appreciably over a period of 2 weeks.

The density or colour change of the image caused by residual thiosulfate is greatly accelerated at high temperature and humidity, allowing potential image degradation to be approximated in a matter of days. The limits for residual thiosulfate permitted in this specification have been selected on the basis of a thiosulfate 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 7.10). 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 thiosulfate 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 thiosulfate 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.

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 sulfur produced by the decomposition of the thiosulfate or can react with atmospheric contaminants to produce discolouration. The sodium sulfide spot test specified in clause 6 provides a qualitative indication of the level of residual silver compounds. 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 thiosulfate is used generically to include its decomposition products, the polythionates and their silver complexes.

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^[6].

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^{[4][10]}. 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^[5].

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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Annex D

Emulsion adhesion

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

One property of film on poly(ethylene terephthalate) base which requires consideration is that of the adhesion of the photographic and backing layers to this base. For some films, this is not quite as good in all respects or under all conditions as is the case with cellulose ester-type bases. The extreme chemical inertness of this new film base makes the problem of adhesion much more difficult. Of importance is the effect of very low relative humidities which cause the gelatin photographic and backing layers to contract and impose severe stresses on the gelatin adhesion. Cycling relative humidities from moderate to very low cause alternate expansion and contraction of the gelatin layers which can be particularly severe on the adhesive bond. The magnitude of this stress on the bond is very dependent on the thickness and physical characteristics of the gelatin layers which differ with each type of film.

Under extreme conditions of elevated temperature and low or cycling relative humidities, gelatin photographic layers and backing layers on polyester base sometimes develop adhesion defects, such as slight edge peeling, flaking, emulsion cracking, etc., while similar layers on cellulose ester base under the same conditions do not exhibit these defects or do so to a lesser degree. For this reason, the relative humidity is extremely important for permanent record storage. Relative humidities between 30 and 50 % are recommended for optimum storage of archival records on this film base. It is essential that polyester base films for permanent records not only meet the requirements of this International Standard, but that they be stored under proper storage conditions. (See ISO 5466.)

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