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Imaging materials — Processed silver-gelatin-type black-and-white films — Specifications for stability

Matériaux pour l'image — Films noir et blanc de type gélatino-argentique traités — Spécifications relatives à la stabilité

Reference number
ISO 18901:2010(E)

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18901 was prepared by Technical Committee ISO/TC 42, *Photography.*

This second edition cancels and replaces the first edition (ISO 18901:2002), which has been technically revised.

This International Standard is one of a series of International Standards dealing with the physical properties and stability of imaging materials. To facilitate identification of these International Standards, they are assigned a number within the block from 18900 to 18999.

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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 is not a safety film as specified in ISO 18906. Nitrate film is not acceptable for any record film. The manufacture of nitrate film declined after World War II and was discontinued in most countries in the 1950s.

From about 1908 to 1956, the only safety-type film bases in commercial use were cellulose acetate, cellulose acetate propionate and cellulose acetate butyrate. The useful life of these cellulose-ester-type bases is somewhat conjectural because of limited practical experience. However, the results of laboratory incubation tests indicate a useful life of at least 50 to 100 years when cellulose-ester-base films are stored under recommended conditions (see References [1], [2], [3] and [4]).

A second type of polymer safety film base was introduced commercially in 1956. This is a polyester-class material whose chemical name is polyethylene terephthalate.

NOTE Another type of polyester base, known as polyethylene naphthalate, has been used for APS (Advanced Photo System) type films since 1996.

Polyester base has several advantages over cellulose-ester base, including greater strength, stiffness, tear resistance and dimensional stability, which are important in many photographic applications (see References [5] and [6]). Accelerated ageing tests supplemented by 35 years of practical experience indicate a potential useful life of 500 years.

This International Standard provides image-stability predictions for three classes of black-and-white films in terms of LE (life expectancy) ratings. These three classes are radiographic films, microfilms and all other films. Two or three LE ratings are given for each of these film classes depending on their residual thiosulfate concentrations.

Studies on the stability of silver-gelatin-type films have investigated the effect of residual hypo on the image permanence of radiographic films, microfilms and aerial films (see References [7], [8] and [9]). This work suggested modifications to the residual hypo limits and a more quantitative image-stability test was included in the first edition of this International Standard. Residual hypo limits and image-stability tests are now included for all film categories.

This International Standard identifies certain hazards to permanence attributable to the chemical or physical characteristics of processed film and provides methods for evaluating them. Some of these are inherent film characteristics, some are related to the chemical processing procedure and some are influenced by both factors. However, storage conditions can also have a pronounced influence on film permanence. The essential requirements for longevity are proper storage temperature and humidity as well as protection from the hazards of fire, water, fungus, and atmospheric pollutants. Proper storage conditions are specified in ISO 18902 and ISO 18911.

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Imaging materials — Processed silver-gelatin-type black-and-white films — Specifications for stability

1 Scope

This International Standard establishes the specifications for photographic films intended for the storage of records.

It is applicable specifically to films with a base of safety cellulose ester or polyester having silver-gelatin emulsions, processed to produce a black-and-white silver image by negative or full-reversal processing. It applies to film processed using a monobath that includes thiosulfate as the fixing agent, followed by a conventional wash. It also is applicable to silver films given a stabilizing treatment by partial or full conversion to silver sulfide, silver selenide or gold.

This International Standard is applicable to films having ultrasonic or dielectric (induction-heated) splices. It does not cover films with splices made of adhesive tape or solvent-type splices.

NOTE Solvent-type splices are suspect since they sometimes retain traces of residual solvents containing peroxide, which can pose some risk of oxidative attack on the silver image.

This International Standard is not applicable to films with chromogenic black-and-white images, colour images of any type, or to films with a magnetic recording track. It does not apply to films with silver images produced by dry or thermal processing or by diffusion-reversal processing, or to films that have been processed by a monobath using means other than a thiosulfate-type fixing solution. It is not applicable to films where the silver salts are removed by means other than thiosulfate solutions (see Reference [10]).

This International Standard is not applicable to films to which lacquers have been applied.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5-2, *Photography and graphic technology — Density measurements — Part 2: Geometric conditions for transmittance density*

ISO 5-3, *Photography and graphic technology — Density measurements — Part 3: Spectral conditions*

ISO 527-3, *Plastics — Determination of tensile properties — Part 3: Test conditions for films and sheets*

ISO 18902, *Imaging materials — Processed imaging materials — Albums, framing and storage materials*

ISO 18906, *Imaging materials — Photographic films — Specifications for safety film*

ISO 18911, *Imaging materials — Processed safety photographic films — Storage practices*

ISO 18917, *Photography — Determination of residual thiosulfate and other related chemicals in processed photographic materials — Methods using iodine-amylose, methylene blue and silver sulfide*

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3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
emulsion layer
image or image-recording layer of silver-gelatin-type black-and-white films

NOTE There can be one or several emulsion layers.

3.2
extended-term storage conditions
storage conditions suitable for the preservation of recorded information having permanent value

NOTE The term “medium-term storage conditions” defines storage conditions suitable for the preservation of recorded information for a minimum of 10 years.

3.3
film base
plastic support for the emulsion and backing layers

3.3.1
cellulose-ester base
base for recording materials composed mainly of the cellulose esters of acetic, propionic, or butyric acids, or mixtures thereof

3.3.2
polyester base
base for recording materials composed mainly of a polymer of ethylene glycol and terephthalic acid (also referred to as polyethylene terephthalate), or a polymer of ethylene glycol and 2,6 naphthalene dicarboxylic acid (also referred to as polyethylene naphthalate)

3.4
full-reversal processing
reversal photographic processing that consists of development, bleach, clear, re-exposing and second development, followed by fixing and washing

3.5
life expectancy
LE
length of time that information is predicted to be acceptable in a system after dark storage at 21 °C and 50 % relative humidity (RH)

NOTE In the past, the term “archival” was used to define material that could be expected to preserve images forever, so that such images could be retrieved without significant loss when properly stored. However, as no such material exists, this is now a deprecated term and is no longer used in International Standards for imaging materials or in systems specifications.

3.6
LE designation
rating for the **life expectancy** (3.5) of recording materials and associated retrieval systems

NOTE The number following the LE symbol is a prediction of the minimum life expectancy, in years, during which information can be retrieved without significant loss when stored at 21 °C and 50 % RH, e.g. LE-100 indicates that information can be retrieved after at least 100 years of storage.

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3.7
non-curl backing layer
layer, usually made of gelatin, applied to the side of the photographic film base opposite that of the emulsion layer, for the purpose of preventing curl

NOTE 1 It is comparable to the emulsion layer in thickness and is not removed in processing.

NOTE 2 Antihalation or other layers removed in processing are excluded from this definition.

3.8
safety film
safety photographic film
film that meets the flammability specifications defined in ISO 18906

4 Film base requirements

The base used for record films, as specified in this International Standard, shall be of a safety polyester or a cellulose-ester type and can be identified by the method described in 8.1.

Some films on a cellulose-ester-type base can have a maximum LE rating of 100. Some films on a polyester base can have a maximum LE rating of 500.

NOTE These limitations are based on historical experiences as discussed in the Introduction.

5 Processed film requirements

5.1 Storage conditions

Films shall be stored under the conditions specified in ISO 18902 and ISO 18911.

5.2 Safety film

Film shall meet the requirements specified in ISO 18906.

5.3 Amount of free acid

The cellulose-ester base shall not have an amount of free acid greater than 0,1 ml of 0,1 mol/l sodium hydroxide solution per gram of film. The amount of free acid shall be measured in accordance with 8.3.

NOTE The degradation of cellulose-ester base is autocatalytic and proceeds rapidly when the free acid is greater than 0,5 ml of 0,1 mol/l sodium hydroxide solution.

The volume of 0,1 mol/l sodium hydroxide equivalent to the amount of free acid of the processed film shall not increase by more than 0,5 ml/l over its original value after the accelerated ageing described in 8.2.

5.4 Tensile properties and loss in tensile properties

Film specimens shall be processed and dried under the conditions used for film records.

Processed films shall be tested for tensile properties as described in 8.4 and shall have a tensile stress and elongation at break as specified in Table 1 (unheated film). The loss in tensile properties after the accelerated ageing test described in 8.2 shall not exceed the percentage specified in Table 1 (heated film).

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Table 1 — Limits for tensile properties and loss in tensile properties on ageing

Film type		Tensile stress at break	Elongation at break
Unheated film: minimum permissible tensile properties	Cellulose-ester base	80 MPa[a]	15 %
	Polyester base	140 MPa	75 %
Heated film: maximum permissible loss in tensile properties compared with unheated film	Cellulose-ester base	15 %	30 %
	Polyester base	15 %	30 %

[a] 1 MPa = 10^6 N/m^2.

6 Requirements for the emulsion and backing layers of processed film

6.1 Layer adhesion

6.1.1 Tape-stripping adhesion

Processed film shall not show any removal of emulsion layer or backing layer when tested as described in 8.5.

6.1.2 Humidity-cycling adhesion

The emulsion layer or backing layer of processed film shall not show separation or cracking that can impair its intended use when tested as described in 8.6.

6.2 Emulsion flow

Processed film shall not show any visual evidence of emulsion flow (caused by partial emulsion remelting) as a result of the accelerated ageing of the processed film. Emulsion flow shall be determined as described in 8.7, when the accelerated ageing is performed as described in 8.2.

6.3 Blocking

Processed film shall show no evidence of blocking (sticking), delamination or surface damage when tested as described in 8.8. A slight sticking of the film specimens that does not result in physical damage or a change in the gloss of the surface shall be acceptable.

6.4 Thiosulfate concentration

Films shall be fixed in solutions containing either sodium thiosulfate (hypo) or ammonium thiosulfate (see Reference [10]). Hypo-eliminating agents containing oxidizing agents such as peroxides or hypochlorites shall not be used.

NOTE 1 Hypo-eliminating agents contain chemicals, usually strong oxidizing agents, that decompose thiosulfate (see Annex B). These are distinguished from hypo-clearing baths, which are high ionic strength salt solutions. These facilitate the washing of thiosulfate from the film, but do not chemically alter the thiosulfate.

After processing, the concentration of thiosulfite in the film shall be determined by one of the test methods described in ISO 18917. The film shall not contain a greater concentration of residual thiosulfate, calculated as thiosulfate ions ($S_2O_3^{2-}$), than that specified in Table 2.

NOTE 2 Three methods for measuring residual-thiosulfate-based chemicals in film are described in ISO 18917. All three methods are considered to be sufficiently reliable to report thiosulfate concentrations at the level of 0,014 g/m^2 of $S_2O_3^{2-}$. The methylene blue method is considered to be reliable for thiosulfate concentrations of 0,007 g/m^2. The methylene blue and iodine amylose methods measure thiosulfate ions only. They are run within two weeks of processing.

The silver sulfide densitometric test method measures polythionate decomposition products and other residual chemicals in addition to thiosulfate. The method can be run more than two weeks after processing. To determine thiosulfate levels accurately with this method, a calibration curve for the particular film is necessary.

The analysis for thiosulfate shall be made on a film specimen from a clear area and shall be made within two weeks after processing (see Annex B). The test method does not measure any change in the specimen between the time of processing and the time of analysis, but is used to judge the state of the film after the test has been carried out.

6.5 Residual silver compounds

Processed film shall not show more than an increase of 0,02 in Status A blue density when tested in accordance with 8.9 (see Annex C).

Table 2 — Limits for thiosulfate concentration

Film type	**Film classification** (LE designation)	**Maximum permissible concentration of thiosulfate**[b] g/m^{2}[c]
Radiographic films	LE-10	0,100
	LE-100	0,050
	LE-500[a]	0,020
Microfilms	LE-100	0,030
	LE-500[a]	0,014
Other films	LE-10	0,100
	LE-100	0,050
	LE-500[a]	0,014

[a] LE-500 film only applies to polyester-base film.

[b] Values are for each side of the film that has a photographic layer or a non-curl backing layer.

Very low concentrations of thiosulfate due to excessive washing may cause the silver image to be more susceptible to oxidative attack. These concentrations may be below the detection limits of ISO 18917.

[c] 0,010 g/m^2 = 1 µg/cm^2.

7 Image-stability requirements

7.1 General

The specifications and test methods for image stability vary for different product types.

Visual diffuse density in accordance with ISO 5 or Status A blue density shall be measured on a densitometer which has geometric conformance to ISO 5-2 and spectral conformance to ISO 5-3. Processed film specimens shall be incubated as described in 8.10.3.

7.2 Radiographic films

An area of unexposed processed film shall be tested. The Status A blue density change of the unexposed area shall be no greater than 0,05 density units after incubation. This requirement shall apply to LE-10, LE-100 and LE-500 films.

7.3 Microfilms

An area of minimum density and another area having a visual diffuse density of 1,2 ± 0,1 on the processed film specimen shall be tested.

Neither the minimum-density area nor the high-density area shall change by more than ± 0,1 visual diffuse density units after incubation.

7.4 Other films

A minimum-density area and a 1,0 ± 0,1 Status A blue density patch of processed film shall be tested. Neither the minimum-density area nor the high-density patch shall change by more than ± 0,1 Status A blue density units after incubation. This requirement shall apply to LE-10, LE-100 and LE-500 films.

8 Test methods

8.1 Identification of film base

Remove all emulsion and backing layers from a specimen of unknown film, either by scraping or by the use of enzyme solution. Then remove all sublayers by scraping.

Prepare a specimen of the base material by scuffing the surface with a suitable tool, such as a razor blade. The general procedure is to move the scuffing device back and forth over the specimen manually while exerting a very slight pressure. This removes the top layer of the base as a very fine dust. Carefully brush this into a mortar.

Mix the specimen with about 100 times its mass of potassium bromide, previously ground to about 75 µm. Prepare a strip or pellet as described in Reference [11].

Obtain an infrared (IR) absorption curve from the prepared pellet by means of an infrared-absorption spectrometer. By comparing the IR absorption curve for the unknown with curves for known polymers, the identity of the unknown can be established (see Reference [12]).

NOTE It is difficult, although not impossible, to distinguish between cellulose acetate, cellulose acetate propionate and cellulose acetate butyrate bases using this method, but such separation is not necessary for the purposes of this International Standard.

8.2 Accelerated-ageing conditions

Processed film shall be subjected to accelerated-ageing conditions to meet the requirements for an increase in the amount of free acid, loss in tensile properties and emulsion flow.

Test specimens shall be conditioned at (23 ± 1) °C and (50 ± 2) % RH for at least 15 h. After conditioning, place the specimens in a moisture-proof envelope and heat-seal the envelope. To prevent sticking between adjacent specimens, it may be necessary to interleave them with polytetrafluoroethylene or uncoated polyester. Ensure a high ratio of film-to-air volume by squeezing out excess air prior to heat-sealing. Use a separate envelope for each film specimen. Double bagging is recommended to reduce any pinhole effect in the envelopes. Heat the envelopes in an oven for 72 h at (100 ± 2) °C.

NOTE 1 A suitable moisture-proof envelope is a metal-foil bag that is coated on the inside with polyethylene for heat-sealing.

NOTE 2 Incubation is accomplished in a closed environment to prevent escape of any acid that might be produced during incubation. Such acid might catalyse further film-base degradation.

An alternative method of incubating the specimens in a closed environment is by placing them in 25 mm borosilicate-glass tubes (see Reference [13]). Each tube shall have two flanged sections separated by a

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gasket to provide a moisture seal and shall be held together by a metal clamp. Sufficient film specimens shall be used to provide a high ratio of film-to-air volume.

NOTE 3 A suitable inert gasket can be made from polytetrafluoroethylene.

NOTE 4 In the text, specimens subjected to these accelerated-ageing conditions are designated “heated film”. Comparison specimens kept at room conditions are designated “unheated film”.

8.3 Determination of the amount of free acid

8.3.1 Specimen preparation

Cut each specimen, including the emulsion layer and all coatings, into small pieces and accurately weigh the specimen prior to dissolving it in the appropriate solvents.

Measurements shall be made on two unheated and two heated specimens of imaged film that weigh approximately 1 g to 2 g each. Weigh the specimens to the nearest 0,01 g. Heat the films in accordance with 8.2.

8.3.2 Solution preparation

Immerse the film specimens in 100 ml of either distilled or deionized water. Allow the film to soak in the water for 24 h at 38 °C with continuous moderate stirring. Filter the solution to remove film particles. It is important to remove emulsion particles because gelatin is amphoteric and can distort the results. Cool the solution to room temperature.

8.3.3 Titration

Titrate the solution with 0,1 mol/l sodium hydroxide using metacresol purple as an indicator. Also, carry out a blank titration on the same distilled or deionized water used in 8.3.2. For titration of specimens whose acidity level is equivalent to 1 ml of 0,1 mol/l NaOH or less, it is more accurate and convenient to use a microburette with graduations of 0,01 ml. For specimens with higher acidity levels, a conventional burette can be employed.

8.3.4 Calculation

The amount of free acid, A, expressed in equivalent millilitres of 0,1 mol/l sodium hydroxide per gram of film base, is calculated as follows for each specimen:

$$A = \frac{(V_\mathrm{S} - V_\mathrm{B})c_\mathrm{t}}{0{,}1\,m} \qquad (1)$$

where

V_S is the volume, in millilitres, of titrant used for the specimen;

V_B is the volume, in millilitres, of titrant used for the blank;

c_t is the concentration, in moles per litre, of the titrant;

m is the mass, in grams, of the specimen.

Carry out the titration in duplicate on separately prepared solutions. The average amount of free acid for the unheated and heated film specimens shall be calculated and reported separately.

8.4 Tensile property test for processed film

8.4.1 Specimen preparation

Processed film already in 16 mm format may be tested in this width. In the case of perforated 16 mm film, specimens shall be cut from the area between the perforations. Film in other sizes shall be cut into sections 15 mm to 16 mm wide and at least 150 mm long using a sharp tool that does not nick the edges of the specimen.

Five specimens are required for unheated film and five specimens for heated film. The specimens to be heated and control specimens shall be cut alternately and contiguously from a single piece of film.

The thickness of each specimen shall be measured with a suitable gauge to the nearest 0,002 mm and the width to the nearest 0,1 mm.

8.4.2 Accelerated ageing

Five specimens shall be subjected to accelerated ageing as described in 8.2.

8.4.3 Conditioning

All specimens, both unheated and heated, shall be conditioned at (23 ± 1) °C and at (50 ± 2) % RH for at least 15 h. This can be accomplished by means of an air-conditioned room or a conditioning-air cabinet.

Specimens shall be supported in such a way as to permit free circulation of air around the film. The linear air velocity shall be at least 150 mm/s.

8.4.4 Procedure

Film specimens shall not be removed from the conditioning atmosphere for testing.

The tensile stress and percentage elongation at break of unheated and heated film specimens shall be tested alternately by means of a tensile machine, as specified in ISO 527-3. The initial grip separation shall be 100 mm and the rate of grip separation shall be 50 mm/min.

The tensile stress and percentage elongation at break shall be calculated separately for unheated and heated film.

8.5 Tape-stripping adhesion test

8.5.1 Specimen preparation

Although the dimensions of the processed-film specimen are not critical, one dimension shall be at least 150 mm. Four specimens shall be used for the emulsion surface and four specimens for the backing layer, if present.

8.5.2 Conditioning

Specimens shall be conditioned as described in 8.4.3.

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8.5.3 Procedure

Film specimens shall not be removed from the conditioning atmosphere for testing.

Apply a strip of pressure-sensitive, plastic-base adhesive tape1) about 150 mm long to the surface of the processed film. Press the tape 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 specimen or extend to the film perforations.

Hold the specimen firmly on a flat surface and remove the tape rapidly 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 a 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 described in the tape-stripping test. The tape shall be rapidly peeled back from the film surface at an angle of approximately 180°.

The peelback force required to separate the tape from the film shall be measured by a suitable device, such as a strain gauge or spring scale capable of reading the maximum force used. A bonding force of at least 0,9 N/mm of tape width is required.

8.6 Humidity-cycling adhesion test

8.6.1 Specimen preparation

Two specimens of processed film shall be selected from an area of high-silver density. The specimens should be (50 × 50) mm, or 50 mm long multiplied by the film width, but the dimensions are not critical provided all specimens are of uniform size.

8.6.2 Procedure

Mount the test specimens in a specimen rack and place them in a glass laboratory desiccator jar in such a way that they are freely exposed to the required conditioning atmosphere. Place the jar in a forced-air circulating oven for 8 h at (50 ± 2) °C. The atmosphere within the jar shall be maintained at 96 % RH, which can be obtained by keeping a saturated solution of potassium sulfate in water (see Reference [14]) in the bottom of the jar.

NOTE 1 Relative humidity is based on the normal vapour pressure of the salt solution, but RH tolerances cannot be specified.

Take care to ensure that the saturated solution contains an excess of undissolved crystals at 50 °C. Some undissolved crystals shall be above the level of the saturated salt solution, and the surface area of the solution should be as large as is practical. To ensure adequate equilibrium, the jar and salt solution shall be kept at 50 °C for at least 20 h prior to use.

After 8 h, place the specimens and specimen rack for 16 h in a second desiccator jar that is also in the same oven. The atmosphere within the second jar shall be maintained at 11 % RH, which can be obtained by keeping a saturated solution of lithium chloride in water (see Reference [14]) in the bottom of the jar.

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1) An example of a suitable tape available commercially is 3M tape No. 610. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.

Alternatively, the temperature and humidity conditions can be achieved by means of conditioning-air cabinets or air-conditioned rooms.

Time periods of 8 h at high humidity and 16 h at low humidity constitute one cycle. Each film specimen shall be subjected to 12 humidity cycles. After this, remove the film specimens from the specimen rack and examine the emulsion and any backing layer for evidence of peeling, flaking or cracking produced as a result of the humidity-cycling treatment.

NOTE 2 This can be most easily accomplished by placing the specimens in the 96 % RH jar in the morning and in the 11 % RH jar in the evening.

NOTE 3 Films can sometimes exhibit what appear to be small pinholes in the image after processing. These can be caused by dirt or dust particles on the emulsion surface at the time the raw film is exposed and are not to be confused with holes or cracks in the emulsion layer. It is important to note the existence of such pinholes in the image prior to humidity cycling so that their presence does not lead to a false interpretation of adhesion weakness.

Film shall be examined under magnification and lighting conditions that are normal for the end use of the product. During an interruption in the cycling procedure, film specimens shall be kept at (50 ± 2) °C and 11 % RH.

8.7 Emulsion-flow test

Measurements shall be made on two heated specimens of processed film. Each specimen shall contain a photographic image of a grid pattern or of printed material.

Subject the film specimens to accelerated ageing as described in 8.2. After incubation, visually examine the photographic images on the heated specimens for any image change caused by flow of the emulsion. This visual examination can be facilitated by comparison with the images on unheated specimens.

8.8 Blocking test

At least five specimens of processed film shall be conditioned at (40 ± 2) °C and 62 % RH. The preferred specimen size is (50 × 50) mm, but the dimensions are not critical provided all specimens are of uniform size.

Place the specimens in a glass laboratory desiccator jar so that they are freely exposed to the required conditioning atmosphere for at least 15 h. Place the jar containing the specimens in a forced-air circulating oven at (40 ± 2) °C. A relative humidity of approximately 62 % can be obtained by keeping a saturated solution of sodium nitrite (see Reference [15]) in water at the bottom of the jar.

Take care to ensure that the saturated solution contains an excess of undissolved crystals at 40 °C. Some undissolved crystals shall be above the level of the saturated salt solution, and the surface area of the solution should be as large as is practical. To ensure adequate equilibrium, the jar and salt solution shall be kept at 40 °C for at least 20 h prior to use.

After moisture equilibrium is attained, remove the jar from the oven. Without removing the film specimens from the jar, stack at least five specimens so that the emulsion surface of one specimen is against the back surface of the adjacent specimen. Place the stack under a uniform pressure of 35 kPa. This can 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 weighted stack shall be put back into the forced-air circulating oven for 3 days at 40 °C.

Alternatively, the temperature and humidity conditions can be achieved by means of conditioning-air cabinets or air-conditioned rooms.

Remove the film stack from the oven and allow it to cool. Remove the film specimens individually from the stack and observe each one for evidence of film blocking (sticking). See 6.3.

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8.9 Residual silver compound test

Two specimens of processed film of any available size shall be tested on both sides. These specimens shall be taken from the end of the processing run. Blot the film dry, if necessary, and spot a clean image-free area with a freshly prepared 0,2 % aqueous solution of sodium sulfide. After 3 min, wash away or blot up the reagent.

If there is more than a barely perceptible tint, a reference specimen shall be prepared. The reference specimen shall be:

- of the same film type, preferably the same lot;
- fixed successively for 5 min in each of two duplicate fresh fixing baths containing 240 g/l of sodium thiosulfate ($Na_2S_2O_3{\cdot}5H_2O$);
- washed for 30 min in running water that is flowing at a rate that will fill the vessel once every 5 min.

Air-dry the film and test it with the sodium sulfide solution as stated in the previous paragraph. Any stain in the test specimen greater than that of the reference specimen is an indication of residual silver salts.

8.10 Image-stability test

8.10.1 Specimen preparation

Specimens of film shall be exposed to the specified densities as indicated in Table 3. These differ for different film types. An appropriate specimen size is (50 × 50) mm for sheet-film products, or 50 mm in length multiplied by the film width for roll-film products.

8.10.2 Conditioning

All specimens shall be conditioned at ambient temperature and 60 % RH for at least 8 h. This can be accomplished by means of an air-conditioning room or a conditioning-air cabinet.

Specimens shall be supported in the conditioning-air cabinet in such a way as to permit free circulation of air around the film. The linear air velocity shall be at least 150 mm/s.

Heat-seal the film in a moisture-proof metallic-foil envelope after the air has been squeezed out. Double bagging is recommended to reduce any pinhole effect in the bags.

8.10.3 Incubation

Film specimens shall be incubated at the temperatures and times specified in Table 3. A description of how these conditions were chosen is given in Annexes D and E.

8.10.4 Measurements

Both before and after incubation, microfilms shall be measured for visual diffuse density and all other film shall be measured for Status A blue density.

Densities shall be measured on a densitometer having geometric conformance to ISO 5-2 and spectral conformance to ISO 5-3. Specimens shall meet the density criteria given in 7.2 to 7.4.

Table 3 — Conditions for image-stability test

Film type	Specimen density	Film classification (LE designation)	Incubation conditions		
			Time days	Temperature °C	Humidity %
Radiographic films	Unexposed area	LE-10	14	68	60
		LE-100	14	77	60
		LE-500	14	85	60
Microfilms	Area of minimum density and D = 1,2 ± 0,1	LE-100	14	60	60
		LE-500	30	60	60
Other films	Unexposed area and D = 1,0 ± 0,1	LE-10	7	60	60
		LE-100	14	60	60
		LE-500	30	60	60

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Annex A
(informative)

Washing and the effect of residual thiosulfate on the developed silver image

The preservation of silver-gelatin-type photographic film is dependent on the products that are still present after washing. The maximum content of residual thiosulfate and residual silver compounds is specified in 6.4 and 6.5. Water used for washing should be colourless and free from substances in suspension. Drinking water is generally pure enough for washing purposes.

NOTE The term “residual thiosulfate” is used in this annex to include its decomposition products, polythionates, including those complexed with silver.

The temperature of the water used for washing should be maintained between 15 °C and 25 °C as this 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. Washing in well-agitated running water for 15 min 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 temperature of the water used for washing should be within 3 °C of the temperature of the developer.

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

Excessive thiosulfate ions retained by the film produce one or more of the following phenomena:

- yellow-brown discolouration in the low-density areas of the film;
- bleaching or loss of density in the image areas;
- increase in density of the image areas (see References [5] and [6]).

The test for residual thiosulfate ions is specified to be made on non-image or clear areas of the film because this is where the thiosulfate ion concentration, as measured by the methylene blue method, will not change appreciably over a period of two weeks.

Tests have shown that, even at fairly high levels of residual thiosulfate ions, adverse changes are quite gradual for incubations at 60 % RH. The limits of residual thiosulfate ions permitted in this International Standard are based on the incubations described in Annexes C, D and E and are sufficiently low to minimize changes under recommended storage conditions.

The amount of thiosulfate ions retained in a processed film is dependent to a considerable degree on the composition of the fixing bath (see References [16] and [17]). Acid-hardening fixing baths (which contain aluminum salts) condition the gelatin in the emulsion so that the thiosulfate ions are 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 hypo eliminators, which contain oxidizing agents (such as hypochlorite and peroxides), should not be used.

The degree of change that can be tolerated in films depends upon their intended use. For textual records, legibility can be the sole consideration. For picture records, even a slight amount of discolouration or bleaching, particularly if non-uniform, can be unacceptable. It is believed that the residual thiosulfate ion level specified provides a sufficient margin of safety for all levels of life expectancy under the storage conditions specified in ISO 18911.

Annex B
(informative)

Effect of residual silver compounds on the developed silver image

Residual silver compounds and complexes are an important cause of image layer degradation in aged photographic films. Silver ions from the complex ions can combine with the sulfur produced by decomposition of the thiosulfate ions or can react with atmospheric contaminants to produce discolouration.

At the present state of knowledge, it is not possible to specify a safe maximum for residual silver compounds in quantitative terms. However, it is believed that the quantity of residual silver compounds will be satisfactorily low if the film passes the test for residual silver compounds specified in 6.5. Thorough washing of the film to reduce the residual thiosulfate ion level also has the important effect of reducing the level of residual silver compounds.

To ensure that the fixing bath is suitable, it is recommended that the amount of silver be less than 0,5 % (mass fraction) in the case of a bath containing sodium thiosulfate ($Na_2S_2O_3$), or less than 0,8 % (mass fraction) in the case of a bath containing ammonium thiosulfate [$(NH_4)_2S_2O_3$]. In microfilms, in order to minimize defects known as “ageing blemish microspots”, the iodide content (as KI) in the fixing bath for such products shall be between 0,1 g/l and 0,5 g/l.

A high concentration of silver complexes in the fixing bath, oxidants in the water used for washing and film-drying conditions that provide an unclean atmosphere or impart a case-hardening effect on the emulsion are considered undesirable factors in the processing of film.

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Annex C
(informative)

Accelerated image-stability test for radiographic films

The experimental method used to derive the residual thiosulfate ion levels and film lifetimes is based on data that appear in Reference [7].

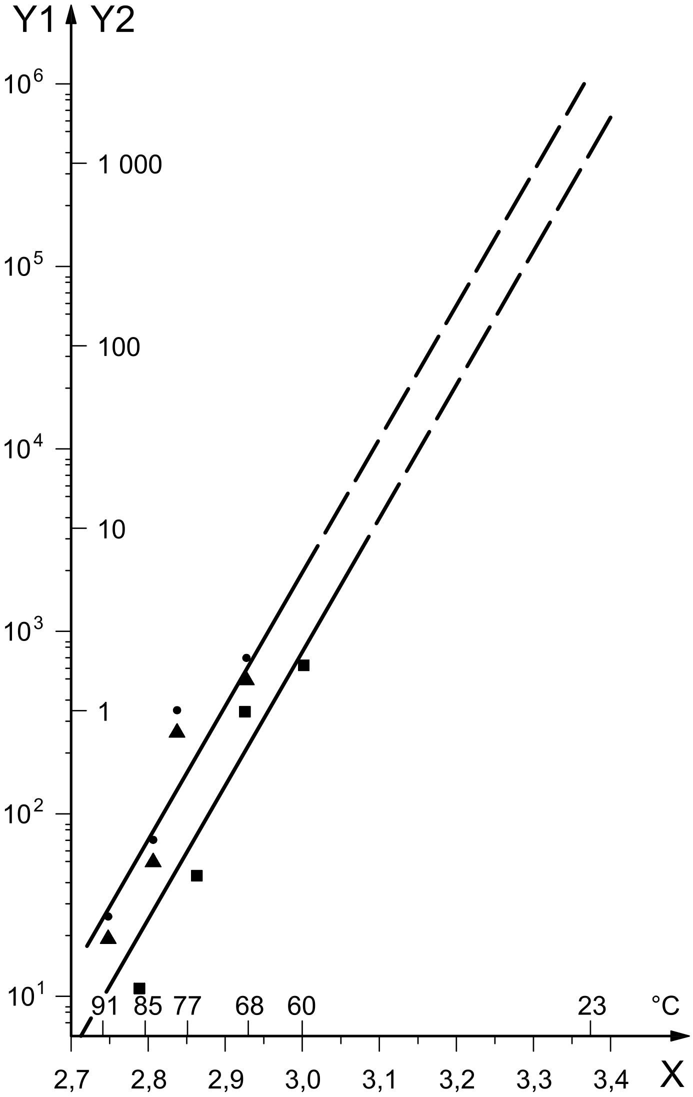
Eight radiographic films were exposed and processed in suitable processors by adding various amounts of fixer to the wash tanks of the processors. This non-recommended processing cycle yielded processed film specimens with a wide range of residual thiosulfate levels. The methylene blue method described in ISO 18917 was used to measure residual thiosulfate levels. The processed film specimens were incubated for a series of times in dark chambers at 60 % RH and temperatures from 93 °C to 52 °C. All specimens were hung freely in chambers where both temperature and humidity were controlled. Status A blue density measurements were made before and after the keeping periods for all specimens.

Status A blue density change in the area of minimum density was shown to be the sensitometric parameter most sensitive to increasing residual thiosulfate ion levels. Practical radiographs that had been incubated were examined by people with experience in X-ray diagnosis. They concluded that a yellow-brown stain increase of 0,05 Status A blue density units would be just noticeable to a diagnostician. For each film and for each temperature, the storage time to produce this sensitometric change was determined for each residual thiosulfate ion level.

The logarithm of the time required for stain build-up to occur for each incubation temperature was plotted against the reciprocal of the thermodynamic (absolute) temperature. Curves are plotted in Figure C.1 for experiments that showed density change.

Extrapolation down to a room temperature of 23 °C indicated the time required for stain build-up to occur for room-temperature storage at 60 % RH. The results of these extrapolations justify the limits specified in Table 2. The limits are conservative and are based on extrapolations of the experimental data. The limits are also conservative because the experiment was carried out at 60 % RH, which is higher than the maximum recommended storage RH for radiographic film.

The incubation criteria for the accelerated tests given in 8.10 were based on results of the eight radiographic films mentioned previously. The times were based on the least stable of these films and are conservative estimates.



Key

X reciprocal of thermodynamic temperature (1 000/*T*), in reciprocal kelvins

Y1 time for 0,05 density increase in area of minimum density, in days

Y2 time for 0,05 density increase in area of minimum density, in years

Residual thiosulfate ions: • 0,035 g/m^2; ▲ 0,11 g/m^2; ■ 0,30 g/m^2.

Figure C.1 — Time versus temperature for radiographic films

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Annex D
(informative)

Accelerated image-stability test for microfilms

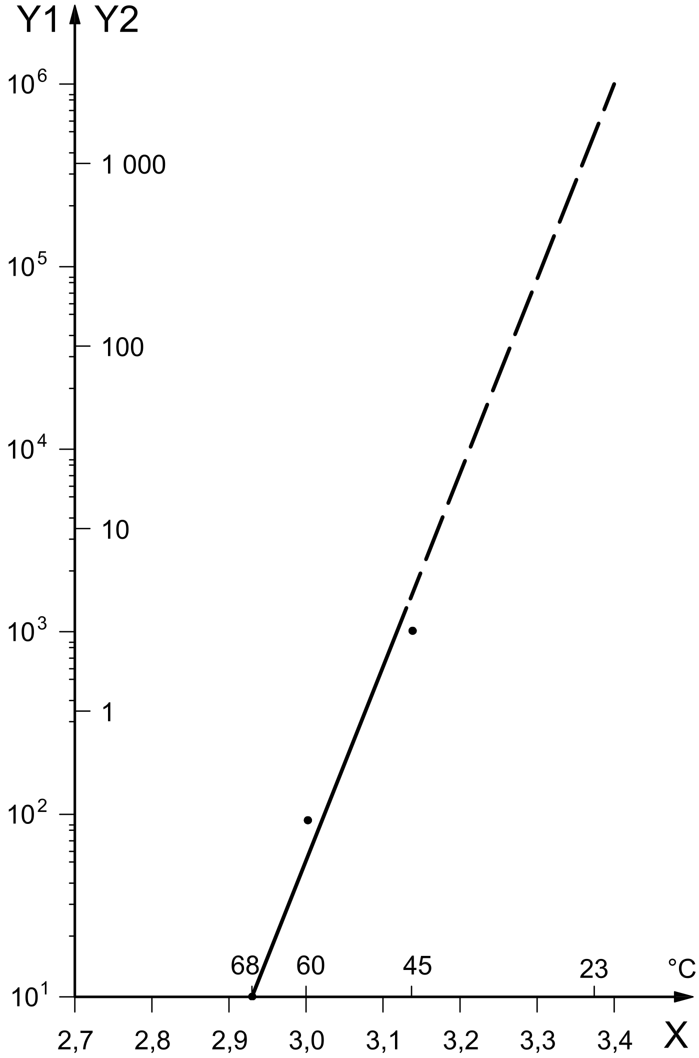
The experimental method used to derive the residual thiosulfate ion levels and film lifetimes is based on data that appear in Reference [8].

Ten microfilms were exposed and processed by adding seasoned fixes to a stagnant wash in the wash tank of the processor. This non-recommended processing cycle resulted in processed film specimens with a wide range of residual thiosulfate ion levels, with values from less than 0,003 g/m^2 to 0,15 g/m^2. The methylene blue method described in ISO 18917 was used to measure residual thiosulfate ion levels. The processed film specimens were incubated for a series of times in dark chambers at 60 % RH and temperatures from 32 °C to 77 °C. All specimens were hung freely in chambers where both temperature and humidity were controlled. Density measurements were made before and after the keeping periods for all specimens.

The density change criteria specified in 7.3 were used. A low-density patch and a 1,2 density patch should not change by more than 0,1 density units. (This level of change is greater than the measurement error.)

The logarithm of the time for a 0,1 density unit increase for each incubation temperature was plotted versus the reciprocal of the thermodynamic (absolute) temperature using the Arrhenius approach. Although the thiosulfate ion levels were as high as 0,15 g/m^2, only the film at the lowest thiosulfate ion level for one of the products changed enough to plot (see Figure D.1).

Extrapolation of high-temperature data down to a room temperature of 23 °C indicated that the time for a 0,1 density change at 60 % RH exceeded 1 500 years. Therefore, the limits specified in Table 2 are conservative. The limits are also conservative because the experiment was carried out at 60 % RH and this is higher than the maximum recommended storage RH for microfilm.



Key

X reciprocal of thermodynamic temperature (1 000/*T*), in reciprocal kelvins

Y1 time for 0,1 density increase in 1,0 density patch, in days

Y2 time for 0,1 density increase in 1,0 density patch, in years

Figure D.1 — Time versus temperature for microfilms

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Annex E
(informative)

Accelerated image-stability test for aerial films

The experimental method used for determining residual thiosulfate ion levels and film lifetimes was based on the study reported in Reference [9].

In three separate experiments, ten aerial films were exposed, developed and fixed, but were either washed in a stagnant water bath to which various amounts of seasoned fixer had been added, or immersed after normal washing in a solution of diluted seasoned fixer. These unorthodox treatments resulted in the retention of residual thiosulfate ions in the emulsion layers ranging from 0,003 g/m^2 to 0,28 g/m^2, as measured by the methylene blue method given in ISO 18917. Processed film specimens were conditioned at 60 % RH, then sealed in foil bags and incubated for different times at temperatures ranging from 32 °C to 77 °C. Density and resolving power measurements were made before and after incubation.

Resolving power values were detemined in accordance with ISO 6328, which involves a visual assessment of target images. None of these resolving power values changed by more than one element.

Changes in density were determined by Status A blue densitometry (see ISO 5-2 and ISO 5-3). The permissible change in blue density was set at 0,1 for both the high-density and minimum-density patches of the specimens. This amount of change is greater than the test system error. Investigation of the density changes as a function of residual thiosulfate level showed that the density change of the high-density patch was the most sensitive parameter.

The logarithm of the time for a 0,1 Status A blue density increase for the high-density patch for each incubation temperature was plotted versus the reciprocal of the thermodynamic (absolute) temperature using the Arrhenius approach. A specimen's curve is shown in Figure E.1.

NOTE The example in Figure E.1 represents a film containing approximately twice the level of thiosulfate ions permited in LE-10 aerial film.

The thiosulfate ion limits for LE-10, LE-100 and LE-500 films are conservative because the experiment was carried out at 60 % RH and this is higher than the maximum recommended RH for the extended-term storage conditions of aerial films.

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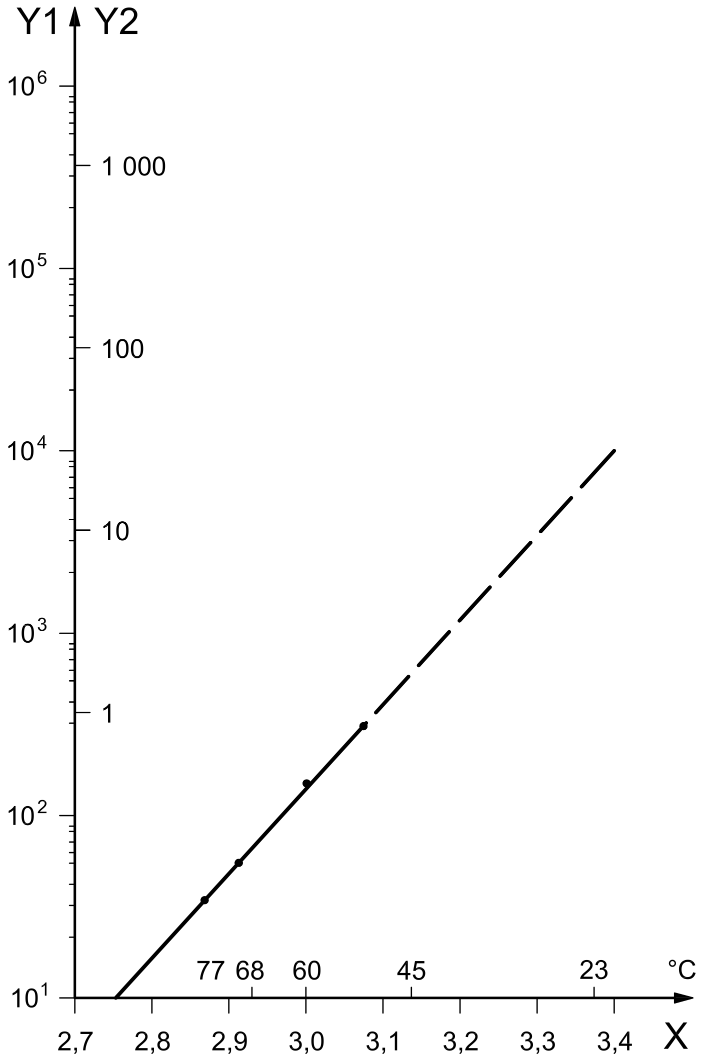

Key

X reciprocal of thermodynamic temperature (1 000/*T*), in reciprocal kelvins

Y1 time for 0,1 density increase in 1,0 density patch, in days

Y2 time for 0,1 density increase in 1,0 density patch, in years

Residual thiosulfate ions: • 0,18 g/m^2.

Figure E.1 — Time versus temperature for aerial films

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