INTERNATIONAL STANDARD

ISO 18901

First edition
2002-02-15

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

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

Reference number
ISO 18901:2002(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 International Standard 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 first edition cancels and replaces the second edition of ISO 10602, of which it constitutes a technical revision.

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

Annexes A to F of this International Standard are for information only.

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 100 years when cellulose-ester base films are stored under recommended conditions (see [1], [2], [3], [4] in the bibliography).

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. [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 [5], [6] in the bibliography). 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 [7], [8], [9] respectively in the bibliography). This work suggested modifications to the residual hypo limits and a more quantitative image-stability test was included in the first edition of ISO 10602. 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 gives methods of 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 also can 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.

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 by a monobath, which 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 by means of adhesive tape or solvent-type splices.

NOTE Solvent-type splices are suspect since they may 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, nor 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, nor to films that have been processed by a monobath using a 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 [10] in the bibliography).

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

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5-2:2001, *Photography — Density measurements — Part 2: Geometric conditions for transmission density*

ISO 5-3:1995, *Photography — Density measurements — Part 3: Spectral conditions*

ISO 527-3:1995, *Plastics — Determination of tensile properties — Part 3: Test conditions for films and sheets*

ISO 18902:2001, *Imaging materials — Processed photographic films, plates and papers — Filing enclosures and storage containers*

ISO 18906:2000, *Imaging materials — Photographic films — Specifications for safety film*

ISO 18911:2000, *Imaging materials — Processed safety photographic films — Storage practices*

ISO 18917:1999, *Photography — Determination of residual thiosulfate and other related chemicals in processed photographic materials — Methods using iodine-amylose, methylene blue and silver sulfide*

3 Terms and definitions

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

3.1
archival medium
recording material that can be expected to retain information for ever, so that such information can be retrieved without significant loss when properly stored

NOTE There is, however, no such material and it is not a term to be used in International Standards or system specifications.

3.2
emulsion layer(s)
image or image-forming layer(s) of photographic films, papers and plates

3.3
extended-term storage conditions
storage conditions suitable for the preservation of recorded information having permanent value

3.4
film base
plastic support for the emulsion and backing layers

3.4.1
cellulose-ester base
base for record materials composed mainly of the cellulose esters of acetic, propionic, or butyric acids, or mixtures thereof

3.4.2
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)

3.5
full-reversal processing
reversal photographic processing that consists of development, bleach, clear, reexposing and second development, followed by fixing and washing

3.6
life expectancy
LE
length of time that information is predicted to be acceptable in a system after dark storage at 21 °C and 50 % RH

3.7
LE designation
rating for the “life expectancy” of recording materials and associated retrieval systems

NOTE The number following the LE symbol is a prediction of the minimum life expectancy in years for 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.

3.8
medium-term storage conditions
storage conditions suitable for the preservation of recorded information for a minimum of 10 years

3.9
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.

3.10
safety photographic film
photographic film which passes the ignition-time test and the burning-time test 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 the equivalent of 0,1 ml of 0,1 mol/l sodium hydroxide solution per gram of film. The amount of free acidity 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.

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 accelerated ageing as described in 8.2 shall not exceed the percentage specified in Table 1 (heated film).

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 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 [10] in the bibliography). Hypo-eliminating agents containing oxidizing agents such as peroxides or hypochlorites shall not be used.

NOTE Hypo-eliminating agents contain chemicals, usually strong oxidizing agents, which decompose thiosulfate (see annex B). These are to be 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 film shall not contain a greater concentration of residual thiosulfate calculated as thiosulfate ions ($S_2O_3^{2-}$) than that specified in Table 2 when determined by one of the test methods described in ISO 18917.

NOTE 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 to be 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 may 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 keeping of the film following the time of the test.

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 [a]	Maximum permissible concentration of thiosulfate [b, c, d, e] g/m^2
Radiographic films	LE-10	0,100
	LE-100	0,050
	LE-500	0,020
Microfilms	LE-100	0,030
	LE-500	0,014
Other films	LE-10	0,100
	LE-100	0,050
	LE-500	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.

[c] The concentration of thiosulfate is expressed in grams per square metre, which conforms to SI units.

[d] 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.

[e] 0,010 g/m^2 = 1µg/cm^2

7 Image stability requirements

7.1 General

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

ISO visual diffuse density 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 [11] in the bibliography.

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 [12] in the bibliography).

NOTE It is difficult, although not impossible, to distinguish between cellulose acetate, cellulose-acetate propionate and cellulose-acetate butyrate bases by this method, but such separation is not necessary for the purpose 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 effect of pinholes 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.

