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Photography — Processed silver-gelatin type black-and-white film — Specifications for stability

Photographie — Film de type gélatino-argentique noir et blanc traité — Spécifications relatives à la stabilité

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

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.

International Standard ISO 10602 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 10602:1993), which has been technically revised.

Annex A forms an integral part of this International Standard. Annexes B, C, D, E, F and G 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 543. **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 1950's.

For many years the only safety films in commercial use were made on some type of cellulose ester base, such as cellulose acetate, cellulose acetate propionate or cellulose acetate butyrate. The useful life of safety cellulose ester-type films is somewhat conjectural, since actual experience with commercial material extends back only to about 1908. However, these materials show severe degradation when exposed to high temperatures and particularly to high humidities. Laboratory incubation studies predict a useful life of at least a century when stored under recommended conditions[1], [2], [3], [4].

A second type of polymer safety film base belonging to the polyester class, known chemically as poly(ethylene terephthalate), was introduced commercially in 1956. This material has a number of advantages over the cellulose ester base such as greater strength, stiffness, tear resistance, flexibility, dimensional stability and other characteristics, which make it superior for many photographic applications[5], [6]. Actual experience with polyester film is considerably less than with cellulose ester film although this material has been used for over 35 years.

Practical experience to date and accelerated ageing tests indicate that this film support is more stable than safety cellulose ester film base and is expected to have a useful life of 500 years[1], [4].

This second edition eliminates the film classifications of "archival", "long-term" and "medium-term" and replaces them with the LE (life expectancy) rating. In addition, residual hypo limits and image-stability tests are given for radiographic film, microfilm and all other films.

Studies on the stability of silver-gelatin-type films have investigated the effect of residual hypo on the image permanence of radiographic films[7], microfilms[8] and aerial films[9]. 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.

Former annex D, "Microsocopic spots and blemishes", and annex E, "Emulsion adhesion", have been eliminated; an annex F, "Accelerated image-stability test for aerial films", has been added.

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 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 keeping behaviour. It is essential to provide proper storage temperature and humidity, and protection from the hazards of fire, water, fungus and certain atmospheric pollutants. It is important that films be stored under the conditions specified in ISO 5466 and ISO 10214.

Photography — Processed silver-gelatin type black-and-white film — Specifications for stability

1 Scope

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

1.2 It applies specifically to films with a base of safety cellulose ester or polyester [poly(ethylene terephthalate)] 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 applies to silver films given a stabilizing treatment by partial or full conversion to silver sulfide, silver selenide or gold.

1.3 This International Standard does not apply to films with 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. It does not apply to films that have been processed by a monobath using 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[10].

1.4 This International Standard does not apply to films to which lacquers have been applied.

1.5 This International Standard applies 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.[1)]

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5-1:1984, *Photography — Density measurements — Part 1: Terms, symbols and notations.*

ISO 5-2:1991, *Photography — Density measurements — Part 2: Geometric conditions for transmission density.*

ISO 5-3:—[2)], *Photography — Density measurements — Part 3: Spectral conditions.*

ISO 417:1993, *Photography — Determination of residual thiosulfate and other related chemicals in processed photographic materials — Methods using iodine-amylose, methylene blue and silver sulfide.*

ISO 543:1990, *Photography — Photographic films — Specifications for safety film.*

ISO 1184:1983, *Plastics — Determination of tensile properties of films.*

ISO 5466:1992, *Photography — Processed safety photographic films — Storage practices.*

1) 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.

2) To be published. (Revision of ISO 5-3:1984)

ISO 10214:1991, *Photography — Processed photographic materials — Filing enclosures for storage.*

3 Definitions

For the purposes of this International Standard, the definitions given in ISO 5-1 and the following definitions apply.

3.1 archival medium: A recording material that can be expected to retain information forever so that it can be retrieved without significant loss when properly stored.

NOTE 1 There is, however, no such material and it is not a term to be used in International Standards or system specifications.

3.2 life expectancy (LE): The length of time that information is predicted to be retrievable in a system under extended-term storage conditions.

NOTE 2 However, the actual useful life of film is very dependent upon the existing storage conditions (see ISO 5466 and ISO 10214).

3.3 LE designation: A rating for the "life expectancy" of recording materials and associated retrieval systems. 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 under extended-term storage conditions.

NOTE 3 For example, LE-100 indicates that information can be retrieved for at least 100 years' storage.

3.4 extended-term storage conditions: Storage conditions suitable for the preservation of recorded information having permanent value.

3.5 medium-term storage conditions: Storage conditions suitable for the preservation of recorded information for a minimum of 10 years.

3.6 film base: The plastic support for the emulsion and backing layers.

3.7 emulsion layers(s): The image or image-forming layer(s) of photographic films, papers and plates.

3.8 noncurl 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 4 Antihalation or other layers removed in processing are excluded from this definition.

3.9 safety photographic film: Photographic film which passes the ignition time test and burning time test as specified in ISO 543.

3.10 safety cellulose-ester base: A film base composed mainly of the cellulose esters of acetic, propionic or butyric acids, or mixtures thereof.

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

3.12 full-reversal processing: Processing that includes development, bleach, clear, re-exposure and second development, followed by fixing and washing.

4 Safety and hazards

4.1 Hazard warnings

Some of the chemicals specified in the test procedures are caustic, toxic or otherwise hazardous. Safe laboratory practice for the handling of chemicals requires the use of safety glasses or goggles, rubber gloves and other protective apparel such as face masks or aprons where appropriate. Specific danger notices are given in the text and footnotes for particularly dangerous materials, but normal precautions are required during the performance of any chemical procedures at all times. The first time that a hazardous material is noted in the test procedure section, the hazard will be indicated by the word "DANGER" followed by a symbol consisting of angle brackets "< >" containing a letter which designates the specific hazard. A double bracket "<< >>" will be used for particularly perilous situations. In subsequent statements involving handling of these hazardous materials, only the hazard symbol consisting of the brackets and letter(s) will be displayed. Furthermore, for a given material, the hazard symbol will be used only once in a single paragraph.

Detailed warnings for handling chemicals and their diluted solutions are beyond the scope of this International Standard.

Employers shall provide training and health and safety information in conformance with legal requirements.

The hazard symbol system used in this International Standard is intended to provide information to the users and is not meant for compliance with any legal requirements for labelling as these vary from country to country.

It is strongly recommended that anyone using these chemicals obtain from the manufacturer pertinent information about the hazards, handling, use and disposal of these chemicals.

4.2 Hazard information code system

< B > Harmful if inhaled. Avoid breathing dust, vapour, mist or gas. Use only with adequate ventilation.

< C > Harmful if contact occurs. Avoid contact with eyes, skin or clothing. Wash thoroughly after handling.

< S > Harmful if swallowed. Wash thoroughly after handling. If swallowed, obtain medical attention immediately.

<< S >> May be fatal if swallowed. If swallowed, obtain medical attention immediately.

< F > Will burn. Keep away from heat, sparks and open flame. Use with adequate ventilation[3].

< O > Oxidizer. Contact with other material may cause fire. Do not store near combustible materials.

4.3 Safety precautions

All pipette operations shall be performed with a pipette bulb or plunger pipette.

Safety glasses shall be worn for all laboratory work.

5 Requirements for film bases

The base used for record films, as specified in this International Standard, shall be of a safety polyester [i.e. poly(ethylene terephthalate)] or a cellulose-ester type, and can be identified by the method described in 9.1.

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 5 These limitations are based on historical experiences as discussed in the introduction.

6 Requirements for processed film

Films shall be stored under the conditions specified in ISO 5466 and ISO 10214.

6.1 Safety film

The film shall meet the requirements specified in ISO 543.

6.2 Amount of free acid

Different specifications and test methods are given for polyester base and cellulose-ester base films. The polyester base shall not have an amount of free acid greater than the equivalent of 1,0 ml of 0,1 mol/l sodium hydroxide solution per gram of film, and the cellulose-ester base shall not have an amount of free acid greater than the equivalent of 0,5 ml of 0,1 mol/l sodium hydroxide solution per gram of film. The amount of free acidity shall be measured in accordance with 9.3.

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 9.2.

6.3 Tensile properties and loss in tensile properties

The film samples shall be processed and dried under the conditions used for the film records. Processed films shall be tested for tensile properties as described in 9.4 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 9.2 shall not exceed the percentage specified in table 1 (heated film).

3) The flammable warning symbol < F > will not be used for quantities of common solvents under 1 litre.

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[1)]	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 %

1) 1 MPa = 10^6 N/m^2

7 Requirements for the emulsion and backing layers of processed film

7.1 Layer adhesion

7.1.1 Tape-stripping adhesion

The processed film shall not show any removal of the emulsion layer or backing layer when tested as described in 9.5.

7.1.2 Humidity-cycling adhesion

The emulsion layer or backing layer of the processed film shall not show separation or cracking that can impair its intended use, when tested as described in 9.6.

7.2 Emulsion flow

The 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 9.7 when the accelerated ageing is performed as described in 9.2.

7.3 Blocking

Processed film shall show no evidence of blocking (sticking), delamination or surface damage when tested as described in 9.8. A slight sticking of the film samples that does not result in physical damage or a change in the gloss of the surface shall be acceptable.

7.4 Thiosulfate concentration

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

NOTE 6 Hypo-eliminating agent contains chemicals, usually strong oxidizing agents, which decompose thiosulfate (see annexes B and D). 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 417[4)].

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 of the film following the time of the test.

4) Three methods for measuring residual thiosulfate based chemicals in film are described in ISO 417. All three methods are considered 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 reliable for thiosulfate concentrations of 0,007 g/m^2. The methylene blue and iodine amylose methods measure thiosulfate ions only. They must be run within 2 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 2 weeks after processing. To determine thiosulfate levels accurately with this method, a calibration curve for the particular film is necessary.

Table 2 — Limits for thiosulfate (SO_2^{2-}) concentration

Film type	Film classification[1]	Maximum permissible concentration of thiosulfate[2] [3] g/m²
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

1) LE-500 film only applies to polyester base film.

2) Values are for each side of the film which has a photographic layer or a noncurl backing layer.

3) The concentration of thiosulfate is expressed in grams per square metre, which conforms to SI units. 0,010 g/m² = 1μg/cm²

7.5 Residual silver compounds

The processed film shall not show more than a barely perceptible tint when tested in accordance with 9.9 (see annex C).

8 Requirements for image stability

The specifications and test methods for image stability are different for the different product types. ISO visual diffuse density or status A blue density shall be measured on a densitometer which has spectral conformance to ISO 5-3 and geometric conformance to ISO 5-2. Processed film samples shall be incubated as described in 9.10.

8.1 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.

8.2 Microfilms

An area of minimum density and another having a visual diffuse density of 1,2 ± 0,1 on the processed film sample shall be tested. The criteria given in 8.2.1 and 8.2.2 shall apply to the different film categories.

8.2.1 LE-100 film

The area of minimum density shall have a visual diffuse density of less than 0,4 after incubation. The difference in visual density between the two test areas shall be at least 0,8 after incubation.

8.2.2 LE-500 film

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

8.3 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.

9 Test methods

9.1 Identification of film base

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