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Imaging materials — Thermally processed silver microfilm — Specifications for stability

Matériaux pour image — Microfilm à l'argent traité thermiquement — Spécifications pour la stabilité

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Contents

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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 18919 was prepared by Technical Committee ISO/TC 42, *Photography*.

This International Standard is one of a series of 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 (see annex B).

Annex A forms an integral part of this International Standard. Annexes B, C, D and E are for information only.

Introduction

Thermally processed silver (TPS) films are used widely for computer-output microfilming (COM) and for document recording. This International Standard is intended to provide the desired information on the stability of IPS images as well as other relevant properties of TPS microfilms. The basic elements of the TPS imaging process are also reviewed.

The first commercial TPS imaging product for the micrographic market was a photothermographic paper, called dry silver paper. It was introduced in 1964. This paper was designed for exposure by projection and for processing with a heated drum in a combination reader-printer processor. A TPS film based on this technology, but adopted for COM recording, followed in 1968. Since then, several other TPS-type films for computer-output and source-document microfilming have been introduced by several manufacturers. Special TPS products for other imaging applications have also been developed, including films for graphic arts and for duplication of aerial photographs, radiographic applications, as well as for line recording and remote sensing systems using laser beam and cathode-ray tube (CRT) imaging devices. However, these special products are not covered by this International Standard. It covers only the currently available TPS microfilms based on the present state of photothermographic technology.

The unique feature of TPS microfilm and its major advantage over conventional silver-gelatin products is its one-step, dry processing method. Another notable difference is that the image-forming components and, therefore, also the final silver image are dispersed in a non-gelatin binder, primarily [poly(vinylbutyral)]. This renders them inert to moisture and its deleterious effects. The support of TPS films is normal, photographic grade PET [poly(ethylene terephthalate)] safety film ([1], [2], [3], [4], [5], [6]).

In most contemporary TPS films, the metallic silver that forms the image is contributed by light-insensitive silver behenate salts that react with an incorporated reducing agent during heat development. This reaction is catalyzed by latent image silver formed during light exposure of silver halide crystals that are also incorporated in the imaging layer. Accordingly, the reaction occurs at a much higher rate in exposed than in unexposed areas, akin to the different rate of reduction of exposed and unexposed silver halide crystals by a chemical developer in a conventional photographic system.

Two important advantages offered by the TPS process include rapid, relatively simple and convenient dry processing and inertness to oxidation of silver images. These images are relatively stable, based on behaviour under normal user and storage conditions as well as on accelerated ageing studies. ([7], [8], [9]). Since TPS films are heat-processed by raising the temperature to between 119 °C and 125 °C, which is well above any expected use and recommended storage temperatures, <u>no chemical fixation is required</u>. Hence, TPS films do not fall within the provisions of ISO 10602 that apply to chemical fixation.

These attractive features should be weighed against the disadvantage that, in the TPS process, the residual image-forming components are not removed during processing. Therefore, the potential for formation of excessive fog exists throughout the life of the record; such fog may render the image unusable. This may occur during dark storage at elevated temperatures, or on prolonged exposure to ambient illumination, or especially on excessive exposure to light and heat in a reader-printer or to heat generated by a nearby fire. In the case of fire, the temperature inside a "fireproof" vault or safe can also rise to cause image degradation. Concerns with these possible causes of degradation have led to the adoption of considerably lower life expectancy ratings of TPS films in these specifications than indicated by accelerated ageing studies.

This International Standard includes all the requirements for the stability of wet-processed silver-gelatin films on safety bases, set forth in ISO 10602. They also include special thermal requirements applicable to TPS films and the requirement of at least ten duplications with a high-intensity mercury vapour lamp, stipulated for diazo and vesicular films. A few other relevant requirements for thermally processed vesicular films (ISO 9718) and ammonia processed diazo films (ISO 8225) are also included.

Imaging materials — Thermally processed silver microfilm — Specifications for stability

1 Scope

This International Standard establishes specifications for the stability of photographic films intended for storage of records; specifically, microfilms with a base of safety polyester [poly(ethylene terephthalate)] having predominantly silver behenate salts dispersed in nongelatinous emulsions, and thermally processed to produce a black-and-white silver image.

This International Standard applies to thermally processed silver (TPS) microfilms having ultrasonic or dielectric (induction-heated) splices. It does not cover films with splices made by means of adhesive tape.

This International Standard does not cover other types of black-and-white TPS films, black-and-white paper, colour images and colour prints that are produced with thermally processed silver behenate systems.

It does not apply to films to which lacquers have been applied.

It also does not apply to conventional black-and-white silver images that are produced by wet processing of silver-gelatin films (see ISO 10602).

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 5-2:1991, *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 543:1990, *Photography — Photographic films — Specifications for safety film*.

ISO 6077:1993, *Photography — Photographic films and papers — Wedge test for brittleness.*

ISO 7565:1993, *Micrographics — Readers for transparent microforms — Measurement of characteristics.*

ISO 8225:1995, *Photography — Ammonia-processed diazo photographic film — Specifications for stability.*

ISO 9718:1995, *Photography — Processed vesicular photographic film — Specifications for stability.*

ISO 10602:1995, *Photography — Processed silver-gelatin type black-and-white film — Specifications for stability.*

3 Terms and definitions

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

3.1
archival medium
recording material that can be expected to retain information for ever so that it 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
life expectancy
LE
length of time that information is predicted to be retrievable in a system under extended-term storage conditions

NOTE However, the actual useful life of film is very dependent upon the existing storage conditions (for example, see ISO 5466 [19] and ISO 10214) [21].

3.3
LE designation
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 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 a permanent value

3.5
medium-term storage conditions
storage conditions suitable for the preservation of recorded information for a minimum of ten years

3.6
film base
plastic support for the emulsion and backing layers

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

3.8
non-curl backing layer
layer, usually made of gelatin, applied to the side of the film base opposite to 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 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 poly(ethylene terephthalate) base
polyester film base composed mainly of a polymer of ethylene glycol and terephthalic acid

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 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 is indicated by the word "DANGER" followed by a symbol consisting of angle brackets "⟨⟩" containing a letter which designates the specific hazard. A double bracket "⟨⟨⟩⟩" is 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) is displayed. Furthermore, for a given material, the hazard symbol is 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 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.

The flammable warning signal ⟨F⟩ shall not be used for quantities of common solvents under 1 litre.

4.3 Safety precautions

All pipette operations shall be performed with a pipette bulb or plunger pipette. This is a critical safety warning. Safety glasses shall be worn for all laboratory work.

5 Requirements for the film base

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

6 Requirements for the thermally processed silver microfilm

6.1 Safety film

The film shall meet the requirements specified in ISO 543.

6.2 Amount of free acid

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. The amount of free acid shall be measured in accordance with the procedure described in 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 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 percentages specified in Table 1 (heated film).

Table 1 — Limits for tensile properties and loss in tensile properties on accelerated ageing of polyester-base film

Film type	Tensile stress at break	Elongation at break
Unheated film Minimum permissible tensile properties	140 MPa [1)]	75 %
Heated film Maximum permissible loss in tensile properties compared with unheated film	15 %	30 %
1) 1 MPa = 10^6 N/m^2		

7 Requirements for the emulsion and backing layers of thermally processed silver microfilm

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 Binder stability

The processed film shall not exceed a 1 mm increase in brittleness after testing as described in 9.7.

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.

8 Requirements for image stability

8.1 General requirements

International Standard (ISO) visual diffuse density or Status A blue diffuse transmission density shall be measured on a densitometer that has spectral conformance to ISO 5-3, and geometric conformance to ISO 5-2. In order to be classified as an LE-100 film, the processed microfilm shall meet the requirements of both the microfilm-reader test and the dark-ageing test described in 8.2 and 8.3 respectively.

8.2 Image stability: Microfilm-reader test

Status A blue diffuse transmission density patches with high-density and low-density levels (see Table 2) of the processed film shall be tested in a microfilm reader as described in 9.9. After testing for 1 h at 70 °C, the low-density patch shall not gain more than 0,4 density units. The difference between the high-density and the low-density patches, before and after testing shall be 0,8 or greater. This test simulates the simultaneous effects of heat and light on thermally processed silver films that are discussed in annex C.

Table 2 — Limits for changes in image density and contrast retention for the microfilm reader test

Parameters	Status A blue diffuse transmission density
Original density levels[1)]	
$D_{\text{low, orig}}$	⩽ 0,4
$D_{\text{high, orig}}$	⩾ 1,2
Net gain in low density, delta D_{low}[2)]	⩽ 0,4
Net contrast retention, delta D	
$D_{\text{high, final}} - D_{\text{low, final}}$	⩾ 0,8

1) The original density levels suggested for the low-density and high-density patches reflect a minimum contrast of at least 0,8 density units to perform this test. A Status A blue diffuse transmission density contrast of thermally processed microfilm is generally 1,6 ± 0,1 units with a D_{low} of approximately 0,2 ± 0,05 units.

2) The final density levels shall be determined with the same densitometer after exposure for 1 h at 70 °C in the microfilm reader.

8.3 Image stability: Dark-ageing test

Four microfilm specimens from the processed film samples shall be tested for dark ageing as described in 9.10, after ten additional exposures in a vesicular or diazo duplicator. After ten exposures in the duplicator, each specimen shall have an area of minimum density of ⩽ 0,4 and an area of high density of ⩾ 1,2. The following criteria shall apply.

LE-100 film: Neither the minimum-density nor the high-density area shall change by more than ± 0,1 in Status A blue diffuse transmission density units after incubation.

9 Test methods

9.1 Identification of film base

Remove all emulsion and backing layers from a sample of the unknown film by scraping. Then remove all sublayers by scraping. Prepare a sample 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 sample 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 sample with about 100 times its mass of potassium bromide, previously ground to about 75 µm. Prepare a strip or pellet as described in reference [10].

Obtain an infrared absorption curve from the prepared strip or pellet by means of an infrared absorption spectrometer. By comparing the infrared absorption curve for the unknown with curves for known polymers, the identity of the unknown can be established (see reference [11]).

9.2 Accelerated ageing conditions

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

The test specimens shall be conditioned at 23 °C ± 2 °C and (50 ± 2) % relative humidity for at least 15 h. After conditioning, place the specimens in a moisture-proof envelope and heat-seal the envelope[1)]. To prevent sticking between adjacent specimens, it may be necessary to interleave them with aluminum foil. 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 sample. Heat the envelopes in an oven for 72 h at 100 °C ± 2 °C[2)].

An alternative method of incubating the specimens in a closed environment is by placing them in 25 mm borosilicate glass tubes. Each tube shall have two flanged sections separated by a gasket to provide a moisture seal[3)], 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 In the subsequent text, samples subjected to these accelerated ageing conditions are designated "heated film". Comparison samples kept under room conditions are designated "unheated film".

Since these are thermally processed silver films, significant differences in appearance due to increase in image density will be noticed between unheated film and heated film specimens.

9.3 Determination of the amount of free acid

9.3.1 Specimen preparation

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 9.2. Remove all coatings from the film base by scraping. Cut each specimen into small pieces and accurately weigh the specimen prior to dissolving it in the appropriate solvents.

9.3.2 Solution preparation

Immerse each specimen in 30 ml of a 70/30 (*m/m*) mixture of purified *o*-cresol/chloroform (DANGER: ⟨B⟩ ⟨C⟩ ⟨S⟩).

WARNING — Chloroform is harmful if inhaled. Avoid breathing vapour, mist or gas. Use with adequate ventilation. If inhaled, move to fresh air. Contact should be avoided between chloroform and the eyes, skin or clothing. If contact occurs, obtain medical attention immediately.

***o*-cresol is toxic if swallowed. Contact should be avoided between *o*-cresol and the eyes, skin or clothing. Wash after handling. In case of contact, flush eyes and skin thoroughly with water and obtain medical attention immediately.**

The use of chloroform and *o*-cresol shall conform to all applicable national and local regulations.

Take care to dispose of chloroform and *o*-cresol in accordance with national and local regulations for hazardous waste disposal.

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

2) Incubation is accomplished in a closed environment to prevent escape of any decomposition products that may be produced during incubation. Such products may catalyse further degradation of the film base.

3) A suitable inert gasket may be made from polytetrafluoroethylene.

Dissolve the polyester support by heating it at 93 °C ± 2 °C for 30 min or until the specimen has dissolved. Precautions shall be taken to prevent excessive evaporation of the solvent. Cool the dissolved specimen to room temperature.

9.3.3 Titration

Titrate the polyester solution potentiometrically with standardized 0,1 mol/l tetrabutylammonium hydroxide using an automatic recording titrimeter and a glass/calomel electrode system. The electrodes shall have been preconditioned for 24 h in the *o*-cresol/chloroform solvent mixture (⟨B⟩ ⟨C⟩ ⟨S⟩) to prevent excessive instrumentation noise.

During titration, the burette tip shall be immersed in the solution as far as possible, and shall also be as far from the electrodes as practical. The stirring rate shall be as rapid as can be maintained without causing bubbles.

Also titrate 30 ml of a blank solution which has been heated for the same length of time as the polyester solution. Details of preparation of the standardized tetrabutylammonium hydroxide are given in annex A.

9.3.4 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:

$$A = \frac{c_T (V_S - V_B)}{0{,}1m}$$

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.

9.4 Tensile property strength for processed film

9.4.1 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 the unheated film and five specimens for the heated film.

The specimens to be heated and the 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.

9.4.2 Accelerated ageing

Five specimens shall be subjected to accelerated ageing as described in 9.2.

9.4.3 Conditioning

All specimens, both unheated and heated, shall be conditioned at 23 °C ± 2 °C and at (50 ± 2) % relative humidity for at least 15 h. This can be accomplished by means of an air-conditioned room or an air-conditioned cabinet. The specimens shall be supported in such a way as to permit free circulation of the air around the film and the linear air velocity shall be at least 150 mm/s.