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Photography — Processed vesicular photographic film — Specifications for stability

Photographie — Film photographique vésiculaire traité — Prescriptions pour assurer 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 9718 was prepared by Technical Committee ISO/TC 42, *Photography*.

Annexes A to G 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 records on film by national, state, and municipal governments, by banks, insurance companies, industry, and other enterprises, has been stimulated by a recognition of the resultant economies in storage space, organization, accessibility, and ease of reproduction. The safe-keeping of pictorial film records having legal, scientific, industrial, medical, historical, military, or other values has also become increasingly important.

The use of film for records having a long-term value necessitated the development of International Standards to specify the characteristics of film suitable for this purpose, and ISO 4331 and ISO 4332 have been written specifiying the requirements for silver-gelatin films which have archival keeping qualities. Archival films have been defined as those suitable for the preservation of records having permanent value. To date, only silver-gelatin type film has been specified as meeting the requirements for archival records.

However, many users of photographic film are not interested in permanence but in film usability after extended periods of time. Accordingly, two additional film categories have been defined: "medium-term" and "long-term" film. The establishment of these two additional categories increases the utility of International Standards to a wider spectrum of interested users. ISO 8225 has been written incorporating these categories in a specification for diazo photographic film.

Criteria for properties of medium-term and long-term vesicular films are based upon the dark-ageing stability of D_{min} processed areas. Different dark-incubation tests are specified for medium-term and long-term film but all other properties and processing requirements are identical.

In addition to tests ensuring that the density of D_{min} areas does not increase to unacceptable levels during storage, a test is also specified on high-density areas. This is to guard against the possibility of vesicle (or bubble) collapse during storage. This test has to be carried out at temperatures below the softening point of the image binder as tests above this temperature have no practical meaning.[1, 2] However, to give confidence of acceptable image stability, the permissible density change was made very small, within the measurement error of the densitometer. Both medium-term and long-term vesicular films must meet the same requirement.

It is recognized that vesicular images may show density changes after exposure to light. However, this International Standard covers only films used as storage copies, not as work copies (as defined in annex B). The light-fading tests in this International Standard ensure satisfactory behaviour for storage copies which are not intended to be subjected to frequent light exposure.

In addition to the characterization of films with respect to their expected storage life, vesicular films are also separated into two classifications which are dependent upon their intended use. Class A films are those which must have density in both the visual and the actinic regions after storage. Such films can be viewed directly or reprinted

on to ultraviolet (UV)-sensitive materials. However, some films are manufactured which are not intended to be reprinted on to UV-sensitive materials. Such films require only visual capabilities after storage and these are designated as Class B films. Obviously, both Class A and Class B films can fall into the medium-term and long-term categories. The properties and processing requirements for Class A and Class B films are identical, with the exception of change in the D_{min} area after dark incubation and after light fading.

Everyone concerned with the preservation of records on photographic film should realize that specifying the chemical and physical characteristics of the material does not, by itself, assure satisfactory behaviour. It is also essential to provide proper storage temperature and humidity and protection from the hazards of fire, water, light and certain atmospheric pollutants. Conditions for the storage of record films are specified in ISO 5466.

Photography — Processed vesicular photographic film — Specifications for stability

1 Scope

1.1 This International Standard specifies requirements for stability of safety polyester base photographic films which have a heat-processed vesicular photographic image formed by nitrogen bubbles.

1.2 The photographic films covered by this International Standard are those intended for medium-term and long-term records.

1.3 This International Standard applies to photographic film in which the image layer is a discrete layer attached to a transparent support.

1.4 This International Standard applies to roll film and sheet film.

1.5 This International Standard characterizes only the inherent keeping behaviour of the film covered. However, the suitability of a film record after extended storage depends on both the inherent ageing characteristics of the film and the original image quality. The latter is discussed in annex A.

1.6 This International Standard applies only to photographic vesicular film intended and used as medium-term and long-term storage copies. It does not apply to vesicular film records intended and used as "work" or "use" copies as discussed in annex B. Most film records used in libraries are work copies and must be durable. Medium-term and long-term storage copies must be stored in accordance with ISO 5466. The effects of heat and pressure are discussed in annex C and of high humidity in annex D.

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 : 1985, *Photography — Density measurements — Part 2: Geometric conditions for transmission density.*

ISO 5-3 : 1984, *Photography — Density measurements — Part 3: Spectral conditions.*

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

ISO 4331 : 1986, *Photography — Processed photographic black-and-white film for archival records — Silver-gelatin type on cellulose ester base — Specifications.*

ISO 4332 : 1986, *Photography — Processed photographic black-and-white film for archival records — Silver-gelatin type on poly(ethylene terephthalate) base — Specifications.*

ISO 5466 : 1986, *Photography — Processed safety photographic film — Storage practices.*

ISO 6077 : 1980, *Photography — Determination of brittleness of photographic film — Wedge brittleness test.*

ISO 7830 : 1983, *Photography — Safety photographic films other than motion picture films — Material specifications.*

ISO 8225 : 1987, *Photography — Ammonia processed diazo photographic film — Specification for stability.*

3 Definitions

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

3.1 medium-term film: A photographic film which is suitable for the preservation of records for a minimum of 10 years when stored under "medium-term" conditions, provided that the original images are of suitable quality.

3.2 long-term film: A photographic film which is suitable for the preservation of records for a minimum of 100 years when stored under "optimum" conditions, provided that the original images are of suitable quality.

3.3 archival film: A photographic film which is suitable for the preservation of records having permanent value when stored under "optimum" conditions, provided that the original images are of suitable quality.

NOTE — At present, only silver-gelatin films as specified in ISO 4331 and ISO 4332 are approved for archival records.

3.4 Class A films: Films which are usable both visually and for printing on to ultraviolet-sensitive materials.

3.5 Class B films: Films which are usable visually but do not have any density requirements for printing on to ultraviolet-sensitive materials.

3.6 film base: The plastic support for the image layers.

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

3.8 safety photographic film: Film that meets the specifications with respect to hazard from fire as defined in ISO 7830.

3.9 medium-term storage conditions: Those storage conditions suitable for ensuring a minimum useful life of 10 years for medium-term films.

3.10 optimum storage conditions: Those storage conditions suitable for the preservation of photographic film having permanent value.

NOTE — Optimum storage conditions will prolong the useful life of both archival and non-archival films.

3.11 transmission density: The radiant energy absorbing quality of a photographic image. It is expressed as the co-logarithm of the transmittance factor determined for specified geometric and spectral conditions (see ISO 5).

3.12 visual transmission density: A density measurement meeting spectral requirements specified in ISO 5-3 for visual density. It is necessary also to describe the geometric conditions of measurement to be specific.

3.13 printing transmission density: A density measurement of a spectrally non-selective film which will produce the same response on the print material as the film measured (see ISO 5-3). The contact printing density of a film specimen is equal to the transmission density of a spectrally non-sensitive modulator when they both produce the same response on the print material when contact printed together.

3.14 projection density: A density measurement in which the angular distributions of the incident and transmitted radiant flux are equal and specified.

For microfilm applications, the angular distribution is a nominal half-angle of 6,4°, which corresponds to an f-number of $f/4,5$ and simulates a microfilm reader.

For motion-picture applications, the nominal half-angle is 18,2°, which corresponds to an f-number of $f/1,6$ and simulates a projector.

4 Physical requirements

4.1 Film base type

The base used for vesicular film shall be safety poly(ethylene terephthalate) type and can be identified by the method specified in 7.1.

4.2 Viscosity retention

The relative viscosity of a solution of film base obtained from processed film shall not show a loss which exceeds 5 % as the result of accelerated ageing of the processed film. The accelerated ageing shall be accomplished as specified in 6.2 and the viscosity determined as specified in 7.2.

4.3 Safety characteristics

The film shall meet the requirements of ISO 7830.

4.4 Base physical property loss

Processed film shall have a tensile strength and elongation at break as specified in line 1 of table 1 when tested as specified in 7.3. The loss in tensile properties after accelerated ageing, as specified in 6.2, shall not exceed the percentage specified in line 2 of table 1.

Table 1 — Limits for tensile properties and tensile properties loss of poly(ethylene terephthalate) base film

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

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

4.5 Layer adhesion

4.5.1 Tape-stripping emulsion adhesion

There shall be no removal of the processed image layer when the film is tested before and after accelerated ageing. The accelerated ageing shall be accomplished as specified in 6.2, and the tape stripping test shall be performed as specified in 7.4.

4.5.2 Humidity-cycling emulsion adhesion

The processed image layer shall show no separation or cracking which would impair its intended use when tested as specified in 7.5.

4.6 Blocking

Processed film shall show no evidence of blocking (sticking), delamination, or surface damage when tested before and after accelerated ageing, as specified in 6.2. Blocking shall be tested as specified in 7.6.

A slight sticking of the film specimens which does not result in physical damage or a change in the gloss of the surface shall be acceptable.

4.7 Binder stability

Processed and imaged film shall not exceed a 1 mm increase in brittleness after accelerated ageing, as specified in 6.2. Brittleness shall be determined at 50 % relative humidity in accordance with ISO 6077. Films shall be tested preferably in low-density areas.

4.8 Thermal sticking

Processed film shall show no evidence of blocking (sticking), delamination or surface damage at high temperature when tested before and after accelerated ageing, as specified in 6.2. Thermal sticking shall be tested as specified in 7.7.

A slight sticking of film to glass which does not result in physical damage shall be acceptable.

5 Image requirements

5.1 Proper development

Processed film shall not show a projection density decrease greater than 20 % when tested as specified in 8.2.

5.2 Residual diazonium salt test

Processed film shall not show a density decrease greater than 0,1 when tested as specified in 8.3.

5.3 Image stability — Light fading

Low-density and high-density patches (see table 2) of the processed film shall be tested in a light exposure apparatus as specified in 8.4. After testing, the low printing and projection density patch shall be 0,7 or less. The difference between the high and low printing density patches shall be 0,8 or greater and between the high and low projection density patches shall be 1,4 or greater (see table 2). These denstiy requirements shall apply to both projection and printing densities for Class A films and to projection densities only for Class B films (see annex E). The same density requirements shall apply for both medium-term and long-term film.

Table 2 — Limits for image density change after light fading test (see 8.4)[1)]

Vesicular density levels	Printing density	Projection density
Original levels		
D_{low}	⩽ 0,4	⩽ 0,4
$\Delta D = (D_{high} - D_{low})$	⩾ 0,8	⩾ 1,4
Final levels[2)]		
D_{low}	⩽ 0,7	⩽ 0,7
$\Delta D = (D_{high} - D_{low})$	⩾ 0,8	⩾ 1,4

1) These requirements apply to both projection and printing densities for Class A films and to projection densities only for Class B films.

2) The same density requirements shall apply for both medium-term and long-term film.

5.4 Image stability — Dark ageing of minimum density area

Minimum density patches of the processed film shall be incubated as specified in 8.5 using the two conditions specified for either medium-term or long-term films. After incubation at each of the two conditions, the low printing and projection density patches shall be 0,6 or less. These density requirements shall apply to both projection and printing densities for Class A films and to projection density only for Class B films.

5.5 Image stability — Dark ageing of vesicular image

A density patch having a projection density of 2,0 shall be incubated as specified in 8.6. After incubation, the projection density shall not show a density change greater than ± 0,03. This density requirement shall apply for both medium-term and long-term film.

6 Accelerated ageing

6.1 Application

Processed film shall be subjected to the following accelerated ageing conditions when determining whether or not the film will meet the requirements specified in clause 4 for viscosity retention, base physical property loss, tape-stripping emulsion adhesion, blocking, binder stability and thermal sticking.

6.2 Accelerated ageing conditions

Mount the processed test specimens in a sample rack so that they are freely exposed to the surrounding air. Place the rack in a glass laboratory desiccator jar. Care shall be taken to prevent contact of the specimens with each other or with the walls of the jar.

Heat the jar in a forced-air circulating oven for 72 h at 100 °C ± 2 °C. The atmosphere within the jar shall be maintained at 20 % relative humidity. This relative humidity can be obtained by keeping a saturated solution of potassium acetate in water in the bottom of the jar.[1] Care shall be exercised to ensure that the saturated solution contains an excess of undissolved crystals at 100 °C. The undissolved crystals shall be completely covered by the saturated solution and the surface area of the solution should be as large as practical.

Maintain the jar and the salt solution at 100 °C for at least 20 h prior to use, to ensure adequate equilibrium.

Alternatively, exposure to these conditions of temperature and humidity may be provided by means of a conditioning air cabinet. The specimens shall be suspended to keep them separated from each other. The air cabinet shall be controlled to (20 ± 2) % relative humidity. No other materials shall be in the same environment as the specimens during the heating period.

Specimens subjected to these accelerated ageing conditions are hereafter designated "heated specimens".

7 Physical test methods

7.1 Identification of film base

Remove all emulsion, backing and sublayers from a specimen of the unknown film by scraping. Prepare a sample of the base material by scuffing the surface with a suitable tool to produce a very fine powder. Mix this powder in a mortar with about 100 times its mass of potassium bromide previously ground to about 200 mesh. Prepare a strip or pellet.[3] Obtain an infra-red absorption curve from this pellet by means of an infra-red absorption spectrometer. Establish the identity of the unknown base by comparison with curves for known polymers.[4]

7.2 Relative viscosity test

Measurements shall be made on the base of two unheated specimens of processed film and two samples that were subjected to accelerated ageing as specified in 6.2. Remove the emulsion and any backing layers by scraping before proceeding with the relative viscosity determination. The samples of base without coatings shall have a mass of 1,00 g each.

Dissolve the film base in approximately 95 ml of a mixture of 60 % (m/m) phenol and 40 % (m/m) chlorobenzene.

WARNING — The mixture of phenol and chlorobenzene should be prepared by stirring the molten phenol into the chlorobenzene. This mixture may be rapidly absorbed through the skin and can cause severe burns. In case of contact, wash the area with water for at least 15 min. Solvent mixture should be used only with adequate ventilation and hot solutions should be used only in a hood.

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

Immerse the flasks containing the dissolved film base in a water bath maintained at 25 °C ± 1 °C until temperature equilibrium has been reached. Add solvent to adjust the volume of the solution to 100 ml, and thoroughly mix the contents. Transfer a portion of the liquid, by filtration (see note) if necessary, to an Ostwald pipette or an equally suitable capillary viscometer immersed in a constant temperature bath at the same temperature.

NOTE — Filtration must be rapid to avoid solvent loss. This may be accomplished by filtering through a porous glass-wool pad.

WARNING — Solutions must not be drawn into pipettes by mouth. Use vacuum or water pumps.

The pipette chosen shall have a flow time between 70 s and 110 s for the solvent. The volume taken shall be sufficient to half-fill the lower bulb of the pipette. Measure the time of flow of the solution through the capillary of the pipette to the nearest 0,2 s. Repeat the measurement for the same volume of the pure solvent. Make three readings for each portion. The relative viscosity is the ratio of the average flow time of the solution to that of the solvent. Duplicate determinations shall be made on both the unheated and heated specimens and the averages shall be calculated separately.

7.3 Base physical property loss

7.3.1 Specimen preparation

Processed film in 16 mm form may be tested in this width. Films in other sizes shall be cut 15 mm to 16 mm wide and at least 120 mm long, using a sharp tool which does not nick the edges of the sample. Ten specimens are required for unheated film and ten specimens for film heated as specified in 6.2. The unheated and heated specimens shall be cut alternately and continuously from a single piece of film.

7.3.2 Conditioning

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

7.3.3 Procedure

The film specimens shall not be removed from the conditioning atmosphere for testing. Test the unheated and heated specimens alternately, and calculate the averages separately.

Determine the tensile strength and percentage elongation at break as specified in ISO 1184. Measure the thickness of each specimen to the nearest 0,002 mm and the width to the nearest 0,1 mm.

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

7.4 Tape-stripping adhesion test

7.4.1 Specimen preparation

Although the dimensions of the processed film specimen are not critical, one dimension shall be approximately 150 mm. Measurements shall be made on two specimens of unheated, processed film and on two heated specimens.

7.4.2 Conditioning

Condition specimens as specified in 7.3.2.

7.4.3 Procedure

7.4.3.1 The film specimens shall not be removed from the conditioning atmosphere for testing.

Apply a strip of pressure-sensitive, plastic-base, adhesive tape about 150 mm long to the image 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 beyond the edges of the film or cover areas of the film perforations.

While holding the film firmly on a flat surface, rapidly remove the tape from the film surface be 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 as failure.

7.4.3.2 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 of 4 N per centimetre of tape width is required.

Determine the bonding force by applying the adhesive tape to the film surface, in the same manner as specified in 7.4.3.1, and peeling it back rapidly from the film surface at an angle of approximately 180°. Measure the peel-back force required to separate the tape from the film by use of a strain gauge or maximum-reading spring scale.

7.5 Humidity-cycling adhesion test

7.5.1 Specimen preparation

A specimen 50 mm square or 50 mm by the film width is convenient, but the dimensions are not critical. Two specimens of processed film shall be selected from a high-density area.

7.5.2 Procedure

Mount the specimens in a rack and place them in a glass laboratory desiccator jar so that they are freely exposed to the atmosphere. Place the jar for 8 h in a forced-air circulating oven maintained at 50 °C ± 2 °C. The atmosphere within the jar shall be maintained at 96 % relative humidity, which can be obtained by keeping a saturated solution of potassium sulphate in water[5] in the bottom of the jar.[1)] After 8 h, place the rack for 16 h in a second desiccator jar in the same oven. The atmosphere within this second jar shall be maintained at 11 % relative humidity, which can be obtained by keeping a saturated solution of lithium chloride in water[5] in the bottom of the jar.[1)] The precautions specified in 6.2 shall be taken to ensure that the proper humidity is obtained.

Times of 8 h at the high humidity and 16 h at the low humidity shall constitute one cycle.

NOTE 1 — This can be most easily accomplished by placing the specimens in the 96 % relative humidity jar in the morning and the 11 % relative humidity jar in the evening.

During a weekend interruption in the cycling procedure, the film shall be kept at 50 °C ± 2 °C and 11 % relative humidity.

Subject the film to 12 humidity cycles, then remove it from the rack and examine the image for peeling, flaking or cracking.

NOTE 2 — Films may sometimes exhibit small pinholes in the image processing. These can be caused by dirt or dust particles on the surface at the time raw film is exposed and should not be confused with holes or cracks in the image layer. The existence of such pinholes in the image prior to humidity cycling should be noted so that their presence does not lead to false interpretation of an adhesion weakness.

Examine the film under the same magnification and lighting conditions as normal for the end use of the product.

7.6 Blocking test

7.6.1 Specimen preparation

At least five specimens of processed film shall be conditioned to 62 % relative humidity at 40 °C ± 2 °C. A specimen size of 50 mm square is convenient where the size of the film permits, but the dimensions are not critical provided that all specimens are of uniform size.

7.6.2 Procedure

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 °C ± 2 °C and about 62 % relative humidity. A relative humidity of approximately 62 % can be obtained by keeping a saturated solution of sodium nitrite[6] in water in the bottom of the jar.[1)] The precautions specified in 6.2 shall be taken to ensure that the proper humidity is obtained.

After moisture equilibrium is attained, remove the jar from the oven. Without removing the specimens from the jar, stack at least five specimens on a smooth surface so that the image

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