INTERNATIONAL STANDARD

ISO 8225

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Photography — Ammonia-processed diazo photographic film — Specifications for stability

Photographie — Film photographique diazoïque traité à l'ammoniac — Spécifications relatives à la stabilité



ISO 8225:1995(E)

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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 8225 was prepared by Technical Committee ISO/TC 42, *Photography*.

This second edition cancels and replaces the first edition (ISO 8225:1987), which has been technically revised.

Annex A forms an integral part of this International Standard. Annexes B, C, D and E 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 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 long-term values necessitated the development of International Standards to specify the characteristics of film suitable for this purpose. ISO 10602 specifies the requirements for silvergelatin films which are suitable for storage. This International Standard (for diazo film) and ISO 9718 (for vesicular film) give the requirements for photographic duplicate films suitable for storage.

The term "archival film" has been discontinued and the new concept of "life expectancy" is introduced. Film life is classified by the LE or life expectancy rating as defined in this International Standard. For example, LE-100 represents film with a life expectancy of 100 years when stored under extended-term storage conditions specified in ISO 5466.

Criteria for properties of LE-10 and LE-100 diazo films are based upon the dark-ageing stability of diazo images. Different dark incubation tests are specified for LE-10 and LE-100 films. All other property and processing requirements for medium and long-term diazo films are identical.

It is recognized that diazo 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 C). The light-fading requirements specified 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, diazo films are also separated into two classes (A and B); these classes are dependent upon their intended use. Class A films are those which retain density in both the visual and actinic region (printing) after storage. Such films can be viewed directly or reprinted onto ultraviolet (UV)-sensitive materials. However, some diazo films are not intended to be reprinted onto UV-sensitive materials. Such films require only visual capabilities after storage and are designated as Class B films. Obviously, both Class A and Class B films can fall into the LE-10 and LE-100 categories. The requirements for Class A and Class B films are identical, with the exception of image-stability tests after dark-ageing 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 the correct 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 and ISO 10214.

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International Organization for Standardization

Photography — Ammonia-processed diazo photographic film — Specifications for stability

1 Scope

- **1.1** This International Standard establishes specifications for the stability of polyester-base safety film which has an ammonia-processed diazo photographic image. It covers photographic film intended for LE-10 and LE-100 records.
- **1.2** This International Standard applies to photographic film in which the image layer is a discrete layer attached to a transparent support.
- **1.3** It applies to roll film and sheet film.
- **1.4** This International Standard characterizes only the inherent keeping behaviour of the film. 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 B.
- **1.5** This International Standard applies only to diazo photographic film intended and used as LE-10 and LE-100 storage copies. Storage copies should be stored in accordance with ISO 5466 and ISO 10214. It does not apply to diazo film records intended and used as "work" or "use" copies (as discussed in annex C).

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 5-3:1995, 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 5466:1992, Photography — Processed safety photographic films — Storage practices.

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

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

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

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

3 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 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):** 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:** 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:** 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 Class A films:** Films which are usable both visually and for printing onto ultraviolet-sensitive materials.
- **3.9 Class B films:** Films which are usable visually but do not have any density requirements for printing onto ultraviolet-sensitive materials.

- **3.10 safety photographic film:** Photographic film which passes the ignition time test and burning time test as specified in ISO 543.
- **3.11 safety poly(ethylene terephthalate) base:** Film base composed mainly of a polymer of ethylene glycol and terephthalic acid.
- **3.12 density:** Degree of light absorption, reflection or scattering characteristics of a photographic image, expressed as the logarithm to the base 10 of the ratio of incident radiant flux to the transmitted, reflected or scattered flux. (See ISO 5-3.)
- **3.13 printing density:** Density of a processed photographic image in which the incident and transmitted radiant flux are evaluated by a receiver having the same spectral response as the photographic material on which the sample is to be printed, and the incident radiant flux has the same spectral energy distribution as the printing light source.
- **3.14 visual diffuse density:** Density of a processed photographic image in which the incident and the transmitted or reflected radiant flux are evaluated by the human eye, or by a receiver having the same spectral response as the human eye.
- **3.15 projection density:** Density of a processed photographic image in which the angular distributions of the incident and transmitted radiant flux are equal and specified.
- NOTE 4 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.

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 procedure 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 " $\langle \ \rangle$ " containing a letter which designates the specific hazard. A double bracket " $\langle \ \rangle$ " will be used for particu-

larly 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.
- $\langle\langle S\rangle\rangle$ 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 1).
- 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 base

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

Some films on polyester base can have a maximum LE rating of 500.

6 Requirements for processed film

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 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 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 percentage specified in table 1 (heated film).

¹⁾ 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 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 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 Blocking

Processed film shall show no evidence of blocking (sticking), delamination or surface damage when tested as described in 9.7. A slight sticking of the film samples that does not result in physical damage or a change in surface gloss shall be acceptable.

7.3 Binder stability

Processed film shall not exceed a 1 mm increase in brittleness after accelerated ageing as specified in 9.2. Brittleness shall be determined at 50 % relative humidity and shall be tested in accordance with ISO 6077. Films shall be tested preferably in low-density areas.

7.4 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 9.2. Thermal sticking shall be tested as specified in 9.8. A slight sticking of film to glass which does not result in physical damage shall be acceptable.

8 Requirements for image stability

8.1 Proper development

Processed film shall not show a visual diffuse transmission density decrease greater than 30 % when tested as specified in 10.2.

8.2 Image stability: Light-fading

Low-density and high-density patches of the processed film shall be tested in a light-exposure apparatus as described in 10.3. After testing, the low-density patches shall have a diffuse density of 0,4 or less, and the difference between the high-density and low-density patches shall be 0,8 or greater (see table 2). These density requirements shall apply to both visual and printing densities for Class A film and to visual density only for Class B films (see annex D). The same density requirements shall apply for both LE-10 and LE-100 films.

Table 2 — Limits for change in diffuse density of image after accelerated testing

Diazo density levels	LE-10 and LE-100 film	
Original Low density High density	0,10 ± 0,05 1,2 ± 0,1	
Final 1) Low density High density — low density	≤ 0,4 ≥ 0,8	

NOTE — These requirements apply to both visual and printing densities for Class A films and to visual density only for Class B films.

1) That is, after the light-fading test (see 10.3) or the dark-ageing test (see 10.4).

8.3 Image stability: Dark-ageing

Low-density and high-density patches of the processed film shall be incubated as specified in 10.4 under the conditions specified for either LE-10 or LE-100 film. After incubation, the low-density patch shall have a diffuse density of 0,4 or less, and the difference between the high-density and low-density patches shall be 0,8 or greater (see table 2). These density requirements shall apply to both visual and printing densities for Class A films and to visual density only for Class B films.

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 μ m. Prepare a strip or pellet as described in reference [1].

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 [2]).

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, binder stability and thermal sticking.

The test specimens shall be conditioned at (23 ± 1) °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 ²⁾. To prevent sticking between adjacent

specimens, it may be necessary to interleave them with aluminium 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 ± 2) °C ³.

An alternative method of incubating the specimens in a closed environment is by placing them in 25 mm borosilicate glass tubes (see reference [3]). Each tube shall have two flanged sections separated by a gasket to provide a moisture seal ⁴⁾ 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 5 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".

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 it prior to dissolving it in the appropriate solvents.

9.3.2 Solution preparation

Immerse the specimens in 30 ml of a 70/30 (m/m) mixture of purified o-cresol/chloroform (DANGER: $\langle B \rangle \langle C \rangle \langle S \rangle$).

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. In case of contact, 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. Obtain medical attention immediately.

²⁾ A suitable moisture-proof envelope is a metal foil bag that is coated on the inside with polyethylene for heat-sealing.

³⁾ Incubation is accomplished in a closed environment to prevent escape of any acid that may be produced during incubation. Such acid may catalyse further film base degradation.

⁴⁾ A suitable inert gasket can be made from polytetrafluoroethylene.

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

Dissolve the polyester support by heating it at (93 + 2) °C for 30 min or until the specimen has dissolved. Precautions should be taken to prevent excessive evaporation of the solvent. Cool the dissolved specimens 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 ($\langle B \rangle \langle C \rangle \langle S \rangle$) to prevent excessive instrumentation noise.

During titration, the burette tip shall be immersed into the solution as far as possible, and shall also be as far from the electrodes as is 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 the preparation of 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:

$$A = \frac{(V_{\rm S} - V_{\rm B})c_{\rm T}}{0.1m}$$

where

- is the volume, in millilitres, of titrant used V_{S} for the specimen;
- is the volume, in millilitres, of titrant used V_{B} for the blank:
- is the concentration, in moles per litre, of c_{T} the titrant;
- is the mass, in grams, of the specimen. m

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 test 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 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 \pm 1) °C and at (50 \pm 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 air around the film and the linear air velocity shall be at least 150 mm/s.

9.4.4 Procedure

The film specimens shall not be removed from the conditioning atmosphere for testing. The tensile stress and percent elongation at break of unheated and heated film specimens shall be tested alternately by means of a tensile machine, as specified in ISO 527-3.

The initial grip separation shall be 100 mm and the rate of grip separation shall be 50 mm/min. The tensile stress and elongation at break shall be calculated separately for the unheated and heated film.

9.5 Tape-stripping adhesion test

9.5.1 Specimen preparation

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

9.5.2 Conditioning

Specimens shall be conditioned as described in 9.4.3.

9.5.3 Procedure

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 surface of the processed film. Press the tape down with thumb pressure to ensure adequate contact, leaving enough tape at one end to grasp. No portion of the tape shall extend to the edges of the film specimens or extend to film perforations. Hold the specimen firmly on a flat surface and remove the tape rapidly from the film surface. This shall be accomplished by peeling the tape back on itself and pulling the end so that it is removed from the film at an angle of approximately 180°. Removal by the tape of any portion of the surface layer on any of the specimens shall be considered failure.

The results of the tape-stripping test may be very dependent upon the adhesive tape used if the bonding force between it and the particular film surface under test is not sufficiently high. For this reason, a minimum bonding force is specified for this test. This bonding force shall be determined by applying the adhesive tape to the film surface in the same manner as described in the tape-stripping test. The tape shall be rapidly peeled back from the film surface at an angle of approximately 180°. The peelback force required to separate the tape from the film shall be measured by a suitable device such as a strain gauge or spring scale capable of reading the maximum force used. A bonding force of at least 0,9 N per millimetre of tape width is required.

9.6 Humidity-cycling adhesion test

9.6.1 Specimen preparation

Two specimens of processed film shall be selected from an area of high density. The specimens should be $50~\text{mm} \times 50~\text{mm}$ or 50~mm long \times the film width where the size of the film permits, but the dimensions are not critical provided all specimens are of uniform size.

9.6.2 Procedure

Mount the test specimens in a specimen rack and place them in a glass laboratory desiccator jar in such a way that they are freely exposed to the required conditioning atmosphere. Place the jar in a forced-air circulating oven for 8 h at (50 ± 2) °C. The atmosphere within the jar shall be maintained at 96 % relative humidity, which can be obtained by keeping a saturated solution of potassium sulfate in water (see reference [4]) in the bottom of the jar 5). Take care to ensure that the saturated solution contains an excess of undissolved crystals at 50 °C. The undissolved crystals shall be completely covered by a layer of saturated salt solution and the surface area of the solution should be as large as practical. The jar and salt solution shall be kept at 50 °C for at least 20 h prior to use to ensure adequate equilibrium.

After 8 h, place the specimens and specimen rack for 16 h in a second desiccator jar that is also in the same oven. The atmosphere within the second jar shall be maintained at 11 % relative humidity, which can be obtained by keeping a saturated solution of lithium chloride in water (see reference [4]) in the bottom of the jar⁵).

Time periods of 8 h at the high humidity and 16 h at the low humidity constitute one cycle ⁶⁾. Each film specimen shall be subjected to 12 humidity cycles. After this, remove the film specimens from the specimen rack and examine the emulsion and any backing layer for any evidence of peeling, flaking or cracking produced as a result of the humidity-cycling treatment.

NOTE 6 Films may sometimes exhibit what appear to be small pinholes in the image after processing. These can be caused by dirt or dust particles on the emulsion surface at the time the raw film is exposed and should not be confused with holes or cracks in the emulsion layer. The existence of such clear spots in the image prior to humidity cycling should be noted so that their presence does not lead to a false interpretation of adhesion weakness.

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

⁵⁾ The relative humidity is based on the normal vapour pressure of the salt solution but the relative humidity tolerances cannot be specified.

⁶⁾ This can be most easily accomplished by placing the specimens in the 96 % relative humidity jar in the morning and in the 11 % relative humidity jar in the evening.

9.7 Blocking test

At least five specimens of processed film shall be conditioned at 62 % relative humidity and (40 + 2) °C. The preferred specimen size is $50 \text{ mm} \times 50 \text{ mm}$ but the dimensions are not critical provided all specimens are of uniform size. Place the specimens in a glass laboratory desiccator jar so that they are freely exposed to the required conditioning atmosphere for at least 15 h. Place the jar containing the specimens in a forced-air circulating oven at (40 ± 2) °C. A relative humidity of approximately 62 % can be obtained by keeping a saturated solution of sodium nitrite (see reference [5]) in water at the bottom of the jar 6). Take care to ensure that the saturated solution contains an excess of undissolved crystals at 40 °C. The undissolved crystals shall be completely covered by a layer of saturated salt solution and the surface area of the solution should be as large as is practical. The jar and salt solution shall be kept at 40 °C for at least 20 h prior to use to ensure adequate equilibrium.

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

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

Remove the film stack from the oven and allow it to cool. Individually remove the film specimens from the stack and observe each for evidence of film blocking (sticking) (see 7.2).

9.8 Thermal sticking test

Measurements shall be made on two unheated specimens of processed film and two heated specimens as specified in 9.2. A specimen 50 mm \times 50 mm is convenient, but the dimensions are not critical.

Place the specimen between two smooth uncoated glass plates that have dimensions slightly larger than the specimen, under a uniform pressure of 35 kPa. This can be accomplished by placing a weight on the upper glass plate, the dimensions of the weight being

greater than that of the specimen. Put each glass-plate/film sandwich in a forced-air circulating oven for 1 h at (65 \pm 2) °C.

Remove the glass-plate/film sandwich from the oven and allow it to cool. After removal of the glass plates, examine the film for evidence of blocking (sticking), film delamination and surface damage.

10 Image test methods

10.1 Densitometry

Image density shall be measured in accordance with 10.1.1 and 10.1.2.

10.1.1 Visual density

Visual diffuse transmission density shall be measured using a densitometer with the geometric requirements specified in ISO 5-2 and the spectral requirements specified in ISO 5-3.

10.1.2 Printing density

Printing transmission density shall be determined as specified for ISO Type 1 density in ISO 5-1, ISO 5-2 and ISO 5-3, designated as D_T (90° opal; S_H : \leq 10°; s_1).

10.2 Proper development test

The diazo film shall be aerated after development for at least 5 min at room temperature in the dark. A test patch of the processed diazo film having a visual diffuse density equal to or greater than 1 shall be read for printing density as specified in 10.1.2. Then place the sample at a distance of 100 mm from a 100 W, clear incandescent lamp. After exposure for 10 min with the lamp turned on, remove the sample.

Again read the density of the identified area for printing density. Express any density decrease as a percentage of the original density.

10.3 Image stability: Light-fading test

10.3.1 Specimens

Measurements shall be made on low-density and high-density areas of two specimens of the processed test film. Class A films shall be measured for visual density as specified in 10.1.1, and for printing density as specified in 10.1.2. Class B films shall be measured only for visual density. The low-density area shall be 0,10 \pm 0,05 and the high-density area shall be 1,2 \pm 0,1 (see table 2) (these densities are typical for diazo microfilm copy).

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10.3.2 Light-exposure apparatus

The light-exposure apparatus shall contain a carbon arc of the solenoid-activated type which operates on a potential of 120 V to 145 V at 15 A. The arc shall be enclosed in a borosilicate glass globe which filters out wavelengths below 275 nm. The film plane surface shall have an irradiance of 1,7 W/mm², 5 W/mm² and 2 W/mm² at wavelengths of 358 nm, 386 nm and 416 nm respectively, and 0,25 W/mm² for the rest of the spectrum 7).

10.3.3 Procedure

Insert the specimens in the light-exposure apparatus. Position the holders in the light-exposure apparatus so that the emulsion side of the specimens is facing the arc. Expose the film for 8 h with the light-exposure apparatus operating between 40 % and 50 % relative humidity and at a temperature between 50 °C and 55 °C. Measure the density values of the low-density and high-density areas using the appropriate methods specified in 10.1.

10.4 Image stability: Dark-ageing test

Measurements shall be made on low-density and high-density areas of two specimens of the processed test film. Class A film shall be tested using a pair of specimens measured for visual density as specified in 10.1.1, and another pair of specimens measured for

printing density as specified in 10.1.2. Class B films shall be tested using only one pair of specimens measured for visual density. The low-density area shall be 0,10 \pm 0,05 and the high-density area shall be 1,2 \pm 0,1 (see table 2) (these densities are typical for diazo microfilm copy).

Condition the film at (50 ± 2) % relative humidity as specified in 9.4.3. Seal the film in a moisture-proof, metallic-foil envelope. Heat LE-10 films for 14 days at (70 ± 2) °C and LE-100 films for 50 days at (80 ± 2) °C (see reference [6]). Measure the density values of the low-density and high-density areas using the appropriate methods specified in 10.1.

11 Classification for reporting

Ammonia-processed diazo films shall be classified as LE-10 or LE-100 film, depending upon their useful life. This is determined by conformance to the darkageing test requirements specified in 8.3. All other property requirements are the same for both classes.

Ammonia-processed diazo films shall be classified as Class A films if they meet visual and printing diffuse density requirements at the end of their projected useful life. They shall be classified as Class B films if they meet visual requirements only. This is determined by conformance to the light-fading test requirements specified in 8.2 and to the dark-ageing test requirements specified in 8.3. Other property requirements are the same for both classes.

⁷⁾ Details on the availability of such a carbon arc light source may be obtained from the Secretariat of ISO/TC 42. (Sources with equivalent emission may be used.)

Annex A

(normative)

Preparation of standard solution of tetrabutylammonium hydroxide

Polyester bases inherently have carboxyl groups incorporated in their molecular structure. In chemical terminology, this is referred to as "free acids" and is expressed as the volume of a standard solution of sodium hydroxide required to neutralize 1 g of polyester base. The intent of the test specified in 6.2 is to ensure that the base is chemically stable and also that it will not be adversely affected by the image-bearing or other layers. Thus, the entire film shall be heated in accordance with 9.2 to simulate longtime ageing. However, remove all the layers from the base prior to determining the acidity to simplify the analytical procedure.

Use a primary 0,1 mol/l standard solution of benzoic acid to standardize the tetrabutylammonium hydroxide (TBAH) titrant. Then titrate the TBAH against the polyester base to determine the amount of free acid. Prepare the nominal 0,1 mol/l primary standard solution of benzoic acid by weighing $(1,22 \pm 0,01)$ g of benzoic acid into a 100 ml volumetric flask. Make up to the mark with a 70/30 (m/m) mixture of purified o-cresol/chloroform (DANGER: $\langle B \rangle \langle C \rangle \langle S \rangle$).

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. Obtain medical attention immediately.

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

The concentration, c_P , in moles per litre, of the primary benzoic acid standard is calculated from:

$$c_{\mathsf{P}} = \frac{\mathsf{0.1} m_{\mathsf{B}}}{\mathsf{1.22}}$$

where $m_{\rm B}$ is the mass, in grams, of benzoic acid used.

Then use the primary standard to determine the molarity of the TBAH base titrant.

Prepare a 25 % (m/m) solution of TBAH by dissolving 25 g of TBAH in 75 g of water. Prepare a nominal 0,10 mol/l TBAH titrant solution by adding 90 ml of isopropanol to 10 ml of the 25 % (m/m) aqueous TBAH solution. Standardize this against the primary benzoic acid solution by pipetting exactly 1 ml of the 0,1 mol/l benzoic acid solution into a 50 ml beaker and adding 30 ml of the 70/30 (m/m) mixture of o-cresol/chloroform $(\langle B \rangle \langle C \rangle \langle S \rangle)$. Titrate this solution with TBAH base titrant using an automatic-recording titrimeter in the specified manner. Carry out a similar titration with a blank 30 ml sample of the o-cresol/chloroform mixture. Make both titrations in duplicate.

The concentration, c_T , in moles per litre, of the TBAH base titrant is calculated from:

$$c_{\mathsf{T}} = \frac{V_{\mathsf{P}} \cdot c_{\mathsf{P}}}{V_{\mathsf{S}} - V_{\mathsf{B}}}$$

where

- $V_{\rm P}$ is the volume, in millilitres, of the 0,1 mol/l benzoic acid solution;
- $c_{\rm P}$ is the concentration, in moles per litre, of the 0,1 mol/l benzoic acid solution;
- $V_{\rm S}$ is the volume, in millilitres, of TBAH titrant used in the titration of the primary standard;
- V_B is the volume, in millilitres, of TBAH titrant used in the titration of the blank.

EXAMPLE

If 1,00 ml of 0,102 mol/l primary standard is titrated with 1,147 ml of TBAH titrant and 30 ml of blank is titrated with 0,032 ml of TBAH titrant, then the concentration of TBAH is

$$c_{\rm T} = \frac{1,00 \times 0,102}{1,147 - 0.032} = 0,091 \text{mol/l}$$

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Annex B

(informative)

Microfilm image quality

B.1 General

If satisfactory image quality is to be retained after storage, the quality of the image to be stored is as important as the keeping characteristics of the stored film. Considerable attention and care should be taken to ensure that the original camera film is properly exposed and is then properly printed onto the duplicate film.

In establishing a microfilm system, three factors should be considered:

- quality of original document,
- exposure of document, and
- image contrast (see reference [7]).

B.2 Quality of original document

B.2.1 General

The three most important qualities of the original document related to image quality are

- a) the height of the characters;
- the stroke width forming the smallest significant character or line;
- c) for non-computer output microfilm, the contrast between the paper and ink.

B.2.2 Character height

The quality requirements needed to enable a microrecording system to record a character of a specific size in a document can be estimated by use of a "quality index". The quality index (QI) relates the reduction ratio and the resolving power of the system to the height of the smallest significant characters in the document in such a way that the level of legibility in the resulting images can be predetermined. The QI is described in reference [8]. In most duplicating processes, there is a loss of information with each succeeding generation. It is therefore essential that the QI of the camera film be high enough so that the QI of the next generation or generations, including hard-copy printouts, will be sufficient to provide images with adequate legibility for storage (see references [9, 10]).

B.2.3 Line or stroke width

Due to optical limitations in most photographic systems, film images of thin lines appearing in the original document will tend to fill in as a function of their width and density. Therefore, as the reduction ratio of a given system is increased, it may be necessary to reduce the background density to achieve an image with relatively low line density so that the copies produced will contain legible characters. No firm rule can, at this time, be written for this relationship.

B.2.4 Ink/paper contrast

With good clear black type on a white background, an image with a high-quality index and high contrast can be achieved at a high reduction ratio. As the original print quality and contrast decrease, the reduction ratio or background density, or both, must be decreased to achieve the same quality image unless the resolving power of the system is increased. Reference [9] gives recommended background densities for achieving satisfactory images.

B.3 Exposure of the document

B.3.1 General

The second important factor to be considered when photographing documents is exposure control (see references [11, 12]).

It is desirable that the contrast of all images in a single roll or microfiche have the same value to facilitate duplicating; i.e. to permit constant printing exposure and speed. Due to the wide variety of documents that must be filmed and the various methods by which

they are filmed, trade-offs must be made in controlling the exposure. Reference [9] gives recommended background densities for camera films based on the contrast of the original documents when using planetary cameras. The same recommendations also apply to rotary cameras. However, even with automatic exposure controls, the image densities from rotary cameras cannot be as well controlled. Computer output microfilmers use higher contrast films, resulting in more uniform and higher contrast images which facilitates duplicating (see references [10, 13-18]).

B.4 Image contrast

The third factor to be considered when photographing documents, the contrast of the micro-reproduction system (see reference [14]), is dependent upon the selection of proper photographic materials for use in each generation. This applies both to the exposure of the camera film and to subsequent printing onto the duplicating film.

The average individual who uses microfilm thinks of contrast as the magnitude of the difference in density between the light and dark areas in a microfilm image.

From the user's point of view, this is a logical interpretation of contrast. However, the quality of a microfilm depends on more than the two ends of the scale: it also depends on the ability of the photographic materials to differentiate between small differences in tones in the document. The characteristic curves of the photographic film define the contrast of the reproduction. Cascading of the characteristic curves of all films used in the system produces its tone-reproduction curve. In a microfilm system, the contrast of the tone-reproduction curve affects the quality of the microfilm reproduction. If the contrast in the image is too high, fine lines and light lines from the original document will tend to fill in and bold black lines from the same original will spread. Open areas in certain characters will also fill in. On the other hand if the contrast is too low, all the graphic information will be reproduced but the lines will appear unsharp and the reproduction will have a flat, muddy appearance.

Film manufacturers have made camera and print films with contrast designed to work effectively together. However, it is up to the user to select the proper films for use in a particular system.

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Annex C

(informative)

Distinction between film storage copies and work copies

The distinction between photographic film records which are intended for storage and those intended for use has not always been clear. Use or work copies are the predominant photographic records found in libraries and records centres. Their value lies in their being available for ready reference. However, as a result of this use, they are subjected to dirt, abrasion, fingerprints, contamination with foreign materials and exposure to excessive light and temperatures. Such use copies may become conditioned to the moisture conditions of the working area, which may be quite different from those of the storage area where they are filed. In fact, physical distortion of use copies can occur if they are not reconditioned to the moisture conditions of the storage area. It is evident that use copies of photographic records are not suitable for extended preservation.

Diazo film is generally used for the work copy of daily information management systems, although it also has found application for storage purposes. The useful life of work copies can be prolonged even in frequent usage by following the recommendations for material selection and handling given in this International Standard.

Where there is a need for extended storage of film records, duplicate storage copies should be made and kept apart in a separate location. Storage copies should meet the appropriate requirements for the photographic material used as outlined in this International Standard and its companion documents. ISO 10602 and ISO 9718. It is equally important that storage copies should be stored according to the recommendations of ISO 5466 and ISO 10214. Of vital importance is the control of temperature and humidity and the protection of the film from dirt and impurities. Unless the appropriate film materials are stored according to recommended procedures, satisfactory preservation will not be obtained. Storage records will occasionally be looked at, otherwise the keeping of these records is pointless. However, the use of storage copies should be infrequent. If more than infrequent use is required, duplicate work copies should be printed from the storage copies.

In conclusion, there are two types of film records: those intended for use purposes and those intended for the preservation of information. This International Standard pertains to the latter only.

International Organization for Standardization

Annex D

(informative)

Light-fading of diazo images

The light-fading test conditions of 8 h in a fadeometer, specified in 10.3, are based on experimental comparisons with practical reader tests. This fadeometer test shows similar image-density changes for a variety of diazo films as found after 3 h of exposure in a commercial reader-printer whose gate temperature corresponded to the maximum specified in some national standards and caused the film to be heated to approximately 65 °C.

The permitted density changes given in 8.2 will result in usable diazo images. It is recognized that diazo im-

ages do change on exposure to light and that use for more than 3 h might cause unacceptable images. However, this International Standard applies only to storage copies and not to work copies as described in 1.5 and annex C. While work copies may be subjected to substantial use during their useful life, storage copies should be used only infrequently. A maximum use of 3 h in a reader or printer seems reasonable over the 10-year life of an LE-10 film or the 100-year life of an LE-100 film.

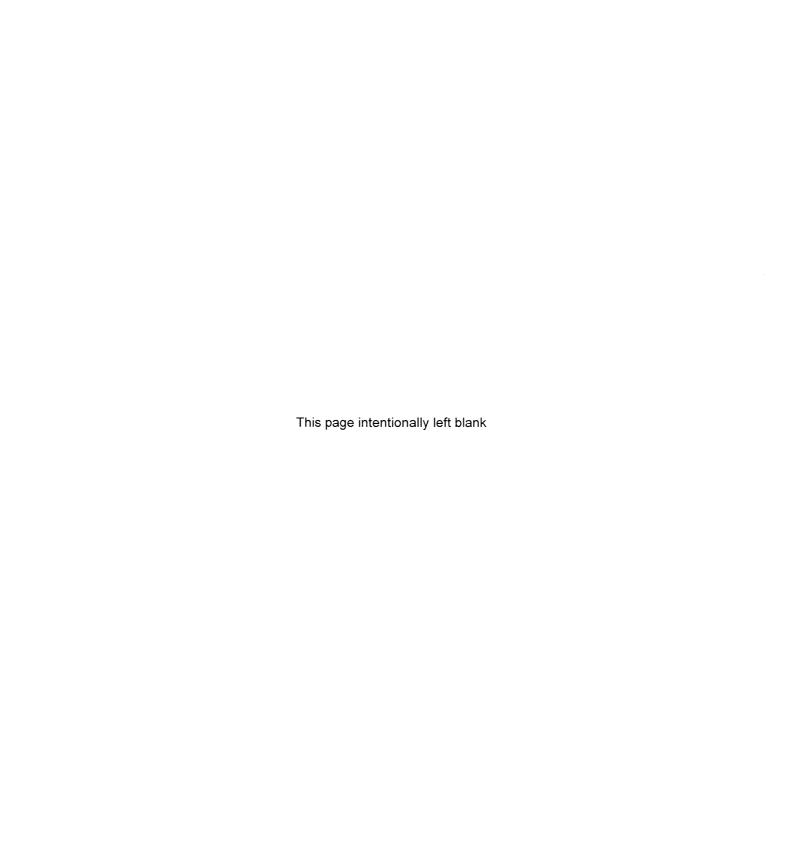
Annex E

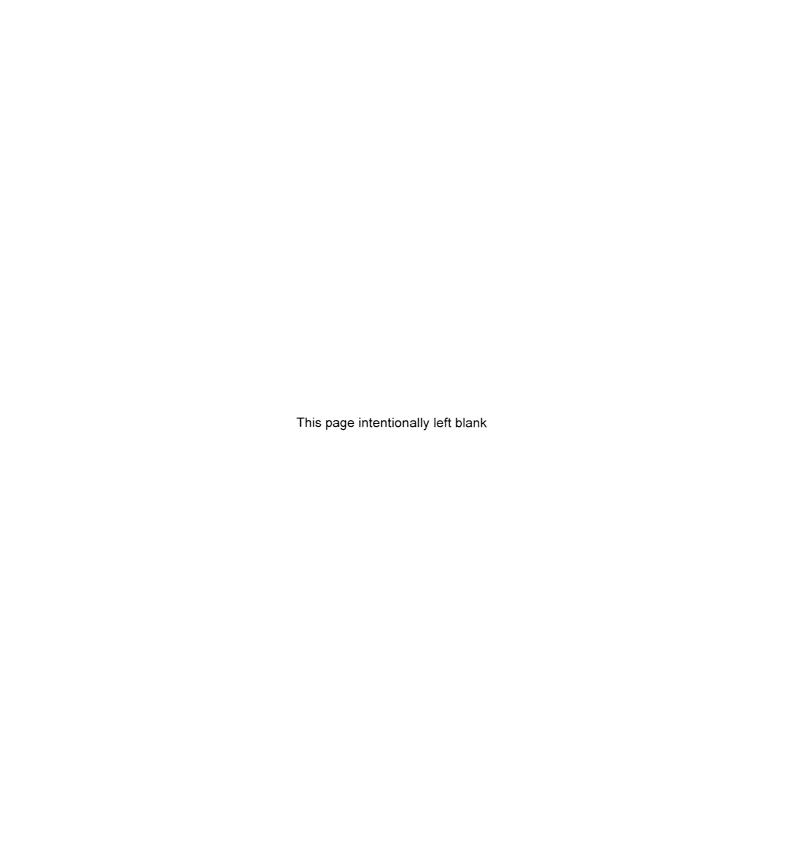
(informative)

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Descriptors: photography, photographic film, diazo film, exposed photographic film, specifications, stability, tests.

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