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Imaging materials — Wet-processed silver-gelatin type black-and-white photographic reflection prints — Specifications for dark storage

Matériaux pour l'image — Tirages photographiques par réflexion par traitement humide gélatinoargentique de type noir et blanc — Spécifications pour le stockage dans l'obscurité

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18929 was prepared by Technical Committee ISO/TC 42, *Photography*.

This International Standard is one of a series of International 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 A).

Introduction

Black-and-white (B & W) silver-gelatin fibre-base papers have been important imaging materials for well over 100 years. Reflection prints, made with such papers, have become increasingly important pictorial and documentary records that are housed in many libraries, archives, museums, universities and other public and private institutions. Although the intrinsic stability of properly processed and stored silver-gelatin prints is very good, there are internal and external factors that can cause degradation of such prints during storage and shorten their useful life. This International Standard provides specifications for tests that evaluate the stability of B & W silver-gelatin prints under prescribed storage conditions. It does not cover tests for the possible harmful effects of extended exposure to light.

B & W prints are laminates, whose main components are a paper base, a white opaque intermediate layer, an image layer, and a protective layer. Paper has been used as a support for prints since about 1840. The history of its evolution into a very stable, high alpha-cellulose type fibre-base in the 1920 s and to a polyethylene resin-coated (RC) paper base in the 1960s has been covered extensively in the literature (see [1], [2], [3], [4] and [5] in the Bibliography).

The intermediate, white opaque layer can be of two types. For fibre paper, it has been a gelatin layer (since the 1880s) containing white barium sulfate pigment (baryta layer) and other additives that enhance the paper's reflectivity, hardness, holdout, adhesion and surface characteristics (glossy, matte, textured). The second type came into use in the 1960s with the introduction of resin-coated (RC) base. This has a paper core coated on both sides with water-impermeable extruded polyethylene layers. The top layer contains white titanium dioxide pigment that provides higher reflectivity than barium sulfate; the bottom layer is transparent and formulated to counteract the curl induced by the emulsion, which is coated on top of the pigmented layer.

Prints on fibre paper have the advantage of having demonstrated long-term stability from years of practical experience in both dark storage and in reasonable display conditions. Proper processing and cleanliness or lack of contamination are important. Dilute toning solutions have been used successfully to enhance stability (see Annex B).

Prints on a RC paper base offer advantages of higher wet strength, reduction in washing and drying times, lower consumption of processing chemicals, better dimensional stability, lower curl, and freedom from cockle and other effects of differential shrinkage. As with fibre-base materials, toning has been used successfully to enhance stability. However they can suffer from stability-related problems such as light-induced image discoloration, silver mirroring, yellowing and cracking of the polyethylene layer (see [6], [7], [8] and [9] in the Bibliography).

This International Standard only applies to prints in dark storage since the ISO Working Group (WG) has not been able to reach consensus on an applicable test for light-induced image deterioration. One reason has been that different brands of RC papers or papers manufactured at different periods have exhibited widely varying stability characteristics. It has been very difficult to reconcile all of the observed differences in behaviour in a relatively simple standardized test. However, the ISO/WG continues to work on this problem (see Annex C).

The image layer of most B & W prints is a gelatin coating containing a dispersion of light-sensitive silver halide crystals that are selectively converted into metallic silver grains by an image-forming exposure and chemical processing. Gelatin is a natural polymer that has been the preferred binder for photographic materials since the 1880s, because it has proven to be the best protective colloid for the precipitation, growth and controlled suspension of silver crystals and for the formation of a dry, flexible image layer during a subsequent coating operation. Gelatin also can be hardened to reduce its swelling and susceptibility to physical damage without blocking the absorption and desorption of aqueous processing solutions required for image formation and removal or stabilization of chemical by-products (see [10] and [11] in the Bibliography).

Converting the metallic silver to a complex or pre-oxidized form of silver will improve image stability. Many toner solutions have been designed to accomplish this, such as selenium, sepia, gold, brown and poly-toners. For example, in the case of sepia toner treatment, the silver is converted from oxidizable metallic silver to a stable silver sulfide.

The storage stability and useful life of reflection B & W prints depends on their physical and chemical properties, as well as on the conditions under which they are stored. For many years, the term “archival medium” was used to designate a recording material that can be expected to retain information forever so that it can be retrieved without significant loss when properly stored. However, there is no such material and it is a term no longer employed for material or systems specifications of International Standards. Another reason for abandonment of the term is the multiplicity of meanings that it acquired over the years, ranging from “preserving information forever” to “temporary storage of actively used information”. The unambiguous term used in this International Standard is “life expectancy” (LE) defined as the length of time that information is predicted to be retrievable in a system after storage at 21 °C and 50 % relative humidity (RH). For example, a designation of LE-100 indicates that the information can be retrieved without significant loss after at least 100 years. Reflection prints are classified according to their LE designation in this International Standard.

Imaging materials — Wet-processed silver-gelatin type black-and-white photographic reflection prints — Specifications for dark storage

1 Scope

1.1 This International Standard establishes the specifications for silver-gelatin photographic reflection prints intended for dark storage and covers silver-gelatin print types of all weights.

1.2 This International Standard applies to wet-processed black-and-white silver-gelatin photographic prints; including those that have been chemically treated (with a gold, selenium, sulfur, or other chemical treatment bath) to improve the permanence of the silver image. It also applies to silver-gelatin prints processed by a monobath, which includes thiosulfate as a fixing agent followed by a conventional wash.

1.3 This International Standard is not applicable to instant black-and-white photographic prints, stabilisation-processed black-and-white prints (those where the undeveloped silver halide has been chemically converted and not removed), and thermally processed black-and-white prints. It does not apply to colour or dye-based photographic prints, nor to prints that have been processed by a monobath using other than a thiosulfate-type fixing solution, or prints where the silver salts are rendered more soluble by means other than thiosulfate.

Laminated and adhesive mounted reflection prints, prints on poly (ethylene terephthalate) support, and prints that have been displayed are also excluded from this International Standard.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 5-4:1995, *Photography — Density measurements — Part 4: Geometric conditions for reflection density*

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

ISO 18907:2000, *Imaging materials — Photographic films and papers — Wedge test for brittleness*

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

ISO 18920:2000, *Imaging materials — Processed photographic reflection prints — Storage practices*

TAPPI T494 om-88, *Tensile breaking properties of paper and paper board (using constant rate of elongation apparatus*

3 Terms and definitions

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

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

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

3.2
emulsion
image-forming layer of photographic materials

3.3
life expectancy
LE
length of time that information is predicted to be acceptable in a system after dark storage at 21 °C and 50 % RH

NOTE However, the actual useful life of black-and-white paper is very dependent upon the existing storage conditions (see ISO 18902 and ISO 18920).

3.4
LE designation
rating for the "life expectancy" of recording materials and associated retrieval systems

NOTE The number following the LE symbol is a prediction of the minimum life expectancy in years for which information can be retrieved without significant loss when stored at 21 °C and 50 % RH, for example, LE-100 indicates that the information can be retrieved after at least 100 years storage.

3.5
reflection print support
white, opaque support for the image forming and auxiliary layers of a print intended for viewing by reflected light

3.6
treated silver images
silver images that have been given a specific treatment, either during or after processing, to increase their stability

4 Safety and operational precautions

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, and in some cases other protective apparel, such as rubber gloves, face masks and aprons. 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 procedures, the hazard will be indicated by the word "**DANGER**" followed by a symbol consisting of angle brackets "〈 〉" containing a letter that designates the specific hazard. A double bracket "〈〈 〉〉" will be used for particularly perilous situations. In subsequent statements involving handling of these hazardous materials, only the hazard symbol consisting of the brackets and letter(s) will be displayed. Furthermore, for a given material, the hazard symbols will be used only once in a single paragraph.

Hazard warning symbols will not be used for common organic solvents when used in quantities of less than 1 litre, unless they are particularly hazardous.

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 code 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 obtains pertinent information from the manufacturer 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.

⟨F⟩ Will burn. Keep away from heat, sparks and open flame. Use with adequate ventilation.

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

⟨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.

4.3 Safety precautions

4.3.1 ALL PIPETTE OPERATIONS SHALL BE PERFORMED WITH A PIPETTE BULB OR PLUNGER PIPETTE. Failure to observe this warning notice can result in cyanide poisoning. THIS IS A CRITICAL SAFETY WARNING!

4.3.2 Digestion procedures shall be performed in an exhaust hood. Hydrogen cyanide or other toxic substances can be evolved.

4.3.3 Safety glasses shall be worn for all laboratory work.

4.3.4 Cyanogen iodide (CNI) may be detoxified by treatment with sodium hypochlorite.

5 Requirements for physical stability of prints

5.1 Wedge brittleness

Unincubated RC paper shall not show a paper crack value greater than 12 mm when tested as described in 8.1.4. Incubated RC paper shall not show an increase in the paper crack value greater than 14 mm when incubated as described in 8.1.2 and tested as described in 8.1.4.

The test is not run on unincubated fibre paper. Incubated fibre paper shall not show a crack value greater than 14 mm when incubated as described in 8.1.2 and tested as described in 8.1.4.

5.2 Tensile energy absorption (see Annex D)

Fibre paper and RC paper shall not show a decrease in tensile energy absorption (TEA) greater than 50 % when incubated as described in 8.2.2 and tested as described in 8.2.4.

5.3 Resin cracking of RC papers (see Annex E)

After 30 d, processed RC papers shall not show any evidence of resin cracking when tested as described in 8.6.5.

6 Requirements for processed image stability (see Annex F)

6.1 Residual thiosulfate concentration

Prints shall be fixed in solutions containing either sodium thiosulfate (hypo) or ammonium thiosulfate. After processing, fibre-base and RC-base B & W prints shall not contain more than 0,014 g/m^2 of residual thiosulfate [calculated as thiosulfate ions ($S_2O_3^{2-}$) when measured by the iodine amylose method in ISO 18917].

NOTE 1 The concentration of thiosulfate is expressed in grams per square metre (g/m^2), which conforms to SI units.

NOTE 2 0,010 g/m^2 = 1 µg/cm^2.

NOTE 3 A very low concentration of thiosulfate due to excessive washing may cause the silver image to be more susceptible to oxidative attack. These concentrations may be below the detection limit of ISO 18917.

Three methods for measuring residual thiosulfate levels are described in ISO 18917, i.e., the iodine amylose, methylene blue, and silver sulfide densitometric test methods. All of them are considered sufficiently reliable to determine thiosulfate concentrations at the 0,014 g/m^2 level, but only the iodine amylose method has been found to give reliable results with all types of fibre-base and RC prints. Therefore, it is the only method chosen for this International Standard.

The iodine amylose method measures only the concentration of thiosulfate ions. It can be run up to two weeks after processing, but the analysis must be carried out with eluent obtained from a minimum density area of the processed print. It is only in such areas that the thiosulfate concentration does not change appreciably over a two-week period.

The methylene blue method shall not be used, because it fails to detect residual thiosulfate in RC-base prints whose unexposed emulsion layers contain a considerable amount of developing agent in order to provide very rapid development during processing.

While the silver sulfide method does detect residual thiosulfate in RC-base prints, it also measures the concentration of polythionate decomposition products and other residual chemicals. Moreover, it also has been shown to give exaggeratedly high thiosulfate values with some RC prints (see [12] in the Bibliography).

Hypo-eliminating agents containing strongly oxidizing substances, such as peroxides or hypochlorites, shall not be used because they contain peroxides that attack the silver image (see Annexes F and G). However, the use of hypo-clearing solutions (such as a 2 % solution of sodium sulfite containing a wetting agent) does facilitate the washing of thiosulfate ions from the fibre prints without chemically altering the thiosulfate. They should not be used for RC prints.

6.2 Residual silver concentration

The processed print shall not contain more than 0,025 g/m^2 of silver when tested in accordance with 8.4.3 or 8.5.3 using the atomic absorption technique.

6.3 Yellowing limit (see Annex H)

The status A blue density of minimum density areas shall not increase by more than 0,08 units on the front (emulsion) surface or the back surface of the prints after incubation under conditions specified in 8.3.2 and tested as described in 8.3.3.

7 LE designation

Prints, that meet all the specifications set forth in this International Standard, shall have a LE rating of 100.

8 Test methods

8.1 Wedge brittleness

8.1.1 Preparation of specimens

Paper samples shall be exposed and processed to yield maximum density. Specimens shall be cut in an atmosphere of (23 ± 2) °C and (50 ± 5) % RH. The cutter shall be of a precision type and shall provide specimen edges that are smooth and free of nicks. Specimens shall be 300 mm long by 15 mm to 16 mm wide.

A set of 10 specimens shall be prepared for the original measurements and another set of 10 specimens for incubation as described in 8.1.2. The operator in handling the specimens, both in their preparation and testing, shall wear moisture-impermeable dust- and powder-free gloves.

NOTE Latex gloves may cause hypersensitivity in some individuals.

8.1.2 Incubation

One set of 10 specimens shall be freely suspended for 30 d in a forced-air oven maintained at (80 ± 2) °C and (50 ± 2) % RH. The short-term fluctuation in relative humidity shall not exceed ± 5 % RH.

8.1.3 Conditioning of specimens

Specimens shall be conditioned at (23 ± 1) °C and (20 ± 2) % RH before measurements. Fibre papers shall be conditioned for at least 1 d and RC papers for at least 7 d prior to measurement. The specimens shall be conditioned in racks that permit free circulation of air around the specimens.

8.1.4 Measurement

Specimens shall be tested at (23 ± 1) °C and (20 ± 2) % RH, as described in ISO 18907 using the wedge apparatus having a 74 mm opening.

8.1.5 Reporting

Specimens shall be evaluated for either paper cracks or paper breaks, depending on which occurs at the wider wedge opening. Specimens which pass through the narrowest point of the wedge (1,5 mm) without failure shall be arbitrarily assigned a value of 1,0 mm, to permit averaging with those specimens which show failure caused by paper cracks or paper breaks.

The average of 10 specimens shall be calculated for both unincubated and incubated specimens. Samples shall meet the criteria given in 5.1.

NOTE Paper cracks may be evident by white paper fibres showing through the emulsion layer processed to maximum density.

8.2 Tensile energy absorption

8.2.1 Preparation of specimens

Paper specimens shall be prepared as described in 8.1.1. A set of 20 specimens shall be prepared for the original measurements and another set of 20 specimens for incubation as described in 8.2.2.

8.2.2 Incubation

One set of 20 specimens shall be hung freely in a controlled forced-air oven maintained at (80 ± 2) °C and (50 ± 2) % RH for 30 d. The set-point accuracy for the RH shall be as described in 8.1.2.

8.2.3 Conditioning

Specimens shall be conditioned as in 8.1.3.

8.2.4 Measurement

Specimens shall be tested at (23 ± 1) °C and (50 ± 2) % RH for tensile energy absorption (TEA) as described in TAPPI T494 om-88.

8.2.5 Reporting

The average TEA of 20 specimens shall be calculated for both unincubated and incubated specimens. Samples shall meet the specification given in 5.2.

Attention shall be given to 6.10 of TAPPI T494 om-88 which pertains to the discarding of tensile measurements.

8.3 Yellowing

8.3.1 Preparation of specimens

Two minimum density specimens shall be prepared for each sample and conditioned as outlined in 8.1.3. The area of each specimen shall be at least 90 cm^2. To prevent contamination by fingerprints, the operator shall wear gloves during the preparation and testing of the specimens.

8.3.2 Incubation

Both specimens shall be freely suspended for 14 d in a forced-air oven maintained at (80 ± 2) °C and (50 ± 2) % RH. The short-term fluctuation in the test chamber shall not exceed ± 5 % RH.

8.3.3 Measurement

The status A blue reflection density of both specimens, backed by a black material, shall be measured before and after incubation (see Annexes H and I). The spectral characteristics of the densitometer shall conform to ISO 5-3, and its geometry to ISO 5-4. Four measurements shall be made of the front and of the back surfaces of the two specimens for a total of sixteen measurements.

8.3.4 Reporting

The average of eight measurements shall be calculated for the front (emulsion) surface and the back surface before and after incubation. The sample shall meet the density criteria specified in 6.3.